THE LOW-CYCLE FATIGUE PROPERTIES OF 20-25-Nb STAINLESS STEEL AT ELEVATED TEMPERATURE

by


A Thesis submitted to the University of Surrey
for the Degree of Doctor of Philosophy
June 1980
The microstructure and low-cycle fatigue properties of 20-25-Nb stainless steel in both the unirradiated and irradiated conditions have been studied over the temperature range 400 to 850°C.

The microstructure of the steel in the as heat-treated and aged, and neutron irradiated conditions is described, followed by a study of the effect of various test variables on the low-cycle fatigue endurance and fracture characteristics, together with some subsidiary work on tensile properties and fracture. A new type of elevated temperature low-cycle fatigue crack nucleation has been identified, and a quantitative model is presented to account for the increased low-cycle fatigue endurances associated with it.

The influence of test variables on the phenomenon of "square" grain formation during low-cycle fatigue is studied, and interpreted in terms of a new model based on dislocation energetics.

The low-cycle fatigue and tensile mechanical properties and fracture characteristics of the irradiated steel is next described and the deficiencies of the existing theory of elevated temperature embrittlement highlighted. The reasons for these deficiencies are discussed, and followed by a description of a qualitative model based on the existence of a "damaged zone" ahead of a growing fatigue crack, which it is believed offers an explanation of the observed mechanical and fracture characteristics of the irradiated steel.
ACKNOWLEDGEMENTS

The experimental work was carried out at the Atomic Energy Research Establishment, Harwell, under an extra-mural contract provided by the United Kingdom Atomic Energy Authority.

The author is indebted to his Industrial Supervisor, Dr. D R Harries of the Structural Materials Group, for his support and encouragement, and also to Professor M B Waldron, under whose Academic Supervision the work was carried out.

Several members of the Harwell staff gave advice and assistance during the course of this project and their help is gratefully appreciated.
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**INTRODUCTION**

Fatigue may be defined as the failure of a material under a reversed stress or strain less than that required to cause failure in a tensile test. Fatigue failure at ambient temperatures occurs by the nucleation and growth of a crack whilst at elevated temperatures other mechanisms, such as grain boundary void formation, may operate.

The fatigue properties and behaviour of a material may be classified either as High-Cycle Fatigue (HCF) or Low-Cycle Fatigue (LCF). In the former the strains in the bulk material are purely elastic but some plastic strain occurs in the latter. Alternatively, LCF may be defined as fatigue in which failure occurs in $10^5$ cycles or less.

Traditional engineering design always involves a "safety factor" so that a component or structure is stronger and thicker than are strictly necessary for the anticipated service conditions. The stress in such designs is nowhere permitted to reach the yield stress so that LCF is thus only a major consideration if the component or structure is subjected to repeated overloading. For this reason, previous research on fatigue has been largely concerned with HCF. Modern technologies, in contrast, sometimes require the greatest possible economy in terms of material usage. In these particular applications some degrees of plastic deformation during service may be allowed in the design and so the LCF properties of the material assume considerable significance.

**LCF in Nuclear Reactor Fuel Cladding**

The fissile fuel in nuclear power reactors is isolated from the coolant by containment in a metal "can", referred to as the "cladding". The cladding thus protects the fuel from corrosion by the coolant and also prevents escape of the highly radioactive fission products into the coolant stream. A variety of metals and alloys, including aluminium, magnesium alloys, zirconium alloys and austenitic steels, have been used as cladding materials in different nuclear reactor systems, the choice being governed by a number of considerations such as operating temperature,
compatibility with the coolant and fuel, resistance to radiation
damage and neutron capture cross-section.

In the U.K. Civil Advanced Gas-Cooled Reactors (CAGR) the
fuel cladding material is a niobium stabilised austenitic
stainless steel containing 20 wt.% chromium and 25 wt.% nickel
and is in the form of a thin walled tube about 15 mm diameter.
The fuel consists of cylindrical pellets of uranium dioxide,
enriched with respect to the fissile isotope, U235, and the
pellets are a close fit in the can so as to maximise the heat
transfer. The fuel pellets are in intimate contact with the
cladding at full reactor power; however, the fuel contracts
away from the cladding when the reactor power is reduced to
meet the requirements of the electricity grid system. The
20-25-Nb steel cladding then collapses by creep on to the fuel
during the early stages of this reduced power operation; on
subsequently increasing the power, the fuel expands and forces
the cladding outwards and repeated power cycles of this type
could give rise to low cycle fatigue failures. This effect may
be exacerbated by cracks in the fuel producing strain concentrations
in the cladding in the regions over the fuel cracks.

Background and Scope of the Present Study

Studies of the LCF properties of 20-25-Nb steel have been
carried out at the Springfields, Nuclear Power Development
Laboratories and at the Atomic Energy Research Establishment,
Harwell so as to provide basic information necessary for the
assessment of CAGR fuel element performance. The present
investigation represents a continuation of this work.

A review of the published literature on the physical
metallurgy of austenitic steels, irradiation effects in metals
and alloys, and the low cycle fatigue behaviour and properties,
particularly of 20-25-Nb austenitic steel, is presented in
Chapter 1 of this thesis.

The experimental investigations carried out and the results
obtained are described in Chapters 2, 3 and 4. The microstructural
studies on 20-25-Nb steel (Chapter 2) were intended to provide the
background data necessary for the interpretation of the LCF properties and behaviour of this steel and do not constitute a detailed investigation of the precipitation kinetics and morphology. The LCF characteristics of the unirradiated 20-25-Nb steel are described in Chapter 3, which represents the principal experimental part of the thesis; the effects of test variables (temperature, plastic strain range and strain rate) on the LCF behaviour are detailed and related to the fracture characteristics and some subsidiary work on the tensile properties is presented. Chapter 4 deals with the influence of prior neutron irradiation on the LCF and tensile properties and behaviour of the steel and attempts to interpret the effects in terms of the current models of high temperature irradiation embrittlement. The general conclusions of the work are summarised in Chapter 5.
CHAPTER 1

Literature Review

1.1 PHYSICAL METALLURGY OF AUSTENITIC STAINLESS STEELS

1.1.1 Introduction

The development, processing and properties of austenitic steels have been described in detail by other authors\(^{(1)}\)(\(^{(2)}\)(\(^{(3)}\)) and only the physical metallurgy aspects of the composition and constitution of these steels, and in particular 20-25-Nb steel, relevant to the experimental work in this thesis will be reviewed here.

The development of the currently available commercial austenitic steels has stemmed primarily from the fact that the high temperature austenite (\(\gamma\)) phase in pure iron is progressively stabilised to lower temperatures with increasing nickel content, coupled with the demonstration that increasing the chromium content imparts excellent oxidation and corrosion resistance to such an alloy. Thus, the initial austenitic steels, typified by the well-known Type 304 steel composition, contained \(\sim 18\) wt.% Cr and \(\sim 8\) wt.% Ni with small additions of other elements such as carbon, manganese and silicon, the balance being essentially iron.

It was subsequently established that reheating such alloys at 600\(^\circ\) to 750\(^\circ\)C (referred to as the "sensitisation" temperature range) produced extensive intergranular chromium carbide precipitation; the associated depletion of the chromium in the zones adjacent to the grain boundaries resulted in enhanced oxidisation and corrosion in these areas. This problem, referred to as "weld-decay", was particularly serious with respect to the properties and behaviour of the heat-affected zones in welded components. The enhanced intergranular corrosion has been avoided either by reducing the carbon content of the steels to \(< 0.03\) wt.% so that the precipitation of chromium carbides was restricted or by additions of titanium or niobium to the steel such that the carbides of these elements were formed preferentially; thus, the chromium
depletion in the grain boundary zones was limited or avoided. Furthermore, the necessity to obtain additional improvements in the oxidation and corrosion resistance of the steels has resulted in increased molybdenum and silicon contents \(^4\) whilst the optimisation of the mechanical strengths has led to more highly alloyed compositions with increased molybdenum and nickel contents \(^5\) and additions of the precipitation hardening elements titanium and aluminium \(^6\). Moreover, the need to conserve the expensive nickel has prompted the development of steels in which manganese, another austenite stabilising element, has been substituted, at least partially, for the nickel \(^7\).

Thus, a whole range of commercial austenitic steels, ranging in composition from the simple to the highly alloyed variety, are available and these possess good combinations of high temperature oxidation/corrosion resistance and long-term creep strength. The compositions of some of the well established American Iron and Steel Institute (AISI) 300 series of austenitic steels are listed in Table 1, whilst the compositions of some austenitic steels, including 20-25-Nb steel, chosen or developed specifically for thermal and fast reactor applications in the U.K. are given in Table 1.2.

1.1.2 Constitution of Austenitic Steels

(a) Thermal Equilibrium Structures

Wrought austenitic steels are either single (\(\gamma\)) or duplex (\(\gamma + \alpha\) or \(\delta\)) phase structures, where \(\gamma\) and \(\alpha\) or \(\delta\) refer to the fcc austenite and the bcc ferrite phases respectively, at the usual solution treatment temperatures of \(> 1000^\circ\text{C}\). This is evident from consideration of the isothermal section of the Fe-Cr-Ni equilibrium diagram at \(1050^\circ\text{C}\) in Fig. 1.1\(^10\) which shows that the compositions of the commercial AISI 300 series austenitic steels lie close to the \(\gamma/(\gamma + \alpha)\) phase boundary at these temperatures; however, the 20-25-Nb steel is single (\(\gamma\)) phase at this temperature due to its high nickel and chromium contents. The following nickel and chromium equivalents of the other principal alloying and impurity elements in the commercial alloys were used in locating the compositions in the Fe-Ni-Cr isothermal sections \(^11\):

\[
\text{Ni equivalent (wt.%)} = \%\text{Ni} + \%\text{Co} + 0.5 \times \%\text{Mn} + 30 \times \%\text{C} + 0.3 \times \%\text{Cu} + 25 \times \%\text{N}.
\]
### TABLE 1.1
Specified Analyses (wt.%) of some of the AISI 300 Series Austenitic Steels*

<table>
<thead>
<tr>
<th>STEEL TYPE</th>
<th>C max.</th>
<th>Si</th>
<th>Mn max.</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>301</td>
<td>0.15</td>
<td>1.00</td>
<td>2.00</td>
<td>16-18</td>
<td>6-8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>302</td>
<td>0.15</td>
<td>1.00</td>
<td>2.00</td>
<td>17-19</td>
<td>8-10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>304</td>
<td>0.08</td>
<td>1.00</td>
<td>2.00</td>
<td>18-20</td>
<td>8-12</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>310</td>
<td>0.25</td>
<td>1.50</td>
<td>2.00</td>
<td>24-26</td>
<td>19-22</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>316</td>
<td>0.08</td>
<td>1.00</td>
<td>2.00</td>
<td>16-18</td>
<td>10-14</td>
<td>2.00-3.00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>321</td>
<td>0.08</td>
<td>1.00</td>
<td>2.00</td>
<td>17-19</td>
<td>9-12</td>
<td>5x%C min.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>347</td>
<td>0.08</td>
<td>1.00</td>
<td>2.00</td>
<td>17-19</td>
<td>9-13</td>
<td>10x%C min.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Balance essentially Fe

### TABLE 1.2
Analyses (wt.%) of Austenitic Steels in U.K. Thermal and Fast Reactors Programmes

<table>
<thead>
<tr>
<th>STEEL GATION</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>B</th>
<th>REACTOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>5-Nb*</td>
<td>0.02</td>
<td>0.45-0.75</td>
<td>0.55</td>
<td>19-21</td>
<td>24-36</td>
<td>0.05-0.75</td>
<td>0.05-0.75</td>
<td>0.05-0.75</td>
<td>0.001</td>
<td>CAGR</td>
</tr>
<tr>
<td></td>
<td>0.03-0.06</td>
<td>1.5-2.0</td>
<td>16.5-17.5</td>
<td>13-14</td>
<td>2.00-2.75</td>
<td>0.05 max.</td>
<td>0.05 max.</td>
<td>0.05 max.</td>
<td>0.003 max.</td>
<td>FAST REACTOR</td>
</tr>
<tr>
<td>8*</td>
<td>0.06-0.09</td>
<td>0.75-1.25</td>
<td>16-17</td>
<td>11-12</td>
<td>1.00-1.75</td>
<td>0.05 max.</td>
<td>1.05 max.</td>
<td>0.05 max.</td>
<td>0.001-0.003 max.</td>
<td>FAST REACTOR</td>
</tr>
<tr>
<td>72HVd</td>
<td>0.10</td>
<td>0.4</td>
<td>1.8</td>
<td>15.0</td>
<td>15.0</td>
<td>1.2</td>
<td>0.4</td>
<td>Balance essentially Fe.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Balance essentially Fe.
Cr equivalent (wt.%) = %Cr + 2 x %Si + 1.5% Mo + 5 x %V + 5.5
x %Al + 1.75 x %Nb + 1.5 x %Ti + 0.75 x %W

The constitution of the steels at ambient temperatures following rapid cooling from the solution treatment temperature can be predicted using the Schaeffler diagram (Fig.1.2) which shows the phase fields again in terms of the nickel and chromium equivalents. The γ and α (or δ) phases present at high temperature in the majority of the austenitic steels are stable at ambient temperature and the 20-25-Nb steel composition is again shown to be fully austenitic; however, partial transformation of the γ + α'-martensite phase occurs in the Type 301 steel during cooling to ambient temperature due to the relatively high $M_s$ temperature of this steel (see section 1.1.4).

The constitution of the austenitic steels at ageing or service temperatures below the solution annealing temperature may also be predicted from the relevant isothermal sections of the Fe-Ni-Cr ternary diagram. An example at 650°C is shown in Fig.1.3 and it is evident that the compositions of the steels, including 20-25-Nb steel, fall within the two phase (γ + γ) field at this temperature, the σ phase being an intermetallic compound with a composition based on FeCr. In the case of the steels with duplex (γ + α) structures following solution treatment, thermal exposure at ~650°C results in rapid transformation of the α to σ phase. However, the σ phase forms directly or indirectly from the austenite at a slower rate than from the ferrite in steels which are single (γ) phase following solution treatment but whose compositions lie within the two phase (γ + σ) field at lower temperatures (see section 1.1.2 (e)).

The isothermal sections of the Fe-Ni-Cr system have not been determined experimentally at temperatures below ~600°C because of the prolonged times required to achieve thermal equilibrium at these temperatures. However, the isothermal sections of the ternary Fe-Ni-Cr equilibrium diagram at temperatures in the range 600°C = 400°C have recently been computed using thermodynamic principles (13) and a typical example at 450°C is reproduced in Fig.1.4. This shows that the compositional range of the two phase (γ + σ) field becomes more restricted with the two phase (γ + σ) and three phase (γ + α + σ) fields extending to higher nickel.
contents at the lower temperatures. Thus, the compositions of the commercial austenitic steels and 20-25-Nb steel are predicted to lie within the three phase (γ + α + σ) or two phase (γ + σ) fields at the lower temperatures, but experimental confirmation of the formation of equilibrium α phase on prolonged holding at these low temperatures has not yet been forthcoming.

(b) Precipitation of Carbides and Other Phases

In addition to the formation of α and σ phases, extensive precipitation of alloy carbides and other phases may occur in commercial austenitic steels at the solution treatment temperature or during ageing or service at lower temperatures. The crystal structures and the phases reported to form in austenitic steels are listed in Table 1.3.

The nature and extent of the phase precipitation depends on composition and on solubility and kinetic considerations.

(c) Solubility Considerations

The experimentally determined solubility of M_{23}C_{6} (where M is Fe, Cr and Mo) in unstabilised austenitic steel (Type 316) is given by (17):

\[ \log (C, \text{ppm}) = 7.771 - \frac{6272}{T^0K} \quad .... \quad (1) \]

This relation yields values for the solubility of carbon of 0.107 and 0.001 wt.% at 1050°C and 650°C respectively.

The solubilities of alloy carbides or nitrides in stabilised austenitic steels are commonly expressed in the form of the Arrhenius equation:

\[ \ln [M^X] = \frac{-H}{RT} + Y \quad .... \quad (2) \]

where \([M]\) is the concentration of the dissolved alloy element
\([X]\) is the concentration of the dissolved C or N
\(H\) is the heat of solution
\(R\) is the gas constant
\(T\) is the absolute temperature
\(Y\) is a constant
<table>
<thead>
<tr>
<th>PHASE</th>
<th>CRYSTAL STRUCTURE</th>
<th>COMPOSITION</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>CARBIDES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MC</td>
<td>f.c.c.</td>
<td>TiC; NbC</td>
</tr>
<tr>
<td>( M_7C_3 )</td>
<td>pseudo-hexagonal</td>
<td>( \text{Cr}_7C_3; (\text{Fe,Cr})_7C_3 )</td>
</tr>
<tr>
<td>( M_{23}C_6 )</td>
<td>f.c.c.</td>
<td>( \text{(Cr,Fe,Mo)}<em>{16}\text{Fe}</em>{5}\text{Mo}_2\text{C}<em>6; (\text{Fe,}\text{Cr})</em>{23}C_6 )</td>
</tr>
<tr>
<td>( M_6C )</td>
<td>f.c.c.</td>
<td>( \text{(Cr,Co,Mo,Ni)}<em>{6}C; (\text{Ti,}\text{Ni})</em>{6}C; (\text{Nb,}\text{Ni})_{6}C )</td>
</tr>
<tr>
<td><strong>NITRIDES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( M(C,N) )</td>
<td>f.c.c.</td>
<td>Nb(C,N); Ti(C,N)</td>
</tr>
<tr>
<td>( M_2N )</td>
<td>f.c.c.</td>
<td>Cr(_2)N</td>
</tr>
<tr>
<td><strong>BOROCARBIDES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( M_{23}(C,B)_6 )</td>
<td>f.c.c.</td>
<td>( (\text{Fe,}\text{Cr,Mo})_{23}(C,B)_6 )</td>
</tr>
<tr>
<td><strong>GEOMETRICALLY CLOSE PACKED PHASES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \gamma' )</td>
<td>f.c.c.</td>
<td>( \text{Ni}_3(\text{Ti,Al}) )</td>
</tr>
<tr>
<td>( \gamma'' )</td>
<td>b.c. tetragonal</td>
<td>( \text{Ni}_3\text{Nb} )</td>
</tr>
<tr>
<td>( \delta )</td>
<td>orthorhombic</td>
<td>( \text{Ni}_3\text{Nb}; \text{Ni}_3\text{Ti} )</td>
</tr>
<tr>
<td>( \beta )</td>
<td>b.c.c.</td>
<td>( \text{Ni}(\text{Al,}\text{Ti}) )</td>
</tr>
<tr>
<td>PHASE</td>
<td>CRYSTAL STRUCTURE</td>
<td>COMPOSITION</td>
</tr>
<tr>
<td>-----------</td>
<td>------------------</td>
<td>-----------------------------------------------------------------------------</td>
</tr>
<tr>
<td><strong>TOPOLOGICALLY CLOSE PACKED PHASES</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>σ</td>
<td>b.c. tetragonal</td>
<td>(Fe,Ni)_x(Cr,Mo)_y</td>
</tr>
<tr>
<td>Laves (η)</td>
<td>hexagonal</td>
<td>Fe₂Mo; Fe₂Ti; Fe₂Nb</td>
</tr>
<tr>
<td>X</td>
<td>b.c.c.</td>
<td>(Fe,Ni)₃₆Cr₁₈Mo₄; Cr₆Fe₁₈Mo₅</td>
</tr>
<tr>
<td>μ</td>
<td>rhombohedral</td>
<td>(Co,Ti)₇(CrW)₆</td>
</tr>
<tr>
<td>G</td>
<td>f.c.c.</td>
<td>(Ti,Zr,V,Nb,Ta,Mn)₆(Ni,Co)₁₆Si₇</td>
</tr>
<tr>
<td>R</td>
<td>hexagonal</td>
<td>Fe-Cr-Mo</td>
</tr>
<tr>
<td><strong>OTHERS</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Boro-Sulphides</td>
<td>h.c.p.</td>
<td>Ti₄S₂C₂</td>
</tr>
<tr>
<td>Cr-Rich</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferrite (α,)</td>
<td>b.c.c.</td>
<td>-</td>
</tr>
<tr>
<td>α-Manganese sulphide</td>
<td>f.c.c.</td>
<td>MnS</td>
</tr>
</tbody>
</table>
TiN is extremely insoluble and forms in the melt as relatively large particles which do not dissolve at least at temperatures up to 1250°C. TiC, however, shows a temperature dependent solubility, which may be expressed as follows: 

$$\log \left[ \frac{[\text{Ti}]}{[\text{C}]} \right] = \frac{6780}{T} + 2.97 \quad \ldots \ (3)$$

Likewise, the experimentally determined solubility relationship for NbC in 20-25-Nb steel is given by the following equation:

$$\log_{10} \left[ \frac{[\text{Nb}]}{[\text{C}]} \right] = \frac{-8358}{T} + 12.07 \quad \ldots \ (4)$$

where \([\text{Nb}]\) and \([\text{C}]\) are in ppm.

This equation may be translated into a solubility curve at 1050°C as shown for 20-25-Nb steel in Fig. 1.5. The stoichiometric line for NbC (Nb:C = 7.7:1) is also included and the amounts of undissolved NbC, dissolved niobium and carbon available for precipitation as NbC at a lower temperature and, where appropriate, the excess niobium and carbon in solution for three typical compositions are indicated.

This approach thus enables the precipitation behaviour to be represented in terms of solubility considerations but, unfortunately, such solubility data are not available for all phases formed in austenitic steels.

(d) Kinetic Considerations

The kinetics of the precipitation have not been elucidated for all the phases formed in austenitic steels but may be represented by the Time-Temperature-Precipitation (TTP) diagrams, as exemplified schematically in Fig. 1.6 for MC, \(M_{23}C_6\) and \(\text{Ni}_3\left(\text{Ti},\text{Al}\right)\) precipitation. It is evident that the addition of a stabilising element such as titanium or niobium to an austenitic steel does not necessarily inhibit the formation of \(M_{23}C_6\) type carbides if the ageing or service is carried out at a relatively low temperature after solution treatment; nevertheless, it is clearly established that MC is the thermodynamically stable carbide phase in stabilised austenitic steels. To ensure that the \(M_{23}C_6\) phase is not formed in stabilised steels, it is necessary to carry out a stabilising heat-treatment at \(\sim 850^\circ\text{C}\) to precipitate the alloy carbide (MC) before ageing or service at a lower temperature.
TTP diagrams have been determined experimentally for matrix precipitation of various phases in Type 316\textsuperscript{(22)} and titanium modified 316\textsuperscript{(23)} steels and for grain boundary precipitation of chromium rich intergranular carbides in Alloy 800\textsuperscript{(24)}.

(e) Second Phases in 20-25-Nb Steel

A detailed description of the formation of second phases in 20-25-Nb austenitic steels is not possible because the Fe-Ni-Cr-Nb-C phase diagram has not been determined experimentally and there are insufficient relevant thermodynamic data to enable the phase stability to be assessed\textsuperscript{(25)}. Consequently, the occurrence and prediction of second phase formation as a function of material composition and heat-treatment are based on metallographic observations and various empirical parameters.

The precipitation of second phases in 20-25-Nb steel, as in the case of other austenitic alloys, is complex. The simultaneous occurrence of five phases, namely NbC, $M_{23}C_6$, $M_6C$, $Fe_2Nb$ and $\sigma$ have been reported in the literature\textsuperscript{(25)}. The nitrogen content is important if the steel has relatively low carbon since nitrogen can replace carbon in the NbC, $M_{23}C_6$ and $M_6C$ phases. Thus, it is more accurate to describe these phases as carbo-nitrides having the general formulae Nb(C,N), $M_{23}(C,N)_6$ and $M_6(C,N)$, where M represents some combination of Fe, Cr and Ni or Nb.

Reference to the solubility data in Fig. 15 shows that the extent of the formation and nature of some of these second phases are dependent on the Nb:(C+N) ratio as well as on the solution and ageing or service temperature. In addition to $\sigma$ phase, and neglecting kinetic considerations, the phases which should form under equilibrium condition in unstabilised, hypo-stoichiometric (Nb:(C+N) < 7.7), stoichiometric (Nb:(C+N) = 7.7) and hyper-stoichiometric (Nb:(C+N) > 7.7) 20-25-Nb steel may be summarised as follows:

<table>
<thead>
<tr>
<th>Nb:(C+N)</th>
<th>Phases</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero (unstabilised)</td>
<td>$M_{23}(C,N)_6$</td>
</tr>
<tr>
<td>&lt; 7.7</td>
<td>$M_{23}(C,N)_6$ and Nb(C,N)</td>
</tr>
<tr>
<td>7.7</td>
<td>Nb(C,N)</td>
</tr>
<tr>
<td>&gt; 7.7</td>
<td>Nb(C,N) and Fe$_2$Nb</td>
</tr>
</tbody>
</table>
The experimental observations are consistent with these predictions. However, $M_6\text{(C,N)}$ also forms in the hyperstoichiometric steel. The composition of this phase, determined using electron beam microanalysis, approximates to the composition $\text{Ni}_x\text{Nb}_y\text{(C,N)}$ but containing small amounts of iron and chromium (26). The formation of this phase thus requires a minimum Nb:(C+N) ratio of 23 if all the carbon and nitrogen are present in the phase.

The formation and morphologies of these respective phases in 20-25-Nb steel will now be discussed in more detail.

$\text{Nb(C,N)}$

This phase has a sodium chloride fcc structure with a lattice parameter of 0.433 nm (27).

$\text{Nb(C,N)}$ is observed as primary eutectic particles aligned in the rolling direction of wrought, solution treated 20-25-Nb steel in which the solubility has been exceeded (28); high densities of dislocations are produced adjacent to the coarse primary NbC particles due to the differences in the coefficients of expansion of the austenite and the carbo-nitride. In addition, precipitation of this phase occurs during ageing at lower temperatures.

The homogeneous nucleation of Nb(C,N) precipitates during ageing is dependent on the degree of supersaturation which is low in niobium stabilised steels because of the reduced carbon solubility in the austenite (19)(29). As a result, this type of nucleation is rarely observed at low carbon supersaturation (30)(31) although it has been noted in a steel with an Nb(C+N) ratio close to the stoichiometric value following solution treatment at 1250°C and direct ageing at 700°C - 750°C (30).

Precipitation occurs on stacking faults in lower nickel austenitic steels such as 18-12-Nb (32). However, since the stacking fault energy increases with increasing nickel content (33) stacking faults and precipitation on stacking faults are less likely to be found in 20-25-Nb steel; nevertheless, stacking fault precipitation of Nb(C,N) has been observed in 20-25-Nb steel during ageing, if the degree of supersaturation is high, and during creep deformation (34). The inhomogeneous precipitation of Nb(C,N) usually occurs on undissociated matrix dislocations and at grain and twin boundaries. The dislocation precipitation sites in 20-25-Nb steel have been classified as follows (25).
a) Dislocations resulting from thermal strain induced during quenching.

b) Dislocations generated by differential contractions between the matrix and particles.

c) Dislocation loops generated by the condensation of vacancies and collapse of vacancy clusters.

d) Dislocations introduced by deformation.

Precipitation on dislocations permits the accommodation of large lattice mis-fit strains (≈ 20%) in the early stages of nucleation and may also allow easier diffusion by mass transport along the dislocation cores. Growth results in the formation of needles or hexagonal plates of Nb(C,N) having the parallel orientation relationship:

\[(100)_{\text{NbC}} \parallel (100)_\gamma \quad \text{and} \quad <100>_{\text{NbC}} \parallel <100>_{\gamma}\]

No evidence of solute clusters of the G-P zone type have been found but strain fields have been observed around particles ≤ 15 nm. diameter. Dislocation loops are emitted from the Nb(C,N) particles ≤ 15 nm. diameter, indicating that the precipitates are incoherent with the austenite matrix. These loops may act as further preferential nucleation sites leading to the formation of long "knotted stringers" of Nb(C,N).

The extent of the grain and twin boundary precipitation of Nb(C,N) appears to be enhanced at lower ageing temperatures and shorter times. Ageing at 700°C produces the fine distribution of grain boundary Nb(C,N) associated with a ribbon-like precipitate whilst dendritic aggregates form at higher temperatures and longer times.

\(M_6(C,N)\)

This f.c.c. phase has a lattice parameter in 20-25-Nb steel of \(1.125 \pm 0.005 \text{ nm}\) and, as stated previously, has a composition consistent with the general formula:

\((\text{Ni} + (\text{Fe} + \text{Cr}))_{\frac{1}{2}}\text{Nb}_{\frac{3}{2}}(C,N)\)

Iron and chromium are only present in small amounts in this carbo-nitride but it has been suggested that chromium enrichment may occur on ageing in the range 500° - 600°C. No solubility data for \(M_6(C,N)\) in austenite are available.

The precipitation of \(M_6(C,N)\) has been studied in detail by Dowey et al in a low-carbon hyper-stoichiometric alloy and
it was suggested that this carbo-nitride forms by transformation of the Nb(C,N). Nb(C,N) initially precipitated on intragranular dislocations and on undissolved primary \( M_6(C,N) \) particles in the grain boundaries; these particles subsequently grew into dendritic aggregates and transformed to \( M_6(C,N) \), the reaction proceeding approximately ten times faster in the grain boundaries than in the grain interiors. \( M_6(C,N) \) with the dendritic flake habit only formed in the grain interior if the initial dislocation density was high. Thin sheets of carbide were observed to grow outwards from grain boundary rods, partly along the boundary and partly into adjacent grains. Diffraction patterns could not be obtained in the early stages of growth, nor were thickness fringes visible, but at a later stage diffraction patterns of \( M_6(C,N) \) were obtained. The habit plane was (100), but no particular orientation relationship was established.

\[
\text{M}_{23}(C,N)_6
\]

The lattice parameter of this carbo-nitride is typically 1.064 nm, and its solubility in austenite is given by equation (1).

The nucleation of this phase at grain and twin boundaries and as a Widmanstätten precipitate within the grains has been described in a series of published papers (37-40) whilst its formation in 20-25-Nb steels containing 0.02 and 0.06\% (C + TiN) has been reported by Cook et al (28). It appears that \( M_{23}(C,N)_6 \) is only a transient phase in stoichiometric and hyper-stoichiometric 20-25-Nb steel and prolonged ageing causes dissolution of the precipitates and the formation of the relatively more stable \( \text{Nb}(C,N) \) phase (29).

\[
\text{Fe}_2\text{Nb}
\]

The lattice parameters of this Laves phase are \( a = 0.4829 \) nm and \( c = 0.7877 \) nm (41).

The phase forms as small hexagonal plates at intragranular sites in 20-25-Nb steels aged for 5000 h at 700°C (29). In general, \( \text{Fe}_2\text{Nb} \) is only formed in niobium stabilised steels with high niobium:(C + N) ratios after ageing for long times (29)(36). However, Rowcliffe and Winton (30) found that \( \text{Fe}_2\text{Nb} \) was the main phase precipitated in a 20-25-Nb steel with \( \text{Nb}:(C+N) \simeq 52 \) at 700°C.

\( \sigma \) Phase

The structure and properties of this electron compound have been reviewed elsewhere (42)(43).
Consideration of the 650°C isothermal section of the Fe-Ni-Cr system shown in Fig.1 indicates that only a small amount of σ phase should form in 20-25-Nb steel at this temperature. The alloy should be fully austenitic at higher temperatures of \(\gtrsim 800^\circ C\)\(^{25}\).

There is relatively little published information on the nucleation of σ phase in 20-25-Nb austenitic steel. σ phase is known to form rapidly by transformation of the α(or δ)-ferrite in duplex austenitic steels but ferrite has not been observed in 20-25-Nb steel. Most of the current understanding of σ formation from austenite has been derived from observations made on other austenitic alloys and it appears that the precipitation behaviour varies markedly with composition. For example, in a 26% Cr: 26% Ni steel the sigma phase forms first at triple grain boundary junctions and subsequently at the grain boundaries away from the triple points and on intragranular inclusions\(^{44}\). However, the preferred σ nucleating sites in an unstabilised 25:20 steel are twin boundaries and stacking faults\(^{45}\); the σ/ matrix orientation relationship is generally close to:

\[(111)_\gamma \parallel (001)_\sigma \quad [\overline{1}10]_\gamma \parallel [\overline{1}10]_\sigma\]

The precipitation of σ from austenite generally occurs at a slow rate but the process may be speeded up by prior cold working\(^{44}(46)\).

The views on the interrelation between carbo-nitride and σ-phase precipitation from austenite are conflicting. For example, some authors\(^{47}\) have concluded that σ phase forms directly from the austenite whilst others\(^{48}(49)\) have suggested that σ forms directly from carbides. An attempt has been made by Knowles\(^{25}\) to resolve these conflicting views and his ideas will not be reiterated here. It suffices to say that, irrespective of whether the σ forms directly from the austenite or from the carbides or carbo-nitrides, it has been clearly established\(^{50}\) in Type 316 steel that the formation of the σ phase is associated with dissolution of \(M_{23}C_6\) type precipitates, the latter process supplying the chromium necessary for the σ phase formation.

1.1.3 The Effects of Boron

Boron additions of up to \(\sim 100\) ppm (wt.) are often made to
austenitic alloys to improve their hot workability and creep-rupture properties, particularly rupture ductility.

The influence of boron on the constitution of and precipitation in austenitic alloys has been fairly extensively investigated but the effects are not completely understood. It has been demonstrated, using an auto-radiographic technique, that the boron may be associated with intragranular primary particles of NbC, inter- and intra-granular f.c.c. carbides such as M_{23}C_6 and segregated in atomic form at the grain boundaries.

The solubility of boron is typically ≤100 ppm (wt.) in austenitic steels at solution treatment temperatures of ~1050°C (39 ppm (wt.) in 20-25-Nb steel) and considerably less than this at the lower temperatures of 650° - 750°C. It was therefore assumed initially that the intergranular atomic segregation was a result of grain boundary equilibrium segregation of boron during annealing at the solution treatment or lower temperatures. However, it has been demonstrated that the enhanced concentrations of boron found in the grain boundary regions of austenitic steels are a consequence of non-equilibrium segregation, the extent of which increases with increasing temperature and decreasing cooling rate. This effect has essentially been attributed to the existence of boron-vacancy complexes at the high temperatures; during relatively slow cooling, these complexes diffuse to the free surface and grain boundaries where the vacancies are annihilated, leaving excess concentrations of boron in these regions.

However, the high concentration of boron at these interfaces do not persist during ageing or creep-rupture testing of austenitic alloys at 650° - 700°C and the boron may be incorporated into carbide phases formed at these temperatures. Thus, it has been observed that boron promotes the formation of intergranular M_{23}(C,B) type precipitates at the expense of MC (where M is niobium or titanium) in stabilised steels and also affects the morphology of the M_{23}C_6 type precipitates; furthermore, there is some evidence that in unstabilised
austenitic steels the replacement of some of the carbon by boron atoms (a) increases the lattice parameter of the M$_{23}$C$_6$ precipitates such that the mis-fit with the austenitic lattice is reduced (62), and (b) increases the stability of the carbide such that $\sigma$ phase formation is inhibited (50).

1.1.4 Martensitic Transformations

It has been established (63)(64) that diffusionless transformation of the austenite ($\gamma$) to martensitic phases may be induced spontaneously in some austenitic steels by quenching below their $M_S$ temperatures, which are usually well below room temperature for these alloy compositions. In addition, the martensitic transformations may be produced by deformation at temperatures well above the $M_S$ and a temperature $M_d$ may be defined above which the transformation cannot be induced irrespective of the deformation strain.

Two martensitic phases have been identified in Fe-Ni-Cr alloys and austenitic steels, namely, the ferromagnetic, bcc $\alpha'$-martensite and the non-magnetic, hcp $\varepsilon$-martensite. The transformation sequence is now generally accepted to be $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ (65) and the lattice parameters of the respective phases are such that the $\gamma$ to $\alpha'$- and $\varepsilon$-martensite transformations result in a volume expansion and contraction of 2.45 and 0.81% respectively.

The $\alpha'$- and $\varepsilon$-martensites are metastable phases and revert to the parent $\gamma$ phase at high temperatures. Reversion of the $\varepsilon$-martensite to $\gamma$ occurs in the temperature range $150^\circ - 400^\circ$C even during short annealing times (65); the $\alpha'$-martensite $\rightarrow \gamma$ reversion is reported to occur in the approximate temperature range $500^\circ - 600^\circ$C for some steels whilst in others the reversion takes place at higher temperatures in the range $570^\circ - 850^\circ$C (65).

The extent of the $\gamma \rightarrow \alpha'$-martensite transformation during quenching to or deformation at room temperature or sub-ambient temperatures is related to composition. It has been established that increasing the contents of all the alloying elements (with
the possible exception of cobalt) in austenitic steels reduces both the $M_s$ and $M_d$ temperatures ($66-69$). However, the compositional dependencies of the $\gamma \rightarrow \alpha'$-martensite transformation temperatures are not as well founded; it appears that the formation of the $\epsilon$ phase is related to the stacking fault energy ($\gamma_{SFE}$) and it has recently been established that $\epsilon$-martensite is only detected in austenitic steels quenched to or deformed at room temperature or $-196^\circ C$ if the $\gamma_{SFE}$ at room temperature $< 35 \pm 5 \text{ mJm}^{-2}$ ($\gamma_{SFE}$).

There are no reported observations of either $\alpha'$- or $\epsilon$-martensite transformation in the quenched or deformed highly alloyed 20-25-Nb austenitic steel and this is consistent with the established $M_s$ and $M_d$ - compositional relationships ($66-69$) and the high $\gamma_{SFE}$ of the steel, estimated from the $\gamma_{SFE}$ - compositional relationships given by Schramm and Reed ($71$) to be $\sim 64 \text{ mJm}^{-2}$. 
FIG. 1.1 1050C ISOTHERMAL SECTION OF THE Fe-Ni-Cr EQUILIBRIUM DIAGRAM WITH COMPOSITIONS OF SOME OF THE AISI 300 SERIES AND 20-25-Nb STEELS INDICATED.
FIG. 1.2 SCHAEFFLER DIAGRAM WITH THE LOCATIONS OF THE COMPOSITIONS OF SOME OF THE AISI 300 SERIES AND 20-25-Nb AUSTENITIC STEELS INDICATED
FIG. 1.3 650°C ISOTHERMAL SECTION OF THE Fe-Ni-Cr EQUILIBRIUM DIAGRAM WITH COMPOSITIONS OF SOME OF THE AISI 300 SERIES AND 20-25-Nb STEELS INDICATED
FIG. 1.4 CALCULATED 450°C Isothermal Section of the Fe-Ni-Cr Equilibrium Diagram with Compositions of Some of the AISI 300 Series and 20-25-Nb Steels Indicated
Ml IU bU L
NIOBIUM & CARBON TO PRECIPITATE AS NbC AT 650C
EXCESS NIOBIUM IN SOLUTION
NIOBIUM & CARBON SOLUBLE AT 650C

FIG. 1.5 SOLUBILITY OF NbC IN Nb STABILISED AUSTENITIC STEEL AT 1050C AND 650C
FIG. 1.6 SCHEMATIC TIME-TEMPERATURE-PRECIPITATION CURVES
1.2 THE EFFECTS OF NEUTRON IRRADIATION ON STRUCTURE AND PROPERTIES

1.2.1 Introduction

It is now well established that significant changes in the structures and mechanical properties of fuel, fuel element cladding and core component materials are produced by neutron irradiation in the cores of thermal and fast reactors. These irradiation induced changes in the structures and properties of austenitic alloys have been investigated in detail and the principal results and observations have been reviewed in a number of publications over the last decade or so (72-80).

1.2.2. Radiation Damage

1.2.2(a) Neutron Energies and Doses

The components in or near the cores of fast reactors are bombarded with neutrons having energies ranging from approximately 500 electron volts (eV) to ten million electron volts (MeV). However, some of the high energy neutrons created during fission of the $^{235}$U isotope in the nuclear fuel (average energy about 2 MeV) are slowed down by repeated collisions with the atoms of the moderator in a thermal reactor and the neutron energy spectrum in this type of reactor extends from 0.025eV to about 10MeV.

The exposure of a non-fissile material to neutron irradiation is usually expressed in terms of the number of neutrons of a given energy (e.g. thermal neutrons with energies of 0.025eV) or within a given range (e.g. greater than 0.1 and less than 1MeV) impinging on unit area of the target for the duration of the irradiation. Thus the unit of exposure, or fluence, is the product of the neutron flux (neutrons per square metre per second) and the irradiation time (in seconds) and is specified as n.m$^{-2}$ (thermal) or n.m$^{-2}$. (greater than 0.1MeV and less than 1MeV).

1.2.2(b) Displacement Damage

Neutrons can produce radiation damage in non-fissile metals and alloys directly by knocking atoms out of their equilibrium lattice positions creating vacant lattice sites and interstitial atoms (81).
The energy \( E_d \) required to displace an atom and create a vacancy and nearby interstitial (a Frenkel pair) is in the range 20 to 50 eV for most metals. Thus, only the high energy neutrons can produce a significant number of vacancy-interstitial pairs because only they are capable of giving a large recoil energy to the struck nucleus.

