IMPACT ATTRITION OF PARTICULATE SOLIDS

by

Zhichao Zhang

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ABSTRACT

Attrition is an unwanted particle size breakdown which occurs widely in processing and handling of particulate solids. It gives rise to dust formation and has harmful effects on product quality and reliable operation of process equipment.

A mechanistic model of impact attrition of particulate solids, having a semi-brittle failure mode, has been developed in order to provide a base to estimate the rate of attrition in process equipment and a capability to produce materials with better attrition resistance.

Observations of the impact damage by high speed photography show that attrition by semi-brittle failure is governed by the chipping process, where material removal occurs from the corners and edges of the particles in the form of platelets. Detailed examination by confocal laser scanning microscopy as well as scanning electron microscopy shows that the platelets are produced by propagation of subsurface lateral cracks. These cracks form during the unloading stage as a result of residual tensile stresses, which are imposed by the relaxation of deformed material around the plastic deformation zone.

Here the analysis of impact attrition is based on the fracture mechanics of subsurface lateral cracks. In this analysis, the volume of material bounded by these cracks and the free surface is considered to be readily detached from the impact site to form the attrition debris. This volume can be estimated from the depth and length of the lateral cracks, which in turn are dependent of the material properties and impact velocity. As a result of the analysis, the fractional loss of material from a mother particle due to an impact is derived as:

\[ \xi = \alpha \eta = \frac{\alpha \rho v^2 l H}{K_c^2 \phi^2} \]

where \( \alpha \) is the proportionality factor, and the dimensionless group \( \eta \) is regarded as the attrition propensity parameter. This parameter includes all the relevant material properties, \( i.e. \) density \( \rho \), particle linear dimension \( l \), hardness \( H \), critical stress intensity factor \( K_c \) and constraint factor \( \phi \), and the impact velocity \( v \).
The above model has been verified by experimental work. Single particle impact testing, which involves striking of a particle against a rigid target at a normal direction, has been used for the determination of attrition rate of particulate solids. Model materials chosen for this study are ionic single crystals of MgO, NaCl and KCl, whose structure and physical properties have been extensively addressed in the past. Both melt-grown crystals and commercially produced solution-grown crystals (i.e. salt crystals) have been used, and attrition of the latter material has important practical implication in process industry. A series of tests on these materials has been carried out to verify the model predictions, in particular for the dependence of attrition rate on material mechanical properties, particle size and impact velocity. It is shown that the trend of the data follows closely the theoretical predictions.

There is an additional complexity in the experimental data arising from the effect of number of impacts. The number of impacts influences the rate of attrition to varying degrees, depending on the impact velocity and particle size. This is considered to be due to the work-hardening effect, where on each impact the hardness increases. Further refinement of the model by taking account of the effect of number of impacts for work-hardening materials is therefore required.
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NOTATION

\( a \) : radius of impression \( \mu m \)
\( A \) : area of cross section of the specimen defined in Fig. 4.9 \( m^2 \)
\( c \) : crack length \( \mu m \)
\( d_{cb} \) : critical particle size below which no breakage occurs \( \mu m \)
\( d_{cl} \) : critical particle size for the formation of lateral cracks \( \mu m \)
\( d_1 \) : diagonal length of indent \( \mu m \)
\( D \) : diameter of a spherical indenter or projectile \( m \)
\( E \) : Young's modulus \( Pa \)
\( E_{100} \) : Young's modulus of <100> crystallographic direction \( Pa \)
\( E_{hkl} \) : Young's modulus of <hkl> crystallographic direction \( Pa \)
\( F \) : force \( N \)
\( h \) : depth at which subsurface lateral cracks initiate \( \mu m \)
\( H \) : hardness \( Pa \)
\( H_p \) : hardness of a particle \( Pa \)
\( H_t \) : hardness of a target \( Pa \)
\( H_v \) : Vickers hardness \( Pa \)
\( G_{hkl} \) : shear modulus of <hkl> crystallographic direction \( Pa \)
\( k \) : experimental shear yield stress \( Pa \)
\( K_c \) : critical stress intensity factor \( N m^{-3/2} \)
\( K_{tc} \) : toughness of a target \( N m^{-3/2} \)
\( l \) : cube dimension \( m \)
\( I \) : mean particle size (see equation 5.7) \( m \)
\( L \) : length of the specimen defined in Fig. 4.9 \( m \)
\( m \) : number of impacts
\( M \) : mass \( kg \)
\( M(N) \) : mass of particles after \( N \) impacts \( kg \)
\( N \) : number of impacts
\( n \) : number of impacts
\( n_p \) : total number of particles
\( p \) : contact pressure \( Pa \)
\( r_p \) : plastic zone size \( m \)
\( r_c \) : critical size of indentation \( m \)
\( R \) : radius of a spherical indenter \( m \)
\( s \) : specific attrition rate
\( S_i \) : specific breakage rate (see equation 1.1)
\( t \) : time \( s \)
\( t_{ce} \) : total time for elastic contact \( s \)
\( t_p \) : peak contact time \( s \)
\( t_{pe} \) : elastic peak contact time \( s \)
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<td>elastic-plastic loading time</td>
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<td>$v$</td>
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<tr>
<td>$V$</td>
<td>volume removed by the formation of lateral cracks</td>
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<td>$w$</td>
<td>Weber number</td>
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<td>$Y$</td>
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<tr>
<td>$Y_R$</td>
<td>representative flow stress</td>
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<td>$z$</td>
<td>component of a Burgers vector</td>
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Greek

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</tr>
<tr>
<td>$\sigma_y$</td>
<td>lower yield point stress in a uniaxial test</td>
</tr>
<tr>
<td>$\zeta$</td>
<td>constant (see equation 7.9)</td>
</tr>
<tr>
<td>$\tau$</td>
<td>shear stress</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
</tr>
<tr>
<td>--------</td>
<td>---------------------------------------------------------------------------</td>
</tr>
<tr>
<td>$\tau_0$</td>
<td>critical resolved shear stress</td>
</tr>
<tr>
<td>$\phi$</td>
<td>constraint factor</td>
</tr>
<tr>
<td>$\chi$</td>
<td>power index (see equation 7.9)</td>
</tr>
<tr>
<td>$\psi$</td>
<td>angle defined in Fig. 4.2</td>
</tr>
<tr>
<td>$\Omega$</td>
<td>proportionality constant (see equation 4.15)</td>
</tr>
</tbody>
</table>
CHAPTER 1

INTRODUCTION

1.1 Background

Attrition is an undesirable particle size breakdown which widely occurs during processing and handling of particulate solids in chemical, agricultural and allied industries. The areas in which attrition is commonly experienced have been reviewed by Bemrose and Bridgwater (1987), and BMHB (1987 and 1990). In process equipment, for example, attrition occurs readily in fluidised beds, cyclones, centrifuges, stirred vessels, pneumatic and hydraulic conveyors.

Attrition has deleterious effects on the product quality and on the reliable operation of process equipment because of changes in particle properties such as particle size distribution, shape and surface area. In fluidised bed systems, for instance, attrition produces fine debris which are then elutriated. In addition to loss of fines, the fluidisation conditions may alter significantly. Furthermore the dust can generally cause serious problems such as health hazards, environment pollution, and dust explosion, e.g. in flour milling and pharmaceutical industry. Prevention efforts such as using filtration devices to collect dust are clearly needed in such circumstances to ensure safety. In addition to the above effects, the wear of containment systems may occur in some situations where the walls of the container suffer a severe erosive and/or abrasive damage because of the collisions of the particles colliding with sliding along the walls. In extreme cases, this can lead to disruption of the operation of the process. However, in relatively uncommon cases, attrition can be beneficial, e.g. for the removal of a particle surface layer which has already been subjected to some reaction. Removal of sulphur dioxide (SO₂), produced by combustion of coal containing sulphur, from gas streams by reaction with lime stone is a good example. This is very important in the power generation industry. Here the reaction blocks the pores in the limestone making the material inside the particle inaccessible for reaction. In this case, attrition is beneficial for efficient use of the limestone (Fieldes et al., 1979).
Overall, the problems associated with particle attrition have significant cost implications in process industry. So far the problem has been addressed in an empirical way. This approach has however proved unattractive because there is a wide variety of material types and properties for which no general conclusions can be drawn on their attrition behaviour. There is therefore a need for developing a more rational approach to the analysis of attrition of particulate solids. This will allow engineers to estimate the level of attrition in process equipment and produce materials with better attrition resistance.

In a number of items of process equipment such as cyclones, centrifuges, conveyors and in the jet region of fluidised beds, attrition results from the particle-particle or particle-wall collisions. Therefore, the study of attrition under dynamic conditions is of great interest in process industry. In this thesis, the mechanical breakdown of single particles, resulting from impact on a rigid target in a velocity range which corresponds to that prevailing in process equipment, is addressed. It is hoped that by understanding the underlying mechanisms of attrition, the important features of a complex system that are responsible for causing attrition are identified.

1.2 Literature Review

Little work has been published on the fundamental mechanisms of particle attrition although a number of empirical models are available. In the following, a literature review is presented with special reference to the models of attrition rate and experimental techniques for the study of attrition.

1.2.1 Models of attrition rate

Various models of the attrition rate are briefly reviewed with a view to considering their relevance to impact attrition. Some of these models were originally developed for comminution processes where the breakage of particles was intentional and desired (Lynch, 1977; van Brakel, 1981; Prasher, 1987). However, they have been adopted for attrition processes because of the similarity of the two processes (e.g. Neil, 1986; Vervoorn and Austin, 1990).

(1) Energy based models

The earliest models considering the required energy for particle breakdown are due to Rittinger (1867), Kick (1885) and Bond (1952). These models have been developed
for comminution processes and are commonly referred to as energy-size reduction relationships. Rittinger's model is based on the energy consumed being directly proportional to the area of new surfaces formed. Kick's model is based on the energy consumption being proportional to the volume of the comminuted product. Bond's model is based on the assumption that the required energy for grinding is directly proportional to the length of new cracks formed. A generalised form of the energy-size reduction relationship is due to Walker et al. (1937), where the energy required to reduce the size of particles is inversely proportional to the particle size raised to some power. Hukki (1961) has noted that the power index proposed by Walker et al. is not a constant but a variable dependent on the representative particle size.

The energy-size reduction relationships are of interest to workers in comminution research (Voller, 1983; Kanda et al., 1986; Austin, 1987; Kapur and Fuerstenau 1987; Weichert, 1988; Fuerstenau et al., 1993, etc.) because of their importance in estimating the energy requirements and improving the efficiency of grinding and crushing processes. The energy-based approaches in general have a simple physical concept, and in principle it should be possible to employ them to derive the rate of attrition in a simple size reduction process provided that the fraction of energy that is actually used for attrition can be reliably estimated. Efforts have been made recently by various workers (Ray et al., 1987; Shamlou et al., 1990; and Werther and Xi, 1993) to study particle attrition in a fluidised bed. In the analysis by Ray et al., the effective breakage energy has been related to the kinetic energy received by the particles by a proportionality factor which represents the efficiency of energy transformation from the gas to the particles. Their analysis shows that the total attrition rate in the bed region is linearly related to the fluidising velocity. In the analysis by Shamlou et al. and Werther and Xi, the rate of attrition in the jetting region is assumed to be directly proportional to the rate of kinetic energy of fluidising gas at its entry to the bed. This implies that the attrition rate should be proportional to the gas velocity raised to the power of 3. However, it has been found that this is not universally applicable, and it depends on particle size and properties (Ghadiri et al., 1994). The application of these energy-size reduction laws to attrition processes, therefore, requires some modifications and further extensive work. This is due to the reasons outlined below.

Firstly, the energy-based laws were obtained from the study of fracture behaviour of brittle solids where the plastic deformation was not significant. Therefore, for ductile or semi-brittle materials, the analysis based on the simple energy-size reduction needs to be modified. Secondly, these energy laws are expressed in the form of a
differential decrease in particle size. However, it is not possible to produce a differential decrease in particle size because size reduction processes generate a full size distribution for which it is difficult to calculate a differential change. Austin (1973) suggested that the mean value of the particle size distribution could be used to calculate the differential decrease in particle size. This however oversimplifies the attrition process. Furthermore, the main interest in the attrition process is the rate of attrition and the size distribution of the product, rather than the input energy, and therefore these models cannot be used directly to describe the attrition process.

(2) Abrasion based models
The model of Gwyn (1969) was originally developed to describe the attrition of silica-alumina catalysts in fluidised beds, but it has since then been applied to a number of other processes, such as attrition in shear cells (Neil, 1986). The basis of this model is surface abrasion rather than fragmentation. Paramanathan and Bridgwater (1983a, b) proposed another abrasion model for their work on attrition in shear cells based on the assumption that the rate of surface grinding is proportional to particle radius with a certain power index. Neil (1986) subsequently showed that the integrated form of the model of Paramanathan and Bridgwater reduces to the Gwyn model.

The abrasion-based models were developed on the assumption that particle attrition was caused by the action of particles sliding over each other rather than inter-particle collisions. Therefore, these models may not be applicable to the impact attrition of particulate solids. Furthermore, these models are conceptual rather than mechanistic and therefore can not take into account the actual mechanism of particle damage. This is further addressed in Chapter 2.

(3) Continuous grinding models
Kelsall et al. (1967) proposed a model based on the concept of a first-order rate process and the definition of specific breakage rate:

$$\frac{-dM_i}{dt} = S_i M_i \quad (1.1)$$

where $M_i$ is the total mass of particles of size $d_i$, which remain unbroken after time $t$, and $S_i$ is the specific breakage rate for particle size $d_i$. $S_i$ depends on both the process conditions and the particle properties, and it is assumed to be a constant with time. The size reduction of a variety of materials in comminution equipment follows closely the first-order rate process (Austin, 1971).
Equation 1.1 provides a useful method to assess the rate of attrition for different processes although it is not mechanistic and therefore it does not have a predictive capability. For instance, it has been used to describe the attrition rate in fluidised beds (Kono, 1981; Patel, 1986) and in shear cells (Neil, 1986). It has also been applied to study the repeated impact attrition of alumina extrudates by Vervoorn and Austin (1990). The validity of the first-order process for impact attrition is assessed in Chapter 5.

(4) Population balance model

Broadbent and Callcott (1956) proposed a comprehensive model of size reduction processes based on the concept of breakage and selection functions. In a given apparatus operating on a continuous steady-state basis, particle breakage is considered to be the result of two processes: a fraction of particles breaks down, depending on the type of equipment and mechanical conditions, which is defined by the selection function. This fraction is then reduced to a size whose relationship with the feed size is defined by the breakage function. These two concepts are then incorporated in a population balance model to describe the rate of formation of the product.

The selection function represents the probability of particles of a given size would break, and the breakage function represents the size distribution of broken particles. Therefore, the breakage and selection functions describe the particle behaviour in a given process equipment, and depend on the physical and mechanical properties of the particles and details of the application of load. It should in principle be possible to describe these two functions by developing models of particle motion and breakdown in a given device, thus providing a powerful method for characterizing the performance of size reduction equipment. Extension of the application of the population balance model to the process of attrition however requires some modification of the concept depending on the modes of breakdown. For example, considering single particle impact attrition, the selection function is essentially unity. However, the damage is so minute that particle size does not change substantially. Consequently, it is more appropriate to define a specific attrition rate which describes the material loss from the mother particles as a result of an impact. In this case, the population model of Broadbent and Callcott then becomes equivalent to the first-order rate model of Kelsall et al. (1967). The interest in this work is on the single particle behaviour, and therefore the development of population balance models for attrition is not pursued.
1.2.2 Experimental techniques for the study of attrition

A number of experimental methods have been applied to investigate attrition processes, to assess the rate of attrition, and to verify the existing models of attrition rate. The test methods can broadly be classified in two categories, i.e. multi-particle and single particle tests.

(1) Multi-particle tests
Typical multi-particle attrition tests are those such as grinding mills, shear cells and fluidised beds which have been mentioned in the last section. The small scale grinding mill tests (Hardgrove, 1932; Bond, 1961) have been applied to quantify the friability of granular materials. The attrition propensity of materials is described by the grindability index, e.g. Hardgrove index, which is based on the quantity of new surface created during a specific grinding process on a closely sized sample of materials. The extent of new surfaces produced is calculated using Rittinger's model after sieving the grinding products, and the value of Hardgrove index is presented as a percentage of the new created surfaces by comparing with that of a standard coal sample. Hardgrove index only describes a relative propensity of attrition and, therefore, its predictability is limited.

Shear cells have been used to study attrition resulting from bulk failure of particles as they are subjected to sliding in a densely packed bed (Paramanathan and Bridgwater, 1983a,b; Neil, 1986; Ouwerkerk, 1991; Kenter, 1992). These techniques are particularly useful for the study of the behaviour of granular materials in processes where the particles undergo considerable shear stresses. However, difficulties in decoupling the effects of particles interactions restrict their contribution to the understanding of attrition mechanisms involved in the processes.

Fluidised bed test is the most common technique for the study of attrition of fine powders because this type of test is directly applicable to processes involving fluidisation such as Fluid Catalytic Cracking (FCC) units (Forsythe and Hertwig, 1949; Gwyn, 1969). Recently, further research has been undertaken in this field (see Patel, 1986; Ray et al., 1987a,b; Shamlou et al., 1990; Ghadiri et al., 1992a,b; Veesler et al., 1993; Wolff et al., 1993), where important factors influencing attrition in fluidised beds, such as the effects of superficial velocity and distributor orifice velocity, have been investigated.
Although multi-particle tests are closer to real processes and are statistically more representative in nature, they are too complex for analysis and the models developed for them are empirical. These techniques are successful in assessing the relative attritability of a particular material in a specified testing device, but fail to correlate the results obtained in different systems, and hence can not provide a sound understanding of the mechanism of attrition (Bemrose and Bridgwater, 1987). This is mainly due to two reasons. Firstly, in the multi-particle tests no account is taken of fundamental physical and mechanical properties of the material which influence attrition. Secondly, the mechanics of particle interaction in both the test device and the real process is not sufficiently well understood to maintain dynamic similarity.

(2) Single particle tests

Single particle tests, in contrast, allow well defined and controlled loading to be applied to a single particle, and therefore enhance the understanding of particle breakdown. They are however difficult to be applied to the prediction of attrition in an actual process. A number of techniques for single particle testing have been developed including: (i) quasi-static indentation (Badrick and Puttick, 1986; Puttick and Badrick, 1987; Arteaga et al., 1992 and 1993), (ii) compression between two platens (Arbiter et al., 1969; Puttick and Badrick, 1987; Shipway and Hutchings, 1993a,b) and (iii) impact on a target (Vervoorn, 1986; Ghadiri and Yuregir, 1987; Yuregir et al., 1987). Indentation has been used as a tool for modelling the conditions of local contact such as that occurring during attrition (Puttick and Badrick, 1987). By applying nano-indentation techniques, Arteaga et al. (1992 and 1993) have recently demonstrated the possibility of assessing potential attrition of particulate solids by measuring the surface hardness of the particles. Uniaxial compression is relevant to crushing of bulk particles where the failure is dominated by fragmentation (Puttick and Badrick, 1987). Single particle impact test involves striking of a particle against a rigid flat target at a required velocity, which is often achieved by the use of compressed air (Yuregir et al., 1987; Scarlett and Mahesh, 1992). With a single particle impact test, the failure of each particle can be characterized individually at well defined and controlled conditions. These tests may be related to processes where attrition occurs by high velocity inter-particle and particle-wall impacts such as in cyclones, centrifuges, jet region of fluidised beds and pneumatic conveyors.

In this work, single particle impact test is chosen to study impact attrition of particulate solids. The main advantage of this technique is that it avoids the complexity of multi-particle interactions and is hence more amenable to rigorous analysis. In this approach, the fracture mechanics can be used to provide a basis for
the characterization of particle breakage. The results obtained in such tests are relevant to processes involving high velocity particle impact such as in cyclones, centrifuges, jet region of fluidised beds and pneumatic conveyors. A complete analysis of attrition in an item of process equipment may then be carried out by combining the findings of the single particle tests with the models describing motion of fluid and particles within the system, as recently shown by Ghadiri et al. (1992b and 1994) for the analysis of attrition in the jetting region of fluidised beds.

1.3 Objectives of the Present Study and Structure of the Thesis

The brief literature review presented in Section 1.2 outlined the techniques for measuring the attrition propensity of particles and described the models of attrition rate. However, none of the existing models are mechanistic and therefore cannot provide an insight into the attrition processes. There is also a lack of understanding of the influence of material properties and loading conditions on the attrition rate. In this work, a new approach to the analysis of impact attrition of particulate solids is developed by the application of fracture mechanics. The breakdown of particulate solids whether in attrition or in comminution processes is brought about by the propagation of cracks on the application of stresses. The application of fracture mechanics allows the conditions of the breakdown of particulate solids to be determined on the basis of energy requirement, i.e. the energy required for an increase in the surface area due to fracture is provided by the elastic strain energy stored in the particles. This approach can therefore provide a fundamental insight into the mechanisms involved in the process of the breakdown of particulate solids.

The objectives of the present study are to investigate the mechanisms of impact attrition, and to identify key parameters that influence the breakdown and their limiting conditions. Impact damage is to be investigated experimentally, with the objective of characterizing the cracks that determine the extent of material removal caused by impact attrition. A predictive model of impact attrition of particulate solids based on fracture mechanics is to be developed, which relates the rate of attrition to the material properties and to the impact conditions. The rate of attrition is to be determined by single particle impact tests, and the results are to be compared with model predictions. The layout of this thesis is described below.

Chapter 2 The mechanism of impact attrition of particulate solids is described first. Then a mechanistic model of impact attrition is developed based on indentation fracture mechanics of subsurface lateral cracks. As a result, a dimensionless attrition
propensity parameter is derived that relates the attrition propensity of particulate solids to the mechanical and physical properties and impact conditions.

Chapter 3 The characteristics of impact damage are studied by the use of techniques such as high speed photography, scanning electron microscopy and confocal laser scanning microscopy. The experimental results are then compared with the theoretical predictions given in Chapter 2.

Chapter 4 This chapter is concerned with a detailed study of material properties, e.g. measurement of indentation hardness and yield stress in order to evaluate the attrition propensity parameter for different materials.

Chapter 5 The experimental results of single particle impact tests are presented, in particular for the dependence of attrition rate on mechanical properties, impact velocity and particle size. The influence of the hardness of target materials on the attrition rate is also addressed here.

Chapter 6 The application of the model of impact attrition, developed in Chapter 2, to the commercial materials is made here, with special emphasis on the effect of particle size on the attrition rate.

Chapter 7 A general discussion of the model developed in this work is given here.

Chapter 8 General conclusions drawn from this work are summarized here.

Chapter 9 Suggestions for further work are described here.
2.1 Introduction

Characterization of the attrition behaviour of particulate solids on a fundamental level requires a detailed knowledge of material properties and modes of stress application to particles. It is necessary to classify materials in terms of their failure mode, i.e. brittle, semi-brittle or ductile, and to consider the mode of stress application, e.g. local loading as in indentation, distributed loading as in compression of flat faces between two platens or high strain rate loading as in impact. This is because crack morphology, and hence the characteristics of the attrition products vary greatly in these cases, and depend on the interaction between the material properties and the process of application of load.

With reference to Fig. 2.1, it is well-known that the strain rate influences the hardness and toughness which in turn can influence the crack morphology (see e.g. Evans (1979) and Chaudhri et al. (1981)). Moreover, the stress pattern in a particle depends on the mode of loading, e.g. local loading and distributed loading can have two different patterns of tensile stresses and hence different fracture morphology. Puttick and Badrick (1987) pointed out that the failure under uniaxial compression has a particular relevance to the fragmentation process of particulate solids, and this is further discussed in Chapter 4. On the other hand, the failure induced by indentation can cause both fragmentation and chipping depending on the crack morphology. It is shown in this chapter that impact attrition by the chipping process can be modelled based on indentation fracture mechanics. In addition, the geometry of the particle can also influence the mechanical breakdown. The geometric effects are reflected in particle size and shape, which in turn determine the size of the contact area. If large particles of a material fail by brittle mode, it is likely that reducing the size can switch the mode of failure to the semi-brittle and eventually to the ductile failure. The characteristics of the three modes of failure are addressed below.
2.1.1 Modes of failure

When the surface of a specimen is loaded by an indenter (as in quasi-static indentation) or by a projectile (as in high strain rate impact), local deformation takes place. Depending on the deformation form, i.e. elastic and/or plastic, the failure mode can be broadly classified as brittle, semi-brittle and ductile failure. Brittle failure is caused by fracture with little or no plastic deformation, where the damage zone is under elastic response regime. If plastic deformation precedes the fracture, this is referred to as elastic-plastic response, hence the term semi-brittle. In contrast, ductile failure is dominated by extensive plastic flow which is responsible for the rupture of the material. Examples of crack formation in brittle and semi-brittle failure are illustrated in Fig. 2.2.
2.1.1.1 Brittle failure

For brittle failure, there are two basic types of fracture, *i.e.* Hertzian ring cracks and circumferential cracks. Hertzian ring crack is probably the best-known type of crack associated with indentation (Lawn and Wilshaw, 1975a). This crack is generally produced by a blunt indenter (*e.g.* a spherical indenter) in a 'brittle solid' such as soda-lime glass. The crack is initiated at the contact boundary where the radial stresses are tensile, and then propagates down into the interior of the material on the loading phase. This is known as Hertzian cone crack (see Fig. 2.2a). A similar type of crack is observed in the surface damage imparted by impact of a highly deformable projectile (Evans and Wilshaw, 1977). The crack forms outside a central undamaged zone. The length and density of the cracks quickly increase to a maximum value as the radius increases and then decrease continuously to zero at the outer edge of the damage zone (see Fig. 2.2b). This is known as circumferential crack. Surface chipping often accompanies circumferential cracks (Evans, 1979).
Brittle failure has been studied very extensively in the past, with a substantial body of work reported in the literature (e.g. see Lawn and Wilshaw, 1975a; Mouginot and Maugis, 1985). The theoretical description of the ring crack initiation and cone crack propagation is due to the Hertzian stress field. A detailed analysis of the elastic stress fields for indentation beneath flat and spherical punches has been given by Mouginot and Maugis (1985). Lawn and Wilshaw (1975a) have evaluated the energy release rate as a function of crack length based on the Griffith's theory (1920), and have outlined the criterion for crack propagation under brittle failure mode. However, the application of this criterion to the breakdown of particulate solids has encountered difficulties due to the problems associated with the characterization of the position and size of pre-existing flaws in the materials.

Recently, Shipway and Hutchings (1993a,b) have presented a theoretical and experimental study of the fracture behaviour of brittle spheres under uniaxial compression and impact, based on the analytical solutions of elastic fields given by Dean et al. (1952) and Hiramatsu and Oka (1966). Their theoretical analysis shows that the stress distributions in elastic spheres are broadly similar under both quasi-static and impact conditions, thus indicating the insensitivity of brittle failure to the strain rate. Significant tensile stresses exist inside the sphere on the axis of the applied load and on the surface of the sphere. High shear stresses are also present on the axis within the sphere. The experimental and theoretical results both show that the failure is very sensitive to contact deformation. Large contact deformations, corresponding to low values of contact stiffness, are more likely to propagate a crack on the surface. On the other hand, high values of contact stiffness produce internal tensile stresses which are much larger than the surface hoop tensile stresses, hence promoting internal failure and the creation of large fragments. Again, a more detailed analysis of breakage in attrition and comminution processes for materials with a brittle failure mode requires a knowledge of the size and spatial distribution of flaws. The characterization of these parameters is difficult at present, hence making a deterministic analysis of brittle failure very difficult at present.
2.1.1.2 Semi-brittle failure

Semi-brittle failure is initiated once a critical condition is achieved. This critical condition depends on the material properties such as hardness and toughness, and is further addressed in Section 2.3.1. When the applied load or deformation size exceeds the corresponding value for the critical condition, crack propagation ensues.

Three basic types of crack have been observed in semi-brittle fracture, i.e. radial, median and lateral cracks as shown in Fig. 2.2 (Evans and Wilshaw, 1976). Radial and median cracks usually initiate during loading as a result of extensive plastic flow. The radial crack forms first from the periphery of the crater produced by indentation and spreads radially outwards. At a higher level of load, the median crack forms on a plane of symmetry containing the contact axis. As the load is further increased, median cracks may merge with radial cracks to form the half-penny shape cracks as shown in Fig. 2.2e. Lateral cracks are commonly observed to initiate near the boundary of the plastic deformation zone during the unloading stage. These cracks then propagate approximately parallel to the surface.

The type and sequence of the formation of cracks are not similar in all materials and depend on the hardness, isotropy of the material, the geometry of the indenter and the loading conditions (Lawn and Wilshaw, 1975a; Evans and Wilshaw, 1976; Cook and Pharr, 1990; Rowcliffe, 1992). For example, for soda lime silica glass under quasi-static indentation no cracks are observed on loading (Cook and Pharr, 1990). During the unloading stage radial cracks form first, and later, almost at the end of this stage, lateral cracks initiate and grow rapidly. Of the various types of crack that exist, the subsurface lateral cracks are primarily responsible for material removal (Evans and Wilshaw, 1976; Marshall et al., 1982, etc.). The mechanism of formation of subsurface lateral cracks is discussed in Section 2.2.2.

