MULTIPLE-MECHANICAL-THERMAL TREATMENT OF AUSTENITIC STEEL

Volume 1 - Text

by

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The effect of multiple-mechanical-thermal-treatment (MMTT) on low- and high-temperature mechanical properties of niobium-stabilised austenitic steels has been studied. MMTT consisted of 2% plastic prestrain at room temperature of the solution treated material followed by ageing at 800°C for 4h to promote the precipitation of Nb(C,N). The 5-MMTT cycle material appears to give best combination of strength and ductility.

Electron microscopy (HVEM) studies have shown that MMTT introduces finely dispersed particles of Nb(C,N) which stabilise and support a comparatively high dislocation density. MMTT strengthening is, therefore, due to the combined contribution of dispersion and dislocation substructure hardening.

The low-temperature strength of the MMTT material was found to be inferior to the cold-worked material, because of extensive recovery occurring during the thermal stage of each MMTT cycle. The high temperature strength (hot tensile and creep) was, on the other hand, found to be appreciably superior for the MMTT material as compared with other comparable treatments. The substantially improved creep resistance is, however, accompanied by poor creep ductility, which has been correlated with void formation at precipitate particles and inclusions during the prestraining stage of each MMTT cycle.

Variable stress creep tests performed during this work revealed inconsistencies in recovery creep measurements and theoretical creep models based on the concept of internal stress. The results indicate that anelastic relaxation processes play a dominant role in such tests.
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Dedicated to my daughter Denise
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1. INTRODUCTION

Before embarking on the main theme of the thesis some general introductory remarks are necessary concerning its layout. This also provides a suitable opportunity to outline the aims and basic principles of the project and to illustrate the development of those concepts essential to its successful conclusion.

The nature of the project, being an attempt to understand the thermal and mechanical behaviour of a complex material subjected to equally complex microstructural manipulation, is not amenable to a conventional treatment. For this reason, the research has been conducted on a wide front, resulting in some degree of fragmentation. Consequently it has been necessary to adopt a rather different format than is usual, particularly with respect to the presentation of the literature review (Chapter 2). This is dictated by the fact that such widely ranging aspects of physical metallurgy as creep and tensile deformation, creep rupture, ductile fracture, precipitate nucleation and coarsening and thermal recovery have all figured prominently in the project. A comprehensive literature survey of the conventional type which successfully encompassed all these aspects would, for a thesis, be a task of prohibitive proportions and, indeed, would detract from the main theme of the research. An endeavour has, therefore, been made to present in the literature survey only those aspects which are fundamental to the main theme of the thesis. The very specific nature of many of the topics makes them more amenable to detailed discussion in the context of the experimental results at a later stage. Still in connection with the literature survey it should be pointed out that a review paper \(^1\) has been published recently by the author, under the supervision of, and in conjunction with, Dr. Z. C. Szkopiak, which covers many of the wide ranging aspects of dislocation substructures and their influence on mechanical properties. This, attached as APPENDIX 1, provides the basis of the first major section of the literature review and only those aspects requiring a more detailed treatment have been presented in Chapter 2.

The terminology used in the literature appears to be rather confusing and it
is appropriate that some comments should be made in an attempt to clarify the situation. Thermomechanical treatments, such as ausforming, have been developed mainly for transformation hardenable steels and this term has become generally applied to other types of treatment. The author has suggested that it would be useful to introduce a more specific classification which describes more clearly the processes involved. Basically, all these treatments derive their strengthening from combined working and heat-treating schedules, but it would be more informative to define them in such a way as to indicate the strengthening mechanism. Clearly, the ways in which these treatments can affect mechanical properties are complex and will vary from one alloy system to another. In pure metals and single phase alloys, which do not undergo phase transformations during treatment, the strengthening is derived solely from the deformation induced dislocation structure. This has been stabilised by some thermal (i.e. annealing) treatment, such as polygonization. Treatments of this type should logically be referred to as Mechanical-Thermal Treatments (MTT) and the mechanism referred to as dislocation substructure strengthening. At the other end of the spectrum are the transformation hardenable alloys, such as steels, in which the major strengthening is derived from the phase transformation. In these circumstances the deformation prior to or during transformation produces a superior distribution of the phases and the term Thermomechanical Treatment (TMT) is more descriptive of the strengthening process. The latter applies to ausforming, in which deformation of metastable austenite, prior to the martensite transformation, is employed to modify the martensitic structure and its subsequent tempering characteristics. Other treatments are more difficult to define as for example isoforming. In the latter it has been shown that, while the deformation has a dramatic effect on the carbide distribution, the main source of strengthening appears to be the fine stable subgrain structure which accompanies this. Similar comments apply to controlled rolling of stainless steels. The latter, like the strengthening of T.D. Nickel by working-annealing treatments, are more conveniently classified as MTT, which is also true of the other treatments.
considered in APPENDIX 1.

Of these treatments, the one with which this research is concerned has been termed Multiple-Mechanical-Thermal Treatment (MMTT). First proposed for bcc metals and alloys, MMTT consisted of the following schedule. The material was plastically deformed to the end of the Lüders strain and then aged at a temperature high enough to allow rapid diffusion of interstitial atoms to dislocations (i.e. strain ageing). This constituted one cycle of MMTT and was repeated up to six cycles in iron and nine cycles in niobium. MMTT appears to confer superior properties, as compared with conventional strain ageing treatments, due to the more uniform dislocation substructure that results. A thorough review of work in this field will be given in Chapter 2.

Because of the proven benefits to be derived from MMTT it was decided to attempt this type of treatment for an fcc alloy, in which the conventional strain ageing processes do not occur. A system was sought in which there was commercial interest and which exhibited dislocation induced precipitation. After an extensive literature survey, which encompassed such considerations as stacking fault energy, precipitation mode and stability, a niobium stabilized austenitic stainless steel was selected as described later. The form of MMTT schedule which should be employed received a considerable amount of attention prior to experimental work. A major program of deformation and ageing studies were performed during this early preparation stage, in order to fill gaps in our knowledge of the material and to check certain assumptions.

Basically the aim of the project as defined in the early stages was to attempt to produce by MMTT a dislocation structure consisting of a uniform network supported by a fine and equally uniform NbC dispersion. A high temperature (1200° - 1300°C) solution treatment, to dissolve a large concentration of niobium and carbon/nitrogen, was to be followed by some form of MMTT, the purpose of which was to introduce dislocations into the structure and lock them by precipitating NbC. In the absence of a clearly defined optimum strain, like the Lüders strain in bcc
alloys, it was arbitrarily decided to use low strains (~2%) at room temperature. It was subsequently decided to investigate the effect of the degree of strain in each MMTT cycle as well as the temperature of prestrain.

The ageing conditions during each MMTT cycle again presented a variable which it was desirable to eliminate if at all possible. Once again it was decided to make what might at first sight appear to be an arbitrary choice of conditions, though in fact one based quite firmly in science. If a regular three dimensional dislocation network is to be stabilized by second phase particles, it has been shown \(^{20}\) that the particle density \((N_v)\) or mean particle size \((d)\) and volume fraction \((f)\) of spherical particles is related to the mean dislocation spacing \((x)\) by

\[
x - d \sqrt{\pi/6f} \sim N_v^{-1/3}
\]

Similarly the mean dislocation spacing in a three dimensional network is given by \(^{21}\)

\[
x = \beta \rho_d^{-1/2}
\]

where \(\rho_d\) is the dislocation density and \(\beta\) a geometrical constant of about unity. It therefore follows that, to stabilise a dislocation density of \(10^{10} \text{ cm}^{-2}\), particles should be about 0.1 \(\mu\)m apart. Such a structure should possess superior recovery resistance and consequently high creep deformation resistance. Since MMTT was to involve room temperature deformation and elevated temperature ageing, a certain degree of recovery could be expected during the first several MMTT cycles. A criterion was therefore sought which might help in determining the optimum ageing conditions without having to resort to a protracted empirical approach.

To this end a very simple model was produced. This is illustrated schematically in Fig. 1-1. It is assumed that precipitates only nucleate at dislocations and that during precipitation each precipitate drains a spherical volume of radius \((r)\) given by

\[
r = \sqrt{D t}
\]
where \( D \) is the rate controlling diffusion coefficient for NbC precipitation and \( t \) the time. As a rough approximation the average dislocation spacing required is 0.1 \( \mu m \) and, therefore, as shown in Fig. 1-1, the radius of the necessary solute depleted zone should be set equal to half the dislocation spacing (i.e. 0.05 \( \mu m \)).

It then follows that during MMTT a newly introduced dislocation lying within the sphere of influence of one already pinned (region A) is unlikely to become locked due to the solute depletion in such regions. If, however, a newly introduced dislocation lies outside the sphere of influence of others (e.g. at B) it may then be locked. In this way a regular dislocation network might be built up after a few cycles of MMTT. Using diffusion data for niobium\(^{22,23}\) which is likely to be rate controlling, a time of about \( \frac{1}{4} h \) ageing per cycle would be necessary at 800\( ^\circ \)C for a regular dislocation distribution and density of \( 10^{10} \) cm\(^{-2} \). The conditions to be used during each cycle of MMTT were then as follows:

(i) 2\% deformation in tension at room temperature followed by

(ii) ageing for \( \frac{1}{4} h \) at 800\( ^\circ \)C.

While the degree of deformation was subsequently varied the ageing conditions were maintained approximately constant as regards \( Dt \), such that at 700\( ^\circ \)C and 750\( ^\circ \)C the ageing times were 10 and 2\( h \) respectively.

Returning once more to the aims of this project it is possible to define several, in addition to the prime motivation of investigating the effectiveness of MMTT as a strengthening process in fcc alloys. This obviously entailed cataloguing the low and high temperature properties and comparing the behaviour with that of other treatments, or their equivalent, which have been used for materials of this type in the past. In addition it was thought desirable to investigate the way the MTTT dislocation structure built up as a function of the number of cycles and to see how this related to the simple model proposed earlier. In the same vein, the contribution of dislocations and precipitates to strengthening were studied and a considerable amount of effort was directed towards developing analytical techniques for this purpose. The assistance of Professor Y. Ishida in
performing some of these rather tedious and time consuming analyses and helpful discussions is gratefully acknowledged. It was also hoped to study the effect of annealing and creep on the MMTT structure, thereby obtaining essential information concerning the nature and magnitude of stabilization of dislocation structures by second phase particles. In common with most researches of this type each step forward produced unexpected effects which were studied in some detail to obtain as wide as possible understanding of all facets of MMTT. These include various aspects of ductile fracture, deformation, ageing and variable stress creep behaviour. The reasons for this will become clear in the relevant sections of the results and discussion.
2. LITERATURE REVIEW

The wide ranging nature of this project has necessitated a slightly modified form of literature review from that traditionally adopted. In the following sections only those topics which bear directly on MMTT and the behaviour of the material studied, austenitic stainless steel, will be dealt with. This should provide the relevant background material to this project. All other aspects such as, for example, creep mechanisms and electron diffraction contrast theory will be presented together with the relevant experimental work and discussion of results. The necessary background material on dislocation substructures and other MTT is contained in Appendix 1.1

2.1 Multiple-Mechanical-Thermal Treatment (MMTT)

A series of mechanical-thermal treatments have been developed recently based on combined deformation and annealing schedules. These are designed to produce materials with a high dislocation density and a uniform and more stable substructure. From such treatments significant improvements in strength and toughness have resulted, which compliment those achieved by traditional alloying and heat treatment methods. This field has been reviewed recently by a number of authors1,24–26. We will return to a discussion of these with specific reference to austenitic stainless steels a little later.

One of these treatments, based on repeated deformation and ageing cycles, was developed for materials exhibiting a discontinuous yield point and low temperature (0.2 Tm) strain ageing phenomena. This, now usually referred to as multiple-mechanical-thermal treatment (MMTT), was developed by Oding, Ivanova and Gordienko9 specifically for materials which strain age, such as low alloy ferritic steels. MMTT appears to have its foundations in the observations, by Bundy and Boxall27, that, during multi-pass wire drawing operations, significant improvements in strength could be achieved, if strain ageing was permitted between passes. This could arise either from inadequate cooling during drawing or from ambient temperature ageing during protracted inter-pass periods. Such observations are numerous, being the familiar phenomenon of stretcher strains
in industrial rolling and drawing operations. These undesirable surface markings may be eliminated by temper rolling, roll levelling or suitable alloying. This whole field of strain ageing in steels has been exhaustively reviewed by Baird and in a wider range of metals and alloys by Hall. The original contribution of Hundy and Boxall was their observation that greater strengthening was achieved by interrupted strain-ageing than by a single cycle for an equivalent total strain and ageing time. This point is fundamental to MMTT.

It is not therefore surprising that MMTT has taken the following form for iron alloys:

(i) Deformation in tension to a degree corresponding to the end of the Lüders strain.

(ii) Ageing at an elevated temperature ($100^\circ - 200^\circ$C) for a period of time corresponding to efficient solute pinning of dislocations.

(iii) This constitutes one cycle of MMTT.

(iv) Repeated cycles of deformation and ageing, as for (i) and (ii), could be applied until the required strength was achieved or the available ductility was exhausted.

In most work ageing has been carried out at $100^\circ$C for 1h in the unloaded condition, which gives adequate strengthening, as well as being a very reproducible temperature. Strengthening has, however, been shown to be a function of grain size, temperature and time of ageing, applied stress during ageing and strain-rate. The deformation in each cycle was almost invariably slightly in excess of the Lüders strain in order to guarantee that the gauge length was completely exhausted of Lüders bands. In fact the most beneficial results appear to have been obtained by ensuring that this excess strain is kept to a minimum. Since the Lüders corresponds to the smallest uniform strain ($\sim 3\%$) that may be applied to alloys exhibiting discontinuous yielding behaviour, the more uniform dislocation structures generated at low prestrains are apparently to be preferred. This point should be stressed as it seems to be fundamental to MMTT strengthening.
The typical variation of stress with strain during MMTT for an armco iron is illustrated in Fig. 2-1. Ageing (100°C, 1 hr) was carried out during each unloading part of the cycle. The step-wise increase in lower yield stress with increasing number of MMTT cycles is apparent. After four MMTT cycles, a final (fifth) straining was performed, this time to fracture. During this final test to failure, it is interesting to point out that the metal yielded at a stress approximately equal to its ultimate tensile strength (UTS). Little or no work hardening occurred prior to plastic instability (necking) and fracture.

For convenience the results of MMTT studies relating to iron-carbon alloys, in which the treatments were very similar, will be considered first. It is in these alloys that the majority of work has been done. The results for niobium alloys\textsuperscript{18,32} will be discussed separately in a later section (2.1.2).

2.1.1 Iron Alloys

2.1.1.1 Room Temperature Tensile Behaviour

A considerable quantity of data, of the type shown in Fig. 2-1, has now been amassed regarding the variation of tensile properties of low carbon steels during MMTT\textsuperscript{9-13,15-17,19}. Here, only some of the more recent work\textsuperscript{15-17,19} will be considered in order to illustrate the effect of structural and treatment variables on MMTT strengthening.

Gordienko et al\textsuperscript{16} have studied the effect of ageing temperature (Fig. 2-2) and strain rate (Fig. 2-3) on the lower yield stress ($\sigma_y$), ultimate tensile stress (UTS) and elongation for an Fe/0.06%C alloy. These parameters are shown as a function of the number of MMTT cycles. In both cases the strength increases continuously with MMTT while the ductility decreases. Increasing the strain rate during MMTT prestraining, while increasing the strength, has little detectable effect on the rate of strengthening (Fig. 2-2). However, by increasing the ageing temperature, in the range 100°C - 200°C, the strengthening rate is observed to increase quite significantly, particularly between 150°C and 200°C (Fig. 2-3). A common feature of all these results is the observation that, at between 4 and 6 MMTT cycles, the degree of strengthening reaches a level such that the values of
\( \sigma_L \) and UTS are effectively equal, as illustrated in Fig. 2-2. Further MMTT cycling is possible beyond this point and further strengthening may be obtained. It is, however, difficult to specify the Luders strain closely, due to the fact that deformation proceeds at the UTS and failure processes are undoubtedly occurring. Working with a similar alloy Gordienko et al.\(^{17}\) observed a comparable strain rate effect. Ageing temperature variations have been shown to give rise to a peak strengthening at \( \approx 150^\circ\text{C} \) for Fe - 0.03\%C\(^{19}\) and at \( \approx 250^\circ\text{C} \) in a 0.3\%C low alloy Cr-Mo-Mn steel\(^{12}\). In the latter material strengths of up to 1400 MPa were achieved. Ageing under a residual or relaxed stress has been found to give rise to increases in both level and rate of strengthening\(^{15}\). A similar effect has been observed during conventional strain ageing of iron alloys\(^{33,34}\). In all these treatments the ductility decreases with number of cycles and under conditions where the strengthening rate increases the ductility exhibits a concomitant decrease.

The degree of prestrain per cycle during MMTT is not constant. The Luders strain varies from \( \approx 2.5\% \) in the as annealed condition to a pronounced minimum of \( \approx 1.0\% \) in the third cycle after which it increases to \( \approx 3.0\% \) during the fifth cycle. A strong strain-rate dependence of this effect has been observed\(^{17}\). The minimum occurs at lower numbers of cycles (and the minimum value increases) with increasing strain rate. The Luders strain has been observed to decrease with increasing prestrain by rolling during conventional strain ageing\(^{35}\), to about 4\%, after which it reaches a fairly constant value. An increase in Luders strain after about 3 cycles of MMTT presumably reflects the low UTS/\( \sigma_L \) ratio (\( \approx 1.0\)).

Further work has been performed by Gordienko\(^{31}\) to correlate the effect of grain size on MMTT in Fe/0.04\%C. The results of this work showed good agreement with the Hall-Petch relation\(^ {36}\)

\[ \sigma_L = \sigma_i + K \frac{1}{d} \]  \( \ldots \) (2.1)

where \( \sigma_i \) is the lattice friction stress, \( K \) a parameter related to the degree of dislocation locking or grain boundary barrier efficiency and \( d \) the average grain
The total MMTT prestrain (Ee) was found to affect $\sigma_i$ and $K_y$ in the manner shown in Fig. 2-4. It is apparent that $\sigma_i$ increases continuously during MMTT while $K_y$ is a rather more complex function of Ee.

### 2.1.1.2 Fatigue Properties

The fatigue properties of armco iron and a low carbon steel (0.15%C) have been studied by Oding et al\(^9\) and Ivanova and Terentyev\(^10\). Significant improvements were gained in both fatigue limit and fatigue strength. For example, 4MMTT cycles\(^10\) gave an improvement of 30% in fatigue limit and an increase of greater than an order of magnitude in service life in the overload region, as compared with annealed material. The fatigue limit and strength increased with number of MMTT cycles.

### 2.1.1.3 Fracture Toughness

The fracture toughness parameters $G_{ic}$, the energy dissipated per unit length of crack growth and $K_{ic}$, the stress intensity factor were determined as a function of the number of MMTT cycles by Ivanova et al\(^14\) for armco iron. Cylindrical, notched tensile specimens were used in this investigation. While open to criticism, their measurements reflect the relative changes in toughness, though the absolute values of $G_{ic}$ and $K_{ic}$ are in doubt. Fig. 2-5 shows the results of this investigation. It is apparent from the figure that both parameters increase during MMTT to 4 cycles, after which an abrupt deterioration occurs. This indicates a 100% improvement in $G_{ic}$ after 4 cycles as compared with the annealed condition. It also shows that, while further strengthening may be achieved by applying more cycles, the optimum degree of strength and toughness are acquired after 4 MMTT cycles.

### 2.1.1.4 Creep Properties

Creep tests have been performed, at 400°C, on armco iron and a low carbon (0.1%) steel\(^9,11\), after MMTT. These tests indicate that the minimum creep rate was reduced by about an order of magnitude for Armco iron after 5 cycles of MMTT as compared to the annealed condition. MMTT of the low carbon steel, to 5 cycles, reduced the minimum creep rate by about 3 orders of magnitude. These improvements
in creep rate illustrate the significant success of MMTT in creating a structure which has good thermal recovery resistance. The stabilizing effect of carbides present in the low carbon steel is also evident. Although no creep data is available, annealing studies\textsuperscript{37} indicate that temperatures in excess of $450^\circ$C would lead to significant losses of creep resistance, due to the 'boiling off' of interstitial atmospheres and coarse carbide precipitation which accompany subgrain formation.

2.1.1.5 Microstructural Studies

The structural changes occurring during MMTT have been studied by both optical\textsuperscript{10,14} and electron microscopy\textsuperscript{17,19,37}. The structures were compared wherever possible with those observed after equivalent strains obtained in normal tensile loading (direct straining).

Optical studies after direct straining and MMTT, in armco iron\textsuperscript{14} and low carbon steel\textsuperscript{10} have revealed that surface slip markings and etch pits are more finely and uniformly distributed after MMTT. This tendency appeared to persist to high strains in the necked region of tensile specimens and at the root of propagating fatigue cracks\textsuperscript{14}. This finer, more uniform dislocation distribution was proposed\textsuperscript{14} as the source of improvements in fracture toughness and fatigue.

Electron microscopy studies\textsuperscript{17,19,37} have provided a clearer and more quantitative description of the structural processes occurring during MMTT as well as providing useful information regarding the strengthening mechanism. To illustrate the typical structures observed, four electron micrographs have been chosen. These are shown in Fig. 2-6 (a) and 2-7 (a) representing the structures after 3 MMTT ($\varepsilon = 7.12\%$) and 4 MMTT ($\varepsilon = 10.0\%$) cycles, respectively. It was a \textsuperscript{1} to attempt to compare MMTT structures with those obtained by direct tensile deformation to an equivalent strain. Structures observed after 7.8 and 11.15\% strain are shown in Fig. 2-6 (b) and 2-7 (b), respectively. It is apparent from Fig. 2-6 that at 7.8\% strain (Fig. 2-6 (b)) diffuse tangles and a grid of fairly straight dislocations is produced at low strains\textsuperscript{1}. By 11.15\% strain (Fig. 2-7 (b))
cell structure has formed consisting of continuous locally dense walls of dislocation between which there are relatively dislocation free areas. This is typical of sub-structures formed at strains in excess of 10% in iron \(^{1,38,39}\) at room temperature. After 3 and 4 MMTT cycles (Fig. 2-6 (a) and 2-7 (a), respectively) dense dislocation tangles and a relatively regular interconnecting network of straight dislocations was observed. The density and number of tangles increased with number of cycles and the background network coarsened. Several conclusions are evident from these observations:

(i) The dislocation density is considerably higher after MMTT

(ii) The density of tangles is higher after MMTT

(iii) No distinct cell structure was observed up to 5 MMTT cycles (\(\varepsilon = 12\%)\)

Further observations of the structure at the UTS showed an extremely well developed cell structure in the direct tension specimens (\(\varepsilon = 25\%)\). Those strained to the UTS after MMTT (Fig. 2-8) showed no evidence for cell formation. The structure instead indicated the complete absence of continuous cell walls. As such it might be referred to as a primitive or incipient cell structure. From such observations it might further be concluded that:

(i) The substructure is more uniform after MMTT, there being only a primitive or incipient cell structure, which is by no means as highly developed as after direct tension and which exhibits remarkably good resistance to mechanical breakdown.

(ii) The spacing of structural features (tangles or cell walls) is considerably reduced by MMTT.

The following tentative model has been proposed\(^{26}\) to explain the formation of the characteristic MMTT substructures. During straining to the Lüder's strain in the first cycle dislocation tangles form randomly throughout the structure (cf. Fig. 2-6 (b)). After the first ageing treatment these tangles are locked by impurity atmospheres and/or fine precipitates and become effectively sessile. During the next cycle of deformation only the more weakly pinned dislocation
mainly between tangles, may move. Dislocation multiplication occurs resulting in two main effects:

(i) The pile-up of dislocations onto existing dislocation tangles giving rise to a 'mushrooming' growth of these tangles

(ii) Interaction of moving dislocations with each other or slip debris to form new tangles.

The net effect is that during subsequent ageing all these new and old growing tangles are again locked. Thus, during MMTT the size and density of tangles are increased. A similar process has been observed by Carson and Weertman during the repeated straining of Fe/Si single crystals at both ambient and elevated temperatures.

To illustrate the way in which the substructural barrier spacing (tangles or cell walls) varied as a function of total prestrain, data obtained from electron microscopy is also recorded in Table 2-1 together with dislocation densities and relevant tensile data for both direct tension and MMTT. It is apparent from this data that in direct tension a limiting substructure barrier spacing (cell size) of 1.0 μm is reached after about 10% strain. MMTT appears to accelerate and enhance tangle formation, as already mentioned, such that after 5 cycles of MMTT the tangle spacing was about 1/5 of the cold worked cell size.

From the dislocation density data presented in Table 2-1 it is apparent that the dislocation generation rate/unit strain during MMTT is greater than in direct tension, as might be expected from the repeated yielding nature of MMTT. This type of structure and level of dislocation generation have been observed during dynamic strain ageing in the range 150° - 250°C in Fe-Mn-N alloys. Recently it has been shown that very similar structures and strengthening are achieved by dynamic strain ageing. Indeed, it was shown by Lloyd and Tangri that a dynamic strain ageing treatment (DSAT) gave slightly better strengthening than MMTT. The latter work also indicated that during DSAT and MMTT at 200°C carbide precipitation occurs without significantly impairing strengthening.
<table>
<thead>
<tr>
<th>DEFORMATION REGIME</th>
<th>Σε (%/o)</th>
<th>σ₁ (MPa)</th>
<th>D (μm)</th>
<th>ρ (cm⁻²×10⁹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DIRECT TENSION</td>
<td>0</td>
<td>182</td>
<td>—</td>
<td>0.09</td>
</tr>
<tr>
<td></td>
<td>3.64</td>
<td>210</td>
<td>~1.5</td>
<td>0.81</td>
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<tr>
<td></td>
<td>5.45</td>
<td>240</td>
<td>—</td>
<td>0.88</td>
</tr>
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<td></td>
<td>7.80</td>
<td>274</td>
<td>1.5</td>
<td>1.23</td>
</tr>
<tr>
<td></td>
<td>9.50</td>
<td>288</td>
<td>—</td>
<td>1.52</td>
</tr>
<tr>
<td></td>
<td>11.15</td>
<td>298</td>
<td>1.00</td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>25.00</td>
<td>355</td>
<td>0.90*</td>
<td>~2.10(est)</td>
</tr>
<tr>
<td>II MMTT CYCLES</td>
<td>4.94</td>
<td>311</td>
<td>0.37</td>
<td>1.36</td>
</tr>
<tr>
<td>III MMTT CYCLES</td>
<td>7.02</td>
<td>378</td>
<td>0.28</td>
<td>1.45</td>
</tr>
<tr>
<td></td>
<td>6.42</td>
<td>356</td>
<td>0.27</td>
<td>1.56</td>
</tr>
<tr>
<td></td>
<td>6.54</td>
<td>360</td>
<td>0.31</td>
<td>1.65</td>
</tr>
<tr>
<td></td>
<td>8.51</td>
<td>378</td>
<td>—</td>
<td>1.45</td>
</tr>
<tr>
<td>III MMTT CYCLES</td>
<td>15.19</td>
<td>419</td>
<td>0.20</td>
<td>1.91</td>
</tr>
<tr>
<td>+ Euts</td>
<td>13.92</td>
<td>416</td>
<td>0.23</td>
<td>2.11</td>
</tr>
<tr>
<td>IV MMTT CYCLES</td>
<td>10.04</td>
<td>394</td>
<td>0.24</td>
<td>1.81</td>
</tr>
<tr>
<td>+ Euts</td>
<td>12.36</td>
<td>433</td>
<td>0.19</td>
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</tr>
<tr>
<td>IV MMTT CYCLES</td>
<td>8.38</td>
<td>424</td>
<td>0.34</td>
<td>2.25</td>
</tr>
<tr>
<td>+ Euts</td>
<td>15.19</td>
<td>419</td>
<td>0.20</td>
<td>1.91</td>
</tr>
</tbody>
</table>

* CELL-SIZE.
† STRAIN RATE = 0.9×10⁻⁴ min⁻¹.

Table 2-1
Summary of dislocation density (ρ) and substructural barrier spacing (D) data for prestrained Fe/0.56%C alloy and after MMTT.
Dislocation density results for direct tension were found to obey the relation

\[ \sigma_f = \sigma_i + \alpha G^b \sqrt{\rho} \]

... (2.2)

where \( \sigma_f \) is the tensile flow stress, \( \sigma_i \) the friction stress, \( \alpha \) a structure (treatment) dependent constant often referred to as the dislocation strengthening efficiency, \( G \) the shear modulus, \( b \) the burgers vector and \( \rho \) the dislocation density. Data from Table 2-1 is presented according to equation (2.2) in Fig. 2-9. The parameters \( \alpha \) and \( \sigma_i \) agreed well with other results obtained on a similar alloy by Dingley and McLean.

If the values of MMTT dislocation density are plotted in a similar manner, it is apparent (Fig. 2-9) that they depart from the as annealed values in a linear and positive manner. This indicates that, for a given dislocation density, the flow stress of MMTT material is greater than that strained by direct tension. Such an effect has been explained as follows. It was postulated, by McLean et al., that the strengthening arises from the more uniform nature of the MMTT dislocation segment length distribution. The number \( N \) of links with lengths lying between \( L \) and \( L + dL \) can be plotted against their flow stress, \( \sigma(\alpha L^{-1}) \), in the manner shown in Fig. 2-10, for the MMTT and direct tension cellular structures. MMTT would be expected to give a narrow, single humped distribution while the cellular structure exhibits a wider double humped distribution. The latter is likely to extend further to the left (i.e. lower flow stress) as indicated in Fig. 2-10.

The model due to McLean and co-workers appears to be sound though somewhat too simplified. However, the concept of the flow stress being controlled by the weakest links in the dislocation distribution is a useful one and will become a constantly recurring theme throughout this thesis. Its importance in the description of strengthening has been discussed in some depth by a number of authors. Distribution of the single humped variety have been measured after creep and are proving useful in the interpretation of creep behaviour. Similar concepts are fundamental to the understanding of dispersion strengthening.
and barrier strengthening in general. In all such models the flow stress of an individual dislocation segment \(\sigma(L)\) of free length, \(L\), between barriers is given by

\[
\sigma(L) = \beta' \frac{\sigma_0}{L}
\]

where \(\beta'\) is a geometrical constant \((\nu_1, 0)\).

Returning now to MMTT it is apparent that the argument of McLean et al. is not completely correct. True, the structure is more uniform after MMTT, but the important factor would seem to have been overlooked, namely the cell wall/tangle spacing. This parameter might be expected to control or limit the free length of dislocations, and hence the flow stress of the weakest segments. It should also be remembered that the local density of dislocations in cell walls produced by direct tension is similar to that in tangles. The distribution might more correctly be represented as in Fig. 2-11 and the flow stress of the weakest links would correspond to their maximum free lengths of \(\nu_1\) \(\mu m\) and 0.2 \(\mu m\) for direct tension and MMTT respectively. This corresponds to a difference in flow stress of \(\nu_100\) MPa, according to equation (2.3) and must be added to that arising from lattice friction and long-range internal stresses (equation (2.2)), assuming that these are relatively constant irrespective of the form of the dislocation distribution. The contribution to strengthening by MMTT may arise from a finer distribution of such barriers to dislocation motion.

An alternative or at least complementary source of strengthening is also expected from the strain ageing itself. This might take the form of dispersion hardening due to fine carbide precipitation or the more effective blocking action of dislocations heavily pinned by fine precipitates or solute atmospheres. The effectiveness of MMTT under conditions where precipitates have been observed suggests that some strengthening might arise from this source. However, since each of these contributions to strengthening is derived directly or indirectly from strain ageing, the question of their relative importance becomes rather academic and remains unanswered.
The much improved fracture toughness and fatigue resistance after MMTT may be a consequence of the unique structure produced. Cellular structures, like those produced during direct tension, would be expected to provide microcrack nucleation sites and relatively easy paths for crack propagation. The MMTT structure, on the other hand, contains few continuous high concentrations of imperfections and the free dislocations in the background network would be expected to have a blunting effect on any cracks formed.

The reason for the dramatic reduction in fracture toughness after 5 MMTT cycles (Fig. 2-5) is unclear at present. It is interesting to point out that 5 cycles of MMTT involves deformation during the final cycle at what is effectively the UTS (Fig. 2-1). In this condition the dislocation density after MMTT and direct tension are very similar implying a treatment independent saturation level. Another noteworthy point concerns the observation of microcracks after ~10% strain in a number of metals by direct and indirect observations (replicas and density change measurements). This latter point will be expanded in the discussion of experimental results from the present work.

Microstructural studies have, therefore, thrown considerable light on the underlying strengthening processes in MMTT. These arise from the finer distribution of substructural barriers which in turn gives rise to a more uniform or narrower dislocation segment length distribution. The incipient cell structure, which forms after MMTT, consists of a random array of discrete dislocation tangles which appear to confer superior crack propagation resistance.

2.1.2 Niobium Alloys

Work on niobium alloys by Smith and Szkopiak has confirmed that MMTT is equally applicable to other materials which exhibit discontinuous yielding. Their alloy contained nitrogen (~130 ppm) and oxygen (~500 ppm) in free solid solution. In niobium the strain ageing processes due to nitrogen and oxygen occur at widely different temperatures due to their differing atomic mobilities. Consequently, ageing at 200°C and 350°C gave rise to dislocation locking by
oxygen and nitrogen, respectively.

As in the work on iron alloys the upper and lower yield stresses and UTS increased with number of cycles and the ductility decreased. This material also showed the same tendency as in iron for \( \sigma_L \) to approach the UTS with increasing number of cycles. The strengthening efficiency of the 350°C ageing treatment was found to be superior to that at 200°C, indicating that nitrogen produced more effective dislocation locking. It is also interesting to point out that a rather dramatic drop in ductility occurred after 5 - 6 cycles, the nature of which is uncertain. This was observed after between 7 and 9% prestrain and may arise from the same source as the fracture toughness decrease above 4 cycles. The Hall-Petch parameters \( \sigma_i \) and \( K_y \) were measured by the method of Rosenfield and Owen. While \( \sigma_i \) was found to increase continuously during MMTT, as it did in iron, \( K_y \) decreased, an observation apparently in contradiction with Gordienko's results. The variation of \( K_y \) with number of MMTT cycles did, however, suggest that the improved strengthening after 350°C ageing arose from the higher value of \( K_y \) during the early stages of MMTT. Attempts were made to interpret these observations in terms of the microstructural observations made on iron alloys. The improved strengthening efficiency of MMTT as compared with conventional strain ageing was also highlighted. Evidence was obtained which suggested that after about 5 MMTT cycles precipitation of nitrides and/or carbides occurred in the 350°C treatment giving rise to an accelerated increase in \( \sigma_i \). This might be associated with the ductility drop at this stage of MMTT.

Apart from the contradictory result concerning \( K_y \), the behaviour of niobium and iron alloys appear to be very similar. This indicates that MMTT is likely to widely applicable to bcc metals and alloys which exhibit discontinuous yielding.

2.1.3 Internal Friction Studies

In addition to the mechanical test data and microstructural studies of MMTT, a number of internal friction studies have been performed to provide more information on the strain ageing process. This technique has been used because
the strength of Snoek relaxation peaks show a linear relationship with the concentration of the corresponding interstitial solute atom\textsuperscript{s} remaining in solid solution. It, therefore, gives an accurate measurement of the amount and rate of segregation to dislocations during strain ageing. Other internal friction relaxation processes were studied, namely the Köster or cold work peak\textsuperscript{53-55}, which generally gives information which compliments the Snoek peak analysis, low and high temperature background damping\textsuperscript{55} and the grain boundary damping peak\textsuperscript{55}. This work has generally provided useful background information on strain ageing processes during MMTT which is in good agreement with observations made using the techniques discussed earlier. However, little new information was obtained about the nature of MMTT from internal friction and since no similar work was done in the context of the present study the results will not be discussed further here.

2.2 Austenitic Stainless Steels

Having discussed background material in previous sections it is now intended to discuss some of those points already raised with special reference to austenitic steels. This will be accomplished by considering two broad aspects of the behaviour of these materials, namely their deformation and ageing characteristics. This will also provide a firm basis for the discussion of the research programme, as well as highlighting the factors taken into account in the planning of the project, at the stage when suitable alloys were being considered.

2.2.1 Deformation Behaviour

2.2.1.1 Stacking Fault Energy (SFE)

As is apparent from Appendix 1, SFE is one of the most important factors influencing the dislocation substructure and mechanical properties of fcc metals and alloys. Consequently it is a parameter which has received a considerable amount of attention in recent years. A number of techniques are available for its measurement and a brief survey is necessary before a detailed discussion of stainless steels is undertaken.
The most commonly used technique for measuring SFE is the node method, first proposed by Whelan. Radii of curvature of partial dislocations at extended nodes in lightly strained crystals are measured by electron microscopy and the SFE calculated using a formula based on the interaction of partial dislocations bounding the fault. The typical dislocation configuration of nodes of this type is shown in Fig. 5 of Appendix 1 and a typical example may also be seen in Appendix 1 (Fig. 13) for an 18 Cr 8 Ni stainless steel. An approximate calculation gave an expression for the SFE ($\gamma_{SF}$) in terms of the radius of curvature (R) as

$$\gamma_{SF} = \frac{T}{R} \ldots (2.4)$$

where T is the partial dislocation line tension. More detailed calculations taking into account the interaction of partial dislocations show that, while giving an approximate figure, this simple relationship yields a value 2.3 times lower than the more realistic one. This fact is important since, for comparisons to be made, data obtained by this method prior to 1964 should be multiplied by a correction factor of 2.3. Unfortunately this technique is limited to materials having SFE less than 60 erg cm$^{-2}$ (0.06 J m$^{-2}$) and is subject to quite large errors due to the excessive image widths of dislocations. This latter objection is less significant with the weak beam imaging technique. Another important factor which can lead to serious underestimation of SFE by this technique is what has been referred to as the solute impedance effect. It has been found that annealing of nodes produced by cold working can lead to a shrinkage which is not completely reversible on cooling. This is attributed to the fact that, when formed, a solute/partial dislocation interaction occurs which does not allow the equilibrium configuration to be achieved. This impedance may be overcome by thermal activation. It is, therefore, clear that the original method proposed by Whelan gives a measure of an apparent SFE only, which may be between 50 and 70% of the true value.

Several alternative techniques have been proposed and used with some success. These are:
(i) Twin boundary energy determinations$^{65,66}$.

(ii) Twin frequency measurements$^{67-70}$.

(iii) Stacking fault probability measurements with X-rays$^{71}$.

(iv) Measurements of single crystal flow stress parameters associated with the ease of cross-slip ($\tau_{III}$)$^{72}$.

(v) Deformation Textures$^{74}$.

Their indirect nature limit most of these techniques and it is often necessary to calibrate against the node method. The latter has been the most widely used technique in the case of austenitic stainless steels.

The SFE of austenitic stainless steels has been the subject of a number of studies$^{58,68,69,75-80}$ in recent years. Before moving on to a discussion of this work the point made earlier about the values determined by the node method prior to 1964 should be amplified. This also applies to all the work done prior to 1967$^{58,68,69,75-80}$ where only the simple model of node forces was used (equation (2.4)) and therefore all values should be multiplied by a factor of 2.

The usual approach has been to correlate variations in SFE with alloying additions and a similar approach will be followed here. Of the alloying elements studied, the effect of nickel has received most attention$^{58,68,69,75-81}$ and is found to cause an increase in SFE as shown in Fig. 2-12. Chromium, on the other hand, at relatively low levels (<15%)$^{68,78}$ causes a decrease in SFE while at higher levels$^{68,69,78}$ an increase is observed (Fig. 2-13). Silicon$^{80}$ has one of the most dramatic effects of all elements causing a decrease of >50% for alloying additions of only 2.5%. The effect of other elements is illustrated in Table 2.2. Copper, niobium and molybdenum are reported to increase SFE, while cobalt and manganese cause a decrease. Niobium$^{77}$, titanium$^{76}$ and molybdenum$^{76}$ in solution have been reported to lower the SFE. Carbon$^{68,75,78}$ has been found to increase SFE only slightly, while nitrogen has a negligible effect$^{75,78}$, although the latter tends to cause planar dislocation structures to form. Phosphorus$^{75}$ appears to behave in a similar manner to nitrogen and it is thought that this characteristic
### Table 2-2

Effect of alloying elements on SFE in Austenitic Stainless Steels ($\Delta_{\gamma SF}$ is the change in SFE per at % of solute)

<table>
<thead>
<tr>
<th>Alloying Element</th>
<th>$\Delta_{\gamma SF}$ (erg cm$^{-2}$ at%$^{-1}$)</th>
<th>Dulieu and Nutting$^{68}$</th>
<th>Silcock et al$^{69}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>+1.4</td>
<td>+0.8</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>-3.4</td>
<td>-3.9</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>-0.55</td>
<td>-0.75</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>+0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>+3.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>+3.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>+0.1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mn</td>
<td>-1.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 2-3

Diffusion data for major elements in austenite (*constant for grain boundary diffusion given as $D_0$, where $\delta$ is the grain boundary width and is usually taken as twice the lattice parameter i.e. $\sqrt{2}$)

<table>
<thead>
<tr>
<th>Alloying Element</th>
<th>Diffusion Path</th>
<th>System</th>
<th>$2D_0$ (cm$^2$ s$^{-1}$)</th>
<th>$Q$ (kcal mole$^{-1}$)</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>Lattice</td>
<td>20/25/Nb</td>
<td>1.74</td>
<td>67.9</td>
<td>143</td>
</tr>
<tr>
<td>Fe*</td>
<td>Gr. Bound.</td>
<td>20/25/Nb</td>
<td>$8.3 \times 10^{-7}$</td>
<td>43.0</td>
<td>145</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20/25/Nb</td>
<td>$1.1 \times 10^{-3}$</td>
<td>57.6</td>
<td>23</td>
</tr>
<tr>
<td>Cr</td>
<td>Lattice</td>
<td>20/25/Nb</td>
<td>0.19</td>
<td>58.8</td>
<td>146</td>
</tr>
<tr>
<td>Ni</td>
<td>Lattice</td>
<td>20/25/Nb</td>
<td>4.06</td>
<td>67.5</td>
<td>146</td>
</tr>
<tr>
<td>Nb</td>
<td>Lattice</td>
<td>$\gamma$-Fe</td>
<td>530</td>
<td>82.3</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20/25/Nb</td>
<td>2.0</td>
<td>64.2</td>
<td>23</td>
</tr>
<tr>
<td>Mn</td>
<td>Lattice</td>
<td>$\gamma$-Fe</td>
<td>0.57</td>
<td>66.0</td>
<td>147</td>
</tr>
<tr>
<td>C</td>
<td>Lattice</td>
<td>&quot;</td>
<td>0.15</td>
<td>33.5</td>
<td>148</td>
</tr>
<tr>
<td>N</td>
<td>Lattice</td>
<td>&quot;</td>
<td>0.20</td>
<td>36.0</td>
<td>149</td>
</tr>
</tbody>
</table>
is due to some other factor than the lowering of SFE. An attempt to rationalise these effects has been made in terms of valency electron to atom ratio which appears to explain, at least empirically, results for copper alloys. Spreadborough using the theory of directed bonding suggested that, by considering electron contributions to the hybrid orbitals necessary to produce the three common structures, it is possible to predict their relative stabilities. Thus increasing the number of d-electrons available for bonding will favour the transition fcc $\rightarrow$ hcp $\rightarrow$ bcc, while with a constant d-electron distribution an increase in s-electrons will favour the transition fcc $\rightarrow$ hcp or bcc. Such changes in electron configuration and relative stability should be reflected in SFE. Dulieu and Nutting attempted to rationalise the alloying effects described above in terms of Spreadborough's hypothesis with some success. Silicon with its higher valency than the solvent metals lowers the SFE as predicted. Interstitial elements such as carbon, nitrogen and probably phosphorus appear in conflict with these predictions, but this is to be expected because of size factor effects.

The solute impedance effect mentioned earlier has not been investigated in austenitic steels and the SFE values quoted here are apparent values only. It is impossible to estimate what effect this will have on SFE but the assumption that these apparent values are approximately 0.7 of the real value, as observed for copper alloys, may not be too much in error.

2.2.1.2 Austenite Stability

One aspect of austenite stability has already been discussed at some length, namely the SFE. This is a measure of the stability of fcc austenite ($\gamma$) with respect to the hcp phase, often referred to as $\varepsilon$-phase. The other types of stability which merit some discussion are those with respect to bcc ferrite ($\alpha$) and the complex $\sigma$-phase. Reference to the ternary Fe-Cr-Ni diagram (650°C isothermal section reproduced in Fig. 2-14) illustrates the extent of the $\gamma$-phase field for iron rich alloys. It is apparent from Fig. 2-14 that alloys of the 18/8 variety are unstable with respect to both $\alpha$ and $\sigma$-phase at this temperature.
From the point of view of good creep and high temperature properties, σ-phase precipitation, during long time exposure at temperatures in excess of 650°C, should be avoided. During room temperature deformation and relatively low temperature treatments, martensite and ferrite formation are generally to be avoided.

The sigma phase occurs in most transition-metal alloy systems usually with deleterious effects on properties. This is especially true of stainless and other high alloy steels. It has a complex tetragonal structure and is characterised mainly by its embrittling effect. A general review of sigma phases has been published in which their occurrence, structure and properties are discussed at length. It is clear from the phase diagram that increasing nickel and decreasing chromium contents stabilise austenite with respect to σ-phase formation. This tendency, namely for ferrite stabilising elements to increase the susceptibility to σ-phase formation and austenite stabilisers to lower it, seems fairly general.

Its formation is, therefore, enhanced by additions of most carbide formers and silicon. Generally forming in the range 500°C - 950°C, it does so at a maximum rate in the range 750°C - 900°C and relatively more rapidly in duplex (ferrite/austenite) structures than in austenite. The effects of σ-phase on mechanical properties have been studied in some detail and may be summarised as follows:

(i) Sigma may increase creep strength up to 750°C in short-term applications with deformation rates <10^{-2} hr^{-1} but during temperature cycling may lead to brittle failure.

(ii) At very low rates of strain, sigma decreases creep strength.

(iii) Sigma increases short-term tensile and yield strength.

(iv) Sigma decreases low temperature ductility and increases tensile strength.

(v) The distribution of sigma particles is important to strength and ductility, its effect being more deleterious to austenitic steels where it occurs at grain boundaries.

The stability of austenite with respect to ferrite introduces factors other than simple equilibrium thermodynamics. For alloys containing greater levels of nickel than indicated by the γ-phase boundary, duplex austenite-ferrite structures
are thermodynamically stable at relatively low temperatures\textsuperscript{94}, although kinetically the transformation is unlikely to occur. Under such conditions martensite may form on cooling, although the $M_s$ temperature is generally well below room temperature for alloys of the 18 Cr 8 Ni type\textsuperscript{95}. For example, a low alloy 12% Cr steel has an $M_s$ around 350°C and is completely martensitic on cooling to room temperature, while for steels of the 18 Cr 8 Ni variety $M_s$ temperatures are around -100°C and little transformation is produced even by refrigeration at -196°C\textsuperscript{95}. The thermodynamics of martensite transformations has been discussed at length by Kaufman and Cohen\textsuperscript{96} and it has been demonstrated by Patel and Cohen\textsuperscript{97} that the application of stress, i.e. mechanical energy, can raise the $M_s$ temperature significantly. This critical temperature has often been referred to as the $M'_d$ or 'deformation induced martensite start temperature'. As might be expected, $\gamma$-stabilising elements reduce the tendency for deformation induced martensite\textsuperscript{95,98-101}, while ferrite stabilising elements have the opposite effect. The structure and mechanisms of martensite formation have been thoroughly reviewed\textsuperscript{102} and the formation of deformation induced martensite widely studied\textsuperscript{98-105}. For steels containing 18% Cr the level of nickel required to eliminate deformation induced martensite has been found to be around 13% during room temperature tensile straining to failure\textsuperscript{106}. Under those conditions where this transformation does occur it is generally found that the proportion of transformation product increases with the degree of deformation\textsuperscript{98,99,103-106}. Also, generally accompanying this transformation, there is the formation of metastable hcp $\varepsilon$-phase which appears consistent with the low SFE of these alloys. The precise mechanism of the transformation is unclear, especially the nature of the nucleus, although a sequence such as stacking fault formation leading to $\varepsilon$-phase nucleation and subsequently martensite nucleation seems widely accepted.

The question of austenite stability is of considerable importance due to its effect on both the dislocation substructures and mechanical behaviour of austenitic stainless steels. It forms the foundation for the choice of the material used in this work.
2.2.1.3 Deformation Substructures

Certain aspects of dislocation substructures have already been reviewed and discussed elsewhere (see Appendix 1). Low temperature substructures are largely dependent on the alloy composition due to its rather drastic effect on austenite stability. In austenitic steels deformation induced martensite may form in low SFE alloys due to the intimate relationship between this phenomenon and ε-phase formation. During room temperature deformation this may occur at SFE below about 30 erg cm$^{-2}$ (0.03 J m$^{-2}$) or for example at nickel contents below 13%. The substructure generally consists of a mixture of bands of ε-phase and martensite which possesses a high dislocation density, having formed by a process similar to deformation twinning. As deformation proceeds ε-phase having reached some maximum level generally disappears at the expense of the martensite, such that ultimately a completely martensitic structure may be achieved. This type of alloy or those of higher SFE deformed at sub-zero temperatures are employed in certain thermo-mechanical treatments and the principle has been used in maraging alloys. However, regardless of the importance of these alloys and their transformation behaviour, further discussion in this section will be confined to austenitic steels which are stable at the deformation temperature.

The other effect of SFE, which is relevant to the present work, is that on the nature of the dislocation substructure. This has already been discussed in APPENDIX 1 where the transition from planar to tangled and cellular dislocation arrays was considered and shown to be a function of SFE (Fig. 14). With SFE in excess of about 30 erg cm$^{-2}$ (0.03 J m$^{-2}$) planar distributions of dislocations are not observed although substructures may possess a slightly banded appearance. This statement requires some qualification although under normal circumstances it holds true. For high nitrogen contents (<0.05 wt%) planar dislocation arrays may be found even in high SFE alloys. The most plausible explanation would seem to be that nitrogen and phosphorous induce short range order interactions which affect rather dramatically the substructure.
Much has so far been said of stacking faults and SFE, but nothing concerning the partial dislocations bounding these faults, particularly with regard to their basic properties and the way in which they affect both dislocation substructure and mechanical behaviour. Slip in crystals is generally discussed in terms of characteristic translation vectors known as Burgers vectors. Where unit slip produces no net change in atomic configuration these vectors define 'perfect' dislocations. Since Peirls-Nabarro forces, required to overcome lattice resistance to dislocation motion, are a minimum on close packed planes, these contain most potential Burgers vectors. Energy considerations limit these still further to the shortest atomic displacements. In fcc crystals the close-packed plane is (111) and the shortest unit translation is $a/2 <110>$, which is the Burgers vector describing unit or 'perfect' dislocations in this structure. If a translation occurs which disrupts the atomic configuration by, for example, introducing a discontinuity in the packing sequence, this is referred to as an imperfect or partial dislocation. Both the crystallography and energetics of dislocation reactions in crystals have been dealt with exhaustively in the literature and thus will not be dealt with further unless it is particularly relevant to this or subsequent discussion.

The most useful way of representing dislocation reactions in fcc structures is with the aid of Thompson's tetrahedron, illustrated in Fig. 2-15. This consists of a regular tetrahedron $ABCD$, where the verticles represent vectors $AB$, $BC$ etc, of type $a/2 <110>$, the normal Burgers vector of slip dislocations. Mid-points of the faces $ABC$, $BCD$ etc, which represent (111) planes, are referred to as $\alpha, \beta, \gamma$ and $\delta$. This then yields two alternative types of possible Burgers vector, both representing partial dislocations. Face diagonals $A\beta$, $B\alpha$ etc, represent the vector $a/6 <112>$, which characterises the glissile Shockley partial dislocation, while the vectors $A\alpha$, $B\beta$, $C\gamma$ and $D\delta$ represent sessile Frank partial dislocations of type $a/3 <111>$. Two important criteria must be borne in mind when investigating potential dislocation reactions. The first, often referred to as 'Frank's' rule, is purely geometrical and requires that the sum of all the Burgers vectors at a
dislocation junction or node, be zero. The second is an energy criterion, which specifies that a given dislocation reaction may only occur if the result is a net reduction in energy. In an elastically isotropic material the energy per unit length of dislocation is directly proportional to the square of its Burgers vector.

The dislocation reaction which has most significance as regards the general appearance of dislocation substructures is the simple dissociation of the type

\[ \frac{a}{2} \langle 101 \rangle \rightarrow \frac{a}{6} \langle 112 \rangle + \frac{a}{6} \langle 211 \rangle \] ... (2.5)

or in the Thompson notation

\[ (DB) a \rightarrow aB + Da \]

Such a reaction leads to splitting of the unit dislocation into two Shockley partials separated by a ribbon of stacking fault and as a consequence extended nodes are formed, as shown in Fig. 5 of Appendix 1. The more general effects of SFE on low temperature dislocation substructures arise from this dissociated nature of dislocations and its influence on cross-slip. Broadly speaking the ease of cross-slip decreases with decreasing SFE or, alternatively, increasing partial dislocation separation, which is given by

\[ w = w_0 \left( 1 - \frac{2\nu}{2-v} \cos 2\alpha \right) \] ... (2.6)

where \( w \) is the partial separation, \( \alpha \) the angle between the dislocations and the resultant Burgers vector of the extended dislocation and \( w_0 \) is the mean width. This is given by

\[ w_0 = \frac{G\bar{b}^2}{8\pi\gamma} \frac{(2-\nu)}{(1-\nu)} \]

where \( \bar{b} \) is the Burgers vector of the partial dislocations bounding the stacking fault and \( \gamma \) the SFE. A more complete calculation has been made by Seeger and Schoeck which takes into account elastic anisotropy. This extended nature of dislocations is the main barrier to cross-slip and in order for them to move the stacking fault must become constricted by the applied stress and back stress of piled-up dislocations. Seeger has calculated that the shear stress \( (\tau) \) required for cross-slip to occur is given by
where \( n \) is the number of dislocations in a pile-up in which the leading dislocation is to cross-slip. This condition holds only at \( 0^\circ \text{K} \) in the absence of thermal energy. It has been shown \(^{72}\) that \( T_{\text{III}} \) in single crystals is a function of SFE. Reasonable estimates of SFE may be obtained from data for the temperature dependence of \( T_{\text{III}} \) using Seeger's model of which equation (2.7) is the central feature. Cellular or tangled dislocation structures are unlikely to arise until the applied stress exceeds this critical value. If both work-hardening and flow stress behaviour in an alloy are relatively insensitive to SFE, higher applied stress or equivalently higher strains would be necessary to produce these structures in alloys of decreasing SFE, as is shown in Fig. 14 of Appendix 1.

More specific features of dislocation substructures may be attributed to the nature of individual dislocation interactions. The most common reaction in fcc crystals between \( a/2 \langle 110 \rangle \) dislocations is of the type

\[
a/2 \left[ 01\bar{1} \right] + a/2 \left[ \bar{0}1\bar{1} \right] \rightarrow a/2 \left[ 1\bar{1}0 \right]
\]

Such a reaction occurs between dislocations lying in their slip plane \((111)\) and, as a result of creep or annealing, gives rise to hexagonal, planar networks described in Appendix 1. A reaction of this general type may occur between dislocations gliding on intersecting slip planes thus

\[
a/2 \left[ 10\bar{1} \right] + a/2 \left[ 01\bar{1} \right] \rightarrow a/2 \left[ 1\bar{1}0 \right]
\]

... (2.8)

Such a reaction was first proposed by Lomer\(^{120}\) and is often referred to as a Lomer lock since the resultant dislocation is sessile, being incapable of moving freely in either slip plane. This dislocation will lie in a \([1\bar{1}0]\) direction having its Burgers vector in a \((001)\) plane. It will therefore be a pure edge dislocation.

A more complicated interaction may occur if both \( a/2 \langle 110 \rangle \) dislocations are dissociated into Shockley partials\(^{121}\). Under these circumstances the leading partial dislocations may associate forming a dislocation of Burgers vector \( a/6 \langle 110 \rangle \) which is often known as a stair-rod dislocation. A typical sequence of reactions leading to the formation of a Lomer-Cottrell lock is

\[
\tau = \frac{2G}{n} \left\{ 0.56 - \frac{\gamma}{Gb} \right\}
\]
\[
a/2 [01\bar{1}] \longrightarrow a/6 [12\bar{1}]^* + a/6 [1\bar{1}2]
\]
\[
a/2 [10\bar{1}] \longrightarrow a/6 [2\bar{1}1]^* + a/6 [1\bar{1}2]
\]
The two leading partials* combine
\[
a/6 [12\bar{1}] + a/6 [2\bar{1}1] \longrightarrow a/6 [1\bar{1}0]
\]
Interactions of this type might alternatively be thought of in terms of the
dissociation of a Lomer lock thus
\[
a/2 [1\bar{1}0] \longrightarrow a/6 [1\bar{1}0] + a/6 [1\bar{1}2] + a/6 [1\bar{1}2]
\]
This type of dislocation interaction may be considered in terms of Saada's model
of attractive junction formation and it seems likely that, as such, they play a
significant role in strengthening. Apart from requiring large stresses to
decompose such junctions, thereby freeing dislocations for glide, multiplication
etc., their sessile character makes them formidable barriers to the movement of
other dislocations, particularly in low SFE materials. In the latter materials,
pile-ups will occur at these barriers until quite high stress concentrations before
cross-slip can occur. In higher SFE materials these barriers may well become
centres for tangling and subsequent cell formation.

The possibility of an alternative reaction for the formation of similar barriers
has been considered and become known as Hirth locks. These form by reactions of
the type
\[
a/2 [101] + a/2 [\bar{1}01] \longrightarrow a/6 [11\bar{2}] + a/3 [00\bar{1}] + a/6 [\bar{1}\bar{1}2]
\]
... (2.9)
where the reacting dislocations have perpendicular Burgers vectors and meet forming
a stair-rod dislocation, a/3 [00\bar{1}], along [1\bar{1}0]. Such dislocation locks have been
observed in only a limited number of fcc single crystal studies which have been
extensively reviewed in recent years, as well as in austenitic steels. Unfortunately, without Burgers vector determinations Lomer, Lomer-Cottrell and
Hirth locks cannot be distinguished. However, this has been attempted in single
crystals of copper and in orientations which should favour Hirth locks none
were observed. This has recently been confirmed in work on a low SFE (\nu0.02 Jm\(^{-2}\))
Cu-10% Al alloy. A typical micrograph of Lomer-Cottrell dislocations taken from the work of Basinski is shown in Fig. 2-16. It is apparent from these figures that Lomer-Cottrell dislocations, whose frequency increases with strain once two or more slip systems become operative, form in regular parallel bands a few microns in length. Hirth locks have been reported recently in copper though the Burgers vector analysis has been criticised as being insufficiently rigorous. While such locks could conceivably form in any fcc crystals they are rarely observed in high SFE metals such as aluminium, where the ease of cross-slip prevents dislocations being forced sufficiently close for such reactions to occur.

It should also be mentioned that a dislocation of Burgers vector $a/6 <310>$ is generally considered a possible stair-rod since it can be formed by combination of two Shockley partials

$$a/6 [\bar{1}2\bar{1}] + a/6 [\bar{2}1\bar{2}] \rightarrow a/6 [0\bar{3}1]$$

as is one of Burgers vector $a/3 <110>

$$a/6 [\bar{1}1\bar{2}] + a/6 [11\bar{2}] \rightarrow a/3 [110]$$

Like the Hirth lock, neither of these has been positively identified experimentally.

The latter three types of dislocation reaction are not directly obvious from the Thompson tetrahedron. One remaining reaction which, although of little significance in the context of deformation behaviour, will be important in the discussion of precipitation at a later stage should be mentioned, since it may be directly inferred from Fig. 2-15. This reaction is that whereby a unit dislocation may dissociate into a Frank and Shockley partial thus

$$a/2 [\bar{0}\bar{1}\bar{1}] \rightarrow a/3 [1\bar{1}1] + a/6 [21\bar{1}]$$

... (2.10)

If the perfect dislocation whose slip plane is (111) lies parallel to the intersection of (111) and (111) then the Burgers vector of the Shockley partial lies in (111), while the Frank partial is sessile. This reaction generally neither increases nor reduces the elastic self-energy of the resulting configuration, although if the elastic anisotropy is high the reaction may become energetically favoured. Other reactions whereby perfect dislocations combine with either
Shockley or Frank partials may also occur although these processes tend only to be of importance during the annealing of faulted defects and like phenomena.

Finally, one further microscopic feature of deformation substructures merits some discussion, namely, the formation of dislocation dipoles. These may occur in arrays or individually as shown in Fig. 2-17, and consist of parallel pairs of positive and negative dislocations. They are characteristically of edge type lying in <112> having lengths of 0.2 – 0.5 μm and widths of \(\sqrt{300}\) (3000 nm). A number of mechanisms have been suggested to account for their formation, two of which are shown schematically in Fig. 2-18. The first illustration (Fig. 2-18(a)) shows the formation of a large jog on a screw dislocation as a result of the coalescence of a number of smaller jogs caused by individual dislocation intersections. If the jog is of edge character it forms a step in the dislocation line between slip planes and as a result can not move conservatively. If the applied stress is sufficiently high the screw segments will glide leaving the edge portion behind so forming a 'cusp' consisting of two parallel segments of edge dislocation with the step trailing at their end. This may be a rate controlling process during creep. Alternatively, two dislocations of opposite sign gliding on parallel slip planes can become aligned over part of their length. Cross-slip at one end of this portion will lead to the formation of a dipole and a jogged dislocation (Fig. 2-18(b)). Such a mechanism would be expected to operate when slip line spacings are \(\sqrt{300}\) (3000 nm). One criticism of the latter type of model involving cross-slip is the observation that dipoles form at 4.2 K where cross-slip can occur only at high stresses. Such models account only for isolated dipoles. However, piled-up arrays of dipoles might naively be expected to form as a result of one such isolated dipole which becomes an obstacle to other dislocations which, by cross-slipping around it, generate more dipoles. This type of process has been reviewed thoroughly by Suzuki who has investigated the effect of stacking fault energy and local order on dipole formation. Clusters of dipoles tend to form during single crystal deformation either in the form of regular piled-up arrays or in irregular arrays as in Fig. 2-17.
This feature like the tendency for pile-ups already discussed is a function of SFE. Like Lomer dislocations these groups of dipoles probably play some, as yet, ill-defined role in the build-up of tangled and subsequently cellular substructures.

These features of dislocation substructures and associated phenomena, while not discussed exhaustively, provide sufficient introduction to this general area, which has been reviewed extensively in the literature, especially for single crystals. While in these cases the processes can be clearly observed their relative contributions to work hardening are still the subject of much discussion.

In polycrystals the situation is still more complex and all these various substructural features can generally be observed in any field of view. It, therefore, seems fruitless to pursue this subject further, since those generalisations made in section Appendix 1 and earlier in this section (2.2) concerning SFE and substructure are of more value than specific work hardening theories, with the possible exception of the simple network models which are, after all, gross oversimplifications.

2.2.2 Low and Intermediate Temperature Ageing Behaviour

The behaviour of austenitic steels during low and intermediate temperature ageing (T<650°C) is, as yet, insufficiently understood. The background in terms of solute/dislocation and other possible interactions (e.g. vacancy/dislocation and solute-vacancy/dislocation interactions) is too broad for the present study. The general characteristics of these processes have been the subject of a number of recent reviews. Therefore, it is intended to discuss only the more specific aspects of this behaviour which are relevant to stainless steels. One of the major problems seems to be the difficulty of being able to relate kinetic data to a particular mechanism. This is due to the complex constitution of these alloys and the relatively limited knowledge of diffusion data, some of which is reproduced in Table 2-3.

The possibility of strain-ageing leading to serrated yielding ('jerky' flow) in austenite was reported in 1950. Tamhanker et al in a study of a 10 Cr-35 Ni alloy observed serrated yielding which was followed as a function of
grain-size, strain rate and testing temperature. They also observed conventional yield point effects and attributed them to chromium or nickel diffusion, associated with Suzuki locking. Subsequently, Barnby\textsuperscript{152} observed serrated yielding in solution treated 316 steel (18 Cr/11 Ni/2.5 Mo/0.05C) tested in the range 300° - 700°C and discontinuous yield points during room temperature tests following 350°C ageing. Using the Cottrell relationship for serrated yielding\textsuperscript{153}

\[ \dot{\varepsilon} = 10^{10}D \]

where \( \dot{\varepsilon} \) is the applied strain rate and \( D \) the diffusion coefficient, he suggested chromium diffusion to explain the effect. It would, however, seem that, from size factor considerations, this is unlikely unless precipitation of \( M_{23}C_6 \) were responsible, which would undoubtedly be possible at elevated temperatures (\( \approx 700^\circ C \))\textsuperscript{154}. Such a situation can only be resolved by electron microscopy, which was not attempted. NbC precipitation has been confirmed by Naybour\textsuperscript{155} for an 18 Cr/12 Ni/Nb steel at 650°C in which case serrated yielding and profuse dislocation multiplication were observed. These observations point towards quench ageing and precipitation effects, which will be dealt with in the next section, but leaves unresolved the effect at temperatures below about 600°C.

Nishino\textsuperscript{156} carried out static strain ageing tests on 18 Cr/8 Ni and 25 Cr/20 Ni steels and obtained an activation energy of 26 kcal mole\textsuperscript{-1} (110 kJ mole\textsuperscript{-1}). The situation was finally resolved by Jenkins and Smith\textsuperscript{157} who obtained an activation energy of 35 kcal mole\textsuperscript{-1} (146 kJ mole\textsuperscript{-1}) on ageing at temperatures of 200° - 400°C and 90 kcal mole\textsuperscript{-1} (376 kJ mole\textsuperscript{-1}) at temperatures in excess of 400°C. These differences in activation energies they ascribed to interstitial and chromium diffusion, respectively. In static isochronal ageing tests they observed two strengthening peaks one at 500° and the other at 800°C. Both peaks disappeared after decarburisation indicating the importance of interstitial elements even at the higher temperatures. This lends support to the precipitation hypothesis at high temperatures and also to the doubtful effect of substitutional elements in terms of size factor interactions. Recent work\textsuperscript{158} on 316 steel confirms these observations concerning low temperature activation energy of about
30 kcal mole\(^{-1}\) (126 kJ mole\(^{-1}\)) and supports Fleischer's view\(^{159}\) that only relatively weak interstitial-dislocation inter-reactions are likely in fcc alloys. Similar effects have been observed in ausforming steels\(^{160-162}\). Rose and Glover\(^{163}\) have observed rapid static ageing effects at temperatures below and serrated yielding above 100°C in a Fe/35 Ni/C alloy. They obtained an activation energy of 20 kcal mole\(^{-1}\) (83.5 kJ mole\(^{-1}\)), and ascribed it to the stress induced ordering\(^{164}\) of carbon-vacancy pairs in the stress field of dislocations. Such a model was postulated because interstitials in fcc lattices occupy octahedral sites hence producing only symmetrical distortions and is arguably the only mechanism which can explain the speed of the ageing process. The static ageing result might, however, be due to unloading yield point processes\(^{165,166}\), while the serrated yielding effect could be explained in terms of conventional interstitial dislocation interactions. Support for the stress-induced ordering model of the static ageing phenomenon exists in the work of Jenkins and Smith\(^{157,158}\), internal friction studies\(^{167,168}\) and the fact that\(^{169}\) a local order reaction has been reported at \(\sim 500°C\).

In conclusion it may be stated that each of these phenomena requires interstitial-dislocation interactions of one form or another and that their effect is not so dramatic as in bcc alloys. Most work appears to exclude the possibility of significant substitutional interactions due either to size factor or modulus effects. Suzuki interactions\(^ {169}\), while being feasible, rarely cause significant ageing peaks and are of doubtful importance in austenitic steels.

2.2.3 High Temperature Ageing Phenomena

Most precipitation reactions in steels of the type under consideration here can be generally classified in terms of the classical theory of nucleation\(^{170,171}\). Precipitation occurs at grain or twin boundaries and on dislocations in a manner consistent with the theory of heterogeneous nucleation\(^{170,171}\). With regard to the present study it is only the latter of these nucleation sites that is relevant, under most circumstances.

2.2.3.1 General Theory of Heterogeneous Precipitation

According to classical nucleation theory\(^{170,171}\), the total free energy change
(ΔF) accompanying the formation of a nucleus of a second phase from a super-saturated solid solution can be expressed by

\[ ΔF = ΔF_v + ΔF_{SF} + ΔF_{ST} \]  \hspace{1cm} (2.11)

The change in volume free energy (ΔF_v) must be negative and is a function of the supersaturation. Changes in surface free energy (ΔF_{SF}) and strain free energy (ΔF_{ST}) due to the formation of the nucleus are positive. The total free energy change is positive increasing with precipitate size to a maximum at a critical size of nucleus. When the radius of the nucleus exceeds this critical value (R_c), ΔF decreases and a stable nucleus is formed capable of growth. Attempts have been made to calculate these free energy changes for both the formation of coherent\(^{172,173}\) and incoherent\(^{174}\) precipitates. In both cases the problem is one of quantifying the strain energy term of equation (2.11).