The irradiation induced defect production rates or the number of times each atom is displaced from its lattice site per unit time during irradiation are dependent on the material composition and the neutron flux, energy and spectrum. The number of displaced atoms, \( n_d \), created by a primary recoil atom of energy, \( E_n \), may be calculated from modified versions of the original formulation of Kinchin and Pease as:

\[
\frac{n_d}{E_d} = 0.8\Delta E_n \frac{n}{2E_d} \quad \text{(1)}
\]

where \( \Delta E_n \) is the fraction of \( E_n \) lost in elastic collisions. The total damage may then be computed from a knowledge of the spectrum of energies of the primary recoil atoms in the particular neutron bombardment regime. Several more sophisticated models have been developed to enable the number of displaced atoms to be estimated; these include the "half-Nelson" \((N/2)\) and Norgett, Torrens and Robinson \((NRT)\) models. However, there are still areas of uncertainty in respect of the calculation of the number of atoms displaced during irradiation, particularly with respect to complex alloys.

The atom displacement rates for iron irradiated in the cores of thermal and fast reactors are typically about \( 1 \times 10^{-7} \) and \( 1 \times 10^{-6} \) displacements per atom per sec (dpa) respectively; the corresponding numbers of displaced atoms produced during an exposure time of one \( 3 \times 10^7 \) s, are thus about 3 and 30 dpa (half-Nelson).

1.2.2(c) Gas Production

Neutrons may produce radiation damage indirectly by being absorbed and producing changes in atomic nuclei creating impurity atoms. A large number of \((n,y)\) nuclear reactions
can occur in non-fissile materials but the \((n,a)\) and \((n,p)\) reactions, which result in the formation of helium and hydrogen respectively, are probably the most important in austenitic alloys. The following are some examples of these gas producing reactions:

**Helium**

\[(1) \quad {^7}_{10}B + n \rightarrow {^3}_{7}Li + 2{^4}_{2}He\]

\[(2) \quad {^{28}}_{58}Ni + n \rightarrow {^{28}}_{59}Ni + \gamma\]

\[2{^{28}}Ni + n \rightarrow 2{^{26}}Fe + 2He\]

\[(3) \quad {^7}_{14}N + n \rightarrow {^5}_{11}B + 2He\]

\[(4) \quad {^{26}}_{54}Fe + n \rightarrow 2{^{24}}Cr + 2He\]

Reactions (1) and (2) are induced by neutrons with thermal energies while (3) and (4) are threshold reactions and are only activated by neutrons with energies greater than about 1MeV.

**Hydrogen**

\[(1) \quad {^{28}}_{58}Ni + n \rightarrow {^{27}}Co + {^1}_1H\]

\[(2) \quad {^{26}}_{54}Fe + n \rightarrow {^{25}}Mn + {^1}_1H\]

Both of these are threshold reactions.

The \((n,p)\) Li\(^7\) and \((n,\gamma)\) Ni\(^{58}\) \((n,\alpha)\) Fe\(^{56}\) reactions are the principal sources of helium in austenitic alloys irradiated in thermal reactors whereas most of the helium generated in these materials during irradiation in the cores of fast reactors arises from the fast neutron "threshold" reactions. The final helium concentrations are in the range 10 to 200 ppm (at) for the austenitic steels currently used and favoured for core component applications in large thermal and fast reactors.

1.2.3 The Effect of Neutron Irradiation on Microstructure

1.2.3(a) Point Defect Clusters

During irradiation the fast neutron imparts up to several hundred KeV of energy to the recoil atom which is thus sufficiently energetic to displace many more atoms (equation (1)). However,
as the primary recoil atom slows down, the mean free path between collisions becomes progressively smaller so that most of the displacement, in all but the lowest density metals, occur in localised regions termed "displacement cascades", typically 1 to 10 nm in diameter. A large fraction of the vacancies and interstitials formed within the displacement cascade or spike immediately recombine. Nevertheless, some of the interstitial atoms escape and are transported away from the centre of the displacement cascade in correlated collision sequences along closely packed rows of atoms leaving a vacancy rich core which collapses to produce a small faulted vacancy loop or stacking fault tetrahedron (Fig.1.7).

Changes in electrical resistivity, lattice parameter, Curie temperature, superparamagnetism, etc. have been measured and used to infer the nature of the irradiation induced microstructure. However, the application of transmission electron microscopy has allowed major advances to be made in the direct observation and identification of point defect clusters larger than about 2 nm (87) and field-ion microscopy has enabled the behaviour of individual point defects to be studied in some instances (88).

The characteristics of the irradiation induced microstructures in austenitic alloys, as determined by electron microscope observations, are dependent on the irradiation temperature and dose (or fluence) and may be summarised as follows: (76)(80)(87)(90).

At irradiation temperatures below 325 C or thereabouts the microstructure consists of a high density of vacancy and/or interstitial defect clusters or loops, where sizes are dependent on material purity but are typically a few nm in diameter. The interstitial loops nucleate homogeneously from the individual migrating interstitials; during prolonged radiation these loops often unfault, coalesce and develop into a uniform dislocation network, characteristic of the irradiation temperature and damage rate. The number of vacancy loops formed heterogeneously in the displacement cascades saturates at a dose of approximately
1 x 10^{-2} dpa; at higher doses the vacancy loops overlap and interact to produce a dislocation network.

In the approximate temperature range 325 to 700°C, the interstitials form a dislocation structure of loops, segments and network. At low doses and low temperatures in this range, the dislocation structure generally consists of faulted loops which increase in size with increasing dose and temperature and may unfault forming a dislocation network. The lifetime of the small vacancy loops decreases as the irradiation temperature increases and becomes insignificant when the rate of thermal vacancy emission from them is large. The vacancies precipitate to form voids in this temperature range (see section 1.2.3(b)). The point defect structures are not stable at temperatures above about 650°C and helium bubbles are nucleated at the grain boundaries and on dislocations within the grains (see section 1.2.3(c)).

1.2.3(b) Void Formation

The phenomenon of irradiation induced void formation and swelling was first discovered experimentally in 1966(91) when it was demonstrated that austenitic steels irradiated in the Dounreay Fast Reactor (DFR) exhibited significant increases in volume, evident as decreases in density. This discovery prompted extensive theoretical and experimental investigations because of the importance of the phenomenon in determining the dimensional and mechanical stability of the materials used for the core components in sodium cooled fast reactors.

A detailed understanding has been achieved as to why voids nucleate and grow within a fairly narrow temperature range (approx. 350 to 700°C) in unstressed austenitic alloys (92-100). The void swelling results essentially from the production of an equal supersaturation of vacancies and interstitials by fast neutron-atom collisions; the dislocations in the structure have a slightly higher preference for the interstitials than the vacancies and the excess vacancies migrate and form clusters which are stabilised as three dimensional voids by residual gases and/or inert gases produced by (n,α) reactions during irradiation. The voids are not formed at temperatures below about 350°C in austenitic alloys because the defect population
is controlled by mutual recombination between the interstitials and vacancies whilst the voids are not stable above 650°C or 700°C because the thermal vacancy concentration exceeds that produced by irradiation.

The extensive experimental data currently available on the neutron induced void swelling in austenitic steels and nickel base alloys have been obtained from dimensional measurement, immersion density and transmission electron microscopy of fuel pin cladding, structural components and material samples irradiated in fast test reactors. These data have been supplemented by the results of charged particle irradiations in accelerators and 1 MeV electron bombardments in a high voltage electron microscope; these simulation approaches have enabled the void formation and swelling to be produced about a thousand times faster than in the highest flux reactor.

The results of the reactor and simulation irradiation experiments have demonstrated that, in addition to its strong dependency on irradiation temperature, the void swelling of austenitic alloys increases progressively with increasing dose and with no evidence of saturation, and is also markedly dependent on the major (nickel and chromium) and minor (particularly carbon, silicon, titanium and zirconium levels) alloying elements in solution, on the structure, and on the initial thermomechanical treatment. However, more limited data suggest that the ferritic steels exhibit relatively high resistance to void swelling while it has been predicted theoretically that the void swelling is enhanced by stress, particularly at temperatures towards the upper end of the void stability range.

1.2.3(c) Helium Bubbles

Inert gases have very low solubilities in metals and so helium formed in (n, α) reactions will tend to precipitate and form helium-filled bubbles, particularly where the lattice is disordered, such as at grain boundaries, dislocation lines, or matrix/precipitate interfaces. It has been suggested that a
bubble is nucleated when two helium atoms enter a vacancy as a result of diffusion migration \(\text{[111]}\). Further vacancies and helium atoms may then diffuse to the bubble nucleus so promoting growth to a size where the bubbles may be resolved by transmission electron microscopy (about 25Å dia.). Under equilibrium conditions, the internal gas pressure \(P\) is balanced by the surface tension \(\gamma\) of the bubble surface according to the relation:

\[ P = 2 \frac{\gamma}{r} \]

where \(r\) is the bubble radius. Although pressure and surface tension are balanced there is an associated spherically symmetric strain field around the bubble which in thin foil electron microscope specimens results in a characteristic ring of dark contrast around the bubble periphery. Consequently bubbles may be distinguished from holes or voids.

In bubbles having radii less than about 1000Å the equilibrium gas pressure is so high that considerable deviation from ideal gas behaviour may be expected. Under these conditions a van de Waal's type of constitutive equation is appropriate: one such equation is:

\[ \left( P + \frac{m^2 a}{\gamma^2} \right) \left( V - m b \right) = m k T \]

where \(P\) = gas pressure, \(V\) = gas volume, \(T\) = gas temperature \(m\) = mass of gas in bubble, \(k\) is Boltzmann's constant, \(a\) and \(b\) are van de Waal's constants.

Since the attraction between molecules of helium is so low, the term including \(a\) may be neglected so that the equation reduces to:

\[ m = \frac{8 \pi \gamma r^3}{3 (k T r + 2 \gamma b)} \]

where \(\gamma\) is the surface energy. Estimates of the amount of helium present in an 18/10/Nb austenitic steel have been made using the above equation and compared with those obtained from calculation of the B\(^{10}\) isotope burnup \(\text{[112]}\) reasonable agreement being found, although the helium contents estimated from bubble measurements were consistently lower than those from the burnup calculations. This work will be discussed further in section 1.2.3 (d) para. (3), page 51.
Bubbles in thin foil copper specimens have been observed to migrate under a thermal gradient \(^{(113)}\); since the solubility of helium is very low in metal it was proposed that migration occurred by the diffusion of matrix atoms across the surface of the bubbles. A theoretical analysis also supported this conclusion \(^{(114)}\). It has alternatively been proposed that the pressure in a bubble is so high that some gas may dissolve in the matrix \(^{(115)}\). Some support for this idea is given by the observed dissolution of krypton in some metals at high pressures \(^{(116)}\).

A matrix bubble will migrate in a random manner unless it is attracted to a dislocation or grain boundary where it will be held. Under a stress, temperature or concentration gradient, however, the bubbles will move down the gradient until a dislocation, grain boundary, or some other trap is reached. Once attached to a dislocation line or grain boundary, the bubble will experience forces if the dislocation is stressed or the grain boundary migrates. The maximum force exerted by a dislocation on a bubble is given by \(Gb^2\) where \(G\) is the shear modulus and \(b\) is the magnitude of the Burger's vector; the maximum force exerted by a boundary is given by \(\pi r \gamma_{gb}\) where \(r\) is the bubble radius and \(\gamma_{gb}\) the grain boundary energy. For steels the grain boundary force exceeds the dislocation force for bubbles larger than about 15\(\text{\AA}\) radius, thus bubbles larger than this will tend to be held at grain boundaries once swept there by dislocations, but if the boundary migrates the bubbles will tend to be carried along with it. Once in the boundary, easier diffusion will enable smaller bubbles to coalesce with the larger ones.

The velocity \(V\) at which the bubbles move under a force \(F\) assuming a surface diffusion mechanism, is given by:

\[
V = \frac{9 \eta \Omega^{4/3} D_s F}{3 \pi r^4 kT}
\]

where \(\eta\) = atomic volume
\(r\) = bubble radius
\(D_s\) = surface diffusion coefficient
\(kT\) has the usual meaning.

The inverse fourth power dependence of velocity on bubble radius shows that helium bubbles will quickly become immobile once they grow to an appreciable size.
1.2.3(d) The Effect of Irradiation on Precipitates and Phase Stability

Irradiation can have a marked effect on processes which bring about structural changes in metals\(^{(117-119)}\). Such processes are:

1. Diffusion
2. Precipitate dissolution and order/disorder effects
3. Re-solution of gases
4. Precipitate nucleation
5. Precipitation of unidentified or non-equilibrium phases

These changes occur as a consequence of the displacement of atoms by collision processes (described in section 1.2.2). The type and magnitude of the changes produced are therefore dependent to some extent on the characteristics of the irradiation treatment.

1. **Diffusion**

The principal diffusion mechanism in metals is the migration of solute atoms by interaction with vacancies. Thermal and fission neutron irradiations can produce a vacancy population greater than the thermal equilibrium value, leading to higher diffusion rates. The irradiation-modified diffusion coefficient is therefore greater than the normal value and varies either linearly or as the square root of the damage rate depending on the type of vacancy sink operating\(^{(120-121)}\). Since the damage rate is much higher in fast reactors the enhanced diffusion effect will be more important here than in thermal reactors. The irradiation-modified diffusion coefficient may vary by several orders of magnitude depending on damage rate and vacancy sink mechanism.

2. **Precipitate Dissolution and Order-Disorder Reactions**

A collision event involving a precipitate atom may result in the atom gaining enough momentum to escape from the precipitate particle altogether, if the collision is energetic enough. (Collisions involving thermal neutrons, which only produce vacancy/interstitial pairs are not likely to result in dissolution). The formation of displacement spikes may result in disordering of a previously ordered structure. On the other hand
the irradiation enhanced diffusion rate may enable precipitation to occur or the structure to reorder faster than it could in the absence of radiation damage, so that a balance may be achieved between these competing processes. This will still be true even at elevated temperatures where normal thermal diffusion predominates, so that the structure may be different from that expected from the equilibrium phase diagram.

Consideration of the balance between precipitate dissolution and growth indicates that whether dissolution or growth predominates depends on the precipitate size; below a critical size all precipitates will grow and above this critical size they will shrink, so that given enough time for equilibrium to be established a distribution of uniformly-sized precipitates should be obtained. However, in the case of carbide precipitates in steels the alloy may not contain enough carbon for the predicted precipitate size to be reached, and furthermore very large doses, corresponding to several hundreds of displacements per atom, may be required to establish equilibrium. Such high doses could be reached using particle accelerators but are not reached in reactor.

In contrast to the foregoing there is one interesting instance where a uniformly sized precipitate structure has been formed after a dose corresponding to only 6 displacements/atom. This is the $\gamma'$ (gamma prime) precipitate in the Nimonic alloy PE16. A more efficient dissolution mechanism has been advanced to explain this effect: the $\gamma'$ precipitate has an ordered structure which is locally destroyed by displacement spikes resulting in local high solute concentrations, which at the precipitate surface result in solute diffusion into the austenitic matrix. Thus dissolution is accelerated, and the equilibrium precipitate size can be relatively quickly reached.

(3) Re-solution of gas

An analogous process to the dissolution of precipitates is the re-solution of gas present in voids or bubbles, a subject first studied in the context of nuclear fuels. A re-solution mechanism has been proposed to explain the discrepancy found
between the quantity of helium present in bubbles and that calculated to be present from burnup of the $^{10}$B isotope during thermal reactor irradiations of an 18/10/Nb steel (referred to in 1.2.3(c)). The amount of helium present from bubble observations was a factor of 5-10 less than that estimated from burnup calculations for boron contents from 2 to 97 ppm (wt). Although this could be partly accounted for by the difficulty of resolving very small bubbles, this could not entirely account for the difference since it implied a dispersion of numerous fine bubbles (<20Å) which would have produced a significant hardening effect and such an effect was not observed. The re-solution was explained as largely due to the energetic products of the $^{10}$B reaction which were able to produce a significant flux of energetic particles in localised regions owing to segregation of boron to grain boundary carbides. This was naturally more pronounced in the steels of higher boron content; in the case of the 97 ppm (wt) boron steel the probability of re-solution was so high near grain boundary carbides that no bubbles were formed within the α-particle recoil distance (≈2.5μm) around the carbide particles. In this case most of the bubbles were intragranular.

(4) Precipitate Nucleation

Point defect clusters are thought to act as nucleation centres in some cases; increased diffusion rates during irradiation may also accelerate homogeneous precipitation of second phases.

Enhanced formation of carbides as a consequence of irradiation has been noted in several stainless steels. Additional precipitation of carbides has been found in 20/25/Nb steel as a consequence of irradiation at temperatures above 600°C and similar results have been found for a 16/12/Nb austenitic steel. In the latter case it appears that vacancies are responsible for the enhanced precipitation since matrix and stacking fault precipitation was observed, while in thermal control specimens only dislocation precipitation was observed. It was proposed that the required vacancy concentration could be formed at the centre of a displacement spike and last long enough to enable stable solute atom clusters to form, which subsequently grow.
into carbide crystals. The precipitate-free zones sometimes found adjacent to grain boundaries in stainless steels may be absent in the irradiated steel, e.g. in 20/25/Nb\textsuperscript{(27)} indicating that irradiation produces sufficient vacancies to counteract the vacancy sink effect of the boundary.

(5) Precipitation of unidentified or non-equilibrium Phases

The formation of non-equilibrium or unknown phases in fast reactor structural alloys has been widely reported\textsuperscript{(128-131)}. An example is an unknown acicular precipitate in AISI 316 austenitic steel\textsuperscript{(131)}. Ferrite formation has been found in several austenitic steels having compositions around 18\%Cr and 10\%Ni. The significance of this for fast reactor structural materials based on this composition has been examined\textsuperscript{(132)}. These steels are near the $\alpha - \gamma$ Fe-Cr-Ni phase boundary at about 600°C before irradiation and it has been suggested that irradiation promotes nucleation and growth of ferrite\textsuperscript{(117)}. It is possible that migration of ferrite stabilising elements such as titanium, chromium or silicon to the ferrite occurs; this segregation may affect the void swelling characteristics of the austenite. (Void swelling is discussed in 1.2.4(c))
1.2.4 The Effect of Neutron Irradiation on Mechanical Properties

1.2.4(a) Irradiation Hardening and Embrittlement

Metals and alloys irradiated and tested at temperatures below about half the absolute melting temperature exhibit marked differences in hardness and tensile properties compared with the unirradiated material. In austenitic stainless steels and nickel base alloys the principal effects are increases in hardness, yield and ultimate strengths and a reduction of ductility. In ferritic steels there is additionally an increase in the notched bar ductile/brittle transition temperature and a decrease in shelf energies. As an example the effect of irradiation at 95°C on the ambient temperature stress/strain curve for an austenitic steel (AISI 347) is shown in Fig.1.8. It may be noted that a distinct yield point is present in the irradiated steel and that the rate of strain hardening is less than in the normal steel.

Some tensile test data for 20/25/Nb steel irradiated at 40°C to a dose of $2.5 \times 10^{20}$ n/cm$^2$ (thermal) and $8 \times 10^{19}$ n/cm$^2$ (fission) and tested at ambient temperature are given in the following table:

<table>
<thead>
<tr>
<th>CONDITION</th>
<th>0.2% PROOF STRESS</th>
<th>UTS</th>
<th>% ELONGATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unirradiated</td>
<td>30 ksi</td>
<td>84 ksi</td>
<td>48</td>
</tr>
<tr>
<td>Irradiated</td>
<td>80 ksi</td>
<td>98 ksi</td>
<td>27</td>
</tr>
</tbody>
</table>

TABLE 1.4
EFFECT OF IRRADIATION ON MECHANICAL PROPERTIES OF 20/25/Nb STEEL

A large increase in the nominal yield stress (the 0.2% proof stress) is apparent, yet there is still considerable ductility in the irradiated steel. The irradiation hardening effect can be explained qualitatively by the interaction between glide dislocations and the damage structure, the damage structure offering obstacles to the progress of glide dislocations on active slip planes. Calculations indicate that single scattered vacancies can only produce a small degree of hardening whereas vacancy clusters produced by annealing quenched f.c.c. metals produce significant yield stress increases. In
lithium fluoride crystals vacancy-interstitial pairs or complexes cause pronounced hardening. Dislocation loops form by the collapse of vacancy clusters or the conglomeration of interstitial impurity atoms. Thus it appears that point defects must occur as clusters, dislocation loops or vacancy-interstitial complexes to have a significant hardening effect.

An unusual feature of the deformation of irradiated metals and alloys is the formation of "channels" about 0.1 - 0.2 μm wide in which intense slip occurs, the damage structure being swept away in these zones. The concentration of slip in narrow channels results in the formation of coarse slip bands at the surface. The slip concentration occurs because the initial dislocation flow results in softening of the channel material as the damage structure is removed, so making an easy path for subsequent dislocation movement. Slip will continue in the channel until the flow stress increases to equal that of the adjacent radiation-damaged material.

The mechanism of channel formation is not fully understood. A model has been proposed based on the operation of a dislocation source in the channel. The theory proposes that the stress required to sustain the growth of a dislocation loop decreases as the loop diameter increases, partly due to a reduction in line tension and partly due to a reduction in back stress as the damaged structure is removed. Later dislocations emitted by the source catch up with the earlier ones due to the softening of the channel material. Thus a pile-up is formed which leads to unstable growth of the slip band. Some mechanisms have been proposed for the elimination of dislocation loops in the channel.

The channeling phenomenon enables qualitative explanations to be advanced for the ductility loss and decrease in strain hardening index found in irradiation-hardened metals and alloys. The elimination of dislocation obstacles in the channel and prevention of dislocation interaction between different slip systems by the confinement of slip to the channel result initially in a low strain hardening index. Strain hardening
may increase at higher strains as dislocations networks form in the channels. The ductility loss can be explained by (1) the exhaustion of slip in the channels, so that the overall deformation before fracture is low, (2) plastic instability caused by the low strain hardening index. Plastic instability is particularly important where ductile fracture occurs by necking of the ductile matrix between hard second phase particles or inclusions. A low strain hardening index in the matrix material will favour rapid necking down and fracture of the ligaments between the particles, leading to early onset of fracture and consequent low ductility.

Irradiation hardening and embrittlement in iron and ferritic steels has recently been reviewed. The increase in the ductile/brittle transition temperature in b.c.c. metals caused by prior irradiation may be accounted for qualitatively by the differing effect of irradiation on the yield stress \( \sigma_y \) and cleavage fracture stress \( \sigma_c \). The cleavage stress is virtually unaffected by irradiation, while the yield stress is markedly increased. The ductile/brittle transition occurs when \( \sigma_c = \sigma_y \); at higher temperatures \( \sigma_c > \sigma_y \) and plastic deformation occurs, at lower temperatures \( \sigma_c < \sigma_y \) and brittle fracture is favoured. In irradiated steels \( \sigma_y \) is increased, so that the temperature of intersection of the \( \sigma_y \) and \( \sigma_c \) curves is raised (Fig. 9) resulting in an increase in the ductile/brittle transition temperature.
Elevated Temperature Irradiation Embrittlement

This subject is extensively reviewed in the references cited earlier. "Elevated temperature" in this context means temperatures at which significant structural changes brought about by diffusion processes can occur over times of the order of hours or days. For austenitic steels, elevated temperatures are 500-600°C and upwards; at these temperatures the defect structure produced by neutron irradiation is removed by point defect diffusion, consequently there is no radiation hardening and the ambient temperature mechanical properties are indistinguishable from those of unirradiated steel. The same effect may be obtained with radiation-hardened steel by post-irradiation annealing. However, a steel both irradiated and subsequently tested at elevated temperature will in general exhibit some degradation of mechanical properties; examples of properties affected are ductility, time to failure in stress-rupture tests, number of cycles to failure in fatigue tests.

The effect of test temperature on the post-irradiation tensile properties of 20/25/Nb steel is illustrated in Fig. 1.10. The radiation hardening effect disappears above test temperatures of 500-600°C, but the ductility is not recovered.

It is generally accepted that elevated temperature irradiation embrittlement is associated with an increased tendency to intergranular fracture over that of the unirradiated material. This tendency may be explained by the unstable growth and coalescence of the helium bubbles located in the grain boundaries: the bubbles absorb vacancies from the boundary and if the resolved normal stress across the boundary is large enough, equilibrium cannot be attained and the bubble enlarges continuously until it impinges on a neighbouring bubble and coalescence occurs.

Experiments in which helium has been injected into stainless steel by a particle accelerator have demonstrated that a ductility loss ensues at elevated temperatures.
The energetics of bubble growth are analysed as follows:

Assuming a spherical grain boundary helium bubble, the equilibrium condition in the absence of any applied stress is given by:

\[ P_0 = \frac{2 \gamma}{r_0} \tag{1} \]

where \( P_0 \) is the gas pressure, \( r_0 \) the bubble radius and \( \gamma \) is the surface energy. If a normal stress \( \sigma \) is applied it assists the gas pressure in expanding the bubble, therefore for equilibrium

\[ \sigma + P = \frac{2 \gamma}{r} \tag{2} \]

where \( r \) and \( P \) are the new equilibrium radius and pressure respectively. Since the mass of gas in the bubble is assumed to remain constant:

\[ P_0 r_0^3 = Pr^3 \tag{3} \]

Hence combining equations (2) and (3)

\[ \sigma = \frac{2 \gamma}{r} \left[ 1 - \frac{r_0^2}{r^2} \right] \tag{4} \]

The relation between applied stress across a grain boundary and equilibrium bubble radius calculated from equation (4) is illustrated in Fig.1.11. The maximum in the \( \sigma/r \) curve represents the critical stress and bubble radius at which growth becomes unstable (i.e. growth continues without any increase in stress). Since \( \sigma/\sigma = 0 \) at the maximum, the critical radius \( r_c \) is given by \( \sqrt[3]{3r_0} \) by differentiation of equation (4). The critical stress is then given by

\[ \sigma_c = 0.76\gamma/r_0 \]

For an empty void \( P=0 \) and so from equation (1) the critical stress for unstable growth would be:

\[ \sigma_c = 2\gamma/r_0 \]

Hence the effect of an internal gas pressure is to lower the stress needed for unstable growth by a factor of approximately three. The grain boundary bubbles will have the greatest
effect on elevated temperature properties for three main reasons:

1. Grain boundaries are preferred bubble nucleation sites.
2. Deformation at or near boundaries can result in stress concentration that will assist bubble growth.
3. Grain boundary bubbles will grow faster because of the higher grain boundary diffusivity compared with the matrix.

The UTS of an austenitic stainless steel at 750°C is typically 30 ksi. Taking the grain boundary energy as 1500 dyne/cm, the minimum bubble diameter for unstable growth is about 100Å; grain boundary bubbles of this size or larger are commonly found in irradiated steels. Consequently if the applied stress is high enough the bubbles can grow continuously at a rate determined by the stress and temperature.

If the mean spacing of bubbles of critical size or larger on a grain boundary is \( a_m \), then the grains elongate by an amount \( a_m \) before bubble coalescence occurs and so the minimum tensile ductility \( \varepsilon \) is given by:

\[
\varepsilon = \frac{a_m}{d}
\]

where \( d \) is the mean grain diameter. This simple relation ignores the contribution of other deformation processes such as intragranular deformation and grain boundary sliding, therefore the calculated ductility represents a minimum value.

The kinetics of bubble growth has been studied using a model developed earlier for the growth of grain boundary cavities during creep. It was found that a reasonable agreement existed between calculated and experimental times to rupture in stress-rupture tests on an 18/10/Nb steel. The calculated and experimental ductilities also agreed fairly well bearing in mind that the calculated values are minimum values. A more sophisticated cavity growth model has been applied to gas bubble growth and again the calculated growth rates are of the right order.
It is difficult to assess the value of these calculated growth rates since both models make simplifying assumptions whose degree of justification is uncertain, for example it is assumed that all bubble nuclei are the same size and are formed at the same time, and also that the grain boundary is isotropic with respect to vacancy formation. In addition the grain boundary energy and diffusivity are not known accurately.

The principal experimental evidence supporting the bubble growth model of elevated temperature embrittlement may be briefly summarised as follows:

(1) The link between boron content, and hence helium content, in thermal reactor irradiated steel, and embrittlement expressed in terms of reduction in elongation at fracture and reduced times to failure in stress-rupture tests is well established\(^{(112)}\).

(2) Helium injected by a particle accelerator produces elevated temperature embrittlement in austenitic steel\(^{(148)}\).

(3) Fractographic examination of chemical-vapour deposited tungsten containing gas bubbles at grain boundaries demonstrated that enough growth could occur under suitable conditions of temperature and stress for bubble coalescence to occur\(^{(153)}\).

In irradiated austenitic stainless steels, however, the mechanism of embrittlement may be more complex than suggested by the bubble growth and coalescence model. In particular, fractographic examination of 20/25/Ni-Ti stabilised steel has indicated that although some growth of the larger grain boundary bubbles occurs\(^{(154)}\) there is no evidence of large cavities forming by bubble coalescence as in tungsten. Both the treatments of bubble growth kinetics require a direct dependence of bubble growth rate on the applied stress, and hence an inverse dependence of time to rupture on applied stress, but no simple relation of this type has been reported for stainless steel in the range of temperature of interest. A study of the fracture behaviour of an irradiated unstabilised 20/25 steel showed that failure occurred by wedge cracking, the loss of ductility being associated with an increase in crack propagation.
rate (155-156). Other work on irradiated niobium-stabilised 20/25 steel (157) has shown that at high stresses and relatively low temperatures the ductility loss is a consequence of increased crack propagation rates, while at higher temperatures and lower stresses failure occurs by grain boundary cavitation. Thus there is some evidence that at least at the lower end of the elevated temperature range the irradiation embrittlement is associated with crack propagation phenomena rather than with cavity growth and linkage. Some explanations advanced for the embrittlement under these conditions are:

1. Impurities other than helium formed by transmutation reactions may reduce surface energies sufficiently to promote intergranular fracture and low ductility. However, this is unlikely to be a primary cause of embrittlement since experiments employing particle accelerators to inject helium into steel have demonstrated that embrittlement results.

2. Intergranular helium bubbles may obstruct the motion of grain boundary dislocations, so preventing sliding and relaxation of stress at triple points, particularly at lower strain rates (158). Thus an increased tendency to wedge crack formation and enhanced propagation rates should be observed.
1.2.4(c) Irradiation Creep

Irradiation creep was first discovered in the uranium metal fuel used in the early thermal reactors but it has subsequently been shown that this phenomenon is also exhibited by graphite, ceramic fuels (UO₂) and non-fissile metals and alloys such as zirconium alloys and austenitic materials. The magnitudes of the irradiation creep in austenitic alloys used for the core components are of major concern with respect to the design, operation and performance of fast reactors, because as in the case of void swelling, the strains can lead to possible interference between adjacent fuel element sub-assemblies.

The irradiation creep of austenitic steels and nickel base alloys has been investigated using helical tension springs, uniaxial tensile tests, internally pressurised tube experiments and stress relaxation tests. The results of these investigations have demonstrated that irradiation creep occurs at significantly lower temperatures and stresses than thermal creep; furthermore, the rates are generally linearly dependent on both the stress and the neutron or displacement dose, at least up to about 30 displacements per atom (dpa), but are only weakly dependent on temperature in the range 280°C to 500°C (approx). However, the steady state irradiation creep rates are markedly less dependent on the material and initial thermomechanical treatment, than the void swelling, but such factors can produce significant variations in the transient creep characteristics.

Numerous mechanisms have been proposed to account for the irradiation creep in non-fissile metals and alloys but as yet no fully accepted and experimentally proven model has been evolved. The currently favoured mechanisms are those based on a pure dislocation climb process such as the stress-induced preferential absorption of point defects by dislocations (see for example ref 168) and a dislocation climb and glide model, termed I-creep.
The basis of the former model is the existence of an induced interaction energy between a point defect and a dislocation under the action of a uniaxial stress which in turn depends on the orientation of the dislocation to the applied stress direction. Thus, it has been shown that the irradiation creep strain due to this process is given approximately by (68):

\[ \epsilon = \frac{0.5 \sigma K t}{\mu} \]

where \( \sigma \) is the applied stress, \( \mu \) is the shear modulus, \( K \) is the atom displacement rate and \( t \) is the time. It is claimed that only this mechanism contributes to irradiation creep at temperatures up to approximately 400°C, the rate being independent of temperature, dislocation density and other material parameters. It has been proposed, however, that thermal emissions of vacancies from vacancy loops, nucleated within the displacement cascades, occurs in the temperature range 400 to 500°C, with the result that the irradiation creep strain of cold worked austenitic steels is approximately doubled on increasing the temperature in this range (170).

The I-creep mechanism is analogous to Cottrell creep in \( \alpha \)-uranium. It basically involves the immobilisation of the irradiation induced vacancies in, for example, voids: the excess interstitials migrate to edge dislocations and produce climb of the dislocation segments into regions where they can bow out under the externally applied stress to produce small amounts of anelastic strain. The sum of the anelastic strain increments produces an irradiation creep rate given by (67):

\[ \dot{\epsilon} = \frac{\sigma \dot{s}}{\mu} \]

where \( \dot{s} \) is the swelling rate and \( \mu \) is the yield stress. This model therefore predicts that the irradiation creep rate is directly proportional to the void swelling rate and also that the creep rate increases by a factor of two on increasing the temperature from about 280 to 450°C (171).

It has been suggested that both the stress-induced preferential absorption and I-creep mechanisms contribute to the irradiation creep of fast reactor irradiated austenitic alloys (172) but insufficient data are available at present to confirm or otherwise the hypothesis that the irradiation creep of austenitic alloys is enhanced by void swelling.
FIG. 1.7 DISPLACEMENT SPIKE

FIG. 1.8 STRESS/STRAIN CURVES FOR IRRADIATED & UNIRRADIATED 347 STEEL
FIG. 1.9 SCHEMATIC DIAGRAM OF THE EFFECT OF IRRADIATION ON DUCTILE/BRITTLE TRANSITION

FIG. 1.10 EFFECT OF IRRADIATION ON PROPERTIES OF 20/25/Nb STEEL
FIG. 1.11 EFFECT OF STRESS ON EQUILIBRIUM BUBBLE RADIUS

FIG. 1.12 CAVITY GROWTH RATE (CALCULATED)
1.3. LOW-CYCLE FATIGUE

1.3.1. Low-Cycle Fatigue in Metals and Alloys

Metal fatigue has been the subject of research for a long period and in this time three fairly well defined areas of study have emerged:

(1) The effect of cyclic loading on the structure and mechanical properties of the material.

(2) The nucleation and propagation of fatigue cracks and the study of their structure.

(3) Empirical correlations and theoretical models of fatigue cracking, the goal being to predict the fatigue life of a specimen or component under given conditions of cyclic loading strain.

In this section the essential features of LCF are discussed according to the above scheme.
The counterpart of the tensile stress/strain diagram in LCF is the "hysteresis loop", so called because of its resemblance to the B/H hysteresis loop of magnetic induction. A schematic LCF hysteresis loop is shown in Fig. 1.13 (page 81) for an elastic-perfect plastic material (no work hardening). At the start of the test, assuming tensile loading, the material follows the normal tensile stress/strain curve until the point A is reached at maximum strain. Here the crosshead motion is reversed and unloading occurs; at point B the load and elastic strain are zero, and only the plastic strain remains. As the crosshead motion continues, compressive elastic and plastic strains are applied until point C is reached at maximum compressive strain. Here the crosshead motion reverses again and unloading occurs down to point D. Finally, tensile strains are applied until point A is reached again. In this example, strain has been the control parameter, but stress could equally well have been chosen. The two LCF testing modes are therefore called strain- and stress-cycling respectively. The hysteresis loop can be considered to be made up of two stress/strain curves, in Fig. 1.13 (page 81) from point A to C, and from point C to A. Consequently it is convenient to define stress and strain in terms of the range rather than in tensile and compressive components. The distance A-C along the strain axis is therefore the total strain range, and along the stress axis the stress range. The distance B-D along the strain axis corresponds to the plastic strain range.

Initially softened materials will strain harden under cyclic straining, and initially cold worked materials will tend to strain soften. If the strain-cycling LCF mode is used, the stress range will increase during the course of the test, for initially soft material, and decrease for initially worked material (Fig. 1.15). In the same way, under stress-cycling conditions the strain range will decrease for softened material and vice-versa for the cold worked condition (Fig. 1.14).
eventually reach a steady-state. The peak stress and strain values, \( \Delta \sigma/2 \) and \( \Delta \varepsilon_p/2 \), may then be plotted to give the cyclic stress/strain curve which can be compared directly with the corresponding tensile stress/strain curve, for material in the same initial condition. Certain metals having moderate to high stacking fault energies have unique cyclic stress/strain curves independent of the starting condition of the material \((173)(174)\).

A cyclic strain hardening exponent, \( \beta \), analogous to the tensile strain hardening exponent, may be defined if the material has a power law type of constitutive equation:

\[
\Delta \sigma = k \Delta \varepsilon^\beta
\]

where \( k \) is a proportionality constant or "strength parameter". Similarly, a strain rate sensitivity exponent, \( m \), may be defined if a power law dependence of flow stress on strain rate exists:

\[
\Delta \sigma = A \varepsilon^m
\]

where \( A \) is again a proportionality constant.

In single phase alloys \( m \) usually increases with decreasing grain size in the micron region.
Effect of Cyclic Strain on Dislocation and Slip Band Structure

Studies of dislocation structures arising from cyclic straining at ambient temperatures have been reviewed by Grosskreutz (174). A very high density of dislocations can be achieved during cyclic deformation, one to two orders of magnitude greater than that attained during tensile testing to the same flow stress. In copper, bundles of dislocations form during cyclic straining and their density increases with increasing cycles. Bundles are believed to form as a result of trapping and annihilations of screw components. Dislocations in the bundles cannot take part in slip, so new ones are generated and pinning in the clear areas can lead to the formation of new bundles, as the strain increases with increasing cycles. If the plastic strain range is greater than about 0.4%, the dislocation bundle structure is replaced by a cell structure when steady state is reached. The cell size appears to be directly proportional to the total strain range. The cell structure formed during cyclic deformation is very similar to that formed during tensile deformation, except that in the former the cell walls consist mainly of loops and dipoles instead of tangles.

A widely observed phenomenon (in materials having moderate to high stacking-fault energies) in both HCF and LCF is the formation of persistent slip bands at temperatures below those at which significant diffusion occurs. Persistent slip bands are softer than the surrounding matrix (175); the dislocation structure within the bands consists of very small cells. It therefore appears that the material in the band experiences locally a higher strain range than existing in the surrounding matrix.

Extrusions and intrusions form within these bands. Extrusions form by the ejection of a thin tongue of material from the persistent slip band during some tens of cycles from the start of cycling (176). Intrusions appear to form by the growth of narrow holes to different depths into the band (177), subsequently joining up to form a microcrack. This microcrack corresponds to the Stage I crack observed initially at 45° to the applied stress direction in HCF (178).
1.3.1(c) Grain Boundary Structure

Changes in grain structure during LCF are observed to occur at elevated temperatures. An unusual effect is the migration and reorientation of the grain boundaries to positions at $45^\circ$ to the applied stress direction, leading to the formation of a "square", "orthogonal" or "diamond" grain structure. Although grain boundary migration (GBM) is observed in HCF at elevated temperatures, it does not result in reorientation of the boundaries. The same is true for creep even when a completely recrystallised grain structure forms. In LCF, GBM does not necessarily stop once the square structure is formed; in very pure copper further migration is observed, leading to a coarser square grain structure. Grain boundary sliding, migration, and square grain formation generally occur concurrently. A dislocation model of GBM is described in Chap. 3. It has been proposed that square grain formation is a consequence of grain boundary sliding. Boundaries oriented at $45^\circ$ experience the maximum shear stress and so grow by sliding at the expense of other boundaries. In pure aluminium, sliding on boundaries in the $45^\circ$ directions produced "folds" (regions of intragranular shear) at triple points. The $45^\circ$ boundary then grew along the fold, so increasing the proportion of boundary oriented at this angle. Folds have not, however, been reported in studies on pure $\alpha$-Fe and copper. Early studies on lead indicated that the square structure formed by a recrystallisation mechanism. Highly deformed material near boundaries oriented close to the $45^\circ$ directions recrystallised to form new strain-free grains having boundaries oriented in these directions. In $\alpha$-Fe, the dislocation cell structure was found to have a square appearance similar to the grain boundaries. The square structure formed very rapidly at 700°C, a stable grain configuration being formed after only 5 cycles at a total strain range of 4%. In all the materials studied the square grain pattern appears to be stable; post test annealing does not lead to a rapid recovery of an equiaxed structure.
1.3.2. **Low Cycle Fatigue Fracture Characteristics**

At ambient temperatures LCF crack nucleation takes place at the surface of the specimen, within persistent slip bands. Growth is rapid and propagation changes to $90^\circ$ to the applied strain direction; the Stage I type of growth observed in HCF is rarely observed in LCF. LCF growth at $90^\circ$ to the applied strain direction (Stage II growth) corresponds to crack propagation rather than nucleation. In contrast to HCF, therefore, very little time is spent in crack nucleation in LCF.