Over the last two decades, substantial effort has been made to gain an understanding of the semi-brittle failure mode, particularly for the failure that is induced by quasi-static indentation. This has led to the development of Indentation Fracture Mechanics (IFM) which has been extended to the analysis of impact fracture (Evans et al., 1978; Rowcliffe, 1992; Ritter, 1992). The application of IFM to the analysis of impact attrition of particulate solids forms the main body of the theoretical work in this thesis. This is further described in Section 2.3.1.
2.1.1.3 Ductile failure

This failure mode normally prevails in metals and polymers. For instance, as the surface of a ductile metal (e.g. mild steel) is impacted by a hard particle (e.g. silicon carbide), the surface is subjected to wear damage. This process is dominated by plastic deformation. The geometry of the deformation is controlled by a number of factors such as particle shape, orientation, impact angle and impact velocity. Hutchings (1992a) identifies two modes of plastic deformation under impact conditions, i.e. ploughing and cutting as shown in Fig. 2.3. The distinction between these two modes lies in the fact that, in the case of ploughing, the material flows to the side and front of impacting particle, whereas in cutting it flows up to the front face of the particle. In the case of cutting, the deformed material may appear as a lip (type I), or it may be cut off as a chip (type II) as shown in Fig. 2.3c. In ductile failure, cracking does not readily occur, but instead the plastic rupture operates. Since only semi-brittle materials are of concern here, ductile failure is not considered further.

![Fig. 2.3 Ductile failure by impact of a hard particle on a metal surface. The impact direction is from left to right. (a) Ploughing deformation by a sphere; (b) Type I cutting by an angular particle, rotating forwards during impact; (c) Type II cutting by an angular particle, rotating backwards during impact (After Hutchings, 1992a).](image-url)
2.1.2 Selection of test materials for the present study

It is clear from the above short review that the mode of failure depends strongly on the material properties. A large number of particulate solids fail by semi-brittle mode. This is because the small size of particles or the presence of sharp corners and edges and asperities can bring about plastic deformation under local loading, where the applied stresses can easily exceed the yield stress due to the small radius of curvature at the contact point. For this reason, materials failing by the semi-brittle failure mode are chosen for the investigation.

Melt-grown single crystals of NaCl, KCl and MgO, and solution-grown crystals of NaCl were chosen for this purpose. Ingots of NaCl and KCl were obtained from Merck (formerly BDH), Poole. The MgO specimens were purchased directly from W&C Spicer, London. The solution-grown crystals of NaCl were obtained from Salt Union Ltd (formerly ICI), Runcorn. These ionic crystals have a rocksalt structure. The choice of these materials is mainly because their structure and physical properties have been studied extensively (Sprackling, 1976). Furthermore, attrition of NaCl salt has commercial importance (see Chapter 6). The impetus for the study of these solids has also come from their practical importance in process engineering of particulate solids. A wide range of materials has a similar attrition mechanism.

The rocksalt structure as shown in Fig. 2.4 consists of two interpenetrating, face-centred cubic lattices, one made of cations and the other of anions. Because these materials are highly ionic, the primary slip does not occur on the close-packed \{100\} cubic planes, as defined by Miller indices, but instead on the \{110\} cubic diagonal planes. For instance, under quasi-static indentation conditions (e.g. a load indenting a \{100\} face), a pattern of displacement of material by dislocation activity on the \{110\}_{45} planes has been described by Chaudhri (1986), and as shown in Fig. 2.5. At small strains, the material displaced by the indenter moves into the bulk along \{110\}_{45} planes (Fig. 2.5a). As the strain is increased (Fig. 2.5b), a limit is reached where the elastic compression of crystal can no longer accommodate the displaced material, following which the dislocations start moving towards the indented surface on another set of \{110\} planes (Fig. 2.5c).

The existence of a limited number of slip systems makes the ionic crystals to be semi-brittle in term of deformation and fracture. In this thesis, the impact attrition of these materials is investigated. To aid the analysis, the relevant mechanical property data are summarized in Table 2.1.
Fig. 2.4 The conventional unit cell in the rocksalt structure (After Sprackling, 1976). ● cation; ○ anion.

Fig. 2.5 A model of displacement of material by dislocation activity on \{110\}_{45} planes as a load on a sphere indenting a \{100\} face of an MgO crystal increases from a low (a), through a moderate (b), to a high value (c). In (a) and (b) material is pushed downwards, whereas in (c) the dislocations move towards the indented surface as well (After Chaudhri, 1986).
Table 2.1 Mechanical properties of melt-grown single crystals of MgO, NaCl and KCl

<table>
<thead>
<tr>
<th></th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
<th>Notes</th>
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</thead>
<tbody>
<tr>
<td>$\rho / 10^3 \text{ kg m}^{-3}$</td>
<td>3.580</td>
<td>2.165</td>
<td>1.984</td>
<td>1</td>
</tr>
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<td>$H_V / 10^9 \text{ N m}^{-2}$</td>
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<td>0.19</td>
<td>0.10</td>
<td>2</td>
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<td>$E_{100} / 10^{10} \text{ N m}^{-2}$</td>
<td>24.82</td>
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<td>3.88</td>
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</tr>
<tr>
<td>$\nu$</td>
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<td>0.209</td>
<td>0.148</td>
<td>4</td>
</tr>
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<td>$\Gamma_{100} / \text{ J m}^{-2}$</td>
<td>1.60</td>
<td>0.34</td>
<td>0.24</td>
<td>5</td>
</tr>
<tr>
<td>$K_C / 10^5 \text{ N m}^{-3/2}$</td>
<td>9.2</td>
<td>1.8</td>
<td>1.4</td>
<td>6</td>
</tr>
</tbody>
</table>

Note 1: Density from Weast (1984).
Note 2: Vickers hardness from Chapter 4.
Note 3: Young's modulus of $<100>$ crystallographic direction from Appendix A.
Note 4: Poisson's ratio from Appendix A.
Note 5: Cleavage fracture energy from Pratt (1980).
Note 6: Critical stress intensity factor from Chapter 4.
2.2 Mechanisms of Impact Attrition in the Semi-Brittle Failure Mode

In this section different attrition mechanisms are classified, with an emphasis on the chipping process. This is followed by a description of the subsurface lateral cracks which are encountered in materials failing by semi-brittle mode.

2.2.1 Classification of attrition mechanisms

As stated previously, attrition is generally considered as an undesirable particle breakdown in process industries, in contrast to comminution which is a desirable process. Occasionally, in literature attrition has been defined more specifically for surface damage processes. However, the former definition is used here. Various mechanisms are responsible for both attrition and comminution depending on the particle mechanical properties, particle shape and mode of loading. In general, the attrition mechanisms can be broadly classified as fragmentation and surface wear.

Fragmentation is a process where a particle splits into several smaller parts by the propagation of radial or median cracks. In principle, fragmentation is the large-scale fracture where the cracks are generated in the areas with high level stresses, and then develop through the whole volume of the particle. Fragmentation is the dominant mechanism of comminution process although in some circumstances it can occur in attrition of bulk solids such as in shear deformations (Neil, 1986). Wear is the surface damage of particles. It can be subdivided into abrasion and erosion. Abrasive wear is due to the action of particles sliding or rolling over each other. Erosive wear results from the particle collisions. Material removal due to surface damage in semi-brittle failure mode is attributed to the formation of subsurface lateral cracks. This process is commonly referred to as chipping, which is the mechanism of interest here in impact attrition of particulate solids.

Ghadiri and Yuregir (1987) have shown that impact attrition of semi-brittle ionic crystals takes place by the formation of lateral cracks, leading to the material removal in the form of platelets from the faces adjacent to the impact corner as shown in Fig. 2.6. Impact causes localised loading on the corners and edges. This leads to significant plastic deformation of the impact site (see Fig. 2.7) followed by the formation of radial cracks (diagonal cracks), and subsurface lateral cracks (see also Fig. 2.7). In the range of velocities tested here, the radial cracks do not contribute to attrition. At sufficiently high velocities, the propagation of the radial cracks causes
Fig 2.6 High speed record of impact of a solution-grown salt crystal in the size range 425-500 µm on a corner at 22 m s\(^{-1}\). Detachment of a platelet from the right face is clearly visible. Sequence: 1st frame on the top right, 2nd on the bottom right, following frames moving to the left in that order (After Ghadiri and Yuregir, 1987).

Fig 2.7 SEM view of impact damage to a corner of a 2 mm melt-grown KCl cube at an impact velocity of 10 m s\(^{-1}\).
splitting of the particle, *i.e.* fragmentation. This is relevant to comminution and is not considered further here. More examples of chipping are shown and discussed further in Chapter 3.

2.2.2 Characteristics of subsurface lateral cracks

A great deal of experimental evidence shows that for single ionic crystals of MgO, NaCl and KCl, under impact conditions, the lateral cracks form during the unloading cycle (Chaudhri and co-workers, 1978, 1981 and 1991). The formation of these cracks is related to residual elastic tensile stresses, caused by the relaxation of deformed material around the plastic deformation zone. However, in general, there is still a lack of understanding of the details of the mechanism of lateral crack initiation and propagation due to the complex nature of the actual stress-strain field under indentation.

**Crack initiation**

For ideal brittle solids, the features that are responsible for crack initiation are the size and density of pre-existing flaws in the material (Griffith, 1920; Lawn and Wilshaw, 1975b). The concept of pre-existing flaws has sometimes been applied also to materials that exhibit semi-brittle failure. For example, Lawn and Evans (1977) have proposed a model for predicting the onset of formation of median cracks below a sharp indenter. This model is based on the assumption of the existence of "fortuitous" subsurface flaws in the vicinity of the elastic/plastic boundary. It is argued that the inherent flaws play a key role for crack nucleation. The above model is inappropriate for crack initiation in materials such as NaCl and KCl because as pointed out by Hagan (1979), the flaw size required for crack initiation is such that, according to the model of Lawn-Evans, they should be readily detectable.

For crystalline materials, *e.g.* semi-brittle ionic crystals of interest here, it has been established that the dislocation pile-up is responsible for the crack nucleation (*e.g.* see Lawn and Wilshaw, 1975b). Therefore, for such materials pre-existing flaws are not considered important in crack formation. Two types of cracks have been observed for ionic crystals as shown previously in Fig. 2.7. The formation of $\{110\}_{90}$ cracks has been investigated extensively (see Keh *et al.*, 1959; Keh, 1960; Armstrong and Wu, 1978; Burnett, 1984; Chaudhri, 1986). In order to illustrate the nucleation of these
cracks, Chaudhri (1986) presented a three-dimensional pattern of displacement of material beneath the indenter as shown in Fig. 2.8. The material moves down initially along \( \{110\}_{45} \) planes OAB, OBC, OCD and ODA, but as the strain is increased further, it moves towards the indented surface along \( \{110\}_{45} \) planes such as CBFG, DCGH, ADHE and BAEF. The adjacent slip planes are inclined to each other at an angle of 120°, and the displacement along these planes will lead to the formation of \( \{110\}_{90} \) cracks.

The second type of crack is the subsurface lateral crack, which in the case of ionic crystals, occurs on \{100\} cleavage planes. Cottrell (1958) describes a possible mechanism of crack nucleation for this type of crack. He proposed that the nucleation of cleavage cracks on a \{100\} plane results from the coalescence of dislocations on two intersecting slip planes. This type of crack has been observed in tension tests (e.g. see Washburn et al., 1959), uniaxial compression tests (e.g. see Ahlquist, 1974), impact tests (e.g. see Pande and Murty, 1974) and double-cantilever beam bending tests (e.g. see Freiman et al., 1975). These authors have explained the nucleation of cleavage cracks by the dislocation activity. The dislocation reactions, involved in the process of crack nucleation are shown schematically in Fig. 2.9, and this can be represented in terms of an edge dislocation reaction of the following form:

\[
\frac{z}{2} [011]_{(011)} + \frac{z}{2} [01\bar{1}]_{(011)} \rightarrow z[010]
\]

The \( z[010] \) product is a pure edge dislocation which lies in the \{001\} cleavage plane.

In addition, other factors such as loading rate or environment may also determine the critical condition for crack initiation (Lawn and Swain, 1975). For example, Pande and Murty (1974) found no cracks for spherical indentations in relatively pure NaCl or KCl. On the other hand, the \{010\} cleavage cracks were indeed observed under dynamic conditions.
Fig. 2.8 A three-dimensional representation of slip on \{110\}_{45} planes leading to formation of \{110\}_{90} cracks, such as OBF, OCG, ODH and OAE (After Chaudhri, 1986).

Fig. 2.9 Crack formation on (001) cleavage plane.
Crack propagation

The mechanism of crack propagation is relatively well understood in comparison with crack initiation. Generally, crack propagation is in such a stage of growth that the fracture mechanics of macro-fracture has been characterized reasonably well. A crack, once initiated, will tend to propagate along trajectories of lesser principal normal stresses, thereby maintaining near-orthogonality to the major tensile component (Lawn and Swain, 1975). For example, it is well-known that the propagation of Hertzian cone crack for a soda-lime glass indented by a spherical indenter, as shown previously in Fig. 2.2a, is driven by the radial tensile stresses.

For ionic crystals, the above crack propagation principle may not be valid for \{110\}_{90} type of radial cracks as the size of these cracks has been observed to be controlled by the dislocation interactions (Burnett, 1984; Chaudhri, 1986). However, Khasgiwale and Chan (1992) found that for their MgO single crystals, the indentation induced radial cracks extended clearly beyond the region controlled by dislocation interactions. Armstrong and Elban (1984) found that the relationship of correlating the load on the indenter with the length of radial crack for MgO follows approximately that predicted by indentation fracture mechanics (Anstis et al., 1981). Nonetheless, the processes which determine the crack length are clearly important, and need to be further characterized.

In contrast, the propagation of cracks on \{100\} cleavage planes for ionic crystals of interest here is catastrophic, i.e. highly brittle. The development of these cracks is not influenced by the dislocation activity as was the case in their nucleation stage. The propagation of cleavage cracks is caused by the tensile stresses (Chaudhri et al., 1981; Chaudhri, 1986). Particularly, for the subsurface \{100\} lateral crack, the driving force results from the residual tensile stresses which are imposed by the relaxation of the elastic region surrounding the irreversible plastic deformation zone. Aspects of lateral crack propagation are addressed further in the next section after exploring the application of IFM to the impact attrition of particulate solids.

In summary, the mechanism of impact attrition under consideration is that which relates to the chipping process, and falls in semi-brittle failure mode. Material removal is caused by the initiation and propagation of subsurface lateral cracks. Therefore, modelling the attrition process, based on the length and depth of lateral cracks, by the use of fracture mechanics is the key point of the theoretical work presented here.
2.3 Development of a Mechanistic Model of Impact Attrition

In developing a model of impact attrition of particulate solids, use is made of the analysis of impact damage to targets by hard projectiles, where a large body of work has been reported in the literature. Impact attrition is, in a way, the reverse process, where the damage is made to the projectile rather than the target. Much can be learned from damage to targets by impacting projectiles in order to interpret impact attrition behaviour (Hutchings, 1993). Furthermore, the analysis of impact damage to targets has in turn been interpreted by quasi-static indentation of 'half-space' specimens by an indenter (Evans and Wilshaw, 1977; Rowcliffe, 1992 etc.). Therefore, the basis of the work described below is essentially a quasi-static indentation analysis, coupled with dynamic impact features as required in the analysis. Earlier work (Yuregir et al., 1987) on impact attrition of NaCl crystals has shown that the crack morphology of the impacting corner of a particle is similar to that produced by indentation of a flat surface by an indenter, thus supporting the validity of the approach.

When a half-space specimen is indented by a sharp indenter (e.g. Vickers pyramid indenter), the front of the indenter acts as a rigid wedge (Tabor, 1951, see also Wedge Cutting Mechanism in Section 4.2.1.1). A similar pattern is present when a blunt indenter like a punch is used (Gilman, 1971). However, in this case the wedge is produced by the deformation of the material itself. Gilman (1971) analysed the plastic flow pattern under a rigid punch, taking into account the friction effects, by the use of slip-line field theory (see Hill, 1950). This is shown in Fig. 2.10a. The slip lines lie at 45° to a shear-free surface, and at 90° to a rigidly clamped surface. Therefore, no flow occurs immediately under the indenter, leading to the formation of a wedge of undeformed specimen material ahead of the indenter.

A similar wedge pattern has been observed in the deformation of a corner of a cubic particle under both quasi-static and impact loading (Johnson, 1985; Ghadiri and Yuregir, 1987). Deformation of a corner of an NaCl particle formed under impact is shown in Fig. 2.11. The plastic region can be clearly seen standing out as a pinnacle, because the material surrounding this zone has chipped off in the form of platelets. The plastic region around the corner appears to behave as a rigid wedge. The wedge acts as an indenter penetrating into the body of the crystal similar to an indentation, thus causing the formation of the cracks. This wedge pattern is similar to that of a plastic wedge formed by a rigid flat (Johnson, 1985) as shown in Fig. 2.10b, and the field of plastic flow in this case is essentially the same as that shown in Fig. 2.10a.
The compressive flow stresses underneath an indentation reflect the resistive pressure of the material to plastic flow. The indentation induced pressure is commonly defined as the indentation hardness of materials (O'Neil, 1934). It is therefore necessary to examine the level of flow stress of a corner of a particle under quasistatic compression, and to compare it with the hardness in order to apply the indentation fracture mechanics. For this reason, the flow stresses of both 'freshly cleaved' and 'chemically polished and annealed' cubes of NaCl crystals were measured at different loads and different strain rates. The details of these tests are given in Appendix B. The important finding of these tests is that the flow stress of a corner of a particle is approximately independent of the load, and is comparable with the Vickers hardness. This suggests that as soon as the sharp corner of a particle is subjected to sufficiently high loading, it flattens very quickly, and meanwhile the constraint for the deformation increases rapidly and becomes comparable with that of indentation on a half-space specimen.

In the following, the required base for the development of a mechanistic model of impact attrition is outlined, where indentation fracture mechanics is used for the characterization of crack formation leading to impact attrition. Impact damage is quantified in terms of the volume bounded by subsurface lateral cracks. Therefore, a quantitative model of lateral crack extension is required. This is given by indentation fracture mechanics, where the crack parameters, i.e. length and depth, are related to the size of plastic impression made on impact. The latter is in turn related to the impact conditions. To outline the development of the model of attrition, the prerequisite knowledge of the above parameters is first described.

Fig. 2.10 Two dimensional slip-line fields: (a) a half-space specimen indented by a rough blunt punch (Gilman, 1971); (b) a wedge deformed by a rigid flat (Johnson, 1985)
Fig. 2.11 Impact damage to a melt-grown NaCl crystal at an impact velocity of 21 m s$^{-1}$: (a) overview of the particle after impact; (b) magnification of the impact site (After Ghadiri and Yuregir, 1987).
2.3.1 Indentation fracture mechanics approach

For indentation fracture, where there is substantial yielding before the formation of cracks, there is a critical size of indentation above which fracture is induced (Puttick, 1978, 1980 and 1987). This is given by:

\[ r_c = \alpha' \frac{E \Gamma}{Y^2} \]  

(2.1)

where \( \alpha' \) is a constant, \( E \) is Young's modulus, \( \Gamma \) is the fracture surface energy and \( Y \) is the yield stress in a uniaxial test. Puttick (1978) gives a value of 30 for \( \alpha' \) based on the fracture mechanics of PMMA, but in general it is found to depend on the type of test, geometry and material properties. The interest here is not in the value of the numerical constant but in the functional relationship between \( r_c \) and the material properties as required for the development of indentation fracture of lateral cracks described in the following section.

In the case of plane strain, it follows that \( E \Gamma = K_c^2 (1 - v^2) \), where \( K_c \) is the critical stress intensity factor and \( v \) is Poisson's ratio. The yield stress can also be expressed in terms of hardness \( H \), as \( Y = H/\phi \), where \( \phi \) is the constraint factor. Generally, \( \phi \) depends on material properties and indenter geometry. Considering that \( v^2 \) is much smaller than unity, the critical size can be rewritten approximately in the following form:

\[ r_c = \alpha' \left( \frac{K_c}{Y} \right)^2 \approx \alpha' \left( \frac{K_c}{H} \phi \right)^2 \]  

(2.2)

It is assumed here that this relation can also describe the onset of indentation fracture when a corner of a particle is compressed. Because the corners and edges of particles of interest here are very sharp, they undergo plastic deformation initially until the size of indentation (corresponding to the plastic zone size) grows sufficiently large to induce fracture.

2.3.1.1 Lateral crack length

When the surface is indented by a sharp indenter, the plastic zone size increases on increasing the load until the dislocation density reaches a critical limit where micro-cracks form. Crack propagation depends on the strain energy release rate. A rigorous analysis of crack propagation in indentation fracture has not been developed so far due
to the complexities of non-linear deformation and anisotropic behaviour. However, semi-empirical approaches to the analysis of indentation fracture have been used, based on the postulate that crack propagation is some function of the critical stress intensity factor $K_c$, which is in turn proportional to $Y \sqrt{c}$. Based on analysis of the elastic stress field outside a semi-spherical cavity, Evans and Wilshaw (1976) proposed that the length of radial and subsurface lateral cracks may be calculated from the following functional form:

$$\frac{c}{a} = f(\frac{K_c}{Y \sqrt{a}}) = f(\frac{K_c \phi}{H \sqrt{a}})$$

(2.3)

where $a$ is the radius of impression. The relationship between the normalized crack length, $c/a$, and the normalized critical stress intensity factor, $K_c \phi / H \sqrt{a}$, has been determined experimentally for a range of materials including sapphire, spinel, silicon nitride and zinc sulphide. This is shown in Fig. 2.12.

![Normalized crack length as a function of the normalized $K_c$ for radial and subsurface lateral cracks (After Evans and Wilshaw, 1976).](image)

Fig. 2.12 Normalized crack length as a function of the normalized $K_c$ for radial and subsurface lateral cracks (After Evans and Wilshaw, 1976).
It is necessary to consider the validity of applying the above results for the study of indentation fracture of the ionic crystals of interest here. This is partly because semi-brittle ionic crystals are much softer than the materials used by Evans and Wilshaw, and also because the theoretical basis of the above approach is the analysis of elastic field of the region outside the plastic zone. It has been reported that the size of \( \{110\}_{90} \) radial cracks of ionic crystals (see Fig. 2.8) is controlled by the dislocation interactions (Burnett, 1984; Chaudhri, 1986). Therefore, it may be argued that the above approach is not applicable to the extension of radial cracks for ionic crystals. However, as pointed out by Chaudhri et al. (1981), unlike \( \{110\}_{90} \) radial cracks, the lateral cracks (i.e. the \( \{100\} \) cleavage cracks) in ionic crystals are highly brittle and are driven by the tensile circumferential stresses. Therefore, it is appropriate to use the results of subsurface lateral cracks in Fig. 2.12. In addition, it should be mentioned that the formation of lateral cracks is sensitive to the strain rate. It will be shown in Section 4.5 that it is very difficult to generate subsurface lateral cracks for single crystals of NaCl and KCl under quasi-static conditions. However, these cracks are easily formed under impact conditions (see e.g. Fig. 2.7). This implies that these ionic crystals become more brittle as the strain rate increases. Evans and Wilshaw (1977) proposed a method, whereby one dimensional wave analysis could be coupled with quasi-static indentation fracture mechanics, to predict the crack extension under impact conditions. Therefore, information from indentation fracture of materials in Fig. 2.12 is used to describe the lateral crack length of ionic crystals under impact conditions. This approach is used for developing the model of chipping of particles due to impact. This approach is further supported by the work of Chaudhri (1984) who found that the shear stress induced by dynamic loading of KCl crystals can be calculated by the use of elastic field of Yoffe (1982).

The use of the complete curve of lateral crack length as shown in Fig. 2.12 does not easily lend itself to the development of a simple analytical model of chipping. Furthermore, impact damage observations in the velocity range of interest here, i.e. typically 5-30 m s\(^{-1}\), show that \( c/a \) is generally greater than 2. Consequently, the length of these cracks is estimated in this work by a linear approximation to the curve shown in Fig. 2.12, i.e.:

\[
\frac{c}{a} = \left[ \frac{5.7 \frac{K_e}{Y \sqrt{a}}}{\sqrt{a}} \right]^{-1}
\]

(2.4)

where \( \frac{K_e}{Y \sqrt{a}} \) is a dimensionless parameter representing the resistance of material to
crack growth proposed by Evans and Wilshaw. Furthermore, Fig. 2.12 and eqn 2.4 are based on materials which are different from the test materials used here. Therefore, it is more appropriate to neglect the numerical factor and assume that:

\[
\frac{c}{a} \propto \left( \frac{K_c}{\sqrt{a}} \right)^{-1} \tag{2.5}
\]

It will be seen later that because of the presence of other unknown proportionality constants, neglecting the numerical factor does not influence the development of the model. The use of eqn 2.5 allows the material loss to be described in an algebraic form, as it will be shown below. Moreover, the resulting error in the value of material loss by the use of eqn 2.5 instead of the curve in Fig. 2.12 is found to be very small for \(c/a>2\).

Evans and Wilshaw (1976) proposed a different relationship of the form: \(c \propto (F/K_c)^{3/4}\), where \(F\) is the load. When \(F\) is expressed as the hardness (contact pressure) times the contact area for a quasi-static approximation (see eqn 2.9), we get

\[
\frac{c}{a} \propto \left( \frac{K_c}{H \sqrt{a}} \right)^{-3/4} a^{1/8}.
\]

This equation is different from the general form of equation 2.3 proposed originally by Evans and Wilshaw, particularly for the dependence on \(a\). Furthermore, the power of 3/4 cannot be right for dimensional reasons. In subsequent publications, Evans et al. (1978) proposed another relationship of the form

\[c \propto \left( \frac{F}{K_c} \right)^{2/3}\]

This relation is consistent with the functional forms of eqns 2.3 and 2.5, because when \(F\) is replaced by its relation to \(H\) and \(a\), we get

\[
\frac{c}{a} \propto \left( \frac{K_c}{H \sqrt{a}} \right)^{-2/3} \tag{2.6}
\]

However, as it will be shown in Chapter 3, eqn 2.5 provides a better approximation for lateral crack propagation than eqn 2.6. Therefore, eqn 2.5 is used here to describe the lateral crack length.

Furthermore, the effect of constraint factor is not reflected in eqn 2.6 when comparing with eqn 2.5. However, Evans and Charles (1976) considered the constraint factor \(\phi\) to equal 3 for sapphire, spinel, silicon nitride and zinc sulphide, and therefore \(\phi\) in the
ordinate of Fig. 2.12 has presumably been taken as 3. As it will be shown later in Chapter 4, this is not universal. For glasses and polymers, $\phi$ has a value smaller than 3 (Marsh, 1964; Henderson and Wallance, 1989). For ionic single crystals $\phi$ can have a value much higher than 3. It is shown in Chapter 4 that for the ionic crystals tested here the constraint factor is about 80. This is considered to be due to the effect of strain-hardening, and it will be discussed in Chapter 4. However, as the value of the constraint factor is nearly the same for the three test materials, its role is not significant in this analysis.

2.3.1.2 Lateral crack depth

As addressed in Sections 2.1.1.2 and 2.2.2, once a lateral crack is initiated, it will propagate on a plane closely parallel to the specimen surface. For homogenous and isotropic materials, the cracks eventually turn to the free surface (Lawn and Swain, 1975). However, for anisotropic materials, this direction depends on the structure of the materials. For example, for the materials of interest here, the cracks propagate along the cleavage planes because these are minimal fracture energy planes. The propagation is controlled by the tensile stresses. It is therefore necessary to identify the location of the peak tensile stresses because this influences the depth at which lateral cracks initiate. Hill (1950) has analysed the elastic-plastic problem of the formation of spherical cavity in an infinite solid medium under quasi-static condition, and shown that the peak tensile stress occurs at the elastic/plastic interface. A numerical dynamic analysis on the elastic-plastic response regime by Evans et al. (1978) has shown a similar tendency. In order to predict the peak tensile stresses, Evans et al. used one-dimensional wave analysis, in conjunction with the use of Lagrangian finite difference method. Their results have shown that the tensile stresses that cause lateral fracture are at a maximum in the vicinity of the elastic-plastic boundary. Consequently, it has been concluded that the depth at which lateral cracks initiate, $h$, is similar to the maximum depth of the plastic zone, $r_p$ as shown in Fig. 2.13.

A relatively rigorous analysis of the peak tensile stresses associated with elastic-plastic indentation is due to Chiang et al. (1982), who have developed an expanding hemispherical cavity model based on Hill's solution and taking account of the free surface boundary condition. Considering the lateral crack system of interest here, the stress $\sigma_{zz}$ that determines lateral fracture is shown in Fig. 2.14 as a function of radial distance along the surface at various depths, where $\zeta$ is the relative distance beneath
Fig. 2.13 Schematic illustration of plastic deformation zone induced by expansion of a spherical cavity.

Fig. 2.14 Variation of stress $\sigma_{zz}$ with the radial distance along the surface at different depth locations for a plastic zone size of $r_p=2.2a$. (a) at peak load; (b) the residual stress (After Chiang et al., 1982).
the surface \((i.e. \zeta = z/a)\) and \(r\) is the radial distance along the surface. The interesting finding of the analysis of Chiang et al. is that the maximum stress for lateral crack propagation occurs not at the depth of plastic deformation zone as proposed by Evans et al. (1978), but at a depth more like that of the radius of impression. This suggests that shallow lateral cracks are more likely to form than deep ones. This is consistent with the experimental trend in this work which is addressed in Section 3.4.3.2.

Nevertheless, it has been observed that the radius of the plastic zone, \(r_p\), is proportional to the radius of impression (Hockey and Lawn, 1975; Evans and Wilshaw, 1976; Swain and Hagan, 1976). Therefore, the depth of lateral cracks may be expressed as follows:

\[
h \propto a
\]

(Eq 2.7)

Evans et al. (1978) showed that the position of lateral cracks for a number of materials including MgO followed this relationship. Ghadiri and Yuregir (1987) examined the thickness of platelets formed by impact of cubes of NaCl on a rigid target, and concluded that the findings of Evans et al. (1978) were also applicable to the projectile damage. Further experimental verification of this point for ionic crystals is given in Chapter 3.