It would be instructive at this stage to consider one of these models in order to illustrate a few of the salient features of dislocation-nucleated precipitation. The model for incoherent precipitates due to Cahn\(^{174}\) will be discussed. Assuming a cylindrical, non-coherent nucleus of radius (R) lying along a dislocation, he identified the strain energy released with the self energy of the dislocation line. This energy reduces the barrier to nucleation. Cahn's model leads to the conclusion that the free energy for precipitation is less at a dislocation than in the perfect lattice, that is by heterogeneous rather than homogeneous nucleation. The numerical value of the free energy per unit length of dislocation occupied by the nucleus can be obtained from the expression

\[ F = -ΔF_v R^2 + 2πγR - AlnR + \text{const.} \]  \hspace{1cm} (2.12)

where

\[ A = \frac{Gb^2}{4\pi(1-\nu)} \]  \hspace{1cm} for an edge dislocation

\[ = \frac{Gb^2}{4\pi} \]  \hspace{1cm} for a screw dislocation

\[ γ \] = interfacial energy between precipitate and matrix. A minimum in free energy was found when the quantity
\[ \alpha = \frac{2A\Delta F_v}{\pi \gamma} < 1.0 \quad \ldots \quad (2.13) \]

This circumstance has been compared to that when a dislocation is surrounded by a Cottrell atmosphere and has been called, by Dehlinger\(^{175}\), a 'performed nucleus'. This corresponds to curve A in Fig. 2-19. If, however, \( \alpha > 1 \) no such minimum is obtained. There is, therefore, no nucleation barrier and the precipitation will proceed instantaneously if no diffusion is necessary (curve B of Fig. 2-19).

If the reaction involves composition changes, the rate of precipitation will be controlled only by the rate of diffusion. Cahn\(^{173}\) showed that the importance of nucleation on dislocations increases with increasing supersaturation \( \Delta F_v \).

For example, at 30% supersaturation the rate of homogeneous nucleation in a typical case is \( \approx 10^{-70} \text{ cm}^{-3} \cdot \text{sec}^{-1} \), whereas the rate of heterogeneous nucleation at dislocations is \( \approx 10^8 \text{ cm}^{-3} \cdot \text{sec}^{-1} \). At very high supersaturations the homogeneous nucleation rate for partially coherent precipitates might be so large as to mask the effect of dislocations. This, however, while being relevant to, for example, aluminium alloys, does not concern us here. In addition to the effect of solute supersaturation, another parameter which will have a significant effect on nucleation is the Burgers vector, \( \vec{b} \). Nucleation of a precipitate will be easiest when \( \vec{b} \) is large. For an incoherent precipitate the relative orientation of precipitate and \( \vec{b} \) will be unimportant since there will be lattice misfit between the precipitate in all directions. This effect of the magnitude of \( \vec{b} \) will be of considerable importance in the present study. A simple calculation is made in Appendix 2 to illustrate this point.

While the Cahn model is physically unrealistic, as regards the alloy system under study, it provides a useful qualitative introduction to this subject.

The models proposed for the nucleation of coherent precipitates are closer to physical reality. In these the elastic strain field of the precipitates is considered and its interaction with the dislocation modify the precipitation process. In order to minimise the strain energy of the system the precipitate strain field will interact with that of the dislocation in much the same way as a
solute atom. It transpires that such an interaction is only likely in the case of an edge dislocation or one with a significant edge component, since only these possess a hydrostatic strain field. In this respect these models differ from that of Cahn in which the mutual strain field effect is ignored. Coherent interfacial energies are significantly lower than in the incoherent case which will change the energy balance equations (2.11) and (2.12) to quite a large extent, as also will the misfit between particle and matrix, which will affect the strain energy term.

A factor not yet considered is the effect of segregation to dislocations. Where large size differences exist between solute and solvent this can lead to a reduction in the strain energy associated with precipitation and the provision of an interface dislocation. If the binding energy to the dislocation is of the order of 0.3 eV the concentration at a dislocation may be \(50\) times greater than the equilibrium solubility in the temperature range where precipitation can occur. A speedy short circuit path also exists for diffusion during the early stages of growth.

2.2.3.2 General Considerations of NbC Precipitation

NbC has a NaCl type lattice, the metal atoms being arranged on an fcc lattice as in austenite. The orientation relationship between NbC and austenite has been determined as

\[
\begin{align*}
\{100\}_{\text{NbC}} & \parallel \{100\}_{\text{AUSTENITE}} \\
\langle 100 \rangle_{\text{NbC}} & \parallel \langle 100 \rangle_{\text{AUSTENITE}}
\end{align*}
\]

The lattice parameter of NbC or, more correctly, niobium carbonitride, Nb(C,N), lies in the range 4.38 - 4.47\(\AA\) depending on the composition and degree of non-stoichiometry. Since the lattice parameter of austenite has generally been found to be 3.58 - 3.59\(\AA\) depending on alloying content, a lattice expansion of \(\sim 24\%\) accompanies the precipitation of NbC.

The precipitation processes are generally connected with mechanisms for the relief of the considerable strains produced as a precipitate nucleates and grows.
For example, the collapse of one vacancy onto the surface of the carbide can accommodate the expansion due to the transfer of 1.1 Nb atoms from matrix to carbide. If a vacancy concentration of $10^{-4}$ were retained on quenching an alloy containing 0.4 at % Nb and (C + N) in solution, only ~3% of the subsequent precipitation could be accommodated. Precipitation might, therefore, be expected to occur in association with some vacancy production process. An alternative or possibly complementary process might be the punching of dislocation loops. In order to relieve the strains for every four atoms precipitated a strain relief of approximately one Burger's vector is required. Therefore, for such an increase in radius one dislocation loop is required.

2.2.3.3 Precipitation Processes

Precipitation of niobium and other refractory carbides has been widely studied over the past decade and has been reviewed recently. The main features of the precipitation process are as follows:

(i) Precipitation of NbC in the temperature range 600° - 900°C appears to occur as a two stage process;

Stage I consists of nucleation and growth of NbC on undissociated dislocations;

Stage II differs from alloy to alloy and with such variables as degree of supersaturation, ageing temperature and dislocation density;

(ii) Depending on the above parameters, further precipitation may occur on precipitate growth dislocations (PGD) punched from precipitates formed on the primary dislocation structure or in association with stacking faults formed by the dissociation of bowing PGD;

(iii) Together with these processes a random matrix precipitation occurs, often referred to as 'matrix' or 'random dot' precipitation;

(iv) Prolonged ageing at temperatures ranging from 600° to 900°C can lead to reversion processes accompanied by the precipitation of $M_{23}C_6$, $M_6C$ and sigma phase, together with the more usual particle coarsening or Oswald ripening process.
Nucleation processes involving dislocations are important in the development of an efficient mechanical-thermal treatment while particle coarsening requires attention because of its potential effect on substructure stability particularly in creep.

Dislocations as preferred nucleation sites for precipitation have been discussed in some detail in the previous section. It is apparent that because of segregation, strain energy reduction and the resulting high rates of nucleation, dislocations play a dominating role in the precipitation processes in austenitic steels, where size factor effects are generally important. Unit dislocations are observed to nucleate precipitates at all temperatures in the range $650^\circ - 900^\circ\text{C}$\textsuperscript{183,196}. Unfortunately, the precise nature of these reactions has not been studied in as much detail in these alloys as the processes occurring in Stage II. At short ageing times discrete precipitates are observed along dislocations. Growth of these precipitates induces large strains which can be relieved by punching of dislocations from their interface. This leads to fresh nucleation on these growth dislocations forming irregular tangled clusters. In some instances, the nucleating dislocations climb providing vacancies for growth and fresh precipitation, thus forming planar colonies of fine precipitates on $\{110\}$ planes. The latter process is sometimes observed in these alloys, although the more tangled arrays are most common. Planar arrays are more clearly observed in Cu-Ag alloys\textsuperscript{197}, which behave in a similar manner as regards the general characteristics of the precipitation reactions but more complex climb modes appear to operate. They are also observed in Fe/Cr/Ni/C alloys during the precipitation of $M_{23}C_6$\textsuperscript{198}. These types of processes occur mostly at high ageing temperatures, where growth rates are more rapid. In solution treated alloys, strains set up during cooling, due to the large differential thermal expansion between austenite and undissolved carbides cause profuse generation of dislocations locally which during subsequent ageing become decorated\textsuperscript{183}. This means that even in relatively slow cooled samples high dislocation densities, in excess of $10^9$ cm$^{-2}$, exist\textsuperscript{183}, and heterogeneous nucleation will play a very important role during precipitation.
Under certain conditions an alternative process may occur in the second stage of ageing. Climbing dislocations emanating from tangles or stringers have been observed to dissociate by the reaction of equation (2.10) producing a segment of Shockley partial dislocation and a Frank partial dislocation\textsuperscript{184,187}. The resulting configuration consists of an extrinsic stacking fault lying in a \{111\} plane \textsuperscript{184,187}. Precipitates nucleate on the Frank partial which climbs emitting vacancies to form a sheet of fine precipitates on a \{111\} plane \textsuperscript{187}. No reduction in dislocation energy accompanies this dissociation process so that the driving force must be in some way associated with the vacancy generation process. It may also be that the energy balance depends on both the line tension of the dislocation and the number of vacancies emitted per unit area of climb\textsuperscript{199}. The number per unit area for an \(a/2[110]\) dislocation is 22\% greater than for an \(a/3[111]\) dislocation, but the line tension is 50\% greater. Since these two will be linearly related in the energy balance, this difference may prove decisive providing that the 'nucleation strength' of the dislocation is sufficient. After long ageing times many patches of precipitate occur, without SF contrast, indicating that the stacking fault may be cancelled by the movement of the Shockley partial dislocation. This type of phenomenon has been observed in a wide range of Fe-Cr-Ni\textsuperscript{182-190, 192-196} and Fe-Mn\textsuperscript{191} austenites containing Nb, Ti, V, Ta carbides and nitrides as well as in Cu-Ag alloys\textsuperscript{200}. This process is often referred to as stacking fault precipitation, a term which can be misleading. Since nucleation has been shown to occur on Frank partials, a more accurate and descriptive term would be Frank Partial Precipitation (FPP). To illustrate this and the other modes of precipitation in these alloys a group of typical micrographs are presented in Fig. 2-20 (a-c) for austenitic stainless steel aged at 800\(^\circ\)C.

It is apparent from the figure that, together with the FPP and unit dislocation nucleated precipitation modes, a random distribution of precipitates exists, which has become known as 'matrix dot' precipitation (MDP)\textsuperscript{196}. This mode of
precipitation has received comparatively little attention and the nature of the nuclei is obscure. It is generally assumed that MDP is a heterogeneous mode of precipitation and that nucleation occurs either at vacancy clusters or more probably due to size factor effects, at solute-vacancy complexes.

A limited amount of work has been done to determine the kinetics of precipitation using dilatometric and electrical resistometric techniques. It is instructive to discuss these results, since apart from the useful kinetic data obtained, they also provide a firm base from which to expand the discussion to the effects of both material and processing variables on the precipitation modes. The dilatometric study was performed on two alloys containing 12% Ni, 1.0% Nb, 0.11% (C,N) and 16% Ni, 0.5% Nb, 0.06% (C,N), respectively. Precipitation of NbC from supersaturated solution is accompanied by a net volume contraction, which is proportional to the volume fraction precipitated. Over the temperature range studied (685° - 825° C) the rate of precipitation was described accurately by the Wert equation

\[ W - 1 = \exp \left[ - \frac{t}{\tau} \right]^n \]

where \( W \) is the fraction precipitated in time \( t \), \( \tau \) and \( n \) are constants for values of \( W < 0.9 \). The time exponent showed two discrete values. In the early stages of precipitation \( n = 0.75 \) while in the later stages its value was near unity. After the initial stages of precipitation an activation energy of 70 kcal/mole was obtained which was invariant at temperatures below 750° C. The early stages of precipitation were interpreted as being similar to strain ageing involving stress induced flow of solute to dislocations and the formation of fine precipitates in the dislocation core. The higher exponent during later stages was thought to relate to precipitate growth, once stacking fault growth had ceased, due to impingement (at \( W \approx 0.4 \)). An upper temperature (\( \approx 750° \)) above which FPP did not occur was observed. This was interpreted as being due to either nucleation difficulties or a change in the relative rates of self and solute diffusion at this temperature. The former would be justified on the basis of Cahn's model of precipitation due to the relative change in the strain and chemical energy terms.
as the ageing temperature changes (Appendix 2). Frank partial dislocations having smaller Burgers vectors can be favourable nucleation sites only at relatively high solute supersaturations. The latter explanation could be feasible if, with the change in diffusion rates, control for the process also changes, as it might under conditions where strain relief during growth is vacancy controlled. While the first explanation seems the most plausible the latter has some support, since only at high temperatures does dislocation punching appear to be an important accommodation mechanism. As there was a critical temperature for FPP there also appears to be a critical time or incubation period before which no stacking faults are observed as inferred earlier in the summary. These conclusions were subsequently confirmed by resistometric studies although at low temperatures (\textlessthan}700^\circ\text{C}) transient effects were observed which obscured the progress of precipitation. These were probably associated with local order reactions apparently unrelated to the precipitation process.

The ageing temperature may have a significant effect on the mode of precipitation via its effect on solute supersaturation. This has been tentatively explained (Appendix 2) in terms of the energy balance derived in equation (2.12). A higher chemical driving force or solute supersaturation is required for nucleation as the strain energy contribution of the dislocation (i.e. Burgers vector) decreases. This also explains the observation that nucleation is never associated with Shockley partial dislocations even during FPP. Similarly, the pre-aged solution treatment can significantly affect the precipitation mode through this same factor. The solute supersaturation decreases with decreasing solution treatment temperature. Subsequent FPP is observed, while for a similar alloy 1300^\circ\text{C} treatments yield profuse FPP. A similar effect may be observed if the alloy has only low Nb (\textlessthan}0.2\%) or low C and N (\textlessthan}0.02\%) contents. The latter effect also applies to MDP, which appears to be suppressed or at least less profuse in alloys with low solute supersaturations. Under all conditions below about 900^\circ\text{C} unit dislocations appear to be favoured nucleation sites. Quenching rate appears to have a significant effect on MDP, this being less profuse or absent after slow and step
cooling or direct cooling to the ageing temperature. This lends support to the view that vacancy clusters aid this mode of precipitation. Similarly, irradiation appears to enhance MDP. Quenching rate does not appear to affect the other modes of precipitation significantly except that the grown-in dislocation densities increase with increasing quenching rate. This may have an effect similar to light prestrains. Low levels of cold working (<5%) prior to ageing appear to reduce the incubation period for FPP and increase the density of stacking faults, while limiting their growth. Higher degrees of prestrain (<5%) tend to inhibit or completely suppress FPP and MDP, producing high densities of decorated dislocations. In those systems, where \(\frac{a}{2}[110]\) climb occurs, prestraining tends to suppress this process. SFE does not appear to significantly affect the precipitation mode in these alloys, FPP being observed in most alloys of the 18/8 to 20/25 types (i.e. SFE 15-55 erg cm\(^{-2}\)). However, Kotval has suggested and obtained evidence for an effect in nickel and cobalt based austenites.

At low SFE where dislocations are dissociated according to equation (2.5) the formation of FPP is unlikely, as it would also be at high SFE. At intermediate values of SFE dislocations would remain undissociated and as a consequence be capable of undergoing the reaction of equation (2.10) during precipitation.

The control and exploitation of the above processes is one of the main aspects of the present project. FPP, while producing the most efficient strengthening would prove difficult to analyse in terms of dislocation-particle interactions and as such should be avoided if at all possible.

### 2.2.3.4 Overageing and Particle Coarsening

Having considered nucleation of precipitates in some detail it is instructive to look briefly at the next stage of ageing. The nucleation rate falls during ageing and by the time peak hardness is reached it is generally assumed to have achieved a relatively low level. Peak hardness corresponds to the point at which the matrix strength reaches a maximum and is due to several competing processes, particularly in the system under discussion. In more simple
systems (e.g. aluminium alloys) it is generally accepted that this maximum occurs when the number of particles and their degree of coherency reach some optimum level. Growth of particles causes a general drop in hardness due, in the main, to the loss of coherency which generally accompanies particle growth. This process tends to occur at a rapid rate in Nb stabilised steels due to the large misfit between NbC and matrix already referred to and may in itself lead to some degree of further strengthening. The loss of coherency is generally accompanied by the punching of dislocations from the particle/matrix interface, which can strengthen the matrix by both increasing the dislocation density and nucleating new precipitates. Nucleation frequencies then fall to negligibly low levels such that further reductions in matrix supersaturation occur mainly by a diffusion controlled growth of those particles already present. This generally results in quite large degrees of matrix softening due to gross reductions in coherency strains, and is generally known as 'overageing'.

While occurring concurrently with 'overageing', the process of particle coarsening or Oswald ripening merits special attention due to its important role in annealing and creep recovery of dislocation structures in the presence of precipitate dispersions. It differs from the process described above as 'overageing' in that continued exposure at high temperatures causes a change in both particle size and density at essentially constant volume fraction.

The driving force for particle coarsening is the difference between the concentration of solute \( S_x \) in equilibrium with small particles of radius, \( r \), and that in equilibrium with larger particles. The variation of solubility with surface curvature is given by the Gibbs-Thompson equation

\[
\ln \left( \frac{S_x}{S} \right) = \frac{2\gamma}{\dot{\Omega}/K_r} \quad \ldots \ (2.14)
\]

where \( S \) is the equilibrium concentration, \( \gamma \) the particle/matrix interfacial energy and \( \dot{\Omega} \) the atomic volume. Since \( 2\gamma \Omega < K_r \) then

\[
S_x = S \left[ 1 + \frac{2\gamma}{\dot{\Omega}/K_r} \right]
\]

Generally the rate controlling process for mass transfer is matrix diffusion.
The rate of change of particle radius can be derived from the equation:

\[ 4\pi r^2 \frac{dr}{dt} = 4\pi DR^2 \frac{dS}{dR} \]

where \( \frac{dS}{dR} \) is the concentration gradient across an annulus at a distance \( R \) from the precipitate. This on integration yields

\[ \frac{dr}{dt} = -D \frac{(S - S_a)}{r} \]

where \( S_a \) is the average concentration far from the particle and \( D \) the solute diffusion coefficient. When the solubility is small the total number of atoms precipitated is approximately constant and independent of particle size distribution such that

\[ \sum 4\pi r^2 \frac{dr}{dt} = 0 \]

From these assumptions it has been shown that the variation of coarsening rate with radius is given by

\[ \frac{dr}{dt} = \frac{2DS\gamma /KTr}{[\frac{1}{r} - \frac{1}{r^*}]} \] \hspace{1cm} (2.15)

From this function it is apparent that particles of radius less than the mean radius \( \bar{r} \) dissolve at increasing rates with decreasing values of \( r \). All particles of radius greater than \( \bar{r} \) grow but the maximum rate occurs for those of twice the mean radius. Over a period of time the number of particles decreases discontinuously when particles dissolve and ultimately the system would tend to form one large particle. However, before this stage is reached the mean radius \( \bar{r} \) increases and the growth rate of the whole system slows down.

A more detailed theory than that, due to Greenwood, outlined above has been derived by Lifshitz and Slyozov, and by Wagner, taking into account the initial particle size distribution. They showed that the mean particle radius varies with time according to

\[ \bar{r}^3 - r_o^3 = 8DS\gamma /9KT \] \hspace{1cm} (2.16)

where \( r_o \) is the mean particle radius at the onset of coarsening. This result is similar to that obtained by integrating equation (2.15) in the elementary theory.

Several limitations to the Liftshitz-Slyosov-Wagner equation should be mentioned here. The first and generally ignored limitation is that of volume
fraction. These theories implicitly assume dilute dispersions or more correctly that the mean particle spacing is large compared with their radius. Hence even for a constant volume fraction extremely fine dispersions may contravene this assumption. The usually accepted upper limit of validity is 5% with the latter reservation. The alloy system studied here falls well within the region of validity on this count. Secondly, it is assumed that the total volume of precipitated phase remains constant throughout the coarsening process. This assumption is inconsistent with the Gibbs-Thompson equation (2.14) but the effect is unlikely to be significant over relatively small changes in particle size.

One of the most important limitations of this model is its restriction to single element systems. In the alloy studied here this is clearly violated, the precipitate of interest being Nb(C+N). Under such circumstances $\Omega$ must represent the molecular volume. The term $DS$ also takes on a different form. Speight and Healey have studied this effect and shown that in principle this may be quantified. Following the analytical treatment of Li, Blakely and Feingold, they showed that, by introducing a coupling force between each migrating metal atom and its associated carbon, the rate limiting process was niobium diffusion. This result is intuitively obvious in the case of stoichiometric NbC. However, where the diffusivities of the elements are similar, deviations from stoichiometry might well change the rate limiting species, although large deviations would be required in order to produce a noticeable change in experimentally determined activation energies. Speight and Healey also investigated the effect of chemical composition and showed that the apparent activation energy would include a heat of solution term for the carbide such that the apparent activation energy

$$\Delta F = -k_d \frac{\ln DS}{d(1/T)}$$

is given by

$$\Delta F = \Omega_{\text{Nb}} + \Delta H/2$$

where $\Omega_{\text{Nb}}$ is the activation energy for niobium diffusion and $\Delta H$ the heat of
solution. This relationship for $\Delta F$ applies only to stoichiometric NbC. For deviations from stoichiometry a more complex form is obtained to include the effect of differing ratios of niobium and carbon. Under these conditions $\Delta F$ will also include a temperature dependent term, since the ratio of metal to carbon atoms varies with solubility.

The degree of coherency of coarsening particles can have a significant effect on kinetics. This may arise from two sources. Firstly, the absolute value of interfacial energy is a function of the coherency of the interface, which in turn is a function of size and mismatch. Coherent interfacial energies are generally of the order of 30 erg cm$^{-2}$ (0.03 J m$^{-2}$) while incoherent interfacial energies have values close to 500 erg cm$^{-2}$ (0.5 J m$^{-2}$). The second source is that of interfacial stresses which arise because of particle-matrix mismatch. These stresses can affect both solubility and diffusivities in the matrix adjacent to a growing particle and have been considered in detail by Oriani$^{212}$ and Speight$^{213}$. As already mentioned these stresses may be relieved by dislocation punching or vacancy condensation. It is argued by Speight$^{213}$ that for the case of substitutional solutions the interfacial stress will be small and that the effect on coarsening kinetics will, therefore, be negligible.

Coarsening kinetics under conditions where transport through the matrix is rate controlling have been considered in detail. The alternative rate process can be the transfer of material across the particle-matrix interface, a process generally referred to as interface-controlled growth. Under these conditions the appropriate rate equation is$^{209}$

$$\frac{dr}{dt} = -C (S_r - S_a)$$

and leads to a coarsening equation of the form

$$r^2 - r_0^2 = 64 \frac{C \gamma \Omega t}{8kT}$$

where $C$ is an interface constant.

2.2.4 Mechanical-Thermal Treatments for Austenitic Stainless Steel

Recently the field of mechanical-thermal treatments has become increasingly active after a period of relatively low activity following the pioneering work
of Sherby et al.\textsuperscript{214} and others\textsuperscript{215-223}. This is reviewed in Appendix 1. Such treatments, usually based on working and annealing or ageing schedules, have now been developed for aluminium alloys\textsuperscript{224-226}, SAP\textsuperscript{227}, T. D. Nickel\textsuperscript{228,229} nickel alloys\textsuperscript{230,231}, low alloy steels (e.g. isoforming\textsuperscript{232,233}) and refractory metal alloys\textsuperscript{234}. These have been shown to produce significant improvements in mechanical properties. Indeed, it may now be possible to 'design' mechanical-thermal treatments which produce the optimum mechanical properties for a given material. It is unfortunate that because of the strong traditions within the steel industry, such treatments have been developed rather slowly. Consequently, the thermo-mechanical treatment of metastable austenitic steels\textsuperscript{235,236} exhibiting deformation induced martensitic transformations have apparently been favoured, due presumably to the similarity with ausforming\textsuperscript{237}. In the following the limited amount of work done in the field of MTT will be described with special reference to austenitic steels.

Apart from the early studies of the effect of cold work on the creep behaviour of austenitic steels\textsuperscript{238} the first serious study of MTT appears to have been that due to Garofalo et al.\textsuperscript{239}. Type 316 austenitic stainless steel was prestrained by varying amounts in tension at room temperature and subsequently heat treated at 483\degree C and 704\degree C to promote precipitation of $\text{M}_2\text{C}_6$ particles at dislocations. It was found that the number of particles increased with prestrain to 25%. For greater prestrains agglomeration was observed so that the density apparently decreased. The as-treated dislocation density similarly increased, though no quantitative measurements were made for either dislocation or particle densities. These simple MTT schedules were found to enhance creep properties at 593\degree C, 704\degree C and 816\degree C. For example, at a stress of 137 MPa (20,000 psi) the minimum creep rate decreased and the rupture life increased with increasing prestrain up to 30\%, beyond which these trends were reversed. A treatment consisting of prestraining by 30\% followed by ageing at 483\degree C for 24h and 704\degree C for 216h produced a 200 fold reduction in minimum creep rate and a 10 fold increase in rupture life. However,
the rupture elongation was found to decrease rapidly with prestrain from 85% without prestraining to 4.5% after the 30% prestrain treatment. Increasing the prestrain further resulted in a slow reduction in ductility to 3.0% after 50% prestrain and ageing.

Following Garofalo et al. similar treatments have been performed on AISI 348 stainless steel in which NbC precipitation occurs. Due to the similarity of this alloy with that studied in the present work it would seem advantageous to discuss the results in more detail than is merited by most of the other work described in this section. The alloy used, AISI 348 is basically a 18 Cr 11 Ni austenitic steel containing 0.6% Nb and 0.04% C by weight. However, as part of the work, certain modifications were made to this alloy composition by increasing both the Nb and C contents but maintaining approximately the same ratio. Two types of treatment, designated STMT and LTMT were considered. STMT consisted of solution treatment at 1065°C 1h air cool followed by 3% deformation at 700°C air cool. LTMT consisted of solution treatment at 1300°C 1h air cool, ageing at 700°C 5 min air cool followed by 3 or 10% deformation at 700°C (LTMT - 3 and LTMT - 10, respectively) air cool and ageing at 700°C 80h. It is apparent from the data presented below (Table 2-4) that considerable strengthening occurred after both STMT and LTMT and that LTMT produced superior strengthening at the expense of ductility. STMT which produced only a slight reduction in ductility did not exhibit stacking fault precipitation (FPP). LTMT produced a complicated substructure with FPP, the amount of which increased with Nb/C content as did the strengthening.

The stability of the LTMT-3 substructure with respect to long term exposure at 700°C is illustrated in Table 2-5 from which the potent stabilising effect of FPP is clearly apparent. The 0.2% proof stress (σ_{0.2}) decreased only slightly after 4,300h exposure as compared with the dramatic reduction for 27% cold worked material. The effect of LTMT on creep at 650°C and 207 MPa is shown in Table 2-6 from which it is apparent that LTMT reduced the creep rate (έ) by as much as an order of magnitude, increased the rupture life (t_{R}) by a factor of about 3 but produced
a dramatic reduction in rupture elongation ($\epsilon_R$). Raising the Nb/C content
(HB alloy) produced a further increase in creep resistance and rupture life.

Using treatments such as those developed by Garofalo et al\textsuperscript{239} and Levy\textsuperscript{240},
which might be considered as the first step in an MMTT schedule, it is clear
that significant improvements in properties may be obtained. These undoubtedly
arise from the combined effects of dislocation substructure and particle strengthen­
ing, though the role of each is not clear from their results.

At about the same time as Garofalo et al\textsuperscript{239}, in Russia a number of workers
notably Ivanova and Oding\textsuperscript{241}, Lozinsky\textsuperscript{252-256} and Sokolkov\textsuperscript{257-260}, were pursuing
a parallel course and much of this work has been reviewed recently by Shorshorov\textsuperscript{263}
and others\textsuperscript{264,265}. The types of treatment used specifically for stainless steels* are summarised below:

(i) Low temperature MTT (LMTT)\textsuperscript{241-251} consisting of deformation at temperatures
below or equal to the polygonization temperature ($\sim 0.4T_m$) followed by
polygonization annealing at about 600°C.

(ii) High temperature MTT (HMTT)\textsuperscript{252-262} consisting of solution treatment
followed by hot deformation at about 1000°C and ageing at about 700°C.

Turning first to LMTT it has been shown that the mechanical properties are
sensitive to the degree and temperature of prestrain, annealing temperature and
duration. While a considerable amount of work has been performed on a wide
range of materials only two stainless steels appear to have been studied. The
optimum treatments for these are shown in Table 2-8. The variation of room
temperature mechanical properties of 1Kh18N9 with treatment is shown in Table 2-9
in which untreated material is compared with the as-strained and LMTT conditions.
It is apparent from the data in the latter Table that the proof stress increases
appreciably after LMTT and that the ultimate tensile strength and ductility are
relatively unaffected. The creep properties after LMTT are compared with untreated
material in Table 2-10. LMTT produced a 5-fold decrease in creep rate, substantial

*Alloy specifications for steels studied shown in Table 2-7
Table 2-4
As-treated tensile properties at 700°C of AISI 348 stainless steel after LTMT

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Nb/C (wt%)</th>
<th>Treatment</th>
<th>$\sigma_{0.2}$ (MPa)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>$\varepsilon$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>348</td>
<td>0.60/0.04</td>
<td>1065°C 1h AC</td>
<td>214</td>
<td>283</td>
<td>33.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>STMT - 3</td>
<td>178</td>
<td>247</td>
<td>19.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1300°C 1h AC + 700°C 600h AC</td>
<td>264</td>
<td>308</td>
<td>9.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>LTMT - 3</td>
<td>317</td>
<td>344</td>
<td>10.1</td>
</tr>
<tr>
<td>HB</td>
<td>0.98/0.08</td>
<td>LTMT - 3</td>
<td>304</td>
<td>362</td>
<td>14.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>LTMT - 10</td>
<td>391</td>
<td>421</td>
<td>9.7</td>
</tr>
<tr>
<td>HJ</td>
<td>2.43/0.22</td>
<td>LTMT - 3</td>
<td>302</td>
<td>356</td>
<td>25.0</td>
</tr>
<tr>
<td>HF</td>
<td>1.96/0.17</td>
<td>LTMT - 10</td>
<td>421</td>
<td>442</td>
<td>14.0</td>
</tr>
</tbody>
</table>

Table 2-5
Effect of thermal exposure at 700°C on tensile properties of AISI 348 stainless steel after conventional treatment and LTMT

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Nb/C (wt%)</th>
<th>Treatment</th>
<th>Exposure Time (h)</th>
<th>$\sigma_{0.2}$ (MPa)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>$\varepsilon$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>348</td>
<td>0.60/0.04</td>
<td>LTMT - 3</td>
<td>0</td>
<td>264</td>
<td>308</td>
<td>9.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1000</td>
<td>258</td>
<td>325</td>
<td>14.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2650</td>
<td>238</td>
<td>297</td>
<td>12.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>4300</td>
<td>237</td>
<td>305</td>
<td>15.6</td>
</tr>
<tr>
<td>348</td>
<td>0.60/0.04</td>
<td>1065°C 1h AC + 27% CW</td>
<td>0</td>
<td>394</td>
<td>454</td>
<td>18.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>50</td>
<td>153</td>
<td>260</td>
<td>50.1</td>
</tr>
</tbody>
</table>
Table 2-6
Creep properties of AISI 348 stainless steel at 650°C and 207 MPa as a function of composition and mechanical-thermal treatment

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Nb/C (wt%)</th>
<th>Treatment</th>
<th>$\dot{\epsilon}$ ($10^{-6}$ h$^{-1}$)</th>
<th>$\varepsilon_R$ (%)</th>
<th>$t_R$ (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>348</td>
<td>0.60/0.04</td>
<td>1065°C 1h AC</td>
<td>20</td>
<td>10.1</td>
<td>324</td>
</tr>
<tr>
<td></td>
<td></td>
<td>LTMT -3</td>
<td>4.0</td>
<td>2.6</td>
<td>585</td>
</tr>
<tr>
<td></td>
<td></td>
<td>LTMT -10</td>
<td>2.0</td>
<td>1.8</td>
<td>967</td>
</tr>
<tr>
<td>HB</td>
<td>0.98/0.08</td>
<td>LTMT -3</td>
<td>1.0</td>
<td>-</td>
<td>2758</td>
</tr>
</tbody>
</table>

Table 2-7
Composition of austenitic steels studied after LMTT and HMTT

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo (wt%)</th>
<th>Mn</th>
<th>W</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>EI 481</td>
<td>0.3</td>
<td>13.0</td>
<td>8.0</td>
<td>-</td>
<td>8.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1Kh18N9</td>
<td>0.1</td>
<td>18.0</td>
<td>9.0</td>
<td>-</td>
<td>-</td>
<td>1.0</td>
<td>0.7</td>
<td>-</td>
</tr>
<tr>
<td>EI 395</td>
<td>0.1</td>
<td>15.0</td>
<td>24.0</td>
<td>6.0</td>
<td>1.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>EI 696</td>
<td>0.06</td>
<td>10.0</td>
<td>20.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3.0</td>
<td>0.4</td>
</tr>
</tbody>
</table>

Table 2-8
LMTT schedules producing optimum mechanical properties for two austenitic steels

<table>
<thead>
<tr>
<th>Alloy</th>
<th>$\varepsilon_P$ (%)</th>
<th>$T_{def}$ (°C)</th>
<th>$T_{ann}$ (°C)</th>
<th>$t_{ann}$ (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1Kh18N9</td>
<td>10.0</td>
<td>600</td>
<td>600</td>
<td>100</td>
</tr>
<tr>
<td>EI 395</td>
<td>2.5</td>
<td>625</td>
<td>625</td>
<td>50</td>
</tr>
</tbody>
</table>
Table 2-9
Comparison of LMTT with other treatments for 1Kh18N9 - room temperature properties

<table>
<thead>
<tr>
<th>Treatment</th>
<th>$\sigma_{0.2}$ (Kgmm$^{-2}$)</th>
<th>$\sigma_{UTS}$ (Kgmm$^{-2}$)</th>
<th>$\varepsilon$ (%)</th>
<th>R of A (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>23.3</td>
<td>73.0</td>
<td>57.5</td>
<td>72.0</td>
</tr>
<tr>
<td>Strained only</td>
<td>29.5</td>
<td>73.6</td>
<td>58.6</td>
<td>69.9</td>
</tr>
<tr>
<td>LMTT</td>
<td>40.2</td>
<td>79.0</td>
<td>53.6</td>
<td>68.2</td>
</tr>
</tbody>
</table>

Table 2-10
Comparison of LMTT with conventional treatment - creep properties (1Kh18N9 600°C)

<table>
<thead>
<tr>
<th>Treatment</th>
<th>$\sigma$ (Kgmm$^{-2}$)</th>
<th>$\dot{\varepsilon}$ (h$^{-1}$)</th>
<th>$\dot{\varepsilon}<em>0/\dot{\varepsilon}</em>{LMTT}$</th>
<th>$\sigma_{100}$ (Kgmm$^{-2}$)</th>
<th>$\sigma_{1000}$ (Kgmm$^{-2}$)</th>
<th>$t_{LMTT}/t_o$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td>15</td>
<td>$2.9 \times 10^{-6}$</td>
<td>5.2</td>
<td>24</td>
<td>19.5</td>
<td>20</td>
</tr>
<tr>
<td>LMTT</td>
<td>15</td>
<td>$5.6 \times 10^{-7}$</td>
<td>28</td>
<td>25.2</td>
<td>20</td>
<td></td>
</tr>
</tbody>
</table>
improvements in rupture strength (100 and 1000h levels) and life. A 20 fold increase in rupture life was observed as compared with untreated material. These improved properties after LMTT have been ascribed to the production of a stabilised subgrain structure. In the case of 1Kh18N9 X-ray diffraction analysis indicated an average subgrain size of 2μm and misorientation of 3 minutes of arc. However, as with the case of previously described work, the precise roles of the substructure and precipitation in promoting strengthening remain unclear.

Similar improvements obtained in nickel, where precipitation effects are unimportant, show the powerful effect of a thermally stabilised dislocation substructure. There is, however, conflicting evidence even here since Yim and Grant have shown that the creep properties of prestrained nickel are superior to prestrained and polygonized nickel and that prestraining adversely affects the rupture life.

HMTT has similarly been attempted in a range of materials of which nickel has been the most widely studied. As with LMTT nickel provides the special case where precipitation does not occur but significant strengthening was still observed. This presumably arises from the thermally stable substructure produced by HMTT. The treatments which produce optimum strengthening, especially at high temperatures with three austenitic steels are shown in Table 2-11. HMTT has been found to produce significant improvements in both low and high temperature tensile properties as indicated for EI 696 in Table 2-12. In addition to the appreciable improvements in strength at both 20° and 550°C as compared with a conventional solution treated and ageing treatment for this precipitation hardened alloy the ductility is not impaired by HMTT and under certain conditions may even be significantly improved. The creep properties have generally been improved by about the same amount as with LMTT i.e. 5 fold increase in rupture life. As with LMTT no definitive experiments have been performed to indicate the source of these improved properties. Consequently it is unclear whether the strengthening arises from the dislocation substructure or a superior particle distribution.
### Table 2-11

**Optimum HMTT schedules for austenitic stainless steels**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>(T_{\text{ann}}^*) (°C)</th>
<th>(t_{\text{ann}}^*) (h)</th>
<th>(\varepsilon_{\text{def}}) (%)</th>
<th>(T_{\text{def}}) (°C)</th>
<th>(T_{\text{age}}) (°C)</th>
<th>(t_{\text{age}}) (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EI 481</td>
<td>1190</td>
<td>0.5</td>
<td>25</td>
<td>1050</td>
<td>700</td>
<td>10</td>
</tr>
<tr>
<td>1Kh18N9</td>
<td>1150</td>
<td>0.5</td>
<td>25</td>
<td>1050</td>
<td>700</td>
<td>12</td>
</tr>
<tr>
<td>EI 696</td>
<td>1120</td>
<td>0.5</td>
<td>30</td>
<td>1120</td>
<td>720</td>
<td>16</td>
</tr>
</tbody>
</table>

### Table 2-12

**Tensile properties of EI 696 at 20° and 550° C after normal solution treatment and ageing and HMTT**

<table>
<thead>
<tr>
<th></th>
<th>20°C</th>
<th>550°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>NORMAL</td>
<td>HMTT</td>
</tr>
<tr>
<td>(\sigma_{0.2}) (Kg mm(^{-2}))</td>
<td>74.0</td>
<td>100.0</td>
</tr>
<tr>
<td>(\sigma_{UTS}) (Kg mm(^{-2}))</td>
<td>110.0</td>
<td>130.0</td>
</tr>
<tr>
<td>(\varepsilon) (%)</td>
<td>17.0</td>
<td>25.0</td>
</tr>
<tr>
<td>R of A (%)</td>
<td>24.0</td>
<td>40.0</td>
</tr>
</tbody>
</table>
Indeed, some interesting microstructural observations on nickel suggest that a major factor in determining creep behaviour after HMTT may be the serrated grain boundary structure, reported by Lozinsky, which appears to be a characteristic feature of HMTT material.

A similar treatment to HMTT has been studied by Gladman et al. who developed a controlled rolling procedure for austenitic stainless steels of the type studied by Levy. Controlled rolling schedules typically involve solution treatment at 1200°C for ½h followed by rolling continuously through about 10 passes with a finish rolling temperature of 800°C - 900°C and involving a total deformation of about 50% R of A. Substantial improvements in room temperature properties have been obtained, the 0.2% proof and ultimate tensile strength being raised by as much as 50 and 20% respectively. Ductilities were generally slightly inferior though this could be improved at the expense of some loss of strength by controlled softening treatments. The strengthening mechanism appears to be principally one of dislocation substructure strengthening, the subgrain size (0.2 - 0.8μm) being the most important factor. Work on AISI 316, 304 and 347 showed that strengthening could be adequately described by the Hall-Petch relationship for subgrains (see Appendix 1 equation (6)). As in the study by Levy, additions of niobium, carbon and nitrogen had a significant effect on strength which was, however, accompanied by a reduction in subgrain size. The role of Nb(CN) particles was thus not clearly defined although it might be expected from the adherence of 0.2% proof stress to the Hall-Petch relationship that the major contribution of particles was through their stabilising influence on the dislocation substructure rather than conventional dispersion strengthening.

Though it is apparent that MMTT is a completely new departure, the treatments described above provide a good indication of the degrees of strengthening achieved by methods with which the present work must be compared to assess its value, if any.

2.3 Materials Selection

The general reasons for the choice of austenitic steels have been briefly outlined in the introduction. Having made this decision, a considerable amount
of thought was given to the suitability of the wide range of possible alloy compositions on the basis of the literature survey. By this means it was hoped to choose an alloy which would benefit from the MMTT approach to strengthening, as well as being simple enough to enable the strengthening processes to be studied in detail.

Taking the first point concerning strengthening it was decided that NbC precipitation would provide a reasonable degree of dislocation locking. Doubts were expressed concerning recovery effects due to the high ageing temperatures required. It was, however, realised that in order to achieve a stabilising effect with high dislocation densities under creep conditions, low temperature ageing would be ineffective. Indeed the recovery effects during NbC precipitation might even be used to advantage in homogenising the dislocation structure.

A fairly uniform three-dimensional network of density \( \approx 10^{10} \text{ cm}^{-2} \) \((10^{14} \text{ m}^{-2})\) could theoretically be achieved by cyclic straining and ageing in a manner analogous to the programmed hardening type of MTT (see Appendix 1). The structure may, in this respect, differ from the MMTT substructures in carbon steels, which consisted of more tangled dislocation distributions characteristic of the low deformation and ageing temperatures. On the basis of the dislocation network model presented in the introduction and discussed in detail later, it is assumed that there should be a one to one correspondence of precipitates to dislocation nodes. To achieve this about 0.2% by volume of NbC would be required. This assumes 250\( \phi \) particles and a dislocation density of \( 10^{10} \text{ cm}^{-2} \). Since some excess would be required to account for insoluble particles and provide a margin for inadequacies in the model, a niobium content of 0.5 wt% was chosen.

Several factors could cause problems as regards interpretation and the alloy composition was designed to circumvent these. To prevent either \( \delta \)-ferrite formation or deformation induced marteniste transformations nickel contents in excess of 12% are required. Sigma phase formation during annealing or creep has a dramatic effect on mechanical properties and is therefore undesirable\(^{90-93}\).
To minimise this effect relatively low chromium and high nickel contents are required. In order to keep the ageing process as simple as possible controlled additions of carbon and nitrogen are necessary to 'neutralise' the niobium and form stoichiometric Nb(C+N). Too little carbon and nitrogen can lead to the formation of intermetallic phases, such as Fe$_2$Nb and Ni$_3$Nb, while too much will lead to an excess and the formation of $M_{23}C_6$ at grain boundaries. Recent research suggests that a Nb:(C+N) ratio of 8:1 is the optimum.268

Stacking fault energy is known to have a significant effect on deformation substructure$^1$ and possibly on precipitation mode$^{204}$. To prevent the formation of extended dislocations, planar dislocation arrays and FPP, a material with as high a SFE as possible is required. This could be achieved with a high nickel content (Fig. 2-12). FPP may be very effectively suppressed by using relatively low solution treatment temperatures ($\sim 1200^\circ C$).

Bearing these points in mind and following discussions with Dr. T. Gladman and Mr. J. Orr of the BSC Special Steels Division, Swinden Laboratories, Rotherham, two nominal alloy compositions were selected which are shown in Table 2-13. The first high SFE alloy (16/25) was chosen to fulfil the requirements discussed above. The low SFE alloy (16/12) was chosen in order to investigate the effect of MMTT on alloys closer to commercial compositions (e.g. AISI series of stainless steels). While the main part of the study centred around the 16/25 alloy, a short program of work was planned on the 16/12 variety but has not been carried out.
Table 2-13

Nominal composition of alloys chosen for MMTT on the basis of the materials selection criteria of section 2.3

<table>
<thead>
<tr>
<th>Alloying Element</th>
<th>Composition (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Low SFE</td>
</tr>
<tr>
<td>Cr</td>
<td>16</td>
</tr>
<tr>
<td>Ni</td>
<td>12</td>
</tr>
<tr>
<td>Mn</td>
<td>1.5</td>
</tr>
<tr>
<td>Si</td>
<td>0.2</td>
</tr>
<tr>
<td>Nb</td>
<td>0.5</td>
</tr>
<tr>
<td>C</td>
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</tr>
<tr>
<td>N</td>
<td>0.02</td>
</tr>
</tbody>
</table>
3. EXPERIMENTAL DETAILS

3.1 Material

The alloy on which most of the work in this investigation was performed was made to the afore mentioned specifications at the Swinden Laboratories of the British Steel Corporation. Two 251b ingots designated RM9209 and RM9775 were prepared. These were air melted in an induction furnace, cast and hot-forged at 1150°C to lengths of 1\(\frac{1}{2}\)" square bar which was then forwarded to NPL where the remainder of the fabrication schedule was completed. The chemical analysis of each of these alloys are shown in Table 3-1. A small quantity of AGR 20Cr/25Ni/0.5Nb alloy used as a canning material was received from Springfields. The composition of this alloy designated BV943 is also shown in Table 3-1.

3.2 Specimen Preparation

The as-received bar was cut into 9" lengths and soaked at 1150°C for 1\(\frac{1}{2}\) hour, prior to further forging. Hot forging was then carried out in order to produce slabs measuring approximately 3" wide, so obviating the need for cross-rolling at a later stage. The number of hot forging passes was four. Between each pass the material was re-heated to 1150°C for about 10 minutes. After the final hot forging operation the original 1\(\frac{1}{2}\)" bars had been reduced to 3" x 3/4" slabs, which were in a suitable form for hot rolling. Prior to rolling, all slabs were soaked at 1150°C for 25 minutes. They were then hot rolled in three stages of two passes, consisting of 1/10" nominal reduction per pass, with intermediate reheating to 1150°C for 5 minutes to about 2/10" thickness. Finally, all sheets were soaked for 10 minutes at 1150°C and hot rolled to 3 mm thickness followed by air cooling.

These sheets were then sand-blasted to remove all traces of oxide scale and cold rolled to 1 mm thick sheet without any intermediate annealing, since edge cracking did not manifest itself. Wires were also prepared from the 3 mm thick hot rolled sheet.

Sheet tensile specimens were prepared in the following manner. Strips, measuring 27 x 127 x 1 mm were cut from the cold rolled sheet in such a way as to
Table 3-1

Chemical composition of alloys used in the present investigation

<table>
<thead>
<tr>
<th>Composition</th>
<th>Alloy Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>(wt %)</td>
<td>RM 9209</td>
</tr>
<tr>
<td>Cr</td>
<td>15.70</td>
</tr>
<tr>
<td>Ni</td>
<td>24.90</td>
</tr>
<tr>
<td>Mn</td>
<td>1.20</td>
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<tr>
<td>Si</td>
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</tr>
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<td>Nb</td>
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</tr>
<tr>
<td>C</td>
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</tr>
<tr>
<td>N</td>
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</tbody>
</table>
obtain three strips from the width. These were then drilled and mounted in a specially designed jig and, using a milling machine capable of reproducing accurately the shape of a former, specimens with the shape and dimensions shown in Fig. 3-1 prepared. Final finishing was achieved using a fine file and emery cloth. These finished specimens had their tensile axis parallel to the rolling direction. To investigate the effect of inclusions on properties some specimens were cut from sheet with their tensile axis perpendicular to the rolling direction.

Prior to mechanical tests or MMTT most specimens were solution treated in the range 1200° - 1300°C. Because of the rather fragile nature of the sheet tensile specimens, the encapsulation method using sealed silica tubes under argon or vacuum was thought inappropriate. No simple means could be devised for ensuring that the specimens did not bend and the sintering of batches of specimens in the same capsule was equally likely to present problems. An alternative method of producing batch solution treatments of about a dozen specimens per heat treatment was sought. One of the most difficult problems to overcome was the requirement that cooling rates of about 20°C sec\(^{-1}\) were necessary to avoid reprecipitation of Nb(C,N) during cooling. To overcome these problems a furnace was designed which enabled specimens to be heat-treated in batches of a dozen in an inert atmosphere and subsequently cooled in a chamber under a blast of cold argon gas.

The main body of the furnace consisted of a 65 mm diameter tube with water cooled end pieces set in ceramic bricks and heated by 6 Crusilite resistance elements. A flow of pure argon gas or argon-hydrogen gas mixture was maintained through the whole of the system. A sliding 'door' was provided between quenching chamber and furnace tube to permit access to the latter during heat treatment and allow the chamber to be sealed off from the furnace during gas quenching. This prevented cold gas entering and possibly cracking the furnace tube. It consisted of a 90 x 150 mm sheet of 1 mm thick stainless steel with a 65 mm diameter hole in its geometric centre. This 'door' slid between 'O' rings set into the end pieces of the furnace tube and the quenching chamber. It could be raised by a foot.
pedal via a pulley system to seal off the chamber. The quenching chamber was mounted on a mobile framework and clamped, holding the door tightly in position, with springs attached to the furnace. This provided a good gas tight seal if vacuum grease was applied liberally to the 'O' rings. The quenching chamber consisted of a 250 mm long, 65 mm diameter brass tube (1.5 mm wall thickness) with end pieces and 'O' ring seals. This was in intimate contact with a water cooling coil wrapped around it. Gas could be fed, under pressure, into the chamber via 2 mm copper tubes three of which were placed vertically above and below the position occupied by the specimen cradle during quenching. Two gas outlets of 5 mm diameter were provided to the rear of the chamber. Both the inlet and outlet tubes were connected to a servo-pneumatic valve assembly which allowed gas to pass only when the door was closed and a microswitch operated. The nimonic alloy specimen cradle was connected to a long length of hollow stainless steel rod through which a thermocouple could be fed to monitor the specimen temperature. This rod, which was used to push the specimens into the hot zone and pull them back into the chamber, was set in a sliding seal. The latter consisted of a rigidly clamped ring of PTFE set in an assembly mounted in the end plate of the chamber. It provided a gas tight seal during the heat-treatment but was badly burned as the specimen boat entered the chamber prior to quenching and therefore a fresh ring had to be fitted before each heat treatment. Specimens were suspended in the boat from nimonic rods inserted through the bolt holes in their heads. Ceramic spacers were used to separate the specimens and prevent sintering. Ceramic rollers were placed across the boat to support the specimen gauge lengths.

A typical sequence of operations of this equipment was as follows. One dozen specimens were hung in the boat and placed in the chamber. The end piece was screwed on and the PTFE seal fixed in position. Usually the furnace would be at the required temperature and sealed off from the chamber with an inert atmosphere. The chamber was then flushed out with pure argon for about 3-4 minutes and the sliding door opened. This automatically turned off the valve and the furnace
gas passed through the whole system. The specimen boat was then pushed into the
furnace hot zone for the required period of the heat treatment. A temperature
gradient of +6°C existed over the length of the boat but over the specimen gauge
lengths this did not exceed +1°C either along each specimen or from one to another.
Because of the high heat capacity of the furnace the desired steady temperature
was reached within 5 minutes and maintained to within ±0.5°C. After the appropriate
period of time the specimen boat was returned to the chamber, the door rapidly
closed and pure argon gas passed through at a pressure of 1.5-2 atmospheres.
This produced an average cooling rate of between 25°C and 35°C sec⁻¹ over the range
1200°C-500°C. The burning of the sliding seal as the boat was retrieved from the
furnace allowed a small amount of air to enter prior to quenching which, however,
only produced slight tarnishing of the specimens.

For some of the preliminary work wires were produced from the hot-rolled and
sand blasted 3 mm sheet. Strips approximately 3 mm wide and 80 mm long were cut
from this sheet and machined to 2.5 mm diameter rods. These were then swaged to
0.75 and 1.0 mm diameter wire and cut into 330 mm lengths, which were annealed
in a direct resistance heating rig, under vacuum. The temperature of the wire
was monitored using an optical pyrometer and was maintained at 1350°C for 1 hour.
During heating and cooling of the wire the length change could be compensated for
in order to prevent undue plastic deformation. This equipment is commonly used
for heat-treating internal friction specimens and has been described in detail
elsewhere. Solution treated wire was then cut into 80 mm lengths and tested
in a Hounsfield 'E' type Tensometer, using grips and other attachments described
elsewhere, at a strain rate of 5 x 10⁻⁴ sec⁻¹.

3.3 Mechanical Testing

3.3.1 Tensile Testing

Both MMTT straining and subsequent tensile tests were performed on a
Hounsfield 'E' type Tensometer in a rig designed to facilitate testing throughout
the temperature range 20°C - 900°C. This rig together with the relevant attachments
is shown in Fig. 3-2 set up for ambient temperature testing. Briefly, the equipment
consisted of a rigid brass framework fixed to the base of the testing machine using conventional fittings. This frame was equipped with four screw adjustors to facilitate accurate alignment of the specimen with the tensile axis of the machine. A similar facility was available with the cage slung from the underside of the framework. This consisted of four stainless steel rods set in a square array, bolted rigidly to the framework at its top and a thick stainless steel plate at its base. The specimen, held in grips as shown in Fig. 3-3, together with a simple mounting jig, was fixed at one end to this base plate and at the other to a load cell mounted on the machine cross-head, via a connecting rod which passed through a hole in the top of the framework. Around each of the cage supports and the connecting rod was placed a water cooling jacket. A three-zone furnace was used during hot-tensile tests and this can be seen in the space below the specimen cage in Fig. 3-2. At the start of such a test, this furnace, which was fixed rigidly to a pair of vertically mounted parallel bars by means of plumbers blocks at the rear of the machine, was raised into position with the cage and specimen immersed in the hot-zone. This design employing the cage and base mounted framework was chosen since it has the advantage that, should overnight tests at low strain rates be required, this could be accomplished with no danger of damage being caused to the furnace tube. Furnace temperature control was achieved using a W. H. Mayes three-zone three-term controller. This was specially designed to give a high power output and fast response to eliminate temperature overshoot problems. With this system the temperature could be maintained to within $\pm \frac{1}{2}^\circ C$ and a gradient of $\pm 1^\circ C$ achieved along the specimen gauge length after approximately 5 minutes heating for temperatures in the range $800^\circ - 850^\circ C$. If electron microscopy was required following hot-tensile testing the specimen was cooled under relaxed load by stopping the cross-head movement and lowering the furnace. In hot-tensile tests to failure the test was continued until a distinct decrease in load was observed and the cross-head stopped. The stress acting on the specimen relaxed under these conditions to some constant value.
If, at the same moment the cross-head movement was stopped, the furnace was
removed, the specimen cooled, generally, under a stress of approximately 70% of the
original applied stress. The cooling rate was such that the specimen temperature
dropped from 800°C to below 500°C within about one minute.

3.3.2 MMTT - Practical Details

MMTT prestraining was carried out at room temperature on the Hounsfield
tensometer described earlier at a strain rate of $3.3 \times 10^{-3} \text{ sec}^{-1}$. Initially
specimens with scribed gauge marks were tested to estimate the real elongation
on a 50 mm gauge length and calibrate this against the total specimen elongation
determined from the machine chart recorder. Though this varied slightly with
strain during tensile deformation it was possible to obtain a reasonable calibration
over a wide range of stress and strain. It was found from these tests that the
ratio of real to total strain applicable to most tests was 0.925. During MMTT
specimens were elongated by an amount corresponding to a given strain, typically
2, 5 or 10%, on the first cycle. Consequently, in subsequent cycles prestraining
to the same nominal elongation resulted in an effective reduction in the
true strain per cycle as MMTT proceeded. During the first cycle it was often
found that the yield stress and work hardening rate varied from one specimen
batch to another. This could lead to a slight though significant difference in
subsequent MMTT behaviour. Consequently, it was decided to strain all MMTT
specimens to the same stress level in the first cycle. This appeared to improve
the treatment reproducibility if a constant strain were applied in each subsequent
cycle and this procedure became standard practice. A computer program was
devised (Appendix 3) to calculate the variation of true stress and strain during
MMTT cycling, taking into account the above factor and allowing the strains in each
cycle to be added together to estimate the total accumulated prestrain during each
treatment. MMTT ageing during each cycle was performed in a silica tube furnace
under dry hydrogen at 1 atmosphere.

A loading jig was used to allow tensile grips to be removed from specimens
during MMTT ageing without damage. This is shown in Fig. 3-3 together with specimen
and grips.
A series of high temperature MMTT runs were performed using the hot tensile facility described earlier (3.3.1). In these treatments straining was performed at the ageing temperature and ageing was carried out either in the stress relaxed or unloaded conditions as discussed in a later section (4.2.6).

3.3.3 Creep Testing

Creep tests were carried out on the sheet specimens described earlier and used for MMTT. These were conducted on Mayes constant load, lever creep machines with standard fixtures and fittings including extensometers. Strain was measured using a pair of capacitance transducers capable of resolving $5 \times 10^{-6}$ in and recorded using the NPL Creep Laboratory data logging system. Temperature was controlled with a Mayes three-zone three-term controller to $\pm 0.1^\circ C$ and the gradient to not more than $\pm 1.0^\circ C$ over the specimen gauge length.

Because of the incompatibility of extensometers with specimen geometry, these being designed for cylindrical bar specimens with machined ridges or knife-edges, sets of adaptors were designed to overcome this problem. These are shown in Fig. 3-4 together with a specimen and Nimonic 80A grips of standard NPL design. The adaptors consisted of 1.0 mm thick 4.5 mm radius semi-circular discs of Nimonic 80A with an accurately machined knife edge around the circumference. These were mounted by means of a press fitting into 2.0 mm thick 25.0 x 5.0 mm stainless steel strips. Pairs of these were clamped, one at each end of the gauge length, across the specimen thickness. The radius of the discs was such that it was identical to the knife edges on the standard cylindrical specimens for which the extensometers were designed. Tests performed on these adaptors by J. S. Herd et al indicated that in variable load tests there was less backlash than with alternative extensometer systems for sheet specimens. Experience showed that each set of adaptors could be used for a number of tests or single tests to in excess of 2000h and inspection of the knife edges revealed that no bending or warping of the knife edges had occurred. All creep specimens were measured, using a travelling microscope with drum micrometer, both before and after testing and extensometer measurements were found to agree with these changes to within 2%.
The clamping system was found to have a negligible effect through stress concentrations and almost always specimens ruptured near to the centre of the gauge length and not at the adaptors.

In creep tests, especially of MMTT material, it is desirable to establish a routine method of testing particularly with respect to initial heating and loading procedures. Variations in the rate of heating to the creep temperature are probably one of the prime sources of scatter in results where recovery or ageing processes can occur. This, of course, applies to tests on both MMTT and solution treated material. The method adopted was to preheat the creep furnace to 50°C above the desired temperature and lower it into position around the specimen. The control temperature was then set and the furnace allowed to come to equilibrium. A steady temperature and gradient were usually obtained within 1h. This procedure was performed under a residual load of 50N (~5.0 MPa) to avoid misaligning or twisting the specimen while asbestos wadding was packed around the base and top of the creep furnace. This alignment was checked prior to heating by elastically loading the specimen and adjusting the grips until the two transducers, one on either side of the tensile axis, gave identical displacements which agreed with those predicted from the elastic modulus of this type of steel.

Variable stress creep tests were performed to measure recovery creep and internal stress parameters. Inconsistencies with other measurements, mainly electron microscopy, and a realisation that anelastic relaxation effects made an important contribution to the strain transients observed, lead to a more complicated series of tests and procedures. These are dealt with later in the relevant section (Appendix 4) together with results and discussions.

3.4 Electron Microscopy

3.4.1 Extraction Replicas

Due to the inadequacy of published solubility data a short program of preliminary work was undertaken to determine the optimum solution treatment conditions. Optical metallography proved inadequate for niobium carbide volume fraction determinations
due to low resolution. Thin foil electron microscopy at 100 kV was also found to be unsatisfactory due to preferential etching in the vicinity of second phase particles. HVEM work was not possible at the time due to 'teething troubles' but the findings of extraction replica studies were subsequently checked by this technique.

Extraction replicas were prepared in the following manner. Samples were mechanically polished as for optical microscopy (to 0.5 \( \mu \)m \( \gamma \)-alumina paste) and then electropolished as for thin foil specimens to ensure a relatively stress-free surface. They were then etched electrochemically for 60 sec in a 7% hydrochloric/3% nitric acid solution at 2 volts and 25°C. A carbon layer \( \approx 500 \AA \) thick was then deposited onto their surfaces in an evaporating unit. The coatings were cut into a number of 1 mm squares and etched in the above solution until the carbon film peeled off and floated to the surface of the liquid. The pieces of replica with the particles imbedded in them were then collected on electron microscope grids and mounted in a conventional specimen holder for inspection in an AEI EM6G microscope operating at an accelerating voltage of 80 kV. A large number (\\( \approx 40 \)) micrographs were taken at random from at least 3 replicas per solution treatment condition at low magnifications (\\( \approx 4000x \)). The area fraction of carbides per micrograph was measured directly from the negatives using a Quantimet with an epidiascope attachment. To avoid making rather doubtful assumptions concerning the volume from which particles were extracted, it was decided to compare solution treated specimens with some standard condition where it could be assumed that all niobium carbide was precipitated. Thus the quantity of niobium carbide after a given solution treatment and hence solubility could be determined by taking ratios of area fractions in each condition with the standard.

Advantage was taken of this work with extraction replicas to determine the nature of carbides and other substances in this material. Consequently, a large number of particles, found fairly randomly throughout the work were analysed by electron diffraction. For this particular piece of work the camera constant
of the microscope was measured using a pure iron specimen of known lattice parameter and care was taken to ensure that as near as possible constant illumination and focusing conditions were used at all times.

3.4.2 Thin Foils

The major portion of the electron microscopy was performed on thin foils prepared from 1 mm thick sheet material. Specimens from the 10 mm wide gauge length of tensile test pieces were cut in 10 mm lengths and lapped to 0.125 mm (0.005 in) from both sides using an automatic lapping machine. Carrington, using an etch pitting technique, has shown that in Fe-3% Si alloy the mechanical damage introduced by this method only extended 0.025 mm beneath the surface. The production of thin foils from these specimens was accomplished electrolytically using a modified Bollman technique. Samples were polished in a 64% phosphoric/15% sulphuric acid/21% water solution at 9.5 volt and 25°C. These were held in tweezers and the edges coated in lacquer to prevent edge attack. They were then placed vertically between two pointed cathodes set approximately 7.5 mm apart. Thinning was continued until two or more holes appeared and the region between them perforated. Thin areas around the holes were cut from the sample with a sharp razor blade. In order to check that this foil handling did not introduce deformation or change in the dislocation structure, on some samples thinning was continued until small pieces fell to the bottom of the thinning bath. These were collected and, like the cut foils, mounted between copper grids for examination in the electron microscope. Thin foils were stored in gelatine capsules and desiccator prior to electron microscopy. It was found that, in this manner, atmospheric contamination of the foils could be avoided for long periods of time.

3.4.3 Quantitative Electron Microscopy

A number of parameters were measured in order to characterise the MMTT substructure. Dislocation and particle densities were measured on large numbers of electron micrographs in order to obtain values of these parameters which were as near as possible characteristic of the bulk material. A program of work was conducted in collaboration with Professor Y. Ishida in which small volumes of material were studied in several orientations and using a variety of imaging
conditions to produce accurate information on the local three dimensional
distribution of dislocations and particles. The latter will be discussed in a
separate section (4.3) together with the relevant experimental results.

The central and possibly most important parameter in any quantitative electron
microscopy study is undoubtedly the foil thickness. In most work this has been
poorly defined and consequently leads to a wide scatter in results which is often
greater than the errors involved in the counting techniques themselves. Simple
assumptions about average foil thicknesses seem completely inadequate particularly
if structures of widely differing dislocation and particle densities are to be
compared. If systematic variations in dislocation density are being studied,
as for example with the number of MMTT cycles and with annealing, it should be
appreciated that the usable thickness tends to decrease with increasing dislocation
density. Variations in instrument illumination and screen brightness can also
give rise to variations in thickness of examined foils. Furthermore, it is to
be anticipated that these errors will be, at best, the same or considerably
larger in HVEM unless a more rigorous approach is adopted. In the present work
a simple though time consuming alternative was adopted. Instead of the average
thickness measurement approach, the thickness of each area observed for dislocation
density measurements was determined so giving much better values of the total
volume of material from which each individual density determination was made.
This was achieved using a variety of techniques, each of which was found to be
useful under specific conditions. With low dislocation densities (<10^{10} \text{ cm}^{-2}),
where thickness fringes could be clearly observed at twin boundaries, the extinction
distance technique was favoured. Multi-beam extinction distances were calculated
by B. Gale$^{273}$ for voltages in the range 100-1000 keV. Experiments have been
performed to determine the variation of extinction distances at the symmetry
position with accelerating voltage by measuring twin boundary fringe contours. Very
good agreement with computer calculations was obtained$^{274}$. An analytical solution
to the three-beam dynamical theory of diffraction contrast was also obtained. This
proved to be a good approximation to multi-beam extinction distances for the case of symmetrical systematic diffraction conditions, which, for low index reflections, gave optimum contrast at high voltages. If the first and second order two beam extinction distances at 100 keV are known, the effective multi-beam extinction distance at a given accelerating voltage \( \Lambda^V \) is given by

\[
\Lambda^V = \xi^V_g V^{-2} - \sqrt{\frac{s^V}{g} - \xi^V_{2g} V^{-2}}
\]

where \( \xi^V_g \) and \( \xi^V_{2g} \) are the first and second order relativistically corrected two beam extinction distances and \( s^V_g \) is the deviation of the symmetry position from the Bragg condition. This is found to agree to within better than 2% with the more accurate computer calculation. Having obtained reasonable agreement with experimentally determined values\(^2\) these were subsequently used in the dislocation and particle density measurements. Experience with the technique, as well as calculations associated with the effect of orientation, indicated that high accuracy (\( \pm 5\% \)) could be obtained providing specimens were oriented to the symmetry position for the low order systematic reflections \(<11\overline{1}>\ and \<200>\). This could be achieved with Kikuchi lines and the accuracy improved with increased accelerating voltages. Under conditions where the foil was known to be within 5° of the horizontal position the trace method was employed. Stereo tilting was the other widely applied alternative and provided large enough tilts (>20°) were employed, a similar degree of accuracy could be obtained.

The gain in useful penetration under normal operating conditions with stainless steel specimens has been studied. The criterion used has been that of visibility and the ability to focus an image. Results, in the light of the more accurately known extinction distances etc, suggest an over estimate by previous workers. At 100 keV penetration is \(~0.30\ \mu m\); at 400 keV it is \(~0.65\ \mu m\) and at 1 MeV it is \(~1.1\ \mu m\). This is in close agreement with the theoretical \( v^2/c^2 \) relationship, which had recently gone into disrepute. Typical average thicknesses employed in the present work were 0.2 \( \mu m \) at 100 KeV, 0.45 \( \mu m \) at 400 KeV and 0.75 \( \mu m \) at 1 MeV.
Dislocation densities were measured by the method of Ham. A series of random lines of total length \( L \) were drawn forming a grid over an area of a micrograph and the number \( N \) of intersections of these lines with dislocations were counted. The dislocation density \( \rho \) was then obtained from the expression

\[
\rho = \frac{2MN}{Lt} ... (3.1)
\]

where \( M \) is the real magnification of the micrograph and \( t \) the specimen thickness.

The particle densities were measured, generally on the same micrographs as dislocation densities and consequently the same foil thickness values were employed. Since the particle size was small being generally less than 0.1 \( \mu m \) the total number of particles \( N' \) in an area was counted and the volume number density obtained from the relationship

\[
N_v = \frac{N'M^2}{At} ... (3.2)
\]

where \( A \) is the area of micrograph on which the measurement was made and \( M \) once again the real magnification. Certain modifications to these methods of measuring dislocation and particle densities were made in the three dimensional analysis performed in collaboration with Professor Ishida (section 4.3).
4. EXPERIMENTAL RESULTS

4.1 Preliminary Studies

The following series of short programs were conducted prior to the main part of the project in order to assess certain material parameters which were essential to the planning of subsequent work on MMTT.

4.1.1 NbC Precipitate Solubilities

The importance of solution treatment conditions in controlling precipitation mode has been emphasised in the literature review (2.2.3). One effect which has been neglected is that on grain size. The temperatures required to obtain high levels of NbC solubility are within about 200°C of the melting point of the present alloy and consequently rapid grain growth can occur. However in the presence of undissolved second phase particles relatively fine grain sizes can be retained. Once the dissolution of particles occurs rapid grain coarsening commences.

Having decided to use 1 mm thick sheet specimens a limit was effectively placed on the maximum grain-size in order to achieve consistent specimen to specimen mechanical properties. This is especially true of ductility. The limit would appear to be exceeded if less than about 10 grains are present in the specimen cross-section.

The purpose of this program was to measure grain-size as a function of solution temperature and, because of the discrepancy between NbC solubilities reported in the literature, obtain values of this parameter for the material under study. From these results optimum solution treatment conditions could be defined bearing in mind the compromise that must be made between niobium/carbon (and nitrogen) solubility, grain-size and subsequent ageing behaviour.

In order to find the optimum conditions a number of 1 cm square 1 mm thick samples were solution treated at temperatures in the range 1200°C - 1350°C for various times. Grain-sizes were determined on optical sections, which were mechanically polished and etched as in the first stage of replica preparation for which they were subsequently used. The measurements were made by a linear intercept method. This was accomplished using a light microscope adapted to allow
a specimen to be traversed automatically at a suitable rate and counting the
number of grain boundaries in a certain length of traverse. For each specimen
three or four traverses were made and approximately 500 grains counted. The mean
grain diameters determined by this technique are presented in Table 4-1. Approximate
values of maximum observed grain diameters are also tabulated. A typical
micrograph showing the general features of the microstructure after the 1h 1200°C
treatment is presented in Fig. 4-1. The range of grain size indicated in Table 4-1
is clearly apparent, as is the relatively high twin frequency (~2 twins/grain) and
inclusion content. This latter parameter was 1.5 and 1.4 vol % in RM 9209 and
RM 9289 respectively and 0.2 vol % in BV 943. Electron probe microanalysis revealed
that the dark inclusions were complex chromium-aluminium-silicon oxides, while
optical activity indicated that many of the light variety were silica.