When a cracked body is loaded, the constraint experienced by the material at the crack tip results in a triaxial stress state there. If the material is ductile, plastic flow will then occur to relax these stresses, and blunting of the crack tip will result. Further straining produced more blunting and so more dispersal of the crack tip strain concentration through a greater volume of material. Thus in a soft, ductile material (e.g. pure aluminium) very large strains must be applied to obtain crack propagation under tensile loading; the material has a low notch sensitivity. Under fatigue loading, however, crack propagation will occur under low applied strains. Under reversed loading, the crack tip is resharpened under the influence of the compressive strain. Thus the strain concentration at the crack tip is maintained and the crack propagates during each tensile half-cycle. It is not necessary for the applied load to be completely reversed during each cycle: fatigue crack propagation will still occur under a mean tensile stress since the compressive residual stress generated during reversed loading will sharpen the crack tip (Fig. 1.16) (page 82).

The crack tip opening and blunting mechanism described above is believed to be responsible for the formation of the striations observed on LCF fracture surfaces, in tests at ambient temperatures. A one-to-one correspondence between fatigue cycles and number of striations has been established for an aluminium alloy in a now classic programme loading experiment (185). Growth rates calculated from striation spacings in titanium were shown to be in excellent agreement with crack
length measurements\(^{(186)}\). In a recent study of LCF in AISI 316 stainless steel\(^{(187)}\), good agreement was found between macroscopic crack growth per cycle and striation spacing for plastic strain ranges above the fatigue limit at 625°C. Below this strain level, i.e. under HCF conditions, each striation corresponded to several fatigue cycles. Other work on HCF of AISI 316\(^{(188)}\) confirms the latter observation and the discrepancy between striation spacings and macroscopic growth rates in HCF has been found also in steels\(^{(189)}\), maraging steels\(^{(190)}\) and HS188 alloy\(^{(191)}\). A variety of explanations have been advanced to explain this effect including the accumulation of strain by repeated cycling ahead of the crack tip\(^{(192)}\), strain induced transformations leading to crack re-initiation\(^{(193)}\) and slip band hardening\(^{(188)}\).

Striation formation does not occur in elevated temperature LCF if the temperature is high enough for diffusion effects to be important: instead intergranular fracture occurs. Grain boundary sliding may nucleate triple point cracks, or intergranular growth of surface nucleated cracks\(^{(194)}\). In addition grain boundary voids may nucleate, grow and coalesce to form intergranular cracks\(^{(195)}\)\(^{(196)}\). Creep processes in general become more important than the purely mechanical processes of lower temperatures. No regular structure is apparent as a rule on the fracture surfaces under these conditions and so fractography offers little information on crack tip mechanisms in this case.
1.3.3. The Empirical and Theoretical Description of Low Cycle Fatigue

1.3.3(a) Empirical Methods

The number of cycles of reversed loading applied before failure occurs is termed the "endurance". Failure here does not necessarily mean complete rupture of the component or test piece; it may instead correspond to the development of a crack of a certain size or the reduction of the net cross-section to some predetermined level. The applied stress or strain range may be plotted against endurance to give a "fatigue curve". In HCF tests the stress range is always the variable employed, the results being presented in the familiar stress log endurance (S/N) curve. In LCF work, strain is the variable most often used, partly because no pre-hardening period is required as in many stress-cycling tests, and also because using strain cycling avoids cyclic creep which can occur in a stress-controlled test especially at high ranges leading to permanent deformation. Coffin and Manson showed that a log, plastic strain range against log, endurance plot yielded a straight line relationship for many materials. Therefore LCF obeys a power law relation (the Coffin-Manson Law) of the form:

$$\Delta\varepsilon_p N^a = \text{constant}$$

where $\Delta\varepsilon_p$ is the plastic strain range (also called amplitude), $N$ is endurance, $a$ is the Coffin-Manson exponent (also called endurance exponent) and the constant is determined by the value of $\Delta\varepsilon_p$ at $N = 1$ cycle. $a$ is the slope and is negative (Fig.1.17) (page 82).

This simple relation between $\Delta\varepsilon_p$ and $N$ in LCF is in contrast to the S/N HCF curve which cannot be described in a simple manner for a wide range of materials in the same way as the Coffin-Manson line. This suggests that although the Coffin-Manson Law is empirical, it has a fundamental significance.

Coffin also proposed that LCF data could be correlated with tensile ductility. A tensile test may be regarded as a LCF test lasting for only $1/4$ cycle. Similarly, the true tensile ductility ($\ln A_o/A_f$) may be regarded as the plastic
strain range in this test. Using this reasoning, Coffin plotted tensile ductilities at 1/4 cycle LCF tests and found quite good correlation with the LCF data for the same material (Fig.1.17). Furthermore, Coffin noted that $\alpha$ for many materials was approximately 1/2, so putting $D$ (ductility) equal to $\Delta \varepsilon_p$, and $N = 1/4$:

$$D \left( \frac{1}{4} \right)^{\frac{1}{2}} = \text{constant} = D/2$$

So that the constant in the Coffin-Manson Law is given by one-half the tensile ductility. Later work, however, showed that the exponent $\alpha$ was more nearly equal to 0.6 for most materials $^{(200)}$, and the identification of the constant with half the tensile ductility appeared to be largely fortuitous. Other attempts have been made to interpret the constant in terms of the tensile ductility but there does not appear to be a convincing relationship $^{(201)}$. It is now generally accepted that the exponent $\alpha$ is a material constant and not a universal one. A relationship similar to the Coffin-Manson Law for total strain range and endurance has been found $^{(179)}$ although the relation breaks down for some materials at low strain ranges (endurances greater than about $10^5$) $^{(202)}$. The LCF properties of strong alloys generally correlate better with total strain range $^{(205)}$. This could be expected since for such materials the ductility is often low, necessitating low plastic strain ranges so that the elastic strain is a large proportion of the total strain.

The influence of temperature and strain rate on the endurance exponent has received some attention. In general, $\alpha$ decreases with increasing temperature and decreasing strain rate $^{(204-206)}$. Consequently there is a more rapid fall in endurance with increasing plastic strain range, although exceptions to this behaviour have been noted $^{(207)}$. The Coffin-Manson constant likewise decreases under these conditions, so that there is an overall general reduction in endurance.
"Crack propagation rate" is defined as the rate of increase of crack length with number of fatigue cycles and often written $\frac{da}{dN}$ where $a$ is the crack length and $N$ the number of fatigue cycles. Crack propagation rate and applied stress range are often correlated in HCF studies using linear elastic fracture mechanics (LEFM). There have recently been attempts to apply this approach to tertiary creep crack propagation and to LCF. The fundamental parameter of LEFM is the stress intensity factor $K$, a constant which describes the overall level of stress in the vicinity of a crack tip. $K$ is not a stress itself nor is it a stress concentration factor. For purely elastic strains the stress field around a sharp crack is given by the Westergaard equation

$$\sigma_{ij} = \frac{K}{(2\pi r)^{1/2}} f(\theta)$$

where $r$, $\theta$, are polar co-ordinates. The stress intensity factor is therefore a constant of proportionality depending on the macroscopic applied stress, specimen geometry and crack shape and dimensions. Standard solutions for $K$ are available for many different conditions. Since the above equation requires that $\sigma \to \infty$ as $r$ tends to zero, the stress field close to the crack tip cannot be determined since there is a singularity here. Also, since the equation is derived using elasticity theory, it is not valid within the plastic zone which must develop at the locus of points where $\sigma_{ij} = \text{yield stress}$. Therefore the LEFM approach is only valid when the plastic zone is small compared to the crack size in practice this means it should be limited to rather strong materials subjected only to elastic loading, known as small scale yielding conditions. As a rule of thumb the applied stress should not be more than $\approx 0.8$ of the yield stress. Some experimental proof of the $\gamma/(2\pi r)^{1/2}$ dependence of stress required by Westergaard's equation is available for an aluminium alloy and a silicon steel. Paris proposed on the basis of strain energy considerations that HCF crack growth rate should be related to stress intensity factor range $\Delta K$ by the equation $\frac{da}{dN} = C \Delta K^{\frac{1}{2}}$, where $C$ is a constant. Thus a log/log plot of crack propagation rate against $\Delta K$ should yield a straight line of slope $\frac{1}{2}$. This is
now a widely established method of presentation of fatigue test data, although the exponent of $\Delta K$ generally lies between 2 and 4. For simple specimen geometries and loading conditions, $K = \frac{\Delta \sigma}{\pi} \sqrt{\pi l}$ where $\sigma$ is the gross section stress, $l$ the crack length and $A$ a proportionality factor. For a through crack of length $2l$ in an infinite plate, $A = 1$, for an edge crack, length $l$, $A = 1.12$. In practice it is found that the $\Delta \sigma / \Delta N$ against $\Delta K$ plot has a sigmoidal rather than a linear shape. At a sufficiently low value of $\Delta K$ crack growth does not occur, i.e. there is a threshold for HCF crack propagation. It has been argued that the threshold corresponds to a propagation rate of one atomic spacing per cycle \(^{(186)}\), this being the least possible crack extension per cycle. At sufficiently high values of $\Delta K$, unstable crack propagation occurs, corresponding to brittle fracture. The limiting value of $K$ is the fracture toughness, $K_c$, at the point where the propagation rate becomes infinite (Fig.1.18).

1.3.3(c) Crack Opening Displacement

The crack opening displacement was proposed by Wells \(^{(213)}\) as a fracture criterion that would not have the same limitations as LEFM. According to this idea, the work done in extending the crack can be directly related to the surface energy and plastic work required to create unit area of new crack surface. The work done per unit volume at the crack tip is $\delta \sigma_y$, where $\delta$ is the crack opening displacement and $\sigma_y$ is the yield stress. Therefore:

$$G = \delta \sigma_y \quad \text{(for small scale yielding)}$$

where $G$ is the "energy release rate" or "crack extension force". Dugdale \(^{(214)}\) has shown that energy release rate, crack opening displacement, and stress intensity factor are related in the small scale yielding case by:

$$G = \delta \sigma_y = \frac{K^2}{E}$$

where $E$ is Young's modulus.

The crack opening displacement (COD), being applicable to loading where plastic strain occurs, should be more suitable for LCF work than stress intensity factors. COD has been applied to LCF in mild steel \(^{(215)}\). In the same work, a dependence of LCF crack propagation rate on COD squared was found; this would be expected since in HCF a fourth power dependence on $\Delta K$ is found.
for this material. There appears to have been little effort to apply the COD approach to LCF in general. This is probably because the measurement of very small crack opening displacements presents considerable practical difficulties.

1.3.3(d) The "J" Integral

This path-independent line integral discovered by Rice constitutes a generalisation of the Griffith energy balance approach to fracture problems. Whereas the Griffith theory is applicable only to elastic problems, the J integral may be applied to elastic-plastic fracture. The integral is:

$$J = \int \left( Wdx_1 - T_{x_1} \frac{\partial u}{\partial x_2} ds \right)$$

where \( W \) is the strain energy density function, \( T_{x_1} \) is the traction vector, and \( u \) is the displacement. \( ds \) is an element of the curve \( \Gamma \) and \( x_1 \) and \( x_2 \) are the axes normal and parallel to the crack plane respectively (page 83). The traction vector is defined by \( T_{x_1} = \sigma_{ij} \frac{n_j}{n_i} \) where \( n \) is a unit vector in the positive sense on the curve \( \Gamma \). The integral \( J \) may be defined as the rate of change of potential energy with crack length. For linear elastic conditions, \( J \) becomes equal to the energy release rate, \( G \). There is therefore a direct relation between \( J, G, \delta, \) and \( K \) under these conditions. The advantage of this energy balance method is that crack tip conditions can be characterised by measurements made remotely from the crack tip itself, since \( J \) is path-independent.

By analogy with the stress intensity factor relation for HCF, a relation incorporating \( J \) can be postulated for LCF:

$$\frac{da}{dN} = C \lambda J^m$$

where \( C \) and \( m \) are constants. An attempt has been made to apply \( J \) to LCF although there is some doubt whether \( J \) is valid for cyclic loading conditions. Unfortunately \( J \) cannot be calculated from simple expressions involving easily determined test parameters, in the way that \( K \) can be. Numerical or experimental techniques must be used which are time-consuming and this reduces the attraction of \( J \) as a means of correlating LCF data. However, methods have been developed for estimating \( J \) for simple geometries and loading conditions.
1.3.3(e) Geometrical Similarity Hypothesis

This relates crack propagation rate to crack length in ductile materials. The argument is basically that if all test parameters are held constant during a fatigue test, then there is only one variable left to which the crack propagation rate can be referred, namely the instantaneous crack length. Therefore propagation rate is proportional to crack length: \( \frac{dL}{dN} = C \) where C is a constant, called the propagation constant. The propagation constant therefore involves the plastic strain range, material properties, and other parameters. Under HCF conditions, \( L \) is related to \( K \) by:

\[
L = \frac{K^2}{(Const)} \frac{\sigma}{\sqrt{\pi}}
\]

(for a small crack). Therefore the geometrical similarity hypothesis corresponds to a \( \Delta K \) exponent of 2 in HCF.

The hypothesis is of limited value in interpretation of fatigue data since important variables such as plastic strain range are not explicitly included in it.

1.3.3(f) Models of Fatigue Crack Propagation

None of the previous treatments of the fatigue crack propagation process involve any model of the deformation mechanism at the crack tip, and consequently they all incorporate constants which must be determined experimentally. A number of fatigue theories have been developed, based on different mechanisms, such as adaptations of the Bilby, Cottrell, Swinden (BCS) dislocation/crack formation model, a crack blunting and sharpening model and a net area stress theory. All these theories are based on small scale yielding conditions and are not therefore directly applicable to LCF. However, a theory of LCF crack propagation has been developed by Tomkins based on the Dugdale equation for plastic zone size and a crack blunting and sharpening model. In this model the cyclic strain hardening exponent plays a central role and a possible connection between fatigue crack propagation rates and strain hardening properties has been suggested.

The LCF crack propagation rate is given by:

\[
\frac{dL}{dN} = \frac{\pi^2}{8} \left( \frac{k}{2\Delta T} \right)^2 \Delta \varepsilon_p (2\beta + 1)
\]

where \( \beta \) is the cyclic strain hardening exponent, \( T \) is the mean
tensile stress in the plastic zone and \( k \) is the strength parameter in the power law hardening equation \( \Delta \sigma = k \Delta \varepsilon^p \) (see section 1.3.1(a) p. 69). \( \bar{T} \) may be approximated by the UTS of the material as determined from a tensile test. The equation may be integrated to yield the endurance in terms of the initial and final crack sizes \( l_0 \) and \( l_f \):

$$
N = \frac{2}{\bar{T}} \left( \frac{K}{2\pi} \right)^2 \Delta \varepsilon_p (2\beta + 1)
$$

This can be written in the form:

$$
\Delta \varepsilon_p N^{1/(2 \beta + 1)} = \text{constant}
$$

This corresponds to the Coffin-Manson Law where the endurance exponent \( \alpha \) is now given by \( 1/(2 \beta + 1) \). The theory has been shown to describe successfully the plastic strain range/endurance behaviour of a number of materials at ambient temperatures.

At elevated temperatures, \( \beta \) tends to decrease, as also do \( k \) and \( \bar{T} \); thus the theory qualitatively predicts an increase in the slope of the Coffin-Manson line and a shift in its position corresponding to an overall lower endurance with increasing temperature. This appears to be true for 20/25/Nb(T6).
FIG. 1.13 HYSTERESIS LOOP PARAMETERS

FIG. 1.14 SCHEMATIC HYSTERESIS LOOP, STRESS CYCLING, MATERIAL ANNEALED BEFORE TESTING

FIG. 1.15 SCHEMATIC HYSTERESIS LOOP, STRAIN CYCLING, MATERIAL ANNEALED BEFORE TESTING
COMPRESSIVE RESIDUAL STRESSES CAUSE CRACK TIP SHARPENING DURING THIS HALF CYCLE.

FIG. 1.16 CYCLING WITH MEAN TENSILE LOAD

TENSILE DUCTILITY

CONSTANT

COFFIN-MANSON LINE

FIG. 1.17 COFFIN-MANSON LINE & TENSILE DUCTILITY

FIG. 1.18 THE HCF Log(da/dN) v. Log(ΔK) SIGMOIDAL PLOT
FIG. 1.19 ILLUSTRATING THE PATH OF THE "J" LINE INTEGRAL
1.3.4. Low-Cycle Fatigue of Unirradiated 20/25/Nb Steel

There appears to be no published work dealing in detail with the effect of cyclic strain on mechanical properties of 20/25/Nb steel or its effect on dislocation and slip band structure. The small literature available on the LCF properties of this steel is chiefly concerned with the fracture aspect and empirical and theoretical treatments of the fatigue endurance; there are some observations on changes in grain boundary structure.

1.3.4(a) Grain Boundary Structure

The formation of both square grain structure and grain growth during elevated temperature LCF of solution treated and aged 20/25/Nb steel tested in push-pull have been observed. Two features of the square formation not noted previously in other metals (see section 1.3.1(c)) were:

(1) The existence of a threshold plastic strain amplitude below which changes in grain structure were not observed. At the test temperature employed (750°C) this threshold amplitude appeared to be about 8%. In work on α-Fe, the rate of formation of the square structure was found to be strain amplitude dependent but no threshold was reported.

(2) The occurrence of grain growth or reorientation of the boundaries to the $45^\circ$ positions was markedly strain rate dependent in different ways for the two phenomena. Grain growth only occurred at the lowest strain rate employed ($10^{-5}$/s) and was not accompanied by boundary reorientation. On the other hand, at the high strain rate ($10^{-4}$/s) reorientation took place without any increase in grain size.

It appears therefore that there is a critical plastic strain amplitude and critical strain rate for square grain formation to occur in 20/25/Nb steel. The rate of formation of the square boundary structure was described qualitatively as follows:

(1) After 20 cycles: Grain boundary migration producing "wavy" grain boundaries.
(2) After 40 cycles: Noticeable reorientation of the boundaries.

(3) After 60 cycles: Complete square structure formed.

This rate of formation is comparable with that of α-Fe at 700°C tested at a total strain amplitude of 4%\(^{(183)}\). In α-Fe the square structure was complete after about 80 cycles. It seems possible therefore that the carbide precipitation in the 20/25/Nb alloy does not have much effect on formation of the square grain boundary structure, since the rate of formation is similar to that of pure iron at a similar temperature.

1.3.4.(b) Low-Cycle Fatigue Fracture Behaviour

At ambient temperature the rate of crack initiation is strongly dependent on the material surface condition\(^{(227)}\), as in high-cycle fatigue, and a similar mechanism to that operating in HCF has been proposed: intrusion/extrusion formation at stress raisers such as surface defects (see section 1.3.1(b)). An alternative mechanism may be sub-surface cracking along slip bands\(^{(228)}\), although if this mechanism is operating it is less obvious why surface condition should have much effect. At high strain amplitudes intergranular fracture has been observed even at 25°C\(^{(227)}\). This may be a manifestation of the extremely high dislocation densities which can be accumulated at grain boundaries in LCF\(^{(174)}\).

Studies of LCF fracture behaviour at elevated temperatures\(^{(227,229,230)}\) have shown that there is an increasing tendency to intergranular fracture with increasing temperature and decreasing strain rate with a concomitant reduction in endurance. The intergranular initiation process may depend on a certain inhomogeneous strain being built up at a boundary, the probability of a boundary acquiring such a strain being dependent on the overall applied plastic strain amplitude: therefore the lower the strain amplitude the fewer intergranular surface cracks should form. Such behaviour has been observed in 20/25/Nb steel\(^{(227)}\).

A detailed metallographic study of LCF specimens tested at elevated temperature has led to an unusual mechanism being advanced to explain crack initiation\(^{(230)}\). Grain boundary groove
formation was observed after about 10% of the fatigue life; a
delta shaped region, interpreted as being a newly created grain,
formed at the root of the groove. The groove then sharpened
into a surface crack which grew down one side of the delta region;
the delta disappeared as the crack propagated and widened. The
formation of the delta was explained either by:

(1) Grain boundary migration to a position at 45° to the applied
stress axis together with shear in the grain behind the
boundary.

(2) Nucleation of the new grain at the groove/boundary junction;
the delta region then growing in the stress field ahead of
the grain boundary groove.

No confirmatory work has been published on this topic. It
is accepted that on annealing cold-worked metals the nucleation
of new stress-free grains is energetically favourable, since
highly deformed material is removed by the recrystallisation
process, but it is less clear why grain nucleation should be
favoured under a crack nucleus. Although plastically deformed
zones form at crack tips at ambient temperatures, it is unlikely
that at elevated temperatures a plastic zone of sufficient size
to promote recrystallisation could exist at the tip of a small
crack nucleus.

Elevated temperature cracking in metals and alloys under
both fatigue and tensile loading is sometimes associated with
the formation of intergranular voids. There is little evidence
for this fracture process in LCF of 20/25/Nb steel, however.
A few isolated intergranular voids have been observed in LCF
at a plastic strain amplitude of 8% but not at lower amplitudes
(229); this result was confirmed by density measurements.

In 20/25/Nb steel, there is evidence that the formation of
the square grain structure is linked with the onset of triple
point cracking (229). Under test conditions which did not produce
the square boundary structure, triple point cracking was not
observed. This result may be compared with the observations of
LCF cracking in a-Fe (183) (see section 1.3.1(c)) where cavitation
was suggested as the chief intergranular fracture mechanism.
It may be significant in both cases that LCF cracking did not
occur until the square grain boundary structure was established.
Possibly the migration prevents grain boundary stress concentrations which lead to crack initiation; this process may be similar to that proposed for the LCF of copper \(^{(179)}\), where the dispersal of boundary sliding by migration has been suggested as the means whereby stress concentrations at triple junctions are prevented. Consideration of the time to failure in a fatigue test, rather than the number of LCF cycles \(^{(229)}\), has shown that at elevated temperature the time to failure tends to a constant value irrespective of strain rate, i.e. the endurance decreases with diminishing strain rate. This behaviour is a characteristic of corrosion-controlled failure mechanisms, so that oxidation at the fatigue crack tip was proposed as the process controlling the rate of crack propagation. This is probably an oversimplification since in tests in simulated reactor atmosphere (which was oxidising) the LCF endurance was greater at a lower strain rate \(2 \times 10^{-5}/s\) than at the higher one \(1 \times 10^{-4}/s\) \(^{(252)}\), so that there was no evidence of corrosion being the rate controlling process in this instance.

1.3.4(c) Empirical Correlations and Theoretical Description of LCF

It has been established that the Coffin-Manson Law (see section 1.3.3(a)) is obeyed by 20/25/Nb steel at both ambient and elevated temperatures \(^{(227,229,231,232)}\) although deviations from the law have been noted at low plastic strain amplitudes (less than about 1\%) \(^{(233)}\).

Tomkins et al \(^{(231)}\) performed combined loading experiments at elevated temperatures on 20/25/Nb steel using three loading combinations:

1. LCF cycling followed by tensile loading to failure.
2. Tensile prestrain followed by LCF cycling to failure.
3. LCF with a superimposed continuously increasing tensile stress.

The experimental endurances were found to be in good agreement with endurances calculated from Tomkin's model of LCF crack propagation \(^{(223)}\) (section 1.3.3(f)). LCF work using thin section (0.5mm) specimens cycled in bending in a simulated
reactor atmosphere at elevated temperature (232) was also interpreted using Tomkin's model and reasonable agreement obtained. However, it has been pointed out that in the combined loading work a relatively high-strain rate was employed and also that the slope and position of the Coffin-Manson line depend to some extent on test atmosphere and strain rate (227). In view of the complexity of the crack propagation process at elevated temperatures, often involving diffusion and corrosion phenomena, it seems unlikely that a LCF crack propagation model based on simple continuum mechanics parameters alone will be capable of describing the LCF fracture process under all combinations of conditions.

Wareing has employed a semi-empirical approach to interpret fatigue data in 20/25/Nb steel (233). After demonstrating that the Geometrical Similarity growth law was obeyed (i.e. \( \frac{dl}{dn} = A_1 \)); where \( l \) = current crack length, \( n \) = number of fatigue cycles, \( A \) = propagation constant) the propagation constant \( A \) was related to the plastic strain amplitude by a power law relationship:

\[
A = B(\Delta \epsilon_p)^\theta \quad \text{...... (1)}
\]

where \( B \) and \( \theta \) are suitably chosen constants.

In addition logarithmic plots of growth rate against stress intensity factor at both 25 and 750C showed that a linear relationship exists, in the same way as for high-cycle fatigue. Therefore another expression for the propagation rate is:

\[
\frac{dl}{dn} = C (\Delta K)^a \quad \text{...... (2)}
\]

where \( \Delta K \) is the stress intensity factor amplitude and \( a \) and \( C \) are constants. Since the stress intensity factor concept is only strictly valid for elastic deformation, equation (2) must be regarded as an essentially empirical relationship.

The constant \( C \) varies with the plastic strain amplitude. Wareing employed the Tomkins analysis to introduce the plastic strain amplitude into equation (2) and obtained the relation:

\[
\frac{dl}{dn} \propto K^2 \Delta \epsilon_p \quad \text{...... (3)}
\]

where \( K \) is a "pseudo stress intensity factor" defined as:

\[
K = \sigma_t \sqrt{1 f (\frac{1}{W})} \quad \text{...... (4)}
\]
in which $\sigma_t$ is the maximum tensile stress in the cycle, $l$ is crack length and $f(1/w)$ is a geometrical factor. Substituting for $K$ in equation (3) gives:

$$\frac{dl}{dn} = \sigma_t^2 r^2 (1/w) A_e \frac{p}{t} l$$

so that $dl/dn$ is proportional to the crack length $l$ as required by the Geometrical Similarity Law.

Equation (3) was shown to be an approximation of a more general case which can also be applied to high-cycle fatigue. Therefore this approach permits some unification of LCF and HCF crack propagation behaviour. Good agreement was found between observed and calculated growth rates at 25°C but at 750°C agreement was not so good. Again, it appears that LCF models oversimplify the elevated temperature crack propagation behaviour.

1.3.4(d) Tensile Properties of 20/25/Nb Steel

With increasing test temperatures, the proof stress, UTS and strain hardening index all decrease as with all non-age hardening alloys \(^{(232)}\). The ductility of the fine-grained material (12-25µm) tends to increase by about 10-20% with test temperature over the range from 600 to 800°C, and also increases with decreasing strain rate at the higher temperature \(^{(232,234)}\). On the other hand, steel having a grain size of 250µm loses ductility over the same temperature range, although the change is not large \(^{(234)}\). The elevated temperature tensile deformation mechanism therefore appears to be noticeably grain size dependent, in the same way as superplastic deformation, although the ductility increases observed are very much less than those found in the superplastic case. This would be expected since the upper grain size limit for superplastic behaviour is generally considered to be about 10µm. Irrespective of grain size, fracture occurred by the growth and coalescence of intergranular voids.

Walkef et al \(^{(235)}\) demonstrated the possibility of obtaining some increases in ductility by straining the specimen incrementally with intermediate anneals. The steel used had a grain size of 30µm; the strain increments employed were 4 and 8%, and a fairly low strain rate was used \((2.8 \times 10^{-5} \text{ Sec}^{-1})\). Other workers
proposed the use of a "recovery parameter" to describe this ductility increase found during incremental strain/anneal cycles and discussed the phenomenon in terms of cavity sintering theory. The kinetics of the ductility recovery process in 20/25/Nb steel were studied by Walker and Evans using different incremental strains. They concluded that the rate of recovery is inversely related to the magnitude of the strain increment applied before the annealing treatment. Variation of recovery parameter with temperature afforded a method of estimating the activation energy of the process. The figure obtained was in good agreement with the published value for grain boundary self-diffusion, indicating that grain boundary diffusion is the rate controlling process. The recovery effect was attributed to cavity sintering. The fact that complete recovery did not occur was explained by stabilisation of the cavities by internal gas pressure. However, it is not proven whether cavities form at the low strain increments employed; plots of cavity volume against applied strain indicate that strains of about 30% or greater are required to produce significant density changes.
1.3.5. Low-Cycle Fatigue Properties of Irradiated 20/25/Nb Steel

The effects of neutron irradiation on the fatigue properties of austenitic stainless steels have received some attention in the USA recently as a consequence of the Liquid Metal Fast Breeder Reactor programme (LMFBR), this work being concentrated mainly on types AISI 304 and 316 stainless steels. These steels are intended for use both as fuel cladding and core structure both in the UK and USA, in fast reactors. Other fatigue studies have been published on AISI 348 steel for light water reactor (LWR) applications and on 20/25/Nb steel currently used as the fuel cladding in the Advanced Gas-Cooled Reactor (AGR).

1.3.5(a) Fracture Properties

Two reviews are available dealing with types AISI 304 and 316 austenitic steels. Fast Reactor irradiation may result in either increases or decreases of endurance depending on irradiation and test temperatures. Fast Reactor irradiation between 427 and 593°C at fluences up to $5 \times 10^{21}$ neutrons/cm$^2$ (energy greater than 0.1 MeV) causes a reduction in the LCF endurance of up to 0.67 of the unirradiated control value at a test temperature of 500°C. Increased fluences and test temperatures in the range 600-700°C result in endurances of approximately 0.4 of the control value. Crack growth studies in AISI 304 and 316 steels have shown that at test temperatures of 427 and 593°C the effect of both thermal and fast neutron irradiations was to increase the rate of crack propagation in HCF.

At sufficiently low irradiation and test temperatures the endurance of the irradiated steel is greater than that of the unirradiated control under stress-cycling (HCF) test conditions. Reversed bending test on AISI 304 steel have indicated that the irradiation and test temperature should be less than about 450°C for the increased endurance to be observed.

Very large endurance increases have been reported: in AISI 348 steel HCF endurances ten times greater than those observed in the unirradiated steel were found after thermal reactor irradiation at 288°C. The enhanced endurance observed under stress cycling conditions may be explained by the irradiation hardening
taking place at sufficiently low irradiation temperatures; there is a fairly well established correlation between strength and endurance under stress-cycling conditions \(^{223}\) though under strain-cycling conditions ductility is the more important parameter.

Prior cold work has been found to have a beneficial effect on the LCF properties of fast reactor irradiated AISI 304 and 316 steels \(^{245}\) afterwards tested at temperatures of 600 and 700°C. This may be explained by the precipitation of helium bubbles on intragranular dislocations, so preventing as much accumulation of intergranular helium as in the previously annealed steel.

The LCF properties of thermal reactor irradiated 20/25/Nb steel have been reported in an investigation which formed a prelude to the present work \(^{232}\). Reversed bend tests were performed in a simulated reactor atmosphere at 700 and 800°C, at a strain rate of \(1 \times 10^{-4}/s\). The irradiations were carried out at 700°C, that is, approximately the fuel cladding temperature in the reactor. Reduced LCF endurances were found, but no clear dependence on irradiation dose was found. In contrast, in tensile tests there was a progressive reduction in ductility with increasing dose in tensile tests carried out under the same test conditions. Empirical correlation of tensile and LCF data (plotting tensile ductilities as one-quarter cycle LCF tests) (see section 1.3.3(a)) yielded reasonable results. The extent of endurance reduction due to irradiation was found to depend on the plastic strain amplitude; below an amplitude of approximately 0.8% there was no irradiation effect, while above this value the reduction in endurance increased with increasing plastic strain amplitude.

The deleterious effect of irradiation on the LCF endurance was explained in a similar way to tensile embrittlement, by the influence of helium bubbles in promoting intergranular cracking (see section 1.2.3(c)). The greater effect of irradiation on endurance at higher plastic strain amplitudes was accounted for by the increased stress, and hence greater bubble growth rates.
1.3.5(b) **Empirical and Theoretical Description of Irradiated LCF Properties**

No physical theory exists at present which describes quantitatively the relation between irradiation treatment and LCF endurance. However, an empirical method based on the Coffin-Manson Law has recently been proposed. According to this method, the influence of irradiation can be regarded as (a) an effect on the material strength, and (b) an effect on ductility. The former is the controlling factor in HCF, and the latter in LCF. The Coffin-Manson Law $\Delta \epsilon_p, N^\alpha = \text{const.}$ can be rearranged:

$$\Delta \epsilon_p = \text{const. } N^{-\alpha} \quad \ldots \ (1)$$

And likewise the elastic strain range can be written:

$$\Delta \epsilon_e = \text{const. } N^{-\delta} \quad \ldots \ (2)$$

Combining (1) and (2):

$$\Delta \epsilon_t = \Delta \epsilon_p + \Delta \epsilon_e = AN^{-\alpha} + BN^{-\delta} \quad \ldots \ (3)$$

where $\Delta \epsilon_t$ is the total strain amplitude. Following the original proposal of Coffin that the tensile ductility can be equated with a one-quarter cycle LCF test, the constant $A$ will depend on the ductility. Similarly, the known dependence of the HCF endurance on UTS implies that the constant $B$ will depend on UTS. The constants $A$, $B$, $\alpha$, and $\delta$, can all be determined from tests on unirradiated material. When the UTS and ductility of the irradiated material are known, then a "fraction modification method" is used to modify the constants $A$ and $B$ in equation (3) by the fractions $\phi_u$ and $\phi_t$; these fractions are given by:

$$\phi_u = \frac{(\text{UTS}) \text{ irrad}}{(\text{UTS}) \text{ unirrad}} \quad \text{and} \quad \phi_t = \left[ \frac{D \text{ irrad}}{D \text{ unirrad}} \right]^{0.6}$$

so that equation (3) becomes:

$$(\Delta \epsilon_t) \text{ irrad} = \phi_u AN^{-\alpha} + \phi_t BN^{-\delta} \quad \ldots \ (4)$$

The exponent 0.6 is the commonly observed value of the Coffin-Manson exponent. The method therefore assumes that the strain amplitude/endurance curve for unirradiated and irradiated steels will have the same slope. Good correlation between fatigue curves generated by equation (4) and experiment has been found for austenitic stainless steels.
1.3.5(c) Tensile Properties

The tensile properties of 20/25/Nb steel irradiated at ambient temperature to thermal neutron doses of \( \lesssim 10^{19} \) n/cm\(^2\) and tested at 750°C were studied by Broomfield et al. A marked loss of ductility was noted. Harries (1972) and Roberts and Harries (1980) reported a similar ductility loss above 600°C in 20/25/Nb steel irradiated to a thermal neutron dose of \( 2.5 \times 10^{20} \) n/cm\(^2\), although both unirradiated and irradiated ductilities showed a trend to higher values with increasing temperature. Other low-temperature irradiation work using thermal neutron doses of \( 2.5 \times 10^{20} \) and \( 6.9 \times 10^{20} \) n/cm\(^2\) demonstrated the beneficial effect of pre- or post-irradiation ageing treatments on ductility at a test temperature of 750°C. The improvement was explained partly by the influence of grain boundary precipitates on grain boundary fracture behaviour and partly on the anchoring effect of intragranular precipitates on small helium bubbles that could otherwise migrate to the boundaries. Wedge cracking and cavitation were both observed in the irradiated steel; the incidence of the former diminished when the steel was aged before irradiation. Further work on a steel irradiated at 40°C to a relatively low dose of \( 5 \times 10^{19} \) n/cm\(^2\) indicated that wedge cracking was the predominant fracture mode irrespective of test conditions, in contrast to the unirradiated controls in which a mixture of wedge cracking and cavitation was observed. The tendency to wedge cracking increased with increasing grain size. No increase in ductility of the irradiated material with increasing test temperature was found, although such an increase was observed above 700°C in the unirradiated material, followed by a slight decline at 900°C. Histograms of crack frequency/crack size for a material grain size of 250 μm showed that there were far fewer small cracks in the irradiated specimens than in the controls, at test temperatures of 600, 750 and 800°C. This was explained by an enhanced rate of propagation of the wedge cracks, so that failure occurred before most cracks had grown to the size that they otherwise would have attained in unirradiated material. Unstable growth of helium bubbles ahead of the wedge crack tip was suggested as the mechanism. The
temperature dependence of the irradiation induced ductility loss was not affected much by the stress. No account was taken of the influence of precipitate distribution and morphology on fracture behaviour.

Irradiation was found to increase the rate of growth of wedge cracks in coarse grained specimens of 20/25/Nb steel\(^1\). Fractography revealed the presence of a dense distribution of small voids, about 0.5 \(\mu\)m dia., on the fracture surfaces of the irradiated specimens, but not on those of the control specimens. It was suggested that the voids nucleated at the sites of grain boundary helium bubbles.
1.4. Literature Survey Summary

The principal facts drawn from the literature bearing on the LCF properties of 20/25/Nb steel are as follows:

1.4.1. Microstructure

Two or three second phases are likely to be present in the steel, their quantity, morphology and distribution depending on composition, irradiation treatment (if any) and thermomechanical history. The carbonitrides may be expected to influence mechanical properties by their interaction with glide dislocations and to influence elevated temperature intergranular fracture behaviour by their presence in the grain boundaries. During elevated temperature LCF tests additional precipitation may occur if the steel is not fully aged; the structure then developing may be different from that of the fully aged steel. If the sigma phase, FeCr, forms the possibility exists that it may affect elevated temperature intergranular fracture (particularly wedge cracking) since it is harder than the matrix austenite, but this is not definitely established. Chromium depletion caused by sigma phase formation may result in reduced corrosion resistance.

1.4.2. Irradiation

Radiation hardening in 20/25/Nb steel will only occur at irradiation temperatures below about 550°C; there will be an accompanying loss of tensile ductility. Irradiation and testing above 550°C will result in a reduction of ductility associated with an increased tendency to intergranular cracking. The extent of ductility loss will be dependent on the quantity of intergranular helium which in turn will depend on boron content, boron segregation and thermal neutron dose. If the boron content is very low (2 ppm (wt) or less) then the Ni\(^{58}\) \(\text{n,\alpha}\) reaction will become the dominant helium formation process at higher doses; this will affect the quantity and distribution of helium compared with higher boron content steels. Void formation and irradiation creep will not be significant during thermal reactor irradiations.

Changes in precipitate morphology and distribution may be expected; the formation of non-equilibrium phases is unlikely since the composition of 20/25/Nb steel is not close to a phase boundary.
1.4.3. **Low-Cycle Fatigue Properties**

Information is not available on the effect of LCF cycling on the bulk mechanical properties and dislocation/slip band structure of 20/25/Nb steel. Grain boundary structure changes may be expected in both unirradiated and irradiated steel at temperatures above approximately 700°C; the changes will be dependent on plastic strain amplitude and strain rate.

During low-temperature LCF, fracture will be transgranular except at very high plastic strain amplitudes when intergranular fracture may be observed. The mechanism of crack initiation is not established with certainty. At elevated temperatures, the initiation mechanism is again not well-established, but there will be a progressive tendency to intergranular fracture with increasing temperature and decreasing strain rate. Intergranular cavitation will not be observed, except possibly at very high plastic strain amplitudes, it will, however, be observed in tensile tests. The grain boundary structure changes will probably influence the fracture behaviour: triple (or quadruple) point cracking may be favoured if a square grain boundary structure is formed. Oxidation of the LCF crack tip may be an important factor governing crack propagation rates especially if:

1. There is a high partial pressure of oxygen or a high oxygen activity
2. The temperature is high
3. The strain rate is low
4. The steel contains a significant fraction of the sigma phase FeCr

The Coffin-Manson Law will be obeyed except at plastic strain amplitudes below about 1%. Low temperature LCF results may be correlated with mechanical properties using the available crack propagation models, but good correlation is less likely at elevated temperatures, probably due to the increased complexity of the fracture process.
Low irradiation and test temperatures will result in a reduced endurance under LCF strain cycling conditions. Similarly, elevated temperature irradiation and testing will produce lower endurances than in the unirradiated steel. This may correlate with an increased tendency to intergranular cracking. The extent of the elevated temperature irradiation-induced endurance loss will depend on:

1. Thermal neutron dose
2. Boron content and distribution
3. Test temperature
4. Strain rate
5. Plastic strain amplitude

There will likewise be a reduction in tensile ductility. Intergranular cavitation may be observed in irradiated LCF specimens.
CHAPTER 2  MICROSTRUCTURE

2.1. Introduction

The following chapter constitutes a brief investigation into the influence of thermomechanical treatments and thermal reactor irradiations on the microstructures of the two compositions of 20/25/Nb stainless steel used in the LCF studies described in chapters 3 and 4.