### 2.3.2. Modelling of contact damage

Contact deformation between the projectile and target is of prime importance in determining the mode and extent of damage imparted to the impacting particle. Contact problems can be classified in terms of elastic, elastic-plastic and fully-plastic. The important contact parameters are the size of impression (related to the contact radius), contact force, and contact time. The contact force is a function of contact time, and this is shown schematically in Fig. 2.15 for elastic and elastic-plastic contact. As discussed in Section 2.2.1, significant plastic deformation and formation of various types of crack occur during the process of impact attrition of particulate solids. Therefore elastic-plastic contact damage is of major concern here. The determination of the contact parameters is described below.
2.3.2.1 Size of impression

The size of impression is the most fundamental contact parameter because it is the direct response of material to impact. It is difficult to measure the contact radius during impact. However, the size of impression left by plastic flow after the event, i.e. after unloading, forms the basis of indentation fracture analysis, and this parameter can be characterized readily using conventional optical microscopy. Among the contact parameters, the size of impression is perhaps the most important parameter for characterizing the crack length and depth, as shown in eqns 2.5 and 2.7.

For a quasi-static approximation, the mean contact pressure, $p$, at any stage of contact may be expressed in terms of the load, $F$, and the size of contact impression, $a$, as:

$$p \propto \frac{F}{a^2}$$

(2.8)
It is assumed that the elastic contribution to the contact size is not significant. This is reasonable in view of the deformation of sharp corners and edges. Therefore, the mean contact pressure is approximated by hardness (see Appendix B). It then follows that the size of contact impression can simply be expressed in terms of hardness as:

\[ a \propto \sqrt{\frac{F}{H}} \]  

(2.9)

The verification of eqns 2.8 and 2.9 has been carried out for quasi-static compression of a corner of ionic crystals. The details are given in Appendix B.

2.3.2.2 Contact force

The contact force under impact conditions can be given by Newton's law. For a cube of length \( l \) and density \( \rho \), impacting at velocity \( v \), we get:

\[ F = M \frac{dv}{dt} = \rho l^3 \frac{dv}{dt} \]  

(2.10)

To calculate the force, a good knowledge of the acceleration, \( \frac{dv}{dt} \), is required. There are several methods to calculate \( \frac{dv}{dt} \) (e.g. see Goldsmith, 1960), but these methods are complex to some extent. In this work, an approximation based on a two point difference method is made:

\[ \frac{dv}{dt} \approx \frac{v}{t_p} \]  

(2.11)

where \( t_p \) is the peak contact time, \textit{i.e.} the impact time at which the force reaches its peak value. Substituting eqn 2.11 into eqn 2.10, the impact force can be expressed, approximately as:

\[ F \approx \rho l^3 \frac{v}{t_p} \]  

(2.12)
Equation 2.12 provides a simple method to estimate the impact force. Equation 2.12 also provides a base for characterizing the size of impression. Substituting eqn 2.12 into eqn 2.9, it then follows that:

\[ a \propto \sqrt{\frac{\rho \, l^3 \, v}{H \, t_p}} \]  

(2.13)

2.3.2.3 Contact time

A rigorous calculation of the contact time is complicated and has not yet been satisfactorily attempted. Simple models are however available in the literature for the cases of elastic and elastic-plastic contacts.

**Elastic contact time**

If the bodies are relatively short, the contact time is large compared to the stress wave transit time across the depth. In this case, the time of contact is determined predominantly by the processes which occur in the actual region of contact. The total time for elastic contact \( t_c \) is given by Johnson (1984) as:

\[ t_c = 2 \, t_p = 3.3 \left( \frac{M^2}{D \, E^* \, v^2} \right)^{1/5} \]  

(2.14)

where \( D \) and \( M \) are the diameter and mass of a spherical projectile, respectively, and \( 1/E^* = (1-v^2)/E + (1-v_t^2)/E_t \). \( E_t \) and \( v_t \) are the Young’s modulus and Poisson’s ratio of the target material, respectively. Equation 2.14 can be rearranged to give the elastic peak contact time (equal to loading time or unloading time) as:

\[ t_p^e = 1.27 \left[ \rho \left( \frac{1-v^2}{E} + \frac{1-v_t^2}{E_t} \right) \right]^{2/5} v^{-1/5} D \]  

(2.15)
Equations 2.14 and 2.15 are valid for a sphere. For a cube, the volume equivalent sphere is considered for which \(D = 1.24 l\). If the target is more rigid than the projectile, the peak elastic contact time can be approximated by:

\[
t_p^e = 1.58 \left( \frac{\rho (1 - v^2)}{E} \right)^{2/5} v^{-1/5} l
\]

(2.16)

It is seen from this equation that \(t_p^e\) is not very sensitive to particle velocity.

**Elastic-plastic contact time**

Because the impact deformation under consideration is elastic-plastic, the peak contact time \(t_p\) consists of both elastic and plastic loading times. It has been suggested that the total contact time can be divided into three distinct periods (Andrews, 1930): elastic, plastic and return periods.

In the elastic period, the stress at the centre of the contact area rises to a value at which plastic flow occurs. In the plastic period, plastic flow occurs until the relative velocity between the two bodies goes to zero under the combined action of both plastic and elastic stresses, and the latter prevailing in the region outside the plastic zone. In the return period, following the stage of dynamic rest, the two bodies move apart under the accelerating force which is now assumed to be fully elastic, caused by the release of the elastic strain energy stored in the elastic region outside the plastic zone.

For the materials of interest here, it can be shown that the plastic contact time is the dominant contact time because the crystals have sharp corners and edges and a low value of the yield stress so that plastic flow occurs at very early stages during the contact. In estimating the peak force and the size of impression, it is therefore only necessary to consider the peak time associated with the plastic flow. The peak contact time dominated by plastic flow has been given by Andrews (1930) and Tabor (1951) as:

\[
t_p^{ep} = \frac{1}{2} \sqrt{\frac{\pi M}{p D}}
\]

(2.17)
where $p$ is the mean contact pressure, and $D$ and $M$ are the diameter and mass of a spherical projectile, respectively. If the mean contact pressure is replaced by the hardness, it then follows that

\[ t_{ep}^p = \frac{1}{2} \sqrt{\frac{\pi M}{H D}} \]  

(2.18)

For a cube, the peak contact time can be approximately estimated as:

\[ t_p = 0.8 \sqrt{\frac{p}{H}} \]  

(2.19)

It is interesting to note that the time of elastic-plastic impact is independent of the impact velocity, in contrast to the elastic contact time (see eqns 2.14 to 2.16). The elastic contact time is affected by the elastic modulus, while the plastic contact time is determined by the hardness of material. The common feature between eqns 2.19 and 2.16 is that the contact time varies linearly with the particle size.

For elastic-plastic contact, the unloading time may be estimated by eqn 2.15 because the unloading process is totally elastic, albeit having a much lower stored energy than the corresponding case for a fully elastic impact. Therefore, the total contact time can be obtained by using eqns 2.17 and 2.15. The experimental investigation of contact time will be presented in Chapter 3.

2.3.3 Outline of model of impact attrition

2.3.3.1 Fractional loss per impact

As discussed earlier in Section 2.2.1, impact damage to a corner of a particle is in the form of plastic deformation, followed by the formation of platelets chipping off from the faces adjacent to the impact site. Considering a single platelet being detached from a corner, this process is approximated by an ideal geometry as shown in Fig. 2.16. A single impact on a corner results in an impression zone, having an area-equivalent radius $a$, and in a subsurface lateral crack of length $c$ and depth $h$, on a plane parallel to a cube face and adjacent to the impact corner. The material bounded by this crack and the free surfaces is considered removed. It then follows that
where $V$ is the volume of material lost per impact. In practice, the shape of the debris may not be a cuboid exactly, and the base area may take a less defined shape than a rectangle. In fact, a triangular base is sometimes observed in the experiments. Furthermore, similar damage may occur on other faces adjacent to the impact corner. However, the volume of debris is still considered to be given by eqn 2.20, where the proportionality factor depends on the geometry of the base and the extent of damage on the other faces. As it will be seen in the derivations below, a knowledge of this factor is not of immediate interest. The fraction of material removed per impact, $\xi$, for a cube of length, $l$, may be given by

$$\xi \propto \frac{c^2 h}{l^3}$$  \hspace{1cm} (2.21)

---

Fig. 2.16  Schematic illustration of the volume of material removed from a corner of a cubic particle.

As it was said previously in Section 2.3.1.1, it is assumed here that the characteristics of these cracks can be obtained from an analysis based on indentation fracture mechanics.
2.3.3.2 Attrition propensity parameter

Based on the above approach, it is possible to estimate the impression size and hence to calculate the fractional loss per impact based on the propagation of subsurface lateral cracks. Substituting eqn 2.19 into 2.13, the size of the impression zone is given by:

\[ a \propto l^{1/2} \left( \frac{\rho}{H} \right)^{1/4} \]  

(2.22)

It is of interest to note that the functional relationship given by eqn 2.22 is identical to that given by Hutchings (1977) which has been derived from a different formulation base. In deriving an equation for the impression size, Hutchings assumed that the work done in forming an indentation is equal to the incident kinetic energy of the impacting particle. The validity of this equation is very important and is examined experimentally in Chapter 3.

The fractional loss per impact, \( \xi \), can now be expressed in terms of the primary variables by substituting for the length and depth of a crack in eqn 2.21 from eqns 2.5, 2.7 and 2.22:

\[ \xi \propto \frac{\rho \sqrt{v^2 - lH}}{K_c^2 \phi^2} \]  

(2.23)

Equation 2.23 relates the fractional loss occurring in one impact to the impact conditions, i.e. velocity, and material properties that are important in describing the extent of damage. It is interesting to note that the fractional loss is proportional to the incident kinetic energy of the particle, and it varies linearly with particle size. The group of parameters \( \frac{H}{K_c^2 \phi^2} \) describes the ratio of material resistances to plastic flow and fracture.

The appearance of the constraint factor \( \phi \) arises from the formulation of the model where it has been assumed that the deformation stress and size of impression of a corner of particles are related to hardness (see eqns B1 in Appendix B and 2.9).
the other hand, eqn 2.5 is in terms of yield stress, hence leading to the presence of $\phi$ in eqn 2.23.

It can be inferred from eqn 2.23 that high values of hardness promote chipping, whilst materials with a low value of hardness tend to deform plastically without shedding much material in the form of platelets. The effect of toughness is the reverse, as high values of toughness prevent propagation of cracks, and hence a low tendency for chipping. However, hardness and toughness are interrelated in indentation fracture mechanics because the flaw responsible for crack propagation is brought about by the extent of plastic flow. It is therefore appropriate to consider the form $\frac{H}{K^2 \phi^2}$ on the whole when analysing the effect of material properties. The validity of eqn 2.23 is assessed in detail in Chapter 5.

Application of eqn 2.23 now requires the determination of the proportionality constant. Estimation of this constant can be done in two ways. In the first method, the proportionality constants in all the relationships leading to eqn 2.23 can be characterized, from which the proportionality constant for eqn 2.23 can be calculated. However, the major difficulty in the determination of the constant in this way is that an assumption needs to be made about the shape of the volume which is shed by the propagation of subsurface lateral cracks. Experimental results show that very often the shape is not as a well-defined as a cuboid, as considered in the development of the model and shown in Fig. 2.16. Therefore a significant error can be made in the calculation of the constant in this way.

The second method is to determine the constant experimentally. This procedure can take a better account of the actual damage imparted to the particle than the above method. Furthermore, as the influence of relevant parameters has already been taken into account through their power indices in eqn 2.23, a unique proportionality constant is expected for different materials exhibiting similar failure behaviour (e.g. where material loss is due to subsurface lateral cracks). This constant should be independent of material properties and impact conditions, and should merely reflect the influence of geometry of chipping. Therefore, with a few statistically reliable tests, the value of the constant can be determined. This approach was selected in this work for the verification of eqn 2.23. In proceeding with this analysis, it is appropriate to define a dimensionless attrition propensity parameter, $\eta$, given by:
\[ \eta = \frac{\rho v^2 l H}{K_c \phi^2} \]  \hspace{1cm} (2.24)

and to assume that the fractional loss per impact, \( \xi \), is proportional to \( \eta \):

\[ \xi = \alpha \eta \]  \hspace{1cm} (2.25)

where \( \alpha \) is the proportionality constant. The determination of this constant from single particle impact tests is described in Chapter 5. To aid the analysis, the values of \( \eta \) calculated from eqn 2.24 for 2 mm particles of MgO, NaCl and KCl for three impact velocities are shown in Table 2.2, based on the values of the parameters from Table 2.1.

Table 2.2 Attrition propensity parameter \( \eta \) predicted from fracture mechanics.

<table>
<thead>
<tr>
<th>( \eta / 10^{-4} )</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>( v = 8.4 \text{ m/s} )</td>
<td>5.41</td>
<td>2.80</td>
<td>2.23</td>
</tr>
<tr>
<td>( v = 5.5 \text{ m/s} )</td>
<td>2.32</td>
<td>1.20</td>
<td>0.96</td>
</tr>
<tr>
<td>( v = 4.1 \text{ m/s} )</td>
<td>1.29</td>
<td>0.70</td>
<td>0.53</td>
</tr>
</tbody>
</table>
2.3.4 Discussion

Several features of the dependence of the attrition propensity parameter, \( \eta \), on the impact velocity and material properties are noteworthy. The power index of 2 for the impact velocity is a direct consequence of the linearisation of the trend of data of subsurface lateral cracks in the range of \( 2 < c/a < 5 \) in Fig. 2.12. The result shows that the fractional loss \( \eta \) is directly dependent on the incident kinetic energy. The work required for plastic flow and surface energy for new surfaces created by fracture take up the major part of the kinetic energy. In practice, for the ionic crystals of interest here, most of the kinetic energy of the particle should be consumed in plastic work because of sharp corners and edges which deform extensively on impact. The fraction of energy required to produce new surfaces is relatively small for the materials tested here because the fracture surfaces are mainly cleavage planes and these are surfaces having minimum fracture energy. Therefore most of input energy is used for the generation and motion of dislocations and the initiation of cracks. Once a crack is initiated, it propagates along a cleavage plane easily with a relatively low resistance.

The linear dependence of \( \eta \) on particle size is also an interesting consequence of the model of breakdown proposed here. For a given velocity, there should be a lower limit for the validity of the linear dependence of \( \eta \) on \( l \). This is because the attrition propensity parameter is based on the assumption that on each impact the velocity is sufficiently high to generate subsurface lateral cracks. As the particle size is reduced, a critical condition is reached where the incident energy is not adequate to initiate any crack, and therefore the attrition propensity parameter is no longer applicable. The limiting particle size depends on the impact velocity, \( i.e. \) as the impact velocity increases the limiting particle size decreases, with the whole process being governed by the energy requirement for the formation of subsurface lateral cracks. This issue will be further discussed in Section 5.5.3.4.

In addition to this limiting particle size which is dependent on impact velocity, there is an ultimate limit for the particle size below which it will not be possible to propagate a crack under any impact velocity or load. At this limit, the failure mode switches from elastic-plastic to fully plastic mode, as addressed by Kendall (1978) and Hagan (1981). However, this ultimate critical size is not of direct concern here because the particle size of interest is much larger than this critical limit, and has therefore not been included in the analysis. However, this limit is very important in grinding of particulate solids to micrometre sizes, commonly referred to as micronization, and will be further discussed in Section 5.5.3.4.
It is also interesting to note that \( \eta \) increases as the hardness is increased. Therefore, it is expected that harder materials will have greater attrition propensity, provided that other material properties remain constant. Experimental evidence for this behaviour has been demonstrated by Ghadiri and co-workers (see Ghadiri et al., 1991; Arteaga et al., 1992 and 1993), who investigated the attrition of commercial NaCl particles. They found that the attrition propensity of commercial solution-grown NaCl crystals depends on the processing route and conditions. The products manufactured under 'rough conditions' in process equipment such as centrifuges and fluidised beds have a higher propensity to attrition than the products that are manufactured in process equipment in which particles are 'gently' handled such as in rotary vacuum filters and dryers. The measurement of hardness on both micro-scale and nano-scale levels, has clearly shown that the NaCl crystals produced under the 'rough conditions' have a higher surface hardness. This trend has been attributed to particle surface damage by inter-particle and particle-wall contacts, where microplastic deformation caused in rough handing of particles leads to the increase of surface hardness. These results support the theoretical model developed here.

The rate of attrition is inversely proportional to the toughness of materials with a power index of 2. This indicates that the fracture toughness plays a strong role in the attrition resistance of particulate solids. For ductile materials, the role of fracture toughness becomes less significant than the hardness (Hutchings, 1993). However, as mentioned previously, in analysing the influence of hardness and toughness on attrition, it is best to consider their combined effect through the form \( \frac{\rho H}{K_c^2 \phi^2} \) which incorporates all the material mechanical properties.
2.5 Conclusions

A mechanistic model of impact attrition of particulate solids having a semi-brittle failure mode has been developed based on indentation fracture mechanics.

The primary mechanism of impact attrition under consideration here is the chipping process, where material removal occurs from the corners and edges of the particles by the propagation of subsurface lateral cracks. Various failure modes have been reviewed, i.e. brittle failure, semi-brittle failure and ductile failure. Semi-brittle failure mode is of interest here. This is relevant to the initiation of lateral cracks because these cracks are induced by extensive plastic flow. Consequently, the parameters required for the description of impact damage, such as contact time have been determined by plastic theory.

Impact damage to particles has been characterized based on an analysis of impact damage to large targets. This in turn has been analysed by the knowledge of quasi-static indentation of half-space specimens. In this analysis, the crack extension and impact pressure are characterized by a quasi-static approach. This approach is appropriate for the low impact velocity range, where the elastic tensile stress waves are not sufficiently strong to cause damage. As a result, a dimensionless attrition propensity parameter, which includes all the relevant physical and mechanical properties of the material, has been derived to describe the attrition propensity of particulate solids. The fractional loss per impact is considered to be proportional to the attrition propensity parameter. This provides a basis for analysis of impact attrition in process equipment and a capability to identify important material properties in order to produce materials with a better attrition resistance.
CHAPTER 3

CHARACTERIZATION OF IMPACT DAMAGE

3.1 Introduction

The validity of the theoretical model of impact attrition requires experimental verification of the mechanisms of particle breakdown, and the characterization of impact damage parameters, such as contact time, impression size and the extent of subsurface lateral cracks. The work on these aspects is described here. Different techniques have been employed for the investigation of impact damage. These techniques include single particle impact testing, high speed photography and impact damage analysis.

The equipment used for impact testing has previously been developed by Ghadiri and Yuregir (1987), and is shown schematically in Fig. 3.1. In this device, particles are accelerated to the required velocity in a vertically downward direction, by the use of an air eductor, and are impacted on a target at a normal angle. The particle velocity is measured by an optical device fixed near the target. The details of the rig will be described in Chapter 5.

The process of impact can be recorded photographically using a high speed camera which is also shown in Fig. 3.1. Details of this camera are described in the following section. With the aid of this technique, the chipping mechanism of impact attrition has been shown by impacting melt-grown NaCl and KCl particles on a rigid flat glass target. The application of high speed photography for observing the impact damage is addressed in Section 3.2.

The techniques involved for the impact damage analysis are scanning electron microscopy (SEM), reflected light microscopy (RLM) and confocal laser scanning microscopy (CLSM). Both SEM and RLM have been applied to characterize the surface damage of the specimens, such as the measurement of the size of impression and the length of radial cracks. SEM has a higher precision and a better depth of field than RLM, thus giving a better image. The specimens for the SEM analysis usually need to be coated with carbon or metal to improve the conductivity. This technique is therefore a destructive process and the specimens cannot be tested any further once they have been coated. In contrast, RLM is a simple, non-destructive technique and can be operated easily. It is very useful when viewing a relatively large object. The
application of SEM and RLM for the study of impact damage, particularly for the characterization of impression size is described in Section 3.3.

Confocal laser scanning microscopy is a relatively new method that is based on scanning optical microscopy. It is particularly appropriate for the observation of subsurface damage. The analysis can be done rapidly and in a non-destructive way. The characterization of subsurface lateral cracks by CLSM is addressed in Section 3.4.

3.2 High Speed Photography of Impact Damage

High speed photography is commonly used in the analysis of fast motion in industrial machinery (e.g. high speed metal forming), military (e.g. ballistics and explosives), aerospace as well as film industry. Various aspects of high speed photography have been reviewed by Lunn (1981) and Field (1982). High speed photography has also been applied to the analysis of impact damage, such as contact time (Chaudhri and Walley, 1978 and Chaudhri et al., 1981), crack propagation in glass (Schardin, 1959), and the displacement field around the crack tip and hence the dynamic stress intensity factor in PMMA (Hu et al., 1984). In this section, the application of high speed photography to the study of impact damage of ionic crystals is presented.

3.2.1 Apparatus

The camera system used in this work is an image converter camera, Imacon 790, manufactured by Hadland Photonics Ltd, Bovingdon, Herts. The light image obtained from a lens in front of the camera is focused on a sensitive photocathode which releases electrons. Further processing is then carried out 'electronically', and hence the potential for very high speeds. A series of deflector and shutter plates in an image tube moves the electron beam to different parts of a phosphor screen, where the electron beam is converted back to a light beam. As a result of the deflection of the electron beam, a set of frames is recorded on a stationary film behind the screen. The camera can produce 8-16 frames of an event at speeds up to 20 million frames per second.

The experimental arrangement for the high speed photography is shown schematically in Fig. 3.2. Because of the short duration of the impact event, the synchronization of the camera trigger with the impact event and lighting is very critical. The camera and the lighting source were triggered by photoelectric sensors, in conjunction with the use of a counter and a time-sequencer. The counter also gave the indication of the
Fig. 3.1 Schematic diagram of impact test rig.

Fig. 3.2 Experimental arrangement for the high speed photography. F denotes a Flash.
impact velocity. A force transducer was mounted underneath the target to detect the moment of impact, and the signals from this and the camera trigger were captured on an oscilloscope. Based on this information, the time delay of the camera trigger was adjusted by the time-sequencer, in order to synchronize the photograph with the impact event. The event was recorded on a Polaroid Instant film which allowed the results to be examined immediately.

3.2.2 Observations of impact damage

Previous work on the observation of impact damage by high speed photography had shown that impact attrition of semi-brittle ionic crystals proceeds essentially by a chipping process (Ghadiri and Yuregir, 1987). The work was carried out at a relatively narrow range of impact velocities and was limited to NaCl crystals only. In order to provide further evidence of chipping for a wider range of impact velocities and for other materials, further work on observation of impact damage by high speed photography was necessary. For this reason, small cubes of melt-grown NaCl and KCl (1 mm in length) were impacted on a soda-lime glass target at different velocities and the events were recorded by the Imacon 790 camera. A frame speed of 100,000 fps was used, with an exposure time of 2 μs for each frame.

Typical records of impact damage for NaCl and KCl particles at different impact velocities are shown in Figs. 3.3 and 3.4, respectively. Eight exposures are obtained for each high speed photograph. The sequence is as follows: 1st frame on the bottom left, 2nd on the top left, the following frames moving to the right in that order. It is clear from the photographs that the impact damage is predominately by chipping, where material removal occurs from the corner of the particle in the form of chips. The amount of damage is, however, related to material properties and impact velocity. For the softer KCl particles, whose hardness is about half of that of NaCl particles, the sharp corner of the impacting particle can sometimes be observed to become blunt after impact as shown in Fig. 3.4a. Furthermore, the amount of damage imparted to KCl particles has been found to be less than that imparted to NaCl particles. This is consistent with the theoretical prediction for semi-brittle failure as described in Chapter 2 (i.e. equation 2.23 for the expression of fractional loss per impact). The quantitative assessment of material losses for ionic crystals including NaCl and KCl will be presented in Chapter 5, when the experimental results of the rate of impact attrition are analysed.
For KCl crystals, complete fragmentation of the particles was occasionally observed at impact velocities above 35 m s\(^{-1}\). This is shown in Fig. 3.5. It should be emphasized here that this behaviour corresponds to the 1 mm melt-grown KCl particles only. For particles having a larger size or for repeated impact tests, the threshold velocity is expected to be lower. KCl crystals are prone to development of small cleavage cracks on their edges during the cleavage process for producing the required size. As it will be seen later, such crystals are discarded from testing as they fail very easily by the propagation of one of these cracks, hence splitting the crystal into two fragments. This process may have been responsible for fragmentation in Fig. 3.5 as the small cleavage cracks on the edges could not have been detected beforehand. During the attrition tests, extreme care was taken to exclude any particle which fragmented. No significant fragmentation took place for the 1 mm melt-grown NaCl particles at this velocity range, with the exception of an instance described below.

In general, impact damage is also influenced by the orientation on impact when the particle approaches the target even at a normal angle (Cleaver et al., 1993). A face-on impact for NaCl is shown in Fig. 3.6. Impact at this orientation produces distributed loading where the stresses are significantly lower than that of a corner impact. Consequently, at low impact velocity, the component of elastic deformation should be significant and the damage to the particle is minute. At sufficiently high impact velocity, the face-on impact as shown in Fig. 3.6, can however cause a severe damage to the particle. This is believed to be due to interfacial friction between the particle and target. Friction results in local tensile stresses which are considered to be responsible for the formation and development of large-scale cleavage cracks from the interface. However, it has been found that for cubic crystals the corner-on impacts are statistically far more frequent than the face-on impacts because of crystal geometry. Therefore, the impact damage is predominately by corner-on impacts. More examples of impact damage to corners of particles will be shown with SEM photographs in Chapter 5.

Another interesting phenomenon associated with single particle impact test is rolling of the particles. This is shown in Fig. 3.7 for a KCl particle where the first impact on the left hand corner has caused damage. The particle subsequently rolls in the frames in a clockwise direction where the contact of a second corner can be seen. This has not produced any visible damage.
Fig. 3.3 High speed photographs of impact of 1 mm melt-grown NaCl crystals. Chipping process is visible for all cases: (a) 10 m s\(^{-1}\) and (b) 23 m s\(^{-1}\).
Fig. 3.4 High speed photographs of impact of 1 mm melt-grown KCl crystals. Chipping process is visible for all cases: (a) 23 m s$^{-1}$ and (b) 34 m s$^{-1}$. 

Fig. 3.5 High speed photograph of impact of a 1 mm melt-grown KCl crystal at impact velocity of 35 m s\(^{-1}\) showing fragmentation of the particle.

Fig. 3.6 High speed photograph of a face-on impact of a 1 mm melt-grown NaCl crystal at impact velocity of 35 m s\(^{-1}\) showing severe damage.
3.2.3 Contact time

It has already been suggested in Chapter 2 that the total contact time for elastic-plastic contact can be divided into two parts: the plastic loading time given by eqn 2.17 and the elastic unloading time given by eqn 2.15. It is therefore necessary to provide experimental data of contact time from high speed photographs to verify the contact time models. However, it is difficult to estimate the contact time from the high speed photographs of Figs. 3.3 to 3.7. This is primarily because the frame speed of $1 \times 10^5$ fps, which gives an inter-frame time of 10 $\mu$s, is not high enough to provide an accurate measure of the short contact time. A higher frame speed of $5 \times 10^5$ fps was therefore used, giving an exposure time of 0.4 $\mu$s and an inter-frame time of 2 $\mu$s. Another feature which causes difficulty for the estimation of the contact time is the shape of the particles. The irregular shape of particles makes the trigger time difficult to control. On the other hand, it is difficult if not impossible to make smooth spherical particles of NaCl and KCl because of the crystal structure. Consequently, it was decided to use the reverse situation, i.e. the materials of interest forming the target
and impacted by rigid spherical particles for which the impact time could be measured more easily. The projectiles were 1 mm lead glass ballotini purchased from Jencons, U.K. The lead glass had a density of 2950 kg m$^{-3}$, elastic modulus of 61 GPa and Poisson's ratio of 0.244, as given by the manufacturer.

In order to get a closer working distance than that used in the photographs shown in Figs. 3.3 and 3.4, and to obtain more details of the object, a 22 mm extension tube combined with a 55 mm Nikkor macro-lens were used in the optical system. This however reverses the sequence of the eight frames and gives the first frame on the top right, second on the bottom right, and following frames moving to the left in that order. Typical records of impact of 1 mm glass ballotini on NaCl and KCl targets are shown in Figs. 3.8 and 3.9, respectively.

In Fig. 3.8, for the case of NaCl, in the first two frames the projectile is approaching the target. The contact starts from the 3rd frame. The departure of the projectile from the target appears to be in the 6th frame. As a result, the contact time is roughly about 5 µs. This value is slightly larger than 3.6 µs which is calculated from eqns 2.17 and 2.15. In Fig. 3.9 for the case of KCl, the contact begins from the first frame. The subsurface damage is visible underneath the contact area from the 2nd frame. In the 3rd frame, the two bodies are still in contact, but the departure of the projectile from the target is obvious in the 4th frame. This also gives the contact time of about 5 µs, which is consistent with the theoretically calculated value of 4.7 µs.

In conclusion, the high speed records of the impact event show that the contact time predicted from the theory in Chapter 2 is not much different from the total contact time observed from the experiments.
Fig. 3.8 High speed record of impact of a 1 mm glass ballotino on an NaCl target at 24 m s\(^{-1}\). Framing speed: \(5\times10^5\) fps.

Fig. 3.9 High speed record of impact of a 1 mm glass ballotino on a KCl target at 23 m s\(^{-1}\). Framing speed: \(5\times10^5\) fps.
3.3 Dependence of the Size of Impression on Impact Velocity

Following the work on high speed photography, the impact damage was analysed by the use of reflected light microscopy (RLM) and scanning electron microscopy (SEM). One of the main characteristics of the surface damage is the size of impact impression. In Chapter 2, it was shown that the length and depth of the cracks were related to the size of impression (see eqns 2.5 and 2.7), and this in turn was related to the impact velocity, as shown in eqn 2.22. The validity of this functional relationship is important and has therefore been assessed.