Solubility measurements were made using extraction replicas by the method
outlined earlier. The standard or reference condition, against which each solution
treated sample was to be compared, was chosen with regard to two main factors.
Firstly, it was necessary to ensure that all NbC was in fact precipitated and
secondly, that the volume of material from which particles were extracted remained
constant from specimen to specimen. The limiting factor in the latter context was
the particle size distribution since the size of the largest particles ultimately
controlled the volume of material which had to be dissolved before the replica
could be floated free of the surface. Smaller particles tended to be attracted to
the replica film while not actually being embedded in it. To fulfil both these
criteria a sample solution treated at 1200°C for 1h and slow cooled to 900°C over a
period of 6h was used as the standard. From the chemical analysis this sample
should theoretically have contained 0.7 vol % NbC. Its area fraction per micrograph
was therefore measured and the volume fraction of each sample determined by
comparison with this standard. The relative values of mean area fraction per
micrograph, estimated volume fraction and solubility products are shown in Table 4-2.
The last parameter was calculated, together with the wt % Nb in solution, assuming
NbC occurred as a stoichiometric compound. This assumption is valid at least in terms of overall chemical composition. The solubility products for the longer times at each of the temperatures are plotted in Fig. 4-2 together with the data obtained by other workers\(^{280-292}\). The cause for confusion as regards reliable solubility data, if published data is consulted, is readily appreciated from the order of magnitude scatter. Much of this scatter has been explained recently\(^{293}\) in terms of deviations from stoichiometry due to varying niobium, carbon and nitrogen contents in the alloys studied. The data determined here seems to be in fair agreement with that determined by chemical analysis techniques\(^{280-282}\). Since this work was never meant to provide very accurate data, but to indicate the most likely range, in all subsequent calculations the data of reference 281, with which these results are in good agreement, was used. This gave a solubility product

\[
\log_{10} [\text{Nb}][\text{C+N}] = \frac{-8850}{T} + 4.35 \quad \cdots (4.1)
\]

where the concentrations are in wt % and \(T\) is the temperature (°K) and was determined on a similar alloy.

Clearly the requirement that approximately 70% of the Nb be in solution is fulfilled by each of these treatments. However, returning to grain-size it is apparent that treatments in excess of 1250° C promote very considerable grain coarsening. The relationship between measured grain-size and insoluble NbC volume fraction can be appreciated from Fig. 4-3. This shows a reciprocal relationship of mean grain-size with particle fraction as predicted by the Zener model\(^{294}\). The compromise between grain size (<0.1 mm) and solubility (>70% total Nb) appears to be obtained quite adequately with the 1h solution treatment at 1200° C. This treatment was consequently used throughout the work except where specified to the contrary.

4.1.2 Tensile Properties

4.1.2.1 Low Temperature Properties

The room temperature tensile properties of all solution treated specimens used in subsequent deformation, ageing or MWTT studies are summarised, together
### Table 4-1

Grain sizes after solution treatment of RM 9209

<table>
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<th>Treatment</th>
<th>Mean Grain Diameter (mm)</th>
<th>Maximum Grain Diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200°C 1hr</td>
<td>0.05</td>
<td>0.10</td>
</tr>
<tr>
<td>1200°C 2hr</td>
<td>0.08</td>
<td>0.20</td>
</tr>
<tr>
<td>1250°C ½hr</td>
<td>0.08</td>
<td>0.25</td>
</tr>
<tr>
<td>1250°C 1hr</td>
<td>0.14</td>
<td>0.45</td>
</tr>
<tr>
<td>1300°C ¼hr</td>
<td>0.15</td>
<td>0.40</td>
</tr>
<tr>
<td>1300°C ½hr</td>
<td>0.20</td>
<td>0.70</td>
</tr>
</tbody>
</table>

### Table 4-2

Solubility data for RM 9209 determined from extraction replicas

| Treatment        | Area Fraction/Micrograph (%) | Vol. % | Wt % Nb in soln | Solubility Product
<table>
<thead>
<tr>
<th></th>
<th></th>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1200°C Slow Cool</td>
<td>11.38</td>
<td>0.7</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1200°C 1hr</td>
<td>3.27</td>
<td>0.20</td>
<td>0.36</td>
<td>0.027</td>
</tr>
<tr>
<td>1200°C 2hr</td>
<td>2.89</td>
<td>0.18</td>
<td>0.37</td>
<td>0.028</td>
</tr>
<tr>
<td>1250°C ½hr</td>
<td>3.52</td>
<td>0.22</td>
<td>0.33</td>
<td>0.025</td>
</tr>
<tr>
<td>1250°C 1hr</td>
<td>1.79</td>
<td>0.11</td>
<td>0.42</td>
<td>0.0315</td>
</tr>
<tr>
<td>1300°C ¼hr</td>
<td>1.92</td>
<td>0.12</td>
<td>0.41</td>
<td>0.031</td>
</tr>
<tr>
<td>1300°C ½hr</td>
<td>0.51</td>
<td>0.03</td>
<td>0.48</td>
<td>0.036</td>
</tr>
</tbody>
</table>
with grain size and specimen dimensions, in Table 4-3. These provide essential background data against which aged and MMTT specimens must be compared, as well as indicating some of the effects of grain-size/specimen diameter ratios mentioned in previous sections.

The first point to be noted is the discrepancy between the tensile properties of wire and sheet specimens. The 1300°C treatment in each case should be compared. Proof stress, ultimate tensile strength (UTS) and ductility were inferior in the wire specimens, although the properties of the larger diameter wires were slightly improved. The reason for this may lie in the statistical distribution of grain-sizes. Reference to Table 4-3 shows that with thicknesses of only 0.75 - 1.00 mm it is highly probable that at some point in the specimen gauge length only one or possibly two grains lie in the specimen thickness. This will clearly influence the ductility and affect the wires more significantly than the sheet specimens which were 10 mm wide. The reason for the discrepancy in UTS may well reflect this trend due to the complex inter-relationship between ductility, work-hardening exponent and UTS via Considère's principle and the almost single crystal nature of the wires. This discrepancy was, however, not considered too important since in the work with wires relative flow stress increases during ageing were to be studied and meaningful overall mechanical behaviour was not desired.

With sheet specimens the effect of solution-treated grain size on ductility is quite apparent as is a corresponding effect on UTS. Another point to be noted is the improved ductility of BV943, the double vacuum melted alloy from British Nuclear Fuels. This improved ductility is probably related to the lower inclusion content in this alloy (0.2 vol % as opposed to 1.5 vol % for RM9209) and their finer distribution. The difference is clearly apparent in Fig. 4-4. The proof stress of these alloys and treatments is, however, little affected. In the case of the 1200°C treatments, which produced similar grain sizes in RM9209, 9289 and BV943 this is to be expected. However, the large difference in grain-size between 1200°C and 1300°C treatments would be expected at first sight to produce a noticeable
<table>
<thead>
<tr>
<th>Property</th>
<th>Wire Specimens</th>
<th>Sheet Specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(1300°C 1/2 hr. ST)</td>
<td>(1200°C 1 hr. ST)</td>
</tr>
<tr>
<td></td>
<td>(RM9209)</td>
<td>(RM9209) (BV 943)</td>
</tr>
<tr>
<td>Grain Size (mm)</td>
<td>0.170</td>
<td>0.044</td>
</tr>
<tr>
<td></td>
<td>0.170</td>
<td>0.048</td>
</tr>
<tr>
<td></td>
<td>0.044</td>
<td>0.048</td>
</tr>
<tr>
<td></td>
<td>0.048</td>
<td>0.195</td>
</tr>
<tr>
<td>Diameter (mm)</td>
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<td>(Thickness)</td>
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<td></td>
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<tr>
<td>Hardness (HV)</td>
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<td>124</td>
<td>133</td>
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<td></td>
<td>131</td>
<td>133</td>
</tr>
<tr>
<td></td>
<td>130</td>
<td></td>
</tr>
<tr>
<td>0.1% PS (MPa)</td>
<td>146</td>
<td>160</td>
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<tr>
<td></td>
<td>142</td>
<td>165</td>
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<td>165</td>
</tr>
<tr>
<td></td>
<td>155</td>
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</tr>
<tr>
<td>0.2% PS (MPa)</td>
<td>162</td>
<td>174</td>
</tr>
<tr>
<td></td>
<td>154</td>
<td>178</td>
</tr>
<tr>
<td></td>
<td>174</td>
<td>178</td>
</tr>
<tr>
<td></td>
<td>170</td>
<td></td>
</tr>
<tr>
<td>2.5% PS (MPa)</td>
<td>220</td>
<td>281</td>
</tr>
<tr>
<td></td>
<td>224</td>
<td>288</td>
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<tr>
<td></td>
<td>281</td>
<td>288</td>
</tr>
<tr>
<td></td>
<td>268</td>
<td></td>
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<tr>
<td>10% PS (MPa)</td>
<td>386</td>
<td>480</td>
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<tr>
<td></td>
<td>400</td>
<td>485</td>
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<tr>
<td></td>
<td>480</td>
<td>485</td>
</tr>
<tr>
<td></td>
<td>458</td>
<td></td>
</tr>
<tr>
<td>UTS (MPa)</td>
<td>550</td>
<td>720</td>
</tr>
<tr>
<td></td>
<td>605</td>
<td>750</td>
</tr>
<tr>
<td></td>
<td>650</td>
<td></td>
</tr>
<tr>
<td>Maximum Uniform Strain (%)</td>
<td>20.3</td>
<td>31.4</td>
</tr>
<tr>
<td></td>
<td>21.8</td>
<td>36.4</td>
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<td></td>
<td>31.4</td>
<td>36.4</td>
</tr>
<tr>
<td></td>
<td>23.8</td>
<td></td>
</tr>
<tr>
<td>True Strain</td>
<td>0.185</td>
<td>0.273</td>
</tr>
<tr>
<td></td>
<td>0.197</td>
<td>0.310</td>
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<tr>
<td></td>
<td>0.273</td>
<td>0.310</td>
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<tr>
<td></td>
<td></td>
<td>0.215</td>
</tr>
</tbody>
</table>
difference in proof stress. From Irvine et al's\textsuperscript{295,296} data the grain-size difference observed here should produce a difference of ~11.5 MPa. According to their work the amount of niobium, carbon and nitrogen in solid solution should be balanced against this. Assuming stoichiometric Nb(C,N) ratios and using the solubility data presented in Table 4-2 the former should just balance the proof stress change due to grain size, being ~12 MPa. Calculations of proof stress and UTS, using the chemical composition, grain size and solubility data for RM 9209 and Irvine et al's regression equations\textsuperscript{296}, are in fair agreement with these results. The UTS and uniform elongation agree with results obtained by Sanderson and Llewellyn\textsuperscript{297} for similar alloys.

4.1.2.2 High Temperature Properties

Hot-tensile tests were conducted on sheet tensile specimens in the solution treated and "stabilised" conditions. The latter refers to those solution treated specimens which were aged at 800\textdegree C for 50 hr prior to testing. Tests have been performed under conditions where precipitation may occur during testing, so contributing to strengthening by a dynamic strain ageing process, and in "stabilised" material where such a process has a negligible effect. These comparative tests were performed at the same strain rate and temperature. Other tests were carried out on the solution treated specimens in order to assess the effect of strain-rate and temperature. Creep tests after the above treatments and a variety of single prestraining and ageing treatments have also been conducted over a range of stresses at 800\textdegree C.

By way of introduction the typical hot-tensile behaviour of solution treated and stabilised specimens are shown in Fig. 4-5. Several differences are apparent from the figure. Yield stress, work hardening rate and ultimate tensile stress are very much higher in the solution treated case, reflecting the significant effect of dissolved solute on strength and the related effect of precipitation. The ductility of stabilised specimens is significantly greater by about 300\% and a very high proportion of the deformation is at ostensibly constant stress.
Presumably this almost complete lack of work hardening is due to the absence of strain ageing processes. It is likely that the latter process, which occurs in the solution treated specimens during straining, raises the stress to such a level that matrix or grain interior strengthening is far in excess of that at grain boundaries and inter-crystalline embrittlement results.

The mechanical properties of solution treated materials over a range of strain rates at 800°C and 850°C are presented in Table 4-4 together with a limited quantity of data for the stabilised condition as a comparison. The tendency for higher strength and work hardening rate and lower ductility in the solution treated condition, for a given strain rate, is clearly apparent as it was in Fig. 4-5. With regard to the dependence of 0.2% proof ($\sigma_{0.2}$) and ultimate tensile stress ($\sigma_{UTS}$) and ductility ($\varepsilon$) on strain rate, it is clear that, for a given temperature and specimen treatment, each increased with increasing strain rate. The proof stress was relatively insensitive to changes in strain rate and appears to approach a limiting value with increasing strain rate. Such behaviour might be expected for a material which at low temperatures exhibits a very low strain rate sensitivity, since thermally activated recovery processes must play little part in deformation at such high strain rates, even at these elevated temperatures. The ultimate tensile stress (or true stress at maximum load) rises much more rapidly with increasing strain rate as shown in Fig. 4-6. These results may be described by a relationship of the form

$$\sigma_{UTS}^n = f \left[ \varepsilon \exp \left( \frac{Q}{RT} \right) \right]$$  ... (4.2)

where $n$, the stress exponent, has values of 7.45 and 14.30 respectively for the solution treated and stabilised conditions and $Q$, the activation energy was approximately 79 kcal/mole after solution treatment. This value of activation energy, being rather higher than that for self diffusion (69 kcal/mole), illustrates the effect of solute elements, notably Nb, already mentioned and is close to that obtained by other workers in similar tests. The variation of ductility with
<table>
<thead>
<tr>
<th>(\dot{\varepsilon} ) (s(^{-1}))</th>
<th>(\sigma_{0.2}) (MPa)</th>
<th>(\sigma_{UTS}) (MPa)</th>
<th>(\varepsilon_{UTS})</th>
<th>(\sigma_{0.2}) (MPa)</th>
<th>(\sigma_{UTS}) (MPa)</th>
<th>(\varepsilon_{UTS})</th>
<th>(\sigma_{0.2}) (MPa)</th>
<th>(\sigma_{UTS}) (MPa)</th>
<th>(\varepsilon_{UTS})</th>
</tr>
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<tbody>
<tr>
<td>(10^{-6})</td>
<td>71.6</td>
<td>79.0</td>
<td>0.031</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>63.5</td>
<td>72.0</td>
<td>0.079</td>
</tr>
<tr>
<td>(10^{-5})</td>
<td>79.5</td>
<td>105.0</td>
<td>0.052</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>(10^{-4})</td>
<td>86.5</td>
<td>162.0</td>
<td>0.061</td>
<td>87.2</td>
<td>136.3</td>
<td>0.089</td>
<td>74.0</td>
<td>103.0</td>
<td>0.172</td>
</tr>
<tr>
<td>(10^{-3})</td>
<td>89.3</td>
<td>214.2</td>
<td>0.136</td>
<td>88.2</td>
<td>182.0</td>
<td>0.119</td>
<td>77.6</td>
<td>121.0</td>
<td>0.207</td>
</tr>
<tr>
<td>(10^{-2})</td>
<td>91.2</td>
<td>272.0</td>
<td>0.194</td>
<td>93.4</td>
<td>234.2</td>
<td>0.172</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>
strain rate is illustrated in Fig. 4-7 from which it is apparent that the ductility in both treatments increases at approximately the same rate. The stabilised condition always exhibits the same degree of improvement. The reduction in ductility (true uniform strain) with decreasing strain rate probably arises from two sources. Firstly, the degree of matrix work hardening decreases with strain rate so concentrating more strain on grain boundary regions leading to earlier failure. Together with this factor, diffusional processes play a more significant part in deformation at low strain rates, so contributing more to cavity and crack growth and thereby effectively accelerating failure.

Hot-tensile tests such as these provide one source of data for comparison with MMTT. Probably the most useful comparisons may be made under creep conditions and therefore a rather more intense programme of the latter tests was performed. Because of the wide variety of treatments used and the limit imposed by time, these tests were conducted at only one temperature, 800°C, and three stress levels in the range 85 - 130 MPa. These were therefore, as will be seen, at high creep rates and consequently short term tests. Minimum creep rates lay in the range $10^{-3} - 10^{-1}$ h$^{-1}$ which in fact corresponds to hot tensile strain rates. The treatments used in these tests included the solution treated and stabilised conditions already discussed as well as a number of prestrained and aged samples. These latter treatments consisted of room temperature tensile prestrains of 2, 4, 6 or 10% followed by ageing for up to 50h at 800°C. A summary of results of the creep tests is presented in Fig. 4-8 and 4-9.

It is apparent from Fig. 4-8, which shows the variation of minimum creep rate as a function of applied stress, that the creep strength of solution treated material decreased after a stabilising treatment as deduced from the hot-tensile behaviour. The stress exponent changed in the same manner, although that obtained in both cases was slightly lower after creep as compared with the hot-tensile values. From the lowest level of creep strength a progressive increase was observed with increasing degree of prestrain prior to ageing. After about 4% prestrain the
creep strength rose to the level of the solution treated material and continued to rise with increasing prestrain. The stress exponent appears to be little affected by the degree of prestrain and remained fairly constant having approximately the same value as in the stabilised condition (-10.0). This is to be compared with 5.9 after solution treatment. It should be noted that the data presented in Fig. 4-8 for the 10% prestrain treatment was obtained after only 2h ageing. The reason for this will be made apparent at a later stage during the discussion of MMTT. Two tests employing a longer ageing time (20h) showed substantially no difference in terms of creep resistance.

As with the hot-tensile test data the best way of representing ductilities appears to be as a function of strain or, in this case, minimum creep rate, as shown in Fig. 4-9. It is apparent from the figure that the solution treated and stabilised ductilities vary in a similar way to the hot-tensile tests. The stabilising treatment generally exhibited higher ductilities. Prestrains in excess of 2-4% prior to ageing had a considerable effect on creep ductility. At levels of 2% prestrain the behaviour was approximately the same as for solution treated material. Prestrains of 4% and greater produced a dramatic reduction in ductility of as much as 50% from the solution treated values. This appears to be relatively independent of the degree of prior deformation. It is thought that this ductility drop is the result of some irreversible damage process which is initiated after a critical prestrain or produces a stable defect, such as cracks, which reach a critical size for growth after this degree of prestrain. The nature of the damage process will be considered at an appropriate stage later in the discussion.

4.1.3 Ageing Behaviour

4.1.3.1 Isochronal Ageing Tests

In order to assess the potential, type and temperature range of the ageing phenomena in the steel to be studied, tensile tests were performed on 0.75 mm diameter wires solution treated at 1300°C/hr., prestrained by 2.5% at 20°C and isochronally aged (2 hr.) at temperatures in the range 20°C - 1000°C. The effects
of these ageing treatments were assessed by tests at room temperature. The results are presented in Fig. 4-10, together with those for a number of double ageing treatments which will be discussed a little later.

At low temperatures (<200°C) ageing phenomena similar to those reported by Rose and Glover were observed. Slight yielding effects, characterised by the presence of a yield plateau, were observed during room temperature ageing after only ~30 sec unloading. The magnitude of the proof or yield plateau stress rose rapidly to the level (4.5 MPa) reported for 2h, in a manner consistent with previous workers findings. The yield plateau extended to a strain of 0.01 and was not apparently preceded by a yield drop. A marginal increase in yield plateau stress was achieved by ageing at 150°C. Ageing at 300°C produced some strengthening, but less than at lower temperatures and a more poorly defined yield plateau. Increasing ageing temperatures produced further strengthening and a return to more distinctive yield plateau effects. A strengthening peak of ~12.5 MPa was observed at 550°C, consistent with the observations of Jenkins and Smith. Clearly neither of these two types of ageing behaviour produces sufficiently powerful dislocation strengthening effects although the 550°C ageing process might as discussed later be employed to produce some degree of recovery resistance or enhanced precipitation at higher temperatures.

Ageing at still higher temperatures lead to a decrease in proof stress and the complete disappearance of the yield point phenomenon. Above 700°C the strengthening once again increased with ageing temperature to a maximum value of ~55 MPa at ~800°C. While no yielding phenomena accompanied this change, a noticeable increase in work-hardening rate was observed qualitatively at low strains. This ageing phenomenon was subsequently studied in some detail and shown to be associated with dislocation nucleated precipitation of Nb(C,N). Further increases in ageing temperature produce a dramatic decrease in strengthening due to overageing and particle coarsening phenomena. No change in grain-size due to either recrystallisation or grain coarsening was observed over this range of
ageing temperatures (20° - 1000°C).

Due to the occurrence of these two characteristic ageing peaks a series of tests were performed to investigate the possible benefits to be gained from double or stepped ageing treatments. The results of these tests are presented in Fig. 4-10. Two of the treatments used (2h 550°C followed by 2hr 800°C and 1h 550°C 1h 800°C) produced no significant change in strengthening as compared with the single step treatment. However, 1h at 550°C prior to the normal 2hr 800°C treatment appears to be most beneficial, raising the strength by a further 27 MPa. Obviously it is unclear, from tensile tests alone, how such an increase arises. Two possible explanations should be considered. Ageing at 550°C leads to the formation of either solute atmospheres or clusters which affect subsequent high temperature ageing behaviour. This may arise from a reduction in the amount of dislocation recovery prior to Nb(C,N) precipitation or an increase in the nucleation rate by providing alternative nuclei. In practice these effects are likely to be complementary.

4.1.3.2 Isothermal Ageing Tests

Hardness and tensile tests were performed on 1 mm thick samples of sheet material in both the as-solution treated and solution treated and prestrained (2%) conditions followed by various isothermal ageing treatments at 800°C. These were thought essential since they provided some of the background data necessary for the successful development of MMTT schedules. The results of the hardness measurements and those for 700°C isothermal ageing of prestrained samples are illustrated in Fig. 4-11.

Several points are evident from the figure. Firstly, the ageing process appears to be considerably accelerated by room temperature prestraining. Peak hardness was reached after 3½h following prestraining by 2%, which is to be compared with 20h after solution treatment only. The peak hardness value increased with prestrain, but the relative increase was less after prestraining. A hardness decrease is apparent during the early stages indicating a strong recovery effect. This is to some extent masked by concurrent precipitation hardening and is
therefore more apparent in the 700°C ageing treatment, where a detectable hardness decrease was observed in the early stages. The time to peak hardness decreased by a factor of 20 for a 100°C temperature change. It is consistent with previous observations\textsuperscript{181-196} and indicates that the rate determining process has an activation energy of 70–80 kcal/mole. Ageing at the lower temperature (700°C) produced a slightly higher peak hardness which might reflect a lower degree of dislocation recovery, a slight increase in particle density or possibly a change in precipitation mode. Also shown in Fig. 4-11 is the variation of 0.2% proof stress with ageing time at 800°C following 1200°C solution treatment and room temperature prestraining. This shows the same trends as the hardness results. The level of strengthening (18.5 MPa) was significantly different to the isochronal ageing result for 800°C (Fig. 4-10). Some difference might be expected since the higher solution treatment temperature was used in the latter case. Thus, potentially more Nb(C,N) was available for precipitation. However, from the solubility data presented in Table 4-2 it is apparent that this increase in volume fraction is not capable of producing an increase of more than ~25% in strength. This discrepancy has not been resolved.

No extensive study has been made of the effect of prestrain on ageing. Because of the decision to age for 4h at 800°C during MMTT, the variation of as-aged proof stress with room temperature prestrain was measured using this ageing condition. The prestrained and aged proof stress followed the cold-work flow stress fairly closely although slight deviations occurred at low and high prestrains. The stress change (Δσ) on ageing is shown as a function of prestrain in Fig. 4-12. With no prestrain, ageing for 4h produced a proof stress increase of 8 MPa. The proof stress increment decreased to zero after about 0.03 prestrain and subsequently became negative. A problem exists in describing this behaviour, since prestrain may have affected the rate of precipitation. This is further complicated by the fact that recovery can accompany ageing at these temperatures. Thus, while with no prestrain ageing proceeded slowly (Fig. 4-11), little or no recovery occurred.
and consequently the proof stress increment was almost entirely due to dispersion strengthening. At prestrains below 0.03 dispersion strengthening clearly exceeded softening due to recovery but at prestrains in excess of this level recovery predominated. This would seem to indicate that the increase in precipitate nucleation rate, which appears to be related to dislocation density and hence prestrain, rose more slowly than the recovery rate which was similarly affected. It is also significant that prestrains up to ~0.26 did not produce large strength changes considering the high levels involved, thereby indicating an absence of recrystallization, a deduction borne out by grain size measurements.

4.1.4 Preliminary Electron Microscope Studies

A considerable amount of preliminary work was performed prior to the MMTT studies. Electron microscopy was used to characterise the microstructures of solution treated and deformed samples and their ageing behaviour. The purpose of this work was to provide the necessary background data from which MMTT schedules could be devised. Much of this data also provides the base properties against which MMTT is to be compared structurally. During the MMTT studies certain observations were made which stimulated closer examination of these phenomena and for the sake of continuity all such work will be presented here.

4.1.4.1 Solution Treated Microstructure

Following the solution treatment optimisation work with extraction replicas it was thought necessary to check the measurements by an alternative method. Because of etching problems, 100 kV microscopy using thin foils was thought to be unsuitable, although Deighton appears to have used the technique successfully. With the arrival and eventual operation of the NPL HVEM it was decided to attempt such a determination together with other work to characterise the microstructure in the solution treated condition. This work like much that follows was performed on the two extreme solution treatments shown in Table 4-1, namely 1200°C 1hr and 1300°C 4hr. A typical low magnification micrograph of the 1200°C treatment is shown in Fig. 4-13 which may be compared with the optical microstructure shown in Fig. 4-1 for general features such as grain size, twin frequency etc. From such areas a
measurement of density of undissolved carbides was made assuming a mean foil thickness of 0.5 μm and making the appropriate correction for mean particle diameter (0.90 μm for 1200°C and 1.60 μm for 1300°C treatments).

This yielded values for particle densities of $4.5 \times 10^9$ cm$^{-3}$ and $1.2 \times 10^7$ cm$^{-3}$ and undissolved carbide volume fractions of 0.0017 and 0.0002 respectively for the 1200° and 1300°C treatments. These values are in fair agreement with those determined by extraction replicas although they appear to be consistently lower by the thin foil method. This discrepancy may arise through the loss of some particles by etching or an error due to the assumptions made in the extraction replica study. Indeed, the etching effect would seem the most likely source, since if the small (-1-5 μm) holes are included in the count as probable carbides the agreement is better. The carbide size distribution is quite wide (-0.3-8 μm) as is to be expected during solution treatment, due to the competing effects of particle coarsening and solution. There is some tendency for clustering of large carbides particularly in the region of grain junctions. This may reflect their strong pinning effect during grain growth.

The dislocation distribution after solution treatment is extremely non-uniform and indeed unlike that generally observed in annealed materials. The average dislocation density in the samples was $3.3 \times 10^9$ cm$^{-2}$ and $1.9 \times 10^{-9}$ cm$^{-2}$ respectively for the 1200° and 1300°C treatments. Areas far from carbides or grain boundaries contain the more usual network of dislocation nodes and segments (Fig. 4-14) which is to be expected in an annealed material. However, the dislocation density is far in excess of the usual annealed value of $\sim 10^8$ cm$^{-2}$ and in the region of grain boundaries or large particles it can rise to levels 3-4 times the average values. It has been proposed$^{183}$ that this high dislocation density results from the high stresses set up during rapid cooling from the solution treatment temperature, due to the large difference in coefficients of expansion between undissolved carbides and austenite. These are relieved by dislocation punching processes at the particle-matrix interface at fast cooling rates or by diffusional mass transfer at low rates.
Clearly the strains must be quite large since structure is observed in carbide particles when the matrix is out of contrast. This observation, which implies plastic deformation of the particles, is not conclusive, since interface structure may produce similar contrast effects. However, the signs of plastic deformation in the matrix are not in the least ambiguous. Fig. 4-15 shows several particles with the commonly observed tangled dislocation structure. One particle (at A) shows what appear to be punched out dislocation loops. Rigorous contrast studies were not performed but these loops closely resemble those typically associated with prismatic punching processes. Silcock has derived a relation between number \( N \) of dislocations generated per particle and the contractional strain on cooling through a temperature range \( \Delta T \):

\[
N = a (\alpha_\gamma - \alpha_P) \frac{\Delta T}{\Delta D} \quad \ldots (4.3)
\]

where \( a \) is the particle diameter and \( \alpha_\gamma \) and \( \alpha_P \) the coefficients of expansion of austenite and particle respectively. Using values quoted by Stuart and Ridley for \( \alpha \) (austenite and NbC), a particle diameter of 1.0 \( \mu \)m and a temperature change of 1180°C the number of dislocations would be 60. Assuming that the mean planar density of particles is given as \( N_v^{2/3} \), cooling should produce a dislocation density of \( 1.6 \times 10^8 \) cm\(^{-2} \). An alternative calculation can be made assuming the material to be divided into cubic cells of mean dimensions \( N_v^{-1/3} \) and each cell to contain a particle of diameter 1 \( \mu \)m, at its centre. The strain per cell is then \( 1.5 \times 10^{-2} \) and is given by the equation

\[
\varepsilon = \rho \frac{\bar{D}}{L} \quad \ldots (4.4)
\]

where \( L \) is the average distance moved by a dislocation \( (-N_v^{-1/3}/4) \). Thus the dislocation density \( \rho \) is given by

\[
\rho = 4\varepsilon N_v^{1/3}/\bar{D}
\]

and would have a value of \( 4.0 \times 10^9 \) cm\(^{-2} \). This of course is an upper bound since the dislocations may move an average distance approximately equal to half the grain.
diameter (20 μm) in which case the dislocation density would be $\sim 3 \times 10^8 \text{ cm}^{-2}$. The actual density would be expected to lie within this range if NbC particles were the sole source of punched dislocations and recovery did not occur. The effect of inclusions which occur as particles 5-10 μm in size and having a volume fraction of between 0.8 and 1.4% should also be taken into account. Indeed the relatively small difference in dislocation density between the 1200°C and 1300°C treatments would seem to suggest that the inclusions have a significant effect. If the punching occurred only at NbC particles the difference should be the same as between the respective volume fractions of undissolved NbC. While it is impossible to predict the very high solution treated dislocation densities quantitatively with any accuracy it nonetheless is apparent that the levels observed are quite reasonable.

4.1.4.2 Aged Microstructure-Unprestrained

Since the object of this work was to compare the effect of MMTT on structure and properties with what might be termed standard treatments a considerable amount of thought was given to the best such standards. Obviously the solution treated condition is such a standard, but one which is susceptible to precipitation during creep. Therefore, it was decided to obtain material with as high a level of stability as possible. To this end it was decided to age solution treated specimens to just beyond peak hardness at 800°C as indicated in Fig. 4-11. Other treatments similar to those used by British Nuclear Fuels were contemplated but these generally produce fine grain structures. This was not thought advisable since for a good comparison it is advantageous to reduce the number of variables to a minimum, grain size being one of the major of these.

Ageing at 800°C for 30 hr following solution treatment at 1200°C produced no significant change in grain size. The general features of the precipitate distribution are apparent in Fig. 4-16. The particle density was measured and found to be $6 \times 10^{12} \text{ cm}^{-3}$ and the dislocation density $1.6 \times 10^9 \text{ cm}^{-2}$. It will be appreciated that the latter parameter is extremely difficult to measure due to the high incidence of dislocation loops particularly in the neighbourhood of particles.
and therefore only isolated dislocation segments and loops have in fact been counted. The most significant feature of the precipitate distribution is its highly banded nature. Trace analysis showed that the bands or stringers lie predominantly along <110> and <112>. Similar behaviour has been observed by Lewis and Hattersley and Beckitt and Clark in unstabilised austenitic steels during the precipitation of $M_{23}C_6$. They described the distribution in terms of primary stringers along <112> and secondary stringers along <110>. The primary stringers were ascribed to precipitation on "grown-in" dislocations of predominantly edge character and formed during the early stages of ageing. To accommodate the high lattice strains during precipitate growth, PGD are punched out which nucleate new precipitates. These PGD are prismatic loops and in this system are of interstitial type and should therefore be punched out along <110> axes giving rise to the secondary stringers. Such a model would seem to explain the general features of the microstructure in niobium bearing alloys of the type studied here. Generally the major proportion of precipitates were in the form of rather short primary stringers with a diffuse distribution of secondary stringers surrounding them. However, in some areas of locally high dislocation density, for example at undissolved carbides (Fig. 4-16) the primary stringers are more pronounced than are the secondary ones. The punching of dislocations from precipitates is clearly apparent but no direct evidence for the prismatic punching process was obtained.

Dislocation punching from Nb(C,N) particles was observed in the HVEM. This was noticed during some early stereo microscopy work and caused problems in Burgers vector and trace analysis. It could, however, be prevented by the simple expedient of avoiding high beam currents and was clearly due to thermal stresses resulting from the differential expansion effect discussed earlier. A typical example of this effect, albeit a contrived one, is illustrated in Fig. 4-17 which shows a region of foil under identical diffraction conditions both before (Fig. 4-17(a)) and after (Fig. 4-17(b)) subjecting the area to an intense focussed beam at 1 MeV. It is apparent from the figure that at several points (A) new dislocations have
appeared in the region of precipitates and that several dislocations (B) have moved. Furthermore, a film of contamination which reduced the clarity of the structural detail is clearly visible. It is possible that internal stresses are introduced by the contamination layer itself and these may contribute. Consequently, high beam currents have the dual effect of heating the foil producing undesirable microstructural changes and accelerating the rate of contamination both of which should be avoided if stereo and Burgers vector analysis are to be successful.

4.1.4.3 Prestrained Microstructures

Another standard condition with which MMTT substructures can be compared is that of work hardened or prestrained specimens directly following the preliminary solution treatment. These are also of importance since these or similar prestrains constitute the first stage of MMTT. Indeed the form of the dislocation structure produced by the first MMTT prestrain, together with the way this affects subsequent ageing behaviour, will control both the structure and mechanical behaviour after MMTT.

To investigate the effect of prestrain on dislocation structure a number of 1200°C solution treated specimens were prestrained by various amounts at room temperature, thinned and their structures characterised by electron microscopy. Some typical micrographs showing structures after 2.0, 4.5, 9.5 and 31% strain are presented in Fig. 4-18, 4-19, 4-20 and 4-21 respectively. The highly banded nature of the dislocation structures is apparent from the HVEM micrographs even at low strains. 100 kV micrographs generally showed the more typical tangled structure described earlier (see Appendix I), in which the dislocation bands were rarely evident (Fig. 4-18(a)). This and other observations relating to the dislocation distribution will be discussed in detail later. First some more quantitative results of dislocation densities will be presented. These are shown in Table 4-5 together with the relevant flow stress data. In Table 4-5 the stress values quoted are the true tensile flow stress prior to unloading at each elongation, with the exception of that for the solution treated condition (ε = 0%). The latter is an
estimated flow stress corresponding to the proportional limit. The dislocation density values quoted for strains of 13% and less are real values determined by the method outlined earlier and are subject to a mean standard deviation of ± 20%. The corresponding standard deviation in \( \rho^b \) is therefore ± 10%. At strains in excess of 13% it was impossible to obtain a reliable measure of dislocation density due to the large number of overlapping images and the uncertainties in measuring thicknesses accurately. The first point is particularly relevant due to the cellular arrangement of dislocations. To obtain the estimated values shown in Table 4-5 certain assumptions had to be made. The first was to assume that the average thickness of areas observed was -70% of that for lower strains. Secondly dislocation counts were made on the very dense, highly tangled areas assuming that dislocation images just overlapped and that the number of intersections with the reference line could be estimated by assuming a value for the average image width (typically ~150 \( \AA \) at 100 kV). The first assumption was made on the basis that during HVEM work acceptable thicknesses, from the point of view of contrast and screen brightness, were lower in the heavily worked samples. These assumptions would be expected to lead to significant underestimates of dislocation density in the case of the 19 and 31% strained samples.

The dislocation density appears to obey relationships of the same form as equations (3) and (4) of Appendix 1 and least squares analysis of this data yields

\[
\rho = (3.1 + 1.71 \times 10^1 \varepsilon^0.84) \times 10^9 \text{ cm}^{-2}
\]

and

\[
\sigma_f = \sigma_o + \alpha GB \rho^b \quad \ldots \quad (4.5)
\]

where \( \sigma_o = -32 \text{ MPa} \) and \( \alpha = 1.5 \). The data is plotted according to equation (4.5) in Fig. 4-22 in which the error bars represent the standard deviation for each result. The deviation from this relationship at high strains presumably reflects the underestimation of dislocation density mentioned earlier. Clearly in this material an upper bound for reproducible measurements of dislocation density appears to be about \( 3.0 \times 10^{10} \text{ cm}^{-2} \) by thin foil microscopy since the dislocation distribution is
so highly heterogeneous. This limit will obviously vary with the nature of the dislocation distribution, foil thickness and consequently accelerating voltage.

Up to this point it has been assumed that the measured dislocation density is the true value for the material. Ignoring such possibilities as dislocation loss during thinning which are difficult to quantify, the measured density will generally be lower than the true density due to dislocation invisibilities under most diffraction conditions. The proportion of dislocations invisible in various diffraction conditions are tabulated in Table 4-6. These are calculated from the expression \( g \cdot b = 0 \) assuming a homogeneous distribution of all Burgers vectors. Providing the diffraction conditions for each micrograph used in the determination are known and that the assumption of homogeneous distribution is valid, a suitable correction factor may be applied to the values in Table 4-5. An alternative and more accurate method would be to use imaging conditions which lead to no invisibilities or to use composite micrographs obtained using several reflections. For most purposes this is a difficult and time consuming procedure and therefore the values quoted in Table 4-6 will be used. Considering all diffraction conditions used in the determination of the values in Table 4-5 and Fig. 4-27 an average invisibility of 40% is obtained. The true dislocation density is therefore approximately 1.65 times the measured value. If this correction is applied to the \( \rho \) values in Fig. 4-27 a new relationship is obtained with \( \sigma_0 \) unchanged and \( \alpha = 1.16 \).

Trace and elementary Burgers vector analyses have been performed on some of these specimens in order to characterise the dislocation structure and be able to relate it to the prestrained and aged microstructures which will be discussed later. All of this work has been done by HVEM. The highly banded nature of the dislocation structures (Fig. 4-18) is consistent with slip on \( \{111\} \) and the bands appear to be debris resulting from dislocation interactions on intersecting slip systems. Viewed in \( [110] \) orientation two prominent bands are often observed along \( [\bar{1}2\bar{1}] \) and \( [\overline{1}2] \) which are the trace of the \( (\bar{1}11) \) and \( (\bar{1}1\bar{1}) \) planes, respectively, viewed edge-on. Generally, a diffuse banded or linear arrangement of dislocations can be seen along
(110) and a rather random network in the background. This indicates slip on one or other of the (111) or (111) planes or more probably both. These planes lie at about 54° to the (110) and many dislocations may be seen bowing out, often along <112> direction traces, implying slip on (111) and (111) of edge dislocations. This observation indicates activity on a large number of slip systems. However, the apparent propensity of slip varies from grain to grain, presumably due to the change in resolved shear stress on the various systems with grain orientation. There also appears to be variations from area to area within a grain such that regions adjacent to grain boundaries and large undissolved carbides exhibit apparently more activity, as for example in Fig. 4-18. This presumably is a consequence of the greater plastic accommodation which must occur in these regions during straining.

As the strain increased the dislocation density increased, as already indicated, and the bands became more pronounced. The bands, apart from becoming continuous and forming a cellular network, also thicken with increasing strain. These observations regarding the general distribution of dislocations are consistent with etch pit and slip line studies and give a more realistic indication of the true bulk distribution because of the increased specimen thickness (0.5 - 0.7 μm) in HVEM. The more tangled irregular structure observed at 100 keV is presumably a result of local rearrangements due to surface effects during thinning, which can occur in thinner (0.2 μm) areas. In thin specimens (0.2 μm) the number of dislocations observed may be insufficient for clearly defined structural features to be observed, hence the impression of a tangled rather than cellular structure.

The trace analysis of dislocation bands discussed earlier has been supplemented by some elementary Burgers vector analysis of specific features such as dipoles. While the principles of trace analysis techniques used here have been discussed generally in sections (4.1.4.4) and (4.3) a few more points should be added concerning Burgers vector analysis. Firstly, the analysis performed here is not as rigorous as is often deemed necessary. The usual criterion of finding at least two lattice reflections in which a dislocation disappears, before its Burgers vector
is uniquely defined, has not been followed. Because of the \([001](110)\) texture the most favourable orientations for this type of analysis\(^{306}\), around (111), were difficult to obtain with conventional goniometers, requiring between \(\pm 30^\circ\) and \(50^\circ\) tilting facilities. If work was performed at or near (110) it is apparent from Table 4-6 that \(-\frac{1}{6}\) of all dislocations, i.e. those with \(\mathbf{b} = a/2[110]\), would be invisible. Using only those lattice reflections which produce reasonable contrast i.e. \([\bar{1}1\bar{1}], [\bar{1}11]\) and \([002]\) it is clear that, while all dislocations should disappear once only one Burgers vector \(\mathbf{b} = a/2 [1\bar{1}0]\) is uniquely determined. Of the other two pairs \((a/2[101], a/2[\bar{1}0\bar{1}])\) and \((a/2[10\bar{1}], a/2[1\bar{1}0])\) it is possible only to distinguish between the pairs, but not individual dislocations. However, other techniques may be used to overcome these limitations without having to tilt large angles from (110). The latter would be both tedious and time consuming for structures of the complexity encountered in most of this work. These techniques which incorporate trace analysis etc. are described and discussed later in this section and (4.3).

To illustrate the analytical methods as well as the more specific characteristics of the dislocation structure after 2% strain at room temperature a series of micrographs are presented in Fig. 4-23. It should be pointed out at this stage that, due to the complexity of these dislocation structures, relatively thin areas (~0.25 \(\mu\)m) have been used. They may not therefore, be truly representative because of the possibility of dislocation rearrangement discussed earlier. A cursory analysis of micrographs such as these revealed that the majority of dislocations were of \(a/2 <110>\) Burgers vector and that in general these appear to be confined to their slip planes. The well defined bands have already been identified with the traces of \(\{111\}\) planes. In the context of the present study the most relevant information was that concerning the nature of these dislocation bands. The most interesting question to resolve is, consequently, whether the bands are slip bands in the traditional sense or whether they are the debris produced by interactions between dislocations from different slip systems, since this might
subsequently affect ageing behaviour. Potentially, such debris might consist of Lomer or Hirth dislocations as already pointed out. The presence of such dislocations may be checked without elaborate Burgers vector analysis by the following method employing a combination of trace and elementary Burgers vector analyses.

The direction traces of dislocations resulting from all potential Lomer and Hirth dislocation reactions may be specified and are summarised in Table 4-7, in which the intersecting planes and the trace and direction in (110) of the resulting Lomer or Hirth dislocations are presented. This information may be used in conjunction with standard dislocation visibility criteria, the relevant details of which are summarised in Table 4-8 for various reflections near (110) orientations. In general dislocation invisibility should result for $|g \cdot b| = 0$ and the image strength is proportional to $|g \cdot b|$. From the tabulated information it follows that if dislocations in the $[112]$ and $[1\bar{1}2]$ bands disappear in only one $<111>$ reflection ($g[111]$ or $g[\bar{1}\bar{1}1]$, respectively) they must be Lomer dislocations. Slip dislocations, on the other hand, lying in these bands will disappear in both $<111>$ reflections. It is apparent from Fig. 4-23 that many dislocations, lying in $[112]$ bands disappear only in $g[111]$. These are Lomer dislocations with $\bar{b} = \frac{1}{2}a[01\bar{1}]$ or $\frac{1}{2}a[10\bar{1}]$. Many lying in $[\bar{1}1\bar{2}]$ bands exhibit similar general behaviour in $g[1\bar{1}1]$ indicating that they too are Lomer dislocations with $\bar{b} = \frac{1}{2}a[10\bar{1}]$ or $\frac{1}{2}a[01\bar{1}]$. Dislocations, which disappear in $g[002]$ with $\bar{b} = \frac{1}{3}a[1\bar{1}0]$, lie in (111) or (11\bar{1}), are clearly glissile and can often be seen bowing out on their slip plane which in both cases is inclined at 54° to the foil plane. Dislocations of other Burgers vectors cannot be unambiguously identified with the combination of diffraction conditions employed here. It should also be pointed out that in this orientation, (110), dislocations with $\bar{b} = \frac{1}{2}a[110]$ are invisible in all $g$. Some dislocations exhibiting weak contrast in most reflections indicating $|g \cdot \bar{b}| \approx 1.0$ and disappearing in $g[002]$ are assumed to be Hirth locks ($1/3a<100>$). No detailed analysis was performed to identify the Burgers vector of the latter unambiguously, though, as will be discussed.
later, similar observations were made in the collaborative work with Professor Ishida after a number of different treatments (Section (4.3)).

An estimate of dipole density and size was made after 2% prestrain. Where foils were observed in {100} orientation all dipoles should have been visible providing two different reflections were used, and no estimate of dislocations in bands was necessary. However, this condition rarely obtained and therefore (112) orientations were used. Here with $g = [11\bar{1}]$ approximately half the dislocations should be invisible and one quarter lie in the dislocation band (i.e. (11\bar{1}) edge on). Counts were made of dipole like features, elongated loops or 'cuspy' dislocations providing these lay along $<112>$ traces. The figure obtained for the dipole density was corrected for dislocation invisibilities and those lying in the band, where they were difficult to detect. To do this it was assumed that the dislocation dipole distribution was homogeneous in terms of slip system and Burgers vector. From the distribution of Burgers vectors in {11\bar{1}} and their visibility it was clear that of those dislocations lying between dislocation bands (11\bar{1}) about one third were invisible and a further one-quarter could not be counted due to their presence in (11\bar{1}). Correcting for these yielded a value of $7.55 \times 10^{13} \text{ cm}^{-3}$. The average dipole length was 0.16 \text{ μm} and width 0.03 \text{ μm}. This dipole size is of the same order as that commonly observed\textsuperscript{311,312} and the density also appears to be fairly consistent with previous work.

4.1.4.4 Prestrained and Aged Microstructures

A number of prestrained and aged specimens have been studied in order to provide background information relating, where possible, the precipitate structures to the dislocation structure prior to ageing. Like the prestrained structures which constitute effectively one half of the first MMFT cycle, this treatment has received a considerable amount of attention, since it completes the first MMFT cycle and, as will be seen later, has a major influence on the manner in which the MMFT structure builds up. Once again the general characteristics of the structure will be described, in this case as a function of ageing time, temperature and degree of
Table 4-5
Summary of dislocation density data for solution treated (1200°C for 1h) and prestrained RM 9209

<table>
<thead>
<tr>
<th>ε_p (%)</th>
<th>σ_f (MPa)</th>
<th>ρ (x10^9 cm^-2)</th>
<th>ρ_b (x10^5 cm^-1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.30</td>
<td>3.3</td>
<td>0.57</td>
</tr>
<tr>
<td>2.0</td>
<td>252</td>
<td>9.2</td>
<td>0.95</td>
</tr>
<tr>
<td>4.5</td>
<td>350</td>
<td>16.2</td>
<td>1.27</td>
</tr>
<tr>
<td>9.5</td>
<td>455</td>
<td>26.5</td>
<td>1.63</td>
</tr>
<tr>
<td>13</td>
<td>520</td>
<td>33.0</td>
<td>1.82</td>
</tr>
<tr>
<td>19</td>
<td>605</td>
<td>40.0</td>
<td>2.00</td>
</tr>
<tr>
<td>31</td>
<td>720</td>
<td>50.0</td>
<td>2.20</td>
</tr>
</tbody>
</table>

Table 4-6
Proportion of dislocations with \( b = a/2 \langle 110 \rangle \) invisible in various \( g \) reflections

<table>
<thead>
<tr>
<th>( g )</th>
<th>Proportion invisible</th>
</tr>
</thead>
<tbody>
<tr>
<td>\langle 111 \rangle</td>
<td>( \frac{1}{2} )</td>
</tr>
<tr>
<td>\langle 200 \rangle</td>
<td>( \frac{1}{3} )</td>
</tr>
<tr>
<td>\langle 220 \rangle</td>
<td>( \frac{1}{6} )</td>
</tr>
<tr>
<td>\langle 113 \rangle</td>
<td>( \frac{1}{6} )</td>
</tr>
<tr>
<td>\langle 133 \rangle</td>
<td>( \frac{1}{6} )</td>
</tr>
<tr>
<td>\langle 420 \rangle</td>
<td>0</td>
</tr>
</tbody>
</table>
Table 4-7
Summary of potential dislocation interactions of the Hirth and Lomer type in an fcc material and trace analysis

<table>
<thead>
<tr>
<th>Intersecting Slip Planes</th>
<th>Resultant Dislocation Lock</th>
<th>Trace</th>
<th>Direction In (110)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(111) with (111)</td>
<td>4a [011]</td>
<td>[011]</td>
<td>[112]</td>
</tr>
<tr>
<td></td>
<td>1/3a [100]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(111) with (111)</td>
<td>4a [101]</td>
<td>[101]</td>
<td>[112]</td>
</tr>
<tr>
<td></td>
<td>1/3a [010]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(111) with (111)</td>
<td>4a [101]</td>
<td>[101]</td>
<td>[112]</td>
</tr>
<tr>
<td></td>
<td>1/3a [010]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(111) with (111)</td>
<td>4a [011]</td>
<td>[011]</td>
<td>[112]</td>
</tr>
<tr>
<td></td>
<td>1/3a [100]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(111) with (111)</td>
<td>4a [110]</td>
<td>[110]</td>
<td>[110]</td>
</tr>
<tr>
<td></td>
<td>1/3a [001]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(111) with (111)</td>
<td>4a [110]</td>
<td>[110]</td>
<td>[110]</td>
</tr>
<tr>
<td></td>
<td>1/3a [001]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>


Table 4-8
Dislocation visibility for diffraction conditions near (110)

<table>
<thead>
<tr>
<th>$\vec{b}$</th>
<th>$\vec{g}$</th>
<th>[111]</th>
<th>[113]</th>
<th>[002]</th>
<th>[111]</th>
</tr>
</thead>
<tbody>
<tr>
<td>4a [110]</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>4a [101]</td>
<td>2</td>
<td>4</td>
<td>2</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>4a [011]</td>
<td>0</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>4a [110]</td>
<td>2</td>
<td>2</td>
<td>0</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>4a [101]</td>
<td>0</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>4a [011]</td>
<td>2</td>
<td>4</td>
<td>2</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>1/3a [100]</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>1/3a [010]</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>1/3a [001]</td>
<td>1</td>
<td>3</td>
<td>2</td>
<td>1</td>
<td></td>
</tr>
</tbody>
</table>
prestrain. This will be followed by a discussion of the results of trace and elementary Burgers vector analysis. Work has also been performed on specimens aged for very short times (so called "flash anneal") in order to establish the nature of precipitate nucleation sites and the rate of dislocation recovery.

The structural changes occurring during ageing at 800°C after 2% prestrain are illustrated in Figs. 4-24 to 4-26. These should be compared with the prestrained structure shown in Fig. 4-18. It is apparent from these figures that the extent of dislocation recovery during the early stages of ageing (<4h) is considerable. A feature of the aged structures observed even at 100 keV is the highly banded nature of the precipitate distributions. In fact, while such a structure was barely evident in the as prestrained condition at 100 keV, the general crystallographic resemblance to the prestrained structure at 1 MeV is quite striking in certain orientations, notably (110). Some of the precipitate bands lay in [112] directions implying that precipitation occurred on (111) dislocation bands. However, with increasing ageing time, interband precipitation appeared to have occurred on a limited scale, in many cases associated with dislocations apparently punched out from primary precipitate bands or stringers. The precipitate density appeared to rise rapidly in the first 1h of ageing, reaching a saturation level in the early stages. Longer ageing times produced only a marginal increase in particle density which arose primarily from the interband precipitation. As already mentioned the dislocation density decreased appreciably during the first 1h of ageing and remained at this level, though the increased number of punched out interband dislocations gave the impression that the dislocation density at peak hardness (~3h at 800°C) was somewhat higher and that the density decreased during the overageing stage (10h and 42h). These general observations are supported by quantitative measurements which are presented in Fig. 4-27. In the figure the particle and dislocation densities are shown as a function of ageing time at 800°C following 2% room temperature prestrain. It is apparent from the data that the prestrained dislocation density (9.2 x 10⁹ cm⁻²) decreased to the level of solution treated material (~3.3 x 10⁹ cm⁻²) after ageing.
for 1h, and that dislocation punching processes increased in the region of peak hardness, the density then returning to its pre-peak level of about $3.5 \times 10^9 \text{ cm}^{-2}$ during overageing. The particle density apparently reached saturation ($-6.2 \times 10^{14} \text{ cm}^{-3}$) by just beyond peak hardness ($\sim 6h$). The comparatively large reduction in dislocation density during the early stages of ageing accounts for the fact that only a marginal increase in hardness and proof stress accompanied ageing. Actually the strengthening arising from precipitation and softening by recovery effectively balance one another producing only a small increase ($\sim 5 \text{ MPa after 1h ageing}$) in proof stress (Fig. 4-11). Furthermore, the fact that most precipitates nucleated during the first 1h of ageing suggests that the increase in dislocation density at peak hardness may have contributed, as might further recovery during overageing, to the overall strengthening behaviour.

For a comparison with other ageing treatments following 2% prestrain 1 MeV micrographs showing typical microstructures after ageing to peak hardness at $800^\circ (3h), 750^\circ (16h)$ and $700^\circ (72h)$ are presented in Fig. 4-28. It is apparent from the figures that structurally there is little difference between these three ageing treatments. No stacking fault precipitation was observed even after $700^\circ$ ageing.

In summary, the dislocation and particle densities after ageing at $800^\circ$ for 3h, $750^\circ$ for 16h and $700^\circ$ for 72h were $4.15 \times 10^9 \text{ cm}^{-2}$ and $5.95 \times 10^{14} \text{ cm}^{-3}$, $4.35 \times 10^9 \text{ cm}^{-2}$ and $6.4 \times 10^{14} \text{ cm}^{-3}$ and $5.75 \times 10^9 \text{ cm}^{-2}$ and $6.9 \times 10^{14} \text{ cm}^{-3}$, respectively. It is evident that the degree of nucleation increased slightly with decreasing temperature. The level of recovery appeared to decrease or dislocation punching to increase with decreasing temperature. Either might give rise to a slightly higher degree of precipitate nucleation. Dislocation punching is expected to become a more important mechanism for strain relief during precipitate growth at lower temperatures where diffusional accommodation becomes less efficient.

Typical structures after room temperature deformation to 2, 5 and 10% strain each followed by ageing at $800^\circ$ for 4h (i.e. the first complete cycle of each MMTT schedule) are shown in Figs. 4-29. It is apparent from these micrographs that
the structural complexity increased with increasing prestrain. The level of recovery appeared to be less and the density of precipitates greater with increasing prestrain, though the complexity after 5 and 10% treatments was such that quantitative measurements were not possible. However, a close relationship between the prestrain dislocation (Figs. 4-18 to 4-20) and subsequent precipitate structures was a common feature of each treatment. Indeed after the 10% prestrain and ageing treatment (Fig. 4-29(c)) the cold worked cellular dislocation structure was still clearly defined. Precipitates were extremely fine and their density well in excess of $10^{15}$ cm$^{-3}$ in the latter case. It is evident that the only structure amenable to analysis is that produced by the 2% prestrain treatment and since this constitutes the most thoroughly studied MMTT schedule this will be discussed further below.

In addition to the general features of the microstructures outlined above a number of specific observations are noteworthy. Denuded zones ~1 μm wide were often observed adjacent to grain boundaries in material prestrained by 2% prior to ageing, as illustrated in Figs. 4-30. Such regions were characterised by a high density of precipitates in grain boundaries often with an attendant dislocation structure and a region denuded of both precipitates and dislocations. Generally the zone consisted of randomly dispersed particles although where well defined bands of precipitates occurred in the grain interior these often crossed the zone. Precipitates in the boundary at the point of impingement with such bands also appeared to occur in characteristic linear arrays. In those boundaries inclined at an angle to the specimen plane the precipitates appeared as discrete rows implying that they were nucleated at points where, for example, slip bands intersected the boundary. Even in the more usual case, where the zone contained only isolated precipitates, linear arrays of particles could be seen at the boundary. The reason for the denuded zone in the present material is unclear. It could arise from solute depletion due to enhanced grain boundary precipitation though this seems unlikely in the light of the observation that the denuded zone disappeared after 3 MMTT
cycles. It is unlikely that it is due to vacancy depletion of the type observed in aluminium alloys, since high vacancy supersaturations of the right magnitude are not achieved in the present alloy. The final possibility is that recovery occurs more rapidly in the region of grain boundaries and consequently fewer nucleation sites would be available during precipitation.

The occurrence of dislocation punching during particle growth is evident in Fig. 4-30. Dislocations were seen apparently bowing out from rows of particles giving them a scalloped appearance. This effect is even more clearly defined in Fig. 4-31(a). Such behaviour was only observed on a large scale in specimens after 1h to 6h ageing. It has consequently been identified with the rise in dislocation density during this period (Fig. 4-27). In the case of individual particles a single dislocation loop was often observed encircling the particle (Fig. 4-31(b)). These effects are analogous to the punching of dislocations from particles during observations in the HVEM (Fig. 4-17), though the latter arose from strains due to differential thermal expansion between the particle and matrix.

While the precipitate bands illustrated in Fig. 4-24 often, when inclined at an angle to the incident electron beam, appear as raft-like structures they are generally fairly continuous. They have consequently been associated with nucleation on dislocations in slip bands. Occasionally structures like that illustrated in Fig. 4-32 were observed. The latter consisted of raft-like arrangements of closely spaced precipitates which extended over only a few microns. Such arrangements of precipitates have been observed by a number of authors\textsuperscript{197,198,200} (see 2.2.3) and are generally accepted as resulting from repeated nucleation on climbing dislocations, though the Burgers vector in the present case has not been identified.

The three dimensional distribution of particles has been studied using stereo and trace techniques. A typical example is illustrated in Fig. 4-33. The same area is shown in three different orientations imaged using the same lattice reflection. Three prominent bands of precipitates are clearly visible in the micrographs corresponding to (111) in the (110) orientation, (\overline{1}1\overline{1}) in the (011)
orientation and that perpendicular to $g$, $(\overline{1}11)$, in each case. Reference to the $(121)$ orientation which lies at $30^\circ$ to $(110)$ and $(011)$ indicates that the $(\overline{1}11)$ and $(1\overline{1}1)$ traces are indeed bands or more correctly planar arrays and not discrete rows of precipitates. The average spacing between bands ($\sim 0.8 \mu m$) is similar to that between slip bands in prestrained material. Trace analysis of the individual rows of precipitates contained in and between bands has been performed on areas such as that shown in Fig. 4-34 together with the details of the analysis. It is apparent from this analysis that most of the precipitate rows lie along $<110>$ or $<112>$ traces. Furthermore the $<110>$ precipitate rows, often with associated dislocations, were $\sim 1.0 \mu m$ long, while $<112>$ rows were almost invariably much shorter ($0.1 - 0.3 \mu m$). The density of $<112>$ rows was also measured in the case of $\frac{t}{4}$h ageing and found to be $5.85 \times 10^{13} \text{ cm}^{-3}$ which, though slightly lower than the dipole density in the prestrained material, is in reasonably good agreement. From these distinct size and density correlations it is proposed that the $<110>$ rows were nucleated predominantly at Lomer or Hirth dislocations and the $<112>$ rows at edge dipoles.

Though stereomicrographs of this type were obtained primarily for trace analysis, as performed above in the particular case illustrated in Fig. 4-33, a number of other imaging reflections were used. From these (Fig. 4-35) one can speculate further on the nature of the nucleation sites. First it is proposed that long straight dislocations exhibiting fuzzy contrast and having $[\overline{1}10]$ traces are either Hirth or Lomer dislocations on which precipitate nucleation was occurring at the end of the ageing treatment. It is readily apparent that the diffraction conditions in Figs. 4-33 and 4-35 are not adequate for obtaining an absolute determination of Burgers vector. Consequently the methods employed in the previous section (4.1.4.3) and illustrated in Tables 4-7 and 4-8 were used. The Burgers vectors of Lomer or Hirth dislocations with $[\overline{1}10]$ traces should be $4a[110]$ and $a[001]$, respectively. (Table 4-7). It is apparent from Table 4-8 that in the reflections employed ($[\overline{1}11]$, $[002]$ and $[\overline{1}12]$) these dislocations did not disappear in any reflection. Consequently, their general contrast behaviour was
consistent with being Hirth dislocations with Burgers vector $a[001]$ and not Lomer dislocations. It is also apparent from Fig. 4-35 that the (111) precipitate bands had a similar structure to the (111) and (111) bands discussed earlier. Furthermore, since most possible $<112>$ and $<110>$ traces were observed it is concluded that all \{111\} planes were active during deformation and ageing.

In order to study in more detail the nature of the precipitate nucleation process, one 2% prestrained sample was annealed in air for 3 min. at 800°C ("flash anneal"). Typical structures observed after this treatment are shown in Figs. 4-36 to 4-37. It is apparent from Fig. 4-36 that the microstructure viewed at (110) resembles the cold worked structure though the dislocation density was considerably reduced. In this figure two reflections are shown in which all dislocations except $a[110]$ are visible in one or other micrograph. Certain dislocations lying in $[112]$ bands are visible in $g[111]$ implying that they are Lomer dislocations. Although the level of recovery was high the dislocation density was $5.8 \times 10^9 \text{ cm}^{-2}$ and the particle density $3.2 \times 10^{14} \text{ cm}^{-3}$ as compared with $4.6 \times 10^9 \text{ cm}^{-2}$ and $5.05 \times 10^{14}$, respectively, after 1h ageing. It is clear that the major portion of recovery occurred in the first few minutes of ageing as did most nucleation. Fig. 4-37 illustrates the typical structure in three different \{111\} reflections (i.e. \{112\} orientations). The rotation between the three orientations were so large that it was very difficult to follow the various structural features from one micrograph to another. It is apparent from the figure that the majority of precipitate rows lie along [110] traces and that at several rows weak dislocation contrast is evident along their length in certain reflections. However, under these circumstances Burgers vector analysis was difficult because of the large translations of various structural features during tilting. Another complicating factor was the tendency for some precipitates to disappear under certain conditions. Many of the particles apparently exhibited strong strain contrast of the type described by Ashby and Brown. The invisibility phenomenon suggests that either these particles had highly assymmetric strain fields or that they had a bounding dislocation which
exhibited conventional contrast. This point was not resolved in the present work.

A number of regions were found where, unlike that in Fig. 4-37, the various structural features could be readily identified from one orientation to another. One such region is shown in Fig. 4-38 from which it is possible to determine unambiguously the Burgers vectors of dislocations comprising the central band. This has been done and the results are presented in the figure. It is apparent from the figure that most dislocations had precipitates associated with them and that often there appeared to be a merging of the contrast implying that their strain fields interacted as predicted by certain models of dislocation nucleated precipitation\(^1\)\(^2\),\(^1\)\(^3\). Furthermore, certain dislocations have been positively identified as Lomer dislocations. While the incidence of Lomer and Hirth dislocations appeared to be rather low it may be that their effect is more complex in that they could stabilize the neighbouring dislocation structure preventing recovery during the very early stages of ageing. No evidence for dipole nucleated precipitates was obtained and it may be that the precipitate bands having \(<112>\) traces were nucleated by punched out PGD\(^1\)\(^8\).

The results of this study show clearly the intimate relationship between prestrained dislocation structure and precipitation. This is a material property which cannot be changed. It is consequently a limiting factor in achieving an ideally uniform substructure and, thus, on the degree of strengthening produced by MMTT.

4.1.4.5 Hot-Tensile Tests

Two specimens were selected from the hot-tensile tests at 800°C and strain-rates of \(10^{-6}\) and \(10^{-3}\) s\(^{-1}\) for examination by electron microscopy. As well as providing a useful comparison with MMTT specimens subsequently tested under similar conditions, the results of this investigation also yield information relevant to the high temperature MMTT schedules which will be discussed later.

The typical structure after straining beyond the maximum load at \(10^{-6}\) s\(^{-1}\) is shown in Fig. 4-39. Long segments of dislocations can be seen bowing between
irregular arrays of precipitates, some arranged in short rows along [001]. Many of the precipitates appear to have a square shape indicating an effect due either to anisotropy of elastic modulus or surface energy. Many of these long dislocation segments are probably screw in character. There is also evidence of dislocation interactions resulting in the formation of a coarse three dimensional network.

The average dislocation density is no greater than the value in the solution treated condition \(3.1 \times 10^9 \text{ cm}^{-2}\) and the precipitate density is \(7.5 \times 10^{13} \text{ cm}^{-3}\). There is little evidence for large scale subgrain formation although, in a few isolated areas, sub-boundaries were observed particularly in the region of grain boundaries (Fig. 4-40). It may be that this phenomenon is associated with the formation of a denuded zone adjacent to grain boundaries. Not all boundaries have this latter characteristic and it is thought that this might be a function of grain boundary structure \(^{313}\).

The structure after straining to beyond the maximum load at \(10^{-3} \text{ s}^{-1}\) is rather more complex than that after \(10^{-6} \text{ s}^{-1}\). Very crude cellular or subgrain structures are apparent in certain orientations (e.g. Fig. 4-41) while in others, near (110), there is evidence of large scale dislocation motion and slip band formation (Fig. 4-42). The average dislocation density is \(12.5 \times 10^9 \text{ cm}^{-2}\). While some long segments of bowing dislocations are observed (Fig. 4-42), the vast proportion exhibit cusps implying jog formation. Most dislocations are, however, dispersed throughout a fine, irregular network. Precipitates are not as clearly visible as in the \(10^{-6} \text{ s}^{-1}\) structure being considerably smaller and apparently much more randomly dispersed. However, as may be seen in Fig. 4-43 some long rows of fine precipitates are observed along <112> traces. These may be a result of repeated precipitation on jogs along moving screw dislocations. The precipitate density here is difficult to measure due to the fineness of the dispersion, but it was estimated to be approximately \(10^{15} \text{ cm}^{-3}\). No evidence for grain boundary denuded zones was obtained in this case, the grain interior structure extending right up to the boundaries. The structure after the \(10^{-3} \text{ s}^{-1}\) straining is then apparently much more uniform and
of higher density than that after $10^{-6}$ s$^{-1}$.

Straining at the lowest rate leads to no overall increase in dislocation density, while that at the highest leads to gross dislocation generation and multiplication. This reflects the effect of recovery during straining. As the rate of deformation increases so recovery has less effect and presumably at some very high strain rate its effect would be minimal in terms of dislocation density, such that no significant difference would be observed from structures produced at lower temperatures. At the lower strain rate the ductility was only 3% while at the higher it was 14%. For equivalent strains at room temperature dislocation densities of about $10^{10}$ and $3.3 \times 10^{10}$ cm$^{-2}$ were observed. This difference in behaviour illustrates the point concerning recovery. Interpreted in terms of the recovery theory of high temperature deformation, this would indicate a relatively strain-rate independent recovery rate and a high work hardening rate sensitivity. At $10^{-6}$ s$^{-1}$ the 3% strain occurs in 8½h, while the 14% at $10^{-3}$ s$^{-1}$ takes only 2 min. As well as causing the widely differing dislocation densities this fact explains both the precipitate density and size. With higher densities of mobile dislocations the nucleation rate is higher at $10^{-3}$ s$^{-1}$ and due to the vastly reduced duration of the test precipitate particles have time only to barely exceed the critical nucleus size, being only $\sim 100 \AA$ in diameter. The precipitate density is well in excess of that observed in prestrained (2%) and aged material.

Clearly hot deformation of solution treated material has certain advantages over some of the simple prestrain and ageing treatments of section 4.1.4.4 providing high strain rates are used. This observation is consistent with the results obtained in controlled rolling of similar steels$^{267,268}$. However, certain of the higher prestrain treatments, in excess of 5%, give superior substructures and properties as will be seen later. The results presented here do, however, suggest that, even compared with the high prestrain treatments, superior properties might be obtained by the use of industrial strain rates ($\sim 10^1$ s$^{-1}$).
4.1.4.6 Creep Testing

Only one creep tested sample was studied electron microscopically, namely that after creep at 110 MPa and 800°C following the stabilising treatment. Conventional creep subgrain structures were observed as for example those shown in Fig. 4-44. It is evident that the material was adequately stabilised by ageing for 50h at 800°C prior to testing since no signs of fine scale precipitation were observed. In general the subgrain boundaries were associated with precipitate particles and an average subgrain size of 5 µm was measured. The average dislocation density, which includes both subgrain interior dislocations and those in sub-boundaries, was found to be $3.7 \times 10^9$ cm$^{-2}$.

It is difficult to justify the use of these parameters as a realistic comparison with other treatments after creep, since the specimens was not cooled under load due to the short duration of the test. In other words, the specimen failed and was at the creep temperature (800°C) for approximately $\frac{1}{4}$h in an unloaded state prior to cooling. During this period a certain amount of recovery could have occurred. However, it is possible to say that, while not being truly typical of the substructure formed during creep under these conditions, it does represent a reasonable qualitative comparison with other treatments with regard to the general characteristics of the substructure.

This result illustrates that in this stabilised condition creep structures similar to those observed in pure materials are formed. These consist of relatively equiaxed subgrains within which a relatively coarse three dimensional dislocation network is observed. In contrast, the hot-tensile substructures discussed in section 4.1.4.5 do not exhibit this characteristic, although, in terms of strain rate and applied stress, they should be roughly equivalent. The ragged cellular structures or, at low strain rates, irregular dislocation networks form as a result of the dynamic strain ageing processes which accompany deformation and reduce the dislocation mobility. In this respect the hot-tensile tested solution treated samples resemble the structure of Fe/C alloys strained at 150°C - 250°C.
4.2 Multiple-Mechanical-Thermal Treatments

4.2.1 MMTT Schedules - General

To avoid a protracted search for the optimum MMTT conditions it was decided to concentrate on a limited number of MMTT schedules only. The ageing time during each cycle was kept constant while three different prestrain treatments were used. It was arbitrarily decided that the ageing times used should be chosen on the basis of a rather simple diffusion calculation.