The thermomechanical treatments are conveniently classified according to the grain size of the final condition of the steel. Three different treatments were used to produce a range of grain sizes, one of these being chosen to reproduce that applied to the steel during fuel element manufacture. Most of the work described in this and later chapters was carried out using steel in this condition, in order that the results should be applicable to actual fuel cladding. The irradiation temperatures were likewise chosen to approximate those experienced by the fuel cladding in the reactor.
2.2. Composition and Thermomechanical Treatments

2.2.1 Composition

Two casts of 20/25/Nb steel, manufactured using the double vacuum melting process, were employed in the investigation described in this and later chapters and had the following compositions (in wt.% unless otherwise indicated):

<table>
<thead>
<tr>
<th>Cast No.</th>
<th>Cr</th>
<th>Ni</th>
<th>Nb</th>
<th>C</th>
<th>N</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z240</td>
<td>20.2</td>
<td>24.7</td>
<td>0.57</td>
<td>0.03</td>
<td>0.02</td>
<td>0.66</td>
<td>0.67</td>
<td>0.005</td>
<td>0.005</td>
<td>0.2ppm+0.05ppm</td>
</tr>
<tr>
<td>Z227</td>
<td>20.3</td>
<td>25.1</td>
<td>0.7</td>
<td>60ppm</td>
<td>-</td>
<td>0.7</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1ppm(nominal)</td>
</tr>
</tbody>
</table>

Specimens made from cast Z227 were only used in the carburisation study described later in Section 2.5.

2.2.2 Thermomechanical Treatments

The preparation of strip has previously been described elsewhere (232). Strip 2.5 mm thick was prepared from the cast material by hot rolling, with intermediate anneals at 1150°C, followed by cleaning and vacuum annealing at 1050°C for 1 hour. Cold rolling was used to prepare strip 0.75 and 0.5 mm thick from which thin section tensile and fatigue specimens were die stamped.

The specimens were sealed in evacuated silica glass capsules and given one of the following heat treatments:

(1) Anneal at 1050°C for 3s. then water quench.

(2) Anneal at 960°C for 1/4 hr. then air cool.

(3) Anneal at 1050°C for 1/2 hr. then air cool.

The mean grain sizes after these treatments were 3, 12 and 30µm, respectively. The treatment producing the 12 µm grain size corresponded to that used for actual fuel cladding.
2.2.3 Irradiation and Ageing treatments

Specimens enclosed in irradiation cans were irradiated in the DIDO reactor. Neutron irradiation was carried out at a temperature of 700°C ± 10°C for periods of 25, 75 or 125 d. The neutron doses corresponding to these times were (in neutrons/sq. cm).

- 25d. \(3 \times 10^{20}\) thermal, \(1 \times 10^{20}\) fission
- 75d. \(1 \times 10^{21}\) thermal, \(3 \times 10^{20}\) fission
- 125d. \(1.5 \times 10^{21}\) thermal, \(5 \times 10^{20}\) fission.

Specimens having grain sizes of 3 or 30 μm were only irradiated for the 125d period, while the 12 μm grain size specimens were irradiated for 25, 75, or 125d. Control specimens were given ageing treatments at 700°C ± 10°C without irradiation for corresponding periods. 20/25/Nb steel in the as heat-treated condition (i.e. without any ageing treatment) of each grain size was also examined. Some specimens were given ageing treatments of shorter duration than those above in order to study the early stages of precipitation; the influence of temperature was included in the study by additionally giving ageing treatments to some specimens at temperatures of 600°C and 800°C. These are summarised below:

<table>
<thead>
<tr>
<th>Grain Size (μm)</th>
<th>Ageing Temperatures (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>600</td>
</tr>
<tr>
<td>3</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>6d</td>
</tr>
<tr>
<td></td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>75</td>
</tr>
<tr>
<td></td>
<td>125</td>
</tr>
<tr>
<td>30</td>
<td></td>
</tr>
</tbody>
</table>

Some additional specimens of the 12 μm grain size steel were given a secondary ageing treatment at 800°C after first ageing at 700°C.
2.3 Specimen Preparation and Examination

2.3.1 Optical Metallography

Microsections were prepared using standard methods, i.e. mounting in thermosetting plastic, grinding, and diamond polishing. Electrolytic etching to reveal grain boundaries and general structure was performed using 10% oxalic acid aqueous solution at 80 to 90°C. Sigma phase was selectively electrolytically etched using 10N aqueous potassium hydroxide at room temperature.

2.3.2 X-ray Powder Photographs

Powder samples were prepared either by electrolytic dissolution in 20% aqueous HCl or in 20% HCl in methanol, or by immersion in Villela's reagent for about 24hr. The residues were washed, centrifuged and dried. The powder was placed in Lindemann glass capillaries and X-ray powder photographs taken using a Debye-Scherrer camera and CuKα radiation.

2.3.3 Electron Microscopy

Carbon extraction replicas were prepared using conventional methods, the replica being removed by overnight immersion in a solution of 20% HCl in alcohol. Thin foils of the aged steel were prepared by punching 2mm or 3mm diameter discs from specimens, followed by "dishing", i.e. jet machining with 30% HNO₃ in methanol to produce a shallow depression in the surface, and then electropolishing at 6V in a solution of 2 parts perchloric acid (specific gravity 1.5) and 1 part glacial acetic acid, at room temperature. A dense anodic layer forms with this solution, so in order to see the moment of perforation more easily a stainless steel screen with a 2mm diameter hole punched in it, was placed about 2mm behind the disc. The Bainbridge "Unithin" equipment gave satisfactory results with the as heat treated steel and was quicker to use than the method described in the previous paragraph. Replicas and foils were examined at a potential of 100KV in either a Siemens or Phillips electron microscope.
2.3.4 Boron Autoradiography and Nuclear Microprobe Analysis

Specimens for these examination techniques were prepared by conventional metallographic methods.

The boron autoradiographic technique\textsuperscript{(251)} provides a method for determining the degree of segregation of boron in the specimen (boron segregation is discussed in section 1.1.3 on page 33 of the Literature Review) and correlating it with microstructural features. The method depends on the formation of alpha particle tracks in a cellulose acetate film attached to the metal surface, previously ground and polished. The alpha particle tracks are formed during irradiation with thermal neutrons from the reaction:

$$\text{Li}^7 + \text{He}^4$$

The reaction is instantaneous for practical purposes. After irradiation the plastic film is stripped off and etched in 20\% potassium hydroxide solution to reveal the tracks and photographed with a phase contrast microscope. A conventional micrograph of the same field is then taken.

The nuclear microprobe technique of analysis uses measurements of prompt radiation charged particles, neutrons, gamma or X-rays resulting from nuclear reaction or elastic scattering of an incident ion beam with the sample. The ion beam is generally a few μm in diameter to obtain sufficient resolution to analyse selected microstructural features. The carbon analysis method\textsuperscript{(252)} employs a beam of 1.3MeV deuterons from an accelerator to generate protons from carbon according to the reaction:

$$\text{C}^{12} \text{(d.p)} \text{C}^{13}$$

The emitted protons are detected by a solid state device and counted.
2.4. The Microstructure of as Heat Treated, and Heat Treated and Aged Steel

2.4.1 As Heat Treated Steel

Grain Size 3 μm

Annealing twins were prominent in the grain structure. Considerable amounts of primary Nb(C,N), about 0.2 μm diameter were distributed fairly uniformly throughout the structure (Plate 2.1). No needles or plates of secondary Nb(C,N) were observed. Very little dislocation contrast could be found, although a few grain boundary dislocations were seen. Grain boundaries did not etch clearly.

Grain Size 12 μm

Particles of primary Nb(C,N) about 0.2 μm diameter were visible (Plate 2.2); no needles of secondary Nb(C,N) were observed. X-ray powder photographs showed that residues were composed of Nb(C,N) and \( \text{M}_6 \text{(C,N)} \). Intensity measurements were made and compared with values calculated from the formula for \( \text{M}_6 \text{(C,N)}: \text{Ni}_2 \text{Nb}_3 \text{(C,N)} \) (see Literature Review, page 30). The mean ratio Nb(C,N): \( \text{M}_6 \text{(C,N)} \) by weight was found to be 80:20. Thin foils showed some dislocation contrast, though the dislocation density was low. Grain boundaries did not etch clearly.

Grain Size 30 μm

Fewer twins were seen than in the finer grain size steels. Particles of primary Nb(C,N) were observed, about 0.3 μm diameter, together with needles of secondary Nb(C,N) about 1 μm long (Plate 2.3). Frequently, two or more needles were associated to form a cross pattern, sometimes in small clusters (Plate 2.4). Grain boundaries etched clearly in this steel.

2.4.2 Heat Treated and Aged Steel, Grain Size 12μm

Aged at 600°C for 6d.

Particles of sigma phase about 2 μm across were present at triple points (Plate 2.5). Little secondary Nb(C,N) was observed, this phase being present chiefly as globular particles of primary Nb(C,N). Thin flakes of \( \text{M}_6 \text{(C,N)} \) were clustered around the
particles of sigma phase, appearing to have nucleated at the sigma-grain boundary junction and then grown along the boundary (Plate 2.6). Some grain boundary flakes were not associated with sigma phase, and a little intragranular $M_6(C,N)$ was present as globular particles.

**Aged at 700°C for 1 to 125d.**

After ageing for 1d, considerable precipitation had occurred (Plate 2.7). The flakes were larger and thicker than in the steel aged at 600°C, in general only being transparent to the 100KV electron beam at the edges. Although $M_6(C,N)$ was clustered at the sigma phase particles as before, there was more $M_6(C,N)$ away from sigma phase particles and intragranular globular $M_6(C,N)$ was more in evidence than in the specimen aged at 600°C. Many small crystals of secondary Nb(C,N) were present, of no definite shape and sometimes occurring in clusters of about 5μm diameter. Examination of thin foils did not reveal the presence of this fine secondary Nb(C,N) in grain boundaries.

Ageing for 7d resulted in a noticeable increase in the quantity and size of both sigma phase and carbonitrides (Plate 2.8). The secondary Nb(C,N) crystals were thicker than those in the steel aged for 1d and in some cases were sufficiently large to have the characteristic acicular and lamellar habits of this carbonitride (see Literature Review, page 29).

Further ageing treatments of 25, 75 and 125d resulted in continued precipitation of carbonitrides and sigma phase (Plate 2.9). Precipitate free zones were observed at grain boundaries at ageing times longer than 25d (Plate 2.10). The size of the sigma phase particles continued to increase with increasing ageing time up to the maximum of 125d, the size range after this latter time being about 5 to 15μm (Plate 2.11). No change in the grain size was found after ageing for 125d. There was little difference in the amount of carbonitrides in the 75 and 125d aged specimens.

X-ray powder photographs were taken of residues from the as heat treated steel and from steel aged for 125d at 700°C and subsequently heat treated at 880°C for 60hr to remove the sigma phase precipitate. This post-ageing heat treatment was necessary since the high iron content of the sigma phase (composition approx. FeCr) caused fogging of the X-ray film by secondary emission when
CuKα radiation was used. A cobalt target X-ray tube was tried to overcome this, but exposure times became inconveniently long. Attempts to remove the sigma phase by electrolytic dissolution in aqueous 20% HCl were not successful.

Intensity measurements were made in the same manner as for the as heat treated steel. The mean Nb(C,N):M6(C,N) weight ratio (3 determinations) was found to be 40% Nb(C,N): 60%M6(C,N) and the ratio of Nb(C,N) in the as heat treated and heat treated and aged for 125d conditions was 0.75.

The observed relative intensities of the different orders of reflection of the M6(C,N) were in reasonable agreement with those calculated from the published formula and structure but the agreement was not as good as expected. Weak lines were detected in the powder pattern corresponding to space group extinctions in the space group Fd3m. These are probably super-lattice lines due to ordering of the nickel and niobium atoms in the f positions (see International Tables for X-Ray Crystallography).

**Aged for 125d at 700°C, Then Further Ageing at 800°C**

Further ageing at 800°C resulted in slow dissolution of the sigma phase precipitates. About 75% of the sigma phase had dissolved after 15d at 800°C, and solution was virtually complete after ageing for 40d. No alteration in the quantities or distribution of the carbonitrides could be detected after this time or any change in grain size.

**Aged for 1 to 7d at 800°C**

After 1d ageing at 800°C, noticeably more Nb(C,N) had precipitated than in specimens aged for 1d at the lower temperature of 700°C, although the amount of M6(C,N) formed appeared to be about the same (Plate 2.12). Flake M6(C,N) in grain boundaries was less apparent in this ageing treatment than in the specimen aged for the corresponding time at 700°C. After 4d at 800°C there was little change in the amount of secondary Nb(C,N), although there was an increase in the size and number of intra- and intergranular particles of M6(C,N). Ageing for 7d resulted in
a structure in which the size and distribution of carbonitrides was similar to that of the steel aged for the same time at 700°C, (Plate 2.13) except that the flake $M_6$($C,N$) clusters associated with sigma phase particles in the 700°C aged specimens were much less apparent in the 800°C aged steel.

**Grain Size 3 μm**

**Aged 125d at 700C**

No change in grain size could be detected after ageing. The distribution of the sigma phase was similar to that in the 12 μm grain size steel, being only found at triple points, though the particles tended to be smaller in the 3μm grain size steel. Flake $M_6$($C,N$) showed a stronger tendency to be associated with sigma phase particles than the same carbonitride in the 12 μm grain size specimens, sometimes forming clusters 10μm in diameter (Plate 2.14). Precipitate free zones around the grain boundaries were particularly prominent. The intragranular carbonitrides consisted of acicular and lamellar Nb($C,N$) with globular $M_6$($C,N$) and primary Nb($C,N$); these tended to become coarser towards the grain interior, the smallest crystals being at the precipitate free zone boundary.

**Grain Size 30 μm**

**Aged for 125d at 700C**

As before, no effect of ageing on grain size was found. The quantity of sigma phase was similar to that found previously in the 12 μm grain size steel, namely 2 to 4% by volume, and likewise located at triple points although the particles were fewer and coarser. There was a strong tendency for $M_6$($C,N$) flakes to form large clusters, sometimes occupying 30% or more of the grain boundary length. Flakes within these clusters showed an alignment towards certain orientations (Plates 2.15 and 2.16). Smaller globular $M_6$($C,N$) crystals were present in the grain interiors. Nb($C,N$) was again present as an intragranular precipitate, often in the form of "knotted stringers" (see Literature Review, page 30 ) consisting of acicular and lamellar crystals (Plt. 2.17). The precipitate free zone was less well-defined than in the 3 μm and 12 μm steels.
2.5. The Microstructure of Heat Treated and Irradiated Steel

Grain Size 3 µm

Irradiated 125d at 700°C

No significant differences in the amount and distribution of sigma phase and carbonitrides were found between steel irradiated for 125d at 700°C and corresponding thermal control specimens. In particular, the precipitate free zone was still present in the irradiated specimen (Plate 2.18). Occasional stringers of acicular and lamellar Nb(C,N) were observed (Plate 2.19). Helium bubbles, 100 to 200Å diameter, were occasionally found in grain boundaries; smaller bubbles, 20 to 100Å diameter, were sometimes attached to Nb(C,N) or M₆(C,N) crystals in the grain interiors, less frequently they were not associated with precipitates or other structural features. Thin foils prepared from specimens irradiated at 700°C and subsequently LCF tested at 800°C appeared to have rather more bubbles than those tested at 700°C, though the overall bubble density was so low that reproducible quantitative data could not be obtained. The largest bubbles found occurred in electron microscope specimens prepared from the test areas of LCF specimens which had been tested at 800°C (Plate 2.20). The largest bubbles found were about 500Å diameter, i.e. about twice the diameter of the largest bubbles observed in thin foils prepared from the unstrained areas of the LCF specimens.

Grain Size 12 µm

Irradiated for 25, 75 and 125d at 700°C

As a rule the quantity and distribution of carbonitrides and sigma phase was found to be similar to that of the corresponding thermal control specimens for all the irradiation treatments (Plt. 2.21). However, examination of transverse microsections etched in potassium hydroxide solution in some cases revealed the presence of an unknown phase in a thin layer (about 50µm) near the surface, both in LCF specimens (Plate 2.22) and tensile specimens (Plate 2.23). The appearance was similar to that of sigma phase, but the etching characteristics differed from sigma phase in that the KOH etchant attacked the unknown phase less readily. For this reason the unknown precipitate was thought to be the complex nitride, Z phase. A specimen was heat treated at 880°C for 60 hr to remove sigma phase, as described in
Section 2.4.2, and residues prepared for X-ray powder photographs. Examination of the powder photographs showed that the unknown phase was, in fact, the carbonitride $M_{23}(C,N)_6$. Since this carbonitride is associated with hypostoichiometric compositions in 20/25/Nb steel (see Literature Review, page 28) this indicated that carburisation had occurred during irradiation. This was confirmed by a nuclear microprobe analysis for carbon (carried out in the Applied Chemistry Division, AERE); this technique has been briefly described on page 103.

The carbon profile obtained by the microprobe analysis on specimens irradiated for 25, 75 and 125d is shown in Fig. 2.2. The carbon content clearly increases by a factor of about 5 towards the surface of the 125d irradiated sample. An increase of about 2 in carbon content occurs in the 75d irradiated sample, while no change is apparent in the 25d irradiated sample. Optical microscope examination of the 25d and 75d irradiated specimens failed to show any $M_{23}(C,N)_6$ precipitates at the specimen surfaces. Similar examinations of a number of LCF specimens irradiated for 125d showed that there was considerable variation in the degree of $M_{23}(C,N)_6$ precipitation, in some instances none was observed. Prior to the study described here it had been observed that anomalously low endurances were obtained in LCF tests in argon atmosphere. The fact that carburisation had occurred in the argon atmosphere LCF tests and may have been responsible for the reduced endurances found.

Consequently, LCF tests on unirradiated specimens were carried out in an argon atmosphere at a test temperature of 800°C and a strain rate of $1 \times 10^{-4}$ sec$^{-1}$. Endurances were much lower than in corresponding tests done in vacuo (Fig. 2.3). $M_{23}(C,N)_6$ was found to have precipitated under the surfaces and around LCF cracks, after testing for 4 or 5 hr. Tests were performed using specimens made from the low-carbon cast Z227 (see page 100) to see whether the lower initial carbon content would be sufficient to suppress $M_{23}(C,N)_6$ formation, but $M_{23}(C,N)_6$ still precipitated, as shown in Plate 2.24; the LCF test results are shown in Fig. 2.4. Tests conducted at a temperature of 800°C and a strain rate of $3.5 \times 10^{-4}$ sec$^{-1}$ indicate
that similar endurances are obtained irrespective of initial
carbon content (Fig. 2.5) and tests at 700C and a strain rate
of 1 x 10^{-4} sec^{-1} show that the loss of endurance caused by
carburisation persists at this lower test temperature (Fig. 2.6).

A micrograph of a Z240 cast specimen is shown in Plate 2.25.
Examination of the entire length of LCF specimens showed that the
M_{23}^{(C,N)} content under the surface decreased rapidly with
increasing distance from the test area, and furthermore the
carbonitride was not present in the unstressed portion of the
test piece beyond the knife edge contact point (Plates 2.26 to
2.28). Sigma phase was never present in areas containing
M_{23}^{(C,N)}). The appearance of the M_{23}^{(C,N)} precipitate at
high magnification is shown in Plate 2.29. In all cases there
was no apparent internal cracking; LCF fracture proceeded by
crack growth from the surface.

Tensile specimens irradiated for 125d tended to be fairly
heavily carburised and showed stringers of M_{23}^{(C,N)} along
grain boundaries, and also showed extensive intragranular pre-
cipitation, often along twin boundaries (Plate 2.30). In the
steel containing 60ppm carbon (Z227) fatigued in argon at 800C,
the M_{23}^{(C,N)} was present chiefly as small (0.2\mu m) particles
near the surface and adjacent to cracks, but there were grain
boundary stringers and clusters of particles towards the interior
(Plate 2.31). In the steel containing 0.03% carbon (Z240) the
M_{23}^{(C,N)} formed small particles near the surface and around
cracks, the particles becoming coarser towards the interior and
resembling small sigma phase particles (Plate 2.32).

The size, quantity, and distribution of helium bubbles in
the 125d irradiated specimens was similar to that of the 3\mu m
grain size steel (Plates 2.33, 2.34). Again, the infrequent
occurrence of bubbles in the electron transparent areas of the
thin foils precluded any reliable measurements of bubble densities
and distributions. Bubbles of 50 to 100A diameter were rarely
seen in the 75d irradiated specimens, and the number of small
bubbles seen (those less than 50A) was likewise small. In the
case of the 25d irradiated steel only very few bubbles could be
found, either in grain boundaries or attached to intragranular
precipitates.
Grain Size 30 µm

Irradiated 125d at 700C

The morphology and distribution of the carbonitrides after 125d irradiation was similar to that of the corresponding thermal control specimens, although there appeared to be a somewhat greater amount of Nb(C,N) in the irradiated steel. The Nb(C,N) was frequently present as the "knotted stringers", discussed in the Literature Review, page 30.

Few grain boundary helium bubbles were observed, though occasional groups of bubbles of 100Å diameter or less were observed in precipitate-free areas (Plate 2.35). An occasional bubble of about 300Å diameter was observed in the grain boundaries in the strained areas of specimens tested at 800C. Bubbles attached to intragranular precipitates were much more in evidence than in the 3µm and 12µm grain size steels. Although the majority were of less than 50Å diameter, a few bubbles of approximately 100Å diameter were observed (Plate 2.36).

The amounts of helium from the $^{10}$B and $^{58}$Ni reactions, expressed in atoms per atom (i.e. the number of helium atoms per atom of the steel) taken from Fig. 2.1 are given below for the three irradiation periods employed.

<table>
<thead>
<tr>
<th>Irradiation Time (d)</th>
<th>Thermal Dose n/cm²</th>
<th>He from $^{10}$B a/a</th>
<th>He from $^{58}$Ni a/a</th>
<th>Total He a/a</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>$3 \times 10^{20}$</td>
<td>$1.4 \times 10^{-7}$</td>
<td>$4 \times 10^{-7}$</td>
<td>$5.4 \times 10^{-7}$</td>
</tr>
<tr>
<td>75</td>
<td>$1 \times 10^{21}$</td>
<td>$2 \times 10^{-7}$</td>
<td>$4.5 \times 10^{-6}$</td>
<td>$4.7 \times 10^{-6}$</td>
</tr>
<tr>
<td>125</td>
<td>$1.5 \times 10^{21}$</td>
<td>$2 \times 10^{-7}$</td>
<td>$1 \times 10^{-5}$</td>
<td>$1.02 \times 10^{-5}$</td>
</tr>
</tbody>
</table>

Because of its relatively large grain size (30 µm) this steel was chosen for autoradiographic examination to determine whether any segregation of boron to grain boundary carbonitrides had occurred (the technique is described on page 103). No such segregation was detected, however.
2.6. Discussion

2.6.1 Carbonitride precipitation

The Nb(C,N) solubility ratio of 0.75 determined experimentally for 20/25/Nb steel solution treated at 960°C for 1/2 hr. and subsequently aged at 700°C (see section 2.4.2, page 104) is in reasonable agreement with the value of 0.84 determined from the solubility curves from the results of Déighton, discussed in the Literature Review, page 24. The solubility of Nb(C,N) at the ageing temperature of 700°C is negligible (see Literature Review, Fig.4.5, page 40). The principal sources of error in this determination were probably the measurements of the integrated intensities of the X-ray reflections, and the lack of close agreement between the observed and calculated structure factors for M6(C,N). The latter is difficult to account for since a different structure factor would usually indicate that the assumed composition was not correct, but in this case the composition appears to be well established by electron probe microanalysis (see Literature Review, page 30). It would appear though, that in the absence of an alternative explanation, the possibility exists that the composition of the M6(C,N) carbonitride in the steel used here differs from the accepted one, to a small extent.

2.6.2 As Heat-treated Steel

Owing to the absence of comprehensive kinetic data (Literature Review, page 28) the occurrence of the carbonitride phases can only be described here in a qualitative manner.

The relatively large amount of primary Nb(C,N) present in the 3 μm and 12 μm grain steels (section 2.4.2) compared with the 30 μm grain size steel probably reflects the influence of solution treatment temperatures and times. The solution treatment to produce the 30 μm grain size involves either higher temperature or longer time than the other treatments, (see section 2.2.2) there is therefore rather less primary Nb(C,N) but more secondary Nb(C,N) plates and needles, due to the greater carbon supersaturation. The reason for the poor grain boundary etching in the 3 μm and 12 μm grain size steels is not understood (section 2.4.1, page 104).
Electron microscope examination of thin foils did not show any differences in grain boundary precipitation compared with the 30 \( \mu m \) grain size steel, which showed normal grain boundary etching characteristics.

Although X-ray powder photographs showed that a little primary \( M_6(C,N) \) was present it could not easily be distinguished from primary Nb(C,N) during electron microscope examination of replicas or thin foils owing to the globular form of both.

Lack of solubility data precluded the determination of a solubility ratio for the \( M_6(C,N) \) phase (see Literature Review, page 24).

2.6.3 Heat-Treated and Aged Steel

The precipitation of the Nb(C,N) phase during ageing (section 2.4.2) was observed to follow the broad features described in the Literature Review, page 29. No evidence for stacking fault precipitation of Nb(C,N) during ageing was found, in agreement with the results discussed in the Literature Review. Stacking fault contrast has not been observed in this investigation; the nickel and niobium contents were evidently too large for any significant separation of Frank partials to occur. Matrix (i.e. homogeneous) precipitation has not been looked for in this work, in view of the conclusion of Dewey et al (Literature Review, page 29.) that this mode of precipitation did not occur in a steel of similar composition to those used in this study. The principal locations for Nb(C,N) precipitation were twin and grain boundaries and undissociated matrix dislocations. Nb(C,N) particles on grain and twin boundaries tended to be fairly large (\( \sim 1 \mu m \)), the intragranular precipitate consisting of small plates and needles.

The 3 \( \mu m \) and 12 \( \mu m \) grain size specimens showed similar Nb(C,N) precipitation behaviour, with random distributions of intragranular crystals, growing coarser towards the grain centre, and a marked precipitate free zone at the grain boundary. The precipitate free zone in this steel has not been reported by other workers.
The precipitate free zone was less apparent in the 30 \( \mu \text{m} \) grain size steel and the intragranular Nb(C,N) was often organised into "knotted" stringers or clusters. The difference in Nb(C,N) precipitation behaviour between the 30 \( \mu \text{m} \) grain size steel and the finer grain sizes probably resulted from the greater degree of carbon supersaturation during heat treatment.

Dislocation loops emitted during growth were easily able to nucleate new Nb(C,N) crystals, so forming "knotted stringers", because the carbon supersaturation was high. Similarly, solute and vacancy depletion near the grain boundary should have had less effect than in the 3 \( \mu \text{m} \) and 12 \( \mu \text{m} \) grain size specimens where the low degree of carbon supersaturation made nucleation correspondingly more difficult. \( \text{M}_6\text{(C,N)} \) showed a marked preference for nucleation and growth of flakes along grain boundaries near sigma phase particles. This was presumably due to the depletion of the matrix in iron and chromium, so encouraging formation of the nickel rich \( \text{M}_6\text{(C,N)} \) assuming a composition close to \( \text{Ni}_2\text{Nb}_2\text{(C,N)} \). These flakes have not been observed in the grain interiors, in agreement with the observation that high dislocation densities are required for their formation (see Literature Review, page 31) and no evidence has been obtained for the growth of \( \text{M}_6\text{(C,N)} \) flakes into adjacent grains; with the steel composition (i.e. cast Z240, page 100) and heat treatments employed (see page 100) \( \text{M}_6\text{(C,N)} \) appeared to be confined to the grain boundaries.

The reason for the lack of intragranular growth of the \( \text{M}_6\text{(C,N)} \) compared with the observations of other workers is not clear, unless carbon supersaturation was again influencing precipitation behaviour.

The X-ray diffraction work on the 12 \( \mu \text{m} \) grain size steel has indicated that the amounts of both Nb(C,N) and \( \text{M}_6\text{(C,N)} \) increased on ageing but that the quantity of \( \text{M}_6\text{(C,N)} \) increased more rapidly, so that after 125d at 700°C the carbonitrides in the 12 \( \mu \text{m} \) grain size steel consisted of 60\%(wt) \( \text{M}_6\text{(C,N)} \), compared with 20\%(wt) in the as heat-treated condition. It has been suggested that \( \text{M}_6\text{(C,N)} \) forms by transformation of Nb(C,N) (Literature Review, page 31) and \( \text{M}_6\text{(C,N)} \) crystals nucleate on a grain boundary spine of Nb(C,N). No clear evidence of a Nb(C,N) to \( \text{M}_6\text{(C,N)} \) transformation has been obtained from the present study. Electron diffraction examination of very thin flakes of
\(M_6(C,N)\) precipitated during ageing at 600°C has not always shown that they are associated with \(\text{Nb}(C,N)\) (see Section 2.4.2). An analysis of the absolute rather than the relative \(\text{Nb}(C,N)\) and \(M_6(C,N)\), as determined here by X-ray powder methods, is necessary to resolve this point.

The amount of the sigma phase, FeCr, present after 125d ageing at 700°C agreed reasonably well with that predicted from the 650°C isothermal section of the Fe-Ni-Cr phase diagram (see Literature Review, page 32) though the influence of the niobium is not, of course, included in the diagram. No published data are available on the influence of carbon on sigma phase formation, although carbon is known to restrict the range of sigma phase precipitation, since it is an austenite stabiliser. In the carburised steel (discussed in Section 2.5) sigma phase was not present in zones containing the \(M_{23}(C,N)_6\) carbonitride, though it was present in carburised samples where the carbon content had not increased sufficiently to bring the steel into the hypostoichiometric composition range, for example, the 75d irradiated specimens (Fig. 2.2). A zone free of sigma phase was generally observed close to the surface of uncarburised specimens tested in an oxidising atmosphere. It seems likely, therefore, that loss of chromium from the austenite, in the first case by \(M_{23}(C,N)_6\) formation and in the second by chromium-rich oxide scale formation, leading to a shift in alloy composition out of the \(\gamma + \sigma\) phase field in a similar manner to the behaviour of AISI 316 steel (see Literature Review, page 32), resulted in resolution of the sigma phase.

Resolution of sigma phase induced by carburisation evidently proceeds rapidly, since aged fatigue specimens tested in argon for 4 or 5 hr showed no sigma phase in the carburised surface zone. This contrasts with the 40d heat treatment at 800°C in non-carburising conditions required to achieve complete dissolution of the sigma phase.
2.6.4 Irradiated Steel

The absence of any clear effect of neutron irradiation on the morphology and distribution of precipitates in the alloy used in this investigation is unexpected when the fact that a considerable irradiation effect on precipitation behaviour in stainless steels has been reported by several workers (see Literature Review, page 50). In the case of the 75d and 125d irradiated steel this may be partly explained by the fairly long irradiation times, since unirradiated controls aged for these times showed virtually complete carbide precipitation (see page 105). However, the persistence of the precipitate free zone after 125d irradiation, at least in the 3\(\mu\)m and 12\(\mu\)m grain size steels, is at variance with the observations of other workers on irradiated 20/25/Nb steel (see Literature Review, page 52). This may indicate that carbon depletion of the matrix near the grain boundary was the controlling factor in the formation of the precipitate free zones, enhanced nucleation due to increased vacancy concentration being of little consequence. Since the degree of carbon supersaturation was low in the 3\(\mu\)m and 12\(\mu\)m grain size heat treatments (discussed in section 2.2.2) the zone near the grain boundary may have had such a low carbon content that nucleation of Nb(C,N) did not occur even if local vacancy supersaturations were available from the displacement spikes (see page 42 of the Literature Review).

2.6.5 Helium Bubbles

The very small numbers of helium bubbles observed in the irradiated steel even after 125d irradiation treatment correlates with the exceptionally low boron content of the 2240 cast (page 100). However, the Ni\(^{58}\) (n,\(\alpha\)) reaction should be the principal source of helium when the boron content is low and the thermal neutron dose high. The contribution of the B\(^{10}\) and Ni\(^{58}\) reactions are shown in Fig. 2.1; the reactions are discussed in the Literature Review, section 1.2.2(c) page 44.

Electron micrographs of the 3\(\mu\)m and 12\(\mu\)m steels used in this investigation did not show the large numbers of intragranular bubbles of about 100A diameter reported by Manley and Rhodes.
in a study of 20/25/Nb steel having a grain size of 30 μm, a boron content of approximately 2 ppm, and subjected to a similar irradiation treatment. The 30 μm grain size steel used in the study described here did show, however, noticeably more intragranular bubble formation than the two finer grain sizes, for a given irradiation treatment, so that possibly the intra- and intergranular gas distribution is influenced by grain size, the finer grain sizes favouring an increase in the extent of intergranular gas bubble formation. This may be because there is a shorter distance for the small bubbles to travel before meeting a grain boundary in fine grained material, there being a correspondingly smaller chance of intragranular formation of larger immobile bubbles (see Literature Review, page 47).

The square relationship between thermal dose and helium contents in the case of the Ni$^{58}$(n,a) reaction (Literature Review, section 1.2.2(c)) implies a factor four difference in the helium contents of 75d and 125d irradiated specimens, whereas the B$^{10}$(n,a) reaction should have been virtually exhausted after 75d, so that if the boron reaction were solely responsible for helium bubble formation there should have been little difference in the bubble contents of the two steels irradiated for the foregoing times. The fact that the steel irradiated for 75d had considerably fewer and smaller bubbles than the 125d irradiated steel lends support to the view that the Ni$^{58}$(n,a) reaction was the principal source of helium. The helium generated from this reaction will be initially distributed uniformly throughout the structure, as also will that from the B$^{10}$(n,a) reaction, with the proviso that there is no segregation to carbonitride particles (boron segregation is discussed in the Literature Review, page 33).

The observation of increased size and numbers of bubbles in specimens irradiated at 700°C and subsequently tested at 800°C may have indicated that a significant proportion of the gas was held in solution at 700°C by the knock-out resolution mechanism (discussed in the Literature Review, page 51 ) and subsequently precipitated out at the higher test temperature of 800°C. However, helium precipitation on annealing generally appears to be associated with rather lower irradiation temperatures (approx 500°C) than that employed in this study and so an alternative explanation may be the thermally activated coalescence of very small bubbles into larger ones.
The reason for the observation of greater numbers of grain boundary bubbles away from precipitate particles (see section 2.5) is not clear. This was not likely to be due to resolution, caused by fast alpha particles from boron segregated into grain boundary carbonitrides (Literature Review, page 33) in view of the negative result of the autoradiographic examination (section 2.5, p. 111). Also, since it appears that the \( \text{Ni}^{58} (n,\alpha) \) reaction is the principal source of helium, carbonitrides would not be expected to influence bubble distribution. The effect may simply have been due to the obscuring of bubbles by electron opaque carbonitride particles, giving an impression of fewer bubbles around them, or to easier bubble nucleation away from carbon-nitride precipitates due to less competition for vacancies from precipitate nuclei.

2.6.6 Carburisation

In the unirradiated specimens tested in argon atmosphere (see section 2.5), in which carburisation occurred, the carburising atmosphere was probably derived from cracked hydrocarbons from the vacuum seals. The degree of carburisation was often greatest on the side of the specimen facing the furnace vacuum seal. In the case of the carburised irradiated specimens, the helium atmosphere of the reactor container may have had trace amounts of hydrogen, possibly from hydrogen-producing nuclear reactions, which may have acted as a carrier gas transporting carbon from cooler parts of the container to the specimens. Carburisation of this kind has previously been detected in tensile test pieces of the irradiated Nimonic alloy PE16(25).

Since the Nb: (C+N) ratio of the cast Z240 is approximately 13:1 (see page 100) a doubling of the amount of carbon should be necessary to obtain the \( \text{M}_{23} \text{(C,N)}_6 \) carbonitride (see Literature Review, page 28). The surface of the 75d irradiated test pieces showed an increase of approximately this extent (Fig. 2.2) but since \( \text{M}_{23} \text{(C,N)}_6 \) was not detected in the metallographic examination the amount present, if any, must have been small. The differences in \( \text{M}_{23} \text{(C,N)}_6 \) precipitation behaviour between irradiated and unirradiated specimens probably reflect the different lengths of time in which carburisation had occurred; 125d in the irradiated
specimen (Plate 2.30) and 5hr in the unirradiated specimens tested in argon atmosphere (Plates 2.31, 2.32). In the former, carburisation proceeded slowly so that extensive intragranular precipitation, on twin boundaries and on dislocations, was possible. In the latter case the short time was evidently only sufficient for predominantly grain boundary precipitation to occur so that the carbonitride was present chiefly as coarse grain boundary particles.

The faster carburisation in the unirradiated argon tested specimens compared with the irradiated specimens was probably due to the easier access to the metal surface caused by fracture of the surface oxide film during fatigue (Plt.2.27). The higher initial carbon content of the cast Z240 steel (see page 100) favoured the formation of $M_{23}(C,N)_6$ during carburisation, so that many large carbonitride particles were present in this steel (Plate 2.32). In the low carbon steel, cast Z227, the $M_{23}(C,N)_6$ clusters observed were probably due to carbonitride precipitation around the chromium-rich sigma phase particles in the early stages of precipitation.

The tendency for $M_{23}(C,N)_6$ to be present as globular particles in the cast Z240 steel but spread out along grain boundaries in the low-carbon Z227 steel (Plates 2.31, 2.32) may perhaps be related to the carbonitride-austenite interfacial energy. The coarsening and globular morphology of the carbonitride particles on heating at 880°C for 60hr (see page 108) suggests that the interfacial energy is sufficiently large for there to be a significant overall reduction in total free energy by minimising the carbonitride-austenite interfacial area. This behaviour has not been observed with Nb(C,N) and $M_6(C,N)$ carbonitrides in the centres of the carburised specimens, however.

The $M_6(C,N)$ carbonitride appeared to redissolve rapidly in the carburised zone since it was not detected there in X-ray powder photographs or residues from the surface of the specimen heat-treated for 60hr at 880°C to remove sigma phase, as mentioned in the previous paragraph.

The surface carburisation of the unirradiated steel clearly had a deleterious effect on LCF endurance (discussed on page 109). In tests at 800°C in argon atmosphere at strain rates of $1 \times 10^{-4}$ and $3.5 \times 10^{-4} \sec^{-1}$ the endurance was reduced by a factor of approximately 10, and in tests at 700°C at a strain rate of $3 \times 10^{-4} \sec^{-1}$, by a factor of five.
The initial carbon content of the steel had no detectable effect on endurance (Fig. 2.4) and likewise prior ageing for 125d at 700°C of the higher carbon Z240 cast steel had no perceptible effect on the LCF endurance over that of the as heat treated steel (Figs. 2.3i, 2.4). These results may be explained by the considerable increase in carbon content of the carburised layer, which swamped any influence of prior carbon content or ageing treatments.

The reduction in LCF endurance of the unirradiated carburised steel appeared to be brought about by an increase in crack propagation rate of cracks generated at the specimen surface (See page 109), a similar effect has been reported in AISI 316 stainless steel\(^{(258)}\). There was no evidence for internal cracking or void formation at M\(_{23}(C,N)\)-austenite interfaces. The increase in propagation rate may have been a result of poorer adhesion between carbonitride and austenite matrix as a consequence of higher interfacial energy. Alternatively the presence of many M\(_{23}(C,N)\)\(_6\) particles in the grain boundary may have influenced the deformation characteristics of the steel. This is discussed further in section 3.4.

Carburisation resulting in formation of M\(_{23}(C,N)\)\(_6\) in the irradiated steel was confined to those specimens subjected to the 125d irradiation treatment at a temperature of 700°C. Assessment of the effect of this carburisation on the LCF endurance of the irradiated steel was complicated by the variation in the surface carbon contents of specimens occupying different positions in the irradiation can and further complicated by the lack of 125d irradiated control specimens free from carburisation for comparison tests. If 75d irradiated specimens were used as controls, it is evident that at 800°C and a strain rate of \(2 \times 10^{-5} \text{ sec}^{-1}\) the small amount of carburisation present in the 125d irradiated specimens (Plate 2.37) had at most only a small effect on LCF endurance (Fig. 2.7) at a plastic strain amplitude of 2%. In fact, the very small reduction in endurance may equally well be ascribed to the greater irradiation dose in the latter specimens. At a plastic strain amplitude of 1%, the LCF endurance of the carburised irradiated steel was almost the same as that of the 125d unirradiated thermal control test, so that under these conditions neither the presence of M\(_{23}(C,N)\)\(_6\) or irradiation embrittlement were having any influence on the LCF properties.
The irradiated specimens used for the LCF tests at 800°C and a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ had a greater degree of carburisation than the specimens used for the tests described in the previous paragraph, again, probably due to their position in the irradiation can. It is clear, however, from the LCF test results shown in Fig. 2.8, that prior surface carburisation had no effect on the endurance of the steel at a plastic strain amplitude of 1.5%.

At lower plastic strain amplitudes, at approximately 0.8%, there may have been slight reduction of endurance.

The absence of any significant influence of carburisation on the LCF endurance of irradiated specimens may be accounted for in two possible ways:

1. The overall smaller degree of carburisation of the irradiated specimens compared with the unirradiated argon atmosphere tested specimens.

2. Irradiation effects brought about the principal loss of LCF endurance and so tended to mask any influence of carburisation.