The power index of the impact velocity for the impression size is predicted to be 0.5, as shown in eqn 2.22. Experimental evidence for this dependence has been obtained by investigating the damage imparted to flat target plates of NaCl, KCl and MgO crystals impacted by 1 mm glass ballotini at different velocities. The size of the targets is about 10 mm x 10 mm x 5 mm, produced by manual cleaving. The damage pattern observed by SEM at high impact velocities for NaCl, KCl and MgO is shown in Figs. 3.10(a)-(c). The impression zone is clearly visible in the case of KCl and NaCl, the former producing a larger size (note the difference in the magnification), whereas for MgO essentially no impression zone can be observed. The square grids produced on the surface of MgO crystal are due to slip on the secondary set of (110)_{45°} slip planes. These are less noticeable in the case of KCl and NaCl, as more plastic flow can be accommodated on the first set of (110)_{45°} slip planes, as shown in Fig. 2.5 (see also Chaudhri, 1986). In some of the experiments, it was found that the glass projectile could be broken when the impact velocity was higher than about 30 m s^{-1}. Therefore, for the purpose of studying the impact damage of MgO, it was appropriate to use stronger materials for the projectile instead of glass. Tungsten carbide (WC) balls having a diameter of 1 mm, supplied by Atlas Ball & Bearing, U.K., were used in the tests. The impact impression on MgO using WC balls as projectiles is shown in Fig. 3.10(d), where the impression is just about visible. [100] surface cleavage cracks as well as [110] radial cracks can be seen in this figure.

The size of impression has been measured by the use of an optical microscope. The results are shown in Fig 3.11(a) for NaCl-glass, in Fig. 3.11(b) for KCl-glass, and in Fig. 3.11(c) for MgO-WC. The experimental data are fitted with straight lines by the use of least-squares analysis. The results confirm the power index of 0.5 for the dependence of the size of impact impression on impact velocity as given by eqn 2.22.
Fig. 5.10. Scanning electron micrographs of damage patterns: (a-c) impacted by 1 mm glass ball; (a) NaCl and (b) KCl at 30 m/s², (c) MgO at 34 m/s²; (d) MgO impacted by a 4 mm tungsten carbide ball at 18 m/s².
Fig. 3.10 Scanning electron micrographs of damage pattern: (a-c) impacted by 1 mm glass ballotini, (a) for NaCl and (b) for KCl at 30 m s\(^{-1}\), (c) for MgO at 34 m s\(^{-1}\); (d) for MgO impacted by a 1 mm tungsten carbide ball at 16 m s\(^{-1}\).
Fig. 3.11 Dependence of impression size on impact velocity: (a) for NaCl and (b) for KCl impacted by 1 mm glass ballotini; (c) for MgO impacted by 1 mm tungsten carbide balls.
3.4 Characterization of Subsurface Lateral Cracks

It was proposed in Chapter 2 that the formation of lateral cracks is responsible for material loss during impact. In this section, subsurface lateral cracks are characterized by the use of confocal laser scanning microscopy (CLSM).

3.4.1 Techniques for studying the extent of subsurface lateral cracks

For transparent or translucent materials such as zinc sulphide, silicon carbide, glass and yttria, subsurface lateral cracks have been characterized by the use of reflected polarised light microscopy (RPLM) by different workers (Marshall et al., 1982; Naylor and Page, 1982; Srinivasan and Scattergood, 1987; Cook, 1990). This technique is simple and suitable for measuring the length of lateral cracks, but it cannot easily be used to measure the depth at which the cracks form. In order to obtain cross-sectional views of subsurface deformation and damage produced during indentation, different mechanical sectioning methods have been used. For example, Smith and Scattergood (1992) obtained a cross section of soda-lime glass by breaking the specimens in four-point bending tests. Hagan and Swain (1978) obtained a cross section of soda-lime glass by making indentations on and near the tip of a pre-existing crack, following the method developed by Peter (1970). The pre-existing crack was first introduced into a plate specimen by gently and repeatedly tapping a scratch mark on the face. The specimen was then aligned in such a way that an indentation could be made on, near or at the tip of the crack so that the crack could section the indentation impression. The sectioned indents were finally obtained by snapping the specimen along the pre-existing crack with finger pressure. Recently, a sequential mechanical sectioning technique has been used by Powell et al. (1993) for the study of subsurface damage of ceramic-matrix composites, where approximately 1 µm layers of material were polished away per sequence by standard metallographic techniques (e.g. see Phillips, 1971). Each section of the specimen can be examined by a reflected light microscope.

The mechanical method for obtaining views of subsurface cracks is a destructive and time-consuming process. Furthermore, the mechanical action to the specimen might obscure the pattern of subsurface damage. These shortcomings have recently been overcome by the use of a new technique, i.e. confocal laser scanning microscopy (CLSM). The earliest application of CLSM can be found in medical and biological areas (see e.g. Brakenhoff et al., 1985; Takamutsi and Fujita, 1987), and since then it has been extended to the field of materials science (see e.g. Russ et al., 1989;
CLSM is the latest generation of light microscopes and incorporates the principle of confocality as shown in Fig. 3.12. Objects in the focal plane of an objective are illuminated by a point source, and the light reflected by the specimen is seen by a detector. The optics are arranged in such a way that the detector and illumination pinholes form conjugated foci hence ensuring that only information from the focal plane reaches the detector. The high precision focusing mode and the use of powerful computers allow the user to produce a whole series of sectional images at various depths by scanning the surface and storing the information on optical disks. The recorded data can be used to construct two or three-dimensional images, where features underneath the surface can be observed with good resolution. CLSM is therefore a unique technique which can provide a rapid and non-destructive means of revealing the subsurface lateral cracks. Wilson (1990) gives a more detailed description of confocal microscopy.

Fig. 3.12 The confocal principle.
3.4.2 Experimental

In Section 3.3 impact damage to single ionic crystals of MgO, NaCl and KCl was investigated by subjecting plates of these materials to impacts by rigid projectiles (i.e. 1 mm WC balls and glass ballotini). Surface damage was then analysed by the use of scanning electron microscopy or conventional optical microscopy. Because ionic crystals of MgO, NaCl and KCl are transparent, it is possible to examine the subsurface damage of the specimens by the use of CLSM. The work on this aspect is reported here for the same materials as tested in Section 3.3.

The CLSM system used for this work was a Carl Zeiss LSM 30 equipped with an internal He-Ne 633 nm laser source. The objective lenses used were 10× and 20×, with numerical apertures of 0.3 and 0.5, respectively. The microscope was operated in confocal incident light conditions. The optical sectioning was computer controlled via a stepping motor with a step size of 250 nm. The images with a resolution of 512 ×512×8 bit were shown on a monitor and transferred to an IBM computer equipped with an image memory board. Two main kinds of sectioning image are produced for the present study. The first kind is an image of a specific depth obtained by scanning the area of interest. These images are assembled in a gallery of images from different depths. The second kind is related to the case where images from various depths are colour coded and superimposed on each other. These images can then be printed and analysed to obtain quantitative information about crack length and depth.

3.4.3 Results

The confocal laser scanning micrographs of impact damage are shown in Figs. 3.13 to 3.15 for MgO, KCl and NaCl, respectively. The image galleries are produced by optical sectioning of the depth of the specimen at certain intervals. The values of the intervals given in the captions of these figures correspond to the distance that the objective has moved each time. Therefore, the true positions of the images in the gallery, and accordingly the positions in the colour depth map, need to be calculated by multiplying the distance by the value of refractive index of the specimen (Powell et al., 1993). The values of the refractive index of MgO, NaCl and KCl crystals are 1.73, 1.54 and 1.50, respectively (Weast, 1984). The sequence of the sections in the galleries is as follows: the free surface on the top left, and the subsequent sections moving to the right, with the following rows being in the same order.
Visual examination of the gallery of images (cf. e.g. Fig. 3.13a) shows the impression of impact on the surface and some small cracks at the boundary of the impression. The presence of cracks can be seen better in Fig. 3.15a. Moving progressively to images of sections below the surface, a number of features start appearing in the images. These are believed to be the subsurface lateral cracks formed at various depths. They can be seen better in Fig. 3.13b, where the pattern of the crack boundary can be better observed. Evidence for the presence of these cracks can be shown by reflected light microscopy, by orienting the plane of the crack in such a way as to get a good reflection for viewing, and also by mechanical sectioning of the specimen (see Section 3.4.4 for the latter). The results have therefore shown the formation of subsurface lateral cracks in all three materials at different impact velocities. The pictures in the galleries show clearly the radial extension of subsurface cracks, which is otherwise very difficult to quantify. The colour depth maps, which are generated by overlapping the section images, give 3-D information on the lateral cracks. The dark blue colour, identified as 0 µm, relates to the position of the last frame in the gallery. Other colours show the damage at positions above this level, identified by colours succeeding from blue to red, with the top position being several micrometers below the free surface (except in Fig. 3.15b where the top position is the free surface). The reason why most of the colour depth maps did not include the free surface was to avoid the influence of surface glare.

Besides the sectioning images, CLSM can also provide direct information of damage pattern of an impact at various depths. A typical example is shown in Fig. 3.15c for NaCl which was impacted at 30 m s⁻¹. The plane of the picture is a φ-z plane in a cylindrical coordinate system, referred to as phi-z-scan. The image shows a considerable dip over a region of the flat surface, which is the impact crater. It has a diameter of about 300 µm and a depth of about 30 µm. Underneath the impact crater, significant features exist down to a depth of about 50 µm, which are thought to be cross-sections of lateral cracks. The true depth is 77 µm, which is calculated by multiplying the figure 50 µm by the refractive index 1.54. Phi-z-scan is therefore a powerful method of revealing impact damage in the orthogonal section of the specimen. However, a φ-z scan provides information from one plane only, and is therefore statistically unreliable for use for quantitative analysis of the crack parameters. The assessment of the length and depth of lateral cracks is described below.
Fig. 3.13 Confocal laser scanning micrographs of an MgO target impacted by a 1 mm WC ball at 16 m s⁻¹: (a) Gallery of images created by sectioning 20 sections at 7.5 μm intervals; (b) Colour depth map obtained by overlapping images from Section No. 3 to No. 20.
Fig. 3.14 Confocal laser scanning micrographs of a KCl target impacted by a 1 mm glass ballotino at 38 m s\(^{-1}\): (a) Gallery of images created by sectioning 20 sections at 6 \(\mu\)m intervals; (b) Colour depth map obtained by overlapping images from Section No. 4 to No. 20.
3.4.3.1 Dependence of lateral crack length on impact velocity

Close examination of impact damage by CLSM and standard optical microscopy shows that as the impact velocity increases, the extent of subsurface damage (i.e. the length and depth of lateral cracks) increases. Furthermore, there appears to be a threshold velocity for the formation of subsurface lateral cracks under impact conditions. For MgO plates impacted by 1 mm tungsten carbide, this is about 5 m s\(^{-1}\), and for NaCl and KCl plates impacted by 1 mm glass ballotini, they are about 20 m s\(^{-1}\) and 25 m s\(^{-1}\), respectively. Below the threshold velocity no lateral cracks can be detected, and the damage consists only of plastic deformation and formation of \{110\}\(_{90}\) type of surface radial cracks.

The CLSM micrograph, shown in Fig. 3.15c, show that lateral cracks form at various depths and have different lengths. Therefore, there is a need to define these features...
more specifically in order to enable a quantitative analysis to be carried out. Considering that the chipping damage is dominated by the length of lateral cracks, it is appropriate to take the radius of the largest crack to represent the length of the subsurface lateral crack. This is defined schematically in Fig. 3.16. The depth of the lateral cracks is taken to be represented by the depth of the largest crack, as shown in Fig. 3.16 (a). The lateral crack length is then measured from the colour height maps.

![Fig. 3.16 Schematic illustration of subsurface lateral cracks: (a) Side View; (b) Top view.](image)

The dependence of lateral crack length on impact velocity for MgO, NaCl and KCl has been obtained from the analysis of many CLSM micrographs. The results are shown in Fig. 3.17. Straight lines have been fitted to the data points by linear regression. The slopes of the straight lines indicate the power index of lateral crack length on impact velocity which can be expressed as:

\[ c \propto v^\beta \quad (3.1) \]
Fig. 3.17 Dependence of lateral crack length on impact velocity: (a) MgO impacted by 1 mm WC balls; (b) and (c) for NaCl or KCl impacted by 1 mm glass ballotini.
The results for MgO and NaCl are reliable and show less scatter than those of KCl. Power indices in the range of 0.78 to 0.9 have been obtained for the three materials. This is consistent with the experimental work of Srinivasan and Scattergood (1987) for the study of lateral cracks in glass, where \( c \) has been taken as the mean lateral crack extension and the exponent \( \beta \) is found to be 0.77.

It has been proposed, in Chapter 2 on the theoretical work of impact attrition, that the lateral crack length is a function of the size of impression as given by eqn 2.5, and the size of impression is in turn related to the impact velocity by eqn 2.22. Substituting eqn 2.22 into eqn 2.5 yields a power-law relationship between lateral crack length and impact velocity as

\[
c \propto v^{0.75}
\]  

(3.2)

The power index of 0.75 is close to the experimental results obtained here. Alternatively, the model of Evans et al. (1978) for the length of lateral cracks can be used, i.e. eqn 2.6. Substituting eqn 2.22 into eqn 2.6, it then follows that

\[
c \propto v^{0.67}
\]  

(3.3)

The power index of 0.67 is much lower than that indicated by the experimental results, and hence provides a poorer agreement than that given by eqn 2.5 as used in the theoretical analysis in this work. In conclusion, the experimental work supports the theoretical approach adopted in Chapter 2, i.e. the quasi-static approach can be used in the analysis of crack propagation under impact conditions. Furthermore, eqn 2.5 provides a better approximation for the lateral crack length than eqn 2.6 due to Evans et al. (1978).

In addition to the lateral cracks, radial cracks have also been observed in all the tests here, e.g. as shown in Fig. 3.15b and that shown previously by SEM in Fig. 3.10. It is useful to examine the relationship between the lateral crack length and the radial crack length here, since it has been assumed by Evans et al. (1978), and Ruff and Wiederhorn (1979) that the length of lateral cracks is proportional to that of radial cracks for the problems encountered in the area of erosion. A typical set of results is shown in Fig. 3.18 for MgO plates impacted by 1 mm WC balls, where a good linear relationship is obtained, hence confirming the validity of the above assumption. As the lateral crack system is of interest in this work, the radial crack system will not be discussed further.
3.4.3.2 Dependence of lateral crack depth on size of impression

The lateral crack depth is based on the most extensive cracking. It is estimated from the gallery of images and the colour height maps, e.g. those shown in Figs. 3.13 to 3.15. The image in the gallery which gives the best focus and brightness for the most extensive crack is taken to represent the depth of the crack. It is found that the depth of lateral cracks increases as the impact velocity increases. It has been proposed in Chapter 2 that the lateral crack depth is proportional to the size of impression given by eqn 2.7. It is therefore necessary to assess this assumption. This is done by plotting the depth of lateral cracks as a function of the size of impression and comparing the results with the theory. This is shown in Fig. 3.19 for MgO and NaCl. The data are scattered, particularly for NaCl. The velocity range clearly needs to be extended in order to increase the confidence in the data. It can however be concluded that the assumption of the linear dependence of the lateral crack depth on the radius of impression is fairly realistic. The velocity range for KCl is not sufficiently wide to
enable a clear trend of the dependence of lateral crack depth on size of impression to be observed, and therefore the results are not reported here.

The depth of lateral cracks, as shown in Fig. 3.19, is in the same range as that of the radius of impression. This implies that the theoretical model of crack depth, proposed by Chiang et al. (1982) as addressed in Chapter 2, is more applicable here than that proposed by Evans et al. (1978). Shallow lateral cracks are more likely to form than deep lateral ones. This is shown clearly in the phi-z-scan picture of Fig. 3.15c for NaCl.

![Diagram showing the depth of lateral crack as a function of the radius of impression.]

Fig. 3.19 The depth of lateral crack as a function of the radius of impression.
3.4.4 Discussion

The experimental results of CLSM have shown that subsurface lateral cracks can form in all the three test materials, i.e. MgO, NaCl and KCl under impact conditions. Little work on the characterization of subsurface lateral cracks has been reported in the literature. Therefore, CLSM has great potential for such an application. To confirm the findings of subsurface lateral cracks by CLSM, it is necessary to compare the results with those obtained by the mechanical sectioning method. Some work was therefore carried out for the NaCl specimen, which was prepared by a standard metallographic technique. The examination of polished samples showed similar patterns of lateral cracks as those observed by CLSM. A recent work by Powell et al. (1993) on the characterization of subsurface damage by the use of both CLSM and mechanical sectioning has also confirmed that the two techniques provided similar data for lateral cracking in ceramic-matrix composites.

There is considerable scatter in the data for KCl as shown in Fig. 3.17c. The velocity range for KCl (i.e. 25-38 m s⁻¹) is perhaps not large enough to produce a clear trend. As the power indices for the dependence of lateral crack length and depth on impact velocity are smaller than unity, i.e. 0.75 for length and 0.5 for depth, the extent of lateral cracking is therefore not very sensitive to the impact velocity. It is therefore necessary in the future work to use a wider velocity range than that tested here, although higher velocities are of little interest to impact attrition.

3.5 Conclusions

Various techniques have been used for the characterization of impact damage. Observations of the impact damage by high-speed photography have confirmed that chipping is significant in impact attrition of semi-brittle ionic crystals. The contact time estimated from the high-speed photographs is in agreement with that predicted by the theory. A dependence of the size of impression on impact velocity to the power of 0.5 has been verified by the use of SEM and RLM. Subsurface lateral cracks, whose formation leads to the chipping process, have been characterized for MgO, NaCl and KCl crystals by the use of confocal laser scanning microscopy. Experimental results have shown that the dependence of lateral crack length on impact velocity is close to the power index of 0.75 predicted by the theoretical work in Chapter 2. The depth of these cracks is close to the impression size, and this is in agreement with the model proposed by Chiang et al. (1982).
CHAPTER 4

CHARACTERIZATION OF MATERIAL PROPERTIES

4.1 Introduction

The model of impact attrition developed in Chapter 2 requires a sound knowledge of several material properties, i.e. the indentation hardness ($H$), constraint factor ($\phi = H/Y$) and fracture toughness ($K_C$). There is a substantial amount of work on the measurement of indentation hardness. There are however inadequate data on the constraint factor and fracture toughness. For example, it is found that for the materials of interest here, the constraint factor has been poorly characterized. This is due to several factors: (i) the complexity of plastic flow arising from work-hardening and anisotropy, (ii) the sensitivity of the yield stress to the level of chemical impurities present, (iii) the history of the specimen.

The theoretical model of impact attrition is based on the indentation fracture mechanics. Therefore, the topic of indentation is briefly reviewed in Section 4.2, followed by the presentation of results of indentation hardness measurement for MgO, NaCl and KCl, using a Vickers indenter.

A detailed study of uniaxial compression of ionic crystals for determining the yield stress is presented in Section 4.3. The effect of work-hardening on the constraint factor for ionic crystals is also addressed. In the process of characterization of material properties, some observations were made on the failure of ionic crystals under uniaxial compression. This type of failure is relevant to the fragmentation and is addressed briefly in Section 4.4.

Fracture toughness plays a crucial role in the model developed here. Various aspects of fracture toughness are therefore discussed in Section 4.5.
4.2 Indentation Hardness, Yield Stress and Constraint Factor

Indentation hardness of a solid represents its resistance to plastic deformation, and is given by the resistive pressure, $p$, \textit{(i.e.} load over projected area\textit{)} when a permanent impression is made. It is measured after a certain amount of plastic strain has been induced, typically around 8\% (Tabor, 1948). The yield stress of a solid relates to the transition from the elastic state to the plastic state under the uniaxial stress-strain condition. Therefore, the indentation hardness is related to its yield behaviour. Quantitatively, the ratio of indentation hardness to yield stress is commonly defined as the constraint factor. The physical meaning of "constraint", which has clearly been explained by Tabor (1986), gives an insight into the indentation process. When a material is subjected to indentation, plastic deformation occurs beneath the indenter. The displaced material is accommodated to a large extent by elastic strains surrounding the local plastic deformation zone. These strains impose a "constraint" on the flow.

Indentation hardness and constraint factor depend on the properties of deformed material, the indenter geometry (see Johnson, 1970) and the coefficient of friction (see Gilman, 1971 and Tabor, 1970). The material properties of concern here are the elastic modulus, yield stress and rate of work-hardening. The yield stress is a function of temperature and strain rate (Hull and Bacon, 1984). As the temperature is decreased, the dislocation mobility is reduced and hence the yield stress increases. A similar behaviour is observed when the strain rate is increased. Because of the increased yield stress under these conditions, the material failure approaches that of the brittle mode, with a concomitant reduction in the fracture surface energy. Consequently, the attrition propensity of materials having a semi-brittle failure is expected to increase at high strain rates or low temperatures. In the following, various theories of indentation hardness are briefly described, and then the idealised models of work-hardening are outlined. The latter provides a base for the interpretation of the experimental results of the materials tested here.

To aid the description, the characteristics of the uniaxial stress-strain curve for several types of material failure are shown in Fig. 4.1. Figures 4.1a and 4.1b represent materials that do not work-harden; elastic deformation is included in Fig. 4.1b, whilst ideal plastic behaviour is shown in Fig. 4.1a. The effect of work-hardening is included with and without an elastic deformation in Figs. 4.1d and 4.1c, respectively. It will be shown that the rate of work-hardening has a very strong influence on the value of the constraint factor.
4.2.1 Theory of indentation hardness

4.2.1.1 Rigid-perfectly plastic indentation

As stated in Section 2.3, the indentation process for a blunt indenter can be approximated for certain materials by a rigid flat punch penetrating a semi-infinite rigid-perfectly plastic medium as shown in Fig. 2.10a. A more general treatment of the indentation process has been described by Hill (1950), based on the wedge cutting mechanism. The plastic flow is described with the aid of slip-line field theory. This theory deals with the plane strain problem of rigid-perfectly plastic materials of the type shown in Fig. 4.1a, where the slip lines are the characteristics of the differential equations of static equilibrium. According to the wedge cutting mechanism, the indentation is treated as cutting by a smooth wedge as shown in Fig. 4.2. It is assumed that slip occurs along planes of maximum shear. The trajectory of the maximum shear stresses is shown on the right hand side of Fig. 4.2. Consider two points, O and J, which after indentation move to locations K and A on the indenter face. Hill (1950) has shown that the amount of slip is characterized by the angle $\psi$, and that the pressure on the wedge is distributed uniformly in the form of:

$$p = H = 2k(1+\psi)$$

(4.1)

where $k$ is the shear yield stress which can be determined from a simple torsion experiment. If $\psi=0=\pi/2$, as of a rigid flat punch, and if von Mises' yield criterion is used, then eqn 4.1 yields a simple relationship between indentation hardness and uniaxial yield stress as:
\[ \phi = H/Y \equiv 3 \]  

(4.2)

Tabor (1948) tested various ductile metals such as aluminum, copper, and mild steel and found that the experimental results are in good agreement with the above theory. However, as it will be seen below, the above simple relationship does not apply to anisotropic and work-hardening materials such as those tested here.

The displacement of material around the indentation produces two modes of impression as shown in Fig. 4.3, *i.e.* the 'piling-up' impression as in aluminum or 'sinking-in' impression as in bearing steel (Marsh, 1964). The rigid-perfectly plastic theory is more suited to the piling-up impression mode. This is because the volume of an element of rigid-plastic material does not alter during flow, and each incremental distortion in a state of plane strain is of pure shear type. Consequently, there is an upward extrusion of the displaced materials which forms a raised crater. For materials whose plastic and elastic deformations are comparable, the sinking-in impression mode is more prevalent than the piling-up mode, and the elastic-plastic theory, described below, is applicable as suggested by Marsh.

![Fig. 4.2 Indentation on a plane surface by a smooth wedge, showing the slip-line field on the right and the main features of the distortion on the left (After Hill, 1950).](image-url)
Fig. 4.3 Schematics of deformation around indentations made by a spherical indenter: (a) 'piling-up' impression mode as in aluminum; (b) 'sinking-in' impression mode as in bearing steel.

4.2.1.2 Elastic-perfectly plastic indentation

Hill (1950) investigated the expansion of a spherical cavity in an infinite elastic-plastic medium, and showed that the mean pressure, $p$, at which the cavity expands is given by

$$\frac{p}{Y} = \frac{2}{3} \left[ 1 + \ln \left( \frac{E}{3(1-\nu)Y} \right) \right]$$  \hspace{1cm} (4.3)

where $E$ is Young's modulus and $\nu$ is Poisson's ratio. This model has since then been widely used to interpret the problems associated with elastic-plastic indentation.

Experimental observations made by Samuels and Mulhearn (1957) have shown that the wedge-cutting mechanism operates only when the wedge angle is less than $30^\circ$. For wedge angles greater than $30^\circ$, it was found that plastic deformation occurred by radial compression producing hemispherical surfaces of constant strain. The centre of the hemispheres was close to the first point of contact between the indenter and specimen. Therefore, Hill's theory of expansion of a spherical cavity to describe the radial compression is more applicable than the wedge cutting mechanism. Figure 4.4 shows the indentation of an elastic-perfectly plastic solid by the expansion of a hemispherical core. The indentation pressure acts over the core, beyond which the plastic strains gradually reduce until they match the elastic strains in the bulk material.

Following Hill but eliminating certain simplifying assumptions, Marsh (1964) derived a new expression relating the indentation hardness ($H$) to the yield stress for a wide range of materials as follows:
\[
\frac{H}{Y} = C_1 + C_2 \left\{ \frac{3}{3 - \kappa} \ln \left( \frac{3}{\kappa + 3\mu - \kappa\mu} \right) \right\}
\]

(4.4)

where \( \kappa = (1-2v)Y/E \), and \( \mu = (1+v)Y/E \). Here the constants \( C_1 \) and \( C_2 \) are no longer the same as those given by Hill's analysis because the constraint around a hemispherical cavity is less than that around a spherical cavity. Furthermore, Marsh estimated \( C_1 = 0.28 \) and \( C_2 = 0.60 \) from experimental data for a number of materials such as glass, some polymers, bearing steel and soft lead alloys.

Equations 4.3 and 4.4 suggest that \( p/Y \) (or \( H/Y \)) is a function of \( E/Y \). For this reason, Tabor (1970) plotted Marsh's data in such a coordinate system as shown in Fig. 4.5. The onset of plastic flow occurs at a ratio of \( p/Y \) (i.e. pressure exerted by the indenter to the yield stress) of about 1 and the rigid-perfectly plastic theory predicts \( p/Y = 3 \). The approximate positions of various materials have been indicated in this figure. It can be seen that for some materials \( p/Y \) is smaller than 3, and therefore Marsh's expression is more reliable.

The hardness of polymers is more difficult to define than other materials because creep prevails even at normal temperatures. Henderson and Wallace (1989) have recently presented a more detailed study of the creep behaviour of high density polyethylene, and have shown that the ratio of time dependent hardness \( H(t) \) to compression yield stress \( Y_c \) is much smaller than 3. This is in agreement with the Marsh's results.

In practice, the indentation is often made with a conical or pyramidal shape indenter where the angle of indenter affects the indentation pressure. A model of indentation hardness for this case has been presented by Johnson (1970) based on the spherical cavity expansion as:

\[
\frac{H}{Y} = \frac{2}{3} \left( 1 + \ln \frac{E \cot \theta}{3Y} \right)
\]

(4.5)

where \( \theta \) is the indenter semi-angle. For a spherical indenter, \( \cot \theta \) may be replaced by \( a/R \), where \( a \) is the radius of the impression and \( R \) is the indenter radius. This is a simple correlation between the indentation hardness and the indenter angle. However, equation 4.5 is not valid for very sharp indenters having a narrow apex angle (see, e.g. Hirst and Howse, 1969, with wedges and Atkins and Tabor, 1965, with cones).
Mean contact pressure $p$

$$\frac{2a}{p} \text{N}$$

Hydrostatic $P$

Core $P$

Plastic

Elastic

Elastic-plastic boundary

Fig. 4.4 Indentation of an elastic-perfectly plastic solid by the expansion of a hemispherical core (After Tabor, 1970).

Fig. 4.5 Variation of indentation pressure, $p$, with the ratio $E/Y$ (After Tabor, 1970).
4.2.1.3 Work-hardening model

The constraint factor, \( \phi \), for the rigid-perfectly plastic and elastic-perfectly plastic models has a value that cannot be greater than 3. However, for some work-hardening materials (e.g. single crystals or high purity well-annealed metals) \( \phi \) has been found to have a value much greater than 3 (Gerk, 1977). Westbrook (1958) has reported a value of around 35 for ionic crystals of MgO, NaCl and LiF. Swain and Lawn (1969) presented a value of about 50–60 for LiF crystals. It will be shown in the next section that the constraint factor may even be as high as about 80 for MgO, NaCl and KCl single crystals.

For materials which exhibit significant work-hardening, it is very important to include this effect in the determination of the constraint factor. This is because hardness is conventionally measured at a strain of about 8% (Tabor, 1986). Therefore significant work-hardening can take place. It is possible to use the analysis of expansion of a spherical cavity, and to incorporate the effect of work-hardening in order to develop a model for the constraint factor. This is described below and later applied to MgO, NaCl and KCl crystals.