The object of MMTT is to lock dislocations to produce a comparatively uniform network. Such a network with a dislocation density of $10^{10} \text{ cm}^{-2}$ was thought to be a sensible goal since this should, if adequately stabilised by pinning particles, possess good flow resistance especially at high temperatures. Using a simple network model, it can be shown (see chapter 1) that the particle spacing required for stabilisation against recovery is approximately equal to the dislocation node separation, which is turn is proportional to the reciprocal square root of the dislocation density ($\rho^{-1/2}$), i.e. 0.1 $\mu$m. To achieve this and have a low solute supersaturation in the matrix would require a time ($t$) of approximately $t = x^2/2D$, where $x$ is half the mean particle separation and $D$ the diffusion coefficient of the solute (Nb) at the ageing temperature. In other words, each particle must drain solute from a sphere of radius equal to half the mean particle spacing. At temperatures of 700°, 750° and 800°C these times are 10, 2 and $\frac{1}{4}h$ respectively. This calculation would seem to be a valid approximation for the low prestrain MMTT, where dislocation densities induced by the 2% prestrain were of the order of $10^{10} \text{ cm}^{-2}$. For the high strain MMTT (5 and 10% per cycle) this is unlikely to be true. However, to provide a fair comparison these ageing times were used throughout the work. The choice of 2, 5 and 10% prestrains was arbitrary, since, unlike the bcc metals and alloys, no convenient indicator, such as the discontinuous yield point, existed in the alloy studied.

It would seem sensible at this point to summarise the main points about MMTT together with details of the treatments studied. A number of MMTT cycles each
consisting of a room temperature prestrain followed by ageing in the range 700° - 800°C were applied to sheet tensile specimens in the solution treated condition. Two solution treatments were used prior to MMTT (1200°C 1h and 1300°C 1h). Treatments were used which involved 2% prestrain per cycle and ageing at either 800°C for 1h, 750°C for 2h or 700°C for 10h. A limited number of high strain MMTT (5 and 10% per cycle) were used together with the 800°C ageing treatment. Up to 13 cycles of the 2% MMTT, 5 of the 5% and 3 of the 10% treatments were applied and the microstructures and properties studied. It should also be appreciated that the level of strain per cycle is subject to a margin of error such that 2%, 5% and 10% prestrains lie in the ranges 1.5 - 2.5%, 4 - 6% and 9 - 12%, respectively, although when averaged over a large number of tests they lie fairly close to these specific values.

4.2.2 Room Temperature Mechanical Behaviour

The variation of 0.2% proof stress and uniform elongation with number of MMTT cycles for the 2% prestrain, 800°C 1h ageing treatment are shown in Fig. 4-45. Also shown is the unloading stress at the end of each prestrain, prior to ageing. The stress rises during each prestrain and follows the course indicated by the arrows during the cycles of straining and ageing. During the first cycle of MMTT there is little change on ageing, the proof stress is only slightly higher than the unloading stress prior to ageing. However, during subsequent MMTT cycles softening occurs during ageing although the proof stress increases steadily with the number of MMTT cycles. The rate of increase in proof stress diminishes and the degree of softening appears to approach a constant value with increasing number of cycles. It would appear that both stress parameters tend to some limiting or saturation level as the number of cycles increases and that some form of dynamic equilibrium is approached. This behaviour is more readily appreciated from Fig. 4-46 in which the increase in flow or unloading stress (Δσ_f) and increase in proof stress (Δσ_p) are shown as a function of the number of MMTT cycles. The proof stress behaviour of both RM 9775 and BV 943 was identical to this (for RM 9209 in Fig. 4-45) after the 1200°C solution treatment. The variation of ductility, also shown in Fig. 4-45, appears to
behave in a rather complex manner. It decreases with increasing number of cycles at a gradually reduced rate to 3 MMTT cycles at which point an apparent discontinuity occurs, followed by a similar reduction with increasing number of cycles. This discontinuity appears to coincide with the point at which the total strain \((\varepsilon_c = \text{MMTT prestrain} + \text{ductility})\) deviates in a positive manner from the solution treated ductility. The total strain rises continuously after 3-4 cycles at a rate which decreases slowly with increased MMTT cycling. RM 9779 behaved in an identical manner once again, but BV 943, under all MMTT conditions, exhibited a higher ductility by 0.03 - 0.04.

The effect of both solution treatment and ageing temperature on proof stress were investigated and the results are presented in Fig. 4-47. It is apparent from the figure that raising the solution treatment temperature (to 1300°C) improved the proof stress by 20 MPa after 5 cycles, reflecting the higher solubility at 1300°C and consequently the larger volume fraction of fine carbides after MMTT. No comparison could be made of ductilities with the 1200°C treatment due to the lower overall ductility after high temperature solution treatment (see Table 4-3). Ageing at 750°C 2h had a negligible effect on proof stress, while the lowest ageing temperature schedule (700°C 10h) produced about the same improvement as the higher solution treatment temperature after 5 MMTT cycles. However, with increasing number of MMTT cycles the margin of improvement over the higher ageing temperatures appears to diminish, in which respect it differs from the higher temperature solution treatment schedule. This apparently enhanced strengthening during the first 5 MMTT cycles may be due to a reduced level of dislocation recovery at 700°C or more profuse precipitate nucleation. No significant difference in ductility was observed after either ageing treatment as compared with the data shown in Fig. 4-45. The ultimate tensile stress of all specimens treated using the 2% schedule appeared to be completely unchanged within the limits of experimental scatter (+ 15 MPa).

The mechanical properties resulting from the 5% MMTT schedule are illustrated in Fig. 4-48. Proof and unloading stresses follow a similar trend as in the 2%
strain treatment, although the stress levels were generally higher, as was the
degree of recovery. Ageing was always accompanied by at least a small amount of
recovery which increased with the number of MMTT cycles. The ductility and total
strain follow a similar trend, but no discontinuity appears in the former, although
it is evident in the latter. However, the ultimate tensile stress after this
treatment increases steadily with the number of cycles to an extent such that after
5 cycles it has risen by about 10%. The results for the 10% MMTT appear to follow
almost exactly the same trends as shown in Fig. 4-49 although the number of cycles
that can be applied before failure is reduced further and stress levels are
generally higher.

A comparison of the various MMTT treatments is shown in Fig. 4-50 together
with the average tensile stress-strain curve for solution treated specimens, which
represents the degree of strengthening achieved by cold work hardening. As regards
room temperature strength, it is apparent that cold working is more effective than
either of the MMTT schedules used. Furthermore, the MMTT strengthening, while being
inferior, increases with the degree of prestrain per cycle. This indicates that the
major contribution to strengthening after MMTT is gained in the first cycle, at
which point the strength differs little from that achieved by direct cold working.
This leads to the conclusion that, since the levels of recovery are high in the
latter stages of MMTT, a major contribution to strengthening is provided by the
early precipitation. It is also apparent that the first prestrain must affect the
precipitation process in such a way that the decrease in proof stress during the
first cycle is so small. Increasing the degree of prestrain must therefore either
accelerate the rate of precipitation so reducing the level of recovery, which
itself should be accelerated, or increase the nucleation rate so compensating, for
higher levels of recovery with increased particle strengthening. The preliminary
ageing studies (section 4.1.4.4) support a combination of these factors.

A comparison of ductility and total strain as a function of MMTT prestrain for
the various treatments is presented in Fig. 4-51. It is apparent from the figure
that these are relatively treatment insensitive following the same trends as
described earlier. The transition observed after 3 cycles of the 2% MMTT is not
clearly distinguishable because of the considerable random scatter in the results
and the reduced quantity of data with the higher strain treatments. It would,
however, appear that ductility after 1 cycle of 5% MMTT and that after 2 cycles
follows the same trend as the 2% MMTT, providing the same degrees of prestrain are
compared.

4.2.3 Effect of Prolonged High Temperature Exposure on MMTT Strength

A series of annealing tests were performed on specimens after 5 MMTT cycles
(2% prestrain, 800°C, 8 h ageing) in order to assess the effect of prolonged exposure
at 800°C on both mechanical properties and structure. The degree of recovery was
determined by retesting at room temperature and observing the change in 0.2% proof
stress. Structural changes will be discussed in a later section.

The variation of proof stress with annealing time at 800°C is shown in Fig. 4-52.
After 5 MMTT cycles the strength falls from the value of 362 to 336 MPa at 150 h and
315 MPa at 900 h; a decrease (Δσ) of 26 and 47 MPa, respectively. Each of these tests
was compared in Fig. 4-52 with the mean proof stress after 5 MMTT cycles. A more
meaningful parameter would be obtained if the annealed proof stress could be
compared with a specimen which, during the fourth and fifth cycles, received the
same MMTT prestrains and exhibited as near as possible identical flow and proof
stress behaviour. This should eliminate some of the experimental scatter due to
the variation in proof stress values after 5 MMTT cycles (362 ± 8 MPa). The stress
changes (Δσ) accompanying annealing, obtained by applying this correction are shown
as a function of time in Fig. 4-53 and it is apparent that the desired effect of
reducing scatter is achieved.

One commonly used relationship describing the recover of cold worked materials
during annealing employs a fractional residual strain hardening parameter which is
often found to decay logarithmically with time to zero. An attempt was made to fit
the data presented in Fig. 4-53 to such a relationship but little correlation was
obtained. A simpler function of the form

\[ \Delta \sigma = k t^m \]  \hspace{1cm} (4.6)

where \( t \) is the annealing time, \( k \) a rate constant incorporating thermal activation terms and \( m \) a constant was used with more success as may be judged from Fig. 4-54. The constants \( k \) and \( m \) in this relationship (equation (4.6)) were found to be \( 4.7 \times 10^{-2} \) MPa s\(^{-m}\) and 0.42, respectively. For the same treatment, 5 MMTT cycles, annealing for 450h at 800°C resulted in a decrease in proof stress of 32 MPa as already shown, while 420h at 850°C produced a decrement of 123 MPa. This implies an acceleration in recovery rate of about 400% for a 50°C temperature rise. However, an insufficient number of tests were performed and no clear conclusion as to the rate controlling mechanism may be made at this stage.

4.2.4 Hot-Tensile Behaviour

A series of hot-tensile tests have been performed after 5 MMTT cycles (2%) in order to assess the mechanical strengthening derived from MMTT during short term exposure at elevated temperatures. These tests may be compared with equivalent results obtained for solution treated and stabilised material at 800°C (4.1.2.2).

A summary of the mechanical properties after 5 MMTT are presented in Table 4-9 together with data for the solution treated material at 800°C. A typical tensile curve is shown in Fig. 4-55 together with that for the solution treated material at a comparable strain rate and temperature. It is apparent from the figure that after MMTT the tensile behaviour is characterised by a very rapid rise in flow stress followed by deformation at ostensibly constant stress until failure, unlike the solution treated material which exhibited the more conventional type of stress-strain curve. In this respect it is similar to the stabilised material (c.f. Fig. 4-5), but differs in that the flow stress is consistently lower in the latter case where larger strains (~0.025) are necessary before the steady stress regime is reached. The ductility of the MMTT material is slightly lower than that for solution treated material, while both the proof and ultimate tensile stress are considerably higher at this strain rate \( (10^{-5} \text{ s}^{-1}) \). These trends are clearly apparent from Table 4-9 at strain rates of \( 10^{-4} \text{ s}^{-1} \) and below. At higher strain rates the ductility remains
Table 4-9

Summary of hot-tensile test data for solution treated and 5-MMTT material at 800°C

<table>
<thead>
<tr>
<th>( \dot{\varepsilon} ) (s(^{-1}))</th>
<th>Solution treated</th>
<th>5 MMTT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \sigma_{0.2} ) (MPa)</td>
<td>( \sigma_{UTS} ) (MPa)</td>
</tr>
<tr>
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<td>71.6</td>
<td>79.0</td>
</tr>
<tr>
<td>( 10^{-5} )</td>
<td>79.5</td>
<td>105.0</td>
</tr>
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</tbody>
</table>
below that of the solution treated material, but the ultimate tensile stress of the latter now exceeds that of MMTT specimens. While the proof stress of solution treated and stabilised material is little affected by strain rate, that of MMTT specimens increases very rapidly with increasing strain rate. The degree of work hardening as measured by the difference between proof and ultimate tensile stress appears to be relatively constant after MMTT at strain-rates of $10^{-4}$ s$^{-1}$ and below. At higher strain-rates the degree of work hardening rises progressively from the level at low strain rates (15 - 25 MPa) to 42 MPa at $10^{-2}$ s$^{-1}$. This latter behaviour occurs throughout the strain-rate range in both solution treated and stabilised conditions. The ultimate tensile or steady stress is sensitive to strain-rate and Fig. 4-56 illustrates this dependence together with data from Fig. 4-6 for comparison. At strain-rates of less than $10^{-4}$ s$^{-1}$ the relationship of equation (4.2) appears to describe this behaviour adequately. The stress exponent $n$ has a value of 14.70 in this range of strain-rates. It should be noted that at a strain rate of $10^{-3}$ s$^{-1}$ the ultimate tensile stress of solution treated and MMTT materials are about equal and that above this strain-rate the stress exponent apparently decreases to a value of about 9.0. This would seem to correspond to a change in deformation mode and coincides with the change in work hardening behaviour already described.

The variation of ductility with strain-rate after 5 MMTT cycles is shown in Fig. 4-57 together with the $800^\circ$C test data from Table 4-4 for comparison. It is apparent from the figure that at all strain-rates the ductility after 5 MMTT cycles is consistently lower than that of unprestrained, solution treated material. The accumulated prestrain during 5 MMTT cycles lies between 9 and 11% and comparison with the creep data presented for prestrained (>4%) and aged samples shows good agreement, if this is made for equivalent creep or strain-rates. Such an observation is very interesting, particularly since in the hot-tensile test a constant elongation rate is employed, while in a creep test the correlation is made with minimum creep rate. Comparing, for example, ductilities at two strain-rates
$3.6 \times 10^{-3} \, h^{-1} \left(10^{-6} \, s^{-1}\right)$ and $3.6 \times 10^{-2} \, h^{-1} \left(10^{-5} \, s^{-1}\right)$ where the creep data corresponds, in the case of 5 MMTT cycles, to the equivalent prestrain (10%) and ageing treatment (Table 4-10) shows not only similar trends but almost identical behaviour in terms of absolute values of ductility. This implies that the application of 5 cycles of 2% strain with intermediate ageing is equivalent in terms of damage accumulation to 1 cycle of 10% strain and ageing. The nature of the damage must, therefore, be such that even at low prestrains it cannot be completely removed by intermediate temperature annealing. It is also interesting that two apparently different tests should give such similar ductilities when compared in the manner outlined above.

A number of variable strain-rate tests were attempted at $800^\circ C$ after 5 MMTT cycles. Basically these consisted of two types of test. The strain-rate was either raised steadily through the range $10^{-6} - 10^{-2} \, s^{-1}$ or varied in a random manner throughout this range. Two tests (1,2) were conducted using the former procedure, the results of which are shown in Table 4-11. The loading schedule is shown schematically in Fig. 4-58. It is apparent from the figure that the tensile curve follows the same form as described earlier at the first strain rate, namely a transient region of rising flow stress to a steady stress value. Having reached this steady stress, the strain rate was increased and a similar curve obtained. Following this procedure a series of curves were obtained which differed little from those observed in simple constant strain rate tests. The steady stress values obtained in this fashion (see Table 4-11) appear to be in excellent agreement with the constant strain-rate values quoted earlier in Table 4-9. The extent of the work hardening portion of the tensile curve, while being similar for the first strain-rate, apparently decreases in subsequent stages to a more or less constant level as measured by the strain to the onset of the steady stress regime ($\Delta \varepsilon_s$). However, at the highest strain rate ($10^{-2} \, s^{-1}$) $\Delta \varepsilon_s$ increased appreciably. It is probable that, at the intermediate strain rates, this transient behaviour is related to some process other than work hardening such as, for example, anelastic
Table 4-10
Comparison of hot-tensile and creep ductilities for stabilised, solution treated and 5-MMTT material at 800°C

<table>
<thead>
<tr>
<th>Test</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(\dot{\varepsilon})</td>
<td>Stabilized</td>
<td>Solution treated</td>
<td>5MMTT cycles</td>
</tr>
<tr>
<td>Creep</td>
<td>(3 \cdot 6 \times 10^{-3} h^{-1})</td>
<td>6%</td>
<td>2.5%</td>
<td>1.2%</td>
</tr>
<tr>
<td></td>
<td>(\equiv 10^{-6} s^{-1})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hot tensile</td>
<td>ditto</td>
<td>8%</td>
<td>3.0%</td>
<td>1.5%</td>
</tr>
<tr>
<td>Creep</td>
<td>(3 \cdot 6 \times 10^{-2} h^{-1})</td>
<td>12%</td>
<td>8.0%</td>
<td>4.0%</td>
</tr>
<tr>
<td></td>
<td>(\equiv 10^{-5} s^{-1})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hot Tensile</td>
<td>ditto</td>
<td>12.5%</td>
<td>5.0%</td>
<td>3.2%</td>
</tr>
<tr>
<td>No</td>
<td>True steady stress (MPa)</td>
<td></td>
<td>Test 1</td>
<td>Test 2</td>
</tr>
<tr>
<td>----</td>
<td>-------------------------</td>
<td>---</td>
<td>--------</td>
<td>--------</td>
</tr>
<tr>
<td></td>
<td>$\dot{\varepsilon}_{1,2}$ (s$^{-1}$)</td>
<td>$\dot{\varepsilon}_{3}$ (s$^{-1}$)</td>
<td>$\dot{\varepsilon}_{4}$ (s$^{-1}$)</td>
<td>$\dot{\varepsilon}_{5}$ (s$^{-1}$)</td>
</tr>
<tr>
<td>1</td>
<td>$1 \times 10^{-6}$</td>
<td>—</td>
<td>138</td>
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<td>$1 \times 10^{-5}$</td>
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<td>216</td>
<td>219</td>
<td>$1 \times 10^{-5}$</td>
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<td>5</td>
<td>$1 \times 10^{-5}$</td>
<td>261+</td>
<td>266+</td>
<td>$1 \times 10^{-3}$</td>
</tr>
<tr>
<td>6</td>
<td>failure</td>
<td>failure</td>
<td></td>
<td>$1 \times 10^{-4}$</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td>$1 \times 10^{-3}$</td>
</tr>
<tr>
<td>8</td>
<td>$1 \times 10^{-2}$</td>
<td>256+</td>
<td></td>
<td>$1 \times 10^{-2}$</td>
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<tr>
<td>9</td>
<td></td>
<td>failure</td>
<td>failure</td>
<td>$1 \times 10^{-3}$</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td></td>
<td>$1 \times 10^{-2}$</td>
</tr>
</tbody>
</table>

* Not strained to steady stress but to between 1 and 2 % only.
† Not steady stress but true stress at maximum load.
relaxation processes. Clearly at the highest strain rate quite a large degree of work hardening occurs as with the constant strain rate test at $10^{-2} \text{ s}^{-1}$. This implies that large scale dislocation generation occurs, a possibility borne out by the other variable strain rate tests and subsequent quantitative electron microscopy.

Two sets of tests were performed in the manner shown schematically in Fig. 4-59. Instead of increasing the strain rate in steps as in Fig. 4-58, here the strain rate was varied in a pseudo-cyclic manner throughout the range $10^{-6} - 10^{-2} \text{ h}^{-1}$. Consequently the strain-rate was decreased at some points and increased at others. In the first set of tests (3,4), which were similar in essence to Fig. 4-59, the strain rate was not allowed to exceed $10^{-3} \text{ s}^{-1}$ until the end. Under these conditions the same comments made concerning tests of the type portrayed in Fig. 4-58 apply. In other words little difference was observed from the constant strain-rate behaviour as regards steady stress values. The comments concerning $\Delta \kappa_s$ apply here also. This behaviour is apparent both from Fig. 4-59 and Table 4-11. It is interesting that the stress relaxed to a steady stress when the strain rate was decreased, in a similar manner to normal stress relaxation tests, although under the conditions studied the decay rate was higher than is usually observed in proper stress relaxation tests. In the final series (5,6) at some points early in the test, straining at $10^{-2} \text{ s}^{-1}$ was performed to between 1 and 2% no steady stress being observed. Prior to the application of the highest strain rate the steady stress behaviour was as described. However, after straining at $10^{-2} \text{ s}^{-1}$ the material still appeared to exhibit steady stress behaviour but the levels were approximately 5-10% higher and after the second straining at $10^{-2} \text{ s}^{-1}$ about 10-15% higher. This behaviour would seem to support the view already expressed that, at the highest strain rate used, structural changes associated with work hardening and dislocation generation occur.

4.2.5 Creep Properties

Creep tests have been performed to evaluate not only the degree of creep resistance introduced by MMTT processing, but also the nature of the structural
strengthening mechanisms. To this end a number of different tests, including variable loading and temperature schedules as well as conventional isothermal constant load testing have been carried out and a wide range of MMTT schedules studied. A summary of creep stresses, temperatures, minimum creep rates, rupture lives and rupture ductilities for all tests on MMTT specimens is presented in Appendix 5 and variable stress creep results in Appendix 4.

4.2.5.1 Constant Load Tests

The majority of tests have been carried out on specimens having received the low strain (2%) MMTT because of its simpler structure and therefore easier structural examination. Specimens in the 5 MMTT cycle condition were chosen for the most extensive study because this treatment gave a good combination of strength and ductility. Smaller numbers of tests have been performed to assess the effect of treatment variables such as number of cycles and prestrain.

The stress dependence of minimum creep rate after 5 MMTT cycles over a wide range of temperatures is shown in Fig. 4-60. Generally it is not a good policy to accept such small number of data points as shown at certain temperatures. However, since the number of data points at 800°C covers the creep rate range more extensively, it is valid to assume a similar behaviour at other temperatures. The minimum creep rate appears to exhibit a dual dependence on applied stress. At high stresses, in excess of about 70 MPa at 800°C, the stress exponent has a value of 6.05 while at low stresses it is 2.80.

From this data the temperature dependence of minimum creep rate has been obtained at 21 and 47 MPa. The variation of minimum creep rate as a function of the reciprocal absolute temperature is shown in Fig. 4-61. At low stresses (21 MPa) the temperature dependence can be described by an activation energy of 72 kcal mole⁻¹. However, at the higher stress (47 MPa) the data appears to deviate quite widely from this straight line relation. Furthermore, the data at 47 MPa shows a consistent trend of increasing activation energy with temperature from ~70 to 85 kcal mole⁻¹. A variable temperature test was performed after 5 MMTT cycles at a stress of 47 MPa.
in order, firstly, to check the above temperature dependence and, secondly, to check the effect of recovery at a high temperature (900°C) on subsequent creep at lower temperatures. The results of this test are shown in Table 4-12. It is apparent from this table that the temperature was raised in a step-wise manner from 700°C to 900°C. At each temperature the test was allowed to continue for a period of time sufficient for the minimum creep rate to be established. This was judged by comparing the creep rate over periods ranging from 10 min to 1 h, depending on creep rate, with that over the preceding period. After three such periods where the creep rate remained constant to within ±10% the next temperature change was made. A comparison with data presented in Fig. 4-60 and 4-61, or that for stresses of approximately 50 MPa in Appendix 5, indicates that for the first run from 700°C - 900°C the data agrees well with the results of constant load tests (i.e. to within a factor of two). This data, therefore, seems to lend support to the concept of a variation in activation energy over this temperature range. Such an observation is difficult to reconcile with the more usual behaviour observed in creep, of a constant activation energy which generally has a value consistent with that for self-diffusion (69 kcal mole⁻¹) or solute diffusion (84 kcal mole⁻¹).

These results indicate that the relationship of equation (4.2) describes the creep behaviour after MMTT, if suitably modified to account for the applied stress and minimum creep rate. One significant difference does, however, present itself, namely the dual stress dependence and the variation in activation energy with temperature. The remaining data in Table 4-12 follows in the same vein as the recovery measurements in 4.2.3. Following the stepped temperature experiment, the test was continued and the temperature decreased to 750°C after 1.5h at 900°C. This resulted in a threefold increase in minimum creep rate at the lower temperature. Raising the temperature to 900°C for a further 2h caused a further rise in the 750°C creep rate of about 70%. A similar effect was observed during a final cycling of temperature although an increase in creep rate of only 25% was obtained. This result confirms the finding that recovery occurs at high temperatures and may
Table 4-12

Results of variable temperature creep test on 5-MMTT material at 47 MPa

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Time (h)</th>
<th>Minimum creep rate (h⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>700</td>
<td>25.0</td>
<td>2.0 x 10⁻⁶</td>
</tr>
<tr>
<td>750</td>
<td>15.0</td>
<td>1.0 x 10⁻⁵</td>
</tr>
<tr>
<td>800</td>
<td>4.5</td>
<td>3.7 x 10⁻⁵</td>
</tr>
<tr>
<td>850</td>
<td>2.5</td>
<td>2.2 x 10⁻⁴</td>
</tr>
<tr>
<td>900</td>
<td>1.5</td>
<td>1.2 x 10⁻³</td>
</tr>
<tr>
<td>750</td>
<td>13.0</td>
<td>4.5 x 10⁻⁵</td>
</tr>
<tr>
<td>900</td>
<td>2.0</td>
<td>2.1 x 10⁻³</td>
</tr>
<tr>
<td>750</td>
<td>10.0</td>
<td>7.8 x 10⁻⁵</td>
</tr>
<tr>
<td>900</td>
<td>1.5</td>
<td>7.2 x 10⁻³</td>
</tr>
<tr>
<td>750</td>
<td>50.0</td>
<td>9.8 x 10⁻⁵</td>
</tr>
</tbody>
</table>
explain the apparently high activation energy at the upper end of the temperature range in both the constant load and variable temperature tests shown in Fig. 4-61 for 47 MPa. At 900°C and possibly 850°C sufficient recovery occurs, prior to the establishment of a minimum creep regime, for this to be significantly accelerated.

The variation of minimum creep rate at 800°C as a function of stress and number of MMTT cycles has been studied for both the low strain (2%) and high strain (10%) treatments. These results provide complementary information to that introduced in section 4.2.2 and are shown in Fig. 4-62 and 4-63 for the low and high strain MMTT, respectively. The creep rate for a given applied stress decreased progressively with the number of MMTT cycles to 5 cycles but effectively no further improvement was observed after 13 cycles. To illustrate this behaviour more clearly the minimum creep rate at an applied stress of 90 MPa is shown as a function of the number of MMTT cycles in Fig. 4-64. This tendency towards some saturation level resembles closely the equivalent behaviour in room temperature tensile tests (Fig. 4-45). The high strain MMTT (Fig. 4-63) follows the same tendency to a limiting or saturation value of creep rate after 3 cycles. It is interesting that these two treatments give almost identical results at or near to saturation (Fig. 4-63). This comes as something of a surprise considering the very large strength differential between them at low temperatures (Fig. 4-50).

As discussed in section 4.1.2.2 a series of tests were performed on a number of differently treated specimens to assess the effect of MMTT on creep resistance as compared with other possible treatments. These treatments are summarised in Fig. 4-65 and consisted of the following:—

(a) Solution treatment 1200°C 1h.

(b) Stabilised condition

(c) Solution treatment 1200°C, 1 h., 10% tensile strain, age 800°C 2½ h, and

(d) MMTT to 5 cycles (2%).

Treatment (c) is included for comparison, since it provides the single cycle equivalent to 5 MMTT in terms of total pre-strain and total ageing time. A number
of points emerge from this work. Firstly, MMTT produces higher creep resistance than any of the other treatments. Secondly, the application of 10% strain in 5 stages of 2% each is more effective than the single stage treatment. Thirdly, solution treated material exhibits higher creep resistance than that in the stabilised condition. This illustrates that considerable solute and dispersion strengthening accompany the dynamic strain ageing process occurring at the creep temperature. A similar effect is evident in hot tensile tests (Table 4-4).

Creep ductilities appear to conform to the pattern already outlined in section 4.2.4 for hot tensile tests. Both stabilised and MMTT ductilities tend to a minimum or at least limiting value with decreasing creep rate, although, as before, the MMTT prestrain lowers the ductility appreciably (Fig. 4-66). All MMTT prestrains correlate well with the behaviour of single prestrain and aged specimens shown earlier in Fig. 4-9. With the lower creep rates observed after MMTT there appears to be some evidence for a ductility minimum as a function of creep rate. Insufficient data is available to clearly define this minimum but the limited data indicates the existence of such a tendency. Several general comments may be made concerning the results. The ductility minimum appears to occur at about 0.5% strain and extend over a wide range of creep rates. At 800°C it appears to occur at a creep rate of about $2 \times 10^{-5}$ h$^{-1}$, while at 850°C and this creep rate the ductility is between 2 and 3%, and the minimum apparently occurs at creep rates in excess of $10^{-4}$ h$^{-1}$. Although no test was performed to failure at 800°C and creep rates of $10^{-6}$ h$^{-1}$, it can be inferred that ductilities well in excess of 1% might be expected. At the end of secondary creep (913h), when the 21 MPa test was terminated, a creep strain of 0.56% had accumulated. In other tests exhibiting this behaviour ductilities of about 1.5 - 2.0% resulted. It is also apparent from the figure (Fig. 4-66) that at 750°C the minimum probably occurs at even lower creep rates. It therefore appears that the ductility minimum occurs at higher creep rates with increasing creep temperature, an observation not inconsistent with ductility minima reported in constant strain rate, variable temperature tests on a number of
A similar ductility minimum has been observed as a function of applied creep stress in Nimonic 80A.

Rupture ductilities have been reported for a number of MMTT and alternative treatments. The variation of rupture life with treatment has so far received no mention. Because of the use of step-load testing for a number of the preliminary tests, this data is not available for some of the 'standard' treatments. However, sufficient data is available for a comparison to be made between MMTT rupture lives and those after the stabilising treatment.

Such a comparison is shown in Fig. 4-67 where the rupture life \( t_R \) is plotted as a function of minimum creep rate \( \dot{\varepsilon}_M \). A near linear relationship is found to describe this variation, i.e.

\[
T_R = K \dot{\varepsilon}_M \tag{4.7}
\]

where \( K \) is a constant which for a given treatment appears to be independent of temperature. MMTT is considered in this case as a single treatment since no systematic variation of rupture life with MMTT prestrain or number of cycles was observed, as is clear from the random scatter around the line in Fig. 4-67. From the figure it is apparent that the rupture life at a given minimum creep rate is appreciably reduced by MMTT. The factor not yet considered and which changes the impression given by Fig. 4-67 is the strength differential between the MMTT and stabilised conditions. The more conventional stress-rupture life (Fig. 4-68) representation puts things into a better perspective. With applied stresses exceeding ~65 MPa at 800°C MMTT produces superior creep rupture lives. This improvement continues with increasing stress. However, at stresses below this level the stabilising treatment yields higher rupture lives. The temperature dependence of rupture life for a given stress is such that a decrease is observed with increasing temperature, yielding an activation energy of 72 kcal mole \(^{-1}\) (Fig. 4-69) at a stress of 50 MPa. Because of the scatter in rupture-life and ductility at rupture, the trends observed previously in stress and temperature dependence of minimum creep rate do not apply to these parameters. Consequently, the stress exponent and
temperature dependence (activation energy) reflect average behaviour. Presumably
the relative magnitude of rupture life after MMTT and stabilising treatment is in
some manner associated with the rupture ductility. This is considerably reduced
by MMTT, particularly at low stresses. The rupture life at a stress of 21 MPa at
850°C (indicated by arrow in Fig. 4-67) is considerably in excess of the value
expected from the rest of the data. This reflects the increased ductility under
the latter conditions. Consequently at very low stresses, i.e. at creep rates below
the ductility minimum, enhanced creep rupture behaviour might generally be expected.
It is interesting to note that this corresponds to levels of stress and creep rate
which are encountered in typical engineering applications, namely stresses and
creep rates in the range 10^-6 - 10^-2 MPa and 10^-8 - 10^-6 h^-1, respectively.

4.2.6 High Temperature MMTT

A series of tests were devised employing high temperature tensile
prestraining and ageing at 800°C in order to investigate the feasibility of
performing MMTT at elevated temperatures. In these tests ageing was carried out
either under stress relaxation conditions or during a period of unloading. A
schematic representation of the first two cycles of a typical treatment, in which
ageing was performed during stress relaxation, is presented in Fig. 4-70. It is
apparent from the figure that the treatment resembles MMTT at low temperatures in
all respects except that ageing is accomplished under relaxed stress conditions.
Consequently the same basic procedure may be employed for computing the variation
of stress and strain during the treatment as in Appendix 3. A small error will be
introduced if the plastic strain during stress relaxation is ignored. Using
reasonable values of modulus (E) and the relationship
\[ \varepsilon_R = \frac{\sigma_S - \sigma_R}{E} \]
which relates the plastic strain during relaxation (\(\varepsilon_R\)) to the stress at the
commencement of relaxation (\(\sigma_S\)) and the relaxed stress (\(\sigma_R\)), it can be shown that
these strains are not expected to exceed 0.1%. This is negligible compared with
the MMTT prestrain. The parameter, $\sigma_s$, is equivalent to the unloading stress of section 4.2.2. In stress relaxation the level $\sigma_k$ after a standard stabilising period (2 min) was measured to provide an alternative indication of the efficiency of MMTT strengthening.

The variation of 0.2% proof stress as a function of the number of cycles and MMTT schedule is shown in Fig. 4-71 and summarised in Table 4-13. It is apparent from the figure that the proof stress rises with the number of cycles in a manner similar to the room temperature treatments (4.2.2), although at considerably lower stresses. Fewer cycles could be performed before the tensile ductility was exhausted. Considering those tests in which ageing was carried out under a relaxed load, the proof stress appears to decrease after the first cycle with increasing relaxation or ageing time. The specimen aged for 10 min under zero load had a superior strength to those aged under a relaxed load. After about 3 cycles there is a tendency for the proof stress to decrease with increasing number of cycles for those treatments employing relaxation times in excess of 10 min. For shorter times and ageing under zero load no such decrease is apparent. These trends seem to be reflected in the relaxed stress ($\sigma_t$) values shown in Fig. 4-72. If it is assumed that the values for the first cycle can be normalised, since this should be treatment independent being related to the unrelaxed stress ($\sigma_s$), the behaviour is almost identical. This is even true for the stress drops after 3 cycles. Reference to Table 4-14 indicates that straining at $10^{-4}$ s$^{-1}$ produces similar behaviour although the stress values are lower due to the strain rate sensitivity of the material.

Also presented in Table 4-13 are the results of the tests to failure after a number of cycles for each treatment at $10^{-3}$ s$^{-1}$. The short relaxation time (2.5 min) test and that conducted with ageing under zero stress exhibit the highest ultimate tensile strengths. The ultimate tensile stresses of the other three tests at $10^{-3}$ s$^{-1}$ each reflect the degree of softening apparently occurring during the penultimate cycle. This softening process may be related to the progress of failure
processes under the relaxed load. Comparison with the tensile data at constant strain rate (4.1.2.2) shows all these treatments to have inferior ultimate tensile strengths and ductilities. In constant strain rate tests these were 214 MPa and 13%, respectively. The total elongation values ($\Sigma \varepsilon$) after MMTT are all virtually constant at about 10% and the final ductility, after $N$ cycles of MMTT ($\varepsilon_{UTS}$), depends on the total MMTT prestrain, such that it has a value of approximately $(10 - \sum_{1}^{N} \varepsilon_{s})\%$. These comments appear to be equally applicable to the $10^{-4}$ s$^{-1}$ tests (Table 4-14) where the final test to failure was performed at $10^{-5}$ and $10^{-3}$ s$^{-1}$.

The idea expressed earlier that the relaxed stress, $\sigma_k$, would act as an indicator of the level of strengthening has been shown to be true in the sense that it follows the trend in proof stress. However, the stress decrement ($\sigma_s - \sigma_k$) observed on relaxation does not appear to show any systematic behaviour during MMTT, other than an increase during the first two cycles (Table 4-15). During subsequent cycles it apparently achieves a treatment independent value of $(65 \pm 4)$ MPa at $10^{-3}$ s$^{-1}$ and $(31 \pm 3)$ MPa at $10^{-4}$ s$^{-1}$. A comparison of the parameter $\sigma_k/\sigma_s$ for the treatments (Table 4-16) indicates for the $10^{-3}$ s$^{-1}$ tests, a treatment and cycle independent value of $(0.64 \pm 0.03)$. For the $10^{-4}$ s$^{-1}$ tests $\sigma_k/\sigma_s$ is slightly higher and appears to exhibit a weak dependence on the number of cycles.

4.2.7 Microstructural Studies of MMTT

Extensive electron and optical microscope studies have been performed on a variety of aspects connected with the MMTT structure. These were conducted in order to relate the manner in which the MMTT substructure built up as a function of cumulative strain and number of cycles to the mechanical behaviour outlined in previous sections. The effect of creep and static exposure at elevated temperatures has also been investigated for the same purpose. These observations may be thought of as generalised studies of average or typical qualitative and quantitative aspects of the substructure. Quite refined analyses have been performed in specific cases involving Burgers vector, trace and orientation analysis. Finally, a considerable amount of optical metallography was carried out to investigate certain aspects of
### Summary of high temperature MMTT at 800°C and $10^{-3}$ s$^{-1}$

<table>
<thead>
<tr>
<th>CYCLE</th>
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<th>$t_r=10$ min</th>
<th>$t_r=10$ min*</th>
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<th>$t_r=60$ min</th>
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<td></td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td></td>
<td>$C_{0.5}$ (MPa)</td>
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<td>90</td>
<td>82</td>
<td>79</td>
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<td>138</td>
<td>129</td>
<td>132</td>
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<td>87</td>
<td>82</td>
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<td>148</td>
<td>148</td>
</tr>
<tr>
<td></td>
<td>$C_{1.5}$ (MPa)</td>
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<td>170</td>
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<td>117</td>
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<td>187</td>
<td>179</td>
<td>169</td>
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<td>$\varepsilon_{r}$ (%)</td>
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<td>177</td>
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<td>132</td>
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<td>113</td>
<td>105</td>
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<td>$\varepsilon_{r}$ (%)</td>
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<td>$C_{1.5}$ (MPa)</td>
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<td>$C_{2.5}$ (MPa)</td>
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<td>$\varepsilon_{r}$ (%)</td>
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<td>$C_{1.5}$ (MPa)</td>
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<tr>
<td></td>
<td>$\varepsilon_{r}$ (%)</td>
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<td>3.62</td>
<td>0.4</td>
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<td>$\Sigma \varepsilon$</td>
<td>0.097</td>
<td>0.108</td>
<td>0.097</td>
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</tr>
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</table>

* Ageing carried out in unloaded condition
**Table 4-14**

Summary of high temperature MTT at 800°C and 10⁻⁴ s⁻¹ with tests to failure at 10⁻¹⁰ and 10⁻⁸ s⁻¹.

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<tr>
<td></td>
<td>$\sigma_{02}^*(MPa)$</td>
<td>82</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0s}^*(MPa)$</td>
<td>116</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0k}^*(MPa)$</td>
<td>83</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_{0s}^*(%)$</td>
<td>0.93</td>
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<tr>
<td>II</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>$\sigma_{02}^*(MPa)$</td>
<td>126</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0s}^*(MPa)$</td>
<td>131</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0k}^*(MPa)$</td>
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<td></td>
<td>$\varepsilon_{0s}^*(%)$</td>
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<tr>
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<tr>
<td></td>
<td>$\sigma_{02}^*(MPa)$</td>
<td>132</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0s}^*(MPa)$</td>
<td>131</td>
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<tr>
<td></td>
<td>$\sigma_{0k}^*(MPa)$</td>
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<tr>
<td></td>
<td>$\varepsilon_{0s}^*(%)$</td>
<td>1.14</td>
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<tr>
<td>IV</td>
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<tr>
<td></td>
<td>$\sigma_{02}^*(MPa)$</td>
<td>131</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0s}^*(MPa)$</td>
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<td>$\varepsilon_{0s}^*(%)$</td>
<td>1.17</td>
</tr>
<tr>
<td>V</td>
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<tr>
<td></td>
<td>$\sigma_{02}^*(MPa)$</td>
<td>130</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{0s}^*(MPa)$</td>
<td>128</td>
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<td></td>
<td>$\sigma_{0k}^*(MPa)$</td>
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<td></td>
<td>$\varepsilon_{0s}^*(%)$</td>
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<td>$\sigma_{ult}(MPa)$</td>
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<td></td>
<td>$\dot{\varepsilon} (s^{-1})$</td>
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### Table 4-16

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<th>( \sigma_k / \sigma_s )</th>
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<td>I</td>
</tr>
<tr>
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<td>(10^{-3})</td>
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</tr>
<tr>
<td>10</td>
<td></td>
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</tr>
<tr>
<td>20</td>
<td></td>
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<td>60</td>
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<td>0.71</td>
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<tr>
<td>60</td>
<td></td>
<td>0.69</td>
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</tbody>
</table>

### Table 4-15

<table>
<thead>
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<th>( t_r ) (min)</th>
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<th>( \sigma_s - \sigma_k ) ((MPa))</th>
</tr>
</thead>
<tbody>
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<td>CYCLE</td>
</tr>
<tr>
<td></td>
<td></td>
<td>I</td>
</tr>
<tr>
<td>2.5</td>
<td>(10^{-3})</td>
<td>51</td>
</tr>
<tr>
<td>10</td>
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<td>42</td>
</tr>
<tr>
<td>20</td>
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<td>60</td>
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</tr>
<tr>
<td>10</td>
<td>(10^{-4})</td>
<td>33</td>
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<tr>
<td>60</td>
<td></td>
<td>34</td>
</tr>
</tbody>
</table>
cavity nucleation and growth during MMTT prestraining and subsequent creep testing. This investigation was augmented by some scanning and transmission electron microscopy.

4.2.7.1 General Features of MMTT

The structural characteristics of room temperature prestrained and aged material (1 complete MMTT cycle) have been described in sections 4.1.4.3 and 4.1.4.4. The effect of subsequent prestraining and ageing cycles on precipitate and dislocation densities and distributions will now be described. Prestrained dislocation distributions were highly banded as is evident in Fig. 4-23. This was shown to be related to the precipitate distribution resulting from ageing to complete the first cycle of MMTT (Fig. 4-33). Because of the complexity and density of substructures formed during the high strain (5% and 10%) MMTT, reliable quantitative data is available only for the low strain (2%) treatment. It is, therefore, the latter treatment that will be discussed first.

A sequence of typical micrographs illustrating the changes occurring during MMTT to 13 cycles is shown in Fig. 4-73. Each micrograph in this series is taken near (110) and consequently illustrates the degree to which the banded structure of the first cycle is modified by subsequent MMTT. Fig. 4-73(a) shows the typical dislocation and precipitate substructure produced by 2% deformation after 1 MMTT cycle. It is apparent that the dislocations generated during straining have a distinct preference for clustering or tangling at particles in the {111} bands. However, this tangling is such that the bands have a considerably more ragged appearance than in material prestrained by 2% in the solution treated condition. Furthermore, the interband dislocation structure is more complex. Continued MMTT to 3 and 5 cycles (Figs. 4-73 (b) and 4-73 (c), respectively) causes a progressive increase in dislocation and particle density and the formation of a finer substructure in interband regions. However, this seems to be accompanied by an increase of dislocation density in the original 1 MMTT cycle precipitate bands. The overall picture does, however, appear to consist of a more homogeneous distribution of substructural features with increasing number of cycles. To investigate the
behaviour during 1 cycle, at a later stage during MMTT, a specimen was strained 2% after 6 MMTT cycles and samples studied in this condition and after subsequent ageing at 800°C for ½ hr (7 complete MMTT cycles). After 6 MMTT cycles and 2% strain (Fig. 4-73(d)) a very ragged form of cellular structure formed in which tangles were associated with precipitate bands. Ageing to complete the seventh MMTT cycle (Fig. 4-73(c)) produced a structure qualitatively similar to that after 5 cycles. The high dislocation density features, associated with precipitate bands, disappeared during ageing, leaving a structure consisting of a considerably reduced overall dislocation density. This, like that after 5 MMTT cycles, consisted of relatively high density regions corresponding to the 1 MMTT cycle precipitate bands, in which dislocations were distributed in a crude planar network. This was interconnected by long segments running from band to band. After the first few cycles it was observed that the major changes in distribution occurred in the interband region. Such changes consisted of a gradual increase in the interband dislocation density and the nucleation of a few precipitates producing a relatively looser network than that in the bands. Qualitatively no further changes appeared to occur during MMTT to 13 cycles, with the exception of the formation of rather diffuse arrays of dislocations, which may have been primitive sub-boundaries, in localised areas (Fig. 4-73(f)). This would seem to suggest some form of mechanical or thermal instability in the MMTT network.

The banded nature of the substructure, even after MMTT, is inherited from the prestrained and aged structure, in other words the first cycle. This characteristic has been shown to be reduced by MMTT, but not eliminated. It is most clearly observed in (110) orientation as in Fig. 4-73(c). Orientations may be selected which give an entirely different impression of dislocation and precipitate distributions. The most notable is (100) in which the structure appears very uniform, as for example in Fig. 4-74. Wherever possible (100) was chosen for dislocation and precipitate density measurements, since average values could then be obtained, without the difficulties associated with counting in the densely
populated bands encountered in most other orientations. Often, however, this was not possible due to limitations inherent in the goniometer stages used.

Finally, some MMTT specimens tested to failure at room temperature were studied. A typical structure observed after 7 MMTT cycles and a post-MMTT strain of 15% is shown in Fig. 4-75. It is apparent from the figure that a well defined high density cellular structure develops which is closely related to the precipitate bands. It is consequently somewhat finer than that developed by cold working solution treated material. The cell walls are somewhat better defined and some degree of streaking and even asterism is observed in SAD patterns (Fig. 4-76) indicating quite large misorientations between cells. In this respect the cold-worked MMTT structure differs considerably from the solution treated and deformed case where cell walls are much broader and more diffuse. This would seem to indicate that lattice strains are more homogenously distributed in the latter case where little streaking or asterism was observed in SAD patterns. The constraining effect of the precipitate bands in MMTT structures may give rise to the localisation of strains necessary to produce such an effect. No systematic work has been performed to accurately measure the misorientations across these cell walls, but diffraction patterns such as that shown in Fig. 4-76 indicate values of about one degree. This type of behaviour contrasts sharply with that of the Fe-0.05%C alloy studied by Liberov et al., in which no well defined cell structures were observed during post-MMTT deformation.

Turning to the higher strain MMTT schedules, the general electron microscopic features after 5 and 10% prestrain and subsequent ageing (1 cycle) have been presented in 4.1.4.3 and 4.1.4.4 (see Figs. 4-19, 4-20 and 4-29). In Fig. 4-77 is shown a typical structure after 4 cycles of 5% prestrain and ageing (800°C ½ h). A very much more complex structure is observed than that found after the low strain MMTT. It is evident that this structure has again inherited many of the features which characterise the 1 MMTT cycle microstructure. However, the bands of precipitate-dislocation structure are less well defined, having a more tangled
appearance. There are also indications that there was greater dislocation activity in the interband region during the second cycle and an associated increase in precipitate density in these areas. These general comments are equally applicable to the 10% MMTT treatment (Fig. 4-78). Furthermore, it appears that the interband activity was so great as to increase the number of these bands formed, reducing their spacing from ~0.70 μm to ~0.30 μm. In this treatment the bands are, however, less ragged in appearance, and the interband structure is considerably more complex.

4.2.7.2 Quantitative Microscopy of MMTT Substructure

The qualitative substructural changes discussed so far give only an incomplete description of the processes occurring during MMTT. Quantitative measurements of dislocation and precipitate densities have been made which allow the structural processes to be described more clearly. A number of these measurements were made at various stages during MMTT schedules up to 13 cycles. These are summarised in Table 4-17 and shown diagramatically in Fig. 4-79. The data is presented corresponding to two stages during MMTT cycles. The first, denoted Nth MMTT cycle, refers to those cases where the examination was carried out after a complete number (N) of MMTT cycles. Secondly, a limited number of studies were performed after N cycles and a further 2% prestrain or what might be called (N + 1) incomplete MMTT cycles. This distinction applies only to the dislocation density data, since the precipitate density is unlikely to be affected by room temperature prestrain.

The rise to saturation levels of both dislocation and particle densities with increasing number of MMTT cycles is apparent in Fig. 4-79. This is in keeping with the mechanical behaviour discussed earlier. Although insufficient data is available for the (N + 1) incomplete cycles condition the trend towards saturation in stress decrement on ageing appears to be reflected in an equivalent dislocation recovery effect after 6 MMTT cycles. At this point both the Nth cycle dislocation and precipitate densities have reached near saturation levels. The precipitate density increased very rapidly during the first cycle and subsequently rose at a steadily decreasing rate with increasing number of cycles, reaching saturation after about
5 cycles. Furthermore, it reached 50% of the saturation level after only 1 MMTT cycle. It is, therefore, not surprising that the precipitate structure formed after the first cycle changes little and that the banded precipitate arrays persisted even at saturation. This behaviour is relatively easily understood in terms of the reduction in chemical driving force for precipitation and, hence, the reduced nucleating efficiency of new dislocations with increasing numbers of MMTT cycles. Such effects are a consequence of the high degree of matrix solute depletion occurring during the first cycle of MMTT. The variation of dislocation density during MMTT appeared to behave in a slightly more complex manner. During the first cycle the net dislocation density increased only slightly indicating a high degree of recovery prior to dislocation locking by precipitates. However, having formed a fine, non-uniformly distributed dispersion during the first cycle, subsequent MMTT cycles produced a comparatively rapid increase in dislocation density. This subsequently appeared to behave in a similar manner to the precipitate density, rising gradually to a limiting or saturation level. Unlike the precipitate density, it did not seem to reach saturation until a larger number of cycles. Indeed this data implies that the choice of 5 MMTT cycles as the treatment for extensive studies of high temperature behaviour was ill-judged from the structural viewpoint, although creep tests would seem to indicate otherwise (Fig. 4-64).

Together with the dislocation and precipitate density values in Table 4-17 is the standard deviation for each result. Each of these is expressed as a percentage of the mean value. To illustrate the scatter involved in the determination of dislocation densities, four typical distributions are shown in Fig. 4-80 in the form of histograms. These serve to illustrate several points regarding both the nature of the dislocation distribution within the material and the validity of the method of dislocation density estimation. Similar distributions were observed in the measurement of precipitate density. In fact, for the cases shown in Fig. 4-80, as with most others, the number of individual measurements in each condition were
### Table 4-17
Summary of dislocation and Nb(C,N) particle densities for MMTT material in the as treated condition

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Dislocation Density ( \times 10^{-9} )</th>
<th>Standard Deviation (%)</th>
<th>Particle Density ( \times 10^{-14} )</th>
<th>Standard Deviation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As solution treated</td>
<td>3.30</td>
<td>+21</td>
<td>0</td>
<td>-</td>
</tr>
<tr>
<td>2% Prestrain only</td>
<td>9.20</td>
<td>+26</td>
<td>0</td>
<td>-</td>
</tr>
<tr>
<td>1 MMTT Cycle</td>
<td>4.55</td>
<td>+22</td>
<td>5.05</td>
<td>+18</td>
</tr>
<tr>
<td>1 MMTT Cycle +2% Prestrain</td>
<td>10.80</td>
<td>+21</td>
<td>5.05</td>
<td>+20</td>
</tr>
<tr>
<td>3 MMTT Cycles</td>
<td>9.35</td>
<td>+12</td>
<td>6.55</td>
<td>+10</td>
</tr>
<tr>
<td>5 MMTT Cycles</td>
<td>11.05</td>
<td>+4.5</td>
<td>7.85</td>
<td>+5.5</td>
</tr>
<tr>
<td>6 MMTT Cycles + 2% Prestrain</td>
<td>14.80</td>
<td>+9.5</td>
<td>7.85</td>
<td>+5.5</td>
</tr>
<tr>
<td>7 MMTT Cycles</td>
<td>12.70</td>
<td>+5.5</td>
<td>8.15</td>
<td>+6.0</td>
</tr>
<tr>
<td>13 MMTT Cycles</td>
<td>13.40</td>
<td>+9.5</td>
<td>9.05</td>
<td>+8.0</td>
</tr>
</tbody>
</table>
similar, the distributions being identical, as may be deduced from the data in Table 4-17. The histograms illustrate certain characteristics of dislocation density measurements.

Firstly, even with small numbers of counts e.g. Fig. 4-80(b) a distinct maximum is generally observed in the frequency-density distribution. The scatter of data about this peak is generally quite symmetrical and the peak corresponds quite closely to the mean values quoted here and elsewhere in the thesis. Data shown in Fig. 4-80(a) for 1 MMTT cycle is fairly typical of those obtained in the case of cold worked structures discussed in section 4.1.4.3 and aged structures in 4.1.4.4. The data shown for 13 MMTT cycles illustrates the more typical distribution and number of counts (\( \Sigma N = 20-35 \)) for the creep and annealed structures discussed in later sections.

Qualitatively, it is apparent that the distributions shown in Fig. 4-80 reflect the general nature of the dislocation structure. After 1 MMTT cycle dislocation structures are generally highly heterogeneous with extremely well defined bands of precipitates and dislocations as seen in (110) orientations. This is also a characteristic of cold worked structures. Even in (100) orientations large area to area variations in dislocation and precipitate density appear to be the rule. Increasing cycles of MMTT lead to a general homogenisation of the structure, at least when observed in (100) orientations. Indeed a noticeable increase in uniformity was observed even in the case of (110) orientations although the banded structure persisted. However, area to area variations do not seem to be quite so important and this is reflected in the width of the dislocation density measurement distribution which decreases during MMTT. The standard deviation or the half peak width at approximately \( \frac{1}{3} \) peak height seems to follow the qualitative substructure changes discussed earlier. This parameter is apparently sensitive enough to indicate such effects as the increase in dislocation density and ragged cellular formations after 2% prestrain following 6 MMTT cycles and the formation of some ill-defined sub-boundaries after 13 MMTT cycles. These are effects, which are clearly confirmed
by qualitative observations and lend strength to the arguments in favour of dislocation density measurements as a useful quantitative technique.

It is felt that one reason for the high sensitivity that appears to have been obtained lies in the more rigorous use of thickness measurement techniques. To illustrate this point the data shown in Fig. 4-80(c) is replotted using average foil thicknesses of 0.2 and 0.5 μm for 100 keV and 1 MeV studies, respectively, in Fig. 4-81. The increase in overall scatter on results is clearly little affected but the overall appearance of the distribution is changed. It no longer exhibits a clearly defined frequency peak and, indeed, gaussian distribution analysis cannot be used with any validity. Incidentally, these values of average thickness are not the same as the average values for Fig. 4-80 but those calculated over a wide range of specimens and treatments. The average thickness values specifically relating to Fig. 4-80 would be 0.23 and 0.44 μm. Using those values adopted in Fig. 4-81 would consequently change the average values of dislocation and precipitate densities also. This latter effect is small. However, the effect on the standard deviation, if this is now a meaningful parameter, is much more dramatic, changing it from the value of 4.5% to 21%. The advantages of using a more rigorous thickness determination method are, therefore, quite considerable.

4.2.7.3 Post-MMTT Hot-Tensile and Creep Substructures

Qualitatively intermediate and high stress creep (50 - 160 MPa) at 800°C and hot-tensile deformation after 5 MMTT cycles produce essentially the same structural changes. This, at stresses below 160 MPa in creep and hot-tensile strain rates below $10^{-3}$ s$^{-1}$, is characterised by Fig. 4-82 which shows the structure after straining to just beyond the steady stress regime in a hot-tensile test at $10^{-4}$ s$^{-1}$ and 800°C. In all general respects this structure is almost identical to that observed prior to high temperature straining. This conclusion was reinforced by quantitative measurements of dislocation and particle densities as shown in Table 4-18. Also included in the table are values for hot-tensile tested solution treated material for comparison. It is apparent from the table that the particle
Table 4-18
Summary of dislocation and Nb(C,N) particle densities for various treatments and test conditions

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Test</th>
<th>( \dot{\varepsilon} ) (s(^{-1}))</th>
<th>( \sigma^* ) (MPa)</th>
<th>Duration of Test (h)</th>
<th>Dislocation Density (x10(^9) cm(^{-2}))</th>
<th>Particle Density (x10(^{14}) cm(^{-3}))</th>
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</thead>
<tbody>
<tr>
<td>As-treated Hot Tensile</td>
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<td>79</td>
<td>-</td>
<td>3.1</td>
<td>0.75</td>
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</tr>
<tr>
<td></td>
<td>( 10^{-3} )</td>
<td>214</td>
<td>-</td>
<td>12.5</td>
<td>10.0</td>
<td></td>
</tr>
<tr>
<td>MMTT</td>
<td>As-treated</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>11.05</td>
<td>7.85</td>
</tr>
<tr>
<td></td>
<td>Hot Tensile</td>
<td>( 10^{-6} )</td>
<td>134</td>
<td>4.5</td>
<td>11.1</td>
<td>7.95</td>
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<tr>
<td></td>
<td></td>
<td>( 10^{-4} )</td>
<td>178</td>
<td>0.45</td>
<td>13.5</td>
<td>7.70</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( 10^{-2} )</td>
<td>230</td>
<td>0.05</td>
<td>18.9</td>
<td>8.05</td>
</tr>
<tr>
<td></td>
<td>Creep</td>
<td>( 7.8 \times 10^{-9} )</td>
<td>50</td>
<td>80</td>
<td>10.0</td>
<td>6.70</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( 4.2 \times 10^{-8} )</td>
<td>80</td>
<td>17</td>
<td>11.5</td>
<td>7.85</td>
</tr>
<tr>
<td></td>
<td></td>
<td>( 2.7 \times 10^{-6} )</td>
<td>160</td>
<td>0.55</td>
<td>12.5</td>
<td>7.80</td>
</tr>
</tbody>
</table>

Minimum creep rate or hot-tensile strain rate where appropriate applied stress or UTS wherever appropriate
density remained relatively unchanged after MMTT, a conclusion quite readily acceptable in the light of the saturation phenomenon of Fig. 4-79. Only in the case of hot-tensile deformation at $10^{-2}$ s$^{-1}$ was there any appreciable change in dislocation density. In the latter case the structure was as shown in Fig. 4-83 from which it is apparent that the increase in dislocation density was due to the production of tangles at particle bands and a general increase in the interband density with more three-dimensional network and more nodes. It is interesting that such behaviour should occur at the fastest strain rate, since this coincided with a transition in strain-rate sensitivity and work hardening behaviour (4.2.6). Furthermore the dislocation density of solution treated material strained at $10^{-3}$ s$^{-1}$ was approximately the same as after MMTT. At the latter strain rate the UTS of MMTT and solution treated material were almost identical (Fig. 4-56 and Table 4-9) and this corresponded to the transition in hot-tensile behaviour already mentioned.

While no signs of large scale recovery and rearrangement of dislocations were observed in grain interiors, grain boundary regions often exhibited such effects, as is apparent in Fig. 4-84. This generally took the form illustrated in the figure, namely the growth of subgrains in a zone ~2.5 μm wide adjacent to and often normal to the boundary. This zone was often denuded of precipitates and any remaining were associated with subgrain boundaries. There was no evidence that such areas increased either in width or frequency at low strain rates, though they were very rarely observed above a strain rate of $10^{-4}$ h$^{-1}$. Furthermore only a small proportion of boundaries appeared to exhibit this behaviour. No systematic study was carried out to confirm this. Since such an effect appeared to occur quite widely at a strain rate in which the test was terminated after 0.45 h it seems reasonable to conclude that either stress or strain assisted recovery processes are important. This conclusion is reached since under normal annealing conditions (4h 800°C) no such effects were observed. It is strictly incorrect to ascribe this phenomenon unequivocably to stress enhanced recovery since it could also arise from the localisation of deformation to grain boundary regions such as would occur if grain
boundary sliding were important during deformation.

Recovery processes such as this and those involving the grain interior structure became more important in low stress creep and long term annealing as discussed in the following section.

4.2.7.4 Low Stress and Recovery Behaviour

Structural changes resulting from long term exposure at elevated temperatures (800°C - 900°C) were studied using both interrupted creep and standard annealing tests after 5 MMTT cycles. The general characteristics of the structural changes are illustrated in Fig. 4-85 for 800°C, 850°C and 900°C anneals. It is apparent from the figure that the density of dislocations and particles decreased and the particle size increased as a function of time and temperature. For example, the structure after 78h at 900°C is qualitatively the same as that after 748h at 850°C. This may be more readily appreciated from quantitative measurements such as those presented in Table 4-19. As with the data for MMTT (Table 4-17) statistical analysis revealed gaussian-like distributions and similar standard deviations in both measured parameters. The variation of dislocation and particle densities with exposure time is illustrated in Fig. 4-86 and 4.87 respectively. As pointed out the dislocation and particle densities decreased with increasing exposure time. This behaviour suggested that, while both recovery and particle coarsening occurred, these processes were inter-related and that the rate controlling process was the latter. The rates of both processes increased with temperature. It is apparent from the figures that no significant difference could be observed between the rates of these processes in crept or annealed specimens. This indicates that stress assisted recovery and particle coarsening do not occur at such low stresses (21 MPa). Furthermore, creep at 800°C for 80h at a stress of 50 MPa did not appear to produce an acceleration of either recovery or particle coarsening, as is evident by comparing the data of Table 4-19 with that of Table 4-18.

As already pointed out the particle distribution of MMTT material is heterogeneous and it appears that this factor gave rise to a variation in coarsening
Table 4-19

Summary of dislocation and Nb(C,N) particle densities after high temperature exposure at 800°, 850° and 900°C in the stressed (21 MPa) and unstressed condition

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Time (h)</th>
<th>Dislocation density (cm⁻² x 10⁻⁹)</th>
<th>Standard deviation (°/o)</th>
<th>Particle density (cm⁻³ x 10¹⁴)</th>
<th>Standard deviation (°/o)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0</td>
<td>11.05 ± 4.5</td>
<td>7.85 ± 5.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>800</td>
<td>150 (A)</td>
<td>9.60 ± 8.5</td>
<td>6.15 ± 7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>450 (A)</td>
<td>8.90 ± 9.0</td>
<td>5.00 ± 8.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>910 (C)</td>
<td>7.00 ± 7.0</td>
<td>3.57 ± 7.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>850</td>
<td>25 (A)</td>
<td>9.80 ± 10.5</td>
<td>7.00 ± 10.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>50 (A)</td>
<td>8.80 ± 7.5</td>
<td>5.40 ± 8.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>80 (C)</td>
<td>8.20 ± 9.0</td>
<td>4.30 ± 9.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>420 (A)</td>
<td>4.70 ± 9.0</td>
<td>2.00 ± 10.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>748 (C)</td>
<td>3.50 ± 9.5</td>
<td>1.10 ± 9.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1152 (C)</td>
<td>2.65 ± 11.5</td>
<td>0.75 ± 12.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>900</td>
<td>78 (C)</td>
<td>5.30 ± 10.5</td>
<td>2.30 ± 10.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
rates, as illustrated in Fig. 4-88. Particles lying in (111) and (111), which form a band at approximately 54° to the foil plane and perpendicular to g, appear to have coarsened more rapidly. The effect could arise either from the fact that, being closely spaced in precipitate bands, diffusion paths are shorter and chemical potential gradients steeper or, because of the higher local dislocation density, pipe diffusion becomes important.

While within grain interiors recovery generally appeared to take the form of network coarsening, localised regions of raft-like arrangements of dislocations were observed. Such a feature can be seen in Fig. 4-85 and was usually associated with {111} precipitate bands. These are thought to be more regular versions of the planar dislocation structure clearly discernable in Fig. 4-88.

The presence of heavily recovered areas adjacent to grain boundaries discussed in the previous section was considerably more noticeable in material annealed for long periods at 850° and 900°C. These generally consisted of more well defined subgrains, the interiors of which were almost completely dislocation and particle free. Such an area is illustrated in Fig. 4-89. It is apparent from the figure that the subgrain so formed has a clearly defined misorientation with respect to the parent grain and that it forms a bulge into the adjacent grain in much the same way as a classical bulge recrystallization nucleus. However, the restraining effect of both grain boundary and grain interior Nb(C,N) particles is apparent. Furthermore, the effect of such recovery events is expected to be minor due to the inhibition of growth and no measureable changes in grain size distribution were observed.

4.2.7.5 High Temperature MMTT

The program of high temperature MMTT described in 4.2.6 was curtailed following the disappointing mechanical properties which resulted from preliminary treatments. However, one specimen which had received 4 cycles of MMTT at a strain rate of 10^-3 s^-1 and aged under relaxed loading conditions for 20 min. was examined.

Typical structures resembled that produced by conventional hot-tensile
straining of solution treated material (Fig. 4-41). However, there was some
evidence of recovery, the overall dislocation density being somewhat lower than in
solution treated and strained material. Precipitate bands were consistent with
ageing during the periods of relaxation employed in each cycle. There was evidence
of ragged though diffuse cellular structure often in localised regions. In this
respect there are some distinct similarities with substructures formed by controlled
rolling\(^{268}\), though, in the latter, subgrains were generally more clearly defined.

4.2.7.6 Miscellaneous Metallography and Fractography

Following the observation of poor creep ductility after MMTT a series of short
studies of microstructural aspects of fracture were performed in an attempt to
identify the likely cause of reduced ductility resulting from MMTT.

Cavities or voids were observed at large inclusions (~5 \(\mu m\)) after prestraining
to 6 or more percent plastic strain at room temperature. Typical cavities observed
after 10\% strain or 5 MMTT cycles and at UTS in the uniform part of the gauge
length, well away from the region undergoing necking (~32\%), are shown in Fig. 4-90.
Usually the voids form at opposite ends of particles on a diameter parallel to the
tensile axis as indicated. The cavity size increases with increasing prestrain.
Other particles with aspect ratios greater than about 2.0 were often observed to have
cracked and the severed portions separated in the tensile direction as shown in
Fig. 4-91. Similar cavities were observed in HVEM studies of prestrained material
(Fig. 4-92).

Ashby\(^{314}\) has predicted, using a model in which the cavities are assumed to
form as a result of interface decohesion due to stress concentrations in the matrix
surrounding the particles, that the ratio of total cavity (\(V_c\)) to particle volumes
(\(V_p\)) should vary linearly with prestrain. To test this model values of \(V_c/V_p\)
obtained from micrographs such as those in Fig. 4-90 are plotted as a function of
prestrain in Fig. 4-93. Typically measurements were made for about 10-12 particles
and considerable scatter, indicated by error bars, was encountered. It is apparent
from the figure that a linear dependence (continuous line) adequately describes the
present data and that, within the limits of the measurements, no significant
difference was observed between MMTT and direct tension. It is also apparent from
Fig. 4-93 that no cavities were observed below about 7% strain. It is unclear
whether this is a physical limit below which cavitation does not occur or whether
it is simply a limit of resolution. From the appearance of cavities at ~10% strain
it would seem likely that, assuming a linear extrapolation of the data in Fig. 4-93
cavities at large particles would be resolvable at strains of 5%. The implication
is therefore that there exists a critical strain below which cavities do not form.

An attempt was made to obtain information on the manner in which cavities
contributed to fracture at low temperatures. Specimens strained to just beyond the
UTS were unloaded and reloaded at a slower rate with the grips misaligned. During
subsequent straining a bending force was applied to the specimen in addition to the
normal tensile stress. These conditions favoured nucleation of a crack, at or near
to one edge, which propagated slowly across the specimen. Straining was discontinued
at a point when the crack had traversed approximately half the specimen width. The
region containing the crack was then mounted, polished and studied in an optical
microscope.

A typical crack produced in this manner is illustrated in Fig. 4-94. The region
of the crack tip is magnified and shown as an inset to the figure. It appears that
the crack changed direction in order to take the most favourable course, probably
that linking cavitated particles, as it moved forward. This view is substantiated
by observations made on other cracks, one of which is illustrated in Fig. 4-95, in
which the direction of propagation changed to link up with a large cavity developing
in the matrix ahead of the crack tip. Particles may be seen imbeded in the crack
walls. Ahead of the crack a large cavity appears to have formed around some
particles and this was linked to the main body of the crack by a series of very fine
cracks along a line $45^\circ$ to the tensile axis. It is also apparent from Fig. 4-95
that ahead of the crack the size of cavities at particles was greater than those
found away from the necked region. Similarly the incidence of cracked particles
appears to be greater near the crack. Using the extrapolation of the data in Fig. 4-93 it seems probable that in such regions very high strains (0.5 - 0.8) are common, implying high local stress and strain concentrations. The latter point was confirmed by electron microscopy in specimens cut from the region of the crack tip (Fig. 4-96). Selected area diffraction patterns taken from such regions display arcs rather than the more usual spot pattern. Such a pattern is shown in Fig. 4-96. Another interesting feature of Fig. 4-96 is the array of small cracks ahead of the larger crack which appears to have been in the process of linking up by shear at approximately 45° to the tensile axis. This type of process may have occurred in the diffuse region of the crack tips in Fig. 4-94 and 4-95. Concentrated shear bands and branching cracks are also clearly visible at the tip of the larger crack in the bottom of the field of view of Fig. 4-96.

Fracture surfaces were studied using both optical and scanning electron microscopy (SEM). A typical SEM micrograph of a fracture surface is shown in Fig. 4-97. The structure resembles that characteristic of ductile fracture consisting of "dimples" which are generally elongated in the direction of crack propagation. No regions of cleavage fracture were observed. An essential feature of this type of fracture was the association of the dimples with second phase particles. These dimples and their associated particles are also clearly visible in the crack walls of Fig. 4-95.

The most striking feature of as crept MMTT material in this regard was the apparently more uniform size distribution of cavities as compared with stabilised or solution treated material. In the latter the cavity size and area distributions were much more heterogeneous as illustrated in Fig. 4-98 in which the appearance of cavities is shown in regions well removed from the necked region after creep to failure at 50 MPa at 800°C. MMTT specimens strained at low rates (~10^-6 s^-1) in hot tensile tests or creep tested at 160 MPa at 800°C exhibited wedge cracks as illustrated in Fig. 4-99. In MMTT material the cracks exhibited a characteristic scalloped appearance as may be seen in Fig. 4-99. Both observations suggest a
link between prestrain induced cavities and subsequent creep cavities. Wedge cracks of the latter type were commonly observed in the necked region of specimens crept at lower stresses, i.e. 50-110 MPa.