The first possibility appears to be the most likely since the reduction of LCF endurance caused by prior irradiation treatments is generally not as great as the tenfold reduction caused by carburisation in the tests on unirradiated specimens.
FIG. 2.1 CONTRIBUTIONS OF THE $^{10}\text{B}$ AND $^{58}\text{Ni}$ REACTIONS TO THE HELIUM CONTENT IN 20-25-Nb STEEL (BORON CONTENT 0.2 ppm)
FIG. 2.2 CARBON CONCENTRATION PROFILES FOR CARBURISED STEEL, (NUCLEAR MICROPROBE ANALYSIS)
FIG. 2.3 REDUCTION OF ENDURANCE IN ARGON ATMOSPHERE

FIG. 2.4 REDUCTION OF ENDURANCE IN ARGON ATMOSPHERE
LOW CARBON CAST Z227
FIG. 2.5 REDUCTION OF ENDURANCE IN ARGON ATMOSPHERE
EFFECT OF INITIAL CARBON CONTENT

FIG. 2.6 REDUCTION OF ENDURANCE IN ARGON ATMOSPHERE
EFFECT OF 700°C TEST TEMPERATURE
FIG. 2.7 THE MINOR EFFECT OF CARBURISATION ON ENDURANCE AT 2% PLASTIC STRAIN AMPLITUDE

FIG. 2.8 AS ABOVE, $\Delta \varepsilon_p = 1.5\%$

* RESULTS DUE TO G H BROOKFIELD
3.1. Introduction

In this chapter the results of low cycle fatigue and tensile tests on unirradiated 20/25/Nb stainless steel are presented, followed by the results of metallographic examination of the fractured specimens. The influence of various mechanical test variables is dealt with in turn, and a model is discussed which relates low cycle fatigue properties and fracture characteristics. A semi-quantitative analysis of the low cycle fatigue endurance is presented using a modified form of a published fatigue theory. On the basis of this analysis a low cycle fatigue parameter is proposed for the correlation of test results at both ambient and elevated temperature.

Certain changes in grain boundary structure are also interpreted semi-quantitatively using a new dislocation model developed by N. Louat.
3.2. Test Methods

3.2.1 Low Cycle Fatigue Specimens and Test Equipment

Some of the details of the LCF specimens and test equipment used in the investigation have been described elsewhere. The test equipment was designed for elevated temperature, controlled atmosphere LCF tests in the bending mode, on thin section stainless steel specimens of the order of 0.5 mm thickness. The compositions and heat treatments of the steel used have been given previously in Chapter 2, section 2.2.

Two designs of LCF specimens were employed, designated Type 1 and Type 2 respectively, and are illustrated schematically in Figs. 3.1 and 3.2. Type 1 specimens were stamped from 0.625 mm thick strip and the test areas formed by machining to a thickness of 0.5 mm, while Type 2 specimens were simply stamped directly from 0.5 mm strip and were therefore of constant thickness throughout.

In the LCF test equipment, the specimens were held by a clamp in the central area and the two ends deflected by knife edges, so producing a cyclic bending moment (illustrated in Fig. 3.4(a) and (b)). The reduced width and the reduced thickness at the test areas in the case of Type 1 specimens, and the reduced width only in the case of Type 2 specimens, ensured that the cyclic strain was concentrated in the test areas.

Calculation of cyclic plastic strain applied during testing was simplified using Type 1 specimens, since the reduced thickness of the test area ensured that a virtually constant radius of curvature, and therefore strain, was obtained over the test area. However, it was difficult to remove marks and surface irregularities produced by the machining of the test areas; electropolishing was not successful. Type 2 specimens, on the other hand, being of constant thickness, could easily be polished to metallographic standards, but suffered the drawback that the radius of curvature during bending was not constant over the test area, so that the cyclic plastic strain could not be determined as accurately as with Type 1 specimens. (Stress and strain measurements are described later in section 3.3).
Type 2 specimens were therefore used for the surface cracking examinations to be described in section 3.5. Type 1 specimens were used wherever possible for determining the LCF mechanical properties and were not employed for the surface cracking studies.

Referring to Figs. 3.4(a) and (b): The specimen was held in a remotely operated clamp mounted on a stainless steel rod positioned centrally inside a tube on which the knife edges were fixed. The two ends of the specimen projected out of the tube between the knife edges and were deflected up and down by them as the tube moved up and down. The tube itself was attached to a crosshead whose two columns passed through vacuum seals down to the driving gear. The clamp supporting rod likewise passed through a vacuum seal to the load cell underneath. The displacement of the knife edge support tube was measured by a servogenerator attached to the driving mechanism, which consisted of a motor-gearbox combination. Since the specimen deflection was the controlled parameter with this test equipment, the LCF tests were therefore all of the strain cycling type (see Literature Review, section 1.3.1(a)).

A single pen chart recorder was used to record the load and knife edge displacement, but instead of the usual fixed chart speed the output from the servogenerator was used to drive the chart advance mechanism. To avoid a reversal of the chart motion during the compression half cycle of the LCF test, the polarity of the servogenerator output was reversed, so that a continuous record of the load against knife edge displacement was obtained. The appearance of the recorder trace obtained for one LCF cycle is shown schematically in Fig. 3.5(a); the portions of the recorder trace corresponding to the various parts of the LCF hysteresis loop being shown in Fig. 3.5(b) (see also Literature Review, section 1.3.1(a)). The short dwell period at zero load occurred because a small clearance between knife edges and specimen was necessary, partly to facilitate specimen loading and partly to avoid cross binding.

Test temperatures up to 900°C were obtained using a "top hat" type furnace having a stainless steel lining. A conventional oil diffusion pump was used to obtain pressures down to $2 \times 10^{-5}$ torr.
3.2.2 Tensile test specimens and Equipment

Thin section tensile test specimens were used, shown schematically in Fig. 3.3. Instead of the pressure grips normally used for sheet specimens, circular "buttons" were bolted onto the heads of the tensile specimens using a special jig. The buttons slotted into hollow grips, so that the specimen was automatically aligned with the machine axis on loading; this feature was particularly useful when handling irradiated specimens remotely. Test temperatures and pressures similar to those of the LCF testing machines were available.
3.3. Experimental Procedures

3.3.1 Mechanical Testing

Most of the mechanical tests were carried out using the cast Z240 steel, heat treated to produce a grain size of 12 μm, and in some cases subsequently given ageing treatments. A few tests intended to investigate grain size effects were done using the 3 μm and 30 μm grain size steels.

The LCF test variables were temperature, strain rate and plastic strain amplitude (the latter is discussed in the Literature Review, section 1.3.1(a)); all tests were carried out in vacuo. The LCF and tensile properties were studied over the temperature range 400 to 850°C. Strain rates employed ranged from a minimum of $2 \times 10^{-5}$ sec$^{-1}$ up to approximately $1 \times 10^{-3}$ sec$^{-1}$ in the LCF tests, while in the tensile tests they ranged from $2 \times 10^{-5}$ sec$^{-1}$ up to approximately $1 \times 10^{-2}$ sec$^{-1}$.

The plastic strain amplitudes in the LCF tests covered the range 0.5% to 4%, the latter representing the maximum possible with the equipment.

In the LCF tests, the number of cycles required for complete rupture of the specimen was not a satisfactory definition of LCF endurance in reversed bending since the plastic strain amplitude decreased to zero as the crack approached the neutral axis of the specimen; this led to a rapid fall-off in the rate of crack propagation as the specimen approached the point of rupture, and consequently made it difficult to determine the number of cycles precisely because the load decreased gradually. To avoid this problem the point of failure was defined as the number of cycles at which 50% load reduction from the starting value had occurred, and the test was stopped. However, even at 50% load reduction some considerable distortion of the specimen could occur, owing to the presence of a large crack, which resulted in a large degree of scatter in the number of cycles to failure. Therefore the LCF endurance was defined as the number of cycles required to produce a load reduction of 20% of the initial value. Consequently in the following chapters "endurance" has the foregoing meaning and "failure" is the number of cycles required for 50% load drop, at which all the tests were stopped.
Plastic strain amplitudes for Type 1 specimens were calculated assuming that the specimen test area was bent into a cylindrical section. The angle of bending was related to the knife edge displacement and test area to knife edge distance and incorporated into a formula (Appendix 1). The plastic strain amplitude was determined by subtracting the elastic component from the total determined from the hysteresis loop (Fig. 3.6) (see also Literature Review, section 1.3.1(a)) and the results were confirmed by measurements of radii of curvature as mentioned in section 3.2.1.

Maximum plastic strain amplitudes for Type 2 specimens were determined by measurements of radii of curvature along the test zone in specimens bent to known displacements. It was found that the formula derived in Appendix 1 for Type 1 specimens gave reasonable values for the maximum plastic strain amplitude in Type 2 specimens and so the same method of calculation was employed for both specimen types.

Since under strain cycling conditions the total strain varies linearly with time, strain rates in the LCF tests were determined by dividing the total strain by half the cycle time.

Surface stresses were calculated using an adaptation of Nadai's method for bend tests on materials with an unknown stress-strain relationship (Appendix 2). Stress calculations were only performed using data obtained using Type 1 specimens.

No extensometer was employed for the tensile tests. True strains were calculated using the relation: \( \epsilon' = \ln(1 + \epsilon) \) and true stresses calculated from: \( \sigma' = \frac{L}{A_0} (1 + \epsilon) \), where \( L \) = Load, \( A_0 \) = initial cross sectional area, \( \epsilon = \) total strain.

3.3.2 Metallographic examination

The LCF fracture characteristics were studied using three techniques:

(i) Conventional microsections
(ii) Fractography using a Scanning Electron Microscope (SEM)
(iii) Optical microscope and SEM examination of a surface of the specimen metallographically polished before LCF testing.
Etching procedures used for microsections have been described previously in Chapter 2, section 2.3.1. In the elevated temperature tests, an oxide deposit formed on the fracture surfaces which had to be removed before fractographic examination in the SEM. This was accomplished using a molten sodium hydroxide-sodium hydride salt bath. This descaling bath, developed by ICI-Mond Ltd., dissolves many oxides without damaging the metal substrate. Sodium pellets are dissolved in a molten bath of sodium hydroxide at a temperature of about 350°C in order to dehydrate it (detected by a change of colour from brown to black) and then provide sodium hydride by reaction with hydrogen bubbled through, the excess hydrogen being burnt off by a pilot light. Specimens were immersed for about two minutes and then washed in water, followed by ultrasonic cleaning in distilled water and drying in acetone. The descaling treatment was also applied to the pre-polished specimens when necessary.
3.4. Results - Mechanical Properties

3.4.1 Low Cycle Fatigue Tests

The setting of the knife edge gap prior to starting a LCF test could not be done with sufficient precision to ensure reproducible values of the plastic strain amplitude. Consequently, whenever possible, the endurance at a given plastic strain amplitude was determined by interpolating the required value from a plot of plastic strain amplitude against endurance (i.e. a Coffin-Manson plot). Measurements of cyclic strain hardening exponents and cyclic strain rate sensitivity exponents (see Literature Review, section 1.3.1(a)) were carried out by plotting surface stress amplitude against surface plastic strain amplitude and strain rate respectively (using Type 1 specimens). Engineering stresses and strains were used for these determinations since the low strains involved in the LCF tests meant that the engineering and true stress or strain values only differed by an insignificant amount. In the tensile tests, however, true stresses and strains were calculated.

3.4.1(a) As Heat Treated Steel

The test programme is summarised in Table 3.1.

The dependence of endurance on plastic strain amplitude (i.e. Coffin-Manson plots) using Type 2 specimens, is shown in Figs. 3.7 to 3.10 and Fig. 3.12 for temperatures from 500°C to 800°C, and at a constant strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$. The effect of strain rate has been studied primarily at a test temperature of 800°C, and the data are shown in Figs. 3.11 to 3.14.

The effect of test temperature on the endurances at a fixed plastic strain amplitude of 1.5% and fixed strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ is shown in Fig. 3.15. The interpolated endurance values, i.e. those read off from the plastic strain amplitude-endurance plots as described above, cover the temperature range 500 to 800°C. An additional value for a test temperature of 400°C is included at a plastic strain amplitude of 1.49%, and strain rate of $2 \times 10^{-4} \text{ sec}^{-1}$. Two more endurance values at a test temperature of 850°C are also included in Fig. 3.15, the plastic strain amplitudes being 1.58% and 1.64% respectively and the strain rate $1 \times 10^{-4} \text{ sec}^{-1}$. 
<table>
<thead>
<tr>
<th>SPEC. TYPE</th>
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<th>TEMP. (°C)</th>
<th>PLASTIC STRAIN AMPLITUDE (%)</th>
<th>STRAIN RATE (S⁻¹)</th>
<th>FIGURES</th>
</tr>
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<td>2 X 10⁻⁵</td>
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<td>12</td>
<td>75d</td>
<td>800</td>
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<tr>
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<td>12</td>
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<td>800</td>
<td>1.00</td>
<td>2 X 10⁻⁵</td>
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</tr>
<tr>
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<td>800</td>
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<td>(Vacuum 1 X 10⁻⁴ torr)</td>
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<td></td>
<td>800</td>
<td>2.67</td>
<td></td>
<td></td>
</tr>
<tr>
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<td>12</td>
<td>75d</td>
<td>700</td>
<td>1.64</td>
<td>1 X 10⁻⁴</td>
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</tr>
<tr>
<td>1</td>
<td>12</td>
<td>75d</td>
<td>700</td>
<td>1.60</td>
<td>1.4 X 10⁻³</td>
<td>3.28</td>
</tr>
<tr>
<td></td>
<td></td>
<td>700°C</td>
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</table>
The 400C result is included since the somewhat higher strain rate used in the test is not likely to affect the endurance much compared with 1 x 10^-4 sec^-1 at this temperature, in view of the relatively small variation of endurance with strain rate found at higher temperatures to be discussed shortly. The plastic strain amplitudes in the tests carried out at 850C are only marginally greater than 1.5% and so these results can be included in Fig. 3.15.

The most prominent, and unexpected, feature of the endurance-temperature plot in Fig. 3.15 is the minimum which occurred at about 600 to 650C. The endurance recovered above this temperature range and decreased again above 800C. The endurance recovery at 700 to 800C was associated with a further decrease in the slope of the plastic strain amplitude-endurance curve (Fig. 3.16), that is the Coffin-Manson exponent "\( \alpha \)" (see Literature Review, section 1.3.3(a)) decreased over the range 500 to 800C. A value of \( \alpha \) for a temperature of 750C is not shown in Fig. 3.16, since insufficient data are available.

The influence of strain rate on LCF endurance at fixed plastic amplitude is illustrated in Fig. 3.17 to 3.20. At a test temperature of 800C and plastic strain amplitude of 1.9% there was clearly little or no strain rate effect on LCF endurance over the range 2 x 10^-5 to 6 x 10^-4 sec^-1 (Fig. 3.20); but at 800C and plastic strain amplitude of 1.5% (Fig. 3.19) there was apparently some reduction in endurance at the highest strain rates of 6 x 10^-4 and 1.2 x 10^-3 sec^-1. Only two endurance values are available at each of the two lower test temperatures 600 and 700C, and there appears to be no noticeable strain rate effect.

Values of cyclic strain hardening exponent "\( \beta \)" at a strain rate of 1 x 10^-4 sec^-1 and also cyclic strain rate sensitivity exponent "\( m \)" (see Literature Review, section 1.3.1(a)) are plotted as functions of test temperature in Fig. 3.21, the plastic strain amplitude being fixed at 1.5%. The variation of \( \beta \) and \( m \) with strain rate at a test temperature of 800C is shown in Fig. 3.22, where the equivalence of increasing strain rate and decreasing temperature is apparent in the two aforementioned Figures. The results for the variation of strain rate sensitivity exponent with plastic strain amplitude are not presented since the dependence was small over the range of plastic strain amplitude employed in this investigation.
Since the strain rate sensitivity exponent is grain size dependent (see Literature Review, 1.3.1(a)) values were determined for the 3 μm and 30 μm grain size steels, and the results are shown in Fig. 3.23. The increase in strain rate sensitivity exponent with decreasing grain size at 700 and 800°C, and increasing temperature in the range 600 to 800°C is apparent, although the increase was not large.

The strength parameter "K" (see Literature Review, 1.3.1(a)) is shown as a function of temperature in Fig. 3.24 for the 12 μm grain size steel, the strain rate being $1 \times 10^{-4}$ sec$^{-1}$. Cyclic hardening (also discussed in the Literature Review, section 1.3.1(a)) occurred at test temperatures up to 700°C, the hardening resulting in an increase of approximately 15% in the stress amplitude, $\Delta\sigma$ within the first ten cycles of the test. No hardening was observed at higher temperatures.

Estimates of the crack propagation constant "A" were made assuming that the LCF crack propagation behaviour was described by the propagation law $dl/dn=A1$ (see Literature Review, section 1.3.3(e)) where A is the "propagation constant", $dl/dn$ the crack growth per cycle, and l the instantaneous crack length. In view of the small specimen size and tendency for multiple LCF crack formation in them, direct measurement of crack propagation rate was not feasible in this investigation. Instead, mean crack depths were estimated using the reductions in load occurring towards the end of the tests, mentioned earlier on page. The steel was assumed to behave in an elastic-perfectly plastic manner, so that given a distribution of cracks of uniform length, the load would be directly proportional to crack length. These assumptions were considered to be justifiable at least at elevated
temperatures, since under these conditions the strain hardening effect is negligible, and it was known from the metallographic studies described in the next section that multiple cracks of similar size tended to form.

Integration and rearrangement of the propagation equation gives:

\[ N_f - N_0 = \frac{1}{A} \ln \left( \frac{l_f}{l_0} \right) \]

where \( l_0 \) and \( l_f \) are the initial and final crack lengths produced by \( N_0 \) and \( N_f \) LCF cycles respectively. The initial crack length was taken as that at 5% load drop; the mean crack length was then assumed to be 5% of the distance from the specimen surface to the neutral axis, i.e. 12.5 \( \mu \)m. Similarly, the crack lengths at 10, 20 and 50% load drop were taken as 25, 50 and 125 \( \mu \)m. Taking these as values of the final crack size \( l_f \), the function \( \ln \left( \frac{l_f}{l_0} \right) \) was calculated and plotted against \( (N_f - N_0) \) for tests at various strain amplitudes at a temperature of 800°C and strain rate of 3.5 \( \times \) 10\(^{-4}\) sec\(^{-1}\) (Fig. 3.25).

Some deviation from the expected linear relationship is apparent in Fig. 3.25, a decrease in slope and therefore in the propagation constant "A" occurring as \( N_f - N_0 \) increase. This was probably because the plastic strain amplitude would have decreased as the mean crack size increased, since the effective thickness of the specimen would have thereby been reduced, the deflection of the specimen by the knife edges being constant.

For comparison purposes an arbitrary value of A was determined by taking a straight line between the 20% and 50% load reduction points. Since in the LCF test \( N_0 \) is zero and therefore \( N_f = \frac{1}{A} \ln(l_f/l_0) \) a linear relationship should be found between LCF endurance and reciprocal propagation constant, since \( \ln \left( \frac{l_f}{l_0} \right) \) is a constant.
Values of $l/A$ determined from Fig. 3.25 are plotted against endurance in Fig. 3.26 and a reasonably good straight line is obtained.

Crack propagation constants were determined for tests at a plastic strain amplitude of 1.5% and strain rate $1 \times 10^{-4}$ sec$^{-1}$ for a range of temperatures from 500 to 800°C (Fig. 3.27). The decrease in the propagation constant above 600°C correlates with the upturn in the endurance-temperature plot discussed on page 136 and shown in Fig. 3.15.

3.4.1(b) Heat Treated and Aged Steel

Insufficient specimens were available to study the effect of plastic strain amplitude on endurance over the whole range of temperature and strain rate investigated for the steel in the heat treated only condition. Consequently, interpolated values of endurance at a fixed plastic strain amplitude of 1.5%, corresponding to those of the previous section (3.4.1(a)), were not available. However, the influence of test temperature on LCF endurance of aged steel at a constant strain rate of $1 \times 10^{-4}$ sec$^{-1}$ has been investigated with only a relatively small deviation from 1.5% plastic strain amplitude, as follows: (see also Table 3.1)

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Plastic Strain Amplitude</th>
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<tr>
<td>500°C</td>
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</tr>
<tr>
<td>600°C</td>
<td>1.72</td>
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<tr>
<td>700°C</td>
<td>1.64</td>
</tr>
<tr>
<td>800°C</td>
<td>1.51</td>
</tr>
</tbody>
</table>

The steel was given a prior ageing treatment of 75d at 750°C and the results are presented together with those for the as heat treated steel in Fig. 3.15. It may be noted that the results for the two different conditions of the steel differ very little.

Two tests at strain rates of $1 \times 10^{-4}$ sec$^{-1}$ and $1.4 \times 10^{-3}$ sec$^{-1}$ at 700°C are shown in Fig. 3.18, the plastic strain amplitudes were 1.64 and 1.60 respectively. Comparison with the as heat treated steel in Fig. 3.18 shows that there was little difference between the as heat treated steel endurance and the endurance of the aged steel under these test conditions.

In contrast, when tested at 800°C at a lower strain rate of $2 \times 10^{-5}$ sec$^{-1}$, the aged steel showed a markedly inferior LCF endurance to that of the as heat treated steel (Fig. 3.28). In order to see whether sigma phase precipitation was responsible for
this effect an aged specimen was heat treated for 60hr at 880C to dissolve the sigma phase (this treatment is described in Chapter 2, section 2.4.2, page 104) and thereafter fatigued under the previous test conditions. The endurance was not affected by this treatment.

Measurements of the cyclic strain hardening exponent $\beta$ and the cyclic strain rate sensitivity exponent $m$ were made, to see if these parameters would indicate any structural differences between the aged and as heat treated steels that could account for the different LCF endurances at 800C and $2 \times 10^{-5}$ sec$^{-1}$ strain rate. The same values were obtained, however, for both aged and as heat treated steel.

In addition to the aforementioned ageing effect on LCF endurance under the specific test conditions of 800C and strain rate $= 2 \times 10^{-5}$ sec$^{-1}$, there was a dependence on the vacuum quality not observed under other combinations of test conditions. An increase in pressure from $2 \times 10^{-5}$ torr to $1 \times 10^{-4}$ torr (approximately) resulted in reduced LCF endurance at strain amplitudes below about 3% (Fig. 3.29).

A pronounced peak developed at the maximum strain point of the hysteresis loop (hysteresis loops are described in the Literature Review, section 1.3.1(a)) of the specimens tested in the poor quality vacuum of $1 \times 10^{-4}$ torr, towards the end of the LCF test. This was probably due to wedging of the LCF crack by oxide during the compression half of the cycle, resulting in an increase in load as the oxide-coated fatigue crack surfaces were forced together.

Estimates of the crack propagation constant "A", determined as described on page 137, are shown in Fig. 3.27.

3.4.2 Tensile Tests

The test programme is summarised in Table 3.2. The variation of ductility of the as heat treated steel with test temperature at a fixed strain rate of $1 \times 10^{-4}$ sec$^{-1}$ is shown in Fig. 3.31 over the range 300 to 800C. The ductilities at 300 to 500C were slightly reduced relative to those at room temperature but substantial increases occurred at higher temperature so that the ductility at 800C was approximately double the value at 300 to 500C.
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<th>AGEING TREATMENT</th>
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<tr>
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<td></td>
<td>800</td>
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The variation of ductility with strain rate for the as heat treated and heat treated and aged steels at temperatures of 600, 700 and 800°C is presented in Fig. 3.31. At 800°C, the tensile ductilities were not significantly dependent on strain rate in the range $1 \times 10^{-2}$ to $1 \times 10^{-4}$ sec$^{-1}$; there were slight increases at 600 and 700°C with decreasing strain rate in this range. The Ultimate Tensile Stress (UTS) is plotted as a function of temperature for a constant strain rate of $1 \times 10^{-4}$ sec$^{-1}$ in Fig. 3.32. The UTS decreased progressively from room temperature up to about 500°C, and thereafter more rapidly up to 800°C. The variation of UTS with strain rates from $1 \times 10^{-4}$ up to $1 \times 10^{-2}$ sec$^{-1}$ is shown in Fig. 3.33. The UTS was only marginally dependent on the strain rate at 600°C, but decreased markedly with decreasing strain rate in the 700 and 800°C tests.

The tensile strain hardening exponent and strain rate sensitivity exponent were determined for the 12 μm grain size steel at test temperatures from 400 to 800°C for comparison with the corresponding cyclic values and are shown in Fig. 3.34. The effect of grain size is illustrated in Fig. 3.35. The "change rate" technique was used to determine the tensile strain rate sensitivity exponent, as follows:

(i) The specimen is loaded at the lowest strain rate required until the flow stress is reached, i.e. fully plastic conditions exist.

(ii) The strain rate is increased sharply to the next value so producing a corresponding increase in the flow stress.

(iii) The procedure is repeated until the maximum strain rate required has been reached.

A series of steps corresponding to the increased value of flow stress with each strain rate increment will have been obtained. If log (flow stress) is then plotted against log (strain rate) a linear relation will be obtained; the slope of the line is the strain rate sensitivity exponent. In general the linear relation is approximate only, the strain rate sensitivity tending to decrease with increasing strain rate, but over a restricted strain rate range (say one to two decades) the linear approximation is reasonably good. The tensile strain hardening exponent and strain rate sensitivity exponent were very similar to the corresponding cyclic values (cf. Figs. 3.21 and 3.34).
3.5. Results - Metallographic Examination of Tested Specimens

In the case of the as heat treated steel, the influence of test temperature, strain rate and plastic strain amplitude on the LCF fracture characteristics was studied. Test temperature was the only variable examined in the heat treated and aged steel.

Specimens tested to failure (i.e. until a 50% load drop occurred) and others tested for a predetermined number of LCF cycles were examined in order to study both crack initiation and propagation.

The effects of test temperature and strain rate on the tensile fracture characteristics were evaluated by the examination of specimens tested to rupture.

3.5.1. Low Cycle Fatigue Fracture of the As Heat Treated Steel

The programme of metallographic work is summarised in Table 3.3. The examinations were carried out on specimens previously tested under fixed conditions of strain rate \(1 \times 10^{-4}\) sec\(^{-1}\) and plastic strain amplitude (1.5%).

3.5.1(a) The Influence of Test Temperature

400C

Optical microscope examinations of a fractured specimen whose surface had been polished prior to testing (see section 3.3.2) revealed the presence of many small, irregular cracks, generally associated with pronounced slip bands (plate 3.1). Fracture often occurred along the edges of slip bands (plate 3.2) the fractures sometimes linking up by cracking at right angles to the slip band direction. It was sometimes observed that sections of the cracks did not appear to be associated with slip bands. Oxidation was very slight at this temperature and was not sufficient to produce any etching of the grain boundaries.

Surface examination using the SEM confirmed the optical microscope findings and facilitated distinction between slip band boundaries and true fissures. Comparison of the optical and SEM micrographs demonstrated that the dark contrast in the optical micrographs was chiefly due to slip band boundaries (Plate 3.3). The development of a slip band fissure is shown at higher magnification in Plate 3.4.

The fracture surfaces and microsections of the specimens tested at 400C were not examined.
# TABLE 3.3
METALLOGRAPHY PROGRAMME FOR THE AS-HEAT TREATED STEEL

**KEY TO ABBREVIATIONS:**
- **F:** CYCLED TO FAILURE
- **S:** SURFACE EXAMINATION OF PREPOLISHED SURFACES
- **M:** MICROSECTIONS
- **E:** SCANNING ELECTRON MICROSCOPE

<table>
<thead>
<tr>
<th>GRAIN SIZE (μm)</th>
<th>LCF CYCLES</th>
<th>TEST TEMPERATURE (°C)</th>
<th>PLASTIC STRAIN AMP. (%)</th>
<th>STRAIN RATE (s⁻¹)</th>
<th>TYPE OF EXAMINATION</th>
<th>PLATES</th>
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<td>S,E</td>
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<td></td>
<td>2 X 10⁻⁵</td>
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</table>
A specimen fatigued for 30 cycles showed some slip band formation (Plate 3.5) but no fissures were observed. After 60 cycles, small cracks were visible at the edges of the specimen. The specimen cycled to failure (1280 cycles) had a similar appearance to the 400°C tested specimen, with considerable slip band structure around the fractures (Plate 3.6). The surface oxidation at this test temperature was sufficient to etch the grain boundaries, so it could be easily seen that the fracture mode was predominantly intergranular.

Optical examination of a specimen fatigued for 30 cycles showed incipient intergranular fracture (Plate 3.7) and many small oxidation pits; slip bands were not visible. After 60 cycles the intergranular cracking was well developed with some slip band structure evident (Plate 3.8). After failure, (900 cycles) intergranular cracks had propagated across the specimen surface (Plate 3.9) and the slip bands were distributed fairly uniformly and not concentrated adjacent to the fracture, as in the tests at lower temperatures.

SEM examination of the surfaces of the specimen tested to failure showed the presence of pits in the grain boundaries, similar but larger than those in the grain interiors (Plate 3.10). These were not deformation induced cavities since they were also present in the unstrained ends of the specimen; they were therefore oxidation pits.

Fracture surfaces and microsections were not examined.

Optical micrographs of a specimen tested for 30 cycles showed many inter- and intragranular oxidation pits and incipient intergranular cracks (Plate 3.11). After 60 cycles many short intergranular cracks were present (Plate 3.12) and the network of grain boundary oxidation pits was practically continuous. It is tempting to suppose that the oxidation pits acted as crack nuclei but there was no proof of this.

Low power optical examination of the surface of a specimen tested for 130 cycles yielded a quite unexpected result, in that instead of the intergranular fracture expected at this temperature,
the cracks were straight and orientated at right angles to the applied strain direction (Plate 3.13). Examination at higher magnification showed that the surface fracture mode was mixed, intergranular sections being linked by transgranular sections, (Plate 3.14).

The transgranular sections were not always fully developed fractures at this stage but were often only shallow depressions in the grain surface (indicated by an arrow on Plate 3.15). After 530 cycles, however, the transgranular sections were well developed fractures (Plate 3.16). A single large crack, the principal fracture, was present at the specimen edge.

A further difference in the appearance of this type of fracture compared with the normal trans- or intergranular type was that there appeared to have been some displacement of the edges of the cracks, which gave a dark contrast effect at low magnifications, indicated by an arrow in Plt. 3.17. After testing to failure, 1005 cycles, this displacement around the cracks was so great that the surface of the specimen was very distorted, so making focusing at high magnifications difficult.

In the specimen tested to failure, cross-linking of the cracks occurred by the formation of short fractures approximately parallel to the strain direction (indicated by an arrow in Plate 3.18). These cross-linking fractures were particularly prominent near the principal fracture and were evidently instrumental in forming the main crack from a series of shorter ones. The cross-linking cracks in the principal fracture were often surrounded by rectangular patterns of lines which may have been further subsidiary fractures (indicated by an arrow on Plate 3.19). Examination of scratches crossing grain boundaries indicated that no grain boundary sliding had occurred.

SEM examination of the specimen fatigued for 60 cycles confirmed the presence of intragranular embryo cracks linking intergranular fractures (Plate 3.20) while another SEM micrograph of the specimen tested to failure clearly showed the depression of the edges of the cracks below the specimen surface (Plate 3.21). A microsection of the specimen tested for 130 cycles and examined optically at high magnification (approx. X3000) showed the
displacement of the crack edges and formation of an intergranular crack (Plate 3.22). A microsection of the specimen tested to failure showed the distortion associated with the cracking and the interior penetration of the cracks (Plate 3.23). No grain boundary voids either at the crack tip or elsewhere were observed.

800°C

The appearance of the surface cracking after testing for 30 cycles was similar to that at a test temperature of 700°C, except that the intergranular sections of the fractures were shorter and the transgranular embryo portions consequently more in evidence (indicated by an arrow on Plate 3.24). The depths of the cracks appeared to be less than in the 700°C tested specimen. The extent of oxidation pitting was greater, as expected.

After testing for 60 cycles, the transgranular embryos were developing into fractures (Plate 3.25) and a pattern of straight cracks at right angles to the strain direction was apparent (Plate 3.26).

After 120 cycles, considerable surface cracking had occurred (Plate 3.27) but the cracks were shorter than in the corresponding specimen tested at 700°C, shown in Plate 3.13. Displacement of the crack edges was evident at high magnification (Plate 3.28).

Further cycling up to failure (2000 cycles) resulted in cross-linking of cracks to form the principal fracture as at 700°C. SEM examination at a tilt angle of 70° showed the displacement of crack edges more clearly than optical micrographs (Plate 3.29). This displacement gave light contrast along the raised edge, owing to the greater electron emission there (Plate 3.30). The larger grain size steel (30 μm) facilitated the observation of the crack edge displacement which was as much as 5 μm (Plates 3.31 and 3.32); the final cross-linking also showed up well (Plate 3.33).

850°C

The appearance of the surface of the specimen tested for 100 cycles was similar to that of the specimen tested at 800°C for 120 cycles, the fracture being mixed inter- and transgranular (Plate 3.34).
Summary

In the range 400 to 600°C there was a normal progression from transgranular to intergranular fracture with increasing temperature. At temperatures between 700 and 850°C, however, an unusual type of surface fracture occurred which appeared to consist of both transgranular and intergranular components and was therefore called mixed mode fracture. The appearance of the mixed mode cracks differed from the normal types in that they were straight. Initiation appeared to be intergranular, followed by transgranular growth and linking. Displacement of the crack edges appeared to take place in a direction approximately normal to the specimen surface. When cycled near to failure, cross linking of the fractures took place to form the principal fracture. There was no evidence of grain boundary sliding in the plane of the surface, and no evidence of grain boundary void formation.

3.5.1(b) Influence of Plastic Strain Amplitude

Examinations were carried out on specimens prepolished and tested at a fixed strain rate of $1 \times 10^{-4}$ sec$^{-1}$ using plastic strain amplitudes of approximately 0.8% and 3% respectively, over the temperature range 500 to 800°C.

Plastic Strain Amplitude of 7%

500°C

The fracture mode was intergranular, and the slip band structure was more prominent than at the plastic strain amplitude of 1.5% described earlier.

600°C

The fracture mode was intergranular. Slip band structure was present adjacent to the cracks, in contrast to the observations at the 1.5% plastic strain amplitude level (Plate 3.35).

700°C

The fracture mode was intergranular, which again differed from the mixed-mode cracking observed at plastic strain amplitude = 1.5%, (Plate 3.36). Failures occurred by the continuous growth of an intergranular crack from the edge of the specimen with no cross-linking of smaller cracks. Little or no crack edge displacement was detected, and no slip bands were visible.
At this temperature the new type of mixed-mode fracture described in section 3.5.1(a) was observed. The principal fracture formed by cross-linking of the smaller cracks.

Plastic Strain Amplitude of 0.8%

Testing was carried out for 1250 cycles only, owing to the very long times required for failure (i.e. 50% load reduction) to be reached.

500C

The slip band structure and fracture characteristics were similar to those found in the tests at higher plastic strain amplitudes.

600C

At this temperature the smaller cracks were of the new mixed-mode type while the larger cracks were of a chiefly intergranular character (Plate 3.37). There was some slip band structure.

700C

Fracture was of the mixed-mode type, the cracks being somewhat shorter in length (in the surface plane, that is) that at the 1.5% plastic strain amplitude (Plate 3.38).

800C

Mixed-mode fracture was observed as at the higher plastic strain amplitudes.

Summary

The incidence of the mixed-mode type of fracture described in section 3.5.1(a) was markedly affected by the plastic strain amplitude. An increase in plastic strain amplitude to 3% resulted in the disappearance of this fracture mode at a test temperature of 700C, although it persisted at 800C. Conversely, a decrease in plastic strain amplitude to 0.8% resulted in the appearance of the mixed-mode fracture at 600C, though at this temperature it did not occur in the largest cracks observed.
3.5.1(c) Influence of Strain Rate

Surface cracking in prepolished specimens was examined, the test conditions being a plastic strain amplitude of 1.5% and test temperatures 600, 700 and 800°C. The strain rate chosen was $6 \times 10^{-4}$ sec$^{-1}$.

600°C

The fracture mode was intergranular. More slip band formation appeared to occur than at the lower strain rate of $1 \times 10^{-4}$ sec$^{-1}$ employed in the tests described in foregoing sections.

700°C

The fracture mode was intergranular. Some slip bands were present (Plate 3-39).

800°C

The fracture appearance was indistinguishable from that in tests at $1 \times 10^{-4}$ sec$^{-1}$. Two additional specimens tested at 800°C at strain rates of $2 \times 10^{-5}$ and $1.2 \times 10^{-3}$ sec$^{-1}$ were also examined. The specimen tested at $2 \times 10^{-5}$ sec$^{-1}$ had the same fracture characteristics as that tested at $1 \times 10^{-4}$ sec$^{-1}$. The specimen tested at the high strain rate of $1.2 \times 10^{-3}$ sec$^{-1}$ however, showed a significant difference, the mixed-mode fracture now being associated with increased intergranular fracture (Plate 3-40); there was also no evidence of cross-linking of the smaller cracks to form the principal fracture as was the case in the fully mixed-mode fracture at lower strain rates.

Summary

Increasing the strain rate had a similar effect on the fracture characteristics of the surface cracking as a decrease in the test temperature, resulting in a trend to intergranular cracking, in place of the mixed-mode type of fracture.

3.5.2 Low Cycle Fatigue Fracture of Heat Treated and Aged Steel

The programme of metallographic work is summarised in Table 3.4. More attention was given to fractography in this programme for later comparison with the irradiated steel (Chapter 4).
3.5.2(a) Influence of Test Temperature

The surface fracture characteristics of steel aged for 75d at 750°C before testing were examined, together with microsections and fracture surfaces. The test conditions were a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ and a plastic strain amplitude of 1.5%.

500°C

No differences between the surface fracture characteristics of the aged steel and that of the as heat treated steel, described in section 3.5.1, could be detected.

Fractographic examination using the SEM revealed the presence of branched intergranular fracture near the test area surface (Plate 3.41) although many areas of transgranular fracture were present in the interior. These transgranular areas frequently had the characteristic "ripple" pattern of fatigue striations (Plate 3.42) (see Literature Review section 1.3.2, page 72). The striation spacing increased with depth, reflecting the increased crack propagation rate with crack depth. Near the specimen centre, the reduced plastic strain amplitude caused a decrease in the crack propagation rate, so that the striations became closely spaced (Plate 3.43).

Striation spacings were measured and plotted against crack depth up to 100 µm (Fig. 3.36). A good approximation to a linear relationship was found in accordance with the crack propagation law $dl/dn = A_1$ (see also page 137 and the Literature Review, section 1.3.3(e)). The slight deviation from linearity with increased crack size was probably caused by the decrease in plastic strain amplitude as the crack depth increased. In microsections the cracking had a fine, branched appearance (Plate 3.43).

600°C

No differences between the surface fracture characteristics of the aged steel and that of the as heat treated steel, described in section 3.5.1, could be detected.

Fractographic examination indicated that the fracture mode was exclusively intergranular (Plate 3.45). The fracture surface was covered with large numbers of small pits, presumably due to oxidation.

The appearance of the cracking in microsections was similar to that at 500°C (Plate 3.46).
### TABLE 3.4

**METALLOGRAPHY PROGRAMME FOR THE HEAT TREATED AND AGED STEEL**

**KEY TO ABBREVIATIONS:**  
F: CYCLED TO FAILURE  
S: SURFACE EXAMINATION OF PREPOLISHED SURFACES  
M: MICROSECTIONS  
E: SCANNING ELECTRON MICROSCOPE

<table>
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<tr>
<th>GRAIN SIZE (um)</th>
<th>AGEING TREATMT.</th>
<th>LCF CYCLES</th>
<th>TEST TEMP (°C)</th>
<th>PLASTIC STRAIN AMP. (%)</th>
<th>STRAIN RATE (sec⁻¹)</th>
<th>TYPE OF EXAMINATION</th>
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### TABLE 3.5

**METALLOGRAPHY PROGRAMME FOR TENSILE TESTS ON AS HEAT TREATED STEEL**

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<td>S,M</td>
<td>3.61</td>
</tr>
<tr>
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<td></td>
<td>S,M,E</td>
<td>3.62 &amp; 3.64</td>
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<td>1 X 10⁻²</td>
<td>S,M</td>
<td>NOT PRESENTED</td>
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</table>
The cracking on the prepolished surface of the specimen was of the mixed-mode type previously found in the as heat treated steel under the same test conditions (see page 145).

Fractographic examination showed that much pitting was present on the fracture surface (Plate 3.47), again, as at 600C, presumably due to oxidation, but the nodular appearance of the 600C fracture, characteristic of intergranular fracture, was not observed at this temperature. The branched appearance of the crack surface may have been caused by cross-linking of smaller cracks.

A cellular structure was often observed around the centre of the specimen (Plate 3.48).

The cracks had a blunter and wider appearance than in specimens tested at lower temperatures when microsections were examined (Plate 3.49).

As in the tests at lower temperatures, the characteristics of the surface cracking were the same as for the as heat treated steel.

Fractographic examination demonstrated that like the specimen tested at 700C the fracture surface was not of the typical intergranular type found at 600C (Plate 3.50). The crack surface had a branched appearance as in the test at 700C, and was probably again due to cross linking of smaller cracks, (Plate 3.51) many small pits also being present.