It is assumed that for work-hardening materials, the stress after yielding can be expressed in the form of:

\[
\sigma = Y + \Pi \tag{4.6}
\]

where \( \Pi \) is the augmented stress associated with work-hardening, and it is a function of strain \( \varepsilon \). \( \Pi \) is normally approximated by a linear relationship of the form:

\[
\Pi = \Pi' \times (\varepsilon - \varepsilon_0), \quad \varepsilon > \varepsilon_0 \tag{4.7}
\]

where \( \Pi' \) is the constant rate of hardening, and \( \varepsilon_0 \) is the strain corresponding to the onset of work-hardening. For an incompressible material, i.e. no change of volume (\( v = 0.5 \)), a simple relationship between the resistive pressure and the rate of work-hardening has been given by Bishop et al. (1945):

\[
H = p = \frac{2}{3} Y \left[ 1 + \ln \left( \frac{2E}{3Y} \right) \right] + \frac{2\pi^2}{27} \Pi' \tag{4.8}
\]
Consequently, the constraint factor can be determined from the yield stress and the rate of work-hardening. This is described in Section 4.3.2.5.

An alternative approach to determine the constraint factor for work-hardening materials has been suggested by Tabor (1986), where the constraint factor is described as the ratio of the indentation hardness to the representative uniaxial flow stress, \( Y_R \), at an effective strain \( \varepsilon_R \).

\[
\phi = \frac{H}{Y_R} \tag{4.9}
\]

For a Vickers indenter, the average effective strain produced by the indenter is between 8% and 10% (Tabor, 1948). The flow stress at this value of strain is taken to calculate the constraint factor from eqn 4.9. This approach produces significantly different values for \( \phi \) as will be shown below.

In summary, the relationship between the constraint factor and material properties is illustrated in Fig. 4.6. It is clear that the material is subjected to more constraint as its morphology changes from the amorphous state to the crystalline state.

![Diagram showing the relationship between constraint factor, material properties, and hardness to yield stress ratio.]

**Fig. 4.6** Dependence of the constraint factor on material properties.
4.2.2 Vickers hardness of NaCl, KCl and MgO crystals

It was shown in Chapter 2 that hardness is an important material property that influences the attrition propensity. Of the various indenter geometries, Vickers diamond indenter is the most common type, and it has been used here to measure the hardness of the test materials. The materials for the hardness test are from the same sample as those used in the other tests, and whose properties are summarized in Table 2.1. Freshly cleaved specimens were tested at room temperature by using an Instron 4501 mechanical testing machine (see also the next section). A low load cell of 10 N, having a resolution of 0.04 N, was used here. The Vickers indenter was first penetrated into the specimen at a speed of 0.05 mm/min. As the peak load was reached, the indenter was held stationary for 12 seconds, after which the indenter was withdrawn at the same speed. The Vickers square-based pyramid was oriented close to the <100> direction. The diagonal lengths of the impression were measured using an optical microscope with a resolution of about 0.5 µm.

For Vickers hardness, HV, the following relation is used to calculate the hardness from the load, F (N) and diagonal length, d (µm) of indent:

\[
HV = \frac{2F \sin(\theta/2)}{d} = 1.854 \times 10^{12} \times \frac{F}{d^2} \quad \text{(Pa)}
\]  

(4.10)

where the indenter angle \( \theta = 136^\circ \). HV in the above equation is based on the actual contact area rather than the projected area. The results are given in Tables 4.1, 4.2 and 4.3 for NaCl, KCl and MgO, respectively. A low load of 0.2 N (20 g) was applied to the NaCl and KCl specimens. The diagonal length produced by this load was about 45 µm for NaCl, and 62 µm for KCl. In order to get a similar indent size for the harder MgO crystals, loads as high as 5 N had to be applied. However, relaxing the condition of getting approximately the same contact area for MgO tests as those of NaCl and KCl tests, a number of indentations were made for MgO crystals in the load range of 0.5-10 N. These tests are not reported here, but they showed that HV was independent of the load in this load range. This is consistent with Loubet's (1986) results. For NaCl and KCl crystals no significant change of hardness was observed when loads in the range of 0.15-0.5 N were applied. This is in agreement with the earlier results of Sirdeshmukh (1965) and Chin (1972). As the tables show, several indentations were made for each test material, for which the mean value and the standard deviation were calculated. The accuracy of these values is assessed below by an error analysis.
The calculation of the systematic error of the hardness measurement can be deducted from eqn 4.10 as:

\[
\frac{\delta H_v}{H_v} = \frac{\delta F}{F} + 2 \times \frac{\delta d_1}{d_1}
\]

(4.11)

In the measurements, \(\frac{\delta F}{F} = 0.5\%\), and \(2 \times \frac{\delta d_1}{d_1} = 2 \times \frac{0.5 \mu m}{40 \mu m} = 2.5\%\), hence,

\[
\frac{\delta H_v}{H_v} = 3\%
\]

Therefore, the systematic errors may be estimated as:

\[
\begin{align*}
\delta H_v &= 0.2 \text{ GPa, for MgO} \\
\delta H_v &= 0.006 \text{ GPa, for NaCl} \\
\delta H_v &= 0.003 \text{ GPa, for KCl}
\end{align*}
\]

For KCl crystals, the systematic error obtained here is slightly smaller than the random error obtained by doing several measurements of the hardness as given in Table 4.2. For NaCl and MgO, the errors of the hardness values are mainly systematic. Therefore, the Vickers hardness values for MgO, NaCl and KCl crystals may be taken as the following:

\[
\begin{align*}
H_v &= (5.8 \pm 0.2) \text{ GPa, for MgO} \\
H_v &= (0.19 \pm 0.006) \text{ GPa, for NaCl} \\
H_v &= (0.10 \pm 0.004) \text{ GPa, for KCl}
\end{align*}
\]

The measured hardness values for NaCl and KCl are close to those reported by Ghadekar (1982), i.e. 0.192 GPa and 0.099 GPa, respectively. It is interesting to note that Ghadekar's materials were obtained from same source as that in this work. For MgO crystals, the values of Vickers hardness reported in the literature vary from 5 GPa to 8 GPa. The scatter is presumably due to the differences in the level of impurities. This implies that the same sample of material that is used for attrition testing should be used for the characterization of material properties, to reduce the effect of impurities.
Table 4.1 Vickers hardness of NaCl crystals at load $F = 0.2$ N.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_v$/GPa</td>
<td>0.194</td>
<td>0.197</td>
<td>0.199</td>
<td>0.194</td>
<td>0.182</td>
<td>0.185</td>
<td>0.181</td>
<td>0.190</td>
</tr>
</tbody>
</table>

Mean value: 0.190 GPa
Standard deviation: 0.006 GPa

Table 4.2 Vickers hardness of KCl crystals at load $F = 0.2$ N.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_v$/MPa</td>
<td>101</td>
<td>96</td>
<td>95</td>
<td>101</td>
<td>106</td>
<td>95</td>
<td>100</td>
<td>106</td>
<td>95</td>
<td>97</td>
<td>97</td>
</tr>
</tbody>
</table>

Mean value: 0.100 GPa
Standard deviation: 0.004 GPa

Table 4.3 Vickers hardness of MgO crystals at load $F = 5$ N.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_v$/GPa</td>
<td>6.0</td>
<td>6.0</td>
<td>5.4</td>
<td>5.7</td>
<td>5.7</td>
<td>5.8</td>
<td>5.6</td>
<td>5.8</td>
<td>5.8</td>
<td>5.7</td>
<td>6.0</td>
<td></td>
</tr>
</tbody>
</table>

Mean value: 5.8 GPa
Standard deviation: 0.17 GPa
4.3 Uniaxial Compression and Plastic Deformation of Ionic Crystals

In order to evaluate the constraint factor $\phi$, it is necessary to carry out uniaxial compression tests to measure the yield stress, and to investigate the plastic deformation of the test materials. The latter is required because of the work-hardening effect.

4.3.1 Experimental

Large ingots of NaCl, KCl and MgO were cleaved carefully with a fine blade down to 4 mm x 4 mm x 10 mm cuboids. These crystals were not subjected to any further treatment after cleaving, but were always kept under vacuum in a desiccator to be protected from moisture. The length to width ratio of 2.5 was chosen to prevent buckling and to minimise any end effects.

Compression tests were conducted on the samples along the longest axis using an Instron 4501 mechanical testing machine. Loads in the range of 0.04-5000 N were applied and measured by the use of interchangeable load cells. An interface port allows a computer to control the test conditions, data acquisition and analysis. The progress of a test can therefore be monitored easily in "real-time", and previously recorded data can be re-analysed.

The loading speed was controlled at 0.05 mm/min; the corresponding strain rate was about $8\times10^{-5}$ s$^{-1}$. The test procedure was therefore essentially quasi-static. The influence of the compliance of different load cells on the measurement of displacement was also taken into account for the analysis. In order to reduce the friction at the interface between the test specimen and the compression platens and to keep the stress in a homogeneous state, Teflon tapes with the thickness of about 40 µm were used as lubricants. A similar procedure has previously been used by Hsu (1969). The tests were carried out at a controlled temperature of 21±1.5 °C and relative humidity RH = 45%.

4.3.2 Results and analysis

Typical uniaxial stress-strain curves for KCl, NaCl and MgO crystals are shown in Fig. 4.7. The true stress is defined as the ratio of the load on the sample to the instantaneous cross-sectional area, and the true strain is defined by:
Fig. 4.7 Uniaxial compression stress-strain curves:
(a) NaCl, (b) KCl, and (c) MgO.
\( \varepsilon_T = \int_{L_0}^{L_i} \frac{dL}{L} \) \hspace{1cm} (4.12)

where \( L_0 \) and \( L_i \) are the initial and instantaneous lengths, respectively.

To aid the analysis, the general behaviour of the stress-strain curve for work-hardening materials is shown in Fig. 4.8. This curve can be divided into four sections, each representing a distinct deformation behaviour. The initial stage \( I_0 \) represents the elastic deformation stage. This is not observable in Fig. 4.7 because of the scale. Stage I represents plastic flow, the so-called yielding stage, where there is little change in the flow stress due to the low work-hardening rate. In stage II, there is appreciable work-hardening which most often occurs at a constant rate. In stage III, the rate of hardening gradually decreases. Following this stage, the material undergoes buckling or fracture. The characteristics of the \( \sigma-\varepsilon \) curves generally depend on the temperature, strain-rate and sample purity (Sprackling, 1976). In practice, as will be seen below, some of these sections may be absent or combined with the neighbouring sections. Details of the various mechanisms involved in each stage are described further below.

![Stress-Strain Curve](image)

**Fig. 4.8** Characteristic uniaxial stress-strain curve for crystals having a rocksalt structure under room temperature.
4.3.2.1 Yield stress of ionic crystals

Plastic flow is attributed to the action of shear stresses, $\tau$, on the slip planes. The stress component normal to the slip plane does not influence slip within the range of stresses in engineering applications. Large-scale plastic deformation in single ionic crystals occurs at a critical value of shear stress that is resolved in the slip direction. This shear stress is known as the critical resolved shear stress (CRSS), $\tau_0$ (Hayden et al., 1965). With reference to Fig. 4.9, the shear stress $\tau$ in the slip direction is

$$\tau = \frac{F \cos \lambda}{A / \cos \delta} = \frac{F \cos \delta \cos \lambda}{A}$$

or,

$$\tau = \sigma \cos \delta \cos \lambda$$

(4.13)

where $F$ is the applied uniaxial load, $A$ is the cross-sectional area of the specimen, $\sigma$ is the applied compressive stress, $\delta$ is the angle between the slip plane normal and the compressive axis, and $\lambda$ is the angle between the slip direction and the compressive axis. For ionic crystals of NaCl, KCl and MgO, which have the primary slip system of $\{110\}<1\overline{1}0>$, as shown in Fig. 4.9, it follows:

$$\delta = \lambda = 45^\circ$$

whence,

$$\tau = 0.5 \sigma$$

(4.14)

Fotedar et al. (1971) showed that the CRSS can be treated as a well-defined material parameter and can be determined experimentally if the sample under test has a cross-section which is close to being a square. More recently, Goretta et al. (1987) and Soullard et al. (1987) have studied the effect of geometry on the yield stress and have shown that CRSS is independent of the aspect ratio $L/A^{1/2}$ for the condition of $2 < L/A^{1/2} < 5$. Therefore, by analogy with eqn 4.14, we may assume that $\tau_0$ can be related to the uniaxial yield stress $Y$ by a linear relationship of the form:

$$\tau_0 = \Omega Y$$

(4.15)

where $\Omega$ is a proportionality constant which depends on material properties. For the ionic crystals of NaCl, KCl and MgO, $\Omega = 0.5$ because of the angles of the slip planes.
For ionic crystals, there are two kinds of yield point morphology in the uniaxial $\sigma$-$\varepsilon$ curve: (i) a smooth transition region between the initial elastic part and the macroscopic plastic part (as seen for all the three test materials in Fig. 4.7), and (ii) an abrupt yield drop which has been observed in some impure materials by different investigators (Johnston, 1962a,b; Brown and Pratt, 1963). For materials exhibiting a smooth transition, two parameters are often used to calculate the yield stress: (i) a proof stress $\sigma_{0.1}$, which is the stress needed to produce a permanent strain of 0.1%, and (ii) the proportional limit $\sigma_p$, both defined in Fig. 4.10. The yield drop, also known as discontinuous yielding, is due to a sharp increase in the density of mobile dislocations occurring at the start of large-scale plastic deformation. In this case, the lower yield point $\sigma_y$ is commonly used to define the yield stress $Y$ and this is also shown in Fig. 4.10. No yield drop was observed for any of the materials tested here because of the high purity of crystals used.
4.3.2.2 Deformation stages

As discussed earlier, the $\sigma$-$\varepsilon$ curve of single crystals can be partitioned into four distinct stages, each stage describing a particular mechanism. The stage I relates to the range of deformations produced by stresses below the CRSS. In crystals with a rocksalt structure the first dislocation bands are quite isolated and they form on two orthogonal planes identified by Miller indices $\{110\} <1\bar{1}0\>$. Glide develops by an increase in the number of bands and by band broadening until an engineering strain of up to 0.3% is reached (Sprackling, 1976). Because of the glide, albeit small, the behaviour is not rigorously elastic even in this stage, and it is more appropriate to treat the deformation as pseudo-elastic.

Stage I, the initial macroscopic plastic deformation stage, is the easy glide region, and it begins after the pseudo-elastic region and continues until the hardening rate becomes significant. During this stage, the glide is concentrated on the primary slip system (Davidge et al., 1964). This stage has a low and constant hardening rate. In
the experiments on NaCl and KCl reported here, this stage ended quickly by comparison with stage II. The rate of hardening per unit strain in this stage is about 77 MPa for NaCl, and 80 MPa for KCl. For MgO crystals, the rate of hardening in this stage is much smaller than that in stage II.

Stage II is preceded by a short transition region following stage I. In stage II, a linear hardening region with a nearly constant hardening rate prevails, which is two or three times greater than that in stage I. The increased rate during this stage is due to glide occurring on the secondary slip system, identified by Miller indices of (001) <110>, which is oblique to the primary system (Srinivasan et al., 1970).

Stage III is a region of decreasing hardening rate, and it starts with the onset of wavy glide, and is probably associated with macroscopic cross glide of screw dislocations (Sprackling, 1976). For crystals with a rocksalt structure the cross glide occurs on {001} and {111} planes (Matucha, 1968).

4.3.2.3 Results of yield stress and work-hardening rate

The experimental data of uniaxial yield stress \( Y \) and the work-hardening rate \( \dot{\sigma} \) in stage II are summarized in Tables 4.4, 4.5 and 4.6 for NaCl, KCl and MgO, respectively. \( Y \) has been measured here by two methods of proportional limit, \( \sigma_p \), and proof stress, \( \sigma_{0.1} \). The proportional limit \( \sigma_p \) is obviously lower than the proof stress \( \sigma_{0.1} \). An appreciable variation in the yield stress was observed for all the three test materials, and therefore a large number of tests were carried out. The arithmetic mean and standard deviation of the measurements are also given in the tables. The mean value of the yield stress for NaCl is in good agreement with Orozco's (1986) value of 2 MPa, and the mean value for KCl is close to that reported by Ohgaku (1988), i.e. 1.2 MPa. The yield stress of MgO observed here is more scattered than that of NaCl and KCl crystals. This is perhaps due to the sample preparation. Because MgO is very much harder to cleave than NaCl and KCl, there is a chance that micro-cracks are formed in cleaving process (Johnson, 1991). However, the values obtained here are similar to those given by Dew-Hughes (1966) and Auten et al. (1976). The measurements of Stoke (1962) (40-60 MPa) for as-cleaved crystals of MgO tested in tension correspond with the lowest observed values of yield stress (~60 MPa). However, the yield stress in tension and compression are not expected to be the same (see Hayden et al., 1965), and the compression test is more relevant here. The CRSS
(\(\tau_0\)) values of MgO, NaCl and KCl have been calculated from eqn 4.15, and are listed in Table 4.7.

For MgO, the stress in stage I of the plastic flow remains relatively constant, and work-hardening occurs only in stage II. For NaCl and KCl, work-hardening occurs in stage I as well which is at a different rate than in stage II. However, Figs. 4.7a and 4.7b show that work-hardening for NaCl and KCl is much less significant in stage I than in stage II, and therefore the determination of the constraint factor has been based on the rate of work-hardening of stage II (see below).

Table 4.4 Yield stress and hardening rate of stage II for NaCl.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>(\sigma_p) (MPa)</th>
<th>(\sigma_{0.1}) (MPa)</th>
<th>(\Pi') (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.7</td>
<td>2.0</td>
<td>227</td>
</tr>
<tr>
<td>2</td>
<td>1.6</td>
<td>1.9</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>1.8</td>
<td>2.1</td>
<td>230</td>
</tr>
<tr>
<td>4</td>
<td>1.8</td>
<td>2.0</td>
<td>226</td>
</tr>
<tr>
<td>5</td>
<td>1.6</td>
<td>1.9</td>
<td>214</td>
</tr>
<tr>
<td>6</td>
<td>2.0</td>
<td>2.2</td>
<td>230</td>
</tr>
<tr>
<td>7</td>
<td>2.0</td>
<td>2.3</td>
<td>239</td>
</tr>
<tr>
<td>8</td>
<td>1.9</td>
<td>2.1</td>
<td>234</td>
</tr>
<tr>
<td>9</td>
<td>2.0</td>
<td>2.2</td>
<td>242</td>
</tr>
<tr>
<td>10</td>
<td>1.5</td>
<td>1.8</td>
<td>-</td>
</tr>
<tr>
<td>Mean value</td>
<td>1.8</td>
<td>2.1</td>
<td>230</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.15</td>
<td>0.15</td>
<td>8</td>
</tr>
</tbody>
</table>
Table 4.5 Yield stress and hardening rate of stage II for KCl.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>$\sigma_0$ (MPa)</th>
<th>$\sigma_{0.1}$ (MPa)</th>
<th>$\Pi'$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.0</td>
<td>1.2</td>
<td>150</td>
</tr>
<tr>
<td>2</td>
<td>1.3</td>
<td>1.4</td>
<td>146</td>
</tr>
<tr>
<td>3</td>
<td>1.3</td>
<td>1.4</td>
<td>149</td>
</tr>
<tr>
<td>4</td>
<td>1.0</td>
<td>1.2</td>
<td>150</td>
</tr>
<tr>
<td>5</td>
<td>1.1</td>
<td>1.3</td>
<td>154</td>
</tr>
<tr>
<td>6</td>
<td>1.2</td>
<td>1.4</td>
<td>144</td>
</tr>
<tr>
<td>7</td>
<td>1.4</td>
<td>1.5</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>1.3</td>
<td>1.5</td>
<td>153</td>
</tr>
<tr>
<td>9</td>
<td>1.2</td>
<td>1.3</td>
<td>153</td>
</tr>
<tr>
<td>10</td>
<td>1.0</td>
<td>1.2</td>
<td>150</td>
</tr>
<tr>
<td><strong>Mean value</strong></td>
<td><strong>1.2</strong></td>
<td><strong>1.3</strong></td>
<td><strong>150</strong></td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td><strong>0.1</strong></td>
<td><strong>0.1</strong></td>
<td><strong>5</strong></td>
</tr>
</tbody>
</table>

Table 4.6 Yield stress and hardening rate of stage II for MgO.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>$\sigma_0$ (MPa)</th>
<th>$\sigma_{0.1}$ (MPa)</th>
<th>$\Pi'$ ($10^3$ MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>80</td>
<td>86</td>
<td>0.9</td>
</tr>
<tr>
<td>2</td>
<td>56</td>
<td>62</td>
<td>0.9</td>
</tr>
<tr>
<td>3</td>
<td>90</td>
<td>98</td>
<td>1.4</td>
</tr>
<tr>
<td>4</td>
<td>77</td>
<td>80</td>
<td>1.3</td>
</tr>
<tr>
<td>5</td>
<td>92</td>
<td>96</td>
<td>0.9</td>
</tr>
<tr>
<td>6</td>
<td>77</td>
<td>85</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>55</td>
<td>61</td>
<td>1.1</td>
</tr>
<tr>
<td>8</td>
<td>74</td>
<td>76</td>
<td>1.0</td>
</tr>
<tr>
<td><strong>Mean value</strong></td>
<td><strong>75</strong></td>
<td><strong>80</strong></td>
<td><strong>1.1</strong></td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td><strong>12</strong></td>
<td><strong>13</strong></td>
<td><strong>0.2</strong></td>
</tr>
</tbody>
</table>

Table 4.7 Critical resolved shear stress of MgO, NaCl and KCl.

<table>
<thead>
<tr>
<th>CRSS $\tau_0$ (MPa)</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td></td>
<td>1.05</td>
<td>0.65</td>
</tr>
</tbody>
</table>
4.3.2.4 Onset of plastic deformation and work-hardening behaviour

Because of the relatively short period of the initial stage, $I_0$, for NaCl and KCl crystals, it is necessary to examine the strain recovery after unloading to ensure the accuracy and reliability of the yield stress measurements. For this reason, further tests in addition to those given in Tables 4.4 to 4.6 were carried out in which the residual strain was measured after unloading for loads corresponding to different stages. The results are given in Table 4.8, and they corroborate the results in Tables 4.4 to 4.6 obtained from the proof stress approach. In addition to these tests, cyclic loading tests were also carried out for the study of the stress-strain behaviour over several cycles of loading and unloading. The results are shown in Fig. 4.11 for the plastic region of KCl as an example. The yield stress is about 1 MPa, as measured in this work. The first unloading cycle clearly shows that plastic flow has occurred. It is worth noting that, as the material work-hardens, the yield stress increases so that the material behaviour is elastic on re-loading up to the maximum stress to which it was subjected prior to unloading. Following this point, the material flows plastically with a concomitant work-hardening.

Table 4.8 Identification of the onset of plastic strain as a function of compression stress.

<table>
<thead>
<tr>
<th>Materials</th>
<th>$\sigma_{\text{max}}$ (MPa)</th>
<th>$\epsilon_0$ (%)</th>
<th>Stage at end point</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>1.0</td>
<td>0</td>
<td>$I_0$</td>
</tr>
<tr>
<td></td>
<td>2.6</td>
<td>1.0</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>4.9</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>22</td>
<td>13</td>
<td>III</td>
</tr>
<tr>
<td>KCl</td>
<td>0.6</td>
<td>0</td>
<td>$I_0$</td>
</tr>
<tr>
<td></td>
<td>1.7</td>
<td>0.5</td>
<td>I</td>
</tr>
<tr>
<td></td>
<td>3.3</td>
<td>1.7</td>
<td>II</td>
</tr>
<tr>
<td></td>
<td>17</td>
<td>16</td>
<td>III</td>
</tr>
</tbody>
</table>
4.3.2.5 Effect of work-hardening on the constraint factor

It is now possible to estimate the constraint factor $\phi$ from measurements of the uniaxial yield stress and hardness. The values of Vickers hardness $H$, yield stress $Y$ and their ratio $\phi$ for MgO, NaCl and KCl crystals are given in Table 4.9.

The values of the constraint factor $\phi$ shown in Table 4.9 are higher than the previously reported value of 35 by Westbrook (1958), but comparable with those reported more recently by Boyarskaya et al. (1985), i.e. 63-100. However, the yield stress is very sensitive to crystal impurities and to the surface treatment of the specimens. As no information is available on the level of impurities and surface treatment relating to Westbrook's data, any direct comparison must be treated with caution.

The effective strain at which the hardness is measured (i.e. about 8% for the Vickers indenter, according to Tabor, 1986) is significantly larger than that at which the yield stress is measured by e.g. the proof stress approach. Because the test materials work harden significantly during indentation, as a result of increasing plastic strain, the flow
stress at which the hardness is measured is much larger than $Y$. This gives rise to large values of $\phi$. There is a reasonable consistency between the values of $\phi$ obtained for different materials with the same crystal structure, as shown in Fig. 4.12.

Table 4.9 Constraint factor for single crystals of MgO, NaCl and KCl.

<table>
<thead>
<tr>
<th></th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H_v/10^9$ N m$^{-2}$</td>
<td>5.8</td>
<td>0.19</td>
<td>0.10</td>
</tr>
<tr>
<td>$Y/10^6$ N m$^{-2}$</td>
<td>80</td>
<td>2.1</td>
<td>1.3</td>
</tr>
<tr>
<td>$\phi$</td>
<td>73</td>
<td>90</td>
<td>77</td>
</tr>
<tr>
<td>$\phi_{\text{theor.}}$</td>
<td>14</td>
<td>87</td>
<td>90</td>
</tr>
</tbody>
</table>

*From equation 4.8

Fig. 4.12 Correlation line for crystals of MgO, NaCl and KCl relating Vickers hardness and yield stress.
As shown previously in Fig. 4.7, the crystals develop a great resistance to flow during the second strain-hardening stage. Therefore stage II is the dominating stage and has the strongest influence on the constraint factor (see also Gerk, 1977). The rate of hardening during stage II \((H')\) for NaCl, KCl and MgO has been summarized in Tables 4.4 to 4.6.

Using the concept of spherical cavity expansion with work-hardening to describe the indentation process, as outlined previously, it is now possible to obtain a theoretical estimate of the constraint factor by using eqn 4.8. The results are shown in Table 4.9. In view of the assumption in the model that the strain hardening is represented by a single linear rate, as compared to the real case where three stages have been observed, the agreement between the experimental data and theoretical predictions for NaCl and KCl crystals is fair. However the theoretical value of the constraint factor for MgO is much smaller than the experimental value. A possible reason for this is discussed in the next section. It is worth noting that MgO crystals are much more brittle than alkali halide crystals of NaCl and KCl, and fail at strain levels smaller than 8% during the compression tests. Therefore the strain does not reach the extent prevailing in an indentation test. This behaviour may be partially responsible for the observed discrepancy.

### 4.3.3 Discussion

Two factors are considered to be responsible for the high values of \(\phi\) as compared to those of metals and plastics for which \(\phi \leq 3\). These are work-hardening and anisotropy in plastic flow. Plastic anisotropy occurs here because the plastic strain is initially accommodated only by the primary slip systems. As the indentation proceeds further to levels of strain corresponding to that of hardness test, further plastic flow is accommodated by the secondary slip systems (Gilman, 1973). Resistance to shear is very high on the secondary systems, hence giving rise to high values of the constraint factor.

The discrepancy between the theoretical and experimental values for MgO (in Table 4.9) is not clear, but a shortcoming in the theoretical model for neglecting the effect of plastic anisotropy is considered to be responsible. The good prediction for alkali halides NaCl and KCl by work-hardening theory is perhaps because the initial yield stress is very small and the subsequent hardening stages can be approximated as isotropic (see also Gerk, 1977). This implies that the constraint factor for soft ionic
crystals (for which the yield stress is small) may be interpreted by the initial yield stress in conjunction with the work-hardening effect. It appears that this approach is not applicable to hard ionic crystals, where the anisotropy in plastic flow is very significant. Further work is needed to develop a more reliable model of the constraint factor for these materials.

Following Tabor (1986), an alternative method of calculating $\phi$ for work-hardening materials has been attempted. Here, the flow stress, corresponding to the effective strain for the hardness test, is used as defined by eqn 4.9. The representative flow stress, $Y_R$, corresponding to an average effective strain of 8% can be determined for NaCl and KCl from Figs. 4.7a and 4.7b, from which a value of $\phi$ may be inferred from eqn 4.9. These are shown in Table 4.10. The value of $\phi$ determined by this approach is about 10 for NaCl and KCl. This means that the experimentally observed hardness is some ten times greater than the flow stress of the bulk crystals. This approach is not employed to make an estimate of $\phi$ for MgO crystals because these crystals fail well before reaching a strain of 8%.

The reported values of the constraint factor for work-hardening materials have a large variation due to the ambiguity of the relevant yield stress, and due to the fact that the yield stress is sensitive to impurities and material history. However, an important outcome of the above work is that the constraint factor for NaCl and KCl (alkali halide) crystals can be interpreted in terms of the work-hardening effect. A question which naturally emerges from this analysis is: "what is the most appropriate constraint factor for indentation fracture?" For the materials tested so far this is not critical because $\phi$ does not differ significantly between MgO, NaCl and KCl, as expected because of the structural similarity between these crystals. However, an extension of this analysis to other materials with different structures requires an assessment of the role of $\phi$ in indentation fracture, and this aspect can form a topic of future work.

<table>
<thead>
<tr>
<th>Materials</th>
<th>$Y_{R,0.08}$ (MPa)</th>
<th>$\phi$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>18</td>
<td>11</td>
</tr>
<tr>
<td>KCl</td>
<td>11</td>
<td>9</td>
</tr>
</tbody>
</table>
4.4 Failure under Uniaxial Compression

Some of the experiments for obtaining the \(\sigma-\epsilon\) curves for MgO, NaCl and KCl led to the failure of the crystals in compression at high strains. The failure occurred by the formation of (010) or (001) cleavage cracks perpendicular to the (100) loading faces, as shown in Fig. 4.13 for NaCl crystals.