Typical cavity densities after a number of creep tests on MMTT and stabilized material are presented in Table 4-20. In each case the tests were to failure and the cavity densities \( N_c \) (in units of number per unit area of polished surface) measured in areas well away from the fracture are given in Table 4-20. The cavity density was greater in MMTT than in stabilized material and not significantly affected by stress. Average cavity sizes were greater in MMTT material due probably to the more uniform size distribution. Both observations suggest prestrain induced cavity formation rather than creep strain or time dependent nucleation. The latter probably occurs in stabilized material.

Interrupted creep tests were performed at 21 MPa and 850°C after 5 MMTT cycles. The effect of creep under these conditions on precipitate and dislocation structures has already been described. Typical appearances of cavities after 80, 748 and 1158h are illustrated in Fig. 4-100. The latter time corresponds to the rupture life under these conditions. It is apparent from the micrographs that the cavities developed individually during the early stages of the test (Fig. 4-100(a)). However, by 748h (Fig. 4-100(b)) comparatively large fissures had formed at transverse boundaries many of which had the scalloped appearance already described. At fracture (Fig. 4-100(c)) these fissures appeared to have linked up and widened. It is evident that such a process contributed significantly to the measured creep strain. This contribution was estimated from carefully polished surfaces using a Quantimet image analysing microscope. The area fraction of cavities was measured and this, being directly related to the volume fraction, yielded values for the gross volume swelling of the samples. If this dilation is assumed to give rise to a net extension of the specimen under creep conditions without any change in cross-sectional area, then the tensile strain contributed by the cavities will be equivalent to the percentage volume swelling \( \frac{\Delta V}{V} \) by the relation
Table 4-20

Summary of cavity density and size measurements for stabilised and 5-MMTT material

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>$\sigma$ (MPa)</th>
<th>$T$ ($^\circ$C)</th>
<th>$\dot{\varepsilon}$ (x10$^{-6}$ h$^{-1}$)</th>
<th>$\varepsilon_R$ (%)</th>
<th>$\tau_R$ (h)</th>
<th>$N_c$ (mm$^{-2}$)</th>
<th>AVERAGE SIZE ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stabilized</td>
<td>50</td>
<td>800</td>
<td>360</td>
<td>4.0</td>
<td>160</td>
<td>295</td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>70</td>
<td>800</td>
<td>4500</td>
<td>6.9</td>
<td>16</td>
<td>208</td>
<td>1.8</td>
</tr>
<tr>
<td>5 MMTT</td>
<td>50</td>
<td>800</td>
<td>28</td>
<td>0.48</td>
<td>84</td>
<td>470</td>
<td>4.0</td>
</tr>
<tr>
<td></td>
<td>80</td>
<td>800</td>
<td>150</td>
<td>0.70</td>
<td>17</td>
<td>445</td>
<td>2.1</td>
</tr>
</tbody>
</table>
The results of these measurements are plotted as creep strain together with the measured creep curve in Fig. 4-101. It is apparent from the figure that the cavity contribution increases steadily during the test and that the major portion of the creep strain probably arises from this source. For long term tests a discrepancy was observed between measurements made on taper sections and sections cut from the centre of the sheet specimens. This illustrates the fact that at or near the specimen surfaces the cavity volume fraction is higher than at the centre. It was commonly observed, as reported by a number of workers, that cavity sizes and densities were greater near the edges of specimens. The assumption that the cavity growth contributed to creep according to equation (4.9) was checked by measuring the linear fraction of cavity along the tensile direction, once again using the Quantimet. For the 748 and 1158h specimens the assumption appeared to have been justified, the actual linear fractions \(\frac{\Delta L}{L}\) being represented by a relation of the form

\[
\frac{\Delta L}{L} = K \frac{\Delta V}{V}
\]

where \(K\) was found to be a constant having a value of 0.95. Consequently the values calculated from equation (4.9) and plotted in Fig. 4-101 are a slight overestimate of the cavity contribution to creep. However, the error is thought to be insignificant being only 5% and thus well within the limits of reliability of the \(\frac{\Delta V}{V}\) values as indicated by error bars in Fig. 4-101. It is apparent from the figure that the creep strain follows the usual three stage time dependence while the cavity contribution appears to be linear and approximately parallel to the minimum creep rate portion of the total creep curve up to about 700h. From these results it would seem that cavity growth constitutes at least 50% of the total creep strain and that after about 200h dislocation and other forms of creep appear to be negligible. The apparent increase in creep due to other sources than cavities above 700h probably arises from necking and accelerated grain boundary fissure growth near the point of fracture.
4.3 Collaborative Studies*

Detailed analysis of sets of stereomicrographs of the type illustrated in Figs. 4-102 and 4-103 has been carried out by Professor Ishida. The HVEM micrographs were obtained by the author using the N.P.L. EM7 microscope for the 5 MMTT material in the as-treated condition and creep tested at 850°C at a stress of 21 MPa.

Two orientations were used in this work, namely (001) and (110). In the former using [200], [020], [220] and [220] reflections all dislocations were visible in one or another reflection, and the Burgers vectors could have been determined. Two vectors only (a/2 [110] and a/2 [110]) were determined unambiguously. However, it was not possible to distinguish between a/2 [011] and a/2 [011] or a/2 [101] and a/2 [101] vectors. Technically, in this orientation, it should be possible to distinguish between a [100] and a [010] dislocations, even though their contrast behaviour was identical to a/2 [101] and a/2 [011] dislocations respectively. This could be achieved by applying Franks rule to dislocation junction reactions. a [001] dislocations were always invisible in (001) orientation. This particular orientation was used in order to obtain dislocation and particle densities, Burgers vector abundance ratios and measurements of the screw character for a/2 [110] and 1/2 [110] dislocations. (110) orientation was used to determine the characteristics of the banded dislocation-precipitate structure. Using [111], [111], [002] and [110] reflections a/2 [110] dislocations were always invisible, a/2 [110] could be unambiguously distinguished but it was not possible to distinguish between a/2 [011] and a/2 [101] or a/2 [011] and a/2 [101].

Most of the essential features of this type of analysis are illustrated in Fig. 4-103, which shows one of the areas studied after 5 MMTT cycles (2%) and creep at 850°C and 21 MPa for 748h. The same area is shown imaged in the two different <200> reflections in (001) orientation, together with a diagram illustrating the

*This work was carried out in collaboration with Professor Y. Ishida of the Institute of Industrial Science, University of Tokyo, who was a Visiting Professor at the University of Surrey from August 1971 to May 1972.
composition of the dislocation network. A table is also included in the figure which summarises the main parameters measured in the analysis. The area used in this analysis is outlined approximately in the accompanying diagram.

The main points emerging from this work are as follows:

(i) The typical MMTT dislocation network was characterised by between 9 and 13 dislocation nodes per circuit.

(ii) The ratio $\frac{p_j^{1/3}}{p_d^{1/2}}$ which indicates the looseness of the dislocation network, was typically $-1.06$ after creep. This implies that the dislocation network remains tight during creep and recovery.

(iii) A proportion (~5%) of dislocations did not conform to the expected contrast behaviour or Frank's rule. These were tentatively assumed to be $a<100>$ dislocations. Examples of dislocation junctions which exhibited this behaviour are shown in Fig. 4-103 at B. Normal junctions composed of $a/2 <110>$ dislocations are illustrated at A.

(iv) Ignoring the small population of $a<100>$ dislocations, the distribution of Burgers vectors appeared to be fairly homogeneous after MMTT and creep testing, as is apparent from the example in Fig. 4-103.

(v) The screw component of $a/2 [110]$ dislocations was analysed after creep, and it was found that 60% of this type of dislocation lay within $20^\circ$ of the pure screw direction. This is clearly apparent from Fig. 4-103.

(vi) After creep 30% of all dislocation segments were found to have at least one small portion of their length lying in a slip plane.

(vii) The long segments of dislocations which apparently join the parallel $(111)$ precipitate bands (Fig. 4-102) are of near pure screw character and appear to make some attractive intersections with network dislocations lying in $(111)$ planes.

(viii) The dislocation network associated with $(111)$ precipitate bands is approximately planar and appears to be roughly hexagonal in form (Figs. 4-88 and 4-102).
In addition to these specific observations a more general statistical analysis of dislocation – particle interactions was also carried out. The results of this analysis are summarised in Tables 4-21 and 4-22. The distribution of Nb(C,N) precipitate particles with respect to the dislocation network was characterised by counting the number of particles not apparently associated with dislocations (0), those associated with single dislocations (0), triple junctions (Δ) and complex junctions (■). The latter junctions consisted of four or more dislocations. From Table 4-21 it is apparent that:–

(i) the precipitate particles in the 5 MMTT material are almost uniformly distributed between the matrix, single dislocations and dislocation junctions.

(ii) the proportion of isolated (matrix) particles (0) decreases after creep.

(iii) the proportion of single dislocation – particle interactions (0) also decreases during creep.

(iv) the proportion of precipitates associated with triple (Δ) and complex (■) junctions increases during creep. The latter were only observed in the presence of precipitate particles, as is apparent from Figs. 4-103 and 4-104, which shows more clearly a junction of this type. These junctions were always composed of only two different Burgers vectors, which were often of the type \( a/2 [110] \) and \( a/2 [110] \) as in the case illustrated in Fig. 4-103 (at C).

The above observations, together with the results given in Table 4-22, in which the number of precipitates lying between dislocation nodes is shown to decrease appreciably after creep, indicate a potent stabilising effect of Nb(C,N) particles on the dislocation network of the MMTT material.
**Table 4-21**
The distribution of Nb(C,N) particles in 5-MMTT material before and after creep testing for 750 h at 850°C at a stress of 21 MPa.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>5-MMTT (%)</th>
<th>Creep Tested (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(0) Matrix</td>
<td>32</td>
<td>23</td>
</tr>
<tr>
<td>(θ) Single dislocation</td>
<td>37</td>
<td>19</td>
</tr>
<tr>
<td>(Δ) Triple junction</td>
<td>16</td>
<td>34</td>
</tr>
<tr>
<td>(σ) Complex junction</td>
<td>15</td>
<td>24</td>
</tr>
</tbody>
</table>

**Table 4-22**
Effect of creep testing for 750 h at 850°C at a stress of 21 MPa on the number of Nb(C,N) particles lying on dislocation lines between nodes.

<table>
<thead>
<tr>
<th>Number of Precipitates between Nodes</th>
<th>5-MMTT (%)</th>
<th>Creep (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>68</td>
<td>92</td>
</tr>
<tr>
<td>1</td>
<td>24</td>
<td>8</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
<td>0</td>
</tr>
</tbody>
</table>
5. DISCUSSION

5.1 MMTT Substructure and Recovery Resistance

The main factors forming the basis of MMTT and influencing the formation of MMTT dislocation substructures in austenitic steels are the introduction of dislocation into the solution treated material and the subsequent precipitation of Nb(C,N) from the supersaturated solid solution. During the first MMTT cycle most of the dislocations introduced by the pre-straining stage are annealed out. Since the dislocations after straining consisted of mobile dislocations, dipoles, and Hirth or Lomer sessile dislocations (Section 4.1.4.3), those remaining would mainly consist of the sessile fraction. During the ageing treatment the nucleation of Nb(C,N) particles would, therefore, take place predominantly at the sessile dislocations, as suggested in Section 4.1.4.4. Besides nucleation of precipitates at dislocations some precipitation apparently occurs within the dislocation-free matrix (Fig. 4-33). During the second MMTT cycle the largest increase in dislocation density occurs (Fig. 5-1), since the dislocations introduced by pre-straining are effectively stabilised against subsequent thermal recovery by the Nb(C,N) particles introduced in the first cycle. The comparatively small increase in the particle density (Fig. 4-79) during the second MMTT cycle is probably due to the tendency for the existing Nb(C,N) particles to grow in preference to fresh nucleation. On subsequent MMTT cycling the dislocation density and particle density increase at decreasing rates, as the substructure tends to attain equilibrium. The MMTT substructure is, therefore, controlled by the distribution of precipitate particles formed during the first ageing treatment, i.e. the first MMTT cycle. This is illustrated in Fig. 4-103 which shows the close association of dislocations with precipitate particles.

The tendency for the dislocation density to reach a saturation level with increasing number of cycles (Fig. 4-79), suggests that dislocation networks are stabilised in a manner analogous to the inhibition of grain growth by second phase particles. This suggestion is supported by the measurements of the mean particle spacing (D) and dislocation mesh size (L) which are related to the...
average dislocation density ($\rho_d$) and particle density ($N_v$) by equations of the form

$$\bar{D} = \beta N_v^{-1/3} \quad \ldots \quad (5.1)$$

$$\bar{L} = \gamma_d \rho_d^{-\frac{1}{3}} \quad \ldots \quad (5.2)$$

where $\beta$ and $\gamma_d$ are constants (about unity). $\bar{D}$ and $\bar{L}$ will be approximately equal if each dislocation node is associated with a pinning particle and

$$N_v^{1/3} \cdot \rho_d^{-\frac{1}{3}} \cdot \gamma_d \beta = \text{constant} \quad \ldots \quad (5.3)$$

$\rho_d$ and $N_v$ for 5 to 13 MMTT cycles and for annealed specimens (5 cycles + 150 or 450h at 800°C) agree well with equation (5.3), the constant $\gamma_d \beta$ ranging from 0.83 to 0.87. Similar values of $\gamma_d \beta$ were obtained from the creep and annealing data shown in Figs. 4-86 and 4-87. These results are summarised in Table 5-1 from which it is apparent that the general tendency is for $\gamma_d \beta$ to decrease during MMTT and subsequent recovery, or creep, attaining a limiting value of about 0.82 after long term creep exposure at 850°C.

In order to test whether this trend was significant, an extensive statistical analysis of dislocation and particle density results was conducted. The main results and conclusions of this work were presented in the section 4.2.7.2 and standard deviations ($S$) for the MMTT and recovery data in Table 5-1. Assuming that the probable error in a parameter $X^n$ is $nSx$ and in a product $X^n Y^m$ is

$$\left[ (nSx)^2 + (mSy)^2 \right]^{1/2},$$

where $Sx$ and $Sy$ are the standard deviations of $X$ and $Y$, respectively, the 99% confidence for $\gamma_d \beta$ is $\pm 4.5\%$. Statistically, therefore, a change in the value of $\gamma_d \beta$ from 0.88 to less than 0.84 is significant, and the trend shown in Table 5-1 is real.

The average dislocation invisibility for these measurements was estimated and found to be about 40%. Applying a correction to the measured dislocation densities used in the evaluation of $\gamma_d \beta$ yields a correction factor of 0.78. $\gamma_d \beta$ approaches a limiting value of 0.8 after MMTT and creep, which corrected for dislocation invisibilities, now becomes 0.625. If $\beta$ is taken as 0.56, the value for a random array of particles^{315-317} (see Appendix 6), the parameter
Table 5-1

Variation of dislocation ($\rho$) and particle ($N_v$) densities and the ratio ($\beta/\gamma$) with various treatments

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Test Temp. ($^\circ$C)</th>
<th>Time (h)</th>
<th>$\rho$ (x10$^9$ cm$^{-2}$)</th>
<th>$Sp$ (%)</th>
<th>$N_v$ (x10$^{14}$ cm$^{-3}$)</th>
<th>$S_{Nv}$ (%)</th>
<th>$\beta/\gamma$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 MMTT Cycle</td>
<td>25</td>
<td>-</td>
<td>4.55</td>
<td>22</td>
<td>5.05</td>
<td>18.5</td>
<td>1.17</td>
</tr>
<tr>
<td>3 MMTT Cycles</td>
<td>25</td>
<td>-</td>
<td>9.35</td>
<td>12</td>
<td>6.55</td>
<td>9.5</td>
<td>0.91</td>
</tr>
<tr>
<td>5 MMTT Cycles</td>
<td>25</td>
<td>-</td>
<td>11.05</td>
<td>4.5</td>
<td>7.85</td>
<td>5.5</td>
<td>0.88</td>
</tr>
<tr>
<td>7 MMTT Cycles</td>
<td>25</td>
<td>-</td>
<td>12.70</td>
<td>5.5</td>
<td>8.15</td>
<td>6.5</td>
<td>0.83</td>
</tr>
<tr>
<td>13 MMTT Cycles</td>
<td>25</td>
<td>-</td>
<td>13.40</td>
<td>9.5</td>
<td>9.05</td>
<td>5.5</td>
<td>0.83</td>
</tr>
<tr>
<td>5 MMTT Cycles + Anneal</td>
<td>800</td>
<td>150</td>
<td>9.60</td>
<td>9.2</td>
<td>6.15</td>
<td>8.3</td>
<td>0.88</td>
</tr>
<tr>
<td>MMTT Cycles + Anneal</td>
<td>800</td>
<td>450</td>
<td>8.90</td>
<td>7.8</td>
<td>5.00</td>
<td>6.3</td>
<td>0.84</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (21 MPa)</td>
<td>800</td>
<td>910</td>
<td>7.00</td>
<td>6.8</td>
<td>3.57</td>
<td>7.15</td>
<td>0.85</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (50 MPa)</td>
<td>800</td>
<td>80</td>
<td>10.00</td>
<td>8.2</td>
<td>6.70</td>
<td>8.0</td>
<td>0.88</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (80 MPa)</td>
<td>800</td>
<td>17</td>
<td>11.50</td>
<td>9.6</td>
<td>7.10</td>
<td>8.7</td>
<td>0.83</td>
</tr>
<tr>
<td>MMTT Cycles + Anneal</td>
<td>850</td>
<td>25</td>
<td>9.80</td>
<td>10.7</td>
<td>7.00</td>
<td>9.80</td>
<td>0.89</td>
</tr>
<tr>
<td>MMTT Cycles + Anneal</td>
<td>850</td>
<td>50</td>
<td>8.80</td>
<td>7.3</td>
<td>5.40</td>
<td>7.85</td>
<td>0.87</td>
</tr>
<tr>
<td>MMTT Cycles + Anneal</td>
<td>850</td>
<td>420</td>
<td>4.70</td>
<td>9.15</td>
<td>2.00</td>
<td>10.30</td>
<td>0.85</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (21 MPa)</td>
<td>850</td>
<td>80</td>
<td>8.20</td>
<td>8.95</td>
<td>4.30</td>
<td>9.40</td>
<td>0.83</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (21 MPa)</td>
<td>850</td>
<td>748</td>
<td>3.50</td>
<td>9.65</td>
<td>1.10</td>
<td>9.20</td>
<td>0.81</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (21 MPa)</td>
<td>850</td>
<td>1152</td>
<td>2.65</td>
<td>11.50</td>
<td>0.75</td>
<td>12.10</td>
<td>0.82</td>
</tr>
<tr>
<td>MMTT Cycles + Creep (21 MPa)</td>
<td>900</td>
<td>78</td>
<td>5.30</td>
<td>10.30</td>
<td>2.30</td>
<td>9.95</td>
<td>0.84</td>
</tr>
</tbody>
</table>
relating dislocation node spacing to density \( (\gamma_d) \) will be 0.9. This value is
to be compared with that determined by Ishida \( \left( \frac{\rho_j^{1/3}}{\rho_d^{1/3}} \right) = 1.06 \), where \( \rho_j \)
is the distance between dislocation nodes and \( \rho_d \) the spacing between dislocations
by a direct method. Ishida also found that after creep the ratio of particle to
node density was 0.75. The use of the network model and the \( (\beta/\gamma_d) \) parameter
underestimates the value of \( \gamma_d \) by a factor of about \( (0.75)^{1/3} \). The probable
value of \( \gamma_d \) determined by the bulk density measurements, using the assumptions
of the network model, is, therefore, about 1.0.

The theoretical relationship for \( \gamma_d \) of equation (5.2) can be derived by the
same statistical arguments as have been used to determine the nearest neighbour
particle separation. The latter gave \( \beta = 0.56 \) in equation (5.1) as
demonstrated in Appendix 6. Also included in Appendix 6 is a treatment of
dislocation network link length distributions using the same analytical methods.
Theoretical values for the parameter \( \gamma_d \) of equation (5.2) are presented in
Table A6-2 for dislocation networks consisting of triple point nodes each of
which is connected to various combinations of nearest neighbours. The simplest
possible combination consists of the case where neighbouring nodes occur at first,
second and third nearest sites. In each of the calculations a parameter \( \gamma_v \)
was evaluated in the relationship

\[
\bar{L} = \gamma_v \rho_v^{1/2}
\]

where \( \rho_v \) is the true volume dislocation density expressed as line length per
unit volume. Schoeck has shown that the dislocation density \( (\rho_d) \) measured
by a line intercept method, expressed as number per unit area, is related to the
volume density \( (\rho_v) \) by the relation

\[
\rho_d = 0.5 \rho_v
\]

Consequently, the parameter \( \gamma_d \) of equation (5.2) may be obtained from the relationship

\[
\gamma_d = \gamma_v \sqrt{2}
\]

and for the simplest case where the first, second and third nearest neighbours
are joined \( \gamma_v = 0.745 \) and \( \gamma_d = 0.53 \). Other combinations of nearest neighbours
are considered in Appendix 6 and it is apparent from Table A6-2 that both \( \gamma_v \) and \( \gamma_d \).
increase with increasing order of nearest neighbour combinations. For more realistic combinations where the contribution of longer dislocation segments is taken into account $\gamma_v$ and $\gamma_d$ approach 1.0 and 0.7, respectively. Consequently, it follows that in most network theories of dislocation strengthening, such as that due to Saada, the assumption that $\bar{L} = \rho_v^{-\frac{1}{2}}$ is valid providing the distinction between $\rho_v$ and $\rho_d$ is understood and included in any application of these models to experimental data.

We are now in a position to estimate a theoretical value for the parameter $\rho_j^{1/3}/\rho_d^{1/2}$ for a tight 3-dimensional dislocation network. As pointed out in Appendix 6, it can be shown that the volume dislocation density ($\rho_v$) is given by

$$\rho_v = \frac{3}{2} \bar{L} \rho_j$$

where $\rho_j$ is the node density. Substitution from equations (5.4) and (5.5) gives ratios of $\rho_j^{1/3}$ to $\rho_v^{1/2}$ and $\rho_d^{1/2}$ of 0.96 and 1.08, respectively, for the case of first, second and third nearest neighbours. As with $\gamma_v$ and $\gamma_d$ values of the above ratio were computed for other combinations of nearest neighbours as discussed in Appendix 6. The ratios decreased with increasing order of nearest neighbour combinations such that for realistic distributions $\rho_j^{1/3}/\rho_v^{1/2}$ and $\rho_j^{1/3}/\rho_d^{1/2}$ approach 0.90 and 1.00, respectively. The measurements of Ishida on the MMTT network after creep and the estimate from the network model of equation (5.3) are in good agreement with the theoretical values computed from the distribution function in Appendix 6. Values of $\rho_j^{1/3}/\rho_d^{1/2}$ less than the theoretical prediction of 1.00 - 1.08 indicate a looser form of network as might be expected if dislocations bow out between pinning points or, due to interactions with particles or point defect clusters, are prevented from relaxing to form straight segments between nodes. Lower values of $\rho_j^{1/3}/\rho_d^{1/2}$ are also expected if, due to crystallographic constraints, dislocations only lie in preferred orientations, forming kinks along their length. It is difficult to accept values greater than the theoretical since, assuming the validity of the calculation, this would imply that certain segments were absent and in such regions the network terminated abruptly at nodes. The
latter clearly contravenes one of the basic premises of dislocation theory\textsuperscript{116}. Indeed, all values reported\textsuperscript{43,320-322} for $\rho_j^{1/3}/\rho_d^{1/2}$ are lower than the theoretical value. In nickel, deformed at room temperature, Lin and McLean\textsuperscript{320} found the above parameter to be 0.9 - 1.1 while in $\alpha$-Fe at temperatures in the range -78° to 400°C values of 0.66 - 1.00 were reported\textsuperscript{43}. After creep at 500°C Ishida and McLean\textsuperscript{321} obtained values in $\alpha$-Fe, Fe-Mn and Fe-Mn-N of 0.44 - 0.56. More recently a value of 0.7 has been measured in crept MgO\textsuperscript{322}. The reported results for pure metals and alloys indicate that during creep dislocation networks are characterised by low ratios of $\rho_j^{1/3}$ to $\rho_d^{1/2}$, implying that only a loose network structure exists. Cold worked metals seem to be generally characterised by values of near unity, and this level is only approached at relatively high strains. After MMTT even when followed by creep or recovery, the dislocation structure is tighter, resembling that produced by cold working\textsuperscript{320}. This presumably is a major factor in high temperature strengthening, though it is intimately connected with the efficient pinning of the dislocation network by Nb(C,N) particles which also retard recovery.

It is evident from Table 5-1 and Figs. 4-86 and 4-87 that in MMTT material the recovery and particle coarsening processes are closely related. From the simple network model outlined above such a relationship between dislocation and particle densities is understandable because of the stabilising effect of Nb(C,N) particles on the MMTT dislocation structure. Furthermore, it is likely that the kinetics of the process is dominated by particle coarsening, which is characterised by the Liftshitz-Slyozov-Wagner relationship\textsuperscript{207-209}

$$r_t^3 - r_o^3 = (8DS\Omega t/9kT) = k_t$$

where $r_o$ is the average initial radius of the particle and $r_t$ that after a time (t), $k$ is a temperature dependent term which includes the solute diffusion coefficient (D), solubility (S), interfacial energy (\gamma), atomic volume (\Omega) and absolute temperature (T). Since most particles exhibited strain contrast, particle size was a difficult parameter to measure accurately and the particle density was used instead. The average particle radius (r) is related to the
particle density \((N_v)\) by a relationship of the form
\[
r = \left( \frac{6f/8\pi N_v}{T} \right)^{1/3}
\]
... (5.8)
where \(f\) is the volume fraction of spherical particles. Therefore, equation (5.7) expressed in terms of particle density becomes
\[
\left( \frac{N_v}{t} \right)^{-1} - \left( \frac{N_v}{o} \right)^{-1} = K't
\]
... (5.9)
where \(K'\) is a temperature dependent constant given by
\[
K' = 8\pi K/6f
\]
... (5.10)
From the network model, equation (5.3), it is apparent that during particle coarsening and recovery the dislocation density should follow the relationship
\[
\left( \rho_t \right)^{-3/2} - \left( \rho_o \right)^{-3/2} = K''t
\]
... (5.11)
where
\[
K'' = 8\pi \beta^3 K/6f \gamma_d^3 = (\beta^3 / \gamma_d^3) K'
\]
The data from Figs. 4-86 and 4-87 is replotted in terms of the parameters \((N_v)^{-1}\) and \(\rho_d^{-3/2}\) in Fig. 5-2 (a) and (b), respectively. It is apparent from the figure that the data is consistent with equations (5.9) and (5.11). The temperature dependence of \(K'\) has been determined and an activation energy of 79 \(\pm\) 8 kcal.mole\(^{-1}\) obtained for the coarsening process, which is similar to that for \(K''\), i.e. recovery (80 \(\pm\) 8 kcal.mole\(^{-1}\)). The measured values of \(K'\) are summarised in Table 5-2 together with those calculated using equation (5-10) with the parameters \(D\) and \(S\) in equation (5.9) taken from references quoted in the table. Allowing for the variations in the diffusion coefficients available, it is apparent from Table 5-2 that the experimentally determined values of the \(K'\) parameter lie within the range indicated by the calculation and exhibit a similar temperature dependence. This is in good agreement with the theory of Speight and Healey\(^{210}\) which predicts that the activation energy for particle coarsening in systems containing stoichiometric compounds such as NbC is \((Q + (\Delta H/2))\) where \(\Delta H\) is the enthalpy of solution of the compound (\(\approx 38\) kcal.mole\(^{-1}\) for Nb(C,N)).

The model outlined above appears to be consistent with Ishida's three-dimensional analysis of MMTT and crept substructures, and accounts for the tendency
of the network to homogenise after creep. Denser regions of the structure tend to coarsen more rapidly (Fig. 4-88) since diffusion paths are shorter and short-circuit paths along dislocations are more numerous. The proportion of particles at triple and higher order junctions was observed to increase during creep (Table 4-21). This presumably is due to the fact that, as the particle density decreases during coarsening, a concommitant growth of the dislocation network occurs and the general tendency will be for the nodes to relax to particle pinning points. The elastic interaction between hard particles and dislocations are such that dislocations are repelled by Nb(C,N) precipitates. Individual dislocations may climb or cross-slip around particles during recovery and creep. The proportion of particles at individual dislocations actually decreased during creep (Table 4-21) in agreement with this argument. Dislocation nodes in a three-dimensional network are, however, less mobile due to the restraining effects of line tension and are, therefore, more likely to be trapped by particles. Further evidence for the strong pinning effect of particles on nodes is to be found in the fact that the proportion of multiple (four-fold or higher order) nodes increased during creep (Table 4-21). The latter have not generally been observed in dislocation structures and are considered to be unstable with respect to either attractive or repulsive junctions \(^{32}\). Their occurrence in MMTT structures, even after prolonged recovery or creep, further supports the concept of strong nodal pinning effects. The poorer stabilising effect on individual dislocation segments shows that McLean's model for network stabilisation by second phase particles \(^{20}\) is incorrect as regards the precise mechanism. It does, however, predict the right order of magnitude of the effect. The model proposed here, with the modification that nodal pinning is the important factor, appears to more accurately describe the behaviour during MMTT and subsequent creep or recovery. Friedel's model for recovery by network growth \(^{323}\), which has been supported by recent observations in nickel base alloys \(^{324}\), cannot explain the behaviour observed in this work. While the driving force for recovery is the same, namely the reduction in dislocation line energy, in MMTT material particle coarsening appears to be the rate controlling process.
### Table 5-2

Comparison of experimentally determined values of \( K' \) (equations (5.9) and (5.10)) with those calculated theoretically

<table>
<thead>
<tr>
<th>( T \ (^\circ C) )</th>
<th>( D_i^{(22)} ) (cm(^2) sec(^{-1}))</th>
<th>( D_{ii}^{(23)} ) (cm(^2) sec(^{-1}))</th>
<th>( S_{Nb}^{(281)} ) (wt %)</th>
<th>( K'_{I} ) (cm(^3) sec(^{-1}))</th>
<th>( K'_{II} ) (cm(^3) sec(^{-1}))</th>
<th>( K'_{EXPT} ) (cm(^3) sec(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>800</td>
<td>( 9 \times 10^{-15} )</td>
<td>( 1.7 \times 10^{-13} )</td>
<td>( 2.9 \times 10^{-2} )</td>
<td>( 3.3 \times 10^{-22} )</td>
<td>( 6.2 \times 10^{-21} )</td>
<td>( 4.6 \times 10^{-22} )</td>
</tr>
<tr>
<td>850</td>
<td>( 5 \times 10^{-14} )</td>
<td>( 6.4 \times 10^{-13} )</td>
<td>( 4.5 \times 10^{-2} )</td>
<td>( 2.6 \times 10^{-21} )</td>
<td>( 3.4 \times 10^{-20} )</td>
<td>( 2.8 \times 10^{-21} )</td>
</tr>
<tr>
<td>900</td>
<td>( 2.5 \times 10^{-13} )</td>
<td>( 2.2 \times 10^{-12} )</td>
<td>( 6.5 \times 10^{-2} )</td>
<td>( 1.8 \times 10^{-20} )</td>
<td>( 1.6 \times 10^{-19} )</td>
<td>( 1.1 \times 10^{-20} )</td>
</tr>
</tbody>
</table>
The apparent 'saturation' of dislocation density occurring after about 5 MMTT cycles (Fig. 4-79) was due to the reduction in precipitate nucleation rate which became negligible, such that the existing dispersion could not support a higher density at the ageing temperature. It also appears that recovery of the excess dislocation density introduced by MMTT pre-straining is very rapid. Continued exposure in the temperature range 800° - 900°C produces a slow decrease in dislocation density (Fig. 4-86) accompanied by a concommitant reduction in particle density (Fig. 4-87) due to particle coarsening. This interpretation is confirmed by the change in the distribution of Nb(C,N) particles as a result of creep testing a 5-MMTT material (Table 4-19). A simple network model appears to describe adequately the 'saturation' behaviour of dislocation and particle densities during MMTT and the subsequent effects of recovery at high temperatures. This reflects the strong pinning effect of Nb(C,N) particles on nodal junctions in the dislocation network. Consequently the MMTT substructure exhibits a very good recovery resistance at temperatures below 800°C particularly in the presence of the high dislocation density that is sustained. The resistance to recovery is solely due to the stability of the Nb(C,N) dispersion. Two independent determinations of dislocation and particle distributions, each employing a different form of analysis, namely bulk density and refined three-dimensional analysis techniques, yielded essentially equivalent results regarding the formation of the MMTT substructure and its thermal stability.  

5.2 Low Temperature Strengthening Mechanism  

From the microstructural characteristics of MMTT material it might be expected that strengthening arises from two main sources -

(a) the dislocation network, and

(b) the Nb(C,N) dispersion.

This hypothesis may be tested by attempting to isolate the individual components of the flow stress and comparing them with established theoretical models. The simplest assumption that can be tested is that these are independent mechanisms, and that the flow stress \( \sigma_f \) of MMTT material is a linear sum of the individual
components, thus

$$\sigma_f = \sigma_0 + \Delta \sigma_N + \Delta \sigma_\rho$$

... (5.12)

where $\Delta \sigma_N$ and $\Delta \sigma_\rho$ are the network and particle contributions to strengthening, respectively.

Considering first the network contribution, it has already been demonstrated that, in the absence of particles, $\Delta \sigma_N$ is in good agreement with equation (4.5). From the discussion of link length distributions in section (5.1) and Appendix 6 it is apparent that in order to compare the parameter $\alpha$ of equation (4.5) with that predicted by theoretical models, the volume density ($\rho_v$) should be used. Consequently, the value of $\alpha$ should be increased by about 40% i.e. a factor of $\sqrt{2}$. However, in the context of equation (5.12) the values of $\alpha$ obtained directly from measurements (i.e. 1.15) may be used. The value of $\alpha$, namely 0.54 (converted to shear stress and volume density), suggests a forest hardening mechanism in which attractive junctions appear to be important. This is supported by the high density of Lomer and Hirth locks in cold worked samples. Furthermore, this conclusion seems equally applicable to MMTT material in which Ishida found that as many as 5% of the total dislocation content consisted of Hirth dislocations.

The particle contribution to the flow stress or Orowan stress can be estimated, for any MMTT condition, as the difference between the flow stress after MMTT and that of solution treated material with the same dislocation density. This data, extracted from Fig. 5-3 and particle densities from Table 4-17, was compared with a recently derived relationship for the Orowan stress

$$\Delta \sigma_\rho = (1.13 m \overline{Gb}/2 \pi \overline{D}) \ln(2r/r_\rho)$$

... (5.13)

in which $m$ is the Taylor factor (3.0), $r$ the mean particle radius and $r_\rho$ the dislocation core radius. If the mean particle radius is assumed constant during MMTT ($\sqrt{100 \overline{D}}$) and $\overline{D}$ given by equation (5.1), equation (5.13) reduces to

$$\Delta \sigma_\rho = \delta \overline{G} \overline{B} N_v^{1/3}$$

... (5.14)

where $\delta$ (≈2.0) is a constant. Results obtained in this manner are shown in Fig. 5-4. The best fit line drawn through the data points and the origin yields a value for
of 0.65, which is about 30% of the theoretical value.

The above analysis is limited by a number of factors:

(a) The validity of equation (5.12), i.e. the linear addition of flow stress contributions

(b) Matrix solute depletion (C,N,Nb) during MMTT

(c) The heterogeneity of the particle distribution and choice of particle parameters, and

(d) The variation of particle size during MMTT.

The first of these factors was considered by several authors\textsuperscript{328,329}, who proposed two basic types of behaviour which are described by:

\[
\sigma_f = \sigma_1 + \sigma_2
\]

... (5.15)

\[
\sigma_f = (\sigma_1^2 + \sigma_2^2)^{1/2}
\]

... (5.16)

where \(\sigma_f\) is the flow stress of a material in which strengthening arises from two mechanisms and \(\sigma_1\) and \(\sigma_2\) are the individual strengthening effects. It is generally accepted that dislocation drag terms and lattice friction stresses are linearly additive to, for example, dispersion hardening or work hardening terms (e.g. equation (4.5)). This is also true for the case where, for example, a high density of weak and low density of strong obstacles co-exist. The second formulation, equation (5.16), is thought\textsuperscript{329} to be applicable in those cases where the densities and strengths of each type of obstacle are similar. Since dislocations and non-deforming particles produce approximately equal strengthening the latter might be a more appropriate form than equation (5.12). Following the same general method as for equation (5.12), the present data has been re-analysed in terms of a relationship similar to equation (5.16) i.e.

\[
\sigma_f = \sigma_o + (\Delta \sigma_N^2 + \Delta \sigma^2) \frac{1}{2}
\]

... (5.17)

It should be noted that the friction stress is taken to be linearly additive and that equation (4.5) is valid for determining \(\Delta \sigma_N\). The values of \(\Delta \sigma'\) and \(\Delta \sigma''\) obtained using equations (5.12) and (5.17) respectively are given in Table 5-3, from which it is apparent that the particle strengthening contribution is greater for equation (5.17) than for equation (5.12). Furthermore, the correction
Table 5-3
Comparison of particle strengthening in MMTT material as calculated using equation (5.12) ($\Delta \sigma^\prime$) and equation (5.17) ($\Delta \sigma^\prime\prime$)

<table>
<thead>
<tr>
<th>Number of MMTT cycles</th>
<th>$\sigma_f$ (MPa)</th>
<th>$\sigma_o$ (MPa)</th>
<th>$\Delta \sigma_N$ (MPa)</th>
<th>$\Delta \sigma^\prime \rho$ (MPa)</th>
<th>$\Delta \sigma^\prime\prime \rho$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>258</td>
<td>-32</td>
<td>198</td>
<td>92</td>
<td>212</td>
</tr>
<tr>
<td>3</td>
<td>343</td>
<td>-32</td>
<td>278</td>
<td>97</td>
<td>251</td>
</tr>
<tr>
<td>5</td>
<td>382</td>
<td>-32</td>
<td>304</td>
<td>110</td>
<td>279</td>
</tr>
<tr>
<td>7</td>
<td>406</td>
<td>-32</td>
<td>325</td>
<td>113</td>
<td>293</td>
</tr>
<tr>
<td>13</td>
<td>433</td>
<td>-32</td>
<td>339</td>
<td>126</td>
<td>318</td>
</tr>
</tbody>
</table>
factor of about 2.5 required to convert $\Delta \sigma_\rho$ data to the form of equation (5.14) yields a value for $\delta$ of 1.65 which is in better agreement with theory than that for $\Delta \sigma'_\rho$.

The second effect, solute depletion, has two main consequences. Precipitation during MMTT depletes the matrix of strong solid solution hardening elements (Nb, C, N). This is likely to produce a decrease in both $\sigma_0$ and $\alpha$ of equation (4.5). The change in $\sigma_0$ can, in principle, be calculated from the results of Irivine et al. for solid solution strengthening in stainless steels, using the chemical composition (Table 3-1) and solubility data discussed earlier. From calculations using the above results $\sigma_0$ is expected to be reduced by 40-50 MPa. An equivalent calculation for $\alpha$ is not possible although a reduction by 20-30% is feasible, i.e. to the value for pure metals$^1$. The effect of solute depletion on both $\sigma_0$ and $\alpha$ is expected to produce a slight over-estimate of the strengthening contribution due to dislocations after MMTT.

The heterogeneous distribution of particles in MMTT material is apparent from Figs. 4-73 and 4-103 and inevitably brings into question the choice of particle parameters adopted in equations (5.13) and (5.14). Since dislocation motion occurs by glide the most meaningful parameter that can be used in connection with equation (5.13) is generally acknowledged to be the planar particle spacing ($D_\rho$) which is given by

$$D_\rho = \eta N_A^{-\frac{1}{3}}$$

where $N_A$ is the planar particle density and $\eta$ a geometrical factor. In the context of dispersion strengthening and the Orowan equation, the correct values of $\eta$ have been discussed in detail by Kocks$^{330}$ who showed that $\eta$ may have values ranging from 0.5 to 1.23 depending on the form of the particle distribution and the relevant spacing. In general it appears that $\eta<1.0$ for nearest neighbour spacings and $\eta>1.0$ for spacing obtained in direct computer simulations of dislocation motion through arrays of strong obstacles$^{331}$. It should be noted that the spacing parameter ($D$) of equation (5.13) should, according to Ashby$^{327}$, be that obtained by Foreman and Makin$^{331}$. However, it can be seen from equation (5.14) that the
volume spacing, given by equation (5.1) was used and this is reflected in the somewhat lower value of $\delta (\approx 2.0)$ obtained theoretically. If the planar spacing was used, $\delta \approx 3.0$ would have been obtained, since this parameter is somewhat larger than the volume spacing. The main reason for using the volume spacing and hence volume particle density ($N_v$) in equation (5.14) is the fact that planar particle densities ($N_A$) are difficult to measure by thin foil transmission electron microscopy. It is necessary to know the precise relationship between $N_v$ and $N_A$ which is very sensitive to spacial distribution and has not as yet been specified for complex structures of the type studied in this work. Uncertainties in the relationship used as well as in the dislocation core radius and mean particle size make the estimation of exact theoretical values of $\delta$ difficult. Nevertheless it is believed that the probable error in $\delta$ is of the order of 25% only.

Finally, the effect of the variation in particle size during MMTT is relatively small as compared with the two factors just discussed. This is so because of the logarithmic term in equation (5.13).

It is clear from the above discussion that, in addition to the simple description of strengthening based on equation (5.12), a more sophisticated mathematical treatment can be used (equation (5.17)) as proposed by Kocks for work hardening in dispersion strengthened materials. These two approaches, of which the latter is to be preferred on theoretical grounds, lead to widely differing estimates of the particle strengthening contribution ($\Delta \sigma_p$), as shown in Table 5-3 and consequently the particle strengthening parameter ($\delta$) of equation (5.14). The value of $\delta$ (1.65), determined using equations (5.14) and (5.17) is in reasonable agreement with the theoretical prediction based on the relationship proposed by Ashby. Consequently, the foregoing analysis appears to be consistent with the hypothesis that the strengthening produced by MMTT arises from the contribution to the flow stress of both the increased dislocation density and the Nb(C,N) particles. This interpretation of low temperature strengthening can be tested by comparing the recovery data of Fig. 4-53 with the values obtained from
equations (4.5), (5.12), (5.14) and (5.17) together with the relevant dislocation and particle density data (section 4.2.7.4). Results of this analysis are summarised in Table 5-4 in which $\Delta\sigma_{\text{Expt}}$ is the measured stress decrement accompanying $\Delta\sigma'$ and annealing and $\sigma'_{\text{f}}$, and $\sigma''/\Delta\sigma''$ are the values of proof stress and stress decrement predicted by equations (5.12) and (5.17) respectively. It is apparent from Table 5-4 that, while reasonable agreement is obtained between calculated and experimental values of $\Delta\sigma$, it is not possible to distinguish between the two approaches used in the calculation of $\Delta\sigma'$ and $\Delta\sigma''$. However, the degree of agreement obtained confirms that the general approach used in the present study is valid.

It can be concluded from the above discussion that the main contributions to strengthening as a result of MMTT arise from an Orowan stress due to the presence of dispersed Nb(C,N) particles and conventional dislocation strengthening. These two components of the flow stress appear to adequately account for the observed strengthening, and there is no evidence to support the view that a large proportion of the strengthening is due to the more uniform distribution of dislocations after MMTT, as postulated for bcc iron alloys. In other words MMTT in stabilised austenitic steels produces strengthening which, though inferior to conventional cold working because of the recovery occurring during the ageing part of the MMTT cycle, is nonetheless important.

5.3 High Temperature Strengthening

5.3.1 Hot-Tensile Deformation

The considerable strengthening at 800°C achieved by MMTT (Table 4-9), as compared with either solution treated or stabilised material, is believed to be due to the considerably higher dislocation density (Fig. 4-79) supported by Nb(C,N) particles (Section 5.1). This is associated with higher strain rate sensitivity of the proof stress of the MMTT material. The difference in strain rate sensitivity cannot be accounted for simply by the higher internal stress after MMTT. The basic principle of deformation theory requires that strain rate (c) and strain rate (d) due to dislocation motion are given by
<table>
<thead>
<tr>
<th>Time (h)</th>
<th>$\Delta\sigma'_N$ (MPa)</th>
<th>$N_e$ ($x10^9$ cm$^{-2}$)</th>
<th>$\Delta\sigma'$ (MPa)</th>
<th>$\sigma'_e$ (MPa)</th>
<th>$\sigma''_e$ (MPa)</th>
<th>$\Delta\sigma''_0$ (MPa)</th>
<th>$\Delta\sigma''$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>11.05</td>
<td>304</td>
<td>7.85</td>
<td>110</td>
<td>382</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>150</td>
<td>9.60</td>
<td>287</td>
<td>6.15</td>
<td>102</td>
<td>357</td>
<td>24</td>
<td>22</td>
</tr>
<tr>
<td>450</td>
<td>8.90</td>
<td>277</td>
<td>5.00</td>
<td>95</td>
<td>340</td>
<td>42</td>
<td>336</td>
</tr>
</tbody>
</table>

Table 5-4
Comparison of experimental recovery data ($\Delta\sigma'_e$) with those calculated theoretically ($\Delta\sigma'_0$ and $\Delta\sigma''_0$)
\[ \varepsilon = \phi \rho_m \bar{b} l_s \]  \hspace{1cm} \ldots \ (5.19) \\
\[ \dot{\varepsilon} = \phi \rho_m \bar{b} V \]  \hspace{1cm} \ldots \ (5.20)

where \( \phi \) is a geometrical factor reflecting the multiplicity of slip systems \((\sqrt{1/3})\), \( \rho_m \) the mobile dislocation density, \( l_s \) the average distance moved by dislocations and \( V \) their average velocity. From these relationships it follows that, for deformation to occur at the imposed strain rate in a hot tensile test, some dislocations must be mobile and move with a certain average velocity.

It is apparent from section 5.1 that the MMTT substructure possesses high resistance to thermal recovery at \( 800^\circ C \) particularly over the period of time involved in the hot tensile tests (about 6h at the lowest strain rate). The flow stress contributions due to particles and dislocations can be assumed to be athermal, varying only weakly as a function of temperature through the shear modulus. This is generally valid for short range interactions of dislocations with particles and for attractive junctions. However, it is apparent from Fig. 4-103 that the dislocation structure of MMTT material is heterogeneous, having a distribution of dislocation segment lengths. This type of dislocation distribution, illustrated schematically in Fig. 5-5, has been discussed qualitatively earlier (section 2.1.1.5) and in the context of deformation by McLean and others. Its existence has been directly confirmed recently in crept austenitic stainless steel and \( \text{MgO} \). In Fig. 5-5 the number of dislocation segments \((N(L))\) having lengths between \( L \) and \( (L + dL) \) is plotted against the length of dislocation segments. A relationship describing such distributions has been derived in section 5.1 for a random 3-dimensional network. In MMTT material the segment lengths will be determined by both node and particle spacings, while in pure metals and single phase alloys the distribution will be a function of the node spacing only. Techniques are currently being sought to obtain measurements of this distribution for the MMTT material, and a paper discussing the theoretical nature and implications of this statistical model is in preparation. In this report, however, only its main qualitative implications and predictions are considered.
It is apparent from Fig. 5-5 that the total dislocation density \( \rho_L \) will be given by the relationship

\[
\int_0^\infty L N(L) dL = \rho \quad \text{... (5.21)}
\]

which has been evaluated for a number of potential distributions in Appendix 6. In the absence of long range stresses, the dislocations move in response to the applied stress \( \sigma_A \) and the density of mobile dislocations will be related to the number of segments which exceed a critical length \( L_c \) given by

\[
L_c = \frac{aG\bar{s}}{\sigma_A} \quad \text{... (5.22)}
\]

The mobile dislocation density \( \rho_m \) can therefore be defined as

\[
\rho_m = \int_{L_c}^\infty s L N(L) dL \quad \text{... (5.23)}
\]

where \( s \), which will also have its own associated distribution function, is related to the number of dislocations generated by a source and their mean free paths. If long range stresses act on these dislocation segments, the applied stress \( \sigma_A \) should be replaced in equation (5.22) by the effective stress \( \sigma_E \) which is related to the applied stress by

\[
\sigma_A = \sigma_I + \sigma_E \quad \text{... (5.24)}
\]

where \( \sigma_I \) is the long range internal stress opposing dislocation motion. This will introduce additional statistical factors into equation (5.23), and therefore the simpler case of deformation given by equation (5.22) will be considered, particularly since climb controlled creep is likely to be relatively insensitive to internal stress.

The MMTT structure, and hence the dislocation segment length distribution, can be assumed to remain relatively unchanged during low strain rate deformation. It therefore follows that for deformation to occur at a given strain rate a sufficient number of dislocations must be mobile and that the stress required to induce plastic flow (proof stress) will be determined by the distribution function of equation (5.23) and \( L_c \). As the strain rate increases, more mobile dislocations must be produced (equation (5.20)) and consequently the critical length \( L_c \) decreases causing the proof stress to increase (equation (5.22)). This will also be accompanied by an increase in dislocation velocity \( v \) which
for the case of climb of edge dislocations will vary according to the relationship

\[ V = \frac{2\pi D_0 \sigma}{kB \ln (R/b)} \]  

where \( D \) is the self diffusion coefficient, \( \Omega \) the atomic volume and \( R \) the external cut-off parameter of the diffusion area.

During hot-tensile tests at low strain rates (<10^{-3} s^{-1}) the MMTT material showed comparatively little work-hardening followed by deformation at a constant flow stress. The small degree of work-hardening in the early stages of deformation is probably due to the inactivation and exhaustion of some dislocation sources, and minor changes in the dislocation distribution. Subsequent deformation at constant stress occurs due to the dynamic equilibrium between work-hardening and recovery. This is consistent with the observation that the dislocation density introduced by 2% MMTT pre-strain decreased very rapidly during subsequent ageing (4h at 800°C). At high strain rates the proof stress behaves in the same manner but, since considerably higher densities of mobile dislocations are necessary for deformation to occur, the work-hardening rate increases significantly. However, the recovery rate does not increase sufficiently for a steady state to be reached, and consequently a considerable increase in dislocation density arises (Table 4-18). This is accompanied by changes in dislocation distribution, work-hardening behaviour and strain rate sensitivity.

5.3.2 Creep

It is apparent from Figs. 4-60 and 4-101 that the creep behaviour of MMTT material is complex. As outlined in the Introduction, one of the main objectives of MMTT was to achieve the highest possible level of resistance to dislocation creep. At low stresses and high temperatures, however, cavitation appears to be an important source of creep after MMTT (Fig. 4-101). The existence of two regimes has been observed in the stress dependence of the minimum creep rate in a number of materials \(^{339,340}\). This behaviour is generally accepted to arise when two different creep processes occur independently but concurrently, such as dislocation and diffusion creep \(^{340}\). For this to be an adequate description of
creep in MMTT material, a stress exponent of \( n = 1.0 \) would be expected at low stresses, and depending on the dislocation creep mechanism \( n = 3.0 - 8.0 \) at high stresses. This does not appear to be the case with the 800°C results (Fig. 4-60), which are most complete, where at low stresses \( n = 2.8 \). However, the stresses employed may not have been low enough for the \( n = 1.0 \) regime to be reached, the 21 MPa stress possibly being in the transition region.

If the minimum creep rate at 850°C and 21 MPa obtained by subtracting the cavity growth contribution as shown in Fig. 4-101(b) \( (10^{-6} \, \text{h}^{-1}) \) is compared with that obtained by extrapolating from high stresses \( (5 \times 10^{-7} \, \text{h}^{-1}) \) using \( n = 6 \) (Fig. 4-60) reasonable agreement is observed. The grain-boundary cavity growth rate should, if diffusion controlled, like diffusion creep, exhibit a linear stress dependence of the form

\[
\frac{\Delta V}{V} = C \delta z \frac{D_g \sigma \Omega}{kT} \quad \ldots \ (5.26)
\]

where \( \Delta V/V \) is the fractional cavity volume change, \( \delta z \) the grain boundary width, \( D_g \) the grain boundary diffusion coefficient, \( \Omega \) the atomic volume, \( \sigma \) the applied stress and \( C \) a geometrical constant \((\equiv 1.0)\). Substituting the following values, applicable to the 21 MPa, 850°C test, into equation (5.26)

\[
\delta z D_g = 4.0 \times 10^{-21} \, \text{m}^{-1} \, \text{s}^{-1} \quad 145
\]
\[
\Omega = 2.0 \times 10^{-29} \, \text{m}^3
\]
\[
k = 1.4 \times 10^{-23} \, \text{J} \, \text{K}^{-1}
\]
\[
N_c = 10^{13} \, \text{m}^{-3} \equiv 500 \, \text{mm}^{-2}
\]

we obtain a cavity creep rate contribution \( (N_c \Delta V/V) \) of about \( 4.0 \times 10^{-6} \, \text{h}^{-1} \), where \( N_c \) is the cavity density. This prediction compares reasonably well with the observed rate of about \( 10^{-5} \, \text{h}^{-1} \) (Fig. 4-101(a)). It, therefore, seems reasonable to conclude that the change in stress dependence of creep in MMTT material is due to the dominance of cavity creep at low stresses. This conclusion is not in conflict with the observation that an exponential law also adequately describes the stress dependence of minimum creep rate over most of the range of stress studied. In this case a deviation was also observed in the stress dependence below 50 MPa.
The much greater contribution of cavitation to creep at low stresses obscures the true level of dislocation creep. The latter is apparently 3 or 4 times lower than that indicated by creep tests at 800°C and 50 MPa, and as much as one or two orders of magnitude lower at 21 MPa. Due to the weaker temperature dependence of cavity growth as compared with dislocation creep (i.e. grain boundary as compared with lattice diffusion) the former is likely to be even more significant at lower temperatures.

Over most of the stress range the minimum creep rate could equally well be described by the relation

$$\dot{\varepsilon} = A' \exp \left( \beta \sigma \right) \exp \left( -Q/RT \right) \quad \text{(5.27)}$$

where $A'$ and $\beta$ are constants. When only one process is rate controlling the constant $\beta$ is related to the activation volume $V^*$, which is given by

$$V^* = kT \beta' = 2.3 kT \left( \frac{d(\log \dot{\varepsilon})}{d\sigma} \right) \quad \text{(5.28)}$$

For the present material the activation volume was found to be about $65 \bar{b}^3$ where $\bar{b}$ is the Burgers vector. Interpreting the high stress (>50 MPa) creep data in terms of this measured activation volume leads to the conclusion that the most sensible rate-controlling creep mechanism is the dragging of jogs by screw dislocations\(^{132}\). An examination of the character of dislocations in crept MMTT material indicated that the vast majority of dislocations were close to screw orientation. However, it was apparent from the same micrographs that the majority of dislocations were imobile and that very few super jogs were observed. Indeed both the recovery data (Table 4-19) and Ishida's analysis indicate that the strength of dislocation-particle interactions was considerably greater than the dislocation-dislocation interactions, which are a necessary consequence of the jogged-screw model\(^{132}\). Furthermore, the stress-exponent (6.0) in the high stress regime is more compatible with that commonly observed in annealed dispersion hardened materials (6.0 - 8.0\(^{346}\).

Accepted models of creep predict stress exponents in the range 3.0 - 5.0 and generally appear to be adequate only for pure metals and single phase alloys\(^{346}\).
Consequently, many authors have invoked the concept of internal stress to explain the high stress exponents observed in dispersion hardened alloys. Variable stress creep or strain transient dip tests have been proposed to measure internal stress. Such tests were carried out during this work (see Appendix 4), which indicated that the internal stress was typically 90-95% of the applied stress throughout the range of stresses studied. Thus at an applied stress of 50 MPa the internal stress was found to be 47 MPa, while at 96 MPa it was 91 MPa. Electron microscopy indicated that in this regime of stress no significant change in dislocation density occurred after creep of MMTT material (Table 4-18). Consequently, it was concluded that modifications to account for the high stress exponent in terms of internal and effective stresses, equation (5.24), are not applicable to the present material after MMTT. Subsequently, it has been proposed that a more plausible explanation for the behaviour of materials during variable stress creep tests should include anelasticity and dislocation dynamics and that internal stress measurements by the strain transient dip test are of doubtful significance (see Appendix 4).

The simple dislocation model proposed in this report, equations (5.19) - (5-23) and Fig. 5-5, has been shown elsewhere to explain both the variable stress behaviour and high stress exponents observed for dispersion hardened alloys. The main effects in creep are similar to those described for hot-tensile tests, although a slightly different emphasis is necessary due to the nature of the measured experimental variables. In hot-tensile tests a sufficiently high density of mobile dislocations must be generated to maintain the applied strain rate. This factor controls the stress levels and work hardening rates observed. On the other hand, in creep under constant stress the mobile dislocation density and hence creep rate, adjust according to equations (5.20) and (5.23). Consequently, the decreasing creep rate during primary creep may be interpreted as being due to a reduction in mobile dislocation density in the same manner as during the initial stress transients in hot-tensile tests. Secondary creep, in the absence of cavitation, therefore corresponds to a dynamic balance between work-hardening
and recovery, as outlined for constant strain rate behaviour. Furthermore, an exponential stress dependence of the minimum creep rate follows as a natural consequence of this type of model, since the dislocation distribution function (N(L)) has been shown to be an exponential relationship of the form of equation (A6.2). The rate controlling process in the dislocation creep regime is apparently self-diffusion and may involve local climb over particles. It is also interesting to speculate that, if the above model is correct, temperature dependent terms may also enter the creep relationship through the mobile dislocation density due to recovery effects, thermally activated breakaway from pinning points or breaking of dislocation junctions. The latter are generally considered athermal processes. High activation energies (about twice self diffusion) and high stress exponents (~20) have been observed in cold-worked T.D. Nickel and dispersion strengthened copper single crystals. These too follow as a natural consequence of the above formulation, as pointed out by McElroy and co-workers.

Variable stress behaviour outlined in detail in Appendix 4 is similarly consistent with the network distribution model presented here. The dislocation distribution is considered to be composed of two fractions in the presence of an applied stress. These are the immobile fraction of dislocation segments shorter than the critical length, L_c, and the mobile fraction with L>L_c. From the estimates made in Appendix 6 the mobile fraction is likely to be less than 10% of the total population even at the highest creep stresses (160 MPa). Any anelastic relaxation arising from dislocation bowing will consequently be of considerable importance during unloading experiments, as demonstrated in Appendix 4. Furthermore, it is unlikely that all the mobile dislocations will become immobilised during the small load reductions employed in recovery rate and internal stress measurements. Indeed, the behaviour predicted from Fig. 5-5 is similar to that observed when the extrapolation procedure of Fig. A4-13 to A4-15 was used. These revealed continued forward creep at a rate which, immediately following a load
reduction, lay between the steady creep rates at the upper and lower stresses employed in the test. This was followed by a strain transient, the creep rate decreasing steadily to a constant level characteristic of the reduced stress. In order to estimate the instantaneous creep rate upon making such a load change it is assumed that the MMTT network distribution remained unaffected by stress change at least over the period of time involved in such an experiment (\( \tau \)). It is further assumed that the mean dislocation velocity was a simple function of applied stress (e.g. equation (5.25)) and that the dislocation velocity changed immediately the stress change was made. At this instant the number of mobile dislocation segments should be that characteristic of the higher stress. Consequently, the instantaneous change in forward creep rate is expected to arise solely from the reduction in dislocation velocity. Subsequently, the creep rate decreases to a level characteristic of the lower stress. This occurs because dislocations, which were initially mobile, become trapped and cannot be reactivated. Consequently, the strain transient obeys an exhaustion law (i.e. approximately logarithmic time dependence) like primary creep, though, as is apparent, the precise mechanism is slightly different. If the dislocation velocity obeys a linear stress dependence, as in the case of pure climb, then the instantaneous forward-creep rate, \( \dot{\varepsilon}_1 \), on reducing the stress from \( \sigma_1 \) to \( \sigma_2 \) should be given by

\[
\dot{\varepsilon}_1 = \frac{\sigma_2}{\sigma_1} \dot{\varepsilon}_1 \quad \ldots \quad (5.29)
\]

where \( \dot{\varepsilon}_1 \) is the creep rate at the higher stress (\( \sigma_1 \)). The estimated ratio \( \dot{\varepsilon}_1 /\dot{\varepsilon}_1 \) obtained from the data of Fig. A4-13 to A4-15 are presented in Table 5-5 and compared with the respective values of \( \sigma_1 \) and \( \sigma_2 \) to test the prediction of equation (5.29). It is apparent from the results of this analysis that \( \dot{\varepsilon}_1 /\dot{\varepsilon}_1 \) is certainly of a similar magnitude to the ratio of \( \sigma_2 /\sigma_1 \). Bearing in mind the problems associated with accurately measuring these strain transients, especially at or close to the instant of unloading, this appears to be in good agreement with the predictions of the network model outlined above.
Table 5-5
Comparison of forward creep component after stress reduction measured by the method of Fig. A4-13 and theoretical values from equation (5.29)

<table>
<thead>
<tr>
<th>Condition</th>
<th>T (°C)</th>
<th>$\sigma_1$ (MPa)</th>
<th>$\sigma_2$ (MPa)</th>
<th>$\frac{\sigma_2}{\sigma_1}$</th>
<th>$\frac{\dot{\varepsilon}_2}{\dot{\varepsilon}_1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 MMTT (10%)</td>
<td>750</td>
<td>100</td>
<td>50</td>
<td>0.50</td>
<td>0.89</td>
</tr>
<tr>
<td></td>
<td></td>
<td>130</td>
<td>80</td>
<td>0.61</td>
<td>0.77</td>
</tr>
<tr>
<td>5 MMTT (2%)</td>
<td>750</td>
<td>100</td>
<td>80</td>
<td>0.80</td>
<td>1.50</td>
</tr>
<tr>
<td>Stabilised</td>
<td>750</td>
<td>75</td>
<td>50</td>
<td>0.66</td>
<td>0.28</td>
</tr>
</tbody>
</table>
The implications of the analyses of variable stress creep tests in terms of an anelastic contribution to the strain transients with respect to recovery creep and internal stress measurements have been pursued in detail in Appendix 4. It is clear that the latter techniques are of dubious value in studying material behaviour. However, certain of the tests described in Appendix 4 have provided useful information on the degree of recovery resistance and mechanical stability conferred by MMTT as compared with alternative treatments. Measurements of $\Delta \varepsilon_p$ as a function of prior recovery period under reduced stress for MMTT and stabilising treatments indicated (Fig. A4-21) that $\Delta \varepsilon_p$ rose with increasing recovery time to a level equivalent to the anelastic strain expected from unloading experiments after 8h and 2h respectively. A slight hysteresis effect was apparent in agreement with other observations concerning the departure of anelastic strains from the ideal behaviour indicated by the Boltzmann Superposition Principle. However, beyond 8h and 2h for MMTT and stabilising treatments respectively, the behaviour was significantly different. In the case of stabilised material $\Delta \varepsilon_p$ continued to increase indicating a strong recovery effect. MMTT material, on the other hand, exhibited completely reversible behaviour (i.e. $\Delta \varepsilon_p = \Delta \varepsilon_A$ beyond 8h). This indicates that MMTT endowed the material with superior recovery resistance and is consequently in good agreement with static recovery, low stress creep and variable strain rate hot-tensile test results.

Since the most important parameter in recovery appears to be the particle distribution, dislocation-particle interactions are also expected to be the most important in creep. The minimum creep rate at 800°C and 90 MPa 'saturates' at about 5 MMTT cycles, (Fig. 4-64) as does the particle density (Fig. 4-79). Continued MMTT up to 13 cycles, during which the dislocation density continues to increase (Fig. 4-79), produces little or no improvement in creep resistance. Consequently, the major factor in high temperature strengthening is the particle distribution, without which the recovery resistance of MMTT material would decrease. Furthermore, the dislocation segment length distribution is controlled by the particles and the parameters of the distribution will be:
(a) \( \bar{L} = A_{Tn} N_{v}^{-1/3} \) where from Table A6-1 \( A_{Tn} = 0.55 - 1.0 \) depending on the tightness of the network.

(b) \( L_{p} \) = average particle spacing within precipitate bands

(c) \( L_{\text{max}} \) = interband spacing

where \( L_{\text{max}} \) corresponds to the longest segments and \( L_{p} \) to the length corresponding to the peak in Fig. 5-5. It can, therefore, be concluded that, while the ultimate theoretical creep strength* has not been achieved, the maximum possible level of creep resistance has been obtained in the present material due to the heterogeneous nature of Nb(C,N) precipitation and the deleterious effect of prestrain-induced cavities.

5.3.3 General Comments

Hot-tensile and creep behaviour of MMTT material has so far been discussed as if these were two entirely different aspects of high temperature testing. This is probably not the case, since they comprise two closely related facets of mechanical behaviour as was appreciated by Zener and Holloman and others. If the present 800°C creep and hot-tensile test data is plotted in the manner shown in Fig. 5-6, both sets of data coincide over the stress range 130-160 MPa and may be described by a relationship, extending over the stress range 50-250 MPa, of the form

\[
\dot{\varepsilon} = A'' \exp (BQ) \quad \ldots \ (5.30)
\]

where \( A'' \) and \( B \) are temperature dependent parameters having values \( 3.0 \times 10^{-11} \text{s}^{-1} \) and \( 3.5 \times 10^{-3} \text{m}^{3} \text{J}^{-1} \), respectively. This implies that basically the same mechanism operates over the major portion of this range and that the exponential dependence is consistent with the model outlined earlier (Fig. 5-5). This type of behaviour has been taken by previous authors as evidence for the existence of a steady state deformation regime.

The so-called steady state deformation, however, may be interpreted as being due to an entirely different process. The true stress at maximum load (UTS) in

*This should occur for a perfectly uniform (narrow) particle distribution only
tensile tests is generally associated with plastic instability or necking. At high temperatures and relatively lower stresses than those observed at ambient temperatures, the 'steady state' condition may occur at a considerably lower rate and, to a large extent, by internal necking (i.e. the growth of cavities and cracks). This appears to be the case with MMTT material (Fig. 4-101). It, therefore, follows from equation (5.30) and Fig. 5-6 that secondary creep is another manifestation of this phenomenon, namely an inflection in the creep curve preceding tertiary creep, which is due to plastic instability. If this is the case, then, applying Considère's principle the work hardening rate \( h = \partial \sigma / \partial \varepsilon \) is equal to the true stress. This corresponds to the ultimate tensile stress in constant strain rate tests, and to the applied stress during secondary creep.

The recovery creep model postulates that the creep rate is related to the dynamic recovery \( r \) and work hardening rates by

\[
\dot{\varepsilon} = \frac{r}{h} \quad \text{... (5.31)}
\]

Variable stress techniques have been proposed for measuring these parameters from strain transients but these have recently been severely criticised and are discussed at length in Appendix 4. However, if the above hypothesis that secondary creep and maximum load (UTS) behaviour are both manifestations of plastic instability is correct, the work hardening rate may be obtained from the applied stress \( (\sigma_A) \) or UTS \( (\sigma_{UTS}) \) by the relation

\[
h = \sigma_A = \sigma_{UTS} \quad \text{... (5.32)}
\]

An estimate may, therefore, be obtained for the recovery rate in MMTT material during creep and hot tensile tests from equation (5.31). Estimated recovery rates obtained by this procedure are tabulated together with other relevant parameters in Table 5-6, from which it is apparent that \( r \) increases very rapidly with strain rate. These values may be compared with those obtained from recovery tests (Section 4.2.3 and Fig. 4-53) on 5-MMTT material and others inferred from the observation (Fig. 4-45) that the excess strengthening produced by prestraining (2%) decreases rapidly during the annealing period \( (\dot{r}h) \) of MMTT. The recovery rates determined from Fig. 4-53 for the 5-MMTT material vary from \( 2.4 \times 10^{-1} \) to
Table 5-6
Variation of work-hardening (h) and recovery (r) rates for 5-MMTT material with strain rate

<table>
<thead>
<tr>
<th>$\dot{\varepsilon}$ (h$^{-1}$)</th>
<th>$\sigma_a$ (MPa)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>h (MPa)</th>
<th>r (MPa h$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$2.8 \times 10^{-5}$</td>
<td>50</td>
<td>-</td>
<td>50</td>
<td>$1.4 \times 10^{-3}$</td>
</tr>
<tr>
<td>$6.4 \times 10^{-5}$</td>
<td>66</td>
<td>-</td>
<td>66</td>
<td>$4.2 \times 10^{-3}$</td>
</tr>
<tr>
<td>$1.5 \times 10^{-4}$</td>
<td>80</td>
<td>-</td>
<td>80</td>
<td>$1.2 \times 10^{-2}$</td>
</tr>
<tr>
<td>$9.3 \times 10^{-4}$</td>
<td>110</td>
<td>-</td>
<td>110</td>
<td>$1.0 \times 10^{-1}$</td>
</tr>
<tr>
<td>$3.6 \times 10^{-3}$</td>
<td>-</td>
<td>134</td>
<td>134</td>
<td>$4.8 \times 10^{-1}$</td>
</tr>
<tr>
<td>$9.6 \times 10^{-3}$</td>
<td>160</td>
<td>-</td>
<td>160</td>
<td>$1.5 \times 10^{0}$</td>
</tr>
<tr>
<td>$3.6 \times 10^{-2}$</td>
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<td>153</td>
<td>153</td>
<td>$5.5 \times 10^{0}$</td>
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<tr>
<td>$3.6 \times 10^{-1}$</td>
<td>-</td>
<td>178</td>
<td>178</td>
<td>$6.4 \times 10^{1}$</td>
</tr>
<tr>
<td>$3.6 \times 10^{0}$</td>
<td>-</td>
<td>214</td>
<td>214</td>
<td>$7.7 \times 10^{2}$</td>
</tr>
<tr>
<td>$3.6 \times 10^{1}$</td>
<td>-</td>
<td>260</td>
<td>260</td>
<td>$5.4 \times 10^{3}$</td>
</tr>
</tbody>
</table>
2.5 x 10^{-2} \text{ MPa h}^{-1} \text{ over the first 1000h of annealing at 800°C, while prestrained MMTT material exhibits a recovery rate in excess of } 10^2 \text{ MPa h}^{-1} \text{ (Fig. 4-45).}

It has been shown (Tables 4-18 and 5-1) that at stresses below 160 MPa and strain rates less than \(10^{-4} \text{ s}^{-1}\) (3.6 x 10^{-1} h^{-1}) at 800°C no significant changes in dislocation density were observed. Dislocations generated during these tests must, therefore, have been annealed out. It is apparent from Table 5-6 that the recovery rates, estimated in the manner outlined above, for all strain rates below \(10^{-4} \text{ s}^{-1}\) are less than those for excess dislocations introduced by prestraining MMTT material \((10^2 \text{ MPa h}^{-1})\). Consequently, a significant increase in dislocation density should only be observed in such tests, if recovery is not sufficiently rapid as compared with the rates of work hardening or, in other words, the accumulation of dislocations. This appears to be the case at strain rates higher than \(10^{-3} \text{ s}^{-1}\) (Table 4-18).