The appearance of cracks in microsections differed from that in specimens tested at lower temperatures: the cracks were broader, more numerous, and the crack tips were apparently blunted, in contrast to the few, sharp, branched cracks characteristic of LCF fracture at 600C or lower (Plate 3.52). Examination at higher magnification showed that although the majority of the cracks were located in grain boundaries, there were many instances where the crack appeared to be propagating transgranularly (Plate 3.53); in microsections the cracks had a short, blunted appearance (Plate 3.54).
3.5.2(b) Influence of Strain Rate at 800C

In view of the strong effect of strain rate on the plastic strain amplitude-endurance curve at 800C in the aged steel (see section 3.4.1(b) and Fig. 3.28) an examination of the fracture characteristics was carried out on the aged steel tested at 800C at a strain rate of $2 \times 10^{-5}$ sec$^{-1}$.

The steel aged for 100d at 700C and fatigued for 60 cycles at a plastic strain amplitude of 1.5% showed an almost entirely intergranular type of cracking (Plate 3.55). Some small intragranular protrusions were visible but they did not propagate across the grain completely and link up with other intergranular embryo cracks to form the mixed-mode type of fracture found in the as-heat treated steel tested under the same conditions (see page 147 and 149) (Plate 3.56). A few of these transgranular protrusions were seen in microsections of specimens aged 125d at 700C (indicated by an arrow on Plate 3.57), tested at 800C and $2 \times 10^{-5}$ sec$^{-1}$. The cracks were sharp and branched, as at lower temperatures of 600C or so where intergranular fracture occurred in the as-heat treated steel (Compare with Plate 3.46 for example).

An examination of the surface fracture carried out on a specimen heat treated to produce a grain size of 30 μm and thereafter aged for 125d at 700C showed well developed transgranular protrusions (indicated by an arrow on Plate 3.58) which again had not linked up with intergranular embryo cracks to form the mixed-mode fracture.

Summary

Prior ageing had no effect on the surface fracture characteristics of the steel compared with that of the as-heat treated steel, except under a certain combination of test conditions.

Fractographic examination indicated that a conventional LCF fracture surface, with striations, found at 500C, gave way to intergranular fracture at 600C. This in turn was replaced by a relatively structureless fracture surface at test temperatures of 700 and 800C.

Examination of microsections showed that at test temperatures of 700 and 800C the LCF cracks took on a blunter appearance and became more numerous than in specimens tested at lower temperatures but otherwise similar test conditions. Under the particular test conditions of a strain rate of $2 \times 10^{-5}$ sec$^{-1}$ and at a test temperature of 800C the fracture mode in the aged steel became intergranular.
3.5.3 Tensile Fracture of As-Heat Treated Steel

The programme of metallographic work is summarised in Table 3.5. Microsections taken from areas adjacent to the fractures of tensile specimens showed that increasing temperature over the range 500 to 800°C at a fixed strain rate of $1 \times 10^{-4}$ sec$^{-1}$ caused a decrease in the tendency to necking before fracture and an increase in the amount of surface cracking (Plates 3.59 to 3.62). Recrystallisation in the tested specimens was evident at 800°C but not at 700°C. Grain boundary cavitation was present at both 700 and 800°C.

As could be expected, the effect of increasing the strain rate was similar to decreasing the test temperature. At a strain rate of $8 \times 10^{-4}$ sec$^{-1}$ and test temperatures of 700 and 800°C the necking and fracture characteristics were similar. At the same strain rate and a test temperature of 600°C there was some necking at the fracture. At a higher strain rate of $1 \times 10^{-2}$ sec$^{-1}$ pronounced necking occurred in tests at 600 and 700°C and was significant at 800°C. Surface cracking was virtually absent at 700°C and greatly reduced at 800°C, and at 500 and 600°C intergranular fracture was not observed, unlike the corresponding LCF tests. Fractographic examination of the specimen tested at 600°C and a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ showed the typical dimpled fracture surface characteristic of ductile fracture (Plate 3.63). At 800°C a somewhat structureless fracture surface was found covered in small pits, similar to that of the corresponding LCF specimen (Plate 3.64) (compare with Plates 3.50 and 3.51).

3.5.4 Orthogonal Grain Structure

Grain boundary migration was observed after testing under certain conditions at temperatures above 750°C (see Literature Review, section 1.3.1(c) and 1.3.4(a)). The migration resulted in two changes in the grain structure, namely:

(1) an increase in the mean grain size
(2) a tendency for the grain boundaries to be aligned at 45° to the strain axis, and hence, at 90° to each other (Plate 3.65).
3.5.4(a) Influence of Test Temperature

Under fixed conditions of plastic strain amplitude, strain rate, and number of LCF cycles, the extent of the grain boundary migration increased with increasing temperature, and was most prominent in tests conducted at 850°C.

An attempt was made to determine an approximate value of the appropriate activation energy by an Arrhenius plot. Boundary migration rate was defined as the ratio of the increase in grain size to number of fatigue cycles and the logarithm of this ratio was plotted against reciprocal absolute temperature. The estimated activation energy, based on LCF test results at 750, 800 and 850°C was approximately 50 kcal/mol, in reasonable agreement with the published value of 44 kcal/mol for grain boundary self diffusion in 20/25/Nb steel (261).

3.5.4(b) Influence of Plastic Strain Amplitude

The extent of grain boundary migration decreased towards the specimen interior, becoming negligible in a zone around the neutral axis (Plate 3.66) provided that the principal fracture or deep cracks were not present.

The migration was generally at its maximum adjacent to the principal fracture (Plate 3.67). Since the plastic strain amplitude increased linearly from zero at the neutral axis to the applied value at the surface, the final grain size could be used as a measure of the rate of boundary migration and plotted as a function of plastic strain amplitude; this is presented in Fig. 3.37, for a specimen tested for $2 \times 10^4$ cycles at 800°C, plastic strain amplitude of 0.5% and strain rate of $3 \times 10^{-4}$ sec$^{-1}$. Although there is some scatter, a linear relation is apparent up to a plastic strain amplitude of 0.35%, with the grain size doubled at this value.

The decrease in plastic strain amplitude towards the neutral axis is also necessarily accompanied by a decrease in the strain rate (since, of course, the cycle time always remains the same) but decreasing strain amplitude was evidently the most important factor, since the reduction in strain rate would not be accompanied by any increase in the total cycling time.

3.5.4(c) Influence of Strain Rate

Tests conducted on the as-heat treated steel at 800°C and strain amplitudes of 1.64 and 2.00% at strain rates of $3 \times 10^{-4}$ sec$^{-1}$
and $2 \times 10^{-5}$ sec$^{-1}$ for 1530 and 1100 cycles respectively indicated that far more migration occurred at low strain rates (Plates 3.67 and 3.68).

3.5.4(a) Influence of Number of LCF Cycles

As heat treated specimens were fatigued at 800°C at a plastic strain amplitude of 1.5% for different numbers of cycles up to failure (3700 cycles). The grain sizes, within a depth of 125 μm from the surface, are plotted against percentage cycles to failure in Fig. 3.38. There was no measureable change in grain size up to 25% of the fatigue life but a rapid increase occurred up to 50% of the life, after which there was only a small increase in grain size.

The foregoing experiment was repeated at a test temperature of 850°C and a similar pattern of behaviour was observed; no detectable increase in grain size occurred until about one third of the fatigue life had elapsed.

The duration of cycling appears to be the most important factor, since tests conducted on the aged steel (125d at 700°C) at a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ and 800°C showed an increasing tendency for grain boundary migration with decreasing plastic strain amplitude, apparently contradicting the previous data on the effect of plastic strain amplitude (Plates 3.69 to 3.71). However, the effect of increased plastic strain amplitude is to reduce endurance and hence, the duration of the test, thereby tending to offset the effect of increased plastic strain amplitude on the grain size changes.

(220) Pole figures obtained from specimens with well developed orthogonal grain structure did not exhibit any detectable differences in texture compared with control specimens.

Summary

Grain boundary migration has been observed in LCF tests at temperatures above 750°C. Two distinct effects could be identified, an increase in grain size and a re-orientation of the boundaries to directions at 45° to the applied strain direction.

Migration became more pronounced the higher the test temperature, the greater the plastic strain amplitude, and the longer the duration of cycling.
3.6. Discussion

3.6.1 Mechanical Testing

The principal observations made from the test results were:

(1) The Coffin-Manson Law (see Literature Review 1.3.3(a) page 74) was obeyed over the range of test variables employed.

(2) At a strain rate of $1 \times 10^{-4}$ sec$^{-1}$, the LCF endurance decreased with increasing temperature until about 600°C, thereafter the endurance increased up to a test temperature of 800°C, and again decreased at 850°C (see section 3.4.1(a) and Fig. 3.15).

(3) The tensile ductility increased over the temperature range 600 to 800°C, there being a reduced tendency to necking at the fracture with increasing ductility (see section 3.4.2, page 140, and Fig. 3.31.)

(4) The strain rate sensitivity exponent ($"m"$) increased with decreasing grain size and increasing temperature under both cyclic and tensile loading (see section 3.4.2, page 140 and section 3.4.1(a), page 134, and Figs. 3.21, 3.23, 3.34 and 3.35).

The fact that both the LCF endurance and tensile ductility tended to increase at temperatures above about 600°C suggested that the two phenomena were in some way related. Furthermore, since the temperature at which the endurance and ductility increases were observed corresponded approximately to that at which significant diffusion could occur in the course of a test (e.g. the absence of cyclic hardening, Literature Review, section 1.3.1(a)) these effects were probably diffusion controlled ones. It has been shown that 20/25/Nb steel having a grain size of 3 µm exhibits superplastic behaviour at test temperatures of 700 and 800°C (262) so it was considered that the endurance and ductility enhancement observed may have been related to the relatively small grain size of 12 µm of the steel used for the mechanical testing described in the previous section.

The 12 µm grain size steel showed strain rate sensitivity exponents below the generally quoted lower limit of 0.2 for superplastic behaviour (see Figs. 3.23 and 3.35) but it was nonetheless capable of limited superplastic deformation, sometimes referred to as "extended plasticity" (263). This is supported by the observation
that necking at the principal fracture decreased with increasing test temperature over the range 500 to 800°C (see section 3.5.3, page 155, and Plates 3.59 to 3.62).

The very large ductility values associated with superplastic deformation are due to the inhibition of plastic instability; in normal materials local strain rate variations result in the formation of a neck in which deformation is concentrated until fracture occurs. The onset of plastic instability in isotropic metals corresponds to the peak of the load-deformation curve, i.e. at the point where the stress reaches the ultimate value. If the material has a high strain rate sensitivity value the tendency to plastic instability, and therefore to necking, will be reduced because strain rate hardening will occur in regions of locally enhanced deformation. A theoretical relationship between rate of necking and strain rate sensitivity exponent has been established.

From the foregoing reasoning it may be deduced that the increased ductilities observed in tensile tests at 700 to 800°C were a result of increases in strain rate sensitivity exponent and consequent decrease in the tendency to plastic instability. A different explanation is required for the observed increases in LCF endurance, however, since the plastic strains involved were much less than those required for plastic instability, irrespective of the value of the strain rate sensitivity exponent. The observed decrease in crack propagation rate suggested that the extended plasticity phenomenon affected LCF endurance by reducing the crack growth rate (see section 3.4.1(b), page 139, and Fig. 3.27) thus a discussion of fracture behaviour follows.

3.6.2 Fracture Behaviour

The principal observations on the fracture characteristics were:

(1) The LCF fracture at test temperatures of 400°C was transgranular and associated with slip band fissures, the presence of slip bands close to the crack probably indicating the presence of a zone of intense deformation near the crack tip.

(2) In the range 500 to 600°C fracture was intergranular with slip bands again concentrated near cracks.
Above 600°C an unusual type of mixed trans-intergranular fracture occurred, characterised by long, straight cracks orientated at 90° to the strain direction. These observations apply to test conditions of plastic strain amplitude = 1.5% and strain rate = $1 \times 10^{-4}$ sec$^{-1}$ (see section 3.5.1, page 145).

Increases in plastic strain amplitude and strain rate had a similar effect on the occurrence of the mixed-mode fracture as a reduction in temperature; i.e. the fracture mode tended to become intergranular; reducing plastic strain amplitude and strain rate had the converse effect (see section 3.5.1(b), page 148).

Grain boundary cavitation was not observed in LCF specimens, but was observed in the tensile specimens at test temperatures above 600°C, at a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ (see section 3.5.3, page 155).

LCF crack initiation in the mixed-mode appeared to occur by grain boundary shear at an angle to the specimen surface; no grain boundary shear in the plane of the surface was observed (see section 3.5.1(a), page 143). The ends of the intergranular crack nucleus propagated transgranularly into the adjoining grains. In this way the crack remained straight during growth.

Electron fractographs of LCF specimens fractured in the mixed-mode showed that the fracture surface had a rather featureless appearance compared with those of specimens tested under conditions where intergranular fracture predominated (see section 3.5.2, page 150).

The formation of the intergranular nucleus in the mixed-mode appeared to occur by grain boundary shear. The depressions visible in micrographs at the ends of the intergranular nuclei (arrow on Plate 3.24) were evidently developing transgranular extensions of these nuclei and were possibly non-crystallographic since the mixed-mode cracks were straight. If this was the case, a combination of dislocation glide and climb would have been necessary for their formation.

The following three-stage model may be proposed for elevated temperature LCF crack formation in the new mixed fracture mode:
(1) Formation of an intergranular nucleus by cyclic grain boundary shear at an angle to the plane of the surface and at 90° to the applied strain direction (Fig. 3.39).

(2) Transgranular growth of the ends of the intergranular nucleus into the adjoining grains, the transgranular growth being apparently unaffected by orientation of the slip planes in the adjoining grains.

(3) Crack growth into the interior, possibly by the same mechanism as above, but in addition growth by plastic decohesion may also play a part when larger crack sizes are reached (Fig. 3.40).

The formation of the mixed-mode fracture evidently depended on the inhibition of intergranular fracture at angles other than 90° to the applied strain direction in the plane of the specimen surface, since no general grain boundary sliding was detected (see section 3.5.1(a), page 148). This inhibition depended on temperature, plastic strain amplitude and strain rate (section 3.5.1, pages 143 to 148). At 700°C the fracture mode varied markedly with plastic strain amplitude, an increase from 1.5% to 3% at a strain rate of 1 x 10^-4 sec^-1 (page 148) being sufficient to alter the fracture mode from mixed to fully intergranular. At 800°C mixed-mode fracture was observed at all plastic strain amplitudes, though it might have been possible to obtain fully intergranular fracture at this temperature and strain rates, if a greater plastic strain amplitude than 4% had been possible with the equipment.

The association of fully intergranular fracture with lowest LCF endurance suggests that at 700°C there should have been a faster decline in endurance with increasing plastic strain amplitude than at 800°C; that is, the Coffin-Manson exponent "a" (see Literature Review, section 1.3.3) should have decreased with increasing temperature, and this was, in fact, observed (Fig. 3.16). This behaviour contrasts with that found in the great majority of metallic materials, where intergranular cracking is the fracture mode observed at elevated temperatures.

Since the height of the grain boundary shear steps increased as cycling progressed, ultimately leading to a considerably distorted surface structure (see page 145 and Plates 3.32 and 3.33) it is likely that cracking by grain boundary shear was still occurring
when the crack propagated into the specimen interior. It was
difficult to prove this conclusively, mainly because of the fine
grain size, which prevented any easy distinction between trans- and
intergranular fracture in microsections. However, the aged steel
tested at 800°C and a strain rate of $2 \times 10^{-5} \text{ sec}^{-1}$ clearly showed
partly developed transgranular cracks in the specimen interior
(see section 3.5.2(b), page 154, and Plates 3.53 and 3.57). There
was also a change in the fracture surface between 600 and 800°C in
aged steel tested at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ (section 3.5.2,
page 151). At 600°C (Plate 3.45) the nodules of individual grains
were visible in this fully intergranular fracture, while at 800°C
(Plate 3.51) the separate grains were not easily visible, the
whole fracture surface was rather featureless compared with that of
the specimen tested at 600°C.

The fracture surface of the specimen tested at 700°C had a
color character intermediate between those tested at 600°C and 800°C
(Plates 3.47 and 3.48).

The foregoing observations, taken together with the reduction
in the estimated crack propagation rates above 700°C (see section
3.4.1(b), page 139) lend some support to the view that the mixed-
mode type of fracture persisted when the LCF propagated into the
specimen interior.

The length of the mixed-mode surface cracks (i.e. the length
in the surface plane as distinct from crack depth) appeared to be
governed by the number of cracks nucleating per unit area, a large
number of nuclei favouring short cracks and vice versa. This
effect was probably due to the unloading of the region surrounding
the nucleus caused by the growth of adjoining nuclei, so inhibiting
lengthwise growth, illustrated in Fig. 3.41. Test conditions
favouring a low nucleation rate, e.g. a very low plastic strain
amplitude, resulted in the formation of very few surface cracks which
sometimes crossed the whole width of the specimen. Increasing
temperature evidently favoured nucleation since larger numbers of
small cracks formed at 800°C than at 700°C (see section 3.5.1(a),
page 143). This was presumably due to easier grain boundary shear
at the higher temperature and possibly due to increased numbers of
intergranular oxidation pits which may have linked up to form crack
nuclei.
The formation of the "cross-linking" cracks (section 3.5.1(a), page 146) and Plates 3.18, 3.19 and 3.34) approximately parallel to the applied strain direction in specimens close to failure, was probably caused by unloading of the region adjacent to deep cracks. It is suggested that this caused a change in the strain direction in the uncracked ligaments so that the cross-linking cracks formed at 90° to this local strain direction (see Fig. 3.42).

Although the mechanism of superplasticity is still the subject of research, grain boundary sliding is believed to be the principal deformation mode in single-phase superplastic alloys \(^{(165)}\). If grain boundary sliding is the sole deformation mechanism, a strain rate sensitivity exponent of unity should be observed, and in superplastic alloys a value greater than about 0.5 would be typical in practice. The low values of strain rate sensitivity exponent obtained for the 12 μm grain size steel used in this investigation (see section 3.4.1(a), page 136) and also the absence of widespread grain boundary sliding during LCF (see section 3.5.1(a), page 148) are observations weighing against a grain boundary sliding mechanism of the extended plasticity observed in this steel. The formation of intergranular crack nuclei by grain boundary shear, however, as described on page 146, indicated that sliding nonetheless occurred on certain favourably orientated boundaries. In a theoretical study of the motion of grain boundary dislocations, McLean \(^{(165)}\) has shown that rates of grain boundary dislocation climb and therefore rates of grain boundary sliding vary markedly with crystallographic orientation as well as with applied resolved shear stress. This sliding may in the present instance have occurred only on the few favourably orientated boundaries. The number of such boundaries evidently increased with increasing temperature and plastic strain amplitude and the observation that only those boundaries normal to the applied strain direction could nucleate LCF cracks indicates that the resolved shear stress acting on the boundary must have been a maximum. Grain boundary shear can occur most easily in a direction inclined to the surface plane since the surface grains are unconstrained in that direction. The grain boundary shear was accommodated by deformation of the grains rather than rotation by pure sliding (see section 3.5.1(a), page 148, Plate 3.22 and also Fig. 3.39).
It is therefore clear that the deformation properties of the grain interior played as important a role as those of the boundary. If the grain interior could not deform easily, then grain boundary shear would have been reduced, or inhibited. Under these circumstances, a weak grain boundary would not have been able to accommodate shear stresses by deformation, and fracture would have resulted, leading to the familiar elevated temperature intergranular cracking. In addition, the grain boundary shear would have been more easily accommodated by the adjoining grains if the shear strain per fatigue cycle was low. Thus, at test temperatures and strain rates where sliding just started to occur, the fracture mode could have been expected to depend on the plastic strain amplitude. A low plastic strain amplitude would have resulted in small grain boundary shear strains which could have been easily accommodated by the adjacent grains, resulting in mixed-mode fractures. A large plastic strain amplitude would have resulted in large grain boundary shear displacements during each LCF cycle which could not have been accommodated by local deformation and so intergranular fracture would have occurred. This was precisely the observed behaviour at a test temperature of 700°C, a strain rate of $1 \times 10^{-4}$ sec$^{-1}$, and plastic strain amplitudes of 1.5% and 3% respectively (see section 3.5.1(a), pages 145 to 149). A low plastic strain amplitude of 0.8% likewise resulted in mixed-mode fracture at the lower test temperature of 600°C (section 3.5.1(a), page 149).

Increasing the applied strain rate produced a similar effect on the mechanical properties and fracture characteristics as a reduction in the test temperature. At 700°C and a plastic strain amplitude of 1.5%, the surface fracture changed from mixed-mode to intergranular at an increased strain rate of $6 \times 10^{-4}$ sec$^{-1}$ (see section 3.5.1(c), page 150, & Plate 3.39) and there was also some slip band formation, that is, the fracture characteristics were similar to those observed at the lower test temperatures of 500 and 600°C, at the lower strain rate of $1 \times 10^{-4}$ sec$^{-1}$. There was no change of endurance with increasing strain rate (see Fig. 3.18) as might have been expected from the similar endurance at 700°C and at the lower test temperatures of 500 and 600°C at the strain rate of $1 \times 10^{-4}$ sec$^{-1}$ (see Fig. 3.15).
At the test temperature of 800°C the influence of strain rate on the surface fracture characteristics was much less marked than at 700°C. The highest strain rate possible with the equipment (approximately $1 \times 10^{-3} \text{ sec}^{-1}$) was not sufficient to produce pure intergranular fracture (see section 3.5.1(c), page 150 and Plate 3.40) although the proportion of intergranular fracture in the mixed-mode fractures appeared to increase. Thus, while some reduction in endurance could have been expected at the highest strain rates (due to the trend to intergranular fracture) the results were conflicting since a decrease in endurance with increasing strain rate was observed in one series of tests at a plastic strain amplitude of 1.5% (Fig. 3.19) but not in another carried out at a slightly higher plastic strain amplitude of 1.9% (Fig. 3.20).

The effect of strain rate in "push-pull" LCF (i.e. tension to compression axial cyclic loading) of 20/25/Nb steel at a test temperature of 750°C has been studied by Skelton (229). The steel was heat treated to produce a grain size of approximately 26 μm and aged at 820°C for 20 hr. In tests at a plastic strain amplitude of about 1% the endurance decreased by an order of magnitude on decreasing the strain rate from $1.7 \times 10^{-3}$ to $1.7 \times 10^{-5} \text{ sec}^{-1}$. This result is in qualitative agreement with the present work on the aged steel tested at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ and $2 \times 10^{-5} \text{ sec}^{-1}$ where a similar reduction in endurance was observed (see section 3.5.2(b), page 154, and Fig. 3.28).

Skelton suggested that the reductions in endurance with decreasing strain rate may have been due to the increasing effect of crack tip oxidation and increasing creep deformation, resulting in constant times to failure independent of plastic strain amplitude. Such a trend was certainly apparent in results described in section 3.4.1(b) page 139, the vacuum pressure being $10^{-4}$ torr (Fig. 3.28). It is unlikely that the reduction in endurance with decreasing strain rate was due solely to oxidation since it was only found in the aged steel; the occurrence of crack tip oxidation should not have depended markedly on prior ageing treatment. Creep deformation may be expected to influence fatigue crack growth in two main ways:

1. removal of material from the crack tip by diffusion
2. formation of grain boundary voids.
If creep voids formed they must have been of sub-microscopic size, since none were observed (section 3.5.1(a), page 147).

At the test temperature of 800°C and strain rate of $2 \times 10^{-5}$ sec$^{-1}$, intergranular fracture was observed to occur more readily in the aged than in the as heat treated steel (section 3.4.1(b), page 140) and section 3.5.2(b), page 154, and Plates 3.55 and 3.56) so that the presence of carbonitrides evidently promoted intergranular fracture at this strain rate, although not at the higher value of $1 \times 10^{-4}$ sec$^{-1}$. The reason for this is conjectural. It is possible that the grain boundaries were weakly locked by precipitates in the aged steel and a certain minimum shear stress had to be exceeded before sliding commenced; at low strain rates the matrix may have been too soft to permit sufficiently high stresses to build up at the grain boundaries to initiate sliding. The duration of an LCF test on the as heat treated steel at a plastic strain amplitude of 1.5% and strain rate of $2 \times 10^{-5}$ sec$^{-1}$ was about 1000 hr. at 800°C, so that the steel underwent virtually complete ageing during the test (see Chapter 2, section 2.4.2, page 104). In the later stages of the LCF test, therefore, the originally as heat treated steel should have behaved in a similar way to the aged steel, and, in particular, the estimated crack propagation rate as determined by the load reduction technique (see section 3.4.1, page 137) should have been the same. This was not the case, however, the estimated crack propagation rate being higher in the steel aged before testing (see Fig. 3.27).

Cyclic plastic deformation may have modified the precipitation characteristics by, for example, increasing intragranular nucleation, such that there was less intergranular precipitation than in the aged steel with consequently less pinning of grain boundaries. A higher strain rate sensitivity exponent would have been expected, due to the inhibition of grain boundary sliding, but this was not observed.

Metallographic examination has shown that crack nucleation occurred very early on in the LCF test at all test temperatures, by slip band fissure formation at low temperatures and by intergranular crack formation at elevated temperature. Crack nuclei were present after less than about 10% of the LCF life at 500°C and after about 1% of the life at 800°C. The endurance must have been determined, therefore, primarily by the crack propagation behaviour. Owing to the
simplifying assumptions made, the load reduction technique of
determining the crack propagation rate (see page 137) yielded
results useful for comparison purposes only.

3.6.3 Theoretical Models

A published fatigue theory (see Literature Review, section 1.3.3(f), page 79) has been applied to the data obtained in this
investigation. Endurance values calculated for test temperatures
between 400 and 800°C, using a plastic strain amplitude of 1.5% and
a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ are compared with the exper­
imental data in Fig. 3-43 (the "modified theory" points in Fig. 3.43 are discussed in the next section).

The initial crack size was assumed to be 2.5 μm as in previous
work, it being believed that this figure represented a reasonable
 crack nucleus size. The agreement was reasonably good up to
test temperature = 600°C, but at higher temperatures the predicted
endurance values were much lower than the observed ones. The low
endurance values predicted by the theory reflected the fall in the
UTS and the cyclic strain hardening capacity of the steel with
increasing test temperatures (see Fig. 3.21 and Fig. 3.32).

Since the theoretical Coffin-Manson exponent $\alpha$ is $1/(2 \beta + 1)$
the slope of the plastic strain amplitude-endurance curve should have
increased to a limiting value of unity as the cyclic strain hardening
exponent $\beta$ tended to zero at temperatures of 700°C and higher. The
opposite behaviour was observed however, the exponent $\alpha$ decreasing
with increasing test temperature (see Fig. 3.16) in the range where
strain rate hardening effects began to predominate. Since the
theory essentially proposes that LCF crack propagation rates are
governed by the strain hardening capacity of the material in the
plastic zone ahead of the crack tip, it is possible that at elevated
temperatures strain rate hardening can play a similar role to strain
hardening at lower temperatures. There would therefore be a temperature
transition zone in which strain hardening and strain rate hardening
capacities will both be influencing LCF crack propagation rates.

3.6.3(a) Modified Theory of LCF Crack Propagation

Modifying the theory to take strain rate hardening into account
requires that the strain rate in the crack tip zone is known. The
two assumptions made are:
(1) That the zone of intense deformation ahead of the crack tip is formed each tensile half-cycle and removed by diffusion processes during the compression half-cycle.

(2) The accumulation of plastic strain in the crack tip zone is linearly time dependent during the tensile half-cycle.

The mean strain rate in the zone of intense deformation is then given by:

\[ \bar{\varepsilon} = \frac{\varepsilon_f}{t} \]  

Where \( \varepsilon_f \) is the fracture strain and \( t \) is the duration of the tensile half-cycle.

Since the elastic strain component of the total strain amplitude is small at elevated temperatures, equation (1) may be written:

\[ \bar{\varepsilon} = \frac{\varepsilon_f \dot{\varepsilon}}{\Delta \varepsilon_p} \]  

where \( \dot{\varepsilon} \) is the applied (i.e. macroscopic) strain rate.

A constitutive equation relating stress, strain and strain rate of a simple power law type has been proposed by Rossard:

\[ \sigma = (\text{constant}) \varepsilon^m \varepsilon_p^\beta \] .... (3)

Materials having this relationship will show a linear dependence of log (true stress) on log (true strain) and on log (true strain rate). In terms of cyclic parameters (3) becomes:

\[ \Delta \sigma = K_f \varepsilon^m \Delta \varepsilon_p^\beta \] .... (4)

\( K_f \) is the counterpart of the strength parameter \( K \) in the ambient temperature constitutive equation \( \Delta \sigma = K \Delta \varepsilon_p^\beta \) \( \beta \) being the strain hardening exponent. \( \Delta \sigma \) and \( \Delta \varepsilon_p \) are the stress amplitude and plastic strain amplitudes respectively. Therefore \( K \) and \( K_f \) are related by: \( K_f \dot{\varepsilon}^m = K \)

When the LCF crack advances during the tensile half-cycle, the normal true stress across the intense deformation zone at the crack tip may be assumed to correspond to that at fracture under tensile conditions: that is,

\[ \sigma_f = K_f \varepsilon^m \varepsilon_p^\beta \] .... (5)

where \( \sigma_f = \) Fracture stress under tensile conditions

\( K_f = \) Tensile strength parameter, the constant in equation (3)

\( \varepsilon_p = \) Tensile strain rate sensitivity exponent.

\( \beta = \) Tensile strain hardening exponent.
Substituting for the mean strain rate in the intense deformation zone, equation (1) gives:

$$ \sigma_F = \frac{K_T}{\epsilon_F} \left[ \frac{\epsilon}{\Delta \epsilon} \right]^{m_T} \left( \frac{K_1}{2K_T \epsilon_F (m + \beta)} \right)^2 \frac{2(m + \beta) + 1}{\Delta \epsilon} $$

The mean tensile stress distribution in the intense deformation zone at fracture $T$, will be given approximately by $\sigma_F$. Experimentally, it has been shown that the tensile and cyclic values of $m$ and $\beta$ are in close agreement (section 3.4.1(a), page 136 and section 3.4.2, page 142, and Figs. 3.21 and 3.34), so equations (4) and (6) may be substituted into the original endurance equation giving:

$$ N = \ln \left( \frac{1}{1 - \frac{1}{\epsilon_0}} \right) \left[ \frac{\pi^2}{8} \left( \frac{K_1}{2K_T \epsilon_F (m + \beta)} \right)^2 \frac{2(m + \beta) + 1}{\Delta \epsilon} \right] $$

In this equation the combined parameter $(m + \beta)$ takes the place of $\beta$ alone in the original equation. The validity of equation (7) may be checked by putting $m = 0$, in which case it reduces to the unmodified form.

The strain rate dependence of the endurance, $N$, is contained in the terms $K_1$, $K_T$, $m$ and $\beta$. $K_1$ and $K_T$ will be affected to approximately the same degree by changes in strain rate, so the ratio $K_1/K_T$ in equation (7) will be practically strain rate independent. The combined hardening exponent $(m + \beta)$ varied only slowly with strain rate for the steel tested at 800C (Fig. 3.22). Thus the proposed model predicts that strain rate should have had little effect on endurance at 800C, as observed in the as heat treated steel, though not in the aged steel (see section 3.5.2(b), page 154).

The theoretical Coffin-Manson exponent $"a"$ derived from equation (7) is $1/2(m + \beta) + 1$. Theoretical values, and experimental values derived from LCF tests at 800C and a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ are compared in Table 3.6.

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Original Theory $\frac{1}{2(\beta + 1)}$</th>
<th>Modified Theory $\frac{1}{2(m + \beta) + 1}$</th>
<th>Observed $a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>0.63</td>
<td>0.63</td>
<td>0.60</td>
</tr>
<tr>
<td>600</td>
<td>0.67</td>
<td>0.72</td>
<td>0.52</td>
</tr>
<tr>
<td>700</td>
<td>0.83</td>
<td>0.65</td>
<td>0.50</td>
</tr>
<tr>
<td>800</td>
<td>0.86</td>
<td>0.75</td>
<td>0.34</td>
</tr>
</tbody>
</table>

TABLE 3.6

Comparison of Theoretical and Observed Coffin-Manson Exponents
At 500°C, \( m = 0 \), so that strain rate hardening played no part in LCF crack propagation and good agreement is found between the theoretical and observed values of \( \alpha \) at this temperature. The agreement is not so good at the higher temperatures, although the modified theory gives better agreement with the observations than the original theory.

Theoretical endurances for temperatures from 400 to 800°C are compared with the observed endurances in Fig. 3.43 for a plastic strain amplitude of 1.5% and a strain rate of \( 1 \times 10^{-4} \) sec\(^{-1} \). The endurances shown in the figure were calculated using the form of equation (7) appropriate to cases where the applied stress is close to the UTS of the material:

\[
N = \ln \left( \frac{1}{1 - \frac{\Delta \sigma}{\sigma_{UTS}}} \right) \left[ B(1 + B\Delta \varepsilon_p)^{2(m+\beta)}\Delta \varepsilon_p^{2(m+\beta)} + 1 \right] ...
\]

where

\[
B = \frac{2}{\pi} \left( \frac{K_{I}}{2K_{II}} \right)^{2}
\]

In fact the endurances calculated using equation (8) did not differ greatly from those obtained using the simpler equation (7). The predicted endurances were in good agreement in the temperature range 400 to 600°C, as a consequence of the similar values of \( K, \bar{T} \) and \( \beta \) at these temperatures, \( m \) being practically zero.

The calculated endurances were of the right order over the temperature range studied, but the modified theory did not reproduce the characteristic minimum in the temperature-endurance curve in the range 500 to 700°C (see Fig. 3.15) and predicted a sharp increase in endurance at 700°C, whereas this increase was not observed experimentally until a temperature of 750°C was reached. However, the low to elevated temperature transitions in mechanical properties and fracture characteristics which occurred at 700°C led to LCF properties at this temperature which were partly similar to those at lower temperatures, e.g. low endurance, as at 500 and 600°C (although some increase is apparent in the aged steel, Fig. 3.15) and partly similar to elevated temperature properties, e.g. an increased strain rate sensitivity exponent, mixed-mode cracking, and a reduction in crack propagation rate (see Fig. 3.27).
At temperatures below 600°C, where the strain rate sensitivity exponent was zero, and the unmodified form of equation (7) was applicable, the theory did not predict the reduction in endurance observed between 400 and 500°C as would be expected from the similar values of $K_1$, $K_T$, $\beta$, and $\beta$ at these temperatures. In this instance the endurance reduction and change in fracture characteristics from trans- to intergranular were not paralleled in the change in mechanical properties. This was probably due to the grain boundaries being weak in LCF at 500°C but not in the tensile test at this temperature ($T$ is derived from the tensile data and intergranular fracture was not observed in the 500°C tensile tests) (see section 3.5.3, page 155). The theoretical Coffin-Manson exponent, "$a$", which is not dependent on $T$, was in good agreement with experiment at 500°C so that the theory correctly predicted the change in endurance with plastic strain amplitude at this temperature.

A partial success may therefore be claimed for the modified theory presented above. This is probably as good as could be expected in view of the complexity of the LCF fracture processes in this steel at elevated temperatures, and the essential simplicity of the model.

3.6.4 Orthogonal Grain Structure

The two consequences of grain boundary migration in push-pull LCF tests on 20/25/Nb steel described by Skelton\(^{(229)}\), (see Literature Review 1.3.4(a), page 84), namely an increase in mean grain size and the formation of a "square" or "orthogonal" grain structure, have both been observed in the present work (see section 3.5.4, page 155). The former effect has been more apparent than the latter in microstructural examinations and none of the test conditions employed have resulted in square grain formation without grain enlargement, as found by Skelton under test conditions of temperature = 750°C, strain rate = $1 \times 10^{-4}$ sec$^{-1}$. This was possibly because the steel used in this study had a smaller grain size (12 μm) than that employed by Skelton (26 μm) and so the driving force for grain enlargement would have been greater in the former case. In Skelton's work, grain enlargement was only observed at the lowest strain rates, suggesting that only under low strain rate conditions was there enough time for a significant amount of grain enlargement to occur.
A further difference in grain boundary migration behaviour between the bend and the push-pull LCF studies is that in the former case the tendency for both grain enlargement and square grain formation to occur increased with decreasing strain rate, at constant plastic strain amplitude and number of fatigue cycles. In the push-pull experiments, square grain formation occurred at a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ but was not observed at $1 \times 10^{-3}$ and $1 \times 10^{-5} \text{ sec}^{-1}$. The absence of square grain formation at the higher strain rate of $1 \times 10^{-3} \text{ sec}^{-1}$ may also be accounted for by the rate of grain boundary migration being too low at 750°C to produce noticeable changes in grain boundary structure during the test. In the corresponding bend LCF experiment grain boundary migration was likewise not observed in specimens tested at a strain rate of about $1 \times 10^{-3} \text{ sec}^{-1}$ (see section 3.5.4(c), page 156).

It is difficult to account for the occurrence of square grain formation in the bend LCF work at a strain rate of $2 \times 10^{-5} \text{ sec}^{-1}$ and its absence in the push-pull experiments conducted at the same strain rate. The lack of square grain formation in the latter case indicated that a minimum strain rate may have been necessary for the effect to occur, while the absence of a similar strain rate effect in the bend LCF tests may have been a consequence of the smaller grain size, different precipitate distribution (see Chapter 2, section 2.4.2, page 104 and page 108) and, possibly, the higher test temperature (800°C).

The 20/25/Nb steel also exhibited different behaviour with respect to plastic strain amplitude in push-pull and bend LCF. In the former case, no changes in grain boundary structure were found below a plastic strain amplitude of 8%, while some increase in mean grain size was found at a plastic strain amplitude as low as 0.1% in the present study (see section 3.5.4(b), page 156 and Fig. 3.3). This effect may again be ascribed to the finer grain size and higher test temperature.

The effect of the higher test temperature would have been to increase the rate of grain boundary migration by enhancing diffusion, while the finer grain size would have provided a greater driving force due to the increased grain boundary tension.

The induction period observed before grain boundary migration produced a significant increase in grain size (section 3.5.4(d), page 157) (Fig. 3.38) corresponds to the behaviour normally encountered
in recrystallisation of cold worked metals. However, in other LCF studies on pure metals\(^{(179)(182)}\), a similar induction period has not been reported, an initially high rate of grain boundary migration gradually reducing with increasing number of cycles until a stable square grain configuration was formed. The presence of an induction period for migration in 20/25/Nb steel may have been due to the anchoring effect of carbonitrides on the grain boundaries; the cause of migration must have been different from that in recrystallisation of metals and alloys as this is associated with the nucleation of new grains. Possibly, a certain amount of strain energy must be accumulated before the boundary can break away from the carbonitride particles. Such anchoring effects have been observed by other workers during the LCF of impure metals and in alloys\(^{(179)(194)}\).

The extensive grain boundary migration observed at 850°C was associated with an increase in crack propagation rate and, hence, a decrease in LCF endurance (see section 3.4.1(a), page 134, and Fig. 3.15) though the decrease cannot be ascribed entirely to grain boundary migration since the considerable oxidation which occurred at this temperature could have influenced crack propagation rates. The increase in grain size in the 850°C tests would be expected to have resulted in increased LCF crack propagation rates because of the associated reduction in strain rate sensitivity exponent (Fig.3.23). However, the observed increases in grain size caused by grain boundary migration were insufficient to cause a significant change in the exponent. Specimens of both 12 and 30 μm grain size had similar endurances at a plastic strain amplitude of 1.4%, and it would appear therefore that an increase in grain size alone due to migration during the LCF test is unlikely to be responsible for the observed reduction in endurance above 800°C. Possibly cavitation at the crack tip was assisting LCF crack propagation at this temperature.

3.6.4(a) Dislocation Model

Grain boundary sliding, migration, and the formation of an orthogonal grain structure are commonly observed simultaneously during LCF (see Literature Review section 1.3.1(c), page 71) and has led some workers to postulate that square grains form as a consequence of preferred sliding in the directions.
of maximum shear stress \((182)\). This clearly does not explain why square grains are never observed following creep or tensile deformation even when grain boundary migration has resulted in the formation of a completely new grain structure. Furthermore, the steel employed in this investigation did not show general grain boundary sliding in LCF under the test conditions used (see section 3.5.1(a), page 148). In the remainder of this section a dislocation model due to W. Louat \((267)\) is described for the formation of orthogonal grain structure which does not depend on grain boundary sliding.