It is well-known that cleavage fracture is influenced by the occurrence of plastic flow prior to cracking in semi-brittle ceramic crystals and in crystals having a high dislocation mobility but with a limited number of slip systems, such as NaCl, KCl and MgO (Ahlquist, 1974). The crack nucleation is due to the dislocation interactions, following the mechanism proposed in Section 2.2.2 as shown in Fig. 2.9.

It is clear that the fracture of a flat face under uniaxial compression is different from that resulting from the deformation of corners by compression or by impact, where the propagation of the cleavage cracks is driven by residual tensile stresses around the deformation zone. Here the process of fracture has been attributed to the local tensile stresses at the interface between the crystal and the platens, formed by discontinuous sliding at the interface (Puttick and Badrick, 1987). This discontinuous sliding can cause local lateral tension, thus providing the driving force for crack propagation from the interface. In the absence of discontinuous sliding, Hill's (1950) classical model of compression between rough plates applies, where the lateral stresses due to friction are compressive everywhere.

The cleavage cracks form more readily in the case of NaCl than in KCl under uniaxial compression. In the case of KCl, high stresses (above 18 MPa) cause buckling of the specimen. This occurs to a much lesser degree for NaCl. It therefore appears that the yield stress plays a major role here, because the plastic flow does not allow the stresses to build up sufficiently to cause fracture. This is in agreement with the quasi-static compression of a corner of crystals (see e.g. Ghadiri and Zhang, 1992), where a significant pile-up along the \(<110>\) direction was observed in the compressed corners of cubes of KCl crystals, while this was less significant for NaCl. Again, as the yield stress of these materials is a function of temperature and strain rate, fracture under uniaxial compression is profoundly influenced by the loading rate and temperature conditions.

In the uniaxial compression the entire volume of the specimen is subjected to plastic flow before failure (cf. the localised plastic deformation in the cases of quasi-static indentation and impact of corners and edges). It is worth noting that internal cracks can also be generated in the final stage of the test as shown in Fig. 4.13a. Failure
under uniaxial compression therefore causes large-scale fracture and leads to fragmentation. This is contrast to the local chipping of the corners which is of interest in this work. Therefore the issue of the failure under uniaxial compression will not be discussed further.

Fig. 4.13 SEM view of the crack morphology of an NaCl crystal after a uniaxial compression test: (a) top face of the crystal; (b) (010) cracks at the interface between the crystal and the platen.
4.5 Fracture Toughness

The theoretical work of impact attrition of particulate solids in Chapter 2, has produced two important results relating to fracture toughness:

(i) The extension of subsurface lateral cracks is inversely proportional to the fracture toughness, $K_c$, as given by equation 2.5.
(ii) The attrition propensity parameter $\eta$ is inversely proportional to $K_c^2$ given by equation 2.24.

Fracture toughness describes the resistance of material to fracture. It is also referred to as the critical stress intensity factor. The model of impact attrition shows that the fracture toughness plays an important role in the attrition propensity of particulate solids. Therefore, a reliable knowledge of the fracture toughness is necessary for the verification of the theoretical model of impact attrition. Substantial efforts have been made in the past to develop suitable methods for the measurement of $K_c$. The conventional methods to determine $K_c$, such as single-edge notched beam (SENB), chevron notched beam (CVNB) and double cantilever beam (DCB) (see Wiederhorn, 1969; Rice et al., 1981) are classified as "macro-notched" techniques. Fracture toughness $K_{IC}$ determined by these techniques apply to crack opening under tension which is commonly referred to as Mode I stress conditions (Lawn and Wilshaw, 1975b). These tests have in general a high degree of reproducibility. It is well-known that the crack propagation under pure shear (Mode II by sliding and Mode III by tearing) requires different amount of stress at the crack tip and therefore have different values of associated fracture toughness. The interest here is on the fracture toughness associated with Mode I because subsurface lateral cracks propagate in this way. These techniques however require an elaborate experimental procedure and may be costly due to the large specimen size that is required.

An alternative to the conventional methods for determination of $K_c$ is the use of indentation fracture (see Anstis et al., 1981; Chantikul et al., 1981; Ponton and Rawlings, 1989a, b). This is generally considered as a "micro-flaw" technique, and was originally developed for brittle materials. It is the simplest method of measuring the toughness of materials, but it is at the same time the most controversial one as the stress field at the crack tip does not correspond to that in the conventional methods. Differences between the fracture toughness measured by indentation fracture and that measured by the conventional methods have been observed (see e.g. Lee and Burn,
1988; Ponton and Rawlings, 1989b). Furthermore, there is a difficulty in applying the indentation fracture analysis for the measurement of toughness for radial crack propagation in crystalline materials of interest here. As previously discussed in Section 2.2.2, the mechanism of the formation of \{110\}_{90} cracks in rocksalt structures is based on the dislocation interactions. Chaudhri (1986) used the spherical indentation of MgO crystals to demonstrate this mechanism, and concluded that the length of these cracks are determined by dislocation interactions rather than tensile stresses, and therefore should not be used for the determination of fracture toughness. In contrast, Khasgiwale and Chan (1992) have recently suggested that the indentation induced radial cracks could extend beyond the dislocation controlled region due to the residual indentation stresses. Armstrong and Elban (1984) found that the diagonal lengths of this type of crack are very nearly in agreement with a power law dependence of the applied force having an index of 3/2 as predicted by the indentation fracture mechanics (see e.g. Anstis et al., 1981).

In view of the foregoing, an attempt was made to measure the toughness by indentation fracture of NaCl, KCl and MgO crystals using a Vickers indenter, following the procedure described earlier in Section 4.2.2. It was found that, apart from MgO, it was very difficult to initiate cracks for highly pure single crystals of NaCl and KCl. This is in agreement with other observations (see e.g. Pande and Murty, 1974; Badrick and Puttick, 1986; Puttick and Badrick, 1987). \{110\}_{90} radial cracks do not easily form with Vickers indenters.

In any case, the above approach is inappropriate for our case here because $K_C$ measured by indentation fracture relates to crack propagation along the $<110>$ rather than $<100>$ direction. The relevant fracture toughness here is that which is associated with the propagation of subsurface lateral cracks. These cracks are responsible for the formation of platelets, and hence for the impact attrition of particulate solids. For the materials of interest, subsurface lateral cracks are on the cleavage planes, and are therefore highly brittle. Consequently, for this type of cracks it is possible to use double cantilever beam method to determine the fracture toughness. As these cracks are highly brittle, linear elastic fracture mechanics has been used in the literature to calculate the fracture surface energy. Cleavage fracture surface energy $\Gamma_{100}$ is therefore considered to be the most appropriate parameter. In fact, a large number of investigations on the fracture surface energy of ionic crystals have been reported by the use of double cantilever beam method (see Gilman, 1960; Freiman, 1975; Pratt, 1980), whereas very few data are available in the literature for $K_C$, where the details of the measurement have also been given (e.g. Hagan, 1979). Wiederhorn et al. (1967)
made a critical analysis of the theory of DCB method and concluded that the fracture surface energy $\Gamma$, obtained from the strain energy basis, is consistent with the stress intensity factor $K_{IC}$, as determined directly from the solution of elastic stress field. Therefore, it is appropriate to use the data of fracture surface energy $\Gamma_{100}$ reported in the literature, in order to estimate fracture toughness $K_{IC}$, assuming that the linear elastic fracture mechanics is applicable to the lateral crack propagation.

For the present study, the fracture surface energies of MgO, NaCl and KCl crystals were obtained from Pratt (1980) as given below:

\[
\begin{align*}
\Gamma &= 1.6 \text{ J m}^{-2} \text{ for MgO} \\
\Gamma &= 0.34 \text{ J m}^{-2} \text{ for NaCl} \\
\Gamma &= 0.24 \text{ J m}^{-2} \text{ for KCl}
\end{align*}
\]

These values are consistent with Lawn's (1975) data of 1.5 J m\(^{-2}\) for MgO and 0.3 J m\(^{-2}\) for NaCl.

$K_{IC}$ is related to the fracture surface energy by the relationship:

\[
K_{IC}^2 = \frac{2E\Gamma}{1 - v^2}
\]  

(4.16)

where $E$ is Young's modulus and $v$ is Poisson's ratio. The data for $E$ and $v$ are given in Table 2.1 for single crystals of MgO, NaCl and KCl.

It should be mentioned that eqn 4.16 is suitable for the plane strain condition. The choice of plane strain condition rather than the plane stress is based on two reasons. Firstly, the fracture toughness determined from a thick plate (plane strain) is independent of the specimen thickness, and is lower than that obtained from a thin plate (plane stress), thus giving a higher safety factor. In fact the plane strain fracture toughness (PSFT) has been recommended by ASTM and BS in order to obtain a reproducible value for toughness of a material. Secondly, regarding the situation of impact attrition of cubic particles, the size of damage zone surrounding the impact corner is generally very small by comparison with the dimensions of the particle, and therefore the toughness determined from plane strain condition provides a better approximation.
The values of fracture toughness $K_{IC}$ calculated from eqn 4.16 are given below:

\[
\begin{align*}
K_{IC} &= 0.92 \text{ MPa m}^{1/2} \quad \text{for MgO} \\
K_{IC} &= 0.18 \text{ MPa m}^{1/2} \quad \text{for NaCl} \\
K_{IC} &= 0.14 \text{ MPa m}^{1/2} \quad \text{for KCl}
\end{align*}
\]

In conclusion, in view of the difficulty in measuring the fracture toughness of ionic crystals by indentation fracture method, the values of $K_c$ required in the model of impact attrition (i.e. eqn 2.24) have been determined from the cleavage fracture surface energies reported in the literature by the use of linear elastic fracture mechanics.
4.6 Conclusions

An experimental investigation has been carried out for the determination of the constraint factor for ionic single crystals of MgO, NaCl and KCl. The work involved the measurement of hardness by Vickers indentation and of yield stress by uniaxial compression. It has been found that the constraint factor has a much higher value for ionic single crystals than that predicted by the traditional relationship of $\phi = H/Y = 3$ due to the work-hardening effect and plastic anisotropy. A work-hardening model of the constraint factor has been used based on Hill's theory of plasticity. A good prediction for the alkali halides of NaCl and KCl has been observed. It is considered that the assumption of isotropic plastic flow may be a good approximation here as the initial yield stress is very small and the subsequent hardening stages can be approximated as isotropic. This approach was found to be unsatisfactory for determining the constraint factor for MgO. The discrepancy between the theoretical and experimental values for MgO is due to the shortcoming of the theoretical model, i.e. ignoring the effect of plastic anisotropy.

The failure of ionic crystals under uniaxial compression is accompanied by an extensive plastic deformation of the whole specimen prior to the fracture. A few $\{100\}$ cleavage cracks have been observed at the interface between the specimen and compression plates. Crack nucleation is considered to be due to the dislocation reaction. The development of these cracks is controlled by the local tensile stresses which are generated by the discontinuities in sliding between the specimen and the plates. The failure under uniaxial compression is believed to have a particular relevance to the fragmentation of particulate solids.

The fracture toughness is determined from the cleavage fracture surface energy $\Gamma_{100}$ using LEFM. This is applicable to the propagation of the subsurface lateral cracks, as the resistance of material to fracture along $<100>$ direction is of interest.
CHAPTER 5

EXPERIMENTAL WORK ON IMPACT ATTRITION

5.1 Introduction

A mechanistic model of impact attrition of particulate solids having a semi-brittle failure mode has been developed in Chapter 2, where the rate of impact attrition is related to the particle properties and impact conditions. In this chapter, the model predictions are verified by comparison with experimental results, in particular for the dependence of attrition rate on material properties, impact velocity and particle size. The work on these aspects is based on impact attrition testing of single crystals of melt-grown MgO, NaCl and KCl.

5.2 Experimental Apparatus

The schematic diagram of the impact test rig has been previously shown in Fig. 3.1. The device consists essentially of an air eductor which 'sucks' the test particles into a tube, accelerates them to the required velocity and impacts them on a stationary rigid target. The test particles are fed individually to the air eductor manually or by the use of a vibratory queuing feeder, depending on the amount of sample to be used. Compressed air is introduced to an annular nozzle, causing a slight vacuum at the constriction. This produces a secondary air flow, which drags the particles into the tube. The particles are then accelerated in a vertically downward direction in the tube by the cocurrent flow of air. The length of the tube is about 1 meter. The particle velocity is determined by measuring the time of flight of each particle between two sets of photoelectric sensors which are located at the end of the tube, close to the target. After impacting the target, the particles are then collected in a cylindrical chamber. A membrane filter with a diameter of 90 mm is placed at the base of the chamber to retain the particles and capture the debris produced by the impact. The air flowing through the chamber is drawn away by a vacuum system. The pressure in the chamber is monitored by a micromanometer, model FCO11 manufactured by Furness Controls Ltd., Bexhill, England. The pressure is kept slightly negative, e.g. 0.5 mm water gauge in order to ensure that: (i) the air eductor operates in a stable manner, (ii) the air flowing through the chamber goes through the membrane filter, hence retaining all the debris on the filter.
Two impact test rigs, operating on the same mechanism, have been constructed at the University of Surrey. One has a tube size of 10 mm I.D., which is suitable for impact testing of relatively large particles, i.e. 2-5 mm in length. In this case, a rough Whatman filter paper (grade 4) is usually used because of high air flow rate, otherwise a large pressure drop is produced which causes difficulty in operation of the device. The other rig has a tube size of 5 mm I.D., which is used for testing smaller particles, i.e. below 2 mm in length. A fine Millipore filter paper with a pore size of 0.45 μm is used here to retain the debris.

5.3 Sample Preparation and Test Procedure

The materials used for this study were high purity melt-grown NaCl, KCl and MgO crystals. Ingots of NaCl and KCl were cleaved manually down to cubes with different side lengths, i.e. 2 mm, 3 mm, 4 mm and 5 mm, by the use of a fine surgical blade. A great deal of attention had to be paid during the cleaving process to minimise the damage imparted to the faces and corners of the crystals. Gloves were always worn to prevent contamination by moisture and grease from the hands. Particles were placed on an anvil which was covered by a thick layer of plasticine to minimise damage to the corners and edges. After cleaving, each dimension of the cubes was measured by the use of vernier callipers with a precision of 0.02 mm. Particles with a tolerance smaller than 0.2 mm were chosen for the test. Each of these particles was then individually examined by optical microscopy for the presence of cleavage cracks on all the faces. These cracks were sometimes produced during manual cleaving, which made the crystals prone to splitting on impact. Cleaving of MgO crystals was found to be difficult, and therefore cubes of MgO with the same sizes as those of NaCl and KCl, were obtained directly from the manufacturer (W&C Spicer). These particles were also examined by optical microscopy to make sure that no cleavage cracks existed. Scanning electron micrographs of typical specimens are shown in Fig. 5.1.

A highly polished sapphire disc (24 mm diameter and 6 mm height) was used as the target material. It was obtained from Agate Products Limited, UK. The Vickers hardness of sapphire is 23 GPa (Evans and Wilshaw, 1976), and this is about 4 times harder than the hardest material tested, i.e. MgO. Therefore the target can be considered to be effectively rigid. For each test about 20 to 50 particles were impacted on the sapphire target at a normal incident angle. The number of impacting particles was limited by the difficulty of sample preparation, in particular cleaving down to the required size without imparting significant damage to the particles.
Fig. 5.1 Freshly cleaved 2 mm specimens of MgO, NaCl and KCl.
Material loss due to attrition was quantified gravimetrically by the use of a high precision balance with a resolution of 10^{-5} g (Model R160P, manufactured by Sartorius, Switzerland). A single impact event does not usually produce sufficient material loss to be measured. Consequently, the material loss was analysed after a cycle of five impacts. Up to twenty impacts were carried out for each test. After each cycle of five impacts, the mother particles were collected in a glass tray, after which they were transferred to a very light and clean aluminium tray for weighing. The use of an aluminium tray was found to give a more reproducible weight reading, presumably by reducing electrostatic effects. The material loss for cycles of five impacts was generally very small, e.g. sometimes smaller than about 1 mg. In this range of weighing, the balance was very sensitive to environmental conditions; for instance the vibration from the floor and the air flow from the laboratory air-conditioning unit during the day time. Consequently, most of the tests were carried out in the evenings or at weekends to increase the weighing accuracy. During the tests, the temperature of the laboratory was about 23-24°C, and the relative humidity around 40-50%.

5.4 Determination of Attrition Rate

5.4.1 Specific attrition rate and fractional loss per impact
There are two methods available to estimate the material loss for a repeated impact process, i.e. the specific attrition rate, \( s \), following the concept of a first order attrition rate as outlined in Chapter 1, and the fractional loss per impact, \( \xi \). Vervoorn and Austin (1990) have shown that the equation defining a first order rate for a repeated impact attrition process is similar to that for a continuous process given by eqn 1.1. If \( M(0) \) is the mass of a sample of mono-size particles undergoing attrition, and \( M(N) \) is the mass of surviving mother particles after \( N \) impacts, then for the specific attrition rate, it follows:

\[
\frac{-dM(N)}{dN} = s \cdot M(N) \quad (5.1)
\]

After integration, it yields:

\[
s = -\frac{1}{N} \ln \frac{M(N)}{M(0)} \quad (5.2)
\]
In practice, the particles are not mono-size and have a size distribution, and therefore the mass of sample relates to that of a narrow size distribution.

The alternative method is to use fractional loss per impact, as given by:

\[
\xi_{0,N} = \frac{1}{N} \frac{M(0) - M(N)}{M(0)}
\] (5.3)

The fractional loss per impact has a more direct physical meaning and a simpler calculation procedure. However, for a low attrition rate process, the specific attrition rate, \(s\), is approximately equal to the fractional loss per impact \(\xi_{0,N}\). This is shown in Appendix C.

In the following sections, the fractional loss per impact has mainly been adopted for the assessment of attrition, in view of its direct relevance to the theoretical model and the invalidity of the first order attrition process for NaCl and KCl. An analysis of the systematic error in the calculation of the fractional loss per impact by eqn 5.3 is given below.

5.4.2 Systematic error of fractional loss per impact

The chipping mechanism produces a 'small scale' damage in contrast to the fragmentation where the mother particles do not survive. Therefore, the fractional loss per impact is usually very small in the chipping process, e.g. in order of \(1 \times 10^{-4}\). It is necessary in this case to consider the systematic error involved in the determination of the fractional loss per impact. The details of the deduction of the systematic error of \(\xi_{0,N}\) determined from eqn 5.3 is presented in Appendix C, and the result is as follows:

\[
|\delta \xi_{0,N}| = \frac{1}{N} \frac{1}{M(0)} \sqrt{2} \delta M(N)
\] (5.4)

where \(|\delta M(N)| = 2 \times 10^{-5} \text{ g}\) for the weighing balance used. It is clear from eqn 5.4 that an increase in the amount of sample or number of impacts reduces the systematic error.
5.5 Experimental Results and Analysis

In this section the experimental results of the dependence of attrition rate on material properties, impact velocities and particle size are presented.

5.5.1 Dependence of attrition rate on material properties

The three types of test materials, i.e. MgO, NaCl and KCl, were deliberately chosen in this work to provide a wide range of material properties whose influence on attrition could be explored. The relevant material properties are given in Table 2.1. For this study, cubes of MgO, NaCl and KCl, 2 mm long, were impacted on a sapphire target at three different velocities, i.e. 8.4 m s\(^{-1}\), 5.5 m s\(^{-1}\) and a velocity corresponding to the free fall of the particles through the impact attrition rig. The free fall velocities were 4.3 m s\(^{-1}\) for NaCl and KCl, and 4.5 m s\(^{-1}\) for MgO. The fractional loss per impact, \(\xi_{0,N}\), for different impact velocities, is given in Table 5.1 together with the systematic errors. It should be mentioned that the data based on 20 impacts for NaCl and KCl at 8.4 m s\(^{-1}\) impact velocity are not reported in Table 5.1. This is because a significant fragmentation occurred after 15 impacts for NaCl and KCl by the propagation of cleavage cracks, and the material loss in this case was not relevant to the model developed in the work. Further discussion on this issue is given in Section 5.5.2.3. The specific breakage rate \(s\) calculated from eqn 5.2, is given in Table 5.2 for comparison. For these tests \(\xi_{0,N} < 2 \times 10^{-3}\), so that \(\xi_{0,N}\) and \(s\) are nearly equal.

It can be seen from Tables 5.1 and 5.2 that the rate of breakdown increases with the number of impacts. For the MgO particles this increase is small, while for NaCl and KCl, it is significant and sensitive to the impact velocity. Nevertheless, the average values of specific attrition rate \(s\) have been summarized in Table 5.3.

It is now possible to estimate the proportionality loss factor \(\alpha\) as given in eqn 2.25 from the experimental data obtained here. To check for the consistency of theoretical predictions, \(\alpha\) is calculated from all the experimental data. This is shown in Table 5.4 for different impact velocities and number of impacts. As the effects of material properties, particle size and impact velocity are taken into account by the attrition propensity parameter \(\eta\), a unique value for \(\alpha\) is expected from all the experimental data. For MgO, \(\alpha\) has a constant value of about 1 for all the different impact numbers and velocities. For KCl, \(\alpha\) has a value close to 1, similar to that of MgO, for the first five impacts, while it gradually increases with the number of impacts. For NaCl, \(\alpha\) has been observed to have a higher value than those of MgO and KCl. For the first
Table 5.1 Fractional loss per impact $\xi_{0,N}$ for 2 mm particles impacting on a sapphire target at different velocities.

<table>
<thead>
<tr>
<th>Velocity, m s$^{-1}$</th>
<th>$\xi_{0,N} / 10^{-4}$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\xi_{0.5}$</td>
<td>4.0±0.1</td>
<td>8.2±0.1</td>
<td>1.8±0.1</td>
</tr>
<tr>
<td></td>
<td>$\xi_{0.10}$</td>
<td>4.51±0.05</td>
<td>11.9±0.1</td>
<td>3.53±0.06</td>
</tr>
<tr>
<td></td>
<td>$\xi_{0.15}$</td>
<td>5.08±0.03</td>
<td>14.70±0.04</td>
<td>6.60±0.04</td>
</tr>
<tr>
<td></td>
<td>$\xi_{0.20}$</td>
<td>5.53±0.02</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>8.4</td>
<td>$\xi_{0.5}$</td>
<td>1.7±0.1</td>
<td>3.3±0.1</td>
<td>0.62±0.07</td>
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<td></td>
<td>$\xi_{0.10}$</td>
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<td></td>
<td>$\xi_{0.15}$</td>
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<td>$\xi_{0.20}$</td>
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</tr>
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<td>5.5</td>
<td>$\xi_{0.5}$</td>
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<td></td>
<td>$\xi_{0.10}$</td>
<td>1.26±0.05</td>
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<td>1.31±0.03</td>
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<td>$\xi_{0.20}$</td>
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<td>2.0±0.1</td>
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<td>$\xi_{0.15}$</td>
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<td>1.13±0.03</td>
</tr>
<tr>
<td></td>
<td>$\xi_{0.20}$</td>
<td></td>
<td>3.36±0.03</td>
<td>1.32±0.02</td>
</tr>
</tbody>
</table>
Table 5.2 Specific attrition rate for 2 mm particles impacting on a sapphire target at different velocities.

<table>
<thead>
<tr>
<th>Velocity, m s(^{-1})</th>
<th>s / (10^{-4})</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(s_{0.5})</td>
<td>4.0</td>
<td>8.3</td>
<td>1.8</td>
</tr>
<tr>
<td></td>
<td>(s_{0.10})</td>
<td>4.5</td>
<td>12.0</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>(s_{0.15})</td>
<td>5.1</td>
<td>15.9</td>
<td>6.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.20})</td>
<td>5.6</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>8.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(s_{0.5})</td>
<td>5.1</td>
<td>15.9</td>
<td>6.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.10})</td>
<td>5.5</td>
<td>12.0</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>(s_{0.15})</td>
<td>5.6</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>(s_{0.20})</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(s_{0.5})</td>
<td>1.7</td>
<td>3.3</td>
<td>0.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.10})</td>
<td>2.1</td>
<td>3.8</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>(s_{0.15})</td>
<td>2.3</td>
<td>4.5</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.20})</td>
<td>2.5</td>
<td>4.9</td>
<td>2.0</td>
</tr>
<tr>
<td>4.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(s_{0.5})</td>
<td>1.2</td>
<td>3.3</td>
<td>0.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.10})</td>
<td>1.3</td>
<td>3.8</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>(s_{0.15})</td>
<td>1.3</td>
<td>4.5</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.20})</td>
<td>1.4</td>
<td>4.9</td>
<td>2.0</td>
</tr>
<tr>
<td>4.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(s_{0.5})</td>
<td>1.7</td>
<td>3.3</td>
<td>0.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.10})</td>
<td>1.3</td>
<td>3.8</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>(s_{0.15})</td>
<td>1.3</td>
<td>4.5</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>(s_{0.20})</td>
<td>1.4</td>
<td>4.9</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Table 5.3 Average values of specific attrition rate (s/\(10^{-4}\)) for 2 mm particles impacting on a sapphire target.

<table>
<thead>
<tr>
<th>Velocity, m s(^{-1})</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.4</td>
<td>4.8</td>
<td>12.1</td>
<td>4.0</td>
</tr>
<tr>
<td>5.5</td>
<td>2.2</td>
<td>4.1</td>
<td>1.4</td>
</tr>
<tr>
<td>4.5</td>
<td>1.3</td>
<td>2.7</td>
<td>1.0</td>
</tr>
<tr>
<td>4.3</td>
<td>2.7</td>
<td>2.7</td>
<td>1.0</td>
</tr>
</tbody>
</table>
Table 5.4 Proportionality factor $\alpha$ for the test materials at different impact velocities.

(a) based on 5 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>0.8</td>
<td>2.9</td>
<td>0.8</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>0.7</td>
<td>2.8</td>
<td>0.7</td>
</tr>
<tr>
<td>$v = 4.5 \text{ m s}^{-1}$</td>
<td>0.8</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 4.3 \text{ m s}^{-1}$</td>
<td>-</td>
<td>2.7</td>
<td>0.7</td>
</tr>
<tr>
<td>Mean value</td>
<td>0.8</td>
<td>2.8</td>
<td>0.7</td>
</tr>
</tbody>
</table>

(b) based on 10 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>0.8</td>
<td>4.3</td>
<td>1.6</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>0.9</td>
<td>3.2</td>
<td>1.2</td>
</tr>
<tr>
<td>$v = 4.5 \text{ m s}^{-1}$</td>
<td>0.8</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 4.3 \text{ m s}^{-1}$</td>
<td>-</td>
<td>3.4</td>
<td>1.7</td>
</tr>
<tr>
<td>Mean value</td>
<td>0.8</td>
<td>3.6</td>
<td>1.5</td>
</tr>
</tbody>
</table>

(c) based on 15 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>0.9</td>
<td>5.1</td>
<td>3.0</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>1.0</td>
<td>3.7</td>
<td>1.7</td>
</tr>
<tr>
<td>$v = 4.5 \text{ m s}^{-1}$</td>
<td>0.8</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 4.3 \text{ m s}^{-1}$</td>
<td>-</td>
<td>3.9</td>
<td>2.0</td>
</tr>
<tr>
<td>Mean value</td>
<td>0.9</td>
<td>4.2</td>
<td>2.2</td>
</tr>
</tbody>
</table>

(d) based on 20 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>1.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>1.1</td>
<td>4.1</td>
<td>2.1</td>
</tr>
<tr>
<td>$v = 4.5 \text{ m s}^{-1}$</td>
<td>0.9</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 4.3 \text{ m s}^{-1}$</td>
<td>-</td>
<td>4.5</td>
<td>2.3</td>
</tr>
<tr>
<td>Mean value</td>
<td>1.0</td>
<td>4.3</td>
<td>2.2</td>
</tr>
</tbody>
</table>
five impacts, $\alpha$ is about 3 and it increases further as the number of impacts increases. These results show that the proportionality factor, $\alpha$, for all the three materials is nearly constant. The variations are not considered to be great, bearing in mind significant differences in the values of hardness and critical stress intensity factor among these materials. It will be shown below that when $\alpha$ is determined for the first impact for NaCl, then its value is close to unity. The variations of $\alpha$ are therefore considered to be due to the work-hardening resulting from repeated impacts. This is discussed in Section 5.5.1.2.

5.5.1.1 SEM observations of impact damage

Scanning electron microscope (SEM) images of the particles after repeated impacts at the velocity of 8.4 m s$^{-1}$, are shown in Fig. 5.2 for MgO, NaCl and KCl as an example. A number of features are noteworthy:

(i) Plastic deformation has occurred at the corners of the particles for all the three materials tested with various types of cracks emanating from the corners, hence indicating a semi-brittle failure mode. The extent of plastic deformation in the case of MgO (see e.g. Fig. 5.2(a2)) is much less than those of NaCl and KCl because MgO is much harder.

(ii) The material losses are from the corners and edges of the particles by chipping.