It should be appreciated that even if the above argument is valid concerning the Considère effect, the conclusions drawn regarding recovery rates are at best only a crude approximation. The hardening rate measured by the above technique will be a composite function which includes the effect of recovery and in that sense it is not equivalent to \(h\) in equation (5.31). However, the above approximate method of measuring hardening rates is a superior estimate to the recovery creep measurements outlined in Appendix 4 due to the significant contribution of anelastic and dynamical dislocation phenomena in the latter tests.

The behaviour described here implies that classical steady state conditions do not occur during creep and hot-tensile deformation of MMTT material. No 'steady state' substructure exists in MMTT material as is apparent from low stress tests during which recovery controlled by particle coarsening occurs continuously (Fig. 4-86 and 4-87). Furthermore, since in MMTT material creep is mainly due to cavitation (Fig. 4-101), secondary creep is a manifestation of the phenomenon of tensile instability which occurs during deformation under constant strain rate. However, at intermediate stresses, corresponding to strain rates below \(10^{-3} \text{ s}^{-1}\), MMTT material appears to deform in a manner consistent with the recovery creep model and exhibits many characteristic features of an
ideal material, the behaviour of which can be described by a mechanical equation of state\textsuperscript{299}.

5.4 Ductility

5.4.1 Low Temperatures

The links between prestrain induced cavities or voids at second phase particles, by a mechanism similar in principle to that proposed by Ashby\textsuperscript{314} and reduced ductility have been established for low temperature deformation. Such cavities have been observed for form at both inclusions and coarse undissolved particles (Fig. 4-90 and 4-92) and their size varied linearly with strain (Fig. 4-93) as predicted by Ashby's model. Both the sudden decrease in ductility at three MMTT cycles (Fig. 4-45) and the apparent absence of cavities from the microstructure at strains below 0.06 implies that they nucleate only after a critical strain has been attained. Such behaviour has been observed in other materials\textsuperscript{362-363} but it has not yet been determined whether the accumulated strain or tensile stress is the critical parameter\textsuperscript{364}. However, once formed the cavities assume a size determined solely by the total accumulated plastic strain.

Nucleation of cavities occurs at particle–matrix interfaces due to the stress concentration resulting from the non-homogeneous deformation around hard non-deformable particles by a process of interface decohesion, as outlined by Ashby\textsuperscript{314} and others\textsuperscript{264-267}. Thus cavitation occurs as one of the basic mechanisms of stress relaxation in materials containing a dispersed second phase\textsuperscript{268}. Relaxation by secondary slip\textsuperscript{314,369,370} is an alternative mechanism especially at low strains, as is apparent from Fig. 4-18. Cavitation has always been observed to be associated with such a mechanism in α-brass crystals containing SiO\textsubscript{2} particles\textsuperscript{369}. Ashby's model\textsuperscript{314} postulates the formation of prismatic dislocation loops or geometrically necessary dislocations, while being a convenient continuum approximation, does not appear to be an entirely satisfactory physical explanation\textsuperscript{367}. However, it has been shown\textsuperscript{368} that the latter approach gives a satisfactory mathematical description of work hardening in dispersion hardened alloys, as do the microscopic models\textsuperscript{368,370} and that it gives a reasonable macroscopic
explanation. In the present material prismatic loops were never observed in deformed samples, probably because of the complex slip geometry and activity on secondary slip systems which produced the necessary stress concentration and mass transfer for the nucleation and growth of cavities. The particles at which cavitation occurred were generally equiaxed and about 1.0μm for undissolved carbides, and 4-10μm for oxide inclusions. Nucleation occurred at approximately the same level of strain regardless of particle size in conflict with the predictions of Ashby and the observations of Palmer and Smith. The general agreement between the observed behaviour and all aspects of the Ashby model except the latter one suggests that the tensile stress concentration necessary for interface decohesion arises not directly from the applied tensile stress but from the applied shear strain acting in the neighbourhood of second phase particles.

Decreasing the volume fraction of inclusions by about 85% (Alloy BV943) produced only a marginal improvement in uniform ductility (Fig. 4-45). The comparatively large change in the volume fraction of inclusions was not accompanied by a similar change in the number of particles. The particle densities for Alloys RM9209 and BV943 were $1.7 \times 10^5$ and $0.95 \times 10^5$ cm$^{-2}$, respectively. The inclusions in the latter alloy (BV943) were, therefore, more finely distributed. Consequently, the number of cavitated particles during MMTT and subsequent deformation was not significantly different particularly when the density of undissolved Nb(C,N) particles was taken into account. Previous work has shown that the reduction of area or true strain at fracture is very sensitive to volume fraction of particles or voids. The effect on uniform ductility does not appear to have been studied in detail, but it might be expected to be considerably less. If cavity growth during low temperature straining affects the macroscopic work hardening rate, as proposed by Edelson and Baldwin, a slight increase in uniform ductility, and consequently UTS, might be expected for Alloy BV943 as was the case. The particle density is apparently an important factor, since with the comparatively low area densities of particles the spacings between them are
large and the initiation of fracture or necking by the cavity coalescence process is practically unaffected by volume fraction or particle size. In the present material, however, fracture propagated by the movement of a ductile crack through the material ahead of which cavities grew at an accelerated rate by localised necking of the matrix between particles followed by shear on planes of maximum shear stress (45° to tensile axis), (Fig. 4-96). It was evident from HVEM studies of samples cut from the necked region of tensile specimens that the localised shear deformation, introduced during crack propagation, was excessive, as is apparent from the electron diffraction pattern shown as an inset to Fig. 4-96. This indicates that large amounts of energy must be expended in both creating and propagating a crack in ductile materials, and is consistent with macroscopic fracture toughness measurements.

5.4.2 High Temperatures

The links between prestrain induced cavities and reduced creep and hot-tensile ductilities are more tenuous since less systematic work has been performed in this case. However, certain facts may be inferred from mechanical behaviour and other observations. It was found, for example, that prestraining by varying amounts followed by a stabilising treatment (50h 800°C) produced an abrupt reduction in creep ductility corresponding to a prestrain of between 0.04 and 0.06 (Fig. 4-9). At higher levels of prestrain only marginal reductions in creep ductility, for a given creep strength (i.e. minimum creep rate), were observed. Thus, the creep ductility was practically the same for 5- and 13-MMNT materials, and a material prestrained by 0.10 at 25°C followed by the stabilising treatment. The latter observation implies that prestrain-induced cavities do not sinter, at least in 50h at 800°C, and that any other forms of structural damage which might provide nucleation sites for creep cavities also do not anneal out. Furthermore, it supports the contention that the level of prestrain rather than applied tensile stress is the important criterion in the context of void initiation.

Similar prestrain effects on creep ductility have been reported for a variety of materials or may be inferred from mechanical property data.
However, no systematic studies have been performed to date, but a broad programme of work is currently under way at NPL which is throwing some light on the subject. The latter work on prestrain effects has evolved out of some early observations made during the present MMTT studies and the realisation that they might have wider implications. A major proportion of work to date has been performed on Nimonic 80A in which large numbers of small (0.25μm) grain boundary cavities were observed after prestraining and annealing, but none were found prior to annealing. The creep ductility at 750°C decreased continuously with increasing prestrain (>1.0%) and the density of cavities increased. These were considerably larger (>1.0μm) than after annealing and were observed only on transverse boundaries, while annealing apparently produced a completely random distribution and a considerably higher density. Nevertheless the cavity density increased by an order of magnitude after prestraining by 10% and subsequent creep at 750°C as compared with unprestrained material. Contrary to this behaviour for Nimonic 80A the present material did not appear to exhibit a particularly strain dependent ductility change after the initial drop at about 3MMTT cycles (6.0% prestrain) (Fig. 4-9) nor was there any qualitative difference in cavity density or distribution with increasing prestrain beyond 6.0%. This contrasting behaviour may be a consequence of the very high density of grain boundary carbides and their wider size distribution in Nimonic 80A as compared with the rather low, almost unimodal distribution in stainless steel. The extremely restricted or planar nature of slip in Nimonic 80A may also be a contributory factor. In fact, there is evidence that the slip band spacing decreased with increasing prestrain and that each cavity at the grain boundary was associated with a slip band. The influence of particles as the nucleation sites is not clearly established due to their extremely high density in Nimonic 80A. Similar prestrain effects have recently been reported in aluminium alloys, notably RR58, by Wilson.

The distribution of creep cavity sizes after MMTT or prestraining appeared to be considerably more uniform than after conventional treatments, and the cavities were generally spherical (Fig. 4-98). This was taken to be a consequence of the
introduction of cavity nuclei during prestraining, which appears to be true for low stress creep (<110 MPa) at 800°C and 50 MPa at 850°C. However, at low stresses (21 MPa) at 850°C during tertiary creep (Fig. 4-100) and in high stress creep and hot-tensile tests at 800°C transverse grain boundaries consisted of wide fissures (wedge cracks) which generally had a serrated appearance indicating a cavity linking process (Fig. 4-98). The estimated cavity density in these cases was higher than in secondary creep or low stress creep tests at 800°C. In high stress creep and hot-tensile tests this effect is presumably due to tensile tearing as a result of stress concentrations at growing cracks. At low stresses and higher temperatures secondary cavitation may occur at new particles which form as a result of particle coarsening at or in the vicinity of grain boundaries.

The nature of the cavity nuclei in MMTT and prestrained material has not been identified unambiguously. It is, however, believed that they may be undissolved grain boundary carbides. This contention can be examined by assuming that every coarse undissolved carbide particle at grain boundaries is associated with a cavity nucleus after prestraining. Creep at 50 MPa and 800°C produced, at failure after 80h, cavities with average size of about 4μm and density of 470 mm⁻² in regions well away from the point at which fracture occurred. The volume density of coarse carbides was found to be 4.5 x 10⁹ cm⁻³ and their average size was 0.9μm. Furthermore, it was found that only about 25% of the carbide particles lie on grain boundaries. For a unimodal particle size distribution the area density (Nₐ), volume density (Nᵥ) and particle diameter (d) are related by

\[ N_a = N_v d \quad \text{(5.33)} \]

and the area density will be about 4.0 x 10⁵ cm⁻² or 4000 mm⁻². The grain boundary density is consequently about 1000 mm⁻². Since only transverse grain boundaries are observed to cavitate during creep, this line of reasoning suggests that cavity densities of 250-500 mm⁻² should be expected, which is in good agreement with observation. From the average cavity size and density the volume fraction of cavities may be determined, and was found to be 3.8 x 10⁻³. Following a similar argument to that used in Fig. 4-101, the cavity contribution to creep at 50 MPa and
$800{}^\circ C$ in the 5-MMTT material was about 75% of the total creep strain ($5.0 \times 10^{-3}$). This supports the view expressed earlier (5.3.2) that the role of cavity creep becomes more important at higher stresses as the temperature is reduced.

Consequently, it is concluded that the cavity nuclei introduced by MMTT were small voids occurring at undissolved grain boundary carbides (and possibly some inclusions) which formed by interface decohesion during prestraining as outlined in the previous section (5.4.1). It must be made clear that this conclusion is reached on a limited amount of circumstantial evidence and that other potential mechanisms such as the presence of residual stresses or grain boundary ledges cannot be entirely ruled out.

The ductility minimum observed when ductility is plotted against stress or, when comparing different treatments, minimum creep rate (Fig. 4-66) is not confined to prestrained material. It has generally been associated with the changing roles of deformation modes, such as grain boundary sliding and dislocation creep, although their precise interaction is unspecified.

In unprestrained Nimonic 80A a minimum in ductility was found to be associated with a maximum in the rate of cavity generation. The nature of the process in the present material has not been distinguished for either MMTT or prestraining. It may, however, arise from the dual stress dependence (deformation) behaviour discussed earlier (Fig. 4-60). At low stresses cavity creep dominates (Fig. 4-101) and large strains may be achieved before the low applied stress is sufficient to cause fracture by cavity linking which occurs almost entirely by diffusional growth. As the stress is increased, the necking of the regions between cavities can occur more easily by a process of shearing and consequently, lower strains are achieved before fracture. However, as the stress is increased further, the role of dislocation creep dominates and makes an increasing contribution to creep strain. Under these conditions cavity growth and possibly nucleation of new cavities occur by plastic deformation rather than diffusion. Furthermore, the creep and hot tensile ductilities appear to vary in an identical manner as a function of deformation rate (minimum creep or hot tensile strain rates) as shown...
in Fig. 5-7. This behaviour lends itself to this type of interpretation as well as supporting the description, in terms of Considéres principle, of the stress dependence of the minimum creep rate and strain rate dependence of UTS (Fig. 5-6) given in section 5.3.3.

5.4.3 Poor Ductility - Remedies

It is evident from the foregoing discussion that the poor ductility after MMTT or simple tensile prestraining, both at low and high temperatures, is associated, to a greater or lesser extent, with second phase particles. Consequently, the control of particle content is a potentially fruitful area for further investigation.

5.4.3.1 Particle Content

With regard to low temperature ductility it was observed, in the present work, that a reduction in volume fraction of inclusions yielded only marginal improvements in uniform ductility, probably due to the fact that the inclusion density remained comparatively unchanged. Some improvement is to be expected, if the latter could also be decreased. This approach is, however, only of limited application, since, given that all inclusions could be removed, the undissolved carbides would still remain. The latter might be the most important factor affecting creep ductility and presents an interesting dilemma. If the proportion of undissolved carbides is reduced either by lowering the niobium, carbon and nitrogen contents of the alloy or by employing higher solution treatment temperatures, considerable grain growth would occur (Fig. 4-3). For the sheet specimens used in the present investigation an increase in the solution treatment temperature from 1200° to 1300°C resulted in the grain-size being increased from 40 to 150μm, accompanied by an increase in the proportion of Nb(C,N) available for hardening by about 30%. More work on bulkier specimens is necessary to establish whether this achieves better creep ductilities without the loss of creep strength.

An alternative approach is to select a carbide/nitride former which precipitates as particles having stronger interfacial adhesion, or to reduce the content of surface active elements, such as sulphur, phosphorus etc. In such
omplex alloy systems as the one studied in the present work, identification and, ultimately, removal of such elements would be complicated. High work of adhesion or strength of particle-matrix interface has been shown theoretically and experimentally to increase the strain at which void initiation occurs. Ideally, particles should possess sufficient interfacial strength for the total MMTT prestrain to be applied without interface decohesion occurring. This may prove a potentially useful method of suppressing cavitation if the inclusion content could also be significantly reduced. However, a high work of adhesion or carbide-matrix interfacial energy will give rise to a higher driving force for particle coarsening (equation (5.7)) and, consequently, poorer structural stability at high temperatures. It is also apparent that, if it is not possible to suppress cavity initiation to a sufficiently high strain, once formed, the cavities grow to a size solely determined by the prestrain. Under these circumstances no benefits would be gained from a higher work of adhesion.

5.4.3.2 Deformation Mode

It was thought initially that a reduction of the tensile to shear stress ratio during MMTT would decrease the tendency for cavity initiation at low strains. In Nimonic 80A torsional creep deformation has been observed to produce fewer cavities for a given strain than tensile creep. However, the indications are that room temperature torsional prestrain produces qualitatively the same cavity densities and similar ductility effects as tension. This seems to confirm the view expressed earlier (5.4.1) that shear strain is the important factor controlling interface decohesion. It also implies that, resorting to deformation modes in which a strong compressive stress is dominant, e.g. rolling, cavity nucleation will be relatively unaffected while growth may be slower. Rolling would, however, introduce problems regarding inhomogeneous deformation in thin sheet material at low strains.

Whatever the outcome of decisions regarding the potential usefulness of MMTT, this general field encompassing the effect of second phase particles and various deformation modes on ductile fracture should receive further study.
5.4.3.3 Deformation Temperature

It has been suggested\(^{386}\) that, since the processes producing reduced creep ductility after prestraining result from stress concentration either at second phase particles or grain boundaries, deformation at elevated temperatures, where slip occurs more homogeneously (fine slip), would be advantageous. MMTT at 800°C was attempted in this work but, due to reduced ductility at this temperature in hot tensile tests and the high level of dynamic recovery, this produced no tangible benefits. However, deformation at lower temperatures (400°C - 600°C) might provide an adequate compromise between the above factors.

5.4.3.4 Sintering

Sintering of cavities would appear to be an alternative method of improving creep ductility. It has been shown, for example, that this is a useful method of regenerating lost creep ductility in Nimonic alloys which have been crept to a high fraction of their rupture life\(^{390}\). However, it involves a re-solution heat treatment and, since this is likely to be the case for stainless steel also, it would mean a 100% loss of strengthening. It has been shown that annealing low temperature prestrained Nimonic 80A at 750°C produced cavity growth\(^{383}\) and it was suggested\(^{386}\) that this occurred as a result of high residual stresses or due to dissolved gases. Temperatures must, therefore, be high enough to anneal out such residual stresses and hydrostatic pressure must be contemplated if gas has a significant stabilising influence on the cavities formed. There is some evidence that sintering does not occur during post-deformation annealing for 50h at 800°C in the present material where gas induced growth or stabilisation may play an important role.
6. ASSESSMENT OF MMTT STRENGTHENING

In austenitic steels MMTT does not exhibit the considerable benefits obtained in room temperature properties with bcc alloys. The reason is the high temperature required for ageing in the present alloy, so that recovery occurs and it is not possible to build up high dislocation densities. Furthermore, the introduction of irrecoverable fracture damage in the form of cavities at inclusions and coarse undissolved carbides, lowers the ductility drastically as the cumulative prestrain increases with MMTT. Conventional cold working has proved to be more effective in terms of strengthening efficiency and ductility at room temperature. In this respect MMTT appears to produce inferior low temperature strengthening and ductility to controlled rolling in which significant subgrain strengthening has been observed.

However, MMTT does have undoubted beneficial effects on recovery resistance, as shown in Figs. 4-53 and 4-86, at temperatures below 800°C. This is reflected in greatly improved creep resistance in this temperature range, particularly at low stresses where cavity growth has been established as a major creep process. If cavity nuclei could be eliminated from MMTT material by one or another of the methods outlined in 5.4.3 the minimum creep rate at, for example, 50 MPa and 750°C, would be about $10^{-7}$ h$^{-1}$. This is about three orders of magnitude lower rate than the best comparison treatment used in the present study. Unfortunately, it is almost impossible to compare MMTT with other proposed treatments, such as controlled rolling, because of lack of relevant data, or similar treatments where entirely different alloys have been used often with considerably increased volume fractions of dispersoids, or where data is classified (e.g. UKAEA, data on 20Cr-25Ni-Nb alloys). The ductility problems experienced in this work, however, also appear to have been encountered with the above treatments and seem to be a common feature of steels of the type studied in the present work, particularly after mechanical treatment. Only more work and a wider dissemination and discussion of experimental results can lead to a true assessment of MMTT in the light of other treatments and a fuller understanding of the variety of observations made in the present study.
7. **CONCLUSIONS**

The following are the main findings of this work:

(i) Improved creep and recovery resistance has been achieved after MMTT by the introduction of finely dispersed Nb(C,N) particles which stabilize a relatively high dislocation density (5-10 times greater than usually encountered during creep of similar alloys). The structure was novel in the sense that dislocations were distributed in a relatively uniform three-dimensional network in which there was approximately one particle for each dislocation segment. The MMTT substructure was very resistant to recovery in the industrial creep range (650° - 750°C), being controlled by the stability of the particles against coarsening.

(ii) The present work suggests that a scientific approach to the design of high temperature materials is feasible and MMTT appears to produce the upper limit of creep deformation resistance, against which standard the resistance provided by alternative treatments may be compared.

(iii) Low temperature strengthening, which was associated with the high dislocation and particle densities, was on the whole disappointing when compared with results obtained in iron and niobium alloys after MMTT. This was due primarily to the less rapid build up of dislocation substructure in the present material, because of the high ageing temperatures that had to be employed. To produce a large increment in flow stress the particles should precipitate finely and also rapidly enough to prevent dislocation losses by annealing.

(iv) Cavitation at inclusions and coarse Nb(C,N) particles developed during MMTT pre-straining resulted in poor low-temperature ductility especially after about 6% cumulative strain. This or an associated process was even more deleterious at high temperatures where cavity growth accounted for the major proportion (~75%) of creep strain at low and intermediate stresses.

(v) Experiments carried out to determine the behaviour of MMTT and stabilized material under varying load during creep suggest that presently accepted notions
of recovery times and internal stresses require reappraisal in terms of recoverable anelastic strain contributions to the transients observed in creep tests. A survey of recovery creep measurements performed on a wide range of metals and alloys indicated that this is likely to be of general importance.

7.1 Suggestions for Further Work

Further work is suggested in five main areas:

(i) To study the effect of alternative deformation modes in which tensile stresses are minimised with respect to shear stresses, e.g. torsion or rolling. In this manner, prestrain induced cavities and their adverse effect on ductility might be eliminated.

(ii) Use of materials free of coarse second phase particles or containing particles less susceptible to interface decohesion.

Both approaches (i) and (ii) are potentially valuable, though (i) would seem the more practicable. (ii) might be successful in that the choice of a dispersion possessing a high work of adhesion or interfacial cavitation resistance might delay cavitation to high enough strains, such that MMTT might be conducted without cavity formation, even during tensile prestraining.

(iii) Digressing momentarily from the theme of MMTT, but still remaining with prestrain cavitation, this work has highlighted the importance of the latter phenomenon, not just as a design problem but also as a potentially useful tool in the study of creep rupture. Prestrain induced cavities could be studied in order to obtain fundamental data concerning the kinetics and physics of cavity formation, growth and sintering. It might also prove a useful aid in the assessment of the susceptibility of materials to cavitation, at the materials development stage. Indeed, these suggestions are already receiving attention by Dyson, Henn and McLean at NPL and illustrate some unexpected spin-off from the project.

(iv) The long term structural stability, which in the present alloy was adequate to temperatures of 750°C, or even 800°C might be exploited further
by selecting a system with non-coarsening particles. Indeed, the substructural observations suggest that a form of MMTT might be successfully applied to conventional dispersion strengthened alloys.

(v) Another fruitful area for research into material behaviour at elevated temperatures is that associated with variable load tests. The influence of anelasticity on strain transients could most effectively be studied as a function of grain size since anelastic strains are known to increase with decreasing grain size. The utilization of large quantities of recently amassed internal friction data in this context would be desirable and the inter-relation between anelastic creep, stress relaxation and internal friction should be studied further. The latter inter-relationship has been sadly neglected in the field of metallurgy since the pioneering work of [\textsuperscript{393}] K\textsubscript{a}, [\textsuperscript{394}] Zener and others [\textsuperscript{395-398}].
REFERENCES


33. E. A. Almond and D. Hull, Phil. Mag., 1966, 14, 515.


35. B. B. Hundy, Metallurgia, 1956, 53, 203.


47. J. Bilde-Sorensen, private communication.


61. L. M. Brown, Phil. Mag., 1964, 10, 441.
91. A. Lena, Metal Progress, 1954, 66, 94.
98. T. Angel, JISI, 1954, 177, 165.
120. W. M. Lomer, Phil. Mag., 1951, 42, 1327.
121. A. Cottrell, Phil. Mag., 1952, 43, 645.
126. P. M. Hazzledine, private communication.
150. C. Boulanger, Rev. Met., 1950, 47, 547.


268. T. Gladman, private communication.


271. W. Carrington, private communication.


274. R. K. Ham, Phil. Mag., 1961, 6, 1183.


284. R. P. Smith, ibid, 1962, 224, 190.


300. R. Summerling and D. Rhodes, J. Nucl. Mat. 1971, 40, 121.

301. H. Stuart and N. Ridley, JISI, 1970, 208, 1087.


308. Y. Ishida, private communication.


322. J. Bilde-Sorensen, private communication.

325. Ref. 323, p.211.
333. R. V. Hesketh, Phil. Mag. 1963, 8, 1321.
339. S. W. Zehr and W. A. Backofen, Trans. ASM, 1968, 61, 301.
352. G. J. Lloyd, R. J. McElroy and J. W. Martin, Proc. 3rd Int. Conf. on

1968, 2, 201.


356. R. J. McElroy, 'Practical Metallic Composites', Institution of Metallurgists,
in press.

(Macmillan).


373. T. Gladman, B. Holmes and I. D. McIvor, 'Effect of Second-Phase Particles


384. B. F. Dyson, private communication.
391. TRG Reports, UKAEA, (Springfields).
410. F. Garofalo, P. R. Malenock and G. V. Smith, Symposium on Determination of Elastic Constant, ASTM STP No. 129.


418. J. D. Lubahn, Trans. ASM, 1953, 45, 787.


426. J. H. Gittus, ibid, 1972, 6, 481.


433. B. F. Dyson and R. J. McElroy, unpublished work.

434. G. J. Lloyd and R. J. McElroy, Phil. Mag., to be published.

435. B. Gale, private communication.

MULTIPLE-MECHANICAL- THERMAL TREATMENT OF AUSTENITIC STEEL

Volume 2 - Appendices and Figures

by

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A Thesis submitted to the University of Surrey for the Degree of Doctor of Philosophy

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Variation of 0.2% proof stress with number of high temperature (800°C) MMTT cycles (ageing under zero or relaxed stress; straining at 10^-3 s^-1).

Variation of relaxed stress with number of high temperature (800°C) MMTT cycles (ageing under zero or relaxed stress; straining at 10^-3 s^-1).

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(b) 3-MMTT cycles;
(c) 5-MMTT cycles;
(d) 6-MMTT cycles + 2% strain (7 incomplete MMTT cycles);
(e) 7-MMTT cycles;
(f) 13-MMTT cycles.

Structure after 5-MMTT cycles in (001) orientation (HVEM).

Structure after 7-MMTT cycles and straining to the UTS (εc = 29%) (HVEM).

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APPENDIX 1

REVIEW PAPER BY R. J. McELROY AND Z. C. SZKOPIAK

"Dislocation-Substructure-Strengthening and Mechanical-Thermal Treatment of Metals"

Dislocation-Substructure-Strengthening and Mechanical-thermal Treatment of Metals

by R. J. McElroy and Z. C. Szkopiak

Introduction

In the search for ever-stronger materials to meet the constantly increasing service requirements of an advanced technology, several approaches have been followed. The result of these researches have been discussed in general reviews1-2 and in a number of specific papers dealing with

(i) Solution-strengthening3
(ii) Strain-hardening4,6
(iii) Precipitation- and fine-particle-strengthening6
(iv) Quench- or vacancy-strengthening7
(v) Radiation-strengthening8
(vi) Martensitic-strengthening9
(vii) Grain-size control10
(viii) Fibre reinforcement11,12

Each of the above strengthening methods was originally developed on a purely empirical basis. Subsequent attempts to describe them in terms of the theory of crystal dislocations13,14 and point defects15 have led to a fairly clear understanding of the principles involved in the strengthening mechanisms.

Following the considerable success achieved in being able to describe and frequently to predict, at least qualitatively and often quantitatively, the mechanical behaviour of materials, it seems appropriate that attempts should now be made to design, from first principles, materials and treatments to meet the new service requirements. One of the most interesting strengthening mechanisms, from the point of view of the metallurgist, would seem to be that of strain-hardening and associated phenomena, since engineering materials are exposed to mechanical treatments as part of their fabrication schedule. An attractive aspect of such treatments is the presence of dislocations (as promoters of deformation), which could be utilized in strengthening treatments to reduce the emphasis previously placed on expensive alloying additions and tedious annealing treatments with their associated problems. Such an approach has already been followed in the now familiar thermomechanical treatments,16,17 e.g. ausforming and isofoming, in materials undergoing a phase transformation. In these cases, deformation is employed to modify the mode of decomposition of a high-temperature metastable solid solution and to produce a more stable dislocation substructure. However, the resultant improvement in properties is chiefly associated with a better phase distribution, the more stable dislocation distribution aiding it rather than being in itself of major importance.

More recently the concept has been applied to single-phase materials not undergoing phase transformations. In this case hardening is due to a build-up of dislocation substructure. Such treatments are termed mechanical-thermal treatments (MTT),18 to differentiate them from the ausforming type. MTT involves the introduction of a high dislocation density by cold working. The dislocation distribution is then modified by some form of post-deformation recovery heat-treatment to allow solute pinning or to rearrange the dislocations into more stable configurations, if possible, without an excessive reduction in the overall dislocation density. These treatments are essentially based on a form of strain-hardening distinguished only by the attempt to modify a dislocation substructure deliberately so as to obtain a desired combination of mechanical properties. This form of strengthening could alternatively be referred to as 'substructure-strengthening'.

The purpose of this work is to survey the effects of dislocation substructure on strength, to describe mechanical-thermal treatments already proposed and to suggest some possible alternative treatments or modifications to the existing ones. It is felt that this field has been neglected in the past, and it is the present aim not only to survey it but also to stimulate interest among those who have not yet recognized its potentialities.

Dislocations and Mechanical Properties

The effect of dislocations on the mechanical properties of metals has been extensively reported1,2,13,14 and as such is not dealt with in this review. To provide a general background, however, a few relevant points will be made concerning dislocations and strength of metals.

Purely on the basis of dislocation density ($\rho$) (dislocation densities are commonly expressed in terms of the total length of dislocation line per unit volume of material, i.e. cm/cm$^3$ or cm/cm$^2$) there are two obvious choices open to the metallurgist, at least in theory if not in practice, for the selection of high-strength materials.19 One is to produce crystalline materials with a perfect lattice, i.e. $\rho = 0$. The second is to produce materials with a very high dislocation density ($\rho = 10^{11}-10^{13}$ cm$^{-2}$) and with the dislocations randomly distributed throughout the lattice.
The theoretical strength of metals, being $\sim 10^{-1} G \times (G \text{ is the shear modulus})$, while virtually obtainable in nearly perfect single-crystal whiskers, has always eluded the metallurgist in real materials. This is due to the unavoidable introduction of dislocations during solidification by thermal contractional stresses during cooling, and as a result of phase transformations. Even the high-strength single-crystal whiskers are by no means free of dislocations, since the kinetics of the growth process requires a single screw dislocation perpendicular to the growth surface. Actually, it is fortunate that dislocations are present in engineering materials, for without them the ductility necessary in fabrication processes, which are an integral part of the production schedule of most metal products, would not be available. Similarly, in many fabrication processes the ability to work harden by mechanisms involving dislocation generation, multiplication, and mutual interaction is essential, especially in sheet-rolling and wire-drawing operations. Thus, having accepted dislocations and their profuse multiplication (from $\sim 10^5$ to $\sim 10^{11} \text{ cm}^{-2}$) during fabrication as a reality, and recognizing their function in determining the mechanical properties of materials, a constructive solution should become apparent to the problem of producing metals with the best combination of mechanical properties for any particular engineering application.

**Arrangement of Dislocations in Annealed Crystals**

**Historical Note**

The subject of dislocation distributions in crystalline materials, while unknown to the investigators at the time, started with the formulation of the theory of reflected X-ray intensities from large perfect crystals by Darwin and Ewald. The predicted X-ray intensities from well-annealed ‘perfect’ crystals were between one and two orders of magnitude lower than those actually observed, while the angular range of reflection was about an order of magnitude too large. Darwin, to explain this discrepancy, introduced the concept of ‘mosaic’ structures. He suggested that the reflecting lattice was broken up into small, slightly misoriented blocks of truly perfect crystals. His calculations showed that blocks $\sim 10^{-5} \text{ cm}$ across and with misorientations $\sim 2-3 \text{ minutes of arc}$ would account for the observed discrepancy in integrated intensity. While these facts were known, all attempts to explain how such a structural state arose failed until the introduction of dislocation theory in 1934 by Taylor, Orowan, and Polanyi. The problem was finally resolved by Bragg and Burgers, who proposed the theory of low-angle dislocation boundaries. This period, as well as the post-war period to 1956, has been reviewed by Hirsch. Since the war, more refined studies of substructures and their effect on mechanical properties have been carried out, especially following the introduction of X-ray microbeam techniques, improved etch-pit studies, and, more recently, thin-foil electron microscopy. These techniques have revealed that dislocations in annealed crystals are distributed in two basic types of arrangement. These are:

1. Three-dimensional networks (the Frank net)
2. Planar dislocation networks (low-angle or sub-grain boundaries)

**Frank Network**

Elementary dislocation theories specify that a dislocation line may terminate only at a free surface or internal interfaces such as grain boundaries. Any dislocation not fulfilling these conditions must form a continuous loop. However, if comparatively high dislocation densities are present, as is the case even in most annealed materials, dislocation interactions necessarily occur. In most crystals the geometry allows for dislocation segments to form junctions or nodes in much the same way as grain boundaries meet at triple points. The only requirement for such a triple junction to form, or in fact any other configuration, is that the sum of the Burgers vectors of the segments forming the node is zero. This condition is generally fulfilled and, as a consequence, structures similar to that represented diagrammatically in Fig. 1 are present. Such networks, known as Frank networks, have been observed experimentally by transmission electron microscopy (Fig. 2). It is believed that Frank networks have high thermal stability and only tend to coarsen in a manner analogous to grain growth.

It has been proposed that Frank networks are sufficient to account for most aspects of the mosaics reported by crystallographers, especially in cold-worked and annealed single crystals in which an excess of dislocations of one sign is absent. However, in cases where such an excess is introduced, for example by plastic bending or during the growth from the melt (striation substructures), low-angle boundaries are formed. This is also true of polycrystals in which lattice bending and rotation occur, resulting in planar dislocation arrays on annealing. Regardless, however, of whether such arrays exist a Frank network is invariably present.

**Low-Angle Boundaries**

An excess of dislocations of one sign in the lattice can give rise, after annealing, to arrangements of dislocations in planar arrays. The two most common forms are:

1. Simple symmetrical tilt boundaries (Fig. 3)
2. Pure twist boundaries (Fig. 4)

These arrangements occur by a process known as polygonization.

Simple tilt boundaries consist of vertical arrays of parallel edge dislocations and can arise by climb and glide of dislocations from more random distributions, e.g. those produced by lattice bending and kink-band formation. The geometry of these boundaries (Fig. 3) is such that the dislocation distribution is governed by a fundamental relationship

$$\bar{b}/h = 2 \sin \theta/2$$

where $\bar{b}$ is the Burgers vector of dislocations, $h$ the vertical dislocation spacing in the boundary, and $\theta$ the angle of misorientation across the wall. This reduces to

$$\bar{b}/h = 0$$

for small misorientations ($\theta < 5^\circ$), which normally occur. This relationship has been confirmed by etch-pit studies. Similarly, networks of parallel screw dislocations can arise, as shown in Fig. 4, resulting in a pure twist boundary. Equations (1) and (2) hold equally for these boundaries.

The above two types of boundaries are generally found in specially deformed and annealed single crystals. In polycrystals, on the other hand, where grain size, shape, and orientation influence dislocation configurations, more complex dislocation boundaries due to the imposed constraints are observed.

An important feature of dislocation configurations, which has a considerable effect on their thermal and mechanical
Fig. 1 The arrangement of dislocations in a well-annealed crystal: the Frank net.

Fig. 2 Three-dimensional dislocation network in α-iron.

Fig. 3 A pure tilt boundary, viewed parallel to edge dislocations in the wall.

Fig. 4 A pure twist boundary. The boundary is parallel to the plane of the figure and the two subgrains have a small relative rotation about their cube axis, which is normal to the boundary. Open circles represent atoms just above the boundary and solid circles the atoms just below. The subgrains join together continuously except along the two sets of screw dislocations, which form a cross-grid.
Comparison of Subgrain Sizes Produced in Metals and Alloys of Differing Crystal Structure and Stacking-Fault Energy during Creep, Hot Working, and Room-Temperature Tensile Tests

<table>
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<tr>
<th>Crystal Structure</th>
<th>Material</th>
<th>Stacking-Fault Energy, $J/\text{m}^2$ (erg/cm$^2$)</th>
<th>Cold Tensile</th>
<th>Hot Work</th>
<th>Creep</th>
<th>Ref.</th>
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<td></td>
<td>Iron</td>
<td>0.2 (200)</td>
<td>1-2</td>
<td>39, 50-58</td>
<td>71-74</td>
<td>4-12</td>
</tr>
<tr>
<td></td>
<td>Fe/Si</td>
<td>0.2 (200)</td>
<td>1-3</td>
<td>54</td>
<td>74</td>
<td>2-20</td>
</tr>
<tr>
<td></td>
<td>Aluminium</td>
<td>0.2 (200)</td>
<td>1-7</td>
<td>45</td>
<td>77, 78</td>
<td>10-100</td>
</tr>
<tr>
<td></td>
<td>Nickel</td>
<td>0.12 (120)</td>
<td>0-5-2-0</td>
<td>45</td>
<td>—</td>
<td>10-20</td>
</tr>
<tr>
<td></td>
<td>Copper</td>
<td>0.05 (50)</td>
<td>0-6-1-0</td>
<td>45, 83</td>
<td>—</td>
<td>5-10</td>
</tr>
<tr>
<td></td>
<td>Stainless steel</td>
<td>0.015 (15)</td>
<td>0-5-1-0</td>
<td>86, 87</td>
<td>—</td>
<td>2-7</td>
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</table>

The effect of solute atoms on dislocation configuration manifests itself through its effect on the stacking-fault energy (SFE). For example, the SFE of copper and silver may be reduced, by as much as 60%\(^4\),\(^5\) by the addition of aluminium, zinc, or germanium. Such a change in SFE produces significant changes in dislocation arrangement.\(^4\),\(^4\)\(^6\). The resultant structural modifications are caused by the tendency for dislocations to dissociate into sets of partial dislocations, provided the SFE is sufficiently low ($0.03 \text{ J/m}^2$ ($< 30 \text{ erg/cm}^2$). Values of SFE for some common metals and alloys are given in Table I.

The most significant effect, as far as the effect of SFE is concerned, on the character of the network is the manner in which extended dislocations react at nodes. The geometry of these interactions has been described by Whelan\(^4\)\(^6\) and is illustrated in Fig. 5. It is apparent that the partial dislocations bounding the stacking fault become constricted at the nodal junctions, while remaining dissociated over their free length. This will have a considerable effect on the mechanical and thermal stability of the networks and hence on the mechanical properties of the material.

Another effect of solute atoms is their tendency to inhibit growth of networks by the mechanism of solute drag.\(^3\) In materials of medium SFE segregation to dislocations takes place, lowering the effective SFE. This can occur as a result of the differences in structure and therefore solubilities in and on either side of the fault, as suggested by Suzuki.\(^4\)\(^8\) Stabilization of the network will also take place.

The effect of SFE on the formation of low-angle boundaries appears to be incompletely understood. It can, however, be stated that materials having low SFE tend not to form dislocation boundaries. In metals where such boundaries form they are complicated by the dissociated nature of the dislocations.\(^4\)

Dislocation Arrangements in Deformed Polycrystals

Room-Temperature Deformation

The deformation of well-annealed crystals is accompanied by a profuse multiplication of dislocations, the density of which increases progressively with increasing plastic strain from $10^4$ to $10^6$ cm\(^{-2}\). These dislocations interact with one another\(^1\)\(^,\)\(^2\)\(^,\)\(^3\) and with obstacles in their glide planes, such as precipitate particles,\(^6\) causing the flow stress to increase. This phenomenon is known as strain-hardening.\(^4\),\(^5\)
The types of dislocation arrangements exhibited by most common metals and alloys during tensile deformation are in general similar, especially in polycrystalline materials. Ignoring the slight differences between metals of different crystal structures, these substructures are a function of one material variable, SFE, and three experimental variables: the degree of plastic strain, deformation temperature, and strain rate. The main features of deformation substructures are similar in both bcc and fcc metals and their alloys, provided they have SFE higher than \( \sim 0.03 \text{ J/m}^2 \) (30 erg/cm\(^2\)). In all these cases the processes of cross-slip and climb are relatively easy. Thus, to avoid repetition, \( \alpha \)-iron will be discussed in order to describe the low-temperature (\( < 0.4 T_m \)) deformation substructures.

The deformation substructures observed in \( \alpha \)-iron have been the subject of a number of investigations. The structures observed during room-temperature tensile deformation are as follows. At strains below \( \sim 1\% \) comparatively straight dislocations are produced, which interact with one another giving rise to jogs and attractive or repulsive junctions (Fig. 6) that form, with increasing strain, fairly close-knit cross-grids of straight dislocations closely resembling a net (Fig. 7). Continued deformation to \( \sim 10\% \) strain causes dislocations to cluster at network junctions, leading by a 'mushrooming-growth' process to tangles (Fig. 7). These tangles grow both in number and size during progressive straining, producing an 'incipient cell structure', until at \( \sim 10\% \) strain the tangles link up into a fairly well defined cell structure. The cells are characterized by cell walls of high dislocation density, in the form of a rather crude Frank network (Fig. 8). During further tensile deformation there is little tendency for the cell size to decrease. However, with increasing strain the dislocation density within the cell walls increases, resulting in an increase in angular misorientation up to \( 5^\circ \) and in the sharpness of cell walls. During working operations, e.g. rolling, where very much larger deformations (\( \sim 70\% \) reduction in area) are produced, there is some tendency towards cell refinement, though the main trend is to produce cells elongated in the direction of rolling, often exhibiting a marked preferred orientation.

While the precise mechanism of tangle and cell formation is still a subject of debate, their close interrelation (cells are never observed without prior tangle formation) indicates that the same (or closely related) processes are responsible for both.

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While the precise mechanism of tangle and cell formation is still a subject of debate, their close interrelation (cells are never observed without prior tangle formation) indicates that the same (or closely related) processes are responsible for both.
Temperature- and Strain-Rate-Dependence

$T < 0.4 T_m$

It has been observed that the dislocation distribution and arrangement in iron are strongly dependent on temperature. The strain necessary to produce cellular structures decreases with increasing deformation temperature. The cellular arrangement forms almost immediately on straining at 200°C ($0.26 T_m$), while at $-75^\circ$C ($198 K$) ($0.11 T_m$) ~16% strain is required. Keh and Weissmann constructed a diagram to illustrate these observations (Fig. 9, full line). In Fig. 9 the strains at which 50% of the grains exhibited cellular structures are plotted against deformation temperature. It is felt that the structure Keh and Weissmann have designated "cellular" is not truly representative of what is generally recognized by this term. Redefining the cellular arrangement as that where continuous walls of dislocations divide the crystal into numerous distinct 'crystallites' or 'blocks', each being slightly misoriented with respect to its neighbours, it becomes apparent that the structures chosen as being cellular by Keh and Weissmann are of a more primitive nature. They are much more tangled and discontinuous than those shown in Fig. 8. An alternative way of defining this structure is to consider the manner in which the tangle spacing or spacing between localized, high-density configurations of dislocations varies with strain. Generally the spacing follows a curve similar to that shown in Fig. 10, which, for room-temperature deformation, tends to a constant value at ~10%. This value appears to be more correct than that given in Fig. 9 by Keh and Weissmann; it agrees well with their own observations and those of others. If the corrected values are plotted in Fig. 9 (broken line 1) the same trend is evident. Again, if the strain at which simple networks give way to tangle formation is plotted in Fig. 9 (broken line 2), it seems to follow a similar trend. Although the absolute strain values shown in Fig. 9 may be questioned, it is clear that trends of this type occur. It might, therefore, be expected that these structures bear some relation to the behaviour of dislocations in iron, the network structure being due to the high Peierls-Nabarro force and its temperature-dependence and the cellular type being due to the cross-slip process and its temperature-dependence. It should be noted that at temperatures below ~$-100^\circ$C ($173 K$) ($0.09 T_m$) cell formation, as defined above, does not occur during tensile tests to failure, except possibly in the necked region. Even there only tangles or 'incipient cell structures' are generally observed. Furthermore, at very low temperatures, ~$-200^\circ$C ($73 K$) ($0.04 T_m$), thermal energy is insufficient for conventional dislocation motion and deformation twinning occurs, as it does also at higher temperatures and very high strain rates. During the application of more complex stress systems, e.g. by rolling, where larger strains may be achieved before failure, there is a tendency for cells to form at high strains (~50%) even at relatively low temperatures. Similarly, the cell size is a function of deformation temperature, as shown in Fig. 11.

$T > 0.4 T_m$

Under both creep and hot-working conditions and for a considerable proportion of high-temperature straining most materials undergo steady-state or constant-strain-rate deformation at constant stress. In these conditions the substructures produced tend to depend on strain rate, temperature, and stress over the whole range of strain rates involved. These vary from $10^{-8 s^{-1}}$ in creep to $10^{3 s^{-1}}$ during hot working. Creep studies on a wide range of materials have been...
Me Elroy and Szkopiak: Mechanical-Thermal Treatment of Metals

 Courtesy Iron Steel Inst.  

 Fig. 12 Dislocation structures in α-iron after creep at 550 °C and a stress of 45-3 MN/m² (3 tonf/in²). x 30 000.

 Fig. 13 Dislocation structure in a low-SFE material, stainless steel, after light deformation, showing an irregular arrangement of pile-ups and nodes at twin boundaries. x 12000.

 the subject of a number of reviews. Hot working, on the other hand, is only now being studied more extensively because of its relevance to fabrication metallurgy and thermo-mechanical processing. The available evidence from electron microscopy of metals and alloys after creep and hot working indicates that during the initial stages of deformation rapid dislocation generation occurs. Subgrains are formed at strains varying from 1 to 10% in creep and 20 to 30% during hot working. These subgrains grow, attaining a ‘pseudo-equilibrium’ size by the commencement of the steady-state stage of deformation, after which little change in size occurs until the final or tertiary stage leading to failure is reached. During steady-state creep the subgrains remain equiaxed, while in hot-worked metals at ~ 0.6 Tm many elongated grains have been observed, which give way to completely equiaxed structures at above ~ 0.7 Tm. The subgrain size is normally found to increase with increasing temperature of deformation and decreasing strain rate. In general, subgrains produced during creep are about one order of magnitude larger than those produced by hot working. The perfection of subgrains, or the degree of strain associated with them, is similarly related to temperature and strain rate. Subgrains produced during hot working tend to be less perfect than those produced in creep. It appears that the subgrain size and perfection are related to SFE, the former tending to decrease (Table I) and the latter to increase with increasing value of this parameter. Below a value of ~ 0.03 J/m² (30 erg/cm²) it is often found that dynamic recovery is not capable of producing the stress relief necessary to maintain the required balance between work-hardening and recovery during steady-state deformation and dynamic recrystallization occurs. This is, however, true only for strain rates in the hot-working range where the result is often catastrophic, as it would be in creep.

 Typical creep substructures observed in iron are shown in Fig. 12, which illustrate the structures commonly found in most materials. It may be seen that there is a clearly defined subgrain-interior network within a regular subgrain structure, the boundaries of which resemble those produced by polygonization.

 Alloying Effects
 Solid Solutions

 Alloying additions, especially interstitial elements such as carbon and nitrogen in iron, have a considerable effect on dislocation density and distribution. In general, the tendency of alloying is to increase the rates of dislocation generation and multiplication, leading to higher dislocation densities for equivalent strains. Tangle and cell formation are also accelerated to some degree and often this is accompanied by a smaller final cell size. Under certain conditions, for example in the blue-brittle region (180-300 °C) in iron and steels, where the alloying elements or impurities are sufficiently mobile to be able to diffuse to dislocations and pin them, these effects are enhanced. In this temperature range tensile stress/strain curves exhibit the so-called serrated or jerky flow effect. Dislocation structures have a characteristic appearance, consisting of ragged tangles that form an ‘incipient cellular structure’ (Fig. 7). This structure is a consequence of the fact that long-range rearrangements, which are a necessary prerequisite of cell formation, are inhibited owing to premature locking of dislocations by solute atmospheres. The effect is similar in origin to that of strain-ageing and is often referred to as dynamic strain-ageing. The SFE of fcc materials is sensitive to composition and this is reflected in the deformation substructures. In general, during the deformation of these materials planar arrays of extended dislocations form on primary slip-planes. These are often pile-ups at obstacles such as grain boundaries or planar networks with extended nodes (Fig. 13). As the strain
is increased these give rise to bands of extended dislocations and complex pile-ups with apparently little desire to form cell structures at low strains. However, at higher strains these arrangements give way to rather ragged, tangled cellular structures.44-45

However, below this value, and for an increasing degree of deformation, as SFE decreases extended dislocations in planar pile-up arrays are observed. Both these observations are consistent with single-crystal results concerning dislocation structure and τ_{III}.104

**Dispersed Phases**

The effect of dispersed second-phase, incoherent particles, such as carbides in steel,105 is in many ways similar to that of solutes. By forcing screw dislocations to cross-slip they increase the rate of dislocation multiplication. It has been suggested by Barnby and Smith106 that shear stresses acting on coarse particles cause elastic distortions of precipitates, which are relieved by the generation of accommodation dislocations. These rapidly build up during deformation forming cell walls between precipitates. Ashby106 produced a similar model, which, in an analogous manner, is based on the introduction of 'geometrically necessary dislocations' to maintain continuity. This model provides adequate explanations for both the structure and deformation behaviour, especially work-hardening, of dispersion-hardened materials. The above models are applicable mainly to materials containing incoherent or partially incoherent particles. In such materials cell and subgrain sizes are a function of the dispersion spacing.105,107,108 The pinning effect of the particles on subgrains is well known and is responsible for their reduced coarsening rates during annealing and hot deformation.108,109

**Relation between Dislocation Density and Stress and Strain**

Dislocation density (ρ) in deformed metals shows definite relations110 with plastic strain (ε_p) and the corresponding stress (σ_p) of the form

\[ \rho = \rho_0 + C \varepsilon_p \]

and

\[ \sigma_p = \sigma_0 + \alpha G \frac{b \rho}{\varepsilon_p} \]

where \( \rho_0 \) is the original dislocation density of the material before deformation, \( C \) and \( \alpha \) are constants, \( \sigma_0 \) is the friction stress, \( G \) the shear modulus, \( b \) the Burgers vector, and \( \alpha \) a constant frequently referred to as the dislocation-strengthening efficiency. Most modern theories of work-hardening lead to these relations, which have been observed in a variety of metals.88,84,53,57,58,111-113 Furthermore, they appear to apply equally well to total dislocation density, regardless of subgrain structure, mobile dislocation density, and dislocation density within subgrains.

From the dislocation-strengthening viewpoint the dislocation-strengthening efficiency (α) is an important parameter. It has values of 0.5–0.8 for pure metals, 0.9–1.3 for solution alloys, and 1.4–1.8 for precipitation-hardened and dispersion-hardened materials. Since α depends on the rates of dislocation generation or multiplication, its variation follows from the effects of solute atoms and precipitates on these mechanisms.

**Post-Deformation Annealing**

The effects of annealing cold-worked materials at progressively higher temperatures on the defect density and distribution, physical and mechanical properties, and microstructures have been studied extensively in recent years.116-120 It is, therefore, proposed to discuss only some of the more relevant observations regarding substructural changes.

The processes occurring during the annealing of cold-worked metals may for convenience be divided into six groups according to the temperature range. These are as follows:

(i) Low-temperature recovery (< 0.2 \( T_m \))
(ii) Intermediate-temperature recovery (> 0.2–0.3 \( T_m \))
(iii) High-temperature recovery (< 0.4 \( T_m \))
(iv) Recrystallization (> 0.4 \( T_m \))
(v) Grain growth (> 0.5 \( T_m \))
(vi) Secondary recrystallization (> 0.8 \( T_m \))

The last three processes involve drastic reductions in overall dislocation density through the nucleation of new, relatively strain-free grains, followed by their growth to reduce the total grain-boundary energy. They lead to a marked decrease in strength and, generally, an increase in ductility, returning the material to a state substantially similar to that already described for well-annealed crystals. Consequently, these aspects of annealing have little relevance to the present review, other than by virtue of the fact that they permit, by correct choice of post-fabrication treatment, the physical state of the starting material to be varied appreciably. The annealing processes with which this review is concerned are those in which recovery occurs with the minimum change in dislocation density.

**Low-Temperature Recovery (Point-Defect Migration)**

During low-temperature recovery121 large-scale elimination and redistribution occurs of deformation-induced point defects, such as vacancies and interstitials. The processes involved are predominantly the recombination of vacancies and interstitials,12 the clustering of like defects, often forming stacking-fault defects,15 and the migration of vacancies14 and interstitial atoms to dislocations.88,97 The latter process causes the familiar strain-ageing phenomenon94,97 in bcc materials.
Fig. 15 Effect of recovery-annealing temperature on dislocation structures in α-iron after ~8% deformation in tension at room temperature. (a) as deformed, (b) 1 h at 400 °C, (c) 1 h at 500 °C, and (d) 1 h at 600 °C. × 40 000.

Table II

<table>
<thead>
<tr>
<th>Metal</th>
<th>Jog Energy, eV</th>
<th>SFE, J/m² (erg/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>0.8</td>
<td>0.05 (50)</td>
</tr>
<tr>
<td>Ni</td>
<td>0.2</td>
<td>0.12 (120)</td>
</tr>
<tr>
<td>Al</td>
<td>0.1</td>
<td>0.2 (200)</td>
</tr>
<tr>
<td>Pb</td>
<td>0.1</td>
<td>—</td>
</tr>
<tr>
<td>Mg</td>
<td>0.2-0.3</td>
<td>—</td>
</tr>
<tr>
<td>Zn</td>
<td>0.2</td>
<td>—</td>
</tr>
<tr>
<td>Cd</td>
<td>0.3</td>
<td>—</td>
</tr>
</tbody>
</table>

Fig. 16 Effect of annealing at 550 °C on (a) room-temperature flow stress and (b) dislocation density in α-iron, after 16% tensile deformation (25 °C).
During this recovery stage little change in substructure takes place other than the appearance of some stacking-fault defects, dislocation loops, and helical dislocations in fcc materials. However, significant changes may occur in mechanical properties as a result of strain-ageing by locking dislocations, thus reducing the density of mobile dislocations.

Intermediate- and High-Temperature Recovery (Polygonization)

The intermediate-temperature recovery stage, on the other hand, comprises minor dislocation rearrangements and short-range interactions between dislocations in slip bands. This is often accompanied by cell growth and a reduction in the thickness and raggedness of cell walls. These interactions usually consist of dislocation annihilation by glide and cross-slip under the mutual attractions between dislocations of opposite Burgers vector, resulting in reduction of the overall dislocation density from \(10^{10} - 10^{11}\) cm\(^{-2}\) to \(\sim 10^9\) cm\(^{-2}\). Finally, during high-temperature recovery, large-scale rearrangements occur, by climb and cross-slip, into low-energy configurations of low-angle subgrain boundaries. These proceed by the process of polygonization. The formation and coarsening of the Frank network to an 'equilibrium' size can also take place.

Typical structures observed in deformed iron after annealing in the range 20-600°C are illustrated in Fig. 15. These indicate the increasing rate of rearrangement of dislocations and decreasing dislocation density with increasing annealing temperature for a constant degree of prestrain. The dislocation density varies during annealing at 550°C, as shown in Fig. 16. Also shown in Fig. 16 is the variation of flow stress. It is interesting to note that while the dislocation density decreases steadily the flow stress decreases to a constant value after only 2 h, at which point a stable subgrain structure (\(\sim 5\) μm) has been established. This observation and that by Roberts and Jolley are of importance in the field of substructure-strengthening.

In low-SFE metals and alloys, especially where strong solute/dislocation interactions occur, the situation is different. Frequently recovery is suppressed and recrystallization is the first major process to operate, often at considerably lower temperatures than expected. This may be due to the fact that the driving force for the recrystallization process is effectively higher, since no major redistribution of dislocations or reduction in dislocation density occurred at lower temperatures. It appears that in cases where polygonization can, nevertheless, take place a reduction in subgrain size is observed, as reported by Ancker and Parker for nickel and its alloys. However, this is not a general rule since, for example, pure copper does not polygonize but does so in the presence of impurities—an apparent anomaly.

This may be due to the high jog energy of copper, which, taken with its relatively low SFE (Table II), makes jog formation less favourable and hence climb more difficult. This point might explain why certain metals of lower stacking-fault energy than copper polygonize readily, their jog energies being considerably less (Table II).

Summary of Dislocation Substructures

From the foregoing sections, it is apparent that dislocation structures, in all but the lowest-SFE materials, consist of two main types:

(i) A random three-dimensional network of dislocation segments joined at nodes, the Frank network.

(ii) Almost planar arrays of dislocations arranged in subgrain or cell walls.

These two types, often coexisting, are found after both deformation and annealing, though the degree to which they conform to the idealized models may differ. In low-SFE materials such structures are ragged under most circumstances. These arrangements are not formed after low levels of cold work, in which case the classic pile-up of extended dislocations occurs, giving way to cellular structures only at high levels of deformation.

Effect of Substructure on Mechanical Properties

General Observations

Creep

The effect of prestrain on subsequent creep behaviour has been studied in a number of investigations. The results seem to indicate that small amounts of cold work before creep testing increase the creep-resistance, which at a limiting degree of prestrain reaches a maximum and then decreases rapidly. Zschokke and Niehus found that the creep limit of an austenitic steel increases initially, with prestrain, to a maximum after which prior deformation causes a rapid decrease in this property. The degree of prestrain to give the optimum creep life increased with decreasing temperature in the range 550-750°C, as did the relative increase in creep limit (Fig. 17). In recent years similar results have been obtained by Russian workers during their search for optimum mechanical-thermal treatments. Other work in this field on aluminium, nickel, and its alloys was concentrated on the effect of prestraining and recovery-annealing treatments on the shape of the creep curve and the main creep parameters. Sherby et al. showed that, in aluminium, pre-straining by a constant amount at temperatures in the range 270 (3 K) to 450°C, followed by a short recovery anneal at 250°C, had a dramatic effect on the creep behaviour. The creep rate decreased and the rupture life increased as the pre-strain temperature was reduced. Results of a similar nature have been obtained for nickel and its alloys after varying degrees of prestrain at room temperature. Creep recovery-annealing at 800°C the creep-rate decreases and the rupture life increases with increasing prestrain (Fig. 18). McLean and Tate observed similar effects in aluminium where, with a constant degree of prestrain at room temperature followed by annealing at temperatures in the range 250-400°C, the creep-resistance increased with decreasing annealing temperature. The observations were also confirmed by Russian work.

While it is fairly clear that, at relatively high stresses, prestraining and subsequent annealing have a beneficial effect on creep properties, the situation is more complicated at low stresses. Betteridge, in low-stress creep tests on aluminium found that prestraining increased the creep rate, as did Milson working with Al-3.5% Mg. Both these workers used large prestrain deformations (\(\sim 40\)%). It therefore appears that the significant improvements in creep-resistance are obtained at low degrees of prestrain and in relatively high-stress situations. This is probably attributable to recrystallization occurring in materials prestrained by large degrees, the effect being accelerated in the low-stress condition.

Yim and Grant showed that the creep properties of prestrained nickel were superior to those of prestrained and polygonized nickel. These results indicate that dislocation density and not subgrain size is the important factor in influencing creep properties, especially the creep rate. However, in their tests it was observed that while the creep rate
Fig. 17 Effect of cold work on the creep limit of an austenitic stainless steel (18% Cr, 9% Ni, 1% W, 0.7% Ti, 0.1% C).

Fig. 18 Effect of prestrain and recovery anneal at 800°C on the shape of the creep curve of nickel at 700°C.

Fig. 19 Effect of prestrain and recovery anneal at 800°C on the tensile properties of nickel.

Fig. 20 Dependence of flow stress increase of zinc single crystals on misorientation angle of single tilt boundaries introduced by plastic bending and annealing at 400°C.

Fig. 21 Effect of subgrain size on flow stress in aluminium.

Fig. 22 Effect of subgrain size on hardness in aluminium alloys.
was not significantly different the rupture life of pretrained material was inferior, a fact possibly reflecting the effect of internal stresses on cavity formation and microcracking. In general, it appears that the effect of prestrain/anneal treatments on creep properties is unclear and is complicated by the type of test performed.

**Low-Temperature Mechanical Properties**

In a similar manner the low-temperature mechanical properties, particularly the work-hardening rate and tensile strength, varied with the conditions of the prestrain treatment. The general conclusion is that an increase in these parameters occurs that is attributed to the refinement of substructure. It has been established that the tensile properties of steel, aluminium, and nickel and its alloys, may be significantly increased by prestraining and annealing treatments. Typical results are shown in Fig. 19 in which the effect of different degrees of prestrain followed by recovery- or warm working, is illustrated. Similar results were obtained by varying the temperature of the prestrain treatment and recovery- or warm working. Washburn, in a classical experiment, introduced kink boundaries into zinc single-crystal shear-test specimens by room-temperature straining and found that the stress required to flow offered by such boundaries was very low, the strength of the crystal being virtually unaffected. However, specimens annealed at $400\degree\text{C}$ after prestraining showed quite noticeable increases in flow stress. It was also observed that the flow stress of these single crystals increased with the angular misorientation across the boundaries, as shown in Fig. 20 for edge-dislocation walls. A similar result was obtained for pure twist boundaries. This was the first attempt to quantify the effect of polygonized subgrain boundaries on mechanical properties. Subsequent attempts to explain the effect of individual substructural features have followed two main approaches. In order of their coverage in the literature, the effect of subgrain structure are:

(i) Subgrain size and misorientation
(ii) Subgrain-interior network density

**The Effect of Subgrain Structure on Mechanical Properties**

**Historical Note**

Early X-ray studies of metals showed that observed X-ray intensities were incompatible with the idea of the perfect crystal. This led to the concept of the 'crystallographer's mosaic'. The nature of the mosaic remained unknown until the investigation of lattice dislocations in the early 1930's. At that time Taylor introduced the basic concept of generation and interaction of dislocations during deformation, and suggested a relationship between stress and dislocation density. This was confirmed by more refined metallographic experimental techniques developed in the 1950's, which also revealed that deformation the 'mosaic' changed its character and dimensions. Bragg considered the elastic distortion of mosaic fragments during straining and derived a relationship between the mean fragment size ($l$) and flow stress ($\sigma$) of the form

$$\sigma = G \frac{\tilde{b}}{l} \quad \cdots (5)$$

The stress calculated from equation (5) showed reasonable agreement with observed elastic limits for a number of metals. This was the first theoretical and experimental evidence that subgrain structure had an effect on the mechanical properties of crystalline materials.

**Experimental Techniques**

A number of techniques have been used to produce subgrain structures with the object of measuring their effect on mechanical properties. These may be divided into five general groups:

(i) Cold working
(ii) Cold working and annealing
(iii) Cold and warm working followed by annealing
(iv) Hot working followed by rapid cooling to suppress recrystallization
(v) Growth of single crystals from the melt, producing striation substructures, the 'subgrain size' being related to the solidification rate

Methods (i), (ii), and (v) involve single-stage treatment only and have therefore been of limited application. Methods (ii) and (iii) consist of a two-stage treatment and thus are much more versatile. In both cases the annealing treatment is employed to sharpen-up the cell or subgrain boundaries introduced by the deformation and to produce a more or less constant degree of internal stress. However, the annealing temperature is usually varied in method (ii) to produce the desired variation of subgrain size. In method (iii), on the other hand, the annealing temperature is kept constant and the range of subgrain sizes is achieved by varying the deformation temperature and the degree of prestrain.

The techniques generally used for measuring subgrain size are:

(i) X-ray methods
(ii) Etch-pit studies
(iii) Electron microscopy

**Low-Temperature Strength**

The majority of quantitative studies in this field consisted of measuring subgrain size, by a variety of techniques, and relating it to flow stress through the well-known Hall–Petch relationship

$$\sigma_f = \sigma_0 + k_s t^{-1/2} \quad \cdots (6)$$

where $\sigma_f$ is the flow stress, $\sigma_0$ the friction stress, $k_s$ a constant related to subgrain-boundary strength (i.e. stress concentration required to generate dislocations from the boundary), and $t$ is the subgrain diameter. Equation (6) was first proposed to account for the grain-size-dependence of lower yield stress in iron alloys. Since then it has been shown to be almost universally adhered to by all polycrystalline materials. In view of the similar structural characteristics of grain boundaries and subgrain boundaries, it is not surprising that the relation is equally applicable to both types.

Values of $\sigma_0$ and $k_s$ parameters for some metals and alloys are given in Table III, together with typical values of the equivalent grain-size parameters, $\alpha_l$ and $k_p$, for comparison. Some of the results were obtained from hardness (HV) measurements, which have been converted to flow stress ($\sigma_f$) using the relationship

$$\sigma_f = \frac{HV}{6} \quad \cdots (7)$$

In Fig. 21 are reproduced some of the reported results of the variation of flow stress with subgrain size for pure aluminium. It is apparent that the metal experiences appreciable hardening with decreasing subgrain size. Some workers have found that in aluminium subgrain-hardening is of the same order of magnitude as that due to grain boundaries.
<table>
<thead>
<tr>
<th>Materials and Methods</th>
<th>Subgrains</th>
<th></th>
<th></th>
<th>Grains</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ref.</td>
<td>$\sigma_y$ MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
<td>Ref.</td>
<td>$\sigma_y$, MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
</tr>
<tr>
<td>99.995% Al (iii)*</td>
<td>140</td>
<td>-1.95 (0.25)</td>
<td>0.08 (0.29)</td>
<td>155</td>
<td>15.7 (1.6)</td>
<td>0.07 (0.22)</td>
</tr>
<tr>
<td>99.998% Al (iii)</td>
<td>142</td>
<td>-29.5 (1.6)</td>
<td>0.09 (0.29)</td>
<td>78‡</td>
<td>15.5 (1.6)</td>
<td>0.11 (0.35)</td>
</tr>
<tr>
<td>99.86% Al (iv)</td>
<td>142</td>
<td>-12.75 (1.3)</td>
<td>0.09 (0.29)</td>
<td>77‡</td>
<td>-12.75 (1.3)</td>
<td>0.09 (0.29)</td>
</tr>
<tr>
<td>99.5% Al (iii)</td>
<td>142</td>
<td>6.8 (0.7)</td>
<td>0.07 (0.23)</td>
<td>156</td>
<td>15.7 (1.6)</td>
<td>0.04 (0.13)</td>
</tr>
<tr>
<td>99.5% Al + 1.0% Al₂O₃ (iii)</td>
<td>142</td>
<td>6.8 (0.7)</td>
<td>0.07 (0.23)</td>
<td>156</td>
<td>15.7 (1.6)</td>
<td>0.04 (0.13)</td>
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<tr>
<td>99.9% Al + 4.7% Al₂O₃ (iii)</td>
<td>142</td>
<td>6.8 (0.7)</td>
<td>0.07 (0.23)</td>
<td>156</td>
<td>15.7 (1.6)</td>
<td>0.04 (0.13)</td>
</tr>
<tr>
<td></td>
<td>Ref.</td>
<td>$\sigma_y$, MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
<td>Ref.</td>
<td>$\sigma_y$, MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
</tr>
<tr>
<td>Specpure Cu (iii)</td>
<td>83</td>
<td>0.0</td>
<td>0.20 (0.65)</td>
<td>157</td>
<td>2.6 (0.35)</td>
<td>0.11 (0.36)</td>
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<tr>
<td>Fe-0.03% C (iii)</td>
<td>144</td>
<td>35.3 (3.6)</td>
<td>0.34 (1.04)</td>
<td>158</td>
<td>47.04 (4.8)</td>
<td>0.71 (2.28)</td>
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<tr>
<td></td>
<td>Ref.</td>
<td>$\sigma_y$ MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
<td>Ref.</td>
<td>$\sigma_y$, MN/m² (kgf/mm²)</td>
<td>$k_y$, MN/m³/² (kgf/mm³/²)</td>
</tr>
<tr>
<td>Fe-0.04% C (i)</td>
<td>147</td>
<td>107.8 (1.1)</td>
<td>0.34 (1.45)</td>
<td>159</td>
<td>36.3 (3.7)</td>
<td>0.21 (0.66)</td>
</tr>
<tr>
<td>En 8 (Isoforming)</td>
<td>148</td>
<td>90.0 (9.6)</td>
<td>0.41 (1.31)</td>
<td>160</td>
<td>90.0 (9.2)</td>
<td>0.79 (2.50)</td>
</tr>
<tr>
<td>Drawn pearlite (0.93% C) (i)</td>
<td>147</td>
<td>41.2 (4.2)</td>
<td>0.42 (1.35)</td>
<td>160</td>
<td>90.0 (9.2)</td>
<td>0.79 (2.50)</td>
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<tr>
<td>Rolled pearlite (0.93% C) (i)</td>
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<td>41.2 (4.2)</td>
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<td>160</td>
<td>90.0 (9.2)</td>
<td>0.79 (2.50)</td>
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<td>Fe-0.02% C-0.006% N (iv)</td>
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<td>0.0</td>
<td>0.41 (1.30)</td>
<td>160</td>
<td>90.0 (9.2)</td>
<td>0.79 (2.50)</td>
</tr>
<tr>
<td>Fe-0.002% C-0.0018% N (iv)</td>
<td>73‡</td>
<td>34.3 (3.5)</td>
<td>0.27 (0.87)</td>
<td>72‡</td>
<td>0.0</td>
<td>0.31 (0.98)</td>
</tr>
<tr>
<td>Fe-0.0086-%C-0.014% N (iv)</td>
<td>72‡</td>
<td>0.0</td>
<td>0.31 (0.98)</td>
<td>72‡</td>
<td>0.0</td>
<td>0.31 (0.98)</td>
</tr>
<tr>
<td>Fe-Mn-N (iii)</td>
<td>94</td>
<td>55.8 (5.7)</td>
<td>0.17 (0.55)</td>
<td>123</td>
<td>72.5 (7.4)</td>
<td>0.21 (0.66)</td>
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<tr>
<td>Fe-0.003% C (ii)</td>
<td>123</td>
<td>72.5 (7.4)</td>
<td>0.21 (0.66)</td>
<td>151</td>
<td>33.3 (3.4)</td>
<td>0.20 (0.65)</td>
</tr>
<tr>
<td>Fe-0.004% C (i)</td>
<td>151</td>
<td>33.3 (3.4)</td>
<td>0.20 (0.65)</td>
<td>151</td>
<td>33.3 (3.4)</td>
<td>0.20 (0.65)</td>
</tr>
</tbody>
</table>

* Numbers in parentheses indicate method of producing subgrains and refer to those listed under "The Effect of Subgrain Structure on Mechanical Properties: Experimental Techniques" (p. 186).
† Converted to flow stress from hardness values by means of equation (7).
The work of Hansen, however, shows that subgrain-hardening\(^{142}\) is twice as effective as grain-boundary-hardening\(^{156}\) (Table III). This is also true in the case of pure copper,\(^{83}\) in which subgrain-hardening is more effective by a factor of \(\sim 2\) (Table III). These results clearly illustrate that fine substructure within the grains represents an appreciable contribution to the strength of these metals.