The orientation of the grain boundaries at \(45^\circ\) to the stress axis and the existence of quadruple points is considered to occur as a consequence of grain boundary energy anisotropy caused by a flux of dislocations entering the boundary during plastic deformation. Since the condition of constant volume during plastic deformation requires an effective value of Poisson's ratio of \(\frac{1}{3}\), slip occurs as though the Burgers vectors are directed along the maximum shear stress directions, at \(45^\circ\) to the tension axis. During elevated temperature deformation, a flux of dislocations will therefore move at \(45^\circ\) to the tension axis by combined glide and climb. A grain boundary receives a flux on each side of \(A\cos \Theta \) and \(A\sin \Theta \) where \(A\) is the flux density (Fig. 3.44). The total flux is therefore \(A(\cos \Theta + \sin \Theta)\). The term in brackets is a minimum when \(\Theta = 0^\circ\) or \(90^\circ\) and has its maximum value when the boundary is either parallel or normal to the applied stress direction. In these positions the flux is \(\sqrt{2}A\) so that the ratio between the maximum and minimum possible fluxes is \(\frac{1}{\sqrt{2}}\). The grain boundary energy is considered to be the sum of the two terms \(E_0\) and \(E_d\), where \(E_0\) is the normal isotropic energy and \(E_d\) is the additional energy due to the dislocation flux. Because of this geometrical effect, the grain boundary has its minimum energy during plastic deformation when positioned at \(45^\circ\) to the applied stress direction. The square grain configuration therefore has a lower energy than the usual random distribution. The stable array of square grains will have a steady state dislocation density \(C_s\). The dislocation density depends on the dislocation flux entering the boundary and the rate of annihilation there. The flux entering the boundary is given by: \(\hat{\alpha} \cdot \dot{\varepsilon}\) where \(\dot{\varepsilon}\) is the strain rate and the factor \(\hat{\alpha}\) incorporating the
geometrical term and other constants. The annihilation rate is given by $b C^2$, where $C$ is the dislocation concentration and $b$ is a temperature dependent constant.

Therefore: \[
\frac{dC}{dt} = a |\dot{\epsilon}| - b C^2 \quad \ldots (1)
\]

At steady state: \[
\frac{dC}{dt} = 0, \quad \text{so} \quad \frac{c_s}{b} = \left( \frac{a |\dot{\epsilon}|}{b} \right)^\frac{1}{2} \quad \ldots (2)
\]

If $E$ is the specific energy associated with a dislocation:

\[
E^d = C_s E \quad \ldots (3)
\]

It is postulated that the square grain structure will be sufficiently stable if $E^d$ is large enough, consequently the steady state dislocation concentration $C_s$ must also be large enough for square grain formation to be energetically favourable.

To find the value of $C_s$ required, the breakup of a fourfold point into two triple points is next considered (Fig. 3.45).

The new section of boundary, length $2a$, is assumed to be in equilibrium with the dislocation flux and therefore has a dislocation concentration $C_s \delta$, where $\delta$ accounts for the higher dislocation density in the non-$45^0$ boundary orientation. The energy of the new section of boundary is therefore $21(E_o + C_s \delta)$, $\delta$ consists of two factors: a factor 2 because two dislocation fluxes now contribute and a factor $(1/\sqrt{2})^\frac{1}{2}$ because the dislocation flux along one $45^0$ direction is reduced when the boundary is tilted at an angle to the flux direction (Fig. 3.46).

From Fig. 3.46 it may be seen that $a$ is reduced by a factor $1/\sqrt{2}$ and so $C_s$ will be reduced by a factor $(1/\sqrt{2})^\frac{1}{2}$. $\delta$ is thus given by $2/(1/\sqrt{2})^\frac{1}{2}$. The formation of a new length $2a$ of new boundary involves the reduction of the length of the $45^0$ boundary by a factor $41/\sqrt{2}$. The energy reduction is therefore $41(E_o + C_s \bar{E})$. Breakup of the quadruple point will consequently not occur if the energy of the new boundary created is greater than the energy of the $45^0$ boundary removed. That is, if

\[
21(E_o + C_s \delta \bar{E}) > \frac{41}{\sqrt{2}} (E_o + C_s \bar{E}) \quad \ldots (4)
\]

which simplifies to:

\[
C_s > 1.55E_o/\bar{E} \quad \ldots (5)
\]
Taking typical values of $E_0 = 200 \text{ erg/cm}^2$ and $E = 10^8 \text{ eV/cm}$ the critical dislocation spacing required to satisfy equation (5) is of order $50\text{A}$. If the critical concentration is denoted by $C_{so}$ then from equation (2):

$$|\varepsilon|_0 = \frac{bc^{2}_{so}}{a} \quad \cdots \quad (6)$$

Therefore there should be a critical strain rate below which square grain formation will not occur. This result is in agreement with Skelton's observation that square grain formation did not occur at a low strain rate (see page 84). It also accounts for the observation that square grain formation is not found during creep deformation, even though extensive grain boundary migration may occur. The model also predicts that the critical strain rate should increase with increasing temperature owing to the enhanced rate of annihilation, but no experimental data are available to check this point.

The absence of square grain formation during elevated temperature tensile deformation may be accounted for as follows:

(1) The kinetics of the process may not permit significant re-orientation of the boundaries during the time available before specimen failure. For the same strain rate, time to failure is naturally greater during LCF than in tensile tests unless very large strain amplitudes are employed.

(2) Large plastic deformations without change in grain size require an increase in the length of boundary orientated in the applied stress direction, owing to stretching of the grains. Under these conditions it could be expected that boundaries lying parallel to the tensile axis would have the least energy. Therefore when large changes of grain shape must occur, as in tensile deformation, square grain formation does not occur.

(3) The model of square grain formation described here requires a high grain boundary dislocation density for the square configuration to be stable. High grain boundary dislocation densities are possible in fatigue. During tensile deformation much lower densities are found.
Fig. 3.1 Type 1 Fatigue Specimen

Fig. 3.2 Type 2 Fatigue Specimen

Fig. 3.3 Tensile Specimen

All Dimensions in MM.
Fig. 3.4a  Bend-fatigue Equipment (Schematic)
Fig. 3.4b Low Cycle Fatigue Testing Equipment
(Vacuum Chamber and Furnace not shown)
Fig. 3.5a
Recorder Trace of LCF Hysteresis Loop (Schematic)

Fig. 3.5b
Portions of Trace as Parts of the LCF Hysteresis Loop (Schematic)

Fig. 3.5c
Portions of Trace corresponding to the elastic, plastic & total strain amplitude
Fig. 3.7 Endurance plot for as heat treated steel 500°C

Fig. 3.8 Endurance plot for as heat-treated steel 600°C
Fig. 3.9 Endurance plot for as heat treated steel 700°C

Fig. 3.10 Endurance plot for as heat treated steel 750°C
Fig. 3.11 Endurance plot for as heat treated steel
strain rate $2 \times 10^{-5}$ sec$^{-1}$

Fig. 3.12 Endurance plot for as heat treated steel
strain rate $1 \times 10^{-4}$ sec$^{-1}$.
Fig. 3.13 Endurance plot for as heat treated steel
strain rate $3.5 \times 10^{-4}$ sec$^{-1}$

Fig. 3.14 Endurance plot for as heat treated steel
strain rate $6 \times 10^{-4}$ sec$^{-1}$
Fig. 3.15 Effect of test temperature on endurance

Fig. 3.16 Variation of Coffin-Manson exponent with temperature
Fig. 3.17 Endurance plot for as heat treated steel effect of strain rate, 600°C

Fig. 3.18 Endurance plot for as heat treated steel effect of strain rate, 700°C
Fig. 3.19 Endurance plot for as heat treated steel effect of strain rate, 800°C

Fig. 3.20 Endurance plot for as heat treated steel effect of strain rate, 800°C
Fig. 3.21 Variation of cyclic strain hardening exponent ($\beta$) and strain rate sensitivity exponent ($m$) with temperature.

Fig. 3.22 Variation of cyclic and tensile hardening exponents with strain rate.
Fig. 3.23 Variation of cyclic strain rate sensitivity factor with grain size & temperature.

Fig. 3.24 Variation of cyclic strength parameter with temperature.
Fig. 3.25 Determination of the Propagation Constant

Fig. 3.26 Linear variation of reciprocal propagation constant with endurance.
FIG. 3.27 VARIATION OF PROPAGATION CONSTANT WITH TEMPERATURE

FIG. 3.28 REDUCED ENDURANCE OF AGED STEEL AT 800°C
PLASTIC STRAIN AMPLITUDE

\( \Delta \varepsilon_p \) (%)

800°C AGED 75d 700°C

\( \bigcirc \) VACUUM \( 2 \times 10^{-5} \) TORR

\( \bigtriangleup \) VACUUM \( 1 \times 10^{-4} \) TORR

ENDURANCE (CYCLES)

FIG. 3.29 EFFECT OF VACUUM QUALITY ON ENDURANCE

FRACTURE STRAIN (DUCTILITY)

\( \varepsilon_F \) (%)

ROOM TEMPERATURE VALUE

AS HEAT-TREATED.

STRAIN RATE \( 10^{-4} \) SEC\(^{-1}\)

GRAIN SIZE 12um

FIG. 3.30 AS HEAT TREATED STEEL, DUCTILITY v TEMPERATURE
FIG. 3.31 VARIATION OF DUCTILITY WITH STRAIN RATE

FIG. 3.32 VARIATION OF UTS WITH TEMPERATURE
FIG. 3.33 VARIATION OF UTS WITH STRAIN RATE

FIG. 3.34 VARIATION OF TENSILE HARDENING EXPONENTS WITH TEMPERATURE
FIG. 3.35 VARIATION OF TENSILE STRAIN RATE SENSITIVITY EXponent WITH GRAIN SIZE AND TEMPERATURE

FIG. 3.36 VARIATION OF CRACK PROPAGATION RATE WITH CRACK LENGTH
FIG. 3.37 INCREASE IN GRAIN SIZE WITH DISTANCE FROM NEUTRAL AXIS

FIG. 3.38 INDUCTION PERIOD BEFORE START OF GBM
Fig. 3.39 Schematic view of formation of surface crack nucleus. Arrows indicate direction of movement.

Fig. 3.39 Sectional schematic view of surface crack nucleus.

Fig. 3.40 Formation of an intergranular section of crack in elevated temperature mixed-mode fracture: (a) Intergranular nucleus, (b) More crack surface, (c) Intrusive crack formed by sliding during compression, (d) Characteristic surface depression with crack at the root, as in specimen tested to failure.
surface cracks

Unloaded zone due to cracks "A" inhibits growth of cracks "B"

Fig. 3.41 Unloading effect of surface cracks on one another

local strain directions

Fig. 3.42 Formation of cross-linking cracks normal to local strain directions.

(a)

(b)

cross-linking cracks

Experimental Points

Original Tomkins Theory

Modified Theory

Fig. 3.43 Observed and predicted endurances
Fig. 3.44 Dislocation Flux: elevated temperature deformation.

Fig. 3.45 Breakup of a fourfold point into a pair of triple points

Fig. 3.46 Effect of boundary tilt on flux
4.1. Introduction

In this chapter the results of low cycle fatigue and tensile tests on irradiated 20/25/Nb stainless steel are presented, followed by the results of metallographic examination of the fractured specimens. The mechanical test results and fracture characteristics are compared with those of the unirradiated steel, the deficiencies of the existing model of elevated temperature irradiation embrittlement are highlighted, and a model proposed which qualitatively accounts for the principal features of the embrittlement phenomenon.
4.2. Test Methods

4.2.1 Low-Cycle Fatigue Specimens and Test Equipment

Cast Z240 steel, heat treated to produce a grain size of 12 μm, was used exclusively in the investigation into the mechanical properties and fracture characteristics of the steel in the irradiated condition. Details of composition and heat treatment are given in Chapter 2, section 2.1, page 100.

Both Type 1 and Type 2 specimens were used (the specimen types are described in Chapter 3, section 3.2.1, page 128 and illustrated in Figs. 3.1 and 3.2). The Type 2 specimens were metallographically polished on one face before the irradiation treatment, followed by a further fine polishing treatment after irradiation to remove the slight surface oxidation and discolouration acquired during the period in the irradiation rig.

Irradiations were carried out in the DIDO reactor as follows:

(1) Type 1 specimens: 75d at 700°C, to a neutron dose of $1 \times 10^{21}$ n/cm$^2$ (thermal) and $3 \times 10^{20}$ n/cm$^2$ (fission).

(2) Type 2 specimens: 75d at 750°C to the same neutron doses as in (1).

The terms "thermal" and "fission" doses are explained in the Literature Review, section 1.2.2(a), page 42. The different irradiation temperatures were a consequence of the availability of space in different irradiation rigs. The LCF test equipment was identical to that described in section 3.2.1, page 128, and was installed in a lead shielded cell.

4.2.2 Tensile Test Specimens and Equipment

The cast Z240 steel, heat treated to produce a grain size of 12 μm, was used for the tensile specimens. Specimens were of the same design as described in section 3.2.2, page 130, and illustrated in Fig. 3.3. The irradiation treatment consisted of 75d at 700°C to the same neutron doses as described in the previous section for the LCF specimens.

The tensile test equipment was identical to that used for the unirradiated steel and was installed in a lead shielded cell.
4.3. **Experimental Procedures**

4.3.1 **Mechanical Testing**

The LCF test variables were temperature, plastic strain amplitude and strain rate, as for the unirradiated steel, described in section 3.3.1, page 131, and all tests were carried out in vacuo. Temperatures ranged from 500 to 800°C. Plastic strain amplitude was employed as a test variable at one temperature only, 800°C, covering the approximate range 1 to 3%. Strain rates from $2 \times 10^{-5}$ sec$^{-1}$ to $1.5 \times 10^{-3}$ sec$^{-1}$ were employed at two test temperatures of 700°C and 800°C. Plastic strain amplitudes and strain rates were calculated using the same methods described in section 3.3.1.

The tensile test variables were temperature and strain rate, as for the unirradiated steel (section 3.3.1), test temperatures covering the range 600°C to 800°C and strain rates $2 \times 10^{-5}$ sec$^{-1}$ to $1 \times 10^{-2}$ sec$^{-1}$. All tests were carried out in vacuo.

Some incremental strain tests were performed on tensile specimens in order to investigate possible recovery effects from elevated temperature irradiation embrittlement. Enhanced ductility, possibly due to void sintering, has been observed in the unirradiated steel (see Literature Review, section 1.3.4(d), page 89), subjected to incremental straining. The test procedure was as follows:

1. A fixed amount of strain was applied at a strain rate of $1 \times 10^{-4}$ sec$^{-1}$.
2. The specimen was unloaded, and held in the unloaded state for a period equal to the strain increment time.
3. The above procedure was repeated until failure occurred.

The test temperature for these incremental strain tests was 800°C.

4.3.2 **Metallographic Examination**

The LCF fracture characteristics were studied using four techniques:

1. Conventional microsections.
2. Fractography using scanning electron microscopy (SEM).
3. Optical microscope and SEM examination of a surface of a Type 2 specimen with one face metallographically polished before testing.
4. Transmission electron microscopy (TEM) of thin foils.
All except (4) had been employed previously in the metallographic examination of the unirradiated LCF tested steel (see section 3.3.2, page 132).

The TEM examination was intended chiefly for studying helium bubble structure (helium bubbles are discussed in the Literature Review, section 1.2.3(b), page 47). The method of preparation of thin foil specimens for TEM examination has been described in Chapter 2, section 2.3.3, page 102.

Some studies were made of the subsurface helium bubble structure at a depth of approximately 5 μm, corresponding to the mean depth of the subsidiary surface cracks in unirradiated LCF specimens tested at 700 and 800°C, by the following method.

The approximately 5 μm thick surface layer was removed by a vibropolishing method (268) the polished surface then being protected by "Lacomit" varnish. Foil specimens were then prepared by punching out 3 mm diameter discs and electropolishing to perforation from the unpolished side only, using the technique described in section 2.3.3, page 102, the vibropolished surface being still protected by its coat of "Lacomit" during this operation.

Thin foil specimens for the examination of helium bubble structure close to the tip of the principal crack were prepared by the technique shown schematically in Fig. 4.1. A single LCF crack of reproducible depth and location was produced by growing the crack from a notch introduced by manually drawing a razor blade across the specimen surface. LCF crack growth from the notch root was carried out by cycling at a plastic strain amplitude of 1.5%, strain rate of \(1 \times 10^{-4}\) sec\(^{-1}\) at a temperature of 700°C, until a load drop of 20% was reached (i.e. fatiguing to the endurance). Measurements of the rate of removal of material during electropolishing indicated that the thin areas of the foil were within 10 μm of the crack tip (i.e. approximately one grain diameter).
4.4. Results: Mechanical Properties

4.4.1 Low-Cycle Fatigue Properties

The LCF test programme is summarised in Table 4.1.

The effect of test temperature on the LCF endurance of the steel irradiated at 750°C for 75d is shown in Fig. 4.2 together with the results for unirradiated control specimens aged for the same time and temperature (the data for the controls is described in section 3.4.1(b), page 139, and also shown in Fig. 3.15). The reduced endurance of the irradiated steel at 600°C and upwards is apparent, compared with the control specimens, although the curve for the irradiated steel still displays a minimum at 700°C in a similar manner to that of the unirradiated steel at 600°C.

Some difficulty was experienced with presetting the required plastic strain amplitude as mentioned in the previous chapter (section 3.4.1, page 134) so that in the 800°C test on the irradiated steel the plastic strain amplitude was somewhat lower than in the other tests (1.18%, see Table 4.1); this raises the question whether the higher endurance value at 800°C was simply due to the lower plastic strain amplitude. Assuming that the slope of the plastic strain amplitude-endurance plot of irradiated steel at 800°C and a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ is similar to that of the unirradiated as heat treated steel (the only steel condition for which complete data are available) the endurance of the irradiated steel at a plastic strain amplitude of 1.5% is predicted to have been 470 cycles. This is still substantially greater than the corresponding endurance at 700°C (260 cycles, shown on Fig. 4.2), so that the upturn in the endurance-temperature curve of the irradiated steel was not due solely to the lower plastic strain amplitude in the 800°C test.

Crack propagation constants were determined as described in Chapter 3, section 3.4.1(a), page 137, and are plotted for the irradiated steel and the control specimens in Fig. 4.3. The propagation constant of the irradiated steel increased up to 700°C and then decreased, whereas in the unirradiated control specimens the increase in propagation constant reached a maximum at 600°C and then
## Table 4.1

Low-Cycle Fatigue Tests on Irradiated Steel

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Irradiation Treatment</th>
<th>Test Temp. (°C)</th>
<th>Plastic Strain Amplitude (%)</th>
<th>Strain Rate (sec⁻¹)</th>
<th>Figures</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>75d 750°C</td>
<td>500</td>
<td>1.5</td>
<td>1 X 10⁻⁴</td>
<td>4.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>600</td>
<td>1.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>700</td>
<td>1.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>800</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>75d 700°C</td>
<td>800</td>
<td>1.3</td>
<td>2 X 10⁻⁵</td>
<td>4.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>75d 700°C</td>
<td>700</td>
<td>1.6</td>
<td>2 X 10⁻⁵</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.6</td>
<td>1 X 10⁻⁴</td>
<td></td>
</tr>
<tr>
<td></td>
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<td></td>
<td>1.5</td>
<td>5.5 X 10⁻⁴</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.6</td>
<td>1.5 X 10⁻³</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>75d 700°C</td>
<td>800</td>
<td>1.8</td>
<td>2 X 10⁻⁵</td>
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<td>1.8</td>
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<td>5.5 X 10⁻⁴</td>
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<td>1.6</td>
<td>1.5 X 10⁻³</td>
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## Table 4.2

Tensile Tests on Irradiated Steel

<table>
<thead>
<tr>
<th>Irradiation Treatment</th>
<th>Test Temp. (°C)</th>
<th>Strain Rate (sec⁻¹)</th>
<th>Figures</th>
</tr>
</thead>
<tbody>
<tr>
<td>75d 700°C</td>
<td>600</td>
<td>7.5 X 10⁻⁵</td>
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<tr>
<td></td>
<td></td>
<td>9 X 10⁻⁵</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2 X 10⁻²</td>
<td></td>
</tr>
<tr>
<td>75d 700°C</td>
<td>700</td>
<td>2 X 10⁻⁵</td>
<td>4.7</td>
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<tr>
<td></td>
<td></td>
<td>7.5 X 10⁻⁵</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9 X 10⁻³</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.1 X 10⁻²</td>
<td></td>
</tr>
<tr>
<td>75d 700°C</td>
<td>800</td>
<td>1.7 X 10⁻⁵</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8.2 X 10⁻⁵</td>
<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.1 X 10⁻²</td>
<td></td>
</tr>
<tr>
<td>75d 700°C</td>
<td>800</td>
<td>1 X 10⁻⁴</td>
<td>4.8</td>
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</tbody>
</table>

*Incremental Strain Tests*
decreased. The effect of plastic strain amplitude on the LCF endurance of the irradiated steel at a test temperature of 800°C and a strain rate of $2 \times 10^{-5} \text{ sec}^{-1}$ is shown in Fig. 4.4. The test conditions (800°C; $2 \times 10^{-5} \text{ sec}^{-1}$) were chosen in order to see whether the effect of irradiation on endurance disappeared at plastic strain amplitudes of about 1% or so in a similar manner to that observed in previous work at a test temperature of 700°C and strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$ (352), the higher temperature and lower strain rate in the present case being more favourable to diffusion processes. It is clear from Fig. 4.4, however, that the influence of irradiation on endurance at 800°C and $2 \times 10^{-5} \text{ sec}^{-1}$ diminished with decreasing plastic strain amplitude and was negligible at 1%, in the same way as in the tests at 700°C and a strain rate of $1 \times 10^{-4} \text{ sec}^{-1}$.

The influence of strain rate on endurance at test temperatures of 700°C and 800°C is illustrated in Figs. 4.5 and 4.6, the mean plastic strain amplitudes being approximately 1.6% and 1.7%. These test temperatures were again chosen to correspond to those where diffusion processes and elevated temperature embrittlement were prominent.

At a test temperature of 700°C, there was an increase in LCF endurance of the irradiated steel with increasing strain rate (Fig. 4.5). The endurance of the unirradiated control specimens, on the other hand, was apparently insensitive to strain rate (Fig. 4.5) see also section 3.4.1(b), page 139, and Fig. 3.28. Strain rate had no effect on LCF endurance of the irradiated steel in tests conducted at 800°C, although this may have been fortuitous since the plastic strain amplitude again differed between the different tests, being 1.8% at the lowest strain rate and 1.6% at the highest (see Table 4.1). If the tests had been conducted at a plastic strain amplitude of exactly 1.5%, the endurances at the lower strain rates would therefore have been somewhat greater than those at the higher strain rates. This is rather unexpected since the opposite behaviour is implied for the irradiated steel at this test temperature than for unirradiated control specimens (also shown in Fig. 4.6). Furthermore, the insensitivity of LCF endurance to strain rate at 800°C was shown also by the as heat treated (unirradiated) steel (see section 3.4.1(a), page 136, and Figs. 3.19 and 3.20). Therefore the as heat treated and
irradiated steels showed a similar response to strain rate at 800°C, whereas the unirradiated aged steel showed a quite different response to the other two, in that the endurance at a strain rate of 2 x 10^{-5} \text{ sec}^{-1} was markedly less than that at higher strain rates (Fig. 4.6 and also section 3.4.1(b), page 139, and Fig. 3.28).

4.4.2 Tensile Tests

The tensile test programme is summarised in Table 4.2.

The tensile ductilities at test temperatures of 600, 700 and 800°C and strain rates ranging from 2 x 10^{-5} \text{ sec}^{-1} to 1 x 10^{-2} \text{ sec}^{-1} were determined for the irradiated steel; the results for these and as heat treated tensile specimens are plotted in Fig. 4.7. Unirradiated aged control specimens were not available to cover the whole strain rate range of the tests on irradiated specimens, though some results are given in section 3.4.2, page 140, and Fig. 3.31.

The overall lower ductility of the irradiated steel is apparent in the results presented in Fig. 4.7, as also is the increase in ductility of the irradiated steel with increased strain rate at all three test temperatures; the ductility of approximately 10\% at a strain rate of 2 x 10^{-5} \text{ sec}^{-1} at test temperatures of 700°C and 800°C increasing to approximately 20\% at a strain rate of approximately 1 x 10^{-2} \text{ sec}^{-1}. Conversely, the ductility of the as heat treated steel declined with increasing strain rate at test temperatures of 600 and 700°C, the net effect being that at the highest strain rate of approximately 1 x 10^{-2} \text{ sec}^{-1} there was little difference between the ductility of the unirradiated and irradiated steels.

The fracture stresses were similar in both the unirradiated and irradiated steels.

The results of the incremental strain tensile tests are presented in Fig. 4.8. A pronounced increase in ductility occurred at strain increments of less than 2\%; at strain increments of 0.5\% this amounted to almost double the ductility in a conventional continuous strain tensile test. It was not practicable to use strain increments of less than 0.5\% with the equipment available, so that it was not certain whether still larger ductilities could have been obtained at smaller strain increments than this.
4.5. **Results: Metallographic Examination of Tested Specimens**

The programme of metallographic work is summarised in Table 4.3.

### 4.5.1 Low-Cycle Fatigue Fracture of Irradiated Steel

#### 4.5.1(a) The Influence of Test Temperature

The fracture characteristics of four specimens irradiated for 75d at 750°C were examined after testing to failure at temperatures from 500°C to 800°C, using a plastic strain amplitude of approximately 1.5% and a strain rate of $1 \times 10^{-4}$ sec$^{-1}$.

**500°C**

The surface fracture characteristics were identical to those of the corresponding unirradiated aged control specimen tested to failure (see section 3.5.2(a), page 151) and also identical to those of the unirradiated as heat treated steel (section 3.5.1(a), page 143); there being considerable slip band structure not closely associated with cracks (Plate 4.1, compare with unirradiated as heat treated steel, Plate 3.6).

Examination of a microsection showed that the interior cracking was fine and branched, similar to the unirradiated, aged steel.

Fractography (using SEM) likewise showed that the unirradiated and irradiated steels had very similar fracture surfaces, the characteristic pattern of striations being present in both, together with areas of intergranular fracture (Plate 4.2, compare with unirradiated, aged steel, Plate 3.42).

**600°C**

The surface fracture characteristics were identical to those of the unirradiated aged control specimen tested to failure (see section 3.5.2(a), page 151) and also identical to those of the unirradiated as heat treated steel (section 3.5.1(a), page 145); a fully intergranular fracture mode.

Examination of a microsection showed that the appearance of the interior cracking was similar to that of the specimen tested at 500°C.

Fractographic examination confirmed that the fracture mode was intergranular, as in the unirradiated aged steel (Plate 4.3, compare with Plate 3.45).
METALLOGRAPHY PROGRAMME FOR THE IRRADIATED STEEL

KEY TO ABBREVIATIONS:
F: CYCLED TO FAILURE
S: SURFACE EXAMINATION OF PREPOLISHED SURFACES
M: MICROSECTIONS
E: SCANNING ELECTRON MICROSCOPE
T: TEM OF SUBSURFACE THIN FOILS
C: TEM OF CRACK TIP THIN FOILS

<table>
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<tr>
<th>LCF CYCLES</th>
<th>TEST TEMPERATURE (°C)</th>
<th>PLASTIC STRAIN AMP. (%)</th>
<th>STRAIN RATE</th>
<th>TYPE OF EXAMINATION</th>
<th>PLATES</th>
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<tr>
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<td>S,M,E</td>
<td>4.1, 4.2</td>
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<tr>
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<td>600</td>
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<td></td>
<td>S,M,E</td>
<td>4.3</td>
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<td>700</td>
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<td></td>
<td>S,M,E</td>
<td>4.4 to 4.12</td>
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<td></td>
<td>800</td>
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<td>T,C</td>
<td></td>
</tr>
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<td></td>
<td></td>
<td></td>
<td>S,M,E</td>
<td>4.13 to 4.17</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>T,C</td>
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TABLE 4.4
METALLOGRAPHY PROGRAMME FOR TENSILE TESTS, IRRADIATED STEEL

<table>
<thead>
<tr>
<th>IRRADIATION TREATMENT</th>
<th>TEST TEMPERATURE (°C)</th>
<th>STRAIN RATE (SEC^-1)</th>
<th>TYPE OF EXAMINATION</th>
<th>PLATES</th>
</tr>
</thead>
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<tr>
<td>75d at 700°C</td>
<td>600</td>
<td>1 X 10^-4</td>
<td>E</td>
<td>4.16 &amp; 4.17</td>
</tr>
<tr>
<td></td>
<td>700</td>
<td></td>
<td>E</td>
<td>4.18</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td></td>
<td>E</td>
<td>4.19 &amp; 4.20</td>
</tr>
</tbody>
</table>
The surface fracture characteristics were similar, but not identical, to those of unirradiated aged steel tested to failure (see section 3.5.2(a), page 153) and the as heat treated steel (section 3.5.1(a), page 145), the fracture being mixed-mode (Plate 4.4 and 4.5), but the cracks appeared to be at an earlier stage of development than in the unirradiated steel. The irradiated specimen tested to failure (Plate 4.4) may be compared with the as heat treated steel tested for about half the endurance (Plate 3.17), in the latter the cracks were longer and more developed, and more displacement of crack edges and surface distortion had occurred, as evinced by comparing the microsection of the irradiated steel, Plate 4.6, with that of the unirradiated as heat treated steel, Plate 3.23. The greater development of the cracks in the unirradiated steel was no doubt a consequence of its greater endurance more LCF cycles resulting in more grain boundary shear. It is also apparent on comparing Plates 4.6 and 3.23 that less development of the subsidiary cracks occurred in the irradiated specimen.

The fracture surface, although damaged by oxidation, appeared to be intergranular in character (Plate 4.7) and therefore differed from that of the unirradiated aged specimen (section 3.5.2(a), page 153 and Plate 3.47) which did not show a distinct intergranular fracture mode.

Thin foils for TEM examination were prepared by the vibro-polishing technique described earlier in section 4.3.2. Few helium bubbles were visible and those seen in the grain boundaries were generally small, about 50A or so in diameter. Crack tip thin foils were prepared as described in section 4.3.2, although only about one third of these specimens were satisfactory, chiefly because of the difficulty of positioning the acid jet precisely under the depression in the polished face (see Fig. 4.1) when carrying out the "dishing" operation (see Chapter 2, section 2.3.3, page 102). If the dished area was slightly off centre then perforation on subsequent electropolishing did not occur at the bottom of the depression, and so no electron transparent area was obtained. Some bubbles having diameters of the order of a couple of hundred A were observed (Plates 4.8 to 4.10) in or near grain boundaries. In two instances, clusters of large bubbles were seen on grain boundaries (Plates 4.8 and 4.9):
The LCF fracture characteristics of the irradiated steel tested at this temperature were similar to those at 700°C. Little crack edge displacement and surface distortion had occurred and subsidiary surface cracks had only penetrated a short distance into the interior (Plate 4.11). No orthogonal grain structure was present. The fracture surface had a somewhat more intergranular character than that of the unirradiated aged specimen, a network of branched intergranular cracks being present (Plates 4.12 and 4.13). The unirradiated steel is discussed in section 3.5.2(a), page 151, and Plates 3.50 and 3.51 may be compared with 4.12 and 4.13.

Thin foils were prepared for TEM examination using the vibro-polishing technique, as for the irradiated steel tested at 700°C. Again, only a few grain boundary helium bubbles were seen, of about 50Å diameter.

Crack tip thin foils were prepared, as for the 700°C tested specimens, and some large bubbles were seen in or near grain boundaries, up to a couple of hundred Å or so (Plates 4.14 and 4.15) but no clusters of bubbles like those in the 700°C tested steel were found, however.

4.5.1(b) The Influence of Strain Rate

In view of the marked effect of strain rate on the endurance of the unirradiated aged steel, notably the decline in endurance with decreasing strain rate from $1 \times 10^{-4}$ sec$^{-1}$ to $2 \times 10^{-5}$ sec$^{-1}$, (see section 3.5.2(b), page 154) in tests conducted at 800°C, an examination was made of the fracture characteristics of irradiated steel tested at 700 and 800°C at a strain rate of $2 \times 10^{-5}$ sec$^{-1}$ to see whether any changes occurred in a similar way to those observed in the unirradiated steel. However the fracture characteristics at the lower strain rate were the same as those at a strain rate of $1 \times 10^{-4}$ sec$^{-1}$.

Summary

No significant differences between the fracture characteristics of the irradiated steel and unirradiated steel at test temperatures of 500 and 600°C were found. At 700 and 800°C, the new type of mixed-mode fracture was observed on examination of prepolished surfaces, but the fracture surfaces had a more intergranular appearance than in the unirradiated steel; the extent of surface grain boundary
shear was also less in the irradiated specimens. No orthogonal grain structure developed in the irradiated specimens.

No increase in the size of helium bubbles was found in sub-surface thin foils, but large bubbles were found in crack tip foils from specimens tested at 700 and 800°C.

Reduction of strain rate to $2 \times 10^{-5}$ sec$^{-1}$ at 700 and 800°C had no perceptible effect on the surface fracture characteristics.

### 4.5.2 Tensile Fracture of Irradiated Steel

The programme of metallographic work is summarised in Table 4.4.

The fracture characteristics of irradiated tensile specimens tested over the range 600 to 800°C at a strain rate of $1 \times 10^{-4}$ sec$^{-1}$ were examined. Corresponding metallographic examinations on unirradiated as heat treated steel are described in section 3.5.3, page 155.

#### 600°C

The fracture surface had the characteristic "dimpled" appearance of ductile fracture, although the dimples were smaller and their shape was less well defined than those of the fracture surface of the as heat treated steel (Plate 4.16, compared with Plate 3.63, page 271). The fine structure of the fracture surface was rougher than that of the unirradiated steel (Plate 4.17). Some necking had occurred before rupture.

#### 700°C

Examination of the specimen surface showed that many intergranular cracks were present, especially around the principal fracture, in contrast to the unirradiated as heat treated tensile specimen which had little surface cracking. The fracture was of an intergranular character (Plate 4.18; there is no corresponding fractograph for the unirradiated steel).

#### 800°C

Very extensive intergranular cracking had occurred (Plate 4.19) and the fracture surface was likewise of an intergranular character (Plate 4.20, compare with the unirradiated steel, Plate 3.64).

The fracture thus differed from that of the unirradiated steel, since intergranular fracture was not observed in the latter.
4.6. Discussion

4.6.1 Mechanical Testing

The principal observations made from the test results from the irradiated steel were:

1. The elevated temperature LCF endurance was markedly reduced by prior irradiation at elevated temperature, under the specific test conditions of plastic strain amplitude = 1.5\% and strain rate = 1 \times 10^{-4} \text{ sec}^{-1} (see section 4.4.1, page 204) (Fig. 4.2). However, the temperature-endurance plot of the irradiated steel had a similar minimum at 700°C to that of the unirradiated steel at 600°C (see also section 3.4.1(b), page 139) (Fig. 4.2).

2. Under test conditions of temperature = 800°C and strain rate = 2 \times 10^{-5} \text{ sec}^{-1} the difference between the endurances of unirradiated and irradiated steels diminished with decreasing plastic strain amplitude, becoming negligible at a plastic strain amplitude of about 1\% (section 4.4.1, page 204) (Fig. 4.4).

3. The influence of strain rate on the endurance of the irradiated steel varied with test temperature (see section 4.4.1, page 204) the plastic strain amplitude being approximately constant at 1.5\%: at 700°C the endurance increased with increasing strain rate (Fig. 4.5) while at 800°C strain rate had little influence on endurance (Fig. 4.6).

4. Over the temperature range 600 to 800°C the tensile ductility of the irradiated steel was lower than that of as heat treated steel, throughout a considerable range of strain rates (Fig. 4.7, see section 4.4.2, page 207). The difference between the ductility of the unirradiated and irradiated steels diminished with decreasing test temperature and increasing strain rate.

5. In the incremental strain tensile tests, an increase in ductility of the irradiated steel was found at strain increments of approximately 1\% or less (Fig. 4.8) (Section 4.4.2, page 207)
In general, prior neutron irradiation brought about a reduction of LCF endurance at elevated test temperatures, and similarly a reduction of ductility in tensile tests, in conformity with the established effects of irradiation embrittlement (See Literature Review, section 1.3.5, page 91). In the case of the LCF tests, however, a marked increase in endurance of the irradiated steel occurred at temperatures above 700°C (Section 4.4.1, page 206, and Fig. 4.2), a similar characteristic to that of the unirradiated steel, except the minimum in the temperature-endurance curve occurred at about 700°C instead of 600°C as in the unirradiated steel. In view of the association of the minimum of the temperature-endurance plot with intergranular fracture in the unirradiated steel (see section 3.5.1(a) page 143 and also discussion, section 3.6.2, page 159) the trend towards an intergranular fracture surface in the irradiated steel at a test temperature of 700°C (section 4.5.1(a), page 210) would be expected to correlate with a shift in the minimum of the temperature-endurance curve of the irradiated steel to a higher temperature, as observed.

The increase in endurance of the irradiated steel at test temperatures above 700°C did not have a counterpart in increased tensile ductility values, practically the same ductility was obtained at test temperatures from 600°C to 800°C, at a constant strain rate (section 4.4.2, page 207, and Fig. 4.7), in contrast with the enhanced ductility observed in the unirradiated steel at higher test temperatures (also shown in Fig. 4.7). In one respect the absence of enhanced ductility with increasing test temperature in the irradiated steel might be expected, since the rapid intergranular cracking would result in failure before the point of plastic instability was reached, and it was the inhibition of plastic instability by increased strain rate sensitivity exponent that resulted in the enhanced ductility of the unirradiated steel at elevated temperature (see discussion, section 3.6.1, page 158). In other words, since the irradiated steel never got to the point where plastic instability (i.e. necking) could start, irrespective of strain rate sensitivity exponent, then no improvement in ductility could accrue from an inhibition of plastic instability.
In another respect, however, the insensitivity of ductility to test temperature is rather unexpected, since at first sight higher test temperatures would have been expected to result in more rapid embrittlement due to faster diffusion processes. However, if failure occurred principally by intergranular crack propagation, then there could have been two opposing processes:

(1) Increased tendency to irradiation embrittlement by intergranular crack growth with the increased grain boundary diffusion at higher temperatures.

(2) Reduced tendency to crack propagation due to increased strain rate sensitivity exponent at higher temperatures and consequent strain rate hardening of material at the crack tip, i.e. essentially the same mechanism as proposed for the increased LCF endurance found in the unirradiated steel at test temperatures above 700°C (section 3.6.3, page 167).

Increasing test temperatures would have favoured both processes and thus may have resulted in the one tending to counterbalance the other.

The disappearance of the irradiation-induced reduction in LCF endurance at plastic strain amplitudes of 1% or less (at a test temperature of 700°C and strain rate of $1 \times 10^{-4}$ sec$^{-1}$) has been reported elsewhere (252) (see also section 4.4.1, page 206) and a similar disappearance of an irradiation effect at 1% plastic strain amplitude has been found in this work under test conditions of 800°C and strain rate of $2 \times 10^{-5}$ sec$^{-1}$ (see page 4.6 and Fig. 4.4). To explain this phenomenon the idea of a "damaged zone" existing ahead of the crack tip is introduced; this will be discussed in more detail in section 4.6.3. If it is assumed that a damaged zone formed ahead of a propagating LCF crack in the irradiated steel, into which the crack grew, then during the compressive half-cycle of an elevated temperature LCF test it is likely that some removal of the damaged zone occurred, perhaps by annealing or void sintering. At a sufficiently low plastic strain amplitude, the damaged zone formed during the tensile half-cycle may have been small enough for complete recovery to be achieved during the compressive half-cycle, so that no irradiation-induced endurance loss occurred.

Recovery processes have been observed in incremental strain tensile tests on unirradiated 20/25/Nb steel (see section 4.3.1, page 202)
leading to improved ductility, and likewise the incremental strain tests on irradiated steel described in section 4.4.2, page 207, and Fig. 4.8, showed a considerable ductility recovery over the conventional tensile test at strain increments of approximately 1% or less, similar to the value of the LCF plastic strain amplitude at which the irradiation effect on endurance disappeared. Since failure of the irradiated steel in the conventional tensile test occurred by formation and propagation of intergranular cracks (see previous page) the results of incremental strain tensile tests tend to provide additional support for the idea of a damaged zone ahead of the crack tip, the removal of which by an annealing treatment could result in improved LCF endurance or tensile ductility by reducing crack propagation rates.

The effect of strain rate on LCF endurance at 700°C and tensile ductility over the range 600 to 800°C of the irradiated steel was qualitatively what would have been expected from a diffusion controlled embrittlement process, namely the endurance and ductility both tended to increase with increasing strain rate (Fig. 4.5 and 4.7) since less time was available for grain boundary vacancy diffusion and consequent bubble growth. However, the LCF results at a test temperature of 800°C were inconsistent with this, the endurance being apparently independent of strain rate (at a plastic strain amplitude of approximately 1.5%), (see Fig. 4.6). This strain rate independence may be accounted for qualitatively by assuming that the crack tip damaged zone formation occurred non-linearly with respect to time, being initially rapid and then tailing off to a slow, or perhaps even zero, rate. In this way at a sufficiently high test temperature, or sufficiently low strain rate, the damaged zone would have formed at the start of the tensile half-cycle and thereafter have been of much the same structure throughout the rest of the half-cycle. Varying the strain rate, and therefore the cycle time, would not have affected the extent or structure of the damaged zone significantly and so crack propagation rates would have been strain rate independent. At lower test temperatures, diffusion processes would have been slower, so that at a sufficiently high strain rate there would not have been enough time for a stable damaged zone to form and so the effect of irradiation would have lessened, as in fact was observed (Fig. 4.5).
4.6.2 Fracture Behaviour

The principal observations made from the fracture characteristics of the irradiated steel were:

1. The characteristics of the LCF surface crack nucleation appeared to be identical to those of the unirradiated steel (section 4.5.1(a), page 208). In particular, the transition to the "mixed-mode" type of fracture (described previously in section 3.5.1(a), page 146) occurred at a test temperature of 700°C.