(iii) The damage morphology of the corners is very complicated for the repeated impacts in contrast to that of a single impact. The size and position of the $\{100\}$ lateral and $\{110\}$ radial cracks vary from corner to corner, and from particle to particle. However, the lateral cracks are primarily responsible for the material removal (see e.g. Fig. 5.2(a2)). The radial cracks propagate from the corner into the particles, and therefore play a minor role for the chipping process. It is however noted that the radial cracks could be a boundary for the development of lateral cracks as shown in Fig. 5.2(b2) for NaCl. In this case, the volume of material removal is bounded by lateral cracks and free surfaces as well as radial cracks. However, the volume depends on the depth and length of the lateral cracks. In comparison with NaCl, there is little chipping in the corner of KCl but there is an indication of extensive plastic deformation.
Fig. 5.2 Repeated impact damage to MgO, NaCl and KCl crystals at 8.4 m s⁻¹ impact velocity. (a1): MgO after 20 impacts; (b1): NaCl after 15 impacts; (c1): KCl after 15 impacts. (a2), (b2) and (c2) show corners of (a1), (b1) and (c1) at higher magnifications.
5.5.1.2 Effect of the number of impacts

An important feature of the results in Table 5.1 is the increase in the fractional loss per impact, $\xi_{0,N}$, as the number of impacts increases. In order to illustrate the variation of attrition rate with the number of impacts at different impact velocities, it is useful to calculate the fractional loss per impact based on instantaneous values of material loss because $\xi_{0,N}$ given by eqn 5.3 provides a cumulative average value. The instantaneous values are obtained from the weight loss for a cycle of five impacts instead of using original mass of material, and this is given by:

$$
\xi_{N,N+5} = \frac{1}{5} \frac{M(N) - M(N+5)}{M(N)}
$$

(5.5)

where $M(N)$ is the mass of mother particles of a given size after $N^{th}$ impact which is also the mass of feeding material for the $(N+1)^{th}$ impact.

The instantaneous fractional loss per impact, $\xi_{N,N+5}$, for different impact velocities is given in Table 5.5. Again, the fractional loss per impact increases with the number of impacts, particularly for NaCl and KCl. For MgO the increase is not significant in comparison with NaCl and KCl. This suggests that the impact attrition of MgO at this velocity range follows approximately a first order process. In order to visualize the effect of number of impacts, the data in Table 5.5 are plotted in Figs. 5.3, 5.4 and 5.5 for NaCl, KCl and MgO, respectively. The increase of attrition rate for NaCl and KCl with the number of impacts is considered to be due to an increase in the hardness. It has been shown in Chapter 4 that soft alkali halide crystals such as NaCl and KCl work-harden significantly when subjected to plastic deformations. Consequently, the corners that are subjected to repeated impacts work-harden and become more brittle. This is not so noticeable for MgO. It is possible, in principle, to account for this effect in eqn 2.24 by determining the changes of hardness as a function of the number of impacts. However, this is a secondary feature which should be addressed at a later stage. The effect of the number of impacts becomes even more significant at higher impact velocities (see Section 5.5.2.1) and greater particle sizes (see Section 5.5.3.2) than these reported here.

The results show that the extent of influence of the number of impacts on material loss is different for different test materials. This makes the determination of the proportionality factor $\alpha$ more difficult. It is most appropriate to determine $\alpha$ based on the first impact because the effect of work-hardening on hardness resulting from
Table 5.5 Fractional loss per impact $\xi_{N, N+5}$ for 2 mm particles impacting on a sapphire target at different velocities.

<table>
<thead>
<tr>
<th>Velocity, m s(^{-1})</th>
<th>$\xi_{N, N+5} / 10^{-4}$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.4</td>
<td>$\xi_{0.5}$</td>
<td>4.0</td>
<td>8.2</td>
<td>1.8</td>
</tr>
<tr>
<td></td>
<td>$\xi_{5.10}$</td>
<td>5.1</td>
<td>15.6</td>
<td>5.2</td>
</tr>
<tr>
<td></td>
<td>$\xi_{10.15}$</td>
<td>6.2</td>
<td>20.5</td>
<td>12.7</td>
</tr>
<tr>
<td></td>
<td>$\xi_{15.20}$</td>
<td>6.9</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5.5</td>
<td>$\xi_{0.5}$</td>
<td>1.7</td>
<td>3.3</td>
<td>0.62</td>
</tr>
<tr>
<td></td>
<td>$\xi_{5.10}$</td>
<td>2.5</td>
<td>4.3</td>
<td>1.73</td>
</tr>
<tr>
<td></td>
<td>$\xi_{10.15}$</td>
<td>2.7</td>
<td>5.7</td>
<td>2.40</td>
</tr>
<tr>
<td></td>
<td>$\xi_{15.20}$</td>
<td>3.0</td>
<td>6.2</td>
<td>3.20</td>
</tr>
<tr>
<td>4.5</td>
<td>$\xi_{0.5}$</td>
<td>1.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{5.10}$</td>
<td>1.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{10.15}$</td>
<td>1.4</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{15.20}$</td>
<td>1.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.3</td>
<td>$\xi_{0.5}$</td>
<td>2.0</td>
<td>0.37</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{5.10}$</td>
<td>3.0</td>
<td>1.59</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{10.15}$</td>
<td>3.6</td>
<td>1.41</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\xi_{15.20}$</td>
<td>4.8</td>
<td>1.89</td>
<td></td>
</tr>
</tbody>
</table>
Fig. 5.3 Variation of attrition rate with number of impacts at different impact velocities for 2 mm NaCl.

Fig. 5.4 Variation of attrition rate with number of impacts at different impact velocities for 2 mm KCl.
repeated impacts has not been taken into account in evaluating $\eta$. However, this is not easy, because the material losses on one impact are usually too small to be gravimetrically measured with a good accuracy. The lowest number of impacts required for the determination of a reliable value of the fractional loss per impact for KCl and MgO as shown in Table 5.1 is about 5, where the relative systematic error for KCl has reached about 20%. The proportionality factor $\alpha$ for MgO is approximately constant and about unity through the whole repeated impact process. It is also found that $\alpha = 1$ for KCl for the first five impacts. NaCl has the highest attrition propensity, for which $\alpha$ varies from 2.7 to 5.1. Because $\alpha$ increases significantly with the number of impacts for NaCl, it was decided to determine its value for the first impact in view of the fact that the fractional loss per impact was sufficiently large in this case so as to make the measurement sufficiently reliable. This is shown in Table 5.6 for NaCl at different impact velocities, where a mean value of about 1 is obtained. About 25 particles were used for each testing. It is noted that the relative error is high, particularly for the lowest velocity.
In conclusion, the increase of the fractional loss per impact with the number of impacts is considered to be due to an increase of the hardness. This is consistent with the prediction of the model, i.e. attrition rate is proportional to the hardness, $\xi \propto H$. The change of hardness is reflected in the variation of proportionality factor $\alpha$. However, the important finding here is that $\alpha$ has the same lower limit of about 1 for all the materials tested. Therefore, the existence of a nearly constant value for $\alpha$ for different semi-brittle materials supports the theoretical work in Chapter 2.

Table 5.6 Fractional loss per impact $\xi$ and proportionality factor $\alpha$ for 2 mm NaCl particles based on the data of the first impact.

<table>
<thead>
<tr>
<th>velocity</th>
<th>$\xi / 10^{-4}$</th>
<th>$\alpha$</th>
<th>Mean value of $\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4$ m s$^{-1}$</td>
<td>2.4±0.6</td>
<td>0.9</td>
<td>1.1</td>
</tr>
<tr>
<td>$v = 5.5$ m s$^{-1}$</td>
<td>1.3±0.5</td>
<td>1.1</td>
<td>1.1</td>
</tr>
<tr>
<td>$v = 4.3$ m s$^{-1}$</td>
<td>1.1±0.6</td>
<td>1.5</td>
<td>1.1</td>
</tr>
</tbody>
</table>

5.5.2 Dependence of attrition rate on impact velocity

The impact attrition model presented in Chapter 2 predicts that the attrition propensity is proportional to the square of impact velocity. The velocity range for the tests reported in Section 5.5.1 was not sufficiently wide to enable a good verification of the effect of velocity. Further experimental work was therefore carried out for 2 mm MgO, NaCl and KCl in a wider velocity range. This was also required for the identification of the velocity range in which the chipping mechanism prevailed.

5.5.2.1 Power law dependence of attrition rate on impact velocity

The data presented here include those reported previously in Table 5.1. The results are shown in Figs. 5.6, 5.7 and 5.8 for MgO, NaCl and KCl, respectively. In these figures, a power law dependence is assumed and the experimental data are analysed by the linear regression method. Consequently, the slopes of the straight lines on the logarithmic scales represent the power indices of the dependence of attrition rate on impact velocity.
The power indices are listed below:

\[ \gamma = 2.0-2.4 \quad \text{for MgO} \]
\[ \gamma = 2.0-2.5 \quad \text{for NaCl} \]
\[ \gamma = 1.9-3.0 \quad \text{for KCl} \]

The experimental results show that the power index increases as the number of impacts increases. This effect is considered to be due to an increase of hardness as the number of impacts increases because of work-hardening. At high impact velocities particles undergo a higher rate of work-hardening. It can be concluded that the power index \( \gamma \) is close to 2.0 for the first few impacts, hence confirming the theoretical predictions. Work-hardening makes the response more complex for repeated impacts, where departure from power index 2 is observed. It will be possible to modify the model to take account of work-hardening in which case \( \alpha \) would be expected to remain constant. This can form an interesting line of future research.

![Graph](image)

**Fig. 5.6** Dependence of fractional loss per impact on impact velocity for MgO.
Fig. 5.7 Dependence of fractional loss per impact on impact velocity for NaCl.

Fig. 5.8 Dependence of fractional loss per impact on impact velocity for KCl.
5.5.2.2 SEM observations of impact damage

SEM photographs of the particles after repeated impacts near the upper boundary of the velocity range of chipping are shown in Fig. 5.9 for MgO, NaCl and KCl. The morphology of impact damage is the same as that shown previously in Fig. 5.2, but its extent is much larger than that of the lower velocity. For example, the extent of the crack-opening displacement for lateral and radial cracks is much greater than that shown in Fig. 5.2 (see e.g. Fig. 5.9(b2) for NaCl and Fig. 5.9(c2) for KCl). Furthermore, the size of damage zone here is also larger than that shown in Fig. 5.9 (e.g. comparing Fig. 5.9(a1) with Fig. 5.2(a1) for MgO, and comparing Fig. 5.9(b1) with Fig. 5.2(b1) for NaCl). It is also noted that in Figs. 5.9(b1)-(b2) for NaCl a long lateral crack propagated along a {100} cleavage plane parallel to the left face and had connected the two adjacent corners. It is expected that when more repeated impacts test is carried out, or the impact velocity is increased further, the long cleavage crack will produce a larger platelet from that face.

5.5.2.3 Transition velocities

Chipping to fragmentation It was found that at high impact velocities, in addition to chipping, other attrition mechanisms such as fragmentation by propagation of radial and cleavage cracks became operative. It appears that there is a threshold velocity, \( v_c \), for the transition from chipping to fragmentation. However, the determination of the exact threshold velocity was found to be difficult because of the particle shape being cubic. The loading conditions of the corner on impact may also differ from test to test, hence producing a variation of the observed transition velocity. Another complicating factor is the relative contribution of radial and cleavage cracks to fragmentation. In addition, the transition velocity is also dependent on the number of impacts. Repeated impacts promote nucleation and propagation of cleavage cracks, and therefore they could lower the threshold velocity for the transition from chipping to fragmentation, particularly when the impact velocity is near the upper boundary of the velocity range for chipping. At these velocities, the damage mechanism is initially by chipping, but as the number of impacts increases there is a sudden switch to fragmentation. Based on damage observations for ten impacts, it is concluded that the threshold velocities for the transition from chipping to fragmentation for 2 mm cubic particles of MgO, NaCl and KCl are about 18 m s\(^{-1}\), 12 m s\(^{-1}\) and 10 m s\(^{-1}\), respectively. In the attrition tests reported here, once a particle was fragmented in the test, it was taken out from the sample for subsequent impacts. However, the velocity range was carefully selected so as to avoid fragmentation in the testing.
Fig. 5.9 Impact damage to MgO, NaCl and KCl crystals after 10 impacts near the upper boundary of the velocity range of chipping. (a1): MgO at 18 m s\(^{-1}\); (b1): NaCl at 12 m s\(^{-1}\); (c1): KCl at 10 m s\(^{-1}\). (a2), (b2) and (c2) show corners of (a1), (b1) and (c1) at higher magnifications.
**Plastic flow to chipping** At very low impact velocities, the stress generated on impact may not be sufficient to exceed the yield strength, and therefore the response will be purely elastic and no permanent damage is expected. However, the materials used here are cubic with sharp corners because the surfaces are cleavage planes. Therefore, when a corner or an edge of a particle is subjected to impact, it is likely that local stresses exceed the plastic yield stress and hence plastic deformation ensues. However, this process does not produce attrition debris and therefore the attrition rate is effectively zero. As the impact velocity is increased, the plastic zone size increases, and similar to the indentation fracture, a critical size and hence a critical load are reached where the propagation of lateral cracks occur, thus producing debris. This point is considered to be the transition from the plastic deformation to chipping, $v_0$. The transition velocities are shown schematically in Fig. 5.10.

![Fig. 5.10 Schematic illustration of the dependence of attrition mechanism on impact velocity and number of impacts.](image)

Fig. 5.10 Schematic illustration of the dependence of attrition mechanism on impact velocity and number of impacts.
5.5.3 Dependence of attrition rate on particle size

The model of impact attrition presented in Chapter 2 indicates a linear relationship between the fractional loss per impact and the particle size. In the experimental work described in the last two sections, a single particle size of 2 mm was used to determine the dependence of the attrition rate on the material properties (see in Section 5.5.1) and on the impact velocity (see in Section 5.5.2). To examine the effect of particle size, it was necessary to measure the attrition rate for several particle sizes while keeping other parameters constant. The work on impact attrition of 2 mm particles of MgO, NaCl and KCl at the impact velocity of 5.5 m s\(^{-1}\), described in Section 5.5.1, was therefore extended to larger particles, i.e. 3 mm, 4 mm and 5 mm. A photograph of freshly cleaved specimens of NaCl crystals, obtained from optical microscopy, is shown in Fig. 5.11. Particles of the other two test materials are similar to those shown in this figure. Details of the work are described below.

Fig. 5.11 Freshly cleaved 2-5 mm cubes of NaCl for the study of size effect.
5.5.3.1 Determination of mean particle size

Manual cleaving produces unavoidable variations in particle size, and therefore there is always a narrow distribution of the actual particle size for each nominal size. To use the nominal size in the analysis the standard deviation should be sufficiently small for the tests to be meaningful. As stated in Section 5.3, particles with a variation greater than 0.2 mm in any of the three dimensions were discarded during the preparation stage. An average particle size was then calculated to check for deviations from the nominal size. This can be done on a mass or a number basis. The former is used here as the analysis of attrition rate is carried out gravimetrically. The mass mean size is determined from the total number of particles, \( n_p \), and the original mass, \( M(0) \), by the form of:

\[
\bar{d} = \left[ \frac{M(0)}{\rho \cdot n_p} \right]^{\frac{1}{3}}
\]

where \( \rho \) is the particle density. It was found that the differences between the mean sizes and the nominal values, \( i.e. \) 2 mm, 3 mm, 4 mm and 5 mm, were smaller than 0.1 mm. Consequently, the nominal sizes were used in the analysis.

5.5.3.2 Results of size effect

Both \( \xi_{0,N} \) and \( \xi_{N, N+5} \) have been used here for the assessment of fractional loss per impact. The results are given in Tables 5.7 to 5.9 for \( \xi_{N, N+5} \) and in Tables 5.10 to 5.12 for \( \xi_{0,N} \). Equations 5.3 and 5.5 are used for the calculation of \( \xi_{0,N} \) and \( \xi_{N, N+5} \), respectively.

Some of these tests were repeated to examine the reproducibility of the results. This information is also shown in Tables 5.7 and 5.10 for 5 mm NaCl particles, in Tables 5.8 and 5.11 for 2 mm and 5 mm KCl particles respectively, and in Tables 5.9 and 5.12 for 2 mm MgO particles. The results of the original and repeat tests are denoted by the terms 'orig.' and 'rep.' in these tables. The reproducibility is marginally acceptable. It is possible to improve on this by using a larger number of crystals (see below). However, this is limited by the time-consuming process of cleaving the ingots.
Table 5.7 Fractional loss per impact $\xi_{N,N+5}$ for NaCl at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>$2$ mm</th>
<th>$3$ mm</th>
<th>$4$ mm</th>
<th>$5$ mm (orig.)</th>
<th>$5$ mm (rep.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5} / 10^{-4}$</td>
<td>3.3</td>
<td>3.67</td>
<td>5.22</td>
<td>6.08</td>
<td>5.76</td>
</tr>
<tr>
<td>$\xi_{5.10} / 10^{-4}$</td>
<td>4.3</td>
<td>6.87</td>
<td>8.26</td>
<td>10.43</td>
<td>8.41</td>
</tr>
<tr>
<td>$\xi_{10.15} / 10^{-4}$</td>
<td>5.7</td>
<td>8.65</td>
<td>9.12</td>
<td>8.36</td>
<td>12.80</td>
</tr>
<tr>
<td>$\xi_{15.20} / 10^{-4}$</td>
<td>6.2</td>
<td>9.20</td>
<td>9.52</td>
<td>11.74</td>
<td>10.50</td>
</tr>
</tbody>
</table>

Table 5.8 Fractional loss per impact $\xi_{N,N+5}$ for KCl at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>$2$ mm (orig.)</th>
<th>$2$ mm (rep.)</th>
<th>$3$ mm</th>
<th>$4$ mm</th>
<th>$5$ mm (orig.)</th>
<th>$5$ mm (rep.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5} / 10^{-4}$</td>
<td>0.48</td>
<td>0.75</td>
<td>0.83</td>
<td>1.18</td>
<td>1.46</td>
<td>2.18</td>
</tr>
<tr>
<td>$\xi_{5.10} / 10^{-4}$</td>
<td>1.17</td>
<td>2.29</td>
<td>2.43</td>
<td>2.36</td>
<td>4.13</td>
<td>3.17</td>
</tr>
<tr>
<td>$\xi_{10.15} / 10^{-4}$</td>
<td>2.28</td>
<td>2.51</td>
<td>2.95</td>
<td>4.15</td>
<td>3.93</td>
<td>5.06</td>
</tr>
<tr>
<td>$\xi_{15.20} / 10^{-4}$</td>
<td>2.73</td>
<td>3.67</td>
<td>3.95</td>
<td>5.05</td>
<td>4.74</td>
<td>6.61</td>
</tr>
</tbody>
</table>

Table 5.9 Fractional loss per impact $\xi_{N,N+5}$ for MgO at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>$2$ mm (orig.)</th>
<th>$2$ mm (rep.)</th>
<th>$3$ mm</th>
<th>$4$ mm</th>
<th>$5$ mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5} / 10^{-4}$</td>
<td>1.6</td>
<td>1.8</td>
<td>2.14</td>
<td>2.16</td>
<td>2.29</td>
</tr>
<tr>
<td>$\xi_{5.10} / 10^{-4}$</td>
<td>2.4</td>
<td>2.5</td>
<td>2.43</td>
<td>2.96</td>
<td>3.19</td>
</tr>
<tr>
<td>$\xi_{10.15} / 10^{-4}$</td>
<td>2.2</td>
<td>3.2</td>
<td>3.65</td>
<td>3.19</td>
<td>3.30</td>
</tr>
<tr>
<td>$\xi_{15.20} / 10^{-4}$</td>
<td>2.8</td>
<td>3.2</td>
<td>3.09</td>
<td>3.45</td>
<td>3.93</td>
</tr>
</tbody>
</table>
Table 5.10 Fractional loss per impact $\xi_{0,N}$ for NaCl at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>2 mm (orig.)</th>
<th>3 mm (rep.)</th>
<th>4 mm (orig.)</th>
<th>5 mm (orig.)</th>
<th>5 mm (rep.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5}$ / $10^{-4}$</td>
<td>3.3</td>
<td>3.67</td>
<td>5.22</td>
<td>6.08</td>
<td>5.76</td>
</tr>
<tr>
<td>$\xi_{0.10}$ / $10^{-4}$</td>
<td>3.81</td>
<td>5.26</td>
<td>6.73</td>
<td>8.24</td>
<td>7.07</td>
</tr>
<tr>
<td>$\xi_{0.15}$ / $10^{-4}$</td>
<td>4.44</td>
<td>6.38</td>
<td>7.51</td>
<td>8.26</td>
<td>8.70</td>
</tr>
<tr>
<td>$\xi_{0.20}$ / $10^{-4}$</td>
<td>4.87</td>
<td>7.06</td>
<td>7.98</td>
<td>9.09</td>
<td>9.12</td>
</tr>
</tbody>
</table>

Table 5.11 Fractional loss per impact $\xi_{0,N}$ for KCl at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>2 mm (orig.)</th>
<th>2 mm (rep.)</th>
<th>3 mm (orig.)</th>
<th>4 mm (orig.)</th>
<th>5 mm (orig.)</th>
<th>5 mm (rep.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5}$ / $10^{-4}$</td>
<td>0.48</td>
<td>0.75</td>
<td>0.83</td>
<td>1.18</td>
<td>1.46</td>
<td>2.18</td>
</tr>
<tr>
<td>$\xi_{0.10}$ / $10^{-4}$</td>
<td>0.83</td>
<td>1.52</td>
<td>1.63</td>
<td>1.77</td>
<td>2.80</td>
<td>2.68</td>
</tr>
<tr>
<td>$\xi_{0.15}$ / $10^{-4}$</td>
<td>1.31</td>
<td>1.85</td>
<td>2.07</td>
<td>2.56</td>
<td>3.17</td>
<td>3.46</td>
</tr>
<tr>
<td>$\xi_{0.20}$ / $10^{-4}$</td>
<td>1.66</td>
<td>2.30</td>
<td>2.53</td>
<td>3.18</td>
<td>3.56</td>
<td>4.25</td>
</tr>
</tbody>
</table>

Table 5.12 Fractional loss per impact $\xi_{0,N}$ for MgO at velocity of 5.5 m s$^{-1}$.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>2 mm (orig.)</th>
<th>2 mm (rep.)</th>
<th>3 mm (orig.)</th>
<th>4 mm (orig.)</th>
<th>5 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\xi_{0.5}$ / $10^{-4}$</td>
<td>1.6</td>
<td>1.8</td>
<td>2.14</td>
<td>2.16</td>
<td>2.29</td>
</tr>
<tr>
<td>$\xi_{0.10}$ / $10^{-4}$</td>
<td>2.01</td>
<td>2.15</td>
<td>2.29</td>
<td>2.56</td>
<td>2.74</td>
</tr>
<tr>
<td>$\xi_{0.15}$ / $10^{-4}$</td>
<td>2.08</td>
<td>2.46</td>
<td>2.74</td>
<td>2.77</td>
<td>2.92</td>
</tr>
<tr>
<td>$\xi_{0.20}$ / $10^{-4}$</td>
<td>2.25</td>
<td>2.67</td>
<td>2.83</td>
<td>2.94</td>
<td>3.17</td>
</tr>
</tbody>
</table>
In order to visualize the effect of particle size on attrition, the results shown in Tables 5.7 to 5.12 are plotted in Figs. 5.12 and 5.13 in terms of instantaneous and cumulative attrition rates, respectively. The straight lines in these figures have been fitted by least-squares linear regression. The slopes of these fitted lines are also shown in the figures.

The feature which needs to be considered first is the difference between the results of the repeat tests and the original ones given in Tables 5.7 to 5.12. The systematic errors resulting from losses due to handling and weighing are much smaller than this difference. The source of this variance is considered to be the small number of particles used in the tests. The particles impact on the target predominantly on the corners and edges, suffering some damage on each impact. However, every individual impact does not necessarily lead to the detachment of a platelet. The platelet may remain attached until subsequent impacts may detach it by fracture due to bending or by other processes propagating the subsurface crack to a free surface. There is therefore a large variation in the amount of debris produced by different particles. This is perhaps the reason why a relatively larger random error has been found for the instantaneous fractional loss per impact given in Tables 5.7 to 5.9 than the cumulative fractional loss per impact given in Tables 5.10 to 5.12. An increase in the number of particles will produce a more statistically reliable average. However, this is limited by the slow rate of production of small particles by manual cleaving.

Alternatively, the variance can be reduced by increasing the number of impacts. This is however less satisfactory here because of the dependence of the fractional loss on the number of impacts. Nevertheless, the dependence on particle size can be better viewed in this way as shown in Fig. 5.14, where the fractional loss per impact, based on the average of 20 impacts, varies linearly with particle size.

Several features of the results are noteworthy:

(i) The fractional loss per impact increases with an increase in the number of impacts for all the three materials tested, but to a varying degree. This recalls the effect of the number of impacts addressed in Section 5.5.2.

(ii) The slopes of the fitted lines to the experimental data in Figs. 5.12 and 5.13 increase with the number of impacts, i.e. larger particles show more sensitivity to the impact number. This is similar to the effect of velocity reported in Section 5.5.2, where at high impact velocities particles show more sensitivity to the impact number than at low impact velocities.
Fig. 5.12 Dependence of fractional loss per impact $\xi_{N, N+5}$ on particle size: (a) NaCl; (b) KCl; (c) MgO.
- $N=0.5$; • $N=5,10$; + $N=10,15$; ◎ $N=15,20$
Fig. 5.13 Dependence of fractional loss per impact $\xi_{0,N+5}$ on particle size: (a) NaCl; (b) KCl; (c) MgO.
- $\bullet$: $N=0.5$; $\ast$: $N=0.10$; $+$: $N=0.15$; $\ast\ast$: $N=0.20$
Fig. 5.14 Dependence of fractional loss per impact $\xi_{0.20}$ on particle size for all three crystals.

(iii) The above two features are most significant for NaCl, KCl and least significant for MgO.

These features can be explained by the phenomenon of work-hardening. The response of the crystals tested in this work is elastic-plastic, where on each impact some plastic deformation takes place. As it was said before, work-hardening is most significant in NaCl and KCl crystals, and least appreciable in MgO crystals. This effect is reflected in the attrition characteristics of these crystals; as the number of impacts increases, the material becomes harder and hence chipping occurs more readily. Similarly, as the particle size increases, the kinetic energy of particles increases on each impact. Consequently, larger particles become harder at a faster rate than smaller ones. The rate of hardening depends however on the material. For the softest ionic crystal, KCl, the slopes of the straight lines in Figs. 5.12 and 5.13 always increase as the impact number increases. On the other hand, for the hardest ionic crystal MgO, the slopes do
not show this trend consistently. This is because at the low impact velocity of 5.5 m s\(^{-1}\), MgO does not undergo significant work-hardening for the particle size range considered, whilst KCl and NaCl crystals do.

The most striking feature of the theoretical model developed here is that this trend can in principle be predicted because the fractional loss parameter is proportional to hardness, and therefore an increase in hardness can be taken into account. However, the difficulty at present is to quantify the work hardening as a function of particle size, contact geometry, impact velocity, and material properties. This task is not straightforward and requires further attention in future work. However, the linear dependence of the fractional loss per impact on particle size can be clearly observed, thus supporting the model presented in Chapter 2.

**5.5.3.3 SEM observations of impact damage**

The SEM photographs of the particles after impacts for the different sizes are shown in Figs. 5.15, 5.16 and 5.17 for MgO, NaCl and KCl, respectively. The SEM photographs are for particle sizes of 2, 3, 4, 5 mm. The impact velocity here is 5.5 m s\(^{-1}\) which is lower than that used previously for Figs. 5.2 and 5.9 where the effects of material properties and impact velocity were shown for the 2 mm particles. The features of impact damage to the larger particles are similar to those described in Section 5.5.1.1 for the 2 mm particles. Again, the process of material removal is by chipping and with a semi-brittle failure mode. In addition to the region showing losses due to chipping, the presence of lateral and radial cracks can be readily observed. It is however the lateral cracks which are primarily responsible for the material removal (see e.g. Fig. 5.15(b2) for 3 mm MgO, Fig. 5.16(c2) for 4 mm NaCl and Fig. 5.17(b2) for 3 mm KCl).

The extent of impact damage to the particles can be clearly observed from these figures. It can also be seen that the extent of damage to the particles increases when the size of particles increases. For example, for MgO in Fig. 5.15, the detachment of materials due to the formation of lateral cracks is very clear for 4 mm and 5 mm cubes as shown in Figs. 5.15(c2)-(d2), comparing with those of 2 mm and 3 mm cubes as shown in Figs. 5.15(a2)-(b2). For KCl in Fig. 5.17, the material removal is extensive for the 4 mm cube as shown in Fig. 5.17(c2), while the 5 mm cube shows extensive cracking but little detachment as shown in Figs. 5.17(d1)-(d2). There is of course some statistical variation of the extent of damage on each corner of a cube.
Fig. 5.15 Impact damage to MgO crystals after 20 impacts at a velocity of 5.5 m s\(^{-1}\) showing the effect of particle size. (a1): 2 mm; (b1): 3 mm; (c1): 4 mm; (d1): 5 mm. (a2), (b2), (c2) and (d2) show a corner of (a1), (b1), (c1) and (d1) at higher magnifications.
Fig. 5.16 Impact damage to NaCl crystals after 20 impacts at a velocity of 5.5 m s\(^{-1}\) showing the effect of particle size. (a1): 2 mm; (b1): 3 mm; (c1): 4 mm; (d1): 5 mm. (a2), (b2), (c2) and (d2) show a corner of (a1), (b1), (c1) and (d1) at higher magnifications.
Fig. 5.17 Impact damage to KCl crystals after 20 impacts at a velocity of 5.5 m s\(^{-1}\) showing the effect of particle size. (a1): 2 mm; (b1): 3 mm; (c1): 4 mm; (d1): 5 mm. (a2), (b2), (c2) and (d2) show a corner of (a1), (b1), (c1) and (d1) at higher magnifications.
5.5.3.4 Discussion

The experimental work carried out here confirms the theoretical prediction of the effect of particle size within the particle size range tested. There is an added complication arising from the effect of number of impacts, but the results show clearly a linear dependence if the effect of particle size is considered for the same number of impacts.