The effect of substitutional solution-hardening on subgrain-hardening has been investigated by Cotner and Tegart,\(^{78}\) using aluminium–magnesium alloys (Fig. 22). For magnesium additions of 0.3–2% they found a concentration-independent increase in \(k_s\) of \(\sim 10\%\). In the case of dispersion-hardening the \(k_s\) parameter, being of the same order of magnitude as that for pure aluminium (Table III), remains practically unchanged up to 4.7% alumina in aluminium.\(^{148}\) There is some evidence that the relative contribution of subgrain structure to strength in dispersion-hardened metals increases with increasing temperature.\(^{151}\) The variation of \(\sigma_0\) is consistent with the nature of solid-solution-\(^3\) and dispersion-hardening (Table III).

Similar observations concerning the effect of solute additions and dispersions on subgrain-strengthening have been made in Fe–Ni, Fe–Cr, and tempered Fe–Cr alloys. Qualitatively the same trends were observed as in aluminium and its alloys,\(^{78}\) though the tempered Fe–Cr alloy\(^{72}\) exhibits a significant change in \(k_s\) as compared with \(\alpha\)-iron and the solid-solution alloys.

The effect of interstitial solutes on subgrain-hardening in bcc metals has also been studied. In this case there is an additional process that can occur during post-deformation treatments, i.e. strain-ageing.\(^{96,97}\) Consequently the subgrain effects are not so well defined and it is necessary to take into consideration post-deformation heat-treatments and subsequent cooling rates. In Fig. 23 are reproduced some of the reported studies of the effect of subgrain size on the flow stress of \(\alpha\)-iron containing carbon and/or nitrogen. Comparing the results of Ball\(^{144}\) and Warrington\(^{145}\) with those of Baird and Mackenzie,\(^{94}\) which were obtained under similar conditions, it is noted that the \(k_s\) parameter is markedly different (Table III and Fig. 23). The lower \(k_s\) reported by Baird and Mackenzie was later shown to be attributable to the absence of strain-ageing.\(^{146}\) The original specimen treatments of Ball and Warrington involved slow cooling from the post-deformation annealing temperature, whereas those of Baird and Mackenzie consisted of a rapid quench. Baird\(^{146}\) showed that slow cooling had the effect of increasing \(k_s\) from the original value\(^{94}\) of 0.17 MN/m\(^{3/2}\) (0.55 kgf/mm\(^{3/2}\)) to one close to that of Ball and Warrington, namely 0.34–0.39 MN/m\(^{3/2}\) (1.04–1.23 kgf/mm\(^{3/2}\)). (This view has been confirmed by recent work\(^{142}\) on a very pure iron in which a value of \(k_s\) similar to that of Baird and Mackenzie\(^{94}\) was obtained).

The above main observations of subgrain-boundary-strengthening are in general applicable to drawn iron,\(^{147}\) copper,\(^{147}\) eutectoid steel,\(^{147}\) tempered low-carbon martensite,\(^{147}\) Fe–C–Mo steel,\(^{147}\) precipitation-hardened molybdenum,\(^{142}\) and drawn and annealed tungsten.\(^{142}\) Isoforming of an Fe 18 steel\(^{148}\) and controlled rolling of austenitic steels,\(^{149}\) in a like fashion, have led to a Hall–Petch relationship. In many of these cases\(^{147,149–151}\) elongated subgrains, characteristic of high strains, were encountered and under such conditions the transverse subgrain diameter was measured. The work on drawn iron\(^{147}\) wire has recently been repeated by Langford and Cohen,\(^{161}\) who found that their data, obtained in a similar manner to that of Embury \textit{et al.},\(^{147}\) could be better expressed by a relationship of the form

\[ \sigma = \sigma_0 + k r^{-1} \]  \( \ldots (8) \]

Careful analysis of their results\(^{151}\) seems to indicate that, while at high strains the relationship holds with the exponent equal to \(-1\), at strains \(< \sim 0.5\) the conventional Hall–Petch form is as valid.

It has been observed that during cold working of dispersion-hardened materials the subgrain structure assumes the dimensions of the interparticle spacing. This is apparent in the studies of carbon steels;\(^{162}\) internally oxidized Al–Mg,\(^{163}\) Cu–Be,\(^{104}\) Cu–Al,\(^{164}\) and Cu–Si\(^{164}\) alloys; \(\sigma_{\text{B}}\);\(^{108,142,148}\) TD nickel;\(^{105}\) precipitation-hardened molybdenum;\(^{156}\) and stainless steel,\(^{105}\) The dependence of flow-stress parameters on (particle spacing)\(^\frac{1}{4}\) in pearlitic steels\(^{162}\) and in spheroidized steels at carbon contents \(> 0.3\%)\(^{165}\) has been explained in terms of the effect of subgrain size, since this is directly related to interparticle spacing and plastic strain.\(^{162}\)

It should be pointed out that equation (6) is not the only relationship that has been obtained between flow stress and subgrain size, though it is probably the most fully authentica ted. Beck \textit{et al.}\(^{166}\) obtained a relationship of the same form as equation (6) for rolled and annealed aluminium using hardness testing, but the exponent was \(\sim 0.7\) instead of 0.5. Similar work on deformed and annealed molybdenum\(^{167,168}\) implied an inverse relationship between flow stress and subgrain size, but insufficient measurements were made to allow any quantitative conclusions to be drawn. Hultgren,\(^{169}\) working with cold-worked and annealed aluminium, found that for a given grain size the flow stress was a function of both subgrain size and sub-boundary misorientation (6), giving

\[ \sigma_i = \sigma_0 + a G 0^{-1} \]  \( \ldots (9) \]

He demonstrated that this relationship was equivalent to equation (4) for dislocation density and that the results of earlier workers\(^{170,171}\) could be explained in a similar manner.

Measurements of the properties of melt-grown single crystals revealed that the critical resolved shear stress could be
increased by as much as 50% by changing the freezing rate. This has been shown to be due to the presence of striation or interlineage boundaries consisting of arrays of dislocations, resembling those resulting from polygonization. McGrath and Craig have demonstrated that the critical shear stress of aluminium single crystals increases with increasing striation density. Fleischer, analysing their results, established that the critical shear stress was inversely proportional to the square root of the mean misorientation. Montgomery, working with aluminium, obtained a relationship similar to the square root of the mean second-order subgrain size to the power — 0.4. They also noted a relation between subgrain size and angular misorientation. Working with aluminium, Montgomery, Fleischer, and Craig, who obtained a relationship similar to equation (9), proposed by Dorn et al. is also shown in the figure (broken line).

It appears that during fatigue the subgrain or cell size is closely related to the alternating stress through a relationship of the same form as equation (10). The results so far reported indicate that the value of the exponent m is again close to unity, and that the term is close to that determined by equations (8) and (10). This is probably not surprising when it is considered that dynamic-recovery effects could have a major influence on subgrain-strengthening during fatigue, as they do in creep and hot working. A recent study of hot-worked substructures revealed that the room-temperature flow stress of hot-worked and rapidly cooled materials is governed by the relationship of equation (10), with m = 1.

The Effect of Dislocation Networks on Mechanical Properties

The effect of dislocation networks on mechanical properties is not as well documented as that of subgrains. This fact does not reflect the importance of their role in deformation and in strengthening.

Under creep conditions it has been shown that the subgrain-interior dislocation density and subgrain size are a function of the applied stress. This is a reflection of the way in which subgrain boundaries act as 'sinks' for dislocations generated from the network. As dislocations are absorbed by the boundaries, internal stresses associated with them and those of the material are reduced. Under these conditions the factor controlling deformation could be the subgrain-interior network. However there is evidence to show that not only the subgrain-interior dislocation density, but also the total dislocation density are a function of the steady-state stress. This leads to a situation that is difficult to resolve, owing to the apparent interdependence of these two qualities under steady-state creep conditions.

One set of observations might throw some light on this complicated matter, i.e. that in which stress-change creep experiments have been applied. Ishida and McLean, studying iron alloys, have provided some useful information. In work on an Fe-Mn-N alloy they showed that the flow stress was a function of attractive and repulsive dislocation intersections and jogs, each forming an integral part of the substructure. The contributions to the creep strength could be separated into the effect of Mn–N additions on the friction...
stress and recovery rate and the effect of dislocation-network density was the same as in iron.198 This provides confirmation of the recovery-creep model199-201 and illustrates its applicability to practical creep problems. Ishida196 further showed, using stress-change experiments, that the creep rate on lowering the stress was more sensitive to the dislocation density within subgrains than to the subgrain size. The implication is, therefore, that the former parameter is the important one during creep.

As regards room-temperature deformation, the contribution of cell-interior dislocation networks to the strength of materials is less well established. One of the first electron-microscopical investigations of iron28 indicated that the flow stress was directly related to the cell-interior dislocation density \( \rho_c \) through equation (4). In this connection we may ignore the major portion of work of this type,54-5,54,511-115 since in the past the emphasis was placed on substantiating equation (4) for the case of \( \rho = \rho_b \), the bulk or average dislocation density. This includes those present within cell walls and tangles. Lin and McLean202 demonstrated that an equally satisfactory relationship existed between the node spacing \( \theta \) and flow stress:

\[
\sigma = \sigma_0 + \alpha G b n^{\frac{1}{2}} \tag{12}
\]

Fig. 25 Typical dislocation structures in an Fe-0.05\% C alloy after deformation to an equivalent strain (11\%) by (a) direct tensile straining and (b) MMTT,59 \( \times 40000 \).

In nickel this relationship holds for strains of up to 12\%, when cell-formation has begun. Equations (4) and (12), being equivalent, indicate that before cell formation the flow stress is controlled by a three-dimensional dislocation network. This is reasonable, since for a random tightly linked network the following relation should hold:

\[
n^\frac{1}{2} = k \rho^{-\frac{1}{2}} \tag{13}
\]

where \( k \) is equal to unity, which was the case here. It is likely that such a relation is fairly universal, but once cellular structures form the situation is very much more complicated. A recently suggested statistical approach to dislocation-density measurements203 should permit the relative contributions of cell-wall and cell-interior dislocation densities to the flow stress to be assessed.

Multiple Mechanical–Thermal Treatments (MMTT)

Recently a certain amount of interest has been generated in the use of MMTT as a means of improving the strength and toughness of low-carbon steels.58 Basically, MMTT involves the application of strain-ageing cycles, the strain corresponding in the case of iron58 and niobium alloys204 to the Lüders strain. Cycles of straining and ageing (up to five) in the case of iron58 are in essence duplicating the conditions prevailing during dynamic strain-ageing, except that in the latter case the process is continuous as compared to its discontinuous nature during MMTT. An example of the MMTT structure after three cycles is shown in Fig. 25, together with that obtained after direct straining to the same degree. The higher dislocation density in the MMTT case is evident, as is the finer, tangled structure. A similar type of structure has been observed during the interrupted dynamic strain-ageing of carburized iron–silicon single crystals at 50°C.59

From the MMTT work113 at present being carried out by the authors on niobium-stabilized austenitic stainless steel, it
appears that an entirely different type of substructure is produced, which is unique. The MMTT schedule involves the repeated application of small prestrains (~ 2%) followed by ageing in the range 700-800°C to promote dislocation locking by NbC precipitates. The structure consists of a very uniform and fine three-dimensional dislocation network (Fig. 26) possessing high resistance to recovery. The mean dislocation density after seven such MMTT cycles is \(2 \times 10^{10} \text{cm}^{-2}\), which is considerably higher than that normally found in this material in the absence of any cellular or tangled structure.

The type of substructure produced by deformation in the blue-brITTLE region or by MMTT and also that resulting from interrupted dynamic strain-ageing can be regarded neither as a true cellular structure nor as a network. It consists of a fine distribution of tangles with an interconnecting 'cross-grid' of dislocation segments. The available evidence suggests that for this structure the flow stress obeys the conventional \(\gamma\) vs. \(\rho d\) relation (equation (4)) and that some type of additional strengthening is also occurring. Whether this is due to an inherent 'barrier-strengthening' effect or to finely dispersed precipitates is unclear at present. It might be that since no continuous cellular structure exists the strength is a function of the network density and the tangles might be thought of as relatively immobile barriers, which, like dispersions, simply contribute to the friction stress term. Failing such an explanation one must assume that the higher flow stress obtained by MMTT for any given total dislocation density is due to an inherently more efficient dislocation distribution. The structure produced by MMTT in an austenitic steel, which also exhibits the additional hardening, closely resembles the ideal model network envisaged for equation (13) and this too appears to behave in a similar manner.

**Models of Dislocation-Substructure-Strengthening Networks**

In Friedel's analysis of the contribution of Frank networks to the yield or flow stress of materials the network is considered to be fully pinned at nodes (Fig. 27(a)) with the individual dislocation segments acting as Frank-Read sources. If these dislocations leave their slip planes (Fig. 27(b)) or are pinned by impurity atoms or precipitates (Fig. 27(c)) the effective length of each segment may be reduced. The application of a shear stress \(\tau\) along the slip-plane causes dislocation segments to bow out between pinning points. Segments bow out under a stress that increases to a maximum when the loop becomes semicircular and the diameter is equal to the node spacing \((l)\). It can be shown that if the stress exceeds a critical value, given by \(\tau_c\),

\[
\tau_c \approx 2T/lB
\]  

where \(T\) is the dislocation line tension, the loop will continue to expand in the normal manner for a Frank-Read source until the source becomes inactivated either by the back stress of piled-up dislocations against barriers or by the jogging action of dislocations from other sources. It is, therefore, apparent that to account for the observed yield strength of common crystals, assuming the line tension \(T\) is given by

\[
T \approx Gb^2
\]  

a network with a node spacing of ~ 10 \(\mu\)m would be necessary. This is of the same order of magnitude as the observed dimensions of Frank networks in pure, well-annealed crystals, and corresponds to a dislocation density of \(~ 10^5 \text{cm}^{-2}\).

This does not mean that Frank-Read sources in the Frank network contribute uniquely to the flow stress. As pointed out by a number of authors, other dislocation sources exist in materials. Similarly, a number of probable dislocation-multiplication processes based on cross-slip have been proposed to explain the profuse multiplication that occurs during yielding. The failure to observe Frank-Read sources in materials other than semiconductor and ionic crystals, the general absence of pile-ups and the finite width of slip-bands in high-stacking-fault energy materials have variously been cited as reasons for supposing that these play little part in plastic deformation. However, Friedel proposed a modified Frank-Read model to account for jog formation and the propensity for cross-slip normally encountered in such circumstances. This modification counters many of these objections, particularly that concerning slip-bands.

Furthermore, such a model has been extended successfully to explain work-hardening behaviour and is the starting point for several well-established theories in this field. It can be shown that equation (4) is equivalent to that derived on the assumption that the node spacing \((n)\) and the mean mesh length \((l_n)\) are related.

\[
l_n \approx n^{-\frac{3}{2}}
\]  

Similarly, it has been proposed that the dislocation density is related to the mesh length. Thus

\[
p = m/l_m^2
\]  

where \(m\) is a geometrical constant having a value between 3 and 5, depending on dislocation arrangement. It follows, therefore, that combining equations (4), (16), and (17) gives

\[
\sigma \approx a'uGb^n \frac{1}{n} \approx a'uGb\frac{l_m}{l_n}
\]  

where \(u\) is a constant having a value between 1 and 2, which is very close to that found experimentally. A Frank network of the type just described will rarely be completely uniform, as has been assumed. Usually a wide range of mesh lengths would be observed, in the main due to recovery in a manner analogous to grain growth. It follows that distributions of the form shown schematically in Fig. 28(a) would be expected. Here the number of segments \((L)\) with individual lengths lying between \(l\) and \((l + dl)\) is plotted against the reciprocal of their length \((1/L)\) or equivalently the flow stress of the individual segments (equation (14)). One would therefore expect the flow stress of a pure metal, in the absence of all dislocation interactions, to be \(\sigma_0\), or, in other words, the flow stress of the weakest links in the network, i.e. those with the longest free length. Obviously this situation...
cannot exist, even in ultra-pure single crystals, since significant interactions are bound to occur between bowing dislocations and those in the surrounding network. This aspect has been analysed and reviewed elsewhere. It is generally accepted that a dislocation moving through the 'forest' of dislocations will experience two types of interaction—short-range and long-range.

The former are due to simple dislocation cutting and intersection processes characterized by the formation of 'trees' and attractive and repulsive junctions (Fig. 29) that lead to a temperature-dependent component of the flow stress. Saada concluded that the major factor in strengthening of this type was the formation of attractive junctions, which produce a strengthening given by

\[ \sigma_J \approx \frac{Gb_1}{l_r} \]

where \( l_r \) is the average distance between attractive 'trees' in the 'forest' and \( \beta \approx 4 \) for fcc metals. Equation (19) explains satisfactorily the low-temperature elastic limits of pure fcc metals. The latter are due to the interaction between moving dislocations and the long-range stress field of the dislocation forest and lead to a temperature-independent component of the flow stress (\( \sigma_f \)). This is inversely proportional to the mean mesh length. The network model is applicable, in these cases, though the flow stress is now given by

\[ \sigma_f \approx \frac{Gb_0}{l_h} \]
where \( \sigma_0 \) is the friction stress. The academic arguments surrounding this subject centre around the relative importance of the last three terms in equation (20).\(^5\)

It is clear from Fig. 28(a) that while a material may possess a relatively high dislocation density its strength is ultimately determined by the weakest links in the network. This is readily understood if a heavily cold-worked material with cellular structure is considered.\(^5\) In this case the density of dislocations within the cell walls is generally accepted to be \( \sim 4-5 \) times the density in the cell interior.\(^5\) The dislocation distribution is thus even more complicated, exhibiting two distinct peaks (Fig. 28(b)) corresponding to dislocation distributions within cell interiors and cell walls, respectively. However, one would expect that the flow stress would be controlled by the longest segments, namely by the cell-interior networks.\(^2\)

The cell walls, on the other hand, would not be expected to contribute to the flow stress except in respect of their effect as barriers to dislocation motion.

Subgrains

During the past decade, attempts have been made to understand the mechanical and thermal stability of subgrain boundaries.\(^3\) The approach has been to consider the behaviour of individual dislocations and groups of dislocations by analysing their stress fields.\(^3\) The problems involved have been considerable, especially regarding the highly complex situations existing in cold-worked structures.

The main prerequisite of any analysis of this type is to establish the nature, magnitude, and distribution of the stress fields of the interacting species. The stress field of a single edge dislocation in isolation has been known for many years\(^3\) and is shown schematically in Fig. 30. This is a 'contour map', representing the expression for the stress field given by

\[
\sigma_{xy} = \frac{Gb}{2\pi(1-\nu)} \ln \left( \frac{x^2 + y^2}{\pi^2} \right) \quad \ldots (21)
\]

where \( \sigma_{xy} \) is the shear stress at any point \((x, y)\) with respect to an edge dislocation at the origin. In a like manner the stress field of a group of edge dislocations comprising a simple tilt boundary has been derived\(^3\) as

\[
\sigma_{xy} = \frac{G(b_d)}{2\pi(1-\nu)} \ln \left( \frac{\pi^2}{h} \right) \quad \ldots (22)
\]

where \((x, y)\) are now the coordinates of a point in space relative to one dislocation in the wall, taken as the origin, and \(h\) is the dislocation spacing. The stress field may be represented diagrammatically as before and is shown in Fig. 31. Comparing these two cases it is clear that while a single edge dislocation possesses a large short- and long-range components of the stress field, the latter is noticeably absent in the case of tilt boundaries. In fact the stress field diminishes to zero at a distance from the wall equal to the spacing of dislocations in it. The force exerted by such dislocation walls on a gliding dislocation has been discussed briefly by Nabarro\(^4\) and Cottrell.\(^5\) A more complete and quantitative analysis has been carried out by Li,\(^6\) who extended Nabarro and Cottrell's ideas to pinned and partially pinned walls,\(^7\) as well as to more complicated configurations such as twist\(^8\) and and mixed dislocation\(^8\) boundaries.

A dislocation gliding on a slip-plane perpendicular to the boundary experiences a force that is alternately attractive and repulsive, depending on its position relative to dislocations in the wall (Fig. 32). The force exerted on the dislocation varies with the distance \(x\) from the wall and the position \(y\) of the slip-plane intersecting the wall, which is a function of the separation of dislocations \(h\) in the wall. It is apparent from the above that the minimum force \(\sigma_{B(min)}\) is given by

\[
\sigma_{B(min)} = \frac{0.45 \frac{Gb}{h}}{2(1-\nu)} \quad \ldots (23)
\]

when the slip-plane is at a position \(t = h/2\) or midway between dislocations in the wall. In this instance the force would be attractive, and at stresses lower than \(\sigma_{B(min)}\) capture of the dislocation by the wall should occur. This applies to dislocations of like sign and Burgers vector, since for those of opposite sign the stress field is reversed. Therefore, in the latter case a dislocation travelling along \(y = h/2\) would experience the same force, but in the opposite sense (repulsion). Similarly, such a dislocation moving along \(y = 0\) would experience an attractive force, resulting probably in annihilation. \(\sigma_{B(min)}\) may be obtained in a more useful form by substituting for \(h\) from equation (2), giving

\[
\sigma_{B(min)} = \frac{0.45 \frac{Gb}{(1-\nu)}}{2} \quad \ldots (24)
\]

Similarities between the general behaviour of grain and subgrain boundaries and the application of the Hall-Petch relationship (equation (4)) to describe their effect on mechanical properties have led to analogous reasoning in the derivation of theoretical models to account for the effect of subgrains on mechanical properties. Eshelby et al.,\(^9\) using a pile-up model, derived a relationship between the grain-boundary 'rupture' strength \(\sigma_g\) and the \(k_p\) parameter

\[
k_p = \left[ \frac{(\sigma_g \frac{Gb}{h})}{\pi(l_p)} \right]^{-\frac{1}{2}} \quad \ldots (25)
\]

where \(d\) is the grain diameter and \(l_p\) the average length of pile-ups. Cottrell\(^1\) still using a pile-up model, introduced the concept of unpinning dislocation sources and derived the relationship

\[
k_p = \sigma_p l_i \quad \ldots (26)
\]

where \(l_i\) is the mean distance of dislocation sources in one grain from the head of a pile-up in another and \(\sigma_p\) the unpinning stress. Other explanations have been proposed, based on models involving grain-boundary dislocation sources\(^10\) instead of pile-ups. These indicate that the total dislocation density is a function of grain size\(^10\) and 'geometrically necessary dislocations' required to maintain strain compatibility between deformed grains.\(^10\)

\(\sigma_g\) obtained from the theoretical analysis due to Li\(^6\) can be used to test the applicability of the relationship of equation (24) to subgrain structures. The \(k_p\) parameter for subgrains \((k_s)\) can be calculated by means of the model due to Eshelby et al.,\(^9\) assuming that for a pinned or partially pinned sub-
boundary the stress \( G_{B_{(m+n)}} \) required to move an edge dislocation through the wall is the limiting factor. Thus

\[
k_s = \frac{G_b}{2\pi(1 - \nu)} \begin{pmatrix} \frac{2(0.45 t_0)}{f_{ps}} \end{pmatrix}^{\frac{1}{2}} \ldots (27)
\]

where \( t \) is the subgrain size, \( \theta \) the angular misorientation, and \( f_{ps} \) the size of pile-up in the subgrain. This should produce a minimum value for \( k_s \), since if the dislocations in the pile-up lie at an angle other than 90° and parallel to dislocations in the wall, jog or junction formation would be likely to occur. In this event the sub-boundary would act as a network and equation (12) would be more applicable.

Pile-ups appear to be absent in most materials, therefore other processes should be considered as possible explanations of the subgrain effect on mechanical properties. When \( \theta \) is small, free dislocations may be moved leaving the pinned dislocations behind. This requires a stress \( \sigma_{pu} \) given by

\[
\sigma_{pu} = 0.2 G \theta/N(1 - \nu) \ldots (28)
\]

where \( N \) is the ratio of free dislocations to pinned dislocations in the wall. Stroh's calculations \( \ldots \) indicate that when \( \theta \) exceeds a critical value pinned dislocations are also capable of being torn free, requiring a stress \( \sigma_{pu}/N \) (Equation (27)). Lastly, there is the case of all dislocations in the boundary being free or readily unpinned, when an applied stress of \( 2G b/\pi t(1 - \nu) \) would be required to achieve this. \( \ldots \) Under such conditions, to maintain yielding a stress sufficient to force dislocations through the 'forest' formed as a result of the decomposition of the boundaries must be applied. The density of free or weakly pinned dislocations in sub-boundaries is given by

\[
\rho = 8 N \theta/(N + 1) b t \ldots (29)
\]

If, however, all the dislocations are unpinned, complete disintegration of the boundary can occur and the 'forest' dislocation density, disregarding the subgrain-interior dislocations, will be

\[
\rho = 8 \theta/b t \ldots (30)
\]

as is likely to be the case in well-annealed materials. Substitution of equation (30) into equation (4) yields a Hall-Petch relationship with

\[
k_s = \frac{G_b}{2\pi(1 - \nu)} \left( \frac{8 \theta}{\nu b} \right)^{\frac{1}{2}} \ldots (31)
\]

It is interesting to speculate that if the stress given by substituting equation (30) into (4) is lower than either of those required for unpinning \( \sigma_{pu} \) or \( \sigma_{ps} \), the flow stress immediately following unpinning would be a function of the forest density, giving

\[
\sigma_f = \sigma_0 + \frac{G_b}{2\pi(1 - \nu)} \left( \frac{8 \theta}{\nu b} \right)^{\frac{1}{2}} t^{\frac{1}{2}} \ldots (32)
\]

and a yield point would accompany the dislocation-generation process.

The equivalence of the pile-up and 'forest' models for a simple tilt boundary has been indicated. \( \ldots \) Values of \( k_s \) from equations (27) and (31) are \( \ldots \) and \( \ldots \), respectively. It has been pointed out by Li \( \ldots \) that if \( \rho_{ps} \) in Cottrell's analysis \( \ldots \) (equation (26)) is taken as the spacing of dislocations in the wall a relationship for \( \sigma_f \) (0.51 \( G b \theta/0(1 - \nu) \)) is obtained, which is similar to that for \( \sigma_{ps} \) in equation (24). Conrad \( \ldots \) has proposed, on the basis of stored-energy measurements and electron microscopy, that the Hall-Petch relationship is a consequence of slip-distances being restricted to the subgrain size. It has been shown that the pile-up and 'forest' models, together with the Cottrell model, yield relationships of the Hall-Petch form and that each leads to a similar value of \( k_s \). This is the present state of the theoretical understanding of the phenomenon, with the parallel situation for grain-boundary- and sub-boundary-strengthening being apparent.

Most of the foregoing discussion is mainly applicable to simple tilt boundaries. Twist and mixed dislocation boundaries, which are less sensitive to simple stress systems, represent a more complicated case. Calculations \( \ldots \) of the force necessary to displace a dislocation from a twist boundary yield a value of \( \sim G b/2h \). Similarly, the stress \( \sigma \) necessary to disintegrate a twist boundary completely has been derived \( \ldots \) as

\[
\sigma_D = G b (A - \ln \theta)/2\pi R \ldots (33)
\]

where \( A \) is a constant and \( R \) the radius of curvature of a bowing sub-boundary. These are about an order of magnitude higher than the equivalent stress for a pure tilt boundary. Since solute atoms and point defects do not interact with twist boundaries to the same extent as they do with tilt boundaries, \( \ldots \) the difference in mechanical strength between them is likely to become less with increasing levels of impurities. Therefore, for most materials studied, the analysis for pure edge-dislocation walls will suffice.

A different approach has been followed by Holt \( \ldots \) in an effort to explain the way in which dislocation substructure affects the flow stress under conditions where dynamic recovery takes place during deformation, e.g. in creep and hot working. An idealized dislocation distribution was considered, in which no annihilation of dislocations was to occur. The analysis was based on a process reminiscent of that operating in spinodal decomposition. \( \ldots \) By analogy with solute atoms, dislocations cluster together to minimize their elastic energy (cf. polygonization). Several conditions are imposed on the dislocation distribution, the main one being the introduction of a 'gradient-energy' term. From the analysis it transpires that the uniform distribution is unstable and that dislocations move to form a structure possessing a modulated dislocation density. This 'modulated structure' is ultimately equivalent to the subgrain structure and the dominant wavelength of the density modulation is equivalent to the subgrain size. Thus, the wavelength \( \lambda_m \) is inversely proportional to the square root of the mean dislocation density:

\[
t \approx \lambda_m \approx K \rho = r_e \ldots (34)
\]

where \( K \) is a constant and \( r_e \) the dislocation/dislocation interaction distance. \( r_e \) implies that subgrain boundaries will be spaced, at a given stress level, so that the effect of dislocations in adjacent walls is minimal and a function of the nearest-neighbour interactions within a wall. From equation (4) it therefore follows that

\[
t \approx a K G b \ldots (35)
\]
assuming that the friction-stress term is negligible. Inherent in this model is the assumption that dislocation mobilities are high enough for the large-scale rearrangements to occur. Under this condition the main assumption, that no dislocation annihilation takes place during clustering, breaks down. However, under conditions where steady-state deformation is observed, such as in creep and hot working, the constant average dislocation density required by this model is maintained by the opposing processes of work-hardening and recovery. It is, therefore, not surprising that in these circumstances the relationship of equation (35) generally holds.72-74

Substantially similar conclusions arise from Kuhlmann-Wilsdorf's theory of work-hardening for Stages II and III in single crystals.213,214 A network model213,214 similar to that proposed earlier was expanded234 to explain the observations of Langford and Cohen.151 In this model dislocation sources lie in the coarsest regions of cell walls and dislocation mean free paths are assumed to be restricted to the cell diameter. This leads to the relationship of equation (10), with \( m = 1 \). The model resembles, in principle, that proposed by Langford and Cohen151 to explain their results and shows good agreement not only for \( k \) and \( m \) but also for values of angular misorientations.234 In the case of creep and hot working, these models151,234 are probably less applicable than that due to Holt.232

The observation195 that the room-temperature flow stress of hot-worked materials obeys the relationship of equation (10) is consistent with the forest model,234 owing to the effect of total dislocation density. If the hot-worked substructure is 'frozen-in' by rapid cooling, the low-temperature flow stress might be expected to be closely related to subgrain size through the dislocation density, as in equations (34) and (35).

Discussion

Networks

The simple model of the three-dimensional dislocation network illustrated in Fig. 29 provides a reasonable explanation of the effect of low dislocation densities (\(< 10^{10} \text{ cm}^{-2}\)) on the mechanical properties of metals. This is especially true in the case of polycrystals where a large amount of averaging occurs over slip systems, making the more specific single-crystal work-hardening theories4,5 generally inapplicable. Under these circumstances the experimental and theoretical equivalence of the mean mesh or segment length and dislocation-density parameters is established. Closer examination of data in this connection often shows apparently anomalous behaviour. For example, in a number of investigations negative intercepts of the flow stress (\( \sigma_0 \)) are observed when equation (4) is applied.55,58,115 This is because the mean dislocation density is a misleading parameter, since it is the weakest links in the dislocation distribution (Fig. 28(a)) which determine the flow stress. At high strains, on the other hand, where dislocation cell structures predominate (Fig. 28(b)), it appears that the mean dislocation density or mesh length remains a meaningful parameter. This implies that in direct tensile straining cell walls formed during deformation do not significantly affect either the flow stress or work-hardening characteristics.230 In more complex deformation such as rolling, where large shape changes are involved, the cell size can become an important parameter.234 Under these conditions, significant changes occur in the dislocation-segment distribution. The work of Langford and Cohen151 could, therefore, be interpreted on the basis of the network model by assuming that at the large strains achieved by wire drawing the length of the weakest segments became closely related to the cell size.234

The simple model of network strengthening shows merit, not only by its qualitative agreement with experimental facts but also by the way in which it highlights the possible routes to the production of higher-strength materials. The drawback concerning mean dislocation-density measurements, and the tendency to underestimate the friction stress, illustrate the 'negative' effect of the dislocation distribution. A 'positive' approach to strengthening can be argued in an analogous manner. Since for any given dislocation density the weakest links in the network control the flow stress by their effect on \( \sigma_i \). In equation (20), the creation of a more uniform or narrower mesh-length distribution could lead to a significant improvement in flow stress. In this case it is assumed that the stress (\( \sigma_i \)) required to generate dislocations from the network by the Frank-Read mechanism is an important factor. If this is not so, and the flow stress is related only to the stress (\( \sigma_0 + \sigma_j + \sigma_k \)) required to force dislocations through the network, such an argument does not apply, as pointed out for cell structures.230 The relative importance of the parameters \( \sigma_1, \sigma_2, \text{and} \sigma_i \) has, over the last few years, been extensively discussed5,111 and still remains debatable.5 The situation is further complicated by the fact that on applying a stress the dislocation distribution is changed, so that some parts of the network become favoured as dislocation-generation sources, particularly in the presence of attractive and repulsive interactions. This being so, it is reasonable to suppose that the \( \sigma_i \) and \( \sigma_j \) terms, which are most closely related to dislocation distribution, are of major importance, and that the long-range stress of the network (\( \sigma_k \)) merely complements them by producing a 'back-stress' on bowing dislocation segments. Thus, at relatively low dislocation densities (\( 10^8-10^{10} \text{ cm}^{-2}\)) dislocation distribution may be considered to be the most important factor controlling the level of flow stress.

The observation58 that the flow stress of iron for a given dislocation density is higher by \( 78-5 \text{ MN/m}^2 \) (8 kgf/mm²) after MMTT, as compared with direct tensile straining, is cited58 as experimental evidence that dislocation distribution is affecting the flow stress. The MMTT substructure consists of relatively short (\( \approx 0.2 \mu\text{m} \)), uniform mesh lengths, while the directly strained structure is cellular with a mesh of \( \approx 0.5 \mu\text{m} \). However, it is possible that the improved strength is due to formation of fine precipitates on dislocation tangles.

In the presence of precipitates, dislocation networks tend to attain a critical mesh length, depending on the particle size and volume fraction of the precipitates. This can be calculated using a modified form235 of the grain-coarsening model due to Zener.236 Once the critical state is reached, any coarsening of the network is controlled by the rate of particle coarsening. The latter is calculable using the Lifshitz–Wagner relationship.237 This calculation, as applied to austenitic steels, showed that the rate of coarsening of dislocation networks was in good agreement with the rate of coarsening of niobium carbide particles.115 Such substructures (Fig. 26) normally have good thermal-recovery-resistance, and, therefore, should be beneficial in creep.233 This is particularly so if a relatively high dislocation density can be maintained, which appears to be possible by applying MMTT.5,8,235

Dynamic Recovery

The mechanical behaviour of metals under conditions of dynamic recovery shows reasonable agreement in terms of both theoretical models151,233,234 and experimental observa-
tions.\textsuperscript{[72–74,84,151,181–187,192–194]} The model due to Holt\textsuperscript{225} leads to an understanding of the dual dependence of subgrain size and subgrain-interior dislocation density on the applied stress during creep. This is so because of the tendency of a continuous distribution of dislocations to assume a modulated structure.\textsuperscript{225} Reppich\textsuperscript{228} has provided experimental proof of a relationship between subgrain size and total dislocation density in Mg-doped LiF single crystals. These were compressed in creep well into the steady-state region over a wide range of temperatures and stresses. The relationship is of the same form as equation (35), with $K = 10$.

Ishida's\textsuperscript{196} observations that during stress-change experiments in an Fe–Mn alloy the dislocation density alters rapidly and achieves a 'steady-state' value by the commencement of a minimum creep rate, and that the changes in subgrain size lag behind it, are inconsistent with this model. This behaviour is, however, expected in the presence of a friction stress. Langford and Cohen's work\textsuperscript{151} on wire-drawn iron and observations in fatigue are also consistent with the models based either on dynamic recovery\textsuperscript{225} or Kuhlmann-Wilsdorf's theory.\textsuperscript{234}

A factor that is not well understood, but that is of importance in the field of substructure-strengthening, is the effect of substructures produced by prior MTT on, for example, creep behaviour. The implication from work on materials, such as TD nickel, is that working and annealing treatments\textsuperscript{235} before creep testing produce finely dispersed substructures that significantly improve creep strength. The exact role of dislocation substructure and recovery-resistance are, however, unclear, though, in terms of the recovery-creep model\textsuperscript{96,66} significant improvements in creep-resistance are understandable.

**Perfect or Idealized Subgrains**

Relatively simple polygonized and striation substructures, which tend to conform fairly well with the idealized forms of sub-boundaries described earlier (see 'Low-Angle Boundaries') show mechanical behaviour consistent with the theoretical models\textsuperscript{229} for low-temperature flow stress. There appears to be the predicted dual dependence of flow stress on both the subgrain size and the misorientation angle. Indeed, the work of Hultgren\textsuperscript{166} and Lake and Craig\textsuperscript{180} leads to a result closely resembling equation (32). Such cases are, however, in a minority and most materials with less-perfect substructures appear not to conform to this type of relationship (equation (32)). They yield, instead, one of the more familiar Hall-Petch-type relationships, with no apparent dependence on angular misorientation.\textsuperscript{83,94,123,140–150}

**The Hall–Petch Relationship**

The Hall–Petch formulations for subgrains are generally consistent with those for the grain-size analysis. Substitutional solutes\textsuperscript{78,79} and dispersed phases\textsuperscript{42} have little influence on $k_y$ but significant effects on $\sigma_0$, the friction stress (Table III). In these cases the $k_y$ parameter seems in general to have values close to the $k_y$ parameter for grain boundaries. It therefore appears that, within the experimental scatter, subgrain and grain boundaries provide equally efficient barriers to deformation. The values quoted by Hansen\textsuperscript{116} for $k_y$ seem to contradict this statement. However, the agreement for $\sigma_0$ is good and hence these results might be considered questionable in the light of recent work on substructure effects.\textsuperscript{240,241} It has been shown\textsuperscript{240,241} that $k_y$ values decrease during plastic deformation as a result of the increasing role of cells and tangles as barriers. The nature of pretreatments and the presence of impurities and dispersions markedly affect both the annealed dislocation density and the subgrain size. This would, therefore, be expected to change the measured values of $K_y$ significantly, especially since such effects will be greater at small grain sizes.

In the presence of elements that exhibit strong dislocation/solute interactions\textsuperscript{94,123,144–146,147,151} the situation is more complicated, as may be seen from Table III. It appears that in pure iron\textsuperscript{123} samples (or impure samples cooled rapidly from high temperatures\textsuperscript{94}), in which no or very little strain-ageing occurs, the $k_y$ values are consistent with those obtained for aluminium,\textsuperscript{140–146} being of the same order as $k_y$ for grain boundaries under similar conditions.\textsuperscript{156} In the case of a strong strain-ageing effect the $k_y$ value increases\textsuperscript{94,144–146,147} in much the same manner as does $k_y$. However, it does not appear to attain as high a value as the $k_y$ parameter. Its value is $\sim 0.5 k_y$, implying that subgrain boundaries have an inferior strengthening effect when strain-ageing processes occur.

**Solute Segregation**

Li\textsuperscript{225} extended his analysis of dislocation structures to include the effects of impurity segregation and showed that the degree of solute segregation to a simple tilt boundary decreased as the long-range stress field was removed during polygonization. Therefore, the degree of pinning of individual dislocations in the subgrain-interior network is generally greater than for those in subgrain boundaries. Consequently, the tendency for subgrain boundaries to disintegrate during yielding would be greater in the presence of solute segregation. This view is supported by electron-microscopical observations of large numbers of apparently disintegrated subgrain boundaries in annealed molybdenum after only 1% strain. The observed discrepancy between $k_y$ and $k_y$ parameters in iron could probably be accounted for, in part, by the tendency of sub-boundaries to disintegrate during yielding.

The degree of pinning of a subgrain boundary should decrease continuously with increasing misorientation angle or decreasing separation of dislocations in it.\textsuperscript{235} On the other hand, the intrinsic strength of such a wall, if it is of the simple tilt type, increases according to equation (31), i.e. with increasing angular misorientation.\textsuperscript{224} It therefore follows that the effective strength in the presence of solute segregation rapidly decreases to a minimum with increasing misorientation.\textsuperscript{225} Thereafter, the strength is relatively insensitive to this parameter, as shown in Fig. 33. Such an effect has been
reported by Berghezan et al.,248 who observed that the degree of segregation to sub-boundaries in niobium consisting of hexagonal networks decreased as the network spacing increased. Owing to segregation effects, the $k_s$ parameter would be relatively insensitive to misorientation, since most boundaries lie, with respect to strength, in the region of the minimum in Fig. 33.225

Redundant Dislocations

Redundant dislocations in subgrain boundaries are those that, owing to their mutual cancellation over a given length of boundary, do not contribute to the misorientation angle. These dislocations arise because insufficient time or too-low annealing temperatures were used in the production of the substructure. This is probably connected with solute segregation to dislocations. The presence of redundant dislocations modifies the net long-range stress field of sub-boundaries and, in turn, contributes to the strength and work-hardening characteristics of the material. As a consequence, such boundaries should contribute more to the flow stress and the presence of redundant dislocations accounts, at least in part, for the insensitivity to misorientation angle.

The two factors, i.e. solute segregation and redundant dislocations, taken together provide a reasonable explanation for the insensitivity of the Hall-Petch relation to misorientation.

General Comments

Recently a semi-empirical approach has been adopted by Abson and Jonas154 for hot-worked and cold-worked and annealed substructures. Data accumulated from various sources for aluminium, iron, and silicon-iron have been reassessed in terms of a slightly modified Hall-Petch analysis. To obtain a better and more consistent correlation, $\sigma_0$ was calculated from single-crystal data using the Taylor factor ($M = 3.06$), so that the friction stress was always positive. Nevertheless, by plotting ($\sigma - M \tau_0$) against $t^{-\frac{1}{2}}$, where $\tau_0$ is the single-crystal yield stress (which yielded a fairly good straight-line fit) a negative intercept was obtained. This is not too surprising considering the frequency of negative $\sigma_0$ terms obtained in experimental determinations. By plotting against $t^{-\frac{1}{2}}$ no better fit was obtained, but a zero intercept resulted. This plot yielded a modified Hall-Petch relationship of the form

$$\sigma = M \tau_0 + k_s t^{-\frac{1}{2}} \quad \ldots (36)$$

which was explained in the following manner. From Li’s228 analysis $k_s$ is a function of the misorientation angle, which in turn is related to subgrain size. It therefore follows that

$$k_s = k_1 t^{-p} \quad \ldots (37)$$

where $p$ is an exponent lying between $\frac{1}{2}$ and $\frac{1}{3}$. This produces a new equation:

$$\sigma = M \tau_0 + k_s t^{-(p+4)} \quad \ldots (38)$$

where $k_1$ is now a true constant and equations (36) and (38) are assumed to be equivalent, $p$ being equal to $\frac{1}{4}$. While this approach yields a reasonable fit to the experimental data, the necessity for such complicated reasoning is questionable.

Measurements of the misorientation angle ($\theta$) across subgrain boundaries in hot-worked substructures give values ranging from 1 to 9°, so that a variation of $k_s$ with $\theta$ is unlikely. This is so because, if the ‘forest’ mechanism predominates, subgrains with misorientations $< \sim 3^\circ$ probably account for most of the deformation-resistance due to sub-boundaries. Furthermore, the effect of both segregation and redundant dislocations on $k_s$ leads to the conclusion that the flow stress is misorientation-independent. This is borne out by the work of Warrington83 on copper, which showed that levels of dynamic recovery, and hence redundant-dislocation density, were very sensitive to deformation temperature. Likewise, there is no reason why $\tau_0$ should be taken instead of the $\sigma_0$ values determined experimentally. A negative intercept probably results from slight differences in dislocation distribution. In many cases subgrain-interior dislocation densities are greater at fine subgrain sizes,83 thus producing a higher $k_s$ parameter and a negative intercept.

Finally, returning to the question of $k_s$ values in iron alloys and the effect of interstitials, the explanation may lie in the relative effects of networks and sub-boundaries on segregation, as mentioned earlier. The $k_y$ parameter for grains is generally measured under conditions where the grain-interior network is simple and strong locking always occurs. However, strain-ageing studies suggest that $k_y$ decreases as the structure becomes more complicated.241-243 Therefore, with the introduction of subgrain boundaries an additional factor has to be considered, namely the efficiency with which these are locked. Because of the mutual reduction of long-range stress fields of dislocations in subgrain walls, the rate of segregation of pinning species is lower. Thus, the sub-boundaries, being only weakly pinned, provide less strengthening than grain boundaries. It is, however, still difficult to explain the high $k_s$ values reported by Embury et al.147 for cold-worked substructures, especially in the light of more recent work by Langford and Cohen153 on a practically identical commercially pure iron. The difference may lie in the treatment of the material before working. Embury et al. used furnace-cooling following an 800°C heat-treatment, while Langford and Cohen used very slow cooling from a 650°C heat-treatment. The latter treatment allowed most of the carbon to be removed from solution, thus permitting dynamic recovery to occur more rapidly. This implies that the relatively high $k_s$ of Embury et al. was due to higher densities of redundant dislocations within cell walls.

Summary

The general mechanical behaviour of materials can be explained in terms of a relatively simple dislocation model.

All dislocation substructures can be described in terms of a simple three-dimensional network consisting of nodes and attractive and repulsive intersections. The three-dimensional network may exist in its own right, but is generally associated with a more widely dispersed network of two-dimensional or planar arrays of dislocations in the form of a cellular or subgrain structure. In this case the complexity of dislocation arrangement can vary widely. The strength of materials can, therefore, be expressed as the sum of two terms, referred to as $\sigma_{net}$ and $\sigma_{sub}$. The $\sigma_{net}$ term is crudely defined as the resistance of the lattice to dislocation motion or friction stress and is a function of crystallography, composition, and constitution of the metal or alloy. The $\sigma_{sub}$ or substructure-strengthening term is a function of both the total dislocation density and the dislocation arrangement.

A generally applicable relationship exists between flow stress and dislocation density under most working conditions (equation (4)). However, the effect of subgrains is more complicated. In hot-working conditions or those where dynamic recovery is appreciable, a simple inverse relationship holds
Potential of Substructure-Strengthening

Attempts to rationalize the various observations concerning substructure-strengthening have been relatively successful, though a clear understanding of the precise role and interaction of the dislocation density and subgrain size in strengthening is still lacking. Assuming that data for low-temperature strength and subgrain size can be normalized with the aid of a factor \( G \), useful information can be obtained regarding the potential of substructure-strengthening. This is a first-order approximation of equations (32)-(38). The results of such normalization are shown in Fig. 34. It is clear that most materials lie in a narrow band when \( \Delta \sigma / G b \) is plotted against subgrain size \( d \). \( \Delta \sigma \) is equal to \( (\sigma - \sigma_0) \) and the gradient to \( -0.5 \). The data for iron alloys in which strain-ageing occurs are consistent with this analysis. From the master curve of Fig. 34 and that for dislocation density (Fig. 35), it is possible to obtain useful information concerning the likely level of flow stress. However, at high temperatures it is difficult to correlate the dislocation density and dislocation distribution with any parameter except under conditions of steady-state substructure. The possibility of predicting the effect of preformed substructure on creep properties therefore remains uncertain. The value of \( \sigma_y G / b \) so derived is a measure of the \( \sigma_{eq} \) term but, by making appropriate assumptions, the effect of metallurgical variables may be obtained from relationships for solution-hardening and precipitation-hardening.\(^{106}\) Thus, knowing the basic parameters it should be possible to calculate, with a fair degree of accuracy, the possible strength of a material.

A strength of 0.01 G, selected as a desirable level at which to aim, corresponds to a flow stress of 380 MN/m\(^2\) (84.7 kgf/mm\(^2\)), 455 MN/m\(^2\) (46.4 kgf/mm\(^2\)), and 267 MN/m\(^2\) (27.2 kgf/mm\(^2\)) for iron, copper, and aluminium, respectively. From Figs. 34 and 35 it is apparent that such a level of strengthening can be achieved by dislocation substructures alone at a dislocation density of \( \sim 10^{13} \) cm\(^-2\) or a subgrain size of \( \sim 0.1 \) \( \mu m \). For good high-temperature strength the prime consideration is that the structure possesses good recovery-resistance.

Low-Temperature Mechanical-Thermal Treatments (LMTT)

In the discussion of substructure-strengthening several methods of producing subgrain structures were introduced. Two of these (cold working; cold and warm working and annealing), known as low-temperature mechanical-thermal treatments (LMTT),\(^{78,244}\) have been used in practice\(^1\),\(^8\),\(^127-138\),\(^244\) and their effect on strengthening assessed. While improvements in low-temperature strength are in fair agreement with the Hall-Petch formulation, vast discrepancies exist in the case of creep behaviour.\(^{127-133}\) The precise level of improvements in creep strength, life, and ductility is unclear in the absence of long-term test data. Some recent results indicate that the ductility both at low temperatures\(^{115,244,345}\) and in creep\(^246\) is adversely affected by cracking originating at particles and interfaces during the prestraining stage.

To obtain the desired level of low temperature and creep strength, means must be found to produce fine subgrains with reasonable recovery-resistance. These requirements can be met if advantage is taken of dispersion-strengthening. The tendency during deformation of cell or subgrain structures to assume dimensions closely related to the particle distribution has been demonstrated.\(^{106-108}\) It has been shown that by applying LMTT to dispersion-hardened alloys subgrain sizes approaching 0.1 \( \mu m \) can be obtained.\(^{359}\) Thermal stability of the subgrain structure up to \( \sim 0.7 \) \( T_m \) is to be expected, in most cases, owing to the resistance of dispersoids to coarsening and chemical reaction with the matrix.\(^{359}\) Furthermore the substructure-strengthening is complemented by the relatively powerful dispersion-strengthening, at least at ambient temperatures.\(^{161}\)

A combination of fine, inert dispersion and some form of LMTT, to produce a fine, uniform subgrain structure, is one of the more promising applications of substructure-strengthening. Isoforming,\(^{143}\) controlled rolling,\(^{149,160}\) and possible variations of these processes may also be included. In such cases warm or hot deformation is applied during a phase transformation or precipitation process. The early stages of deformation assist in the production of fine and more uniformly distributed precipitates that, during subsequent deformation stages, behave like normal dispersions.\(^{148}\) For low-temperature applications most forms of incoherent precipitates could be used. However, for higher temperatures at
which thermal stability is important refractory carbides are
more suitable.

High-Temperature Mechanical-Thermal Treatments (HMTT)

Mechanical-thermal treatments based on hot working have been suggested by a number of workers. In the case of solution-treated stainless steels and Nimonic-type alloys, they comprise working at 1000-1100°C by rolling, quenching to suppress recrystallization, and subsequently ageing at 750-850°C. Such schedules are known as high-temperature mechanical-thermal treatments (HMTT). The hot working produces a relatively coarse subgrain structure (~5-20 μm), which is subsequently stabilized by precipitates during ageing. HMTT resulted in significant improvements in both low- and high-temperature strength of stainless steels, Nimonic alloys, and even nickel. These improvements were accompanied by relatively large gains in ductility (up to 50%) and increased creep-rupture life (by ~200%) at 600-700°C. HMTT appears to induce appreciable substructure-strengthening and should be studied further, particularly with a view to high-temperature applications.

High-Rate and Low-Temperature Deformation

Attempts have been made to utilize the complementary nature of high-strain-rate and low-temperature deformation for the purpose of substructure-strengthening. Shock loading of nickel and sub-zero deformation of copper produced cell structures with dimensions close to 0.1 μm and the accompanying increase in strength. However, under both conditions most bcc metals undergo twinning during deformation. The mechanical stability of such substructures is good at low temperatures, but at elevated temperatures recrystallization occurs easily. It has, nevertheless, been suggested that substructures can be stabilized by heating slowly through the low-temperature (up to ~0.3 Tm) spectrum of point-defect recovery processes, resulting in higher strengths. However, both low- and high-temperature ductilities were very poor. This approach has, therefore, extremely limited applicability as a substructure-strengthening treatment.

Multiple Mechanical-Thermal Treatment (MMTT)

MMTT involves the repeated application of cycles of deformation and annealing or ageing. It is not confined only to low-carbon (strain-age-hardenable) and austenitic (precipitation-hardenable) steels, but can equally be applied to dispersion-hardened alloys where LMTT is applied a number of times. MMTT, because of its flexibility, is probably the most versatile of the mechanical-thermal treatments so far proposed.

A considerable amount of interest has been shown in MMTT since it was originally proposed by Oding et al. Improvements in strength > 30%, as compared with fully work-hardened material, have been achieved in low-carbon steels while maintaining reasonable ductility (3-5%), in creep properties at 400°C and in fatigue life and endurance limit of ~400, which have been reported. MMTT is therefore potentially a very promising method of producing high-strength materials through substructure-strengthening, which can be supplemented by conventional strengthening methods that further stabilize the MMTT substructure. The process becomes even more versatile when applied to materials such as austenitic steels, in which varying the amount of prestrain, ageing time, and temperature allows widely different forms of substructure to be produced. These can range from the network structure (Fig. 26) at low strains to complex cellular structures (Fig. 36) at high strains.

Programmed Hardening

Recently a treatment often referred to as programmed hardening has been proposed as a means of improving the properties of materials. It is based on the philosophy inherent in the network model, namely that the distribution of dislocation-segment lengths is of major significance. The treatment therefore employs a stepped loading schedule in which, at each step, the load is maintained for a time to allow the dislocation structure to attain a more stable configuration. On increasing the load the weakest links in the network are broken and these microvolumes refined. In this manner more desirable properties associated with a uniform dislocation distribution are obtained. The result is that the 'weakest microvolumes' of material are strengthened and a more uniform distribution of internal stresses obtained. Unfortunately, no structural evidence for this type of effect is available, except in the case of MMTT, which is considered to be a somewhat similar process.

Concluding Remarks

It is apparent from the work reviewed in this paper that we are far from a true understanding of the complex interrelation between dislocation structure and mechanical behaviour. However, using a rather simple model of a three-dimensional dislocation network, on to which a subgrain structure is superimposed, a reasonable qualitative explanation of mechanical behaviour can be obtained. Further research is required to elucidate the true nature of the relationship between the two basic types of dislocation distributions. It is
clear from a large volume of experimental work that flow stress is a function of both dislocation density and subgrain size, and that the strength of materials can be considered as the sum of a substructure term and a term accounting for the effect of metallurgical variables. It is, therefore, possible to enhance the strength of a material by the introduction, under controlled conditions, of a dislocation substructure. This approach is known as mechanical—thermal treatment (MTT) and can be carried out in a number of ways. But there are only two possible courses available. One is the production of small, well-pinned subgrains; the other is the production of dense, uniform dislocation networks.

Frequently, dislocations are considered as undesirable lattice defects drastically reducing the potential strength inherent in most materials. But their presence is essential in fabrication. The traditional approach has, therefore, been to obtruct their mobility with (often) expensive alloying additions and heat-treatments, while little attention has been paid to the potential benefits of MTT schedules that could be conveniently incorporated into existing fabrication and heat-treatment procedures with relatively small increases in fabrication costs. Here this alternative, though complementary, approach has been reviewed. Substructure-strengthening and MTT may be summarized by saying that, to realize more closely the potential strength of materials, we must 'set a thief to catch a thief'.

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References

23. C. G. Darwin, Phil. Mag., 1921, 24, 1413.
24. C. G. Darwin, Phil. Mag., 1922, 43, 800.
27. M. Polanyi, ibid., p. 660.
43. A. Howie and P. R. Swann, Phil. Mag., 1961, 6, 1215.
57. A. S. Keh, Phil. Mag., 1965, 12, 9.
70. 'Deformation under Hot—Working Conditions' (Special Rep. 108), 1968: London (Iron Steel Inst.).
APPENDIX 2

DISLOCATION NUCLEATED PRECIPITATION-CALCULATIONS

Cahn\textsuperscript{174} has derived an equation for the free energy change accompanying the formation of a cylindrical, non-coherent nucleus of radius \( R \) along a dislocation. This is presented in equation (2.12) of the text, from which it can be shown that no barrier to precipitation occurs when the quantity

\[
\alpha = \frac{2\Delta F_v}{\pi Y^2} > 1.0 \quad \ldots \quad (A2.1)
\]

A series of calculations have been performed in order to assess the effect of temperature (i.e. supersaturation) and Burgers vector on nucleation. In these calculations the chemical free energy is assumed to be given by the supersaturation, \( \frac{C_0}{C_T} \), from the relationship

\[
\Delta F_v = -\frac{kT}{V} \ln \frac{C_0}{C_T} \quad \ldots \quad (A2.3)
\]

where \( C_0 \) is the alloy composition assuming all Nb(C,N) to be in solid solution, \( C_T \) the equilibrium solubility at temperature \( T(\text{K}) \), \( V \) the volume per molecule of Nb(C,N) and \( k \) Boltzmanns constant. It is further assumed that \( \frac{C_0}{C_T} \) is given by the ratio of the solubility products obtained from Pickering et al\textsuperscript{281} (see equation (4.1))

\[
\frac{C_0}{C_T} = \frac{[\text{Nb}][C+N]}{[\text{Nb}]_T[\text{C+N}]_T}
\]

where the suffixes have the same meaning as above. The Burgers vectors considered are as follows

\[
\vec{b}_U = a/2\[110\] = a/\sqrt{2} \quad - \text{UNIT DISLOCATION} \\
\vec{b}_H = a\[100\] = a \quad - \text{HIRTH DISLOCATION} \\
\vec{b}_F = a/3\[111\] = a/\sqrt{3} \quad - \text{FRANK DISLOCATION} \\
\vec{b}_S = a/6\[112\] = a/\sqrt{6} \quad - \text{SHOCKLEY DISLOCATION}
\]

where \( a \) is the lattice parameter of the matrix \((2.58 \times 10^{-10} \text{ m})\). The temperature dependence of the shear modulus of the matrix was calculated from Youngs modulus data\textsuperscript{399} using isotropic elasticity theory. The strain energy parameter, \( A \), has been
taken as $gb^2/4\pi(1-\nu/2)$ in order to obtain an average value for edge and screw dislocations. The value adopted for the interfacial energy was 0.55 J m$^{-2}$.

Calculations of the variation of free energy with nucleus size at different temperatures yielded sets of curves for different Burgers vectors similar to that shown in schematically Fig. 2-19. At 750°C nucleation either at Shockley dislocations or homogeneously was characterised by a barrier, while nucleation at Frank, Hirth or unit dislocations behaved as discussed earlier. The free energy should be related to the magnitude of the Burgers vector $(\vec{b}_H \vec{b}_U \vec{b}_F \vec{b}_S)$. Consequently, Shockley dislocations, having the shorter Burgers vector, should not preferentially nucleate precipitates at 750°C due to the inadequate supersaturation.

The temperature range over which dislocation nucleated precipitation is likely to be important has been estimated for each Burgers vector using the parameter $\alpha$ of equation (A2.1) as suggested by Cahn. Results of this calculation are shown in Table A2-1 for the four types of Burgers vector, together with values of the chemical free energy per unit volume and the constant $A_H$ for the Hirth dislocation. Values of $A_F$, $A_S$ and $A_U$ for the Frank, Shockley and unit dislocations respectively, may be obtained by simple proportions from $A_H$. This particular calculation is interesting since it provides an estimate of the transition temperature ($T_c$) below which no nucleation barrier should exist (i.e. $\alpha>1.0$). It is apparent from the table that $T_c$ varies with the magnitude of the Burgers vector:

- **SHOCKLEY PARTIAL DISLOCATION** $T_c \approx 550^\circ$C
- **FRANK PARTIAL DISLOCATION** $T_c \approx 800^\circ$C
- **UNIT DISLOCATION** $T_c \approx 900^\circ$C
- **HIRTH DISLOCATION** $T_c \approx 1050^\circ$C

Consequently, nucleation at unit or Hirth dislocations is expected to be much more profuse than homogeneous nucleation at all ageing temperatures employed in the present work (i.e. below 900°C). This has generally been found to be the case as discussed in 2.2.3.2. Furthermore the calculation indicates that, providing the stacking fault energy is low enough for dissociation of unit dislocations to occur,
producing a Frank-Shockley partial dislocation pair (equation (2.10)), nucleation is likely to be restricted to the Frank partials at temperatures in the range 600° - 800°C. This seems to be consistent with previous work (2.2.3.2), in which only Frank partial dislocations have been observed to nucleate Nb(C,N) precipitates.
Table A2-1

Computed values of the dislocation nucleating efficiency ($\alpha$) for Hirth, Frank, Shockley and unit dislocations in austenite

<table>
<thead>
<tr>
<th>$T \ (^{\circ}C)$</th>
<th>$F_V \ (x10^9 \text{Jm}^{-3})$</th>
<th>$A_H \ (x10^{10} \text{N})$</th>
<th>$\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>HIRTH</td>
</tr>
<tr>
<td>500</td>
<td>4.78</td>
<td>7.14</td>
<td>7.18</td>
</tr>
<tr>
<td>550</td>
<td>4.45</td>
<td>6.94</td>
<td>6.50</td>
</tr>
<tr>
<td>600</td>
<td>4.08</td>
<td>6.74</td>
<td>5.78</td>
</tr>
<tr>
<td>650</td>
<td>3.73</td>
<td>6.54</td>
<td>5.14</td>
</tr>
<tr>
<td>700</td>
<td>3.40</td>
<td>6.34</td>
<td>4.54</td>
</tr>
<tr>
<td>750</td>
<td>3.06</td>
<td>6.14</td>
<td>3.96</td>
</tr>
<tr>
<td>800</td>
<td>2.72</td>
<td>5.94</td>
<td>3.38</td>
</tr>
<tr>
<td>850</td>
<td>2.30</td>
<td>5.74</td>
<td>2.78</td>
</tr>
<tr>
<td>900</td>
<td>2.01</td>
<td>5.54</td>
<td>2.34</td>
</tr>
<tr>
<td>950</td>
<td>1.68</td>
<td>5.34</td>
<td>1.88</td>
</tr>
<tr>
<td>1000</td>
<td>1.27</td>
<td>5.14</td>
<td>1.38</td>
</tr>
<tr>
<td>1050</td>
<td>0.97</td>
<td>4.94</td>
<td>1.00</td>
</tr>
<tr>
<td>1100</td>
<td>0.62</td>
<td>4.74</td>
<td>0.62</td>
</tr>
</tbody>
</table>
APPENDIX 3

COMPUTER PROGRAM FOR CALCULATION OF TRUE STRESS/STRAIN

DATA DURING MMTT

The purpose of the computer program was to calculate true stress and strain during MMTT cycling from load and extension measurements made directly from the chart output of the Hounsfield Tensometer. Due to the large volume of data to be handled, typical sets of tests involving about a dozen specimens and as many as 13 MMTT cycles per specimen, the simplest possible input parameters were chosen, namely the actual displacements on the chart in cm.

The input parameters were as follows:

SPECNO - specimen number
NTEST - number of MMTT cycles
AREA - initial cross-sectional area (mm$^2$)
GLGTH - specimen gauge length (mm)
PLOAD - the load equivalent to the 0.1% proof stress measured in cm on the chart
ULOAD - the load equivalent to the unloading stress measured in cm on the chart
LSCALE - load corresponding to full scale deflection of the chart
STRAIN - elongation measured in terms of the distance of chart travel
STRSCL - distance of chart travel equivalent to an extension of 0.5 mm or, in the case of specimens possessing a 50 mm gauge length, 1.0%

The parameters calculated by computer were:

NPSTRS - nominal 0.2% proof stress
NUSTRS - nominal unloading stress
TPSTRS - true 0.2% proof stress
TUSTRS - true unloading stress
NSTRN - nominal or engineering strain
TSTRN - true or natural strain
STSTRN - total or accumulated strain during MMTT
All MMTT was performed at a crosshead speed of 1 cm min$^{-1}$ (initial strain rate = $3.3 \times 10^{-3}$ sec$^{-1}$) and a chart speed of 25 cm min$^{-1}$. The full scale deflection on the chart measured 24.2 cm.

The sequence of calculations necessary to obtain the parameters described above was as follows. First the nominal proof stress was calculated

\[ \text{NPSTRESS} = \frac{\text{PLOAD} \times \text{LScale}}{0.242 \times \text{AREA}} \]  \hspace{1cm} \ldots \ (A3.1) \]

and from this the true proof stress

\[ \text{TPSTRS} = \text{NPSTRS} \times 1.002 \]

was obtained. The nominal or engineering strain was then calculated

\[ \text{NSTRN} = \frac{\text{STRAIN}}{\text{STRSCL} \times \text{GLGTH}} \]

and the nominal unloading stress

\[ \text{NUSTRS} = \frac{\text{ULOAD} \times \text{LScale}}{0.242 \times \text{AREA}} \]

From these, the true unloading stress was determined

\[ \text{TUSTRS} = \text{NUSTRS} \times (1 + \text{NSTRN}) \]

This completed the computation of the relevant parameters for the first MMTT cycle. Before the calculation could be commenced for the second cycle, the new cross-sectional area was estimated

\[ \text{AREA} = \frac{\text{AREA}}{1 + \text{NSTRN}} \]

together with the new gauge length

\[ \text{GLGTH} = \text{GLGTH} \times (1 + \text{NSTRN}) \]

The true strain was then calculated

\[ \text{TSTRN} = \ln (1 + \text{NSTRN}) \]  \hspace{1cm} \ldots \ (A3.2) \]

The total accumulated MMTT strain was then determined from equation (A3.2), since it is only valid to estimate this in terms of true strain

\[ \text{STSTRN} = \text{STSTRN} + \text{TSTRN} \]

On the first cycle the accumulated strain was given by equation (A3.2) since prior to this cycle \text{STSTRN} = 0. The sequence was then repeated for the second and
subsequent cycles using the new values of AREA and GLGTH generated in the previous cycle.