2. Failure in both LCF and tensile tests appeared to occur by the intergranular propagation of a surface-nucleated crack. Grain boundary void formation was not observed.

3. Transmission electron microscope examination of crack tip thin foils indicate that enlarged helium bubbles were present within one grain diameter of the crack tip, (section 4.5.1(a), page 210), and Figs. 4.8 to 4.10 and 4.14 and 4.15).

Prior irradiation did not bring about any change in the mechanism of LCF crack nucleation; in particular, the new type of mixed-mode surface fracture found in the unirradiated steel at test temperatures of 700°C and above (see section 3.5.1(a), page 146) was likewise observed in the irradiated steel (section 4.5.1(a), page 210).

The reduced LCF endurance of the irradiated steel at elevated temperature appeared to be caused by the increased rate of propagation of the cracks, associated with a tendency to intergranular fracture once the crack was past the nucleation stage (section 4.5.1(a), page 210, and Fig. 4.3). Increased crack propagation rates explain the small extent of subsidiary surface cracking observed in irradiated LCF specimens (section 4.5.1(a), page 210): once a sufficiently large crack had nucleated at the surface it propagated so rapidly through the specimen thickness that there was little time for other cracks to grow to a significant size. Increased crack propagation rates also explain the narrower appearance of cracks in the irradiated steel, there would have been less time for oxidation of the crack faces to produce a crack with a wider, blunter appearance as found in the unirradiated steel.
Prior irradiation produced a change in the tensile fracture characteristics of the irradiated steel. Instead of the cavitation found in the unirradiated steel, there was extensive grain boundary cracking. The absence of cavitation could have been expected in view of the lower ductility of the irradiated steel; published work on unirradiated 20/25/Nb steel indicates that there should be about 30% strain before a significant volume fraction of cavities will form, and the ductility of the steel in the irradiated condition was much less than this. The absence of widespread subsidiary cracking again suggests that tensile failure occurred by the rapid propagation of an intergranular crack, probably nucleated at the specimen surface.

The observation of enlarged helium bubbles lends support to the possible existence of a damaged zone at the crack tip, the helium bubbles possibly acting as void nuclei; no voids were observed in crack tip thin foils prepared from unirradiated specimens.

4.6.3 The Mechanism of Irradiation Embrittlement at Elevated Temperature

In section 4.6.1 the hypothesis of a damaged zone ahead of a propagating crack was introduced in order to account for the differences in behaviour between the unirradiated and irradiated steels. This was found to be necessary because the established theory of elevated temperature embrittlement by helium bubble growth and coalescence on grain boundaries did not appear to give a satisfactory account of the observed results. The theory is discussed in the Literature Review, section 1.2.4(b), page 57. The main reasons for this conclusion are summarised for convenience below:

1. Growth and coalescence of grain boundary helium bubbles should have resulted in widespread internal intergranular crack nucleation. This was not observed, and instead the elevated temperature crack nucleation mechanism in the irradiated steel appeared to be identical to that of the unirradiated steel.

2. The stresses in LCF and tensile tests were in some instances too low to produce unstable growth of even the largest grain boundary helium bubbles observed in thin foil specimens, although a pronounced effect of irradiation on mechanical properties was observed.
(3) The kinetics of bubble growth by vacancy absorption should have resulted in an inverse dependence of time to failure on stress, which was not observed; this lack of an inverse dependence has been commented on in published work (Literature Review, 1.2.4(b) page 60).

(4) The same kinetic considerations as in (3) above should have resulted in approximately linear dependence of LCF endurance and tensile ductility on strain rate, the higher the strain rate the higher the endurance and ductility. However, the endurance and ductility only changed slowly with strain rate, and in the LCF tests conducted at 800°C and a plastic strain amplitude of 1.5% there was no effect of strain rate at all (see Fig. 4.6).

(5) In LCF tests, increased test temperatures between 700 and 800°C should have resulted in reduced endurance, due to faster diffusion and faster bubble growth rates, but the opposite was observed. The slightly lower stress at the higher temperature was unlikely to have had much effect on bubble growth rates.

(6) The change in peak stress with plastic strain amplitude at 800°C and strain rate $2 \times 10^{-5}$ sec$^{-1}$ is very slow, since the cyclic strain hardening exponent is virtually zero under these conditions, hence the disappearance of the irradiation effect at a plastic strain amplitude of about 1% is very difficult to account for, since the stress would have to decrease with decreasing plastic strain amplitude until at the 1% amplitude level it was insufficient to cause unstable bubble growth.

The essential idea of the helium bubble growth and coalescence model of elevated temperature embrittlement, referred to previously, is that unstable growth of bubbles will occur if the applied stress reaches a value given by the equation

$$\sigma_c = 0.76 \gamma/r_0$$

in which $r_0$ is the initial, unstressed, bubble radius, and the stress is the normal stress acting across the grain boundary; growth here is assumed to occur entirely by the absorption of grain boundary vacancies. In the present work the lowest flow stress measured in a tensile test (at 800°C and strain rate approximately $2 \times 10^{-5}$ sec$^{-1}$, Fig. 4.7) was 9.4 ksi (65 MN/m$^2$). Since irradiation embrittlement was observed in this test, a lower bound on the minimum bubble radius
necessary for unstable growth may be obtained. Taking the surface energy \( \gamma \) as 1500 dyn/cm: 
\[
    r_o = 0.76 \times 1500/9.4 \times 0.69 \times 10^8 = 176 \times 10^{-8} \, \text{cm}.
\]
Therefore a bubble must have a diameter of about 350 A or greater in order to undergo unstable growth at an applied stress of 9.4ksi. Since the maximum bubble diameter in foils was of the order of 250 A, it appears that the great majority of bubbles would not have been able to grow in an unstable manner. The large irradiation embrittlement effect observed under the foregoing test conditions is therefore unexpected on the basis of the unstable bubble growth model. Reasons may have been:

1. The surface energy value assumed was too large; the calculated critical stress was also too large as a result.

2. It may not have been possible to see bubbles of 350 A or larger in thin foil specimens since the foil would perforate at the bubble during electropolishing.

3. The nearest neighbour spacing of bubbles larger than about 350 A may have been too large to permit a significant frequency of bubbles of this size to be observed in thin foil specimens.

4. The bubble growth and coalescence model assumes an equilibrium vacancy concentration in the grain boundary; this assumption may not be a valid one on all grain boundaries in a material undergoing deformation at elevated temperature.

Considering each of the above points in turn:

There is considerable uncertainty in surface energy values for metals and alloys but it is well established that most technologically important materials have surface energies in the range 1000 to 2000 dyn/cm. It appears unlikely therefore that the value of 1500 dyn/cm normally assumed for stainless steels is seriously in error.

Metal foil specimens used for transmission electron microscopy normally have thicknesses in the range 500 to 1000 A. It is therefore probable that helium bubbles larger than about 350 A will result in perforation of the foil. The frequency of bubbles of this size must, however, have been low in thin foil specimens since large areas comprising perhaps 25 grains were obtained in the electron transparent areas without holes.
It may be shown that the nearest neighbour spacing of bubbles in a given size range is given by \( a_t = (a_o d)^{1/2} \) where \( a_t \) is the actual spacing, \( a_o \) the measured spacing and \( d \) is the foil thickness (see Appendix 3). This result applies to grain boundary bubbles. If \( a_o \) is large compared to \( d \), the actual spacing will be much less than the observed one. For example, if \( d \) is 500 A and \( a_o \) is 10 \( \mu \)m then the actual nearest neighbour spacing is 0.7 \( \mu \)m. Conversely, a large actual spacing will result in very large apparent spacings, so that bubbles in the largest range of radii may only be seen very rarely. Thus bubbles of 350 A or larger may have been present in a much higher density in the boundary than indicated by measurements on thin foil specimens.

Assuming that failure occurs by the growth and coalescence of helium bubbles in grain boundaries having an equilibrium vacancy concentration, the time to rupture may be estimated according to one analysis \(^{(112)}\) by the relation:

\[
t_r = \frac{a^3 kT}{16 \sigma \Omega D_{gb} \delta z},
\]

where \( \sigma \) is the stress normal to the boundary, \( \Omega \) is the atomic volume, \( D_{gb} \) is the atomic grain boundary diffusion coefficient and \( \delta z \) is the grain boundary width; \( kT \) has its usual meaning. The time to rupture should therefore vary inversely with the applied stress. This implies that rupture should occur sooner in a test conducted at a lower strain rate, provided the stress does not decrease significantly. This has not been observed; although there was a decrease in ductility with decreasing strain rate (see Fig. 4.7) (section 4.4.2, page 207) time to failure did not follow an inverse relation with the applied stress. The absence of this relationship has been noted previously (see Literature Review, section 1.2.4(b), page 60) in irradiated austenitic stainless steels. Furthermore the foregoing equation implies that times to rupture should decrease rapidly with increasing temperature, because of the exponential temperature dependence of the diffusion coefficient \( D_{gb} \). This again is not apparent from the ductility data shown in Fig. 4.7.

In the following it is suggested that the deficiencies of the helium bubble growth and coalescence model illustrated above may be in part caused by the underlying assumption of equilibrium grain boundary vacancy concentration under the applied stress: this may
affect both the energetics and kinetics of the process. Departures from an equilibrium concentration may be caused by (1) grain boundary precipitates (2) generation or absorption of vacancies depending on grain boundary orientation. Intergranular precipitates may influence the transport processes in the grain boundary, so affecting its efficiency as a source or sink for vacancies. A dependence of grain boundary vacancy concentration on relative orientation has been suggested by other workers\(^\text{265, 267}\) to explain the non-uniform grain boundary sliding observed during creep; in particular, the fact that sliding is not found to occur on those boundaries parallel to the maximum shear stress direction, as would be expected in a material having isotropic grain boundary properties. According to this model, grain boundary sliding occurs by the climb of grain boundary dislocations. The climb process in turn results in the creation or annihilation of vacancies depending on the relative orientations of adjacent grains. Therefore the vacancy concentration in deforming material varies from one boundary to another.

Helium bubbles located in the grain boundaries can only grow if the chemical potential, which depends on vacancy concentration, is large enough. Under unstressed conditions, the equilibrium concentration is given by:

\[
\frac{c}{c_0} = e^{\frac{E_v}{kT}} \quad......(1)
\]

where \(E_v\) is the vacancy formation energy. If a normal stress \(\sigma\) acts across the grain boundary, part of the work done in vacancy formation is done by the stress so that the concentration is

\[
c = e^{\frac{E_v - \sigma\Omega}{kT}} \quad......(2)
\]

where the work term \(\sigma\Omega\) is the chemical potential. Combining (1) and (2):

\[
\frac{c}{c_0} = e^{\frac{\sigma\Omega}{kT}} \quad......(3)
\]

(\(\Omega\) is the atomic volume).

At an applied stress of 9.4ksi (65MN/m\(^2\)) the vacancy concentration calculated using equation (3), would be 13% greater than the concentration in the unstressed steel.
If the vacancy concentration in the grain boundary differs from the equilibrium value, then equation (3) may be written:

\[
\frac{c^*}{c} = \frac{e \sigma \Omega / k^T}{\gamma}
\]

where \(c^*\) is the non-equilibrium vacancy concentration and \(\sigma^*\) is the value of applied stress that would be necessary to produce the vacancy concentration \(c^*\) under equilibrium conditions.

In materials capable of grain boundary sliding at the test temperatures involved, the growth or otherwise of bubbles will depend on the value of \(\sigma^*\) in a particular grain boundary and will thus vary from one boundary to another. The applied stress, \(\sigma\), will not therefore necessarily be a criterion of the onset of irradiation embrittlement since the effective stress, \(\sigma^*\), may be higher in a particular boundary than the applied value.

In addition to these energetic considerations, the kinetics of helium bubble growth will be influenced by departures from equilibrium vacancy concentrations, since the chemical potential of the vacancies will be higher in those boundaries where vacancies are being created. Therefore a simple dependence of time to rupture of the type shown on page 221 on applied stress would not necessarily be expected. In particular, if the rate at which a growing helium bubble absorbs vacancies is governed entirely by the rate of vacancy creation in the adjoining boundary or boundaries, then this requires that the rate of volume increase should be constant. Since the infinitesimal volume, \(dv\), of a spherical shell is given by \(4\pi r^2 dr\), where \(r\) is the current radius of the sphere, then the rate of volume increase \(dv/\dot{t}\) is given by \(4\pi r^2 \dot{r}/\dot{t}\). As \(dv/\dot{t}\) is a constant, \(dr/\dot{t} = \text{constant}/4\pi r^2\). Hence the rate of increase of the radius of a spherical bubble will be inversely proportional to the square of its current radius; in other words the larger the bubble grows the slower will be the rate at which its radius increases, even though the growth is unstable, i.e. equilibrium is not being approached. Also in general the rate of vacancy generation in a boundary will depend on the dislocation concentration and rate of climb \((269)\) thus in the zone of intensified strain at the tip of a growing intergranular crack, where a high grain boundary dislocation density will exist, the best conditions will be available for the growth of helium bubbles.
In section 4.6.1, the idea of a "damaged zone" at the crack tip was introduced to explain the observed variations in mechanical properties. This explanation has been advanced by other workers to explain the enhanced intergranular crack propagation rates found in fatigue of AISI 316 stainless steel containing intergranular precipitates. The increased crack propagation rates were attributed to void formation around precipitates ahead of the crack tip: when the particle spacing was of the same order as the fatigue crack advance per cycle then the voids could contribute directly to the formation of new crack surface, and so increased crack propagation rates resulted. The process was considered to be a three-dimensional analogue of the tearing of a sheet along a line of perforations.

A similar mechanism is proposed here to explain qualitatively the features of LCF crack propagation at elevated temperature in irradiated 20/25/Nb steel, as follows:

(1) The nucleation of the LCF cracks occurred at the specimen surface in exactly the same way as in the unirradiated steel, by the new "mixed-mode" fracture process, as described in section 3.6.2 page 159.

(2) Strain concentration at the tips of the growing crack nuclei resulted in high grain boundary dislocation densities in a localised crack tip volume and consequent high rates of vacancy formation in favourably orientated boundaries, so resulting in some degree of vacancy supersaturation during the tensile half-cycle.

(3) The vacancies diffused to the intergranular helium bubbles close to the crack tip and the bubbles began to undergo unstable growth during the tensile half-cycle if their initial radii were large enough. During the compression half-cycle the bubbles tended to emit vacancies and return to their initial radii.

(4) The growth rate of the helium bubbles was controlled by the rate of vacancy formation. Therefore the rate of increase of the bubble radii decreased rapidly as the bubbles grew; the net effect was that bubble coalescence did not occur, but that a small area of grain boundary ahead of the crack became weakened by the presence of enlarged bubbles.
During crack advance tearing of the boundary occurred, the enlarged bubbles contributing directly to the formation of new crack surface; this process was essentially one of plastic deformation of the ligaments between the bubbles, not bubble coalescence. Tensile fracture of the irradiated steel occurred by basically the same mechanism, except that recovery processes (as in (3) above) were involved only in incremental strain tests.

The characteristic features of elevated temperature irradiation embrittlement according to the above model are:

1. Crack nucleation occurs by the same mechanism in both unirradiated and irradiated steel.

2. Embrittlement is a consequence of increased intergranular crack propagation rates.

3. Formation of new crack surface occurs by plastic deformation of ligaments between enlarged helium bubbles and decohesion of the grain boundary, not by unstable growth and linkage of bubbles.

4. The irradiation embrittlement will tend to diminish or disappear if recovery periods are introduced into the deformation regime. In LCF, if the crack advance per cycle is sufficiently low (i.e. the plastic strain amplitude is low enough) compared with the grain boundary bubble spacing, then the enlarged helium bubbles will not be able to contribute significantly to the formation of new crack surface and the influence of irradiation on LCF endurance will tend to disappear.

The above features are consistent with the following experimental observations:

1. The "mixed-mode" type of LCF surface crack nucleation has been observed in both unirradiated and irradiated steel (section 4.5.1(a), page 208 and section 4.6.2, page 217).

2. There was an increased tendency to intergranular fracture in the irradiated steel and a corresponding shift in the minimum of the temperature-endurance curve to a higher temperature; also the crack propagation constant was greater in the irradiated steel (section 4.4.1, page 204). In tensile tests the grain boundary cavitation fracture mechanism of the unirradiated steel was replaced by intergranular cracking in the irradiated steel.
(3) Crack advance by plastic deformation of ligaments, rather than diffusion controlled bubble growth would be expected to result in a relatively insensitive response to changes in test temperature and strain rate, as was in fact observed (section 4.4.1 and 4.4.2) with the proviso that test temperature and strain rate must be such as to allow significant bubble growth during each tensile half-cycle:

(4) Enlarged helium bubbles were observed in electron microscope examinations of thin foil specimens prepared from areas close to the tips of LCF cracks.

(5) Incremental strain tensile tests resulted in a considerable reduction of irradiation embrittlement (section 4.4.2). A reduction in plastic strain amplitude, and hence of crack advance per cycle, resulted in a smaller influence of irradiation on LCF endurance, which was largely independent of test temperature and strain rate as required by paragraph (3) (see section 4.4.1).

Although the mechanism of elevated temperature irradiation embrittlement proposed above appears to be qualitatively satisfactory there are severe difficulties involved in making the transition to a quantitative treatment. Some of the more important are the calculation of mean rates of grain boundary vacancy production in the crack tip zone, calculation of vacancy concentrations there and the determination of numbers and sizes of helium bubbles on the boundaries. The last mentioned should be practicable with extensive experimental work but the first two require the development of a comprehensive theory of grain boundary vacancy generation/annihilation which is lacking at present.
1. Notched-bend fatigue spec. with crack.
2. Notch & part of crack ground off. Spec. stopped off except for cracked face.
3. Successive electro-polishing until crack tip reached; surface depression formed.
4. Polished surface protected with Lacomit. Rim & bottom face ground to leave 50-75μm thickness in centre.
5. Spec. stopped off except for bottom face. Dished & electro polished until perforation.
6. Finished disc profile.

**FIG. 4.1 CRACK TIP THIN FOIL PREPARATION**

**FIG. 4.2 VARIATION OF ENDURANCE WITH TEMPERATURE**

IRRADIATED STEEL & THERMAL CONTROLS
**FIG. 4.3** VARIATION OF PROPAGATION CONSTANT WITH TEMPERATURE.

**FIG. 4.4** VARIATION OF ENDURANCE WITH PLASTIC STRAIN AMPLITUDE.
Fig. 4.5 Influence of strain rate on endurance

- IRRADIATED 75d 700°C
- AGED 75d 700°C

700°C
Mean plastic strain amplitude = 1.6%

Fig. 4.6 Influence of strain rate on endurance

- IRRADIATED 75d 700°C
- AGED 75d 700°C

800°C
Mean plastic strain amplitude = 1.7%
AS HEAT TREATED

800C •
700C △
600C ◆

75d 700C IRRADIATED

800C ○
700C △
600C ◆

700 & 800C

600C

FIG. 4.7 EFFECT OF STRAIN RATE ON DUCTILITY

STRAIN RATE (SEC-1)

STRAIN RATE = 1 x 10^-4 s^-1

IRRADIATED 75d 700C

800C

FIG. 4.8 EFFECT OF STRAIN INCREMENTS ON DUCTILITY
5. General Conclusions


(1) The occurrence and distribution of second phases in the unirradiated steel used in this investigation was broadly in accordance with existing published work.

(2) Irradiation had no apparent effect on the occurrence and distribution of second phases; in particular, the precipitate free zone found in the unirradiated steel adjacent to grain boundaries was present in the irradiated steel.

(3) Owing to the low boron content (approximately 0.2 ppm.wt.) the number of grain boundary helium bubbles was less than that of steel containing the "natural" boron content of approximately 2 ppm.wt.

(4) Carburisation of some irradiated specimens had occurred during the irradiation treatment leading to the formation of $M_{23}(C,N)_6$ carbonitride. Similar carburisation occurred in unirradiated specimens tested in argon atmosphere, probably due to hydrocarbons from vacuum seals. Carburisation had a deleterious effect on LCF endurance.

5.2. Chapter 3. Low Cycle Fatigue Properties of Unirradiated 20/25/Nb Steel

(1) At test temperatures up to 600°C the LCF fracture process was of a conventional nature, involving crack nucleation by slip band fissuring and striation formation on fracture surfaces at lower temperatures, and a trend to intergranular fracture at higher temperatures.

(2) At test temperatures above 600°C a new type of surface fracture was found having a mixed transgranular-intergranular character; the occurrence of the new fracture mode was associated with a marked decrease in crack propagation rates and increase in LCF endurance, and also an increase in the strain rate sensitivity exponent of the steel. The latter effect also resulted in reduced necking and enhanced ductility of the steel in tensile tests.
(3) The new mixed-mode LCF surface fracture was found to be the result of intergranular crack nucleation by grain boundary shear approximately normal to the specimen surface followed by transgranular growth of the ends of the crack nucleus. Grain boundary shear in the plane of the specimen surface did not occur. The reduced LCF crack propagation rates and consequent increased endurance was explained by the strain rate hardening effect at the crack tip, produced as a consequence of the increased strain rate sensitivity exponent.

(4) An existing theory of LCF crack propagation was modified in order to take strain rate hardening at the crack tip into account, and on the basis of this a "combined hardening parameter" has been proposed for the correlation of LCF data for materials exhibiting significant strain rate hardening effects.

(5) Grain boundary migration, leading to both grain enlargement and the formation of a "square" or "orthogonal" grain structure with the grain boundaries orientated at 45° to the strain direction has been observed at test temperatures above 700°C. The main features were similar to those described in published literature but there were significant differences between the present and published work on 20/25/Nb steel. A theoretical model prompted by the experimental work described here has been presented.

5.3. Chapter 4. Low-Cycle Fatigue Properties of Irradiated 20/25/Nb Steel

(1) In general, prior neutron irradiation produced a reduction in LCF endurance and tensile ductility at temperatures of 600°C and above, but the minimum in the temperature-endurance plot found in the unirradiated steel was still present in the irradiated steel. The degradation of material mechanical properties was associated with an increased tendency to intergranular fracture and increased crack propagation rates, although the mixed-mode mechanism of LCF crack nucleation remained the same.
(2) Recovery of LCF endurance and tensile ductility occurred with decreasing plastic strain amplitude in the former case and the introduction of an incremental strain deformation regime in the latter (there was also a tendency for endurance and ductility to increase with increasing strain rate).

(3) Enlarged helium bubbles were found in crack tip thin foil electron microscope specimens. A mechanism has been proposed to account for the preferential growth of helium bubbles in the crack tip zone.

(4) Irradiation embrittlement is considered to be a consequence of increased crack propagation rates. The increased propagation rate is believed to be the result of easier formation of new crack surface in the "damaged zone" ahead of the crack tip by plastic deformation and decohesion of ligaments between enlarged helium bubbles.
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Calculation of Surface Plastic Strain in Low-Cycle Fatigue Specimens.


The notation used for a rectangular section bar is shown in Fig. 1

The length under consideration (i.e. the gauge length) is denoted by \( l \), the thickness by \( h \) and the radius of curvature by \( r \).

Therefore:

\[
2\phi = \frac{l}{r}
\]

for a uniform cross-section the surface strain \( \epsilon \) is given by:

\[
\epsilon = \frac{h\phi}{l} \quad \ldots \ldots (1)
\]

Considering now a bend fatigue specimen, the centre is clamped at A and the ends deflected by knife edges, one of which is located at K (Fig. 2).

The distance from the knife edge to the centre of the gauge length is \( D \). The plastic stroke (i.e. the total deflection minus the elastic deflection) is \( S_p \).

Therefore:

\[
\tan \alpha = \frac{S_p}{D}
\]
Since \( \alpha \) is small (say less than \( 10^0 \)) the approximation \( \alpha = \frac{S_p}{D} \) may be used.

Now \( \alpha = 2 \varphi \) \( (\varphi \) is shown in Fig. 1) 

Therefore \( 2 \varphi = \frac{S_p}{D} \)

and \( \varphi = \frac{S_p}{2D} \)

thus from equation (1) we have:

\[
\Delta \epsilon_p = \frac{h.S_p}{l} \cdot \frac{1}{2D}
\]

where \( \Delta \epsilon_p \) is the plastic strain amplitude.
Stress Calculations in Cantilever Bending.


This method of determining the surface stress during bending does not require prior knowledge of the stress-strain curve for the material.

The stress is given by:

$$\sigma = \frac{2}{bh^2} \left(2M + \phi \frac{dm}{d\phi}\right)$$

where \(M\) is the bending moment and \(b\) is the width of the rectangular section bar. \(\phi\) and \(h\) have the same meanings as in Appendix 1.

The bending moment \(M\) is known; the problem is to find the term \(\phi \frac{dm}{d\phi}\). This may be found from the slope of the load-deflection curve, which is shown in Fig.1 for one half-cycle.

\(S_t\) is the total deflection, at the peak load. The bending moment and load are related by \(M = aL\) where \(a\) is a factor to be determined. Also \(\phi = bS\).

If \(D\) is the distance from the gauge length to the knife edge:

$$2\phi = \tan^{-1}\left(\frac{S}{D}\right)$$

Also:

$$\frac{dM}{d\phi} = \frac{d(aL)}{d(bS)} = \frac{d(aL)}{dS} \cdot \frac{dS}{d(bS)}$$

$$\frac{d(aL)}{dS} = \frac{d(aL)}{dL} \cdot \frac{dL}{dS}$$
Hence: 

\[
\frac{d(aL)}{d(bS)} = \frac{d(aL)}{dL} \cdot \frac{dL}{dS} \cdot \frac{dS}{d(bS)}
\]

and 

\[
\frac{d(aL)}{dL} = a
\]

and 

\[
\frac{dS}{d(bS)} = \frac{1}{b}
\]

Thus: 

\[
\frac{dM}{d\phi} = a \cdot \frac{dL}{dS}
\]

at the peak load: 

\[
\frac{dL}{dS} = \frac{L}{S_t}
\]

and so 

\[
M = \frac{LD}{2}
\]

hence the factor \(a\) is \(D/2\).

If \(\phi\) is small: 

\[
\tan 2\phi = 2\phi = S/D
\]

hence the factor \(b\) is \(1/D\).

Therefore 

\[
\phi \frac{dM}{d\phi} = \frac{a \cdot dL \cdot bS}{b \cdot dS} = \frac{a \triangle S_t}{S_t} = a \frac{\triangle L}{D} = D \frac{\triangle L}{2}
\]

Finally therefore the surface stress is given by:

\[
\sigma = \frac{2}{bh^2} \left( LD + \frac{D \triangle L}{2} \right)
\]

\[
\sigma = \frac{2D}{bh^2} \left( L + \frac{\triangle L}{2} \right)
\]
APPENDIX 3

Calculation of True spacings of Grain Boundary Helium Bubbles.

Helium bubbles in a given size range are assumed to be randomly distributed over the grain boundary. The problem is therefore similar to that of counting blood cells in a microscope sample, and the appropriate distribution function is the same as that employed in haematology, namely the Poisson Distribution. This is defined by the relation:

\[ p = \frac{e^{-m} m^x}{x!} \]  

\[ \text{in which } p \text{ is the probability of an event occurring } x \text{ times in a specified interval, and } m \text{ is the average occurrence of the event over the sum of all the intervals.} \]

If the average number of bubbles per unit area of grain boundary is \( X \), and this unit area is considered to be divided into \( n \) small squares, then on average there will be \( X/n \) bubbles per square. Therefore we have \( m = X/n \), and small \( x \) in equation (1) can be put equal to 1 since the chance of getting 2 or more bubbles in a square can be neglected when \( X/n \) is sufficiently small.

The chance of getting one bubble per square is therefore:

\[ p_1 = e^{-x/n} \frac{x}{n} \]  

\[ \text{The intersection of the plane of the foil specimen with the grain boundary can be represented as a row of the small squares; the number of small squares in the intersection (per unit area) is } \sqrt{n}. \] If the frequency of observation of bubbles is \( f \), then:

\[ f = \sqrt{n} p_1 = \sqrt{n} \frac{x}{n} e^{-x/n} \]  

\[ \text{If the observed mean spacing of the bubbles in the grain boundary is } a, \text{ (per unit length of boundary) } a = \frac{1}{f}. \]

and so

\[ a = \frac{1}{\sqrt{n}} \frac{x}{n} e^{-x/n} \]  

\[ \text{if the foil thickness is } d, \text{ then } d = \frac{1}{\sqrt{n}} \]

(taking foil thickness = length of side of small square)
Therefore: \[ a = \frac{1}{d} \frac{d}{dx} e^{-X/n} \] ...... (5)

since \( n = \frac{1}{d^2} \), eqn. (5) can be written:

\[ a = \frac{1}{d} \frac{d}{dx} e^{-Xd^2} \]

i.e.

\[ Xe^{-Xd^2} = \frac{1}{ad} \] ......(6)

if the term \( Xd^2 \) is sufficiently small:

\[ e^{-Xd^2} = 1 - Xd^2 \]

and so eqn. (6) can be written

\[ X(1 - Xd^2) = \frac{1}{ad} \] ......(7)

Taking some typical values for large bubbles of \( a = 10^{-4} \) cm and \( d = 5 \times 10^{-6} \) cm (500A) (These are not typical of the steel used in the present study) it may be shown that the term \( Xd^2 \) in eqn. (7) only amounts to about 5% of \( X \), so that for the fairly large spacings to be expected for large bubbles, (7) reduces to

\[ X = \frac{1}{ad} \]

The true mean bubble spacing \( S \) is given by \( \frac{1}{\sqrt{X}} \), and so the final equation is:

\[ S = \frac{1}{\sqrt{ad}} \]

Using the previous values for \( a \) and \( d \), an observed spacing of \( 10^{-4} \) cm in a foil 500A thick corresponds to a true mean spacing of \( \sqrt{10^{-4} \times 5 \times 10^{-6}} = 2.24 \times 10^{-5} \) cm, which is about a quarter of the observed spacing. There is therefore considerable error in taking spacings measured from foils as true spacings when the spacing is large compared with the foil thickness.
PLATE 2.17 X 20K
Nb(C,N) "KNOTTED STRINGERS"

PLATE 2.18 X 1K
PRECIPITATE-FREE ZONES

PLATE 2.19 X 8K
Nb(C,N) STRINGERS

PLATE 2.20 X 60K
INTERGRANULAR HELIUM BUBBLES

PLATE 2.21 X 1K
GENERAL PRECIPITATE DISTRIBUTION:
IRRADIATED STEEL
EFFECT OF ELASTIC STRAIN ON CARBURISATION
PLATE 2.29  X 1300
$M_{23}(C,N)_6$ - LCF SPECIMEN -
ARGON TESTED

PLATE 2.30  X 1300
INTRAGRANULAR $M_{23}(C,N)_6$ - IRRADIATED TENSILE SPECIMEN

PLATE 2.31  X 1300
CLUSTERS & STRINGERS OF $M_{23}(C,N)_6$

PLATE 2.32  X 1300
COARSENING OF $M_{23}(C,N)_6$ TOWARDS INTERIOR
PLATE 3.1  X 500
SLIP BANDS & LCF CRACKS, 400°C

PLATE 3.2  X 2000
SLIP BAND CRACKING, 400°C

PLATE 3.3  X 2K (SEM)
SLIP BANDS & LCF CRACKS, 400°C

PLATE 3.4  X 5K (SEM)
SLIP BAND CRACK FORMATION
PLATE 3.5 X 2000
30 CYCLES, 500°C. SLIP BANDS

PLATE 3.6 X 500
TESTED TO FAILURE, 500°C. SLIP BANDS & CRACKS

PLATE 3.7 X 2000
30 CYCLES, 600°C. INCIPIENT INTERGRANULAR FRACTURE

PLATE 3.8 X 2000
60 CYCLES, 600°C. SLIP BANDS & INTERGRANULAR CRACKS
PLATE 3.9  X 500
TESTED TO FAILURE, 600°C.
INTERGRANULAR CRACKING

PLATE 3.10  X 5K (SEM)
OXIDATION PITS & INTERGRANULAR CRACKS, 600°C

PLATE 3.11  X 2000
30 CYCLES, 700°C. OXIDATION PITS & INCIPIENT INTERGRANULAR CRACKS

PLATE 3.12  X 2000
60 CYCLES, 700°C. INTERGRANULAR CRACKS
PLATE 3.13  X 200
130 CYCLES, 700°C. STRAIGHT CRACKS

PLATE 3.14  X 1500
130 CYCLES, 700°C. MIXED MODE CRACK

PLATE 3.15  X 3000
130 CYCLES, 700°C. TRANSGRANULAR DEPRESSION FORMING

PLATE 3.16  X 1500
530 CYCLES, 700°C. TRANSGRANULAR FRACTURE FULLY DEVELOPED
PLATE 3.17  X 200
530 CYCLES, 700°C. DARK CONTRAST DUE TO CRACK EDGE DISPLACEMENT

PLATE 3.18  X 500
TESTED TO FAILURE, 700°C. CROSS-LINKING FRATURES

PLATE 3.19  X 500
TESTED TO FAILURE, 700°C
SUBSIDIARY FRATURES

PLATE 3.20  X 2KK(SEM)
60 CYCLES, 700°C. EMBRYO TRANSGRANULAR CRACK
PLATE 3.21  X 1400 (SEM)
45° TILT. TESTED TO FAILURE, 700°C
CRACK EDGE DISPLACEMENT

PLATE 3.22  X 3000
130 CYCLES, 700°C. INTERGRANULAR
CRACK NUCLEATION

PLATE 3.23  X 200
TESTED TO FAILURE, 700°C. CRACK
PENETRATION

PLATE 3.24  X 2000
30 CYCLES, 800°C. TRANSGRANULAR
EMBRYO CRACK
PLATE 3.25  X 2000
60 CYCLES, 800°C. MIXED MODE CRACK DEVELOPMENT

PLATE 3.26  X 500
60 CYCLES, 800°C. STRAIGHT CRACK PATTERN

PLATE 3.27  X 200
120 CYCLES, 800°C. STRAIGHT CRACK PATTERN

PLATE 3.28  X 1500
120 CYCLES, 800°C. DISPLACEMENT OF CRACK EDGES
PLATE 3.35 X 500
PLASTIC STRAIN AMP. = 3%, 600°C
INTERGRANULAR FRACTURE

PLATE 3.36 X 500
PLASTIC STRAIN AMP. = 3%, 700°C
INTERGRANULAR FRACTURE

PLATE 3.37 X 500
PLASTIC STRAIN AMP. = 0.8%, 600°C
MIXED MODE & INTERGRANULAR CRACKS

PLATE 3.38 X 500
PLASTIC STRAIN AMP. = 0.8%, 700°C
MIXED MODE FRACTURE
PLATE 3.39 X 500
STRAIN RATE 6 x 10^{-4}s^{-1}, 700°C
INTERGRANULAR FRACTURE

PLATE 3.40 X 500
STRAIN RATE 1.2 x 10^{-3}s^{-1}, 800°C
MIXED MODE & INTERGRANULAR CRACKS

PLATE 3.41 X 1K (SEM, TILT=0°)
INTERGRANULAR FRACTURE

PLATE 3.42 X 2K (SEM, TILT=0°)
FATIGUE STRIATIONS

PLATE 3.43 X 2K (SEM, TILT=0°)
FATIGUE STRIATIONS
PLATE 3.44  X 500
500°C. FINE, BRANCHED CRACKS

PLATE 3.45  X 1K (SEM, TILT=0°)
600°C. INTERGRANULAR FRACTURE

PLATE 3.46  X 500
600°C. FINE, BRANCHED CRACKS

PLATE 3.47  X 1K (SEM, TILT=0°)
700°C. PITTED FRACTURE SURFACE
PLATE 3.48 X 200 (SEM, TILT=0°)
700°C. CELLULAR STRUCTURE

PLATE 3.49 X 500
700°C. BLUNT, COARSE CRACKS

PLATE 3.50 X 200 (SEM, TILT=0°)
800°C. FRACTURE SURFACE

PLATE 3.51 X 1K (SEM, TILT=0°)
800°C. PITTED FRACTURE SURFACE
PLATE 3.52 X 150
800°C. BLUNT, COARSE CRACKS

PLATE 3.53 X 1500
800°C. POSSIBLE TRANSGRANULAR CRACK

PLATE 3.54 X 500
800°C. BLUNT CRACKS

PLATE 3.55 X 500
STRAIN RATE 2 X 10^-5 s^-1, 800°C
INTERGRANULAR CRACKS
PLATE 3.56  X 2000
STRAIN RATE 2 x 10^{-5} s^{-1}, 800°C
INTRAGRANULAR PROTRUSION

PLATE 3.57  X 1500
STRAIN RATE 2 x 10^{-5} s^{-1}, 800°C
INTRAGRANULAR PROTRUSION

PLATE 3.58  X 500
GRAIN SIZE 30um. INTRAGRANULAR
PROTRUSION
PLATE 3.59 X 200
500C. NECKING DOWN AT
FRACTURE

PLATE 3.60 X 200
600C. NECKING DOWN AT
FRACTURE

PLATE 3.61 X 200
700C. NECKING DOWN AT
FRACTURE

PLATE 3.62 X 200
800C. NECKING DOWN AT
FRACTURE
PLATE 3.63 X 1K (SEM, TILT=0°)  
600°C. DUCTILE FRACTURE

PLATE 3.64 X 2K (SEM, TILT=0°)  
600°C. OXIDISED FRACTURE SURFACE

PLATE 3.65 X 630  
GRAIN BOUNDARY ORIENTATION  
AT 45° TO STRAIN DIRECTION

PLATE 3.66 X 200  
VARIATION OF GRAIN STRUCTURE  
THROUGH LCF SPECIMEN

PLATE 3.67 X 200  
"SQUARE GRAINS"  
ADJACENT TO  
FRACTURE
PLATE 3.68 X 200
VARIATION OF GRAIN STRUCTURE
THROUGH LCF SPECIMEN

PLATE 3.69 X 200
VARIATION OF GRAIN STRUCTURE
THROUGH LCF SPECIMEN

PLATE 3.70 X 200
VARIATION OF GRAIN STRUCTURE
THROUGH LCF SPECIMEN

PLATE 3.71 X 200
VARIATION OF GRAIN STRUCTURE
THROUGH LCF SPECIMEN
PLATE 4.1  X 500
500°C. SLIP BANDS AND
INTERGRANULAR FRACTURE

PLATE 4.2  X 2K (SEM, TILT=0°)
500°C. FATIGUE STRIATIONS

PLATE 4.3  X 1K (SEM, TILT=0°)
600°C. INTERGRANULAR FRACTURE

PLATE 4.4  X 500
700°C. MIXED MODE FRACTURE
PLATE 4.5 X 2000
700°C, MIXED MODE FRACTURE

PLATE 4.6 X 100
700°C, FINE, BRANCHED CRACKING

PLATE 4.7 X 1K (SEM, TILT=0°)
700°C, INTERGRANULAR FRACTURE SURFACE

PLATE 4.8 X 40K
700°C, INTERGRANULAR HELIUM BUBBLES

PLATE 4.7 X 1K (SEM, TILT=0°)
700°C, INTERGRANULAR FRACTURE SURFACE

PLATE 4.8 X 40K
700°C, INTERGRANULAR HELIUM BUBBLES
PLATE 4.9  X 40K
700°C. INTERGRANULAR HELIUM BUBBLES

PLATE 4.10  X 40K
700°C. INTERGRANULAR HELIUM BUBBLES

PLATE 4.11  X 100
800°C. SMALL SURFACE CRACKS

PLATE 4.12  X 1K (SEM, TILT=0°)
800°C. INTERGRANULAR CRACK BRANCHING
PLATE 4.13 X 2K (SEM, TILT=0°)
800°C. INTERGRANULAR CRACK BRANCHING

PLATE 4.14 X 40K
800°C. INTERGRANULAR HELIUM BUBBLES

PLATE 4.15 X 40K
800°C. INTERGRANULAR HELIUM BUBBLES

PLATE 4.16 X 1K (SEM, TILT=0°)
600°C TENSILE TEST. DUCTILE FRACTURE
PLATE 4.17 X 2K (SEM,TILT=0°)  
600°C TENSILE TEST. DUCTILE FRACTURE

PLATE 4.18 X 1K (SEM,TILT=0°)  
700°C TENSILE TEST. INTERGRANULAR FRACTURE

PLATE 4.19 X 500 (SEM,TILT=0°)  
800°C TENSILE TEST. INTERGRANULAR CRACKING

PLATE 4.20 X 1K (SEM,TILT=0°)  
800°C TENSILE TEST. INTERGRANULAR FRACTURE SURFACE