A copy of the computer program together with some typical input data for some preliminary tests and results are appended to this section. This data was checked against that obtained by direct measurements of cross-sectional area and elongations on these specimens which had scribed gauge marks. The agreement was found to be very good, lying well within the probable error of the cross-sectional area measurements (~5%).
COMPUTER PROGRAM

EX 30N
100: INTEGER SPECNO
200: REAL LSCLAE, NPSTRS, NSTRN, NUSTRS
300: DIMENSION LSCLAE(50), PLOAD(50), ULOAD(50), STRAIN(50)
400: 5 READ(5, 10) SPECNO
500: 10 FORMAT(15)
600: IF(SPECNO.EQ.1066) GO TO 999
700: READ(5,10) NTEST
800: READ(5,20) GLGTH
900: 20 FORMAT(F5.2)
1000: READ(5,20) STRSCL
1100: READ(5,20) AREA
1200: DO 30 I=1, NTEST
1300: READ(5,35) PLOAD(I)
1400: READ(5,35) ULOAD(I)
1500: READ(5,35) STRAIN(I)
1600: 30 READ (5,35) LSCALE(I)
1700: WRITE(6,40) SPECNO
1800: 35 FORMAT(F7.3)
1900: 40 FORMAT('SPECIMEN NUMBER=', I3)
2000: WRITE(2,40) SPECNO
2100: WRITE(2,45)
2200: WRITE(2,45)
2400: 16X, 'NSTRAIN', 4X, 'TSTRAIN')
2500: STSRN=0.0
2600: DO 50 J=1, NTEST
2700: NPSTRS=PLOAD(J)*LSCALE(J)/(24.2*AREA)
2800: TPS TRS=NPSTRS*1.002
2900: NSTRN=STRAIN(J)/(STRSCL*GLGTH)
3000: NUSTRS=ULOAD(J)*LSCALE(J)/(24.2*AREA)
3100: TUSTRS=NUSTRS*(1.0+NSTRN)
3200: AREA=AREA/(1.0+NSTRN)
3300: GLGTH=GLGTH*(1.0+NSTRN)
3400: TSRN=ALOG(1.0+NSTRN)
3500: STSRN=STSRN+TSRN
3600: WRITE(2,60) J, TPS TRS, TUSTRS, AREA, NSTRN, TSRN
3700: 50 WRITE(6,60) J, TPS TRS, TUSTRS, AREA, NSTRN, TSRN
3800: 5 FORMAT(3X, I3, 4X, 5(4X, F7.3))
3900: WRITE(2,70) STSRN
4000: WRITE(6,70) STSRN
4100: 70 FORMAT( ' ', 4X, 'SUM OF TSTRAIN=', F7.3)
4200: GO TO 5
4300: 999 RETURN
4400: END
INPUT DATA

EX. JAIN
100:// JOB ( )
200:// EXEC RUNCG,REGION,G=100K
300://C. ERROUTPUT DD HUFILFILE=ERRORS
400://C. SYSIN DD *
500://C. DD
600://
700://C. FTOFP001 DD HUFILFILE=DATA
800://C. SYSIN DD *
900: 17
1000: 4
1100: 50.00
1200: 2.70
1300: 9.83
1400: 8.600
1500: 13.050
1600: 3.250
1700: 500.000
1800: 12.700
1900: 15.400
2000: 2.800
2100: 500.000
2200: 15.200
2300: 17.800
2400: 2.900
2500: 500.000
2600: 17.000
2700: 19.600
2800: 3.300
2900: 500.000
3000: 18
3100: 4
3200: 50.00
3300: 2.70
3400: 10.18
3500: 9.100
3600: 13.100
3700: 2.750
3800: 500.000
3900: 13.100
4000: 16.200
4100: 3.000
4200: 500.000
4300: 15.900
4400: 18.600
4500: 3.000
4600: 500.000
4700: 17.950
4800: 20.120
4900: 2.700
5000: 500.000
5100: 1066
5200://*
## RESULTS

### SPECIMEN NUMBER = 17

<table>
<thead>
<tr>
<th>CYCLE</th>
<th>PSTRESS</th>
<th>USTRESS</th>
<th>AREA</th>
<th>NSTRAIN</th>
<th>TSTRAIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.112</td>
<td>28.089</td>
<td>9.599</td>
<td>0.024</td>
<td>0.024</td>
</tr>
<tr>
<td>2</td>
<td>27.391</td>
<td>33.819</td>
<td>9.408</td>
<td>0.020</td>
<td>0.020</td>
</tr>
<tr>
<td>3</td>
<td>33.447</td>
<td>39.893</td>
<td>9.219</td>
<td>0.021</td>
<td>0.020</td>
</tr>
<tr>
<td>4</td>
<td>31.176</td>
<td>44.934</td>
<td>9.012</td>
<td>0.023</td>
<td>0.023</td>
</tr>
</tbody>
</table>

**SUM OF TSTRAIN = 0.087**

### SPECIMEN NUMBER = 18

<table>
<thead>
<tr>
<th>CYCLE</th>
<th>PSTRESS</th>
<th>USTRESS</th>
<th>AREA</th>
<th>NSTRAIN</th>
<th>TSTRAIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.506</td>
<td>27.129</td>
<td>9.977</td>
<td>0.020</td>
<td>0.020</td>
</tr>
<tr>
<td>2</td>
<td>27.163</td>
<td>34.280</td>
<td>9.764</td>
<td>0.022</td>
<td>0.022</td>
</tr>
<tr>
<td>3</td>
<td>33.712</td>
<td>40.197</td>
<td>9.560</td>
<td>0.021</td>
<td>0.021</td>
</tr>
<tr>
<td>4</td>
<td>38.870</td>
<td>44.299</td>
<td>9.384</td>
<td>0.019</td>
<td>0.019</td>
</tr>
</tbody>
</table>

**SUM OF TSTRAIN = 0.081**
APPENDIX 4

VARIABLE LOAD TESTS

A program of work to measure such parameters as recovery rates, internal stresses etc. by variable load testing was thought desirable since it could provide supplementary information about creep processes after MMTT. The procedures employed in this work are based on those of Cottrell and Aytekin, Mitra and McLean and Ahlquist and Nix. These techniques have been used extensively to study creep in a wide range of materials.

The recovery creep method derives directly from the hypothesis of Bailey and Orowan that, during steady state creep the creep rate ($\dot{\varepsilon}$) is given by the ratio between the rates of recovery ($r$) and hardening ($h$) thus

$$\dot{\varepsilon} = \frac{r}{h} \quad \ldots \quad (A4.1)$$

Such a model provides a general framework within which more specific mechanistic models of creep have been formulated and has achieved wide acceptance. The explanation of the creep behaviour during small stress changes has been interpreted in terms of Orowan's mathematical formulation of this recovery theory. A mechanical equation of state has been assumed to be an adequate description of creep behaviour such that the flow stress of a material ($\sigma$) is some function of strain ($\varepsilon$), time ($t$) and temperature ($T$)

$$\sigma = f(\varepsilon, t, T) \quad \ldots \quad (A4.2)$$

At constant temperature it can then be shown that

$$d\sigma = \frac{\partial \sigma}{\partial \varepsilon} d\varepsilon + \frac{\partial \sigma}{\partial t} dt \quad \ldots \quad (A4.3)$$

which during steady state creep should reduce to

$$d\sigma = 0.$$ 

In other words, during steady state creep the flow stress of the material is invariant with time. Under these conditions it can be shown that equation (A4.1) results where $r = -\frac{d\sigma}{d\varepsilon}$ and $h = \frac{d\sigma}{d\varepsilon} t$. 

A schematic representation of the strain transients accompanying a small (-10%) reduction in stress and its subsequent reapplication during secondary creep is shown in Fig. A4-1. On reducing the stress by an amount $\Delta \sigma$, an instantaneous elastic contraction is observed, followed by a period ($\Delta t$), the recovery or delay time, during which little or no creep occurs. Creep then accelerates to a new steady rate. If the stress is restored to its original level an instantaneous elongation is observed which is elastic and possibly partly plastic in origin. This is followed by a transient during which the creep rate decelerates to a steady value generally the same as that prior to stress cycling. The transient creep and plastic strain ($\Delta \varepsilon$) is the other important parameter obtained from these measurements.

Gottrell and Aytekin have suggested that, providing the stress changes are small, the recovery rate is given by

$$r = \frac{-\Delta \sigma}{\Delta t} \quad \ldots \quad (A4.4)$$

It has further been suggested that the hardening rate is given by

$$h = \frac{\Delta \sigma}{\Delta \varepsilon} \quad \ldots \quad (A4.5)$$

Mitra and McLean, whilst using the same technique for measuring $r$, employed a room temperature tensile test on previously crept specimens to determine $h$. We will confine our discussion mainly to the former method which has been more widely used.

Each change in stress is accompanied by a corresponding instantaneous change in strain resulting from elastic forces within the material. While not particularly important in the context of recovery creep measurements, the magnitude of this instantaneous strain will have a considerable bearing on work described later. The temperature dependence of elastic moduli have been studied for a wide range of austenitic steels and more specifically for a 20/25/Nb steel. In the present work the latter data has been used in a slightly modified form. The temperature dependence of Young's Modulus quoted in ref. 399 is given by

$$E_T = (19.7 - 0.0069T) \times 10^4 \text{ MPa} \quad \ldots \quad (A4.6)$$
However, a more recent room temperature determination has yielded a value of $20.2 \times 10^4$ MPa and consequently a modified form of equation (A4.6) has been used

$$E_T = (20.2 - 0.0069T) \times 10^4 \text{ MPa} \quad \ldots \quad (A4.7)$$

which gives values close to those determined by Garofalo et al. Recent work has shown that the strain-transient accompanying a stress reduction varies with applied stress. In the context of recovery measurements, using the Bailey-Orowan model, the recovery rate given by equation (A4.4) may vary in a complex manner with stress decrement. In early work the strain measurement sensitivity appears to have been relatively poor by modern standards and behaviour similar to that in Fig. A4.1 was always observed. In this particular case the delay time is clearly defined. However, this behaviour has subsequently been shown to be a gross oversimplification and generally true for only a very small range of stress. If quite small stress reductions are made no horizontal transient region is observed. Characteristically the strain rises continuously to a new steady rate as shown by the middle curve in Fig. A4-2. For large stress decrements, on the other hand, an initial contraction is observed, as shown by the upper curve in Fig. A4-2. The horizontal strain transient is observed only at intermediate stress decrements. For both the large and small stress decrements an extrapolation technique is commonly used, which for small stress decrements gives a fairly unambiguous result. However for large stress reductions this can lead to confusion because two such extrapolations are possible. These are illustrated in Fig. A4-2. On reloading a strain transient occurs the magnitude of which was dependent on stress increment. Extrapolation of the creep rate in the secondary or steady creep stage to the start of the load cycle yields a strain increment $\Delta \varepsilon_p$ from which the hardening rate of the material may be estimated using equation (A4.5).

Recent work has indicated gross departures from the idealized behaviour shown in Fig. A4-1, as already discussed (Fig. A4-2). Rarely does creep stop completely upon making a stress reduction. On the contrary, depending on the magnitude of $\Delta \sigma$, the material may exhibit continued forward creep or a time dependent contraction.
prior to recommencing forward creep, as shown schematically in Fig. A4-2. The idealized case shown in Fig. A4-1 is merely a transition between these two. Generally, for stress reductions of greater than 50-75% no forward creep occurs even after long periods of time. This and the contraction observed at smaller stress decrements have been identified as an anelastic phenomenon. Results of this type may conveniently be represented as shown schematically in Fig. A4-3, where the instantaneous creep rate ($\dot{\varepsilon}_I$) on reducing the stress is plotted as a function of stress decrement ($\Delta\sigma$). The transition between positive and negative creep has been assumed to be due to the partitioning of the applied stress ($\sigma_A$) into two components; namely the internal (athermal) stress, opposing dislocation motion, and the effective (thermal) stress, the driving force for glide. Recent descriptions of high temperature deformation using this concept therefore assume that

$$\sigma_A = \sigma_I + \sigma_E$$

where $\sigma_I$ is the internal stress and $\sigma_E$ the effective stress. In these models it is assumed that, if the applied stress is reduced by an amount greater than $\Delta\sigma = \sigma_E$, creep effectively ceases for a period of time ($\Delta t$) during which the appropriate level of recovery (in $\sigma_I$) occurs permitting creep to resume at the reduced stress. Hence, the value of $\Delta\sigma$ corresponding to $\dot{\varepsilon}_I = 0$ is identified with the effective stress. It is also assumed that the internal stress is a periodic function of position in the structure and that anelastic strains or reverse dislocation motion are negligible for $\Delta\sigma < \sigma_E$.

This type of experiment based on the Ahlquist-Nix strain-transient dip test was performed together with the recovery creep tests. The instantaneous creep rate ($\dot{\varepsilon}_I$) observed on making a stress reduction was measured and as is apparent from Fig. A4-4 the results when plotted against $\Delta\sigma$ exhibit the same behaviour as Fig. A4-3. Similar experiments were performed on specimens after the stabilizing treatment and 2 cycles of the high strain (10%) MMTT. The results for these three conditions are summarised in Table A4-1. It is apparent from the table that for each treatment the internal stress is relatively independent of applied stress ($\sigma_A$), being $~0.94\sigma_A$. 
Table A4-1

Summary of 'internal stress' measurements by the strain transient dip test

<table>
<thead>
<tr>
<th>TREATMENT</th>
<th>$\sigma_A$ (MPa)</th>
<th>$T$ (°C)</th>
<th>$\sigma_I$ (MPa)</th>
<th>$\sigma_I/\sigma_A$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 MMTT</td>
<td>130</td>
<td>750</td>
<td>120</td>
<td>0.92</td>
</tr>
<tr>
<td></td>
<td>96</td>
<td></td>
<td>91</td>
<td>0.95</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td></td>
<td>47</td>
<td>0.94</td>
</tr>
<tr>
<td>2 MMTT</td>
<td>75</td>
<td>750</td>
<td>70</td>
<td>0.93</td>
</tr>
<tr>
<td>STABILISED</td>
<td>70</td>
<td>750</td>
<td>62</td>
<td>0.87</td>
</tr>
<tr>
<td></td>
<td>53</td>
<td></td>
<td>45</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>152</td>
<td>650</td>
<td>135</td>
<td>0.88</td>
</tr>
<tr>
<td></td>
<td>282</td>
<td>550</td>
<td>250</td>
<td>0.89</td>
</tr>
</tbody>
</table>
after MMTT and, surprisingly, $-0.87\sigma_A$ in the stabilised condition. Indeed the latter also appears to be independent of creep temperature.

The magnitude of the internal stress according to these measurements is rather puzzling since electron microscopy revealed an entirely different result. The MMTT material should possess an internal stress approximately 5 times greater than the stabilised condition yet still appears to exhibit creep at stresses well below this value (~150 MPa). This observation together with doubts as to the correctness of the interpretation of strain transients during variable loading stimulated an extensive study of the anelastic or recoverable creep component, which causes the contraction accompanying large stress reductions. From the results of this investigation the implications of anelasticity with regard to strain transients were considered. It is in the context of these measurements that elastic strains are of paramount importance, since at very short times the anelastic component of strain can become indistinguishable from the elastic. For these measurements, large stress reductions were made, generally to a value of stress, which, although often referred to as zero, corresponded to a slight residual stress (~2-5 MPa), in order to minimise machine backlash effects. Thus, where a stress change of $50\to0$ MPa is quoted this may well have corresponded to a real change of $53\to3$ MPa.

Typical anelastic strain-time curves are shown in Fig. A4-5 for stresses of 50 and 100 MPa at $750^\circ$C after 5 MMTT cycles. A considerable amount of systematic work has been performed on a range of materials as a result of which a number of empirical time laws have been proposed. These vary from the complex viscoelastic network model of Lubahn to the simpler logarithmic relationship proposed by Johnson and the power law of Henderson and Snedden. These relationships together with their validity and the phenomenological theories of anelastic behaviour have been reviewed recently by Henderson and Snedden. Both the power law and logarithmic relationships have been used for the data in Fig. A4-5 and the latter appears to give a better description of the data at least for times of less than 20h. This is only true if the time of loading exceeds ~5h as will be discussed later.
Throughout the work an empirical relationship of the form

\[ \varepsilon_A = m \log (1+t) \quad \ldots (A4.9) \]

where \( \varepsilon_A \) is the anelastic strain, \( m \) a temperature dependency term and \( t \) the relaxation or unloading period, has been found to be satisfactory. The data shown in Fig. A4-5 is replotted on log-linear co-ordinates in Fig. A4-6 to illustrate the quality of the agreement with equation (A4.9). More data at 750°C after 5 MMTT cycles is shown in Fig. A4-7 and the same good agreement with equation (A4.9) is apparent.

To illustrate the relative magnitude of the anelastic component of the recovered strain on unloading and elastic strains, the latter is plotted as a function of stress decrement together with the total recovered strain after 10 and 60 min. in Fig. A4-8. The same data is reproduced in Fig. A4-9 with the elastic strains calculated from equation (A4.7) removed to show more clearly the stress dependence of the anelastic strain at 2, 10 and 60 min. This component of the strain exhibits a linear stress dependence as observed by Johnson \(^{414,417}\), Lubahn \(^{418}\) and Henderson \(^{420,221}\) and Snedden \(^{420-422}\).

The magnitude of anelastic or recoverable strains appear to be not only a function of stress but also the loading time prior to removal of the applied stress. This behaviour is illustrated in Fig. A4-10. After loading for times of 20h at 750°C and 75 MPa the anelastic strain exhibits a logarithmic time dependence and a linear stress dependence. Short loading times of, for example, 15 min. lead to a more complex form of behaviour, namely a lower level of anelastic strain and clearly defined deviation from the simple logarithmic relationship at about 10 min. This observation shows the inadequacy of the Boltzmann Superposition Principle and is in good agreement with similar results in the literature \(^{420-422}\). The behaviour illustrated for 15 min. loading is characteristic of that for times below 5h at 750°C. The magnitude of the anelastic strain in this region increased with loading time, as did the point at which the deviation occurred. For loading times in excess of 5 hr. the behaviour was similar to that characterised by the 20 hr. data. This observation has considerable implications with regard to variable stress prediction.
problems, as well as the interpretation of the phenomenon of anelasticity in terms of theoretical models.

A series of anelastic measurements were made on stabilized material over a range of temperatures. The results of these tests are illustrated in Fig. A4-11 for data obtained with 70 MPa (72 + 2 MPa) stress decrements in the range 550° - 800°C. Once again, good agreement with equation (A4.9) was obtained for each set of data. The rate of anelastic strain recovery clearly increases with increasing temperature as observed by other workers. This implies that the process involved is thermally activated. However, before an activation energy may be obtained from data such as this, the precise form of the relationship between anelastic strain (\(e_A\)), time and temperature must be considered carefully. The empirical relationship of equation (A4.9) is inadequate in this respect. However, the activation energy for the process appears to be very low; it is considerably lower, in fact, than the values for most potential diffusion controlled mechanisms.

Measurements have been conducted at 750°C after 2 cycles of the high strain MMTT. Data for this, the stabilized condition and 5 MMTT cycles is shown in Fig. A4-12 for comparison. The anelastic strain recovered as a function of stress decrement, after a recovery time of 5 min. and a loading period in excess of 5h in each case, is apparently strongly structure dependent. 5 MMTT (2%) cycles exhibits the highest anelastic strains, the stabilized condition the lowest with 2 MMTT (10%) cycles producing intermediate levels. In each case a linear stress dependence was observed. Extrapolating recovered anelastic strains to 10h, which is a good approximation to the maximum level to be expected for a given stress at 750°C, and comparing these with the respective elastic strains yielded the following values of \(\frac{e_A}{e_E}\):

- 5 MMTT (2%) cycles: 0.50
- 2 MMTT (10%) cycles: 0.35
- Stabilized: 0.29

Because of the apparent inconsistencies in recovery rate and internal stress measurements it was thought instructive to pursue further the subject of anelastic
strains and their contribution to transients during variable stress tests. Some contribution of anelasticity to strain transients is accepted by most workers, with the proviso that anelasticity is unimportant for stress decrements (Δσ) less than 401 * 418,419 the effective stress (see Fig. A4-3). A few workers418,419 have made limited attempts to interpret these transients solely in terms of anelasticity. These, however, have not involved analyses of data for small stress decrements and have provided inadequate explanations of the multitude of effects generally ascribed to recovery creep processes. They have even failed to provide adequate interpretations of those inconsistencies which defy explanation in terms of the recovery creep model. In the following, the hypothesis that anelasticity produces the major contribution to strain transients will be investigated.

Before embarking on this task certain points concerning the nature of anelasticity ought to be clarified. Anelasticity is defined as a time dependent, recoverable component of strain394. In respect of its recoverable nature it is similar to elastic strain, although the latter occurs instantaneously with change of stress and is independent of microstructure. Both these recoverable components of strain can be assumed to vary linearly with stress change394. Elastic strains may truly be said to obey the Boltzmann superposition principle. However, in the case of anelastic strains this is generally only true to a first approximation as already shown (A4-10). These aspects of anelasticity and their contribution to creep have been exhaustively reviewed recently420 and adequate evidence is now available regarding its importance particularly at low stresses413-423. Furthermore, the major anelastic process is assumed to be similar to that modelled by Granato and Lucke424 to explain low temperature internal friction and formulated in the present context by Gittus425,426. Dislocation segments react to an applied stress by bowing, in a viscous fashion, to an equilibrium position determined by line tension and applied stress. Other potential processes such as grain boundary relaxation394 may also have significant effects in polycrystals. Plastic or creep strains are, in contrast, unrecoverable and a more complex function of stress. The proposed form for the
contribution of forward creep and anelastic contractions to the strain transients immediately following a stress reduction are shown in Fig. A4-3 (dotted lines). In summary, according to Ahlquist and Nix\(^{401}\) (Fig. A4-3(a))

(a) forward creep ceases at $\Delta \sigma = \sigma_E$

(b) anelastic strains are negligible for $\Delta \sigma < \sigma_E$.

It has also been pointed out recently that equations for $r$ and $h$ should more correctly be expressed in terms of $\Delta \sigma$. An alternative view, which is proposed and examined here, is that illustrated in Fig. A4-3(b), namely that anelastic strains contribute to strain transients at ALL levels of $\Delta \sigma$ and that the corresponding anelastic contraction and strain rate increase linearly with $\Delta \sigma$. It will, furthermore, be assumed that forward creep occurs at stresses well below that defined as the internal stress in Fig. A4-3(a). A precise interpretation of the intercept with the axis under these conditions is not so readily apparent since it will be a complex function of forward and anelastic creep rates. In terms of mobile dislocations, the model illustrated by Fig. A4-3(a) implies that at any instant during creep the vast majority of dislocations are mobile. The immobilized fraction is either so small that it cannot contribute significantly to anelastic processes or the dislocation configurations are such as to be incapable of relaxation. That illustrated in Fig. A4-3(b) implies almost the converse, namely a small mobile and large immobile fraction. These are all capable of anelastic relaxation and such a model appears to be more consistent with observations made recently on dislocation distributions during creep\(^{45-47}\) and dynamic in-situ HVEM creep studies\(^{428}\).

Firstly, the effect of anelasticity on strain transients for large (~30%) stress reductions will be analysed. In order to perform this type of analysis a number of assumptions are necessary. These are as follows:

(i) The effects during a period at reduced stress are solely due to an anelastic recovery (contraction) and some unspecified forward creep contribution, which is to be evaluated.

(ii) The anelastic strain may be estimated using the data given earlier for
100% load reductions.

(iii) Anelastic strains are linearly related to stress decrement, and
(iv) Partial stress reductions give rise to the same level and time dependence as equivalent 100% load reductions.

Creep curves typical of these partial stress reduction tests are shown in Figs. A4-13, A4-14 and A4-15 for 2 MMTT (10%) cycles, 5 MMTT (2%) cycles and the stabilized condition respectively. To illustrate the basic principles the results shown in Fig. A4-13 will be discussed in detail. The data points represented in the figure are those determined experimentally for a 100% stress reduction of 50 MPa (©), for the same stress decrement 100 -> 50 MPa (©) and 130 -> 80 MPa (©). Also shown in the figure are the respective forward creep components (continuous lines), ε_{p}(t), obtained by assuming that the measured creep strain, ε_{c}(t), at any instant of time (t) during the stress change is given by

\[ \varepsilon_{c}(t) = \varepsilon_{A}(t) + \varepsilon_{F}(t) \] ... (A4.10)

where ε_{A}(t) is the anelastic strain. ε_{A}(t) will be negative (i.e. a contraction) for all stress reductions. Two very important points emerge from the results shown in Fig. A4-13. Firstly the forward creep component exhibits a decelerating rate, ultimately reaching a steady creep rate in a manner characteristic of primary creep behaviour. Similar behaviour has been noted by Lubahn and others. Finally, no delay time phenomena are evident in the estimated forward creep component. It is apparent from Figs. A4-14 and A4-15 that this appears to be a common phenomenon.

The situation is fairly clear for intermediate and large stress drops where measurements and extrapolations of the type shown in Figs. A4-13 to A4-15 appear to be quite valid. For smaller stress reductions, particularly those corresponding to the condition defined as σ_{e} in Fig. A4-3, the sensitivity of strain measurement and general environmental control required to perform this type of experiment become prohibitive. Unfortunately, it is in this precise range that the model must be checked. A method does, however, exist and this is shown schematically in Fig. A4-16. If the basic behaviour at low Δσ is similar to that shown in Figs. A4-13 to A4-15,
then extrapolating the creep curve in the steady region, following a stress reduction, to the origin should yield the anelastic strain component. This assumes that the forward creep component exhibits no transients i.e. occurs at a constant rate \( \dot{\varepsilon}_L \). Reference to Figs. A4-13 to A4-15 shows this to be a reasonable approximation. Under these conditions the anelastic strain \( \varepsilon_A \) should be approximately equal to the intercept \( \varepsilon_A' \) and the recovery time \( \Delta t \) will be given by

\[
\Delta t = \frac{\varepsilon_A}{\varepsilon_L}
\]  

... (A4.11)

This approach suggests two possible methods for checking the model described in Fig. A4-3(b). It is clearly limited by the magnitude of the forward transient creep strain \( \Delta e_{ERROR} \).

The first method consists of comparing \( \varepsilon_A' \) values determined by the extrapolation technique of Fig. A4-16 \( (\Delta e_A^{\text{Ext}}) \) with \( \varepsilon_A \) values obtained by 100% load reductions \( (\Delta e_A^M) \) as a function of \( \Delta \sigma \). The results of this comparison are presented in Table A4-2 for 5 MMTT (2%) and stabilized material. Some of these results are plotted in Fig. A4-17 as a function of stress decrement and compared with the anelastic strains measured by 100% load reductions (continuous line). It is apparent from both Fig. A4-17 and Table A4-2 that \( \varepsilon_A' \) \( (\Delta e_A^{\text{Ext}}) \) values consistently lie below \( \varepsilon_A (\Delta e_A^M) \), but that they are approximately linear in \( \Delta \sigma \). \( \varepsilon_A' \) \( (\Delta e_A^{\text{Ext}}) \) also appears to be relatively independent of applied stress \( (\sigma_A) \). The data apparently agrees with the interpretation of Fig. A4-3(b) in that anelastic strains can account for strain transients at \( \Delta \sigma < \sigma_E \) which is indicated in Fig. A4-17 (i.e. \( \sigma_E \approx 8 \) MPa for \( \sigma_A = 53 \) and 71 MPa). It might be expected that, due to the strain transient in forward creep (Figs. A4-13 to A4-15), \( \varepsilon_A' < \varepsilon_A \). However, if recovery effects were paramount, anelastic strains being negligible below \( \sigma_E \) (of Fig. A4-3(a)), \( \varepsilon_A' \) would be expected to vary with \( \sigma_A \) and probably exceed \( \varepsilon_A \). Clearly neither of the latter types of behaviour were observed.

The second test that may be applied to this hypothesis was attempted using equation (A4.11). Data from Gibbons et al.\textsuperscript{430} of \( \Delta t \) as a function of reduced stress for nickel at 750°C and \( \sigma_A = 30 \) MPa is shown in Fig. A4-18(a). The creep rate \( \dot{\varepsilon}_L \)
### Table A4-2

Comparison of estimated anelastic strains using extrapolation technique ($\Delta e_A^{\text{Ext}}$) of Fig. A4-16 with unloading measurements ($\Delta e_A^{\text{M}}$).

<table>
<thead>
<tr>
<th>TREATMENT</th>
<th>$T$ ($^\circ$C)</th>
<th>$\sigma_1$ (MPa)</th>
<th>$\sigma_2$ (MPa)</th>
<th>$\Delta \sigma$ (MPa)</th>
<th>$\Delta e_A^{\text{Ext}}$ ($x 10^{-5}$)</th>
<th>$\Delta e_A^{\text{M}}$ ($x 10^{-5}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 MMTT</td>
<td>750</td>
<td>130</td>
<td>123.5</td>
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<td></td>
<td>2.35</td>
<td>2.75</td>
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<tr>
<td></td>
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<td>17.0</td>
<td></td>
<td>3.30</td>
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</tr>
<tr>
<td></td>
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<td>23.0</td>
<td></td>
<td>4.30</td>
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</tr>
<tr>
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<td>75.0</td>
<td>21.5</td>
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<td>3.30</td>
<td>4.65</td>
</tr>
<tr>
<td></td>
<td></td>
<td>91.0</td>
<td>5.5</td>
<td></td>
<td>0.90</td>
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</tr>
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<td></td>
<td></td>
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<td>3.5</td>
<td></td>
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<td>0.76</td>
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<td></td>
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<td>12.5</td>
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<tr>
<td>STABILISED</td>
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<td></td>
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<td>53.0</td>
<td>48.0</td>
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<td></td>
<td>1.88</td>
<td>2.09</td>
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</table>
at the reduced stress was computed using their data for the creep rate at 30 MPa and stress exponent (n). Anelastic strains were calculated using their result for 30 MPa assuming a linear stress dependence. Computed values of $t$ as a function of reduced stress are shown as the continuous line in Fig. A4-18(a) and it can be seen that there is reasonable agreement considering the wide scatter on results and the limitations imposed by the assumptions. Similar or superior agreement was obtained for other data of Gibbons et al.$^{430}$ Fig. A4-18(b) illustrates schematically some further data on nickel$^{430}$, where for a wide range of $\Delta \sigma$, classical "recovery behaviour" apparently occurs. It is interesting to note that the above agreement was obtained under these conditions. The observed range of possible values of $\sigma_E$ would seem to pose a problem as regards interpretation in terms of the effective stress hypothesis of Fig. A4-3(a). However, it is also difficult to reconcile the present model with this behaviour.

It is instructive to pursue this theme of anelastic contributions to creep by analysing their effect during loading transients. This is particularly relevant to the measurement of hardening rates by the method outlined earlier for recovery creep measurements$^{321}$. Using the technique outlined for load decrements in Fig. A4-13, the contribution of anelastic or recoverable strains to transient creep have been studied under a variety of loading conditions. The typical behaviour observed for initial loading and subsequent reloading in secondary creep after a period in excess of 5h at 0 MPa is illustrated in Fig. A4-19 for creep of 2 MMTT (10%) material at 50 MPa and 750°C. Shown in the figure are the measured creep curve, the minimum creep rate contribution extrapolated to $t = 0$ and the anelastic strain contribution determined from 100% load reductions. It is apparent from the figure that during initial loading (Fig. A4-19(a)) most of the transient creep strain can be accounted for by the anelastic component. A significant discrepancy was, however, observed in most cases. This may be identified with the primary plastic strain ($\varepsilon_p$) and in this particular case was $4 \times 10^{-5}$ or about 0.1 $\varepsilon_E$, which should be compared with the anelastic strain of 0.35 $\varepsilon_E$. However, reloading after a period of recovery at zero
stress (Fig. A4-19(b)) produced effectively no such plastic transient. This behaviour probably reflects the high mechanical and thermal stability of the MMTT substructure. The small transient plastic strain observed during initial loading was probably due to minor rearrangements of the more loosely pinned dislocations. The latter appear to constitute only a small proportion of the total dislocation content of MMTT material.

Apparently anomalous behaviour was observed in a minority of cases, one of which is illustrated in Fig. A4-20. The usual practice of plotting the measured creep after an unloading period and the anelastic creep component obtained during unloading together has been followed in Fig. A4-20(a) and the net forward creep component in Fig. A4-20(b). It is apparent from the figure that the behaviour here cannot be interpreted in the simple terms of Fig. A4-19. The plastic creep component is more complex than a simple minimum creep contribution consisting of a transient component with a point of inflexion at 1.4h. It is unclear whether this really reflects true material behaviour or whether it arises from experimental errors associated with machine backlash or similar effects. However, since the vast majority of tests behaved like that illustrated in Fig. A4-19 the latter is a more plausible explanation.

A final series of tests were performed to compare the behaviour of 5 MMTT and stabilised material at 750°C. The transient strain on reloading ($\Delta \varepsilon_p$ in the context of Fig. A4-1) after different periods of time spent at reduced stress levels was measured and the results are illustrated in Fig. A4-21. It is apparent from the figure that after MMTT (Fig. A4-21), at short times, the anelastic strain ($\varepsilon_A$) is greater than $\Delta \varepsilon_p$. However, they converge as the time approaches the initial loading period prior to relaxation at the reduced stress (10h). At times in excess of about 8h the two parameters are essentially equal. Furthermore, this behaviour was exhibited at two different stress levels for $\Delta \sigma = 50$ MPa. It has not been possible to check whether this behaviour was a true hysteresis effect or whether it was due to experimental errors arising from the extrapolation of the minimum creep
contribution to the origin. The latter seems unlikely since this behaviour was observed consistently in a number of similar tests at different stress levels and creep rates, including the limiting case where over the period of testing the minimum creep contribution was effectively zero compared with the anelastic strains (i.e., 40 MPa at 750°C $\varepsilon$~$5 \times 10^{-7}$ h$^{-1}$). Stabilised material, on the other hand, exhibited somewhat different behaviour as shown in Fig. A4-21(b) for $\Delta \sigma$ = 30 MPa at 750°C. At short times the behaviour was similar to that after MMTT. However, instead of $\varepsilon_A$ and $\Delta \varepsilon_p$ reaching a common level, $\Delta \varepsilon_p$ continued to increase after 2h while $\varepsilon_A$ tended to a saturation condition. Consequently at times in excess of 2h $\Delta \varepsilon_p > |\varepsilon_A|$. At this point it should be emphasized that in the context of recovery creep tests the delay time ($\Delta t$) in typical cases ($\Delta \sigma$~10% $\sigma_1$) at 750°C was only of the order of 10 min. and consequently if normal recovery creep procedures were followed the time at the reduced stress was $<<2h$. The continued rise in $\Delta \varepsilon_p$ is probably associated with irreversible deformation due to the relatively weak pinning of dislocations in stabilised material. Similar behaviour has recently been observed in nickel$^{430}$.

The information obtained from the above results relating to MMTT strengthening have been incorporated in the discussion (5.3.3). However, for completeness it is interesting to consider briefly some of the more wide ranging implications which have been discussed at length recently by Lloyd and McElroy$^{334}$.

Having shown that experimental data is consistent with this general line of reasoning, a plausible explanation of the apparently good agreement between creep rate ($\dot{\varepsilon}$) and measured recovery and workhardening rates, obtained via equations (A4.4) and (A4.5), will be outlined. An approximate relationship, equation (A4.11), has been derived for $\Delta t$ and the recovery rate expressed in terms of anelasticity becomes

$$ r = \frac{\Delta \sigma \varepsilon_L}{\varepsilon_A} \quad ... \text{(A4.12)} $$

It may further be assumed that the major portion of the transient "plastic" strain on reloading ($\Delta \varepsilon_p$) is indeed anelastic and approximately equal to that on partial
unloading \( \left( \varepsilon_A \right) \). The values of anelastic strains are typically \((0.1 - 1.0) \varepsilon_E\), which is compatible with observed hardening rates (Table A4-3) if

\[
\dot{\varepsilon} = \frac{\Delta \sigma}{\varepsilon_A} \quad \ldots \quad (A4.13)
\]

As already pointed out in the discussion of Fig. A4-21(b), this is likely to be a good approximation in most recovery creep experiments. The hardening rate \( (h) \) is therefore better described as the anelastic modulus. Indeed, a linear relationship between \( \Delta \varepsilon \) and \( \Delta \sigma \) has in fact been observed \(^{321,407} \). Combining equations (A4.12) and (A4.13) we obtain

\[
\dot{\varepsilon} = \dot{\varepsilon}_L \quad \ldots \quad (A4.14)
\]

which is true providing \( \Delta \sigma \) is small. We conclude that the Bailey-Orowan equation may be obtained by this simple interpretation of anelastic contributions to the strain transients. Therefore it seems more appropriate to refer to \( \Delta t \) and \( h \) as anelastic recovery time and modulus respectively.

Furthermore, certain other observations are consistent with the above interpretation. Although insufficient anelastic data are as yet available to predict these quantitatively, some success is evident in explaining trends.

Deviations have been observed from Garofalo's equation for primary and secondary creep \(^{431} \)

\[
\varepsilon = \varepsilon_o + \varepsilon_T \left( 1 - e^{-mt} \right) + \varepsilon_s t \quad \ldots \quad (A4.15)
\]

where \( \varepsilon_o \) is the instantaneous strain on loading, \( \varepsilon_T \) the limiting transient creep strain, \( m \) a constant and \( \varepsilon_s \) the secondary creep rate. These deviations have been observed for a wide range of metals and alloys \(^{406,408} \) during the initial 10 - 15\% of primary creep, when the creep rate decreases more rapidly than would be expected from equation (A4.15). A schematic representation of this type of behaviour is shown in Fig. A4-22(a). It has been proposed \(^{406} \) that this effect is associated with an increasing hardening rate and that equation (A4.15) adequately accounts for creep once \( h \) assumes a constant value. By its very nature anelasticity, which has been shown to contribute significantly to primary creep \(^{414-423} \), affects the early
Table A4-3

Some workhardening rates in creep

<table>
<thead>
<tr>
<th>Authors</th>
<th>Ref.</th>
<th>Material</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mitra and McLean</td>
<td>361</td>
<td>aluminium</td>
<td>$\frac{h}{G} = 0.2 - 1.0$</td>
</tr>
<tr>
<td>Mitra and McLean</td>
<td>361</td>
<td>nickel</td>
<td>$\frac{h}{G} = 0.2 - 1.6$</td>
</tr>
<tr>
<td>Ishida and McLean</td>
<td>321</td>
<td>Fe/Mn/N alloys</td>
<td>$h \approx E$</td>
</tr>
<tr>
<td>Evans and Wilshire</td>
<td>406</td>
<td>Cu-15% Al</td>
<td>$h \approx 5 \times 10^6 \text{ psi} \approx 0.3E$</td>
</tr>
<tr>
<td>Davies and Williams</td>
<td>407</td>
<td>$\alpha$-Fe</td>
<td>$h \approx 3 \times 10^6 \text{ psi} \approx 0.25E$</td>
</tr>
<tr>
<td>Sidey and Wilshire</td>
<td>408</td>
<td>Nimonic 80A</td>
<td>$h \approx 4 \times 10^3 \text{ tsi} \approx 0.3E$</td>
</tr>
<tr>
<td>Williams and McLauchlin</td>
<td>405</td>
<td>20/25/Nb stabilized stainless steel</td>
<td>$h = 0.2E$</td>
</tr>
</tbody>
</table>


stages of deformation to a greater extent, due to the logarithmic time dependence. If the anelastic contribution were to be included in the empirical form of equation (A4.9) the relationship for creep suggested by Zener\textsuperscript{394} and others\textsuperscript{416,418} is obtained

\[ \varepsilon = \varepsilon_o + \varepsilon_A + \varepsilon_T + \varepsilon_S \quad \cdots (A4.16) \]

These creep components are shown schematically in Fig. A4-22(b). Furthermore, this interpretation is in agreement with that based on \( h \), though the definition of equation (A4.13) invoking anelasticity is to be preferred. Having shown that observed variations of \( h \) with creep strain\textsuperscript{406,408} are compatible with the model, we turn our attention to observations concerning recovery time and rate. It has commonly been observed that the recovery time increases and consequently recovery rate decreases during primary creep\textsuperscript{406-408}. This behaviour is a natural consequence of an anelastic contribution to creep via equations (A4.9), (A4.11) and (A4.16).

The close interrelation between anelasticity and creep is illustrated by recent creep experiments on internally oxidized Cu-Al single crystals\textsuperscript{352}. Incremental loading and unloading tests were performed on well annealed crystals at temperatures in the range 350 - 650°C. The typical behaviour is illustrated in Fig. A4-23. Loading incrementally at stresses below the yield stress (\( \sigma_y \)) produces only an instantaneous elastic extension. Unloading in this regime similarly resulted in an elastic contraction. No creep or anelastic strains were observed. Loading beyond \( \sigma_y \) produced an instantaneous elastic and plastic strain followed by fairly typical primary creep behaviour, the creep rate decreasing progressively to a steady value. If the stress was decreased from this level to below \( \sigma_y \) the type of creep behaviour described in Fig. A4-2 was observed. Measurable creep occurred at \(-0.6 \sigma_y \) providing that the yield stress was first exceeded and anelastic contractions were then also observed. Indeed, the creep rates at stresses below \( \sigma_y \) were approximately equal to those predicted from the stress dependence above \( \sigma_y \). This observation supports the view that the mobile dislocation density is the most important parameter in creep and that the concept of internal stress has little relevance. It is also
consistent with the observation of creep in MMTT material at stresses well below the "internal stress" determined by either the strain transient dip test (Table A4-1) or electron microscopy. This view is reinforced by the strong resistance of both the MMTT and dispersion hardened single crystal materials to recovery.

The anelastic strain ($\varepsilon_A$) arising from the viscous bowing of dislocations in a network has been estimated \(^{432}\)

$$\frac{\varepsilon_A}{\varepsilon_E} = \phi \rho L^2 \quad \ldots (17)$$

where $\varepsilon_E$ is the elastic strain, $\phi$ an orientation factor, $\rho$ the dislocation density and $L$ the mean free length of bowing dislocation. In dispersion hardened systems, at low dislocation densities, $L$ is equivalent to the interparticle spacing ($D_s$). However, at high dislocation densities, where the network node spacing is less than the particle spacing, the anelastic strain is expected to be independent of $\rho$.

This has been suggested by McLean \(^{432}\) for single phase materials where $\rho \sim L^{-2}$. For example, a well annealed Cu-Al\(_2\)O\(_3\) crystal having a dislocation density of $6.0 \times 10^6$ cm\(^{-2}\) and particle spacing ($D_s$) of 0.52 \(\mu\)m exhibited no anelasticity (Fig. A4-23), while a similar crystal stressed above $\sigma_y$, having a dislocation density of $4.5 \times 10^9$ cm\(^{-2}\), exhibited an anelastic to elastic strain ratio of 0.2. This transition should theoretically occur at $\rho \sim D_s^{-2}$ i.e. $\rho \sim 4 \times 10^8$ cm\(^{-2}\) and a systematic study is planned by the author to test this prediction in a Nimonic alloy where preliminary tests\(^{433}\) indicate such an effect. The latter result gives an estimate for $\phi$, the orientation factor, of 0.2 which is close to the value of $\frac{1}{8}$ predicted by Hesketh\(^{333}\).

In addition to the viscous bowing of dislocations, in polycrystals a strong anelastic contribution arises from grain boundary relaxation processes and has been identified with the high temperature background damping phenomenon observed in internal friction studies. The latter have been shown to be a useful source of anelastic data and a simple procedure for obtaining $\frac{\varepsilon_A}{\varepsilon_E}$ values from such data has been outlined elsewhere \(^{434}\). The magnitude of the grain boundary contribution, which increases with decreasing grain size, may be as high as $\frac{\varepsilon_A}{\varepsilon_E} \sim 1.0$ in pure metals, as compared with levels of 0.10 - 0.20 in single crystals \(^{434}\). Therefore, grain
boundary effects may be as much as 500% greater than the dislocation contribution, another factor which tends to discredit internal stress models. Alloys, on the other hand, are generally observed to have $\varepsilon_A/\varepsilon_E$ values of 0.20 - 0.5.

It is apparent that the incorporation of anelasticity into a phenomenological theory of creep as outlined above provides a much improved description of variable stress behaviour than that based on internal stress. This view is reinforced by similarly good agreement between anelastic and stress relaxation measurements\textsuperscript{434}. The latter test has been used by a number of workers\textsuperscript{401,412} to obtain estimates of internal stresses, which must, like recovery creep measurements, be of dubious significance in the light of such correlations.
### APPENDIX 5

#### Summary of Creep Results

<table>
<thead>
<tr>
<th>Creep Temperature (°C)</th>
<th>Applied Stress (MPa)</th>
<th>Minimum Creep rate ($x10^{-6}$ cm h$^{-1}$)</th>
<th>Rupture Elongation (%)</th>
<th>Rupture Life (h)</th>
</tr>
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<tr>
<td></td>
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</tr>
<tr>
<td><strong>(i) Solution Treated (1200°C 1h)</strong></td>
<td></td>
<td></td>
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<tr>
<td>800</td>
<td>90</td>
<td>5,900</td>
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<td>170,000</td>
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</tr>
<tr>
<td>Creep Temperature (°C)</td>
<td>Applied Stress (MPa)</td>
<td>Minimum Creep Rate (x10^-6 m/h)</td>
<td>Rupture Elongation (%)</td>
<td>Rupture Life (h)</td>
</tr>
<tr>
<td>------------------------</td>
<td>----------------------</td>
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<td>(viii) 3 MMTT cycles (2% prestrain, aged 8h 800°C)</td>
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<td>115</td>
<td>4800</td>
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<tr>
<td>(ix) 5 MMTT cycles (2% prestrain, aged 8h 800°C)</td>
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<td>0.57</td>
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<td>0.85</td>
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<td>47</td>
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<td>(x) 13 MMTT cycles (2% prestrain, aged 8h 800°C)</td>
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<td>(xi) 1 MMTT cycle (10% prestrain, aged 8h 800°C)</td>
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<tr>
<td>750</td>
<td>50</td>
<td>36</td>
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<td>-</td>
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<td>-</td>
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<td>-</td>
<td>-</td>
</tr>
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<td>110</td>
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<td>-</td>
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</tr>
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<td></td>
<td>130</td>
<td>14000</td>
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<td>-</td>
</tr>
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<td>(xii) 2 MMTT cycles (10% prestrain, aged 8h 800°C)</td>
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<td>50</td>
<td>10.5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>80</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>800</td>
<td>50</td>
<td>36</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>750</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>900</td>
<td>21</td>
<td>65</td>
<td>0.78</td>
<td>55</td>
</tr>
</tbody>
</table>
(xiii) 3 MMTT cycles (10% prestrain, aged 1h 800°C)

<table>
<thead>
<tr>
<th>Creep Temperature (°C)</th>
<th>Applied Stress (MPa)</th>
<th>Minimum Creep rate ($\times 10^{-6} \text{ h}^{-1}$)</th>
<th>Rupture Elongation (%)</th>
<th>Rupture Life (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>800</td>
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<td>85</td>
<td>0.34</td>
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<tr>
<td>850</td>
<td>31</td>
<td>35</td>
<td>1.11</td>
<td>143</td>
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</tbody>
</table>
A6.1 Distribution Function for Random Points in Space

Consider a volume $V$ containing $N$ points and let the points be randomly situated. If the volume $V$ is divided into $Q$ equal volumes $AV$ such that $QAV = V$ then the probability that a particle lies in a volume element $AV$ is $Q^{-1}$. The probability that only one point lies in the volume element $AV$ is

$$Q^{-1} (1 - Q^{-1})^{N-1}$$

so that when $Q$ is large the probability of the point being in the volume element $AV$ is

$$\frac{N_{AV}}{V} = \frac{N_{AV}}{AV}$$

where $N_v$ is the volume density of points. This is an obvious and well known result.

Now consider a volume $V' < V; V' = Q'AV'$. Then the probability that no point lies in the volume element $AV'$ is

$$(1 - N_{vAV'})^{Q'} = (1 - \frac{N_{vV'}}{Q'})^{Q'}$$

In the limit as $Q \to \infty$ this becomes

$$\exp (- N_{vV'})$$

The probability that a point lies in a volume element $AV'$ and that no other point lies in a complementary volume $V' - AV'$ is

$$N_{vAV'} \exp (-N_{v[V' - AV']})$$

Hence the probability that a single point lies in a volume $V'$ is

$$Q'N_{vAV'} \exp (-N_{v[V' - AV']})$$

which tends to

$$N_{vV'} \exp (-N_{vV'})$$

as $Q'$ becomes large.

In general the probability of $N'$ points lying in a volume $V'$ is
which is a Poisson distribution

\[
\frac{(N V')^N}{N!} \exp(-N V')
\]

where \( M = N V' \) and \( S = N' \).

Now consider a sphere centred on one point of the distribution. The probability that no other point lies in such a sphere of radius \( L \) and that only a single point lies in a spherical shell of radius \((L + dL)\) is

\[
P(L) dL = 4\pi N v L^2 \exp\left(-\frac{4}{3}N v L^3\right) dL \tag{A6.1}
\]

where

\[
\int_0^\infty P(L) dL = 1
\]

Kendall and Moran have shown that the distribution of the \( n \)th neighbour distances \( (L_n) \)

\[
P(L_n) dL_n = \frac{3}{\Gamma(n)} \left(\frac{4\pi v}{3}\right)^n L_n^{3n-1} \exp\left(-\frac{4}{3}N v L_n^3\right) dL_n \tag{A6.2}
\]

A6.2 Estimation of Mean Near Neighbour Distances

A6.2.1 Nearest Neighbours

The mean nearest neighbour distance \((\bar{L}_1)\) is given by the expression

\[
\bar{L}_1 = \int_0^\infty L P(L) dL = 4\pi N v \int_0^\infty L^2 \exp(-\theta L^3) dL \tag{A6.3}
\]

where \( \theta = \frac{4}{3}N v \). It follows that

\[
\int_0^\infty L^3 \exp(-\theta L^3) dL = \frac{d}{d\theta} \int_0^\infty \exp(-\theta L^3) dL \tag{A6.4}
\]

and substituting \( Z^3 = \theta L^3 \) into equation (A6.4)

\[
\int_0^\infty L^3 \exp(-\theta L^3) dL = \frac{d}{d\theta} \left[ \exp(-Z^3) \right] = \frac{d}{d\theta} \frac{\Gamma(\theta)}{3} \tag{A6.5}
\]

Substituting equation (A6.5) into (A6.3) gives

\[
\bar{L}_1 = -4\pi N v \frac{d}{d\theta} \left[ \frac{\Gamma(\theta)}{3} \right]
\]
\[ F = 2.6789385 \]

Consequently

\[ \bar{L}_1 = 0.55396 N_v^{-\frac{1}{3}} \approx 0.554 N_v^{-\frac{1}{3}} \tag{A6.6} \]

which is the result obtained by Hertz\(^{315}\), Chandrasekhar\(^{316}\) and Forscher\(^{317}\).

### A6.2.2 General Case of \( n \) Neighbours

The mean \( n \) neighbour distance (\( \bar{L}_n \)) is given by

\[
\bar{L}_n = \int_0^\infty L_n P(L_n) dL_n
\]

\[ = \frac{3}{\Gamma(n)} \left(\frac{4}{3} \pi N_v\right)^n \int_0^\infty L_n^{3n} \exp (-\theta L_n^3) dL_n \tag{A6.7} \]

Following the same procedure as with equation (A6.3)

\[
\int_0^\infty L_n^{3n} \exp (-\theta L_n^3) dL_n = (-1)^n \frac{d^n}{d\theta^n} \int_0^\infty \exp (-\theta L_n^3) dL_n
\]

\[ = (-1)^n \frac{d^n}{d\theta^n} \left[ \frac{1}{\theta} \int_0^\infty \exp (-Z^3) dZ \right]
\]

\[ = (-1)^n \frac{d^n}{d\theta^n} \left[ \theta^{-\frac{1}{3}} \frac{\Gamma(\frac{3}{3})}{\frac{3}{3}} \right] \tag{A6.8} \]

Substituting equation (A6.8) into (A6.7) gives

\[ \bar{L}_n = (-1)^n \frac{3}{\Gamma(n)} \left(\frac{4}{3} \pi N_v\right)^n \frac{d^n}{d\theta^n} \left[ \theta^{-\frac{1}{3}} \frac{\Gamma(\frac{3}{3})}{\frac{3}{3}} \right]
\]

\[ = (-1)^n \frac{3}{\Gamma(n)} \left(\frac{4}{3} \pi N_v\right)^n \left[ (-1)^n \left(\frac{3(n-1)+1}{3} \right) \right]
\]

\[ \times \theta^{-\frac{3n+1}{3}} \frac{\Gamma(\frac{3}{3})}{\frac{3}{3}} \tag{A6.9} \]

The mean \( n \) neighbour spacing is therefore given by

\[ \bar{L}_n = \left(\frac{\Gamma(\frac{3}{3})}{(3n+1) \Gamma(n)}\right) \left(\frac{3(n-1)+1}{3n} \right) N_v^{-\frac{1}{3}} \tag{A6.10} \]

\[ = 0.55396 \left(\frac{3(n-1)+1}{3n} \right) N_v^{-\frac{1}{3}} \]
It is apparent that for \( n = 1 \) (i.e. nearest neighbour spacing) equation (A6.10) reduces to equation (A6.6) for \( \bar{L}_1 \).

The equation relating the mean \( n^{th} \) neighbour spacing to the volume point density has been shown to have the form

\[
\bar{L}_n = \beta_n N_v^{-\gamma} \quad \ldots \text{(A6.11)}
\]

where \( \beta_n \) is a constant of proportionality. Values of \( \beta_n \) for different near neighbour spacings have been calculated for \( n = 1 \) to \( n = 10 \) and are presented in Table A6-1. The mean of the first \( n \) near neighbour spacings (\( \bar{L}_{1n} \)) may be obtained from the relationship

\[
\bar{L}_{1n} = \frac{1}{n} \sum_{n} \bar{L}_n \quad \ldots \text{(A6.12)}
\]

and computed values of the parameter \( \beta_{1n} \) in the relationship

\[
\bar{L}_{1n} = \beta_{1n} N_v^{-\gamma} \quad \ldots \text{(A6.13)}
\]

are also included in Table A7-1.

In the case of randomly dispersed particles \( \bar{L}_{1n} \) will be equivalent to \( \bar{D} \) of equation (5.1). Consequently the value of \( \beta \) in equation (5.1) will be sensitive to the distribution of neighbours chosen. At present there seems to be no logical means of choosing between the various combinations of nearest neighbours and it will undoubtedly be governed by the way in which the equation (5.1) is employed. Consequently \( \beta \) is likely to lie in the range 0.554 to, for example, 1.00.

Unfortunately the rate of convergence of \( \beta_{1n} \) is not sufficiently rapid for an upper bound to be established in the range \( n = 1 - 10 \) for which the calculation has been performed (Table A6-1).

A6.3 Dislocation Networks

In the following discussion a dislocation network is considered to consist of randomly distributed triple point nodes interconnected by straight dislocation segments. Such a network consists of \( N_L \) segments per unit volume, each segment connecting a pair of dislocation nodes. If the density of nodes is \( p_j \) then the latter two parameters will be related by
Table A6-1

Values of the parameters of equations (A6.11) and (A6.13) for the $n^{th}$ neighbour spacings ($\beta_n$) and first $n$ neighbours ($\beta_{1n}$)

<table>
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<th>n</th>
<th>$\beta_n$</th>
<th>$\beta_{1n}$</th>
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</thead>
<tbody>
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<td>1</td>
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<td>0.55396</td>
</tr>
<tr>
<td>2</td>
<td>0.73861</td>
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<td>0.99124</td>
</tr>
<tr>
<td>10</td>
<td>1.32166</td>
<td>1.02428</td>
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</tbody>
</table>
\[ \rho_j = \frac{2}{3} N_L \]  
\[ \text{... (A6.14)} \]

since three segments meet at each node and each segment connects two nodes. For a dislocation density \( \rho_v \) expressed as length per unit volume the density of segments \( N_L \) is given by the relationship

\[ N_L = \rho_v \bar{L}^{-1} \]  
\[ \text{... (A6.15)} \]

where \( \bar{L} \) is the average segment length.

The dislocation density measured by the method of Ham\textsuperscript{274}, which is the most widely used technique in electron microscope studies, is related to the volume density \( \rho_v \) by the expression\textsuperscript{318}

\[ \rho_d = 0.5 \rho_v \]  
\[ \text{... (A6.16)} \]

where \( \rho_d \) is the measured dislocation density (in units of number per unit area). The two most important parameters describing a dislocation network are defined by the following equations:

\[ \bar{L} = \gamma_v \rho_v^{-\frac{1}{2}} \]  
\[ \text{... (A6.16)} \]

\[ \rho_j^\gamma = \delta_v \rho_v^{-\frac{1}{2}} \]  
\[ \text{... (A6.17)} \]

where \( \gamma_v \) and \( \delta_v \) are geometrical and statistical constants and are dependent on the combination of near neighbours chosen. The latter choice at present appears to be a completely arbitrary one and consequently to obtain an estimate of the effect of this variable only simple combinations have been studied. This has been achieved by performing calculations for combinations \( n_1, n_2 \) and \( n_3 \) where \( n \) lies between 1 and 6. Obviously an exclusion principle applies such that \( n_1 \neq n_2 \neq n_3 \neq n_1 \).

It follows from equations (A6.22), (A6.14) and (A6.15) that

\[ \bar{L} = \left( \frac{3}{2} \beta_m \right)^{\frac{1}{2}} \rho_v^{-\frac{1}{2}} = \gamma_v \rho_v^{-\frac{1}{2}} \]  
\[ \text{(A6.18)} \]

and

\[ \rho_j^\gamma = \left( \frac{2}{3} \gamma_v \right)^{\frac{1}{2}} \rho_v^{-\frac{1}{2}} = \delta_v \rho_v^{-\frac{1}{2}} \]  
\[ \text{... (A6.19)} \]

where \( \beta_m \) is the mean of the three values of \( \beta_n \) from equation (A6.11) corresponding
to \( n = n_1, n_2 \) and \( n_3 \) and is given by

\[
\beta_m = \frac{1}{3} \{ \beta_{n_1} + \beta_{n_2} + \beta_{n_3} \}
\]

In the context of dislocation density, as measured by electron microscopy (\( \rho_d \)), equations (A6.18) and (A6.19) become

\[
\bar{L} = \left( \frac{3}{4} \beta_m \right)^{\frac{3}{2}} \rho_d^{-\frac{1}{2}} = \gamma_d \rho_d^{-\frac{1}{2}}, \quad \text{(A6.20)}
\]

and

\[
\rho_j^{\frac{1}{3}} = \left( \frac{2}{3} \gamma_d \right)^{-\frac{1}{3}} \rho_d^{\frac{1}{3}} = \delta_d \rho_d^{\frac{1}{3}}, \quad \text{(A6.21)}
\]

respectively. Values of the parameters \( \gamma_v, \gamma_d, \delta_v \) and \( \delta_d \) are presented in Table A6-2 for different combinations of neighbours. It is apparent from the table that \( \gamma_v \) and \( \gamma_d \) increase with increasing order of neighbours, \( n \), approaching \(-1.2\) and \(-0.9\) respectively. \( \delta_v \) and \( \delta_d \), on the other hand, decrease with increasing \( n \) to \(-0.8\) and \(-0.9\) respectively. As with the value of \( \beta_n \) in Table A6-1 the rate of convergence is not sufficiently fast for an upper bound to be defined accurately, though the values presented above appear to be reasonable approximations for typical dislocation networks.

The combinations shown in Table A6-2 are idealised and represent varying degrees of tightness of the network, the tightest being that where each of the three nearest neighbours are joined. In a real network the contribution of segments joining varying combinations of the first \( n \) neighbours should be taken into account by obtaining a distribution function for this also. The latter will undoubtedly require a knowledge of the geometrical constraints on the orientation of dislocation lines, which is at present incomplete. Such a distribution would remove the bias to short segment lengths which is inherent in the examples considered here.
Table A6-2

Values of the dislocation network parameters $\gamma_V$, $\gamma_d$, $\delta_V$, and $\delta_d$ for combinations:

<table>
<thead>
<tr>
<th>$n_1$</th>
<th>$n_2$</th>
<th>$n_3$</th>
<th>$\gamma_V$</th>
<th>$\gamma_d$</th>
<th>$\delta_V$</th>
<th>$\delta_d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>3</td>
<td>0.745</td>
<td>0.527</td>
<td>0.963</td>
<td>1.081</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>4</td>
<td>0.795</td>
<td>0.562</td>
<td>0.943</td>
<td>1.058</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>5</td>
<td>0.838</td>
<td>0.593</td>
<td>0.926</td>
<td>1.039</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>6</td>
<td>0.909</td>
<td>0.643</td>
<td>0.902</td>
<td>1.012</td>
</tr>
<tr>
<td>1</td>
<td>3</td>
<td>4</td>
<td>0.862</td>
<td>0.609</td>
<td>0.918</td>
<td>1.030</td>
</tr>
<tr>
<td>1</td>
<td>3</td>
<td>5</td>
<td>0.905</td>
<td>0.640</td>
<td>0.903</td>
<td>1.013</td>
</tr>
<tr>
<td>1</td>
<td>3</td>
<td>6</td>
<td>0.979</td>
<td>0.692</td>
<td>0.879</td>
<td>0.987</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>6</td>
<td>1.190</td>
<td>0.842</td>
<td>0.824</td>
<td>0.925</td>
</tr>
</tbody>
</table>
FIGURES
Schematic representation of dislocation locking during MMTT and the build up of the dislocation substructure.
2-1. Stress–strain curves for annealed Fe/0.5% C alloy and MMTT with test to failure after 4 MMTT cycles.

2-2. Variation of lower yield stress ($\sigma_y$), ultimate tensile stress (UTS) and ductility ($\varepsilon$) with number of MMTT cycles for an iron alloy aged at different temperatures during MMTT.
Variation of lower yield stress ($\sigma_y$), ultimate tensile stress (UTS) and ductility ($\varepsilon$) with number of MMTT cycles for an iron alloy prestrained at different strain rates during MMTT. 

(See figure for graph representation.)
2-4 Variation of the Hall-Petch parameters $K_y$ and $\sigma_y$ with total MMTT prestrain. 

2-5 Variation of the fracture toughness parameters $G_{IC}$ and $K_{IC}$ with number MMTT cycles.
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(b) direct tension to 7.80% strain.

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(a) 4 MMTT cycles (Δε = 10.04%); 
(b) direct tension to 11.15% strain.

Fig. 2-8 Structure after 4 MMTT cycles and straining to UTS (Δε = 12.36%).
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Fig. 2-17 Dipoles and dislocation tangles in deformed copper single crystal (x10,000)\textsuperscript{123}.

Fig. 2-18 Dipole formation:
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(b) Tetelman's mechanism of dipole formation\textsuperscript{130}. 

\textsuperscript{123}123
\textsuperscript{130}130
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(b) unit dislocation precipitation in 15Cr 10Ni 0.8Nb 0.3V 0.1C alloy—
decoration of dislocations formed near undissolved carbides during quenching \(^{183}\) (700°C 7h);
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Fig. 4-14 Grain interior dislocation structure of solution treated RM9209 (1200°C 1h) (100 keV).

Fig. 4-15 Evidence of dislocation punching at undissolved carbides in solution treated RM9209 (1200°C 1h) (HVEM).

Fig. 4-16 Typical structure of solution treated (1200°C 1h) and aged (800°C 30h) RM9209 (HVEM).

Fig. 4-17 Dislocation punching from precipitates during examination in HVEM:
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Fig. 4-18 Structure of solution treated RM9209 after 2% strain at 20°C:
(a) 100 keV
(b) 1 MeV (HVEM)

Fig. 4-19 Structure after 4.5% strain at 20°C (HVEM)

Fig. 4-20 Structure after 9.5% strain at 20°C (HVEM)

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(b) 750°C (26h);
(c) 700°C (72h) (HVEM)

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(b) 5% prestrain;
(c) 10% prestrain (HVEM)
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Fig. 4-31 Dislocation punching from growing precipitates (2% prestrain, 800°C 3h) (100 keV):
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(b) punching from individual precipitates.

Fig. 4-32 Raft-like precipitate structure (2% prestrain, 800°C 6h) (100 keV).
Fig. 4-33 Stereomicrographs showing precipitate distribution after 1 MMTT cycle (2% prestrain) (HVEM).

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Lomer dislocation \( \frac{a}{2}[011] \) at A.

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Fig. 4-41  Typical structure after hot-tensile testing at 800°C and 10^{-2} s^{-1} in solution treated condition (HVEM).

Fig. 4-42  Structure after hot-tensile straining at 10^{-2} s^{-1} (dislocations out of contrast).

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Comparison of MMTT strengthening after different initial solution treatment and MMTT ageing conditions.

Variation of the change in flow stress, $\Delta\sigma$, and the increase in 0.2% proof stress, $\Delta\sigma_p$, per MMTT cycle with the number of MMTT cycles.
Variation of 0.2% proof stress, unloading stress and ductility with number of MMTT cycles (5% strain per cycle).

Variation of 0.2% proof stress, unloading stress and ductility with number of MMTT cycles (10% strain per cycle).

The variation of 0.2% proof stress with total MMTT or cumulative pre-strain for 2%, 5% and 10% MMTT and cold working.

Comparison of post-MMTT ductility and total accumulated strain to failure (\(\Sigma\varepsilon\)) after 2, 5 and 10% MMTT as a function of total MMTT prestrain.
4-52 Change in room temperature 0.2% proof stress accompanying annealing at 800°C of 5-MMTT material.

4-53 Reduction in room temperature 0.2% proof stress accompanying annealing at 800°C of 5-MMTT material (data normalised to account for scatter in as treated properties - see text).
Recovery data of Fig. 4-53 plotted on log coordinates.
200
150
100
50
0

True tensile strain

Tensile stress (MPa)

\[ \dot{\varepsilon} = 10^{-5} \text{ s}^{-1} \]
\[ T = 800 \degree \text{C} \]

5 MMTT cycles

Stabilized

4-55 Typical hot-tensile stress-strain curves for 5-MMTT and stabilized material.

4-56 Comparison of strain rate dependence of UTS for solution treated, stabilized and 5-MMTT material at 800\degree \text{C}.

4-57 Comparison of strain rate dependence of ductility of solution treated, stabilized and 5-MMTT material at 800\degree \text{C}.
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Comparison of stress sensitivity of minimum creep rate for 2 and 10% MMTT material at 800°C.

Variation of minimum creep rate at 800°C and 90 MPa with the number of MMTT cycles.
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Variation of 0.2% proof stress with number of high temperature (800 °C) MMTT cycles (ageing under zero or relaxed stress; straining at $10^{-3} \text{ s}^{-1}$).

Variation of relaxed stress with number of high temperature (800 °C) MMTT cycles (ageing under zero or relaxed stress; straining at $10^{-3} \text{ s}^{-1}$).
Fig. 4-73 Structures (HVEM) after:
(a) 1 MMTT cycle + 2% strain (2 incomplete MMTT cycles);
(b) 3 MMTT cycles;
(c) 5 MMTT cycles;
(d) 6 MMTT cycles + 2% strain (7 incomplete MMTT cycles);
(e) 7 MMTT cycles;
(f) 13 MMTT cycles.
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Fig. 4-75 Structure after 7 MMTT cycles and straining to the UTS ($\varepsilon = 29\%$) (HVEM).

Fig. 4-76 Selected area diffraction pattern from area shown in Fig. 4-75.

Fig. 4-77 Structure after 4 MMTT cycles of 5% strain and ageing at 800°C for $\frac{1}{4}$h (HVEM).

Fig. 4-78 Structure after 3 MMTT cycles of 10% strain and ageing at 800°C for $\frac{1}{4}$h (HVEM).
Variation of dislocation and particle densities with the number of MMTT cycles.

Histograms of typical dislocation density measurements for various MMTT conditions.
Dislocation density histogram for 5-MMTT material obtained using an average foil thickness value.

\[ \Sigma N = 54 \quad \text{Mean} = 12.0 \times 10^9 \text{cm}^{-2} \]

Standard deviation \( = \pm 21\% \)
Fig. 4-82 Structure of 5 MMTT material after hot-tensile straining at $10^{-2}$ s (HVEM).

Fig. 4-83 Structure of 5 MMTT material after hot-tensile straining at $10^{-2}$ s (HVEM).

Fig. 4-84 Grain boundary structure of 5 MMTT material after hot-tensile straining at $10^{-2}$ s (HVEM).
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(a) 800°C 910h; (b) 850°C 748h;
(c) 900°C 78h (HVEM)

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(b) stabilised material

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Fig. 4-100 Cavity and wedge crack growth at 21 MPa and 850°C:
(a) 80h; (b) 748h; (c) 1152h.

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(a) Total creep and cavity growth (ΔV/V);
(b) Estimated creep due to sources other than cavity growth.
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Fig. 4-104 Complex junction consisting of four dislocations meeting at a particle in 5 MMTT material crept at 21 MPa and 850°C for 748 h.
Increase of dislocation density per MMTT cycle as a function of the number of cycles.

(a) Variation with exposure time at 800° - 900°C of the MMTT substructure parameters:
   (a) (Particle density)⁻¹
   (b) (Dislocation density)⁻³/₂
5-3 Variation of room temperature flow stress of 0.2% proof stress with (dislocation density) for solution treated and MMTT samples.

5-4 Variation of Orowan stress with (particle density) $^{1/3}$ after MMTT.
Dislocation link length distribution in deformed metals (schematic).

\[ N_i(\text{arbitrary units}) = \frac{\sigma G b}{\sigma A} \]

- \( N_i \): Distribution function
- \( \sigma \): Stress
- \( G \): Shear modulus
- \( b \): Burgers vector
- \( \sigma_A \): Yield stress

Dislocation segment length (arbitrary units)

**Immobile**

**Mobile**

\( l_{AV} \)
5-7 Relationship between minimum creep rate or hot-tensile strain rate and ductility.

5-6 Relationship between minimum creep rate or hot-tensile strain rate and applied stress for MMTT material.
A4-1 Schematic representation of idealized strain transients accompanying the removal and re-application of a small stress $\Delta \sigma$ during secondary creep.

A4-2 Schematic representation of the diversity of strain transients observed experimentally as a function of stress decrement.
A4-3 Variation of the instantaneous creep rate accompanying small stress reductions and two possible interpretations:

(a) in terms of internal and effective stresses, and

(b) assuming anelastic contributions for the entire range of $\Delta \sigma$.

A4-4 Variation of the instantaneous creep rate accompanying small stress reductions for 5-MMTT material at 750°C.
Anelastic strain as a function of unloading or recovery time for 5-MMTT material at two stress levels.

Data of Fig. A4-5 plotted as a function of log (1+t) according to equation (A4-9).
More data for 5-MMTT material plotted according to equation (A4-9).

Variation of total recovered strain \((\varepsilon + \varepsilon_A)\) observed on unloading with stress decrement - recovered strain after 10 and 60 minutes compared with elastic strain.
Variation of anelastic component of recovered strain with stress decrement after 2, 10 and 60 minutes unloading for 5-MMTT material.

Variation of anelastic strain with log (1+t) for 5-MMTT material - effect of prior loading period.
Comparison of time dependence of anelastic strain for stabilised material at 70 MPa over temperature range 550° - 800°C.

Comparison of stress dependence of anelastic component of recovered strain for stabilised, 5 MMTT (2%) and 2 MMTT (10%) material at 750°C.
Estimation of forward creep component for partial stress reductions in 2 MMTT (10%) material.

Estimation of forward creep component for partial stress reduction in 5 MMTT (2%) material.
Estimation of forward creep component for partial stress reduction in stabilised material.

Schematic representation of the relationship between recovery time and anelastic strain (equation (A4.11)).
A4-17 Comparison of the measured anelastic strain (ε_A or ε_m) with that obtained by the extrapolation of Fig. A4-16 (ε_A or ε_Ext).

(a) Data of Gibbons et al
- Prediction of equation using following data:
  n = 5.0
  σ_A (80 N/mm^2) = 7.75 × 10^-7
  ε = 1 × 10^-5 hr^-1

Nickel 650°C

(b) Schematic representation of the observed response to stress reductions during secondary creep.
Note that the instantaneous creep rate remains constant (and equal to zero) for a wide range of Δσ.
A4-19 Comparison between anelastic strain and primary creep strain during initial loading and subsequent reloading after a period of recovery.

A4-20 Anomalous behaviour observed during reloading experiment.
A4-21 Comparison of primary 'plastic' strain ($\Delta \varepsilon_p$) observed after periods of 'recovery' at a reduced stress with the anelastic strain for stabilised and 5 MMTT material.

A4-22 Analysis of primary and secondary creep: (a) in terms of Garafalo's equation (A4.15) and (b) incorporating anelastic strain contributions.
Schematic representation of the response to stress changes observed by Lloyd et al in copper single crystals containing a dispersion of $\text{Al}_2\text{O}_3$. 