There should be a lower limit of particle size below which the linear dependence of attrition rate on particle size does not hold, as briefly discussed in Section 2.3.4. This is because below this limit the impact energy may be insufficient to initiate and propagate subsurface lateral cracks from the impact zone. Therefore the results presented in figures 5.12 to 5.14 cannot be extrapolated to particle sizes below this limit. In the following, factors affecting this limit are discussed.

Hutchings (1992b) has recently analysed the threshold conditions for the lateral crack formation of a target material impacted by a spherical or rounded particle. This analysis is based on the results of Marshall et al. (1982) of the critical load for the onset of lateral crack formation under quasi-static indentation. In this analysis, the maximum plastic indentation force is considered to be given by the area of the indentation impression times hardness. Assuming that the kinetic energy of the particle is taken by the work done in forming the impact impression, a critical particle size for the formation of lateral cracks in the target is derived, as given by:

\[
d_{cl} \propto \left( \frac{H_t}{K_{tc}} \right)^{-2} \frac{E_t^{1/4}}{H_t^{1/4}} \rho^{-1/4} \nu^{-1/2}
\]  

(5.8)

where \( K_{tc}, H_t \) and \( E_t \) are the fracture toughness, hardness and Young's modulus of the target, respectively, and \( \nu \) is the impact velocity. The critical particle size is strongly dependent on the ratio of hardness to fracture toughness, \( H_t/K_{tc} \), which is commonly regarded as the "brittleness index". 'Brittle' materials are prone to chipping by the formation of subsurface lateral cracks, and have therefore a lower critical particle size than 'ductile' materials. For ductile materials, i.e. materials having low yield stress, plastic deformation occurs without the particle undergoing a substantial elastic compression, and therefore the residual tensile stresses are not sufficiently high to initiate lateral cracking. Therefore, ductile materials are not much prone to chipping. It is also worth noting that the critical size is inversely dependent on the impact
velocity. Therefore, as the impact velocity increases the critical particle size decreases.

The above analysis should equally be applicable to damage imparted to a particle when impacting on a rigid target as in the case of impact attrition considered here. However, extension of the application of eqn 5.8 to the determination of the threshold conditions for the formation of lateral cracks in impacting particles requires further experimental work.

In addition to this critical particle size which is dependent on the impact velocity, it has long been recognised that there is an ultimate critical particle size $d_{cb}$ below which the breakage of particles is impossible (Boddy, 1943; Parish, 1967; Kendall, 1978; Hagan, 1981). For example, Kendall (1978) proposed a model of the ultimate critical particle size $d_{cb}$ based on the uniaxial compression of a brittle particle which contained a pre-existing crack. He concluded that when the particle size was reduced below the critical size $d_{cb}$, the elastic energy stored in the particle was not sufficient to enable crack propagation. However, as pointed out by Hagan, this model overestimates the ultimate critical particle size because it is based on the threshold conditions for crack propagation rather than initiation. Based on the micromechanics of crack nucleation under indentation, Hagan (1981) presented another model of the critical particle size as given by:

$$d_{cb} \approx 30 \left( \frac{K_C}{H} \right)^2 \quad (5.9)$$

Equation 5.9 suggests that the critical particle size is a function of brittleness index.

Using the model of Hagan (1981), the ultimate critical particle size can be estimated for MgO, NaCl and KCl crystals based on the data of Table 2.1:

$$d_{cb} = 0.8 \, \mu m, \quad \text{for MgO}$$
$$d_{cb} = 27 \, \mu m, \quad \text{for NaCl}$$
$$d_{cb} = 59 \, \mu m, \quad \text{for KCl}$$

MgO is relatively brittle, and therefore the ultimate critical particle size is very small. A larger ultimate critical particle size is obtained for KCl which is the softest of the above three crystals. However, these critical sizes are far below the size range tested here, i.e. 2-5 mm. It is therefore difficult to specify the lower limit for which the
linear dependence of attrition rate on particle size is applicable. Because the size of particles which give rise to attrition problems in industry is usually much larger than these limits, the ultimate critical particle size is not much of interest to attrition process. For this reason, this critical particle size given by eqn 5.8 is more applicable. However, further verification of eqn 5.8 is required.

5.6 Influence of Rigidity of Target Materials on Attrition Rate

In the previous sections, sapphire was used as the target. Sapphire has a Vickers hardness of about four times and a Young's modulus of about twice that of MgO. This ensures that the target is rigid and that the contact deformation is predominately from the impacting particle, as required by the theoretical model proposed in Chapter 2. Furthermore, by choosing an essentially rigid target, the extreme case of causing the maximum amount of contact damage is considered. However, in most practical situations in processing and handling of particulate solids, attrition is caused by inter-particle collisions or by particle impact with walls. The hardness of particles is obviously comparable with each other, and in some cases it may also be comparable with those of the walls. Therefore, the influence of the mechanical properties of the target material (e.g. hardness) on the contact damage becomes increasingly important (see Hutchings, 1992a, for abrasive wear; and Wada, 1992, for erosion). For example, Wada (1992) has shown that the erosion rate of ceramic targets decreases remarkably when the hardness of the erosive particles decreases to an extent that it becomes similar to that of the target. In view of reported trends in the literature, it was decided to investigate the influence of hardness of target materials on the attrition rate. For this purpose, the impact attrition of single particles on the soda-lime glass was measured.

The soda-lime glass used here was a circular thin disc having 20 mm diameter and 1 mm thickness. It was obtained by cutting an optical microscopic glass slide. The hardness of the glass was measured by Vickers indentation with a load range of 5-20 N. The average value of Vickers hardness obtained was 5.6 GPa. This value is comparable with that of MgO, i.e. 5.8 GPa, and is also comparable with that of the glass target used by Lawn et al. (1980), i.e. 5.5 GPa. The soda-lime glass has a Young's modulus of 70 GPa which is about 3-4 times smaller than that of MgO (see Table 2.1).
Following the test procedure described in Section 5.3, single crystals of MgO, NaCl and KCl with a size of 2 mm were impacted on the glass target at three different velocities, *i.e.* 8.4 m s⁻¹, 5.5 m s⁻¹ and 4.1 m s⁻¹ in order to compare the results with those obtained in Section 5.5.1. The fractional loss per impact $\xi_{0-N}$ is shown in Table 5.13 for the 10 and 20 impacts basis. The results obtained here compare fairly well with those reported for sapphire (see Table 5.1). The results of MgO are shown in the form of a histogram in Fig. 5.18 for both target materials to facilitate the comparison. Because the hardness of MgO is comparable with that of the glass target, any difference in the impact attrition results between the two target materials should be most noticeable for MgO crystals. For NaCl and KCl crystals the glass target is already sufficiently hard so that no great differences are expected. It should be mentioned that the systematic error in Table 5.13 is relatively larger than that in Table 5.1. This is because the balance used for weighing in these tests had an accuracy of 1 $\times 10^{-4}$ g. This is one order of magnitude lower than that used for the tests reported in the previous sections. The fractional loss per impact was therefore based on a cycle of 10 rather than 5 impacts because the level of attrition obtained after 5 impacts was too low to be measured with any degree of accuracy. It should also be noted that the lowest impact velocity used here is slightly different from that of the previous tests reported in Table 5.1. The proportionality factor $\alpha$, has been calculated by the use of eqn 2.25, and is shown in Table 5.14. It can be seen that the mean values are close to those in Table 5.4.

The results obtained here show that the rate of attrition is not sensitive to the hardness of target materials in the range tested here, *i.e.* $4.35 \times 10^{-3} < \frac{H_p}{H_t} \leq 1$. The case of $\frac{H_p}{H_t}$ greater than unity has not been tested here. The conclusion here is therefore different from the findings of Wada (1992). It is worth noting that the velocity range used here is much lower than that used by Wada for the erosion study, where the impact velocity was about 300 m s⁻¹. It is of interest to explore the influence of the target hardness on attrition in the range of $\frac{H_p}{H_t} > 1$. However, in most practical applications, where $\frac{H_p}{H_t} \leq 1$, this work shows that there is no appreciable influence of $\frac{H_p}{H_t}$ on the rate of attrition.
Table 5.13 Fractional loss per impact $\xi_{0,N}$ for 2 mm particles impacting on a glass target.

(a) based on 10 impacts.

<table>
<thead>
<tr>
<th>$\xi / 10^{-4}$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>5.3 ± 0.3</td>
<td>12.3 ± 0.3</td>
<td>5.4 ± 0.4</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>2.5 ± 0.2</td>
<td>4.7 ± 0.3</td>
<td>1.5 ± 0.3</td>
</tr>
<tr>
<td>$v = 4.1 \text{ m s}^{-1}$</td>
<td>1.2 ± 0.2</td>
<td>2.2 ± 0.3</td>
<td>0.8 ± 0.3</td>
</tr>
</tbody>
</table>

(b) based on 20 impacts

<table>
<thead>
<tr>
<th>$\xi / 10^{-4}$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>5.3 ± 0.1</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>3.0 ± 0.1</td>
<td>5.8 ± 0.1</td>
<td>1.9 ± 0.2</td>
</tr>
<tr>
<td>$v = 4.1 \text{ m s}^{-1}$</td>
<td>1.2 ± 0.1</td>
<td>2.8 ± 0.1</td>
<td>1.3 ± 0.2</td>
</tr>
</tbody>
</table>

Table 5.14 Proportionality factor $\alpha$ based on the data in Table 5.13.

(a) based on 10 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>1.0</td>
<td>4.4</td>
<td>2.4</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>1.1</td>
<td>3.9</td>
<td>1.6</td>
</tr>
<tr>
<td>$v = 4.1 \text{ m s}^{-1}$</td>
<td>0.9</td>
<td>3.1</td>
<td>1.5</td>
</tr>
<tr>
<td>Mean value</td>
<td>1.0</td>
<td>3.8</td>
<td>1.8</td>
</tr>
</tbody>
</table>

(b) based on 20 impacts

<table>
<thead>
<tr>
<th>$\alpha = \xi/\eta$</th>
<th>MgO</th>
<th>NaCl</th>
<th>KCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>$v = 8.4 \text{ m s}^{-1}$</td>
<td>1.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$v = 5.5 \text{ m s}^{-1}$</td>
<td>1.3</td>
<td>4.8</td>
<td>2.0</td>
</tr>
<tr>
<td>$v = 4.1 \text{ m s}^{-1}$</td>
<td>0.9</td>
<td>4.0</td>
<td>2.5</td>
</tr>
<tr>
<td>Mean value</td>
<td>1.1</td>
<td>4.4</td>
<td>2.3</td>
</tr>
</tbody>
</table>
Fig. 5.18 Effect of rigidity of target materials on attrition rate at different impact velocities for 2 mm MgO crystals. The lowest velocity used for tests with glass target is in the brackets.
5.7 Conclusions

Single particle impact tests have been used to assess the rate of impact attrition of particulate solids. The trends of the experimental results agree with the predictions of the mechanistic model of impact attrition developed in Chapter 2. More specifically, the dependence of attrition rate on material properties, impact velocity and particle size have been verified for ionic crystals of MgO, NaCl and KCl.

It has been shown that the impact attrition does not generally follow a first order rate process. For the range of conditions tested here, the attrition rate of crystals of MgO which are the hardest of the three materials tested here follows approximately a first order rate, while those of NaCl and KCl follow higher order rates. The number of impacts influences the rate of attrition to varying degrees, depending on impact velocity and particle size. This is believed to be due to the work-hardening effect, where on each impact the hardness increases. This is in fact aptly predicted by the theoretical model, where the dimensionless attrition propensity parameter \( \eta \) is proportional to the hardness. The change of hardness is reflected in the variation of the proportionality factor \( \alpha \) with the number of impacts. It is in principle possible to account for the change of hardness with the number of impact, but further work is needed to quantify this property. However, the important finding here is the existence of a unique lower limit for \( \alpha \) which is about 1 for all the tested materials. This supports the linear assumption between \( \xi \) and \( \eta \), and the capability of \( \eta \) to describe the attrition propensity.

The impact velocity has a strong influence on the fractional loss per impact. A threshold velocity for the transition of breakage mechanism from chipping to fragmentation has been observed. In the chipping regime, the attrition rate determined experimentally follows a power law dependence on the impact velocity with an index of around 2. There is however a variation in the power index with the number of impacts due to the work-hardening effect. It has been found that the lower limit of the power index, which applies to the lowest number of impacts tested, \( i.e. \ 5 \), has a value of 2, which is in a remarkable agreement with the theory.

Particle size is another factor influencing the fractional loss per impact. A linear relationship between the fractional loss per impact and particle size has been verified for a particle size range of 2-5 mm. A lower limit for the applicability of the linear relationship between the fractional loss per impact and particle size is expected.
However, this limit has not been specified in this work, although it can in principle be determined from the consideration of minimum load or impact velocity that can cause lateral fracture. There is an ultimate critical particle size below which the particles do not break at all. This critical size has been estimated by applying the model proposed by Hagan (1981). However, this limit has little practical significance in attrition, because the sizes of the particles of interest in attrition are usually much larger than this limit.

The influence of rigidity of target materials on the attrition rate was assessed. In contrast to erosion, the rate of attrition was found to be insensitive to the hardness of target materials even when the ratio of hardness of impacting particle to that of target approached unity from a value far below unity. The case of the ratio of hardness of the particle to that of target being greater than unity was not tested.
CHAPTER 6

EFFECT OF PARTICLE SIZE ON IMPACT ATTRITION
OF SOLUTION-GROWN NaCl CRYSTALS

6.1 Introduction

In the experimental work on impact attrition presented in Chapter 5, high purity melt-
grown single crystals have been used for the verification of the model of impact
attrition. However, solution-grown crystals are produced more commonly on a large
scale in industry, but are generally much less perfect than the melt-grown crystals.
Their surfaces, corners and edges are often extensively damaged during the
production stage, in addition to less perfect structure that is produced due to the
presence of impurities, as further discussed below. It is therefore of great interest to
investigate their impact attrition behaviour.

In a parallel research programme the attrition behaviour of several solution-grown
crystals has been investigated (Ghadiri et al., 1991; Arteaga et al. 1993). One of the
test materials was a solution-grown NaCl salt produced commercially by formerly ICI,
and now Salt Union Ltd, at their Weston Point Salt Works, Cheshire. This salt is
commonly known as Pure Dried Vacuum (PDV) salt. Production of this material is
accompanied by the formation of fines with a size below about 100 µm, resulting
from attrition during processing and handling stages. Presence of fines degrades the
product quality and causes environmental and processing problems.

In this work, the effect of particle size on the fractional loss per impact has been
investigated. The results are reported below and are interpreted by the theoretical
model developed in Chapter 2.
6.2 Experimental

6.2.1 Materials
PDV salt is produced by crystallization from a salt solution (i.e. the brine). The salt crystals are then separated from the liquor and dried via three routes: in route 1 the crystals are centrifuged and fluid-bed dried only; in route 2 the crystals are centrifuged, fluid-bed dried and cooled; and in route 3 they are filtered by a rotary vacuum filter and dried in situ. The salt from route 3 (referred to as Type III PDV salt) is less damaged, and receives less thermal shock than the other two because the process conditions are more gentle. This type of salt has therefore been chosen for testing here. PDV salt has a wide size distribution (90 µm to 850 µm) and this allows testing of at least a few size cuts.

PDV salt crystals contain macroscopic flaws such as occlusions and crevasses within the crystal and surface damage. In fact, previous investigations have revealed a structure shown in Fig. 6.1, even for apparently single crystals, where a relatively good quality seed crystal of PDV salt is surrounded by a heavily flawed outer layer (Yuregir et al., 1986). It has been shown that, at impact velocities above 20 m s⁻¹, the outer layer is easily shed in the form of platelets, although complete fragmentation of the crystals can also occur. PDV salt also contains multiple crystals of the type shown in Fig. 6.2. The inter-crystalline bond breaks easily at low impact velocities. It has been found that the platelets form readily during impact of PDV salt particles, similar to the more perfect melt-grown crystals. Therefore, as long as the impact velocity is kept sufficiently low so as not to cause complete fragmentation, this material may be appropriate for verifying the model predictions because the damage results primarily from chipping, i.e. resulting from the formation of subsurface lateral cracks.

The presence of multiple crystals is most abundant in PDV Type III salt, and furthermore the mass fraction of multiple crystals increases with the particle size. This causes some difficulty in interpreting the attrition results as will be seen below, but it has been overcome by adopting a special experimental procedure which is described below.
Fig. 6.1 PDV salt crystal observed under transmitted light, soaked in dibutyl phthalate to enhance the observation of the subsurface flaws within the crystal (After Yuregir et al., 1986).

Fig. 6.2 SEM view of PDV salt showing the multiple crystals (After Ghadiri et al., 1991).
6.2.2 Test procedure

In contrast to the impact attrition tests of large particles presented in Chapter 5, the problem encountered here in testing of PDV salt particles is the handling losses during the test operation. A fine filter has therefore been used in the collection chamber of the test rig as shown previously in Fig. 3.1 so that debris larger than about 0.45 µm are all retained. This ensures the handling losses of particles are reduced to a low level of about 0.04% at velocities below 10 m s\(^{-1}\) tested here.

Samples were prepared by hand sieving (Allen, 1990) using British Standard sieves (BS410/1986). Hand sieving was a time-consuming process, but it was necessary in order to reduce attrition during the sieving operation, and also to prepare particles having 'near mesh' sizes. Samples of different near mesh sizes, i.e. 500 µm, 425 µm and 355 µm were produced by this method. About 5-10 g of near mesh size particles were impacted repeatedly up to 35 impacts at a velocity of 2.7 m s\(^{-1}\). The choice of this low impact velocity was to minimise fragmentation by the propagation of cleavage cracks, particularly at large numbers of impact. After each impact, the products were collected very carefully to minimise the handling losses, and then weighed by the high precision balance which has already been described in Section 5.3. The mother particles were then separated from the debris by using a sieve, whose size was one sieve size below the standard size used for preparing the near mesh mother particles, i.e. for the case of 500 µm particles, the sieve size 425 µm was used.

The procedure for sieving is as follows. The 425 µm sieve is nested on to a catch pan, which has been pre-tared to zero, and the particles are placed on top of the sieve. The sieve is held slightly inclined to the horizontal and rapped gently with a cylindrical piece of wood about 450 mm long and 18 mm diameter. The amount passing through the mesh is weighed after one minute of sieving and this is continued until the passage per minute is about 3-4% of the total amount passed during the previous minute. The particles remaining on the top of the sieve are the surviving mother particles, whose corners have been chipped off. These particles are used as the feed material for the following impact (see Fig. 6.3). The process is repeated for 35 impacts. Following the same procedure, 425 µm and 355 µm near mesh size particles were analysed using 355 µm and 300 µm sieves, respectively. The impact velocity was kept constant at 2.7 m s\(^{-1}\).

Optical observations of PDV salt particles have shown that a large number of multiple crystals exist in the raw material, whose number frequency increases as the particle size increases. The attrition mechanism of interest here is chipping of single particles,
and not splitting of multiple crystals. The bond strength for most multiple crystals is very weak and the bond can be broken easily even at the lowest impact velocities available in the apparatus, which corresponds to the free falling speed of the particles in the tube. This is typically less than 3 m s$^{-1}$ for the particle sizes tested here. However, the method of repeated impacts can overcome this difficulty provided the behaviour of the particles at large numbers of impacts is considered in the analysis, rather than that at the first few impacts, where losses from the mother particles are mainly due to the breakdown of multiple crystals. The repeated impact process is illustrated in Fig. 6.3. The masses of particles at various stages of the test are defined in following:

$M_f(n)$ — Mass of feed particles for the $n^{th}$ impact
$M_a(n)$ — Mass collected after the $n^{th}$ impact
$M_b(n)$ — Mass of surviving mother particles after the $n^{th}$ impact
$M_s(n)$ — Mass of debris after the $n^{th}$ impact and sieving losses
$M_h(n)$ — Mass of handling losses during one impact stage

![Fig. 6.3 Schematic illustration of the repeated impact process.](image)
As seen in Fig. 6.3, the feed material of the \( n^{th} \) impact is the surviving mother particles from the \((n-1)^{th}\) impact. The mass balance for the \( n^{th} \) impact is:

\[
M_f(n) = M_d(n) + M_h(n)
\]

or,

\[
M_f(n) = M_b(n) + M_s(n) + M_h(n) \quad (6.1)
\]

\( M_f(n) \), \( M_d(n) \) and \( M_b(n) \) are measured by the use of the balance, and \( M_h(n) \) and \( M_s(n) \) can then be calculated from eqn 6.1.

### 6.3 Results and Analysis

The results of repeated impact tests for the feed material sizes of 500 µm, 425 µm and 355 µm are shown in Tables 6.1, 6.2 and 6.3, respectively. The unit of mass in these tables is gram. The handling losses are generally small, and with a continuous improvement in the test procedure, they have been reduced to about 0.04%. However, the amount of fines produced per impact (see Tables 6.1, 6.2 and 6.3), although much larger than the handling losses, is nevertheless in some cases in the same order of magnitude as the handling losses. This inadvertently reduces the accuracy of the attrition measurements. Taking account of the handling loss, the fractional loss per impact \( \xi \) may be calculated in two ways. If all the handling losses are considered to be fines, then an upper limit of the fractional loss per impact between the \( m^{th} \) impact and \( n^{th} \) impact can be expressed as:

\[
\xi^{(+)}_{n,m} = \frac{M_f(n) - M_b(m)}{M_f(n)} \cdot \frac{1}{m - n + 1} \quad (m \geq n) \quad (6.2)
\]

On the other hand, if the amount which is lost is considered to be in the form of mother particles, then a lower limit of fractional loss per impact is applicable. This could arise from mishandling of the feed particles, and therefore the particles are not attritted during the process.

\[
\xi^{(-)}_{n,m} = \frac{M_f(n) - \sum_{n}^{m} M_h(n) - M_b(m)}{M_f(n) - \sum_{n}^{m} M_h(n)} \cdot \frac{1}{m - n + 1} \quad (m \geq n) \quad (6.3)
\]
Based on the above formulae, the upper and lower limits of fractional loss per impact can be calculated for each test, and these results are also shown in Tables 6.1, 6.2 and 6.3. The error margin is generally satisfactory. However, it can clearly be seen that in order to improve the accuracy of the measurements, it is essential to reduce the handling losses as much as possible. In practice, the actual fractional loss is somewhere between these limits. Its proximity to one of these limits depends on the size of mother particles and on the amount and size of debris. In this work, extreme care was taken not to lose the mother particles, and experience suggests that the losses are primarily due to the loss of debris and therefore the upper limit of the fractional loss per impact, i.e. \( \varepsilon_{n,m}^{(+)} \) is more applicable.

In order to compare the fractional losses for the three different particle sizes, the results are also shown in Fig. 6.4, where the fractional losses per impact are plotted as a function of the number of impacts. The results show a number of very interesting features. There is a significant decrease in the fractional loss in the early stages of the repeated impact process, but the rate of attrition reduces and eventually reaches an asymptotic value for all the sizes. The very high initial losses are due to the breakage of the multiple crystals. As more multiple crystal particles exist in the 500 \( \mu \text{m} \) size cut than in the 425 \( \mu \text{m} \) and 355 \( \mu \text{m} \) size cuts, the amount of attrition is higher and the slope is steeper for the 500 \( \mu \text{m} \) cut in the first few impacts. This is consistent with the observations made by optical microscopy. After about 20 impacts the fractional losses per impact approach asymptotic values of about 0.255\%, 0.195\% and 0.165\% for the three size cuts of 500 \( \mu \text{m} \), 425 \( \mu \text{m} \) and 355 \( \mu \text{m} \), respectively.

Another interesting feature is that the fractional losses for the PDV salt are about one order of magnitude larger than those of the melt-grown salt, given in Chapter 5, even after reaching the asymptotic value. Moreover the particle size of PDV salt is much smaller than that of the melt-grown particles tested previously. Because of very high attrition rates, a much lower impact velocity was used for the PDV salt than those tested previously. The melt-grown crystals have virtually no macroscopic flaws, and are therefore much stronger than the solution-grown crystals, whose outer layers are dominated by the presence of crevasses.
Table 6.1 Results of repeated impact attrition testing of 500 µm PDV Type III salt at impact velocity of 2.7 m s\(^{-1}\) (value of masses given below is in gram).

<table>
<thead>
<tr>
<th>n</th>
<th>(M_f(n))</th>
<th>(M_a(n))</th>
<th>(M_b(n))</th>
<th>(M_s(n))</th>
<th>(\sum M_h(n))</th>
<th>(\xi(+)), %</th>
<th>(\xi(-)), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10.5349</td>
<td>10.4804</td>
<td>8.4573</td>
<td>2.0231</td>
<td>0.0545</td>
<td>(\xi_{1,1}=19.7)</td>
<td>19.3</td>
</tr>
<tr>
<td>10</td>
<td>8.4573</td>
<td>8.3963</td>
<td>7.3505</td>
<td>1.0458</td>
<td>0.0610</td>
<td>(\xi_{10,1}=1.5)</td>
<td>1.4</td>
</tr>
<tr>
<td>15</td>
<td>7.3505</td>
<td>7.3307</td>
<td>7.1567</td>
<td>0.1740</td>
<td>0.0198</td>
<td>(\xi_{15,1}=0.53)</td>
<td>0.48</td>
</tr>
<tr>
<td>20</td>
<td>7.1567</td>
<td>7.1500</td>
<td>6.9942</td>
<td>0.1558</td>
<td>0.0067</td>
<td>(\xi_{20,1}=0.46)</td>
<td>0.44</td>
</tr>
<tr>
<td>25</td>
<td>6.9942</td>
<td>6.9833</td>
<td>6.8977</td>
<td>0.0856</td>
<td>0.0109</td>
<td>(\xi_{25,1}=0.28)</td>
<td>0.25</td>
</tr>
<tr>
<td>30</td>
<td>6.8977</td>
<td>6.8839</td>
<td>6.8040</td>
<td>0.0799</td>
<td>0.0138</td>
<td>(\xi_{30,1}=0.27)</td>
<td>0.23</td>
</tr>
<tr>
<td>35</td>
<td>6.8040</td>
<td>6.7934</td>
<td>6.7118</td>
<td>0.0816</td>
<td>0.0106</td>
<td>(\xi_{35,1}=0.27)</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Table 6.2 Results of repeated impact attrition testing of 425 µm PDV Type III salt at impact velocity of 2.7 m s\(^{-1}\) (value of masses given below is in gram).

<table>
<thead>
<tr>
<th>n</th>
<th>(M_f(n))</th>
<th>(M_a(n))</th>
<th>(M_b(n))</th>
<th>(M_s(n))</th>
<th>(\sum M_h(n))</th>
<th>(\xi(+)), %</th>
<th>(\xi(-)), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.0032</td>
<td>5.0026</td>
<td>4.7245</td>
<td>0.2781</td>
<td>0.0006</td>
<td>(\xi_{1,1}=5.6)</td>
<td>5.6</td>
</tr>
<tr>
<td>5</td>
<td>4.7245</td>
<td>4.7189</td>
<td>4.5713</td>
<td>0.1476</td>
<td>0.0056</td>
<td>(\xi_{5,1}=0.81)</td>
<td>0.78</td>
</tr>
<tr>
<td>10</td>
<td>4.5713</td>
<td>4.5650</td>
<td>4.4716</td>
<td>0.0934</td>
<td>0.0063</td>
<td>(\xi_{10,1}=0.44)</td>
<td>0.41</td>
</tr>
<tr>
<td>15</td>
<td>4.4716</td>
<td>4.4595</td>
<td>4.3943</td>
<td>0.0652</td>
<td>0.0121</td>
<td>(\xi_{15,1}=0.35)</td>
<td>0.29</td>
</tr>
<tr>
<td>20</td>
<td>4.3943</td>
<td>4.3879</td>
<td>4.3403</td>
<td>0.0476</td>
<td>0.0064</td>
<td>(\xi_{20,1}=0.25)</td>
<td>0.22</td>
</tr>
<tr>
<td>25</td>
<td>4.3403</td>
<td>4.3359</td>
<td>4.2958</td>
<td>0.0401</td>
<td>0.0044</td>
<td>(\xi_{25,1}=0.21)</td>
<td>0.19</td>
</tr>
<tr>
<td>30</td>
<td>4.2958</td>
<td>4.2894</td>
<td>4.2507</td>
<td>0.0387</td>
<td>0.0064</td>
<td>(\xi_{30,1}=0.21)</td>
<td>0.18</td>
</tr>
<tr>
<td>35</td>
<td>4.2507</td>
<td>4.2401</td>
<td>4.2031</td>
<td>0.0370</td>
<td>0.0106</td>
<td>(\xi_{35,1}=0.22)</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Table 6.3 Results of repeated impact attrition testing of 355 µm PDV Type III salt at impact velocity of 2.7 m s\(^{-1}\) (value of masses given below is in gram).

<table>
<thead>
<tr>
<th>n</th>
<th>(M_f(n))</th>
<th>(M_a(n))</th>
<th>(M_b(n))</th>
<th>(M_s(n))</th>
<th>(\sum M_h(n))</th>
<th>(\xi(+)), %</th>
<th>(\xi(-)), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.0022</td>
<td>3.9980</td>
<td>3.8411</td>
<td>0.1569</td>
<td>0.0042</td>
<td>(\xi_{1,1}=4.0)</td>
<td>3.9</td>
</tr>
<tr>
<td>5</td>
<td>3.8411</td>
<td>3.8183</td>
<td>3.7599</td>
<td>0.0584</td>
<td>0.0228</td>
<td>(\xi_{5,1}=0.53)</td>
<td>0.31</td>
</tr>
<tr>
<td>10</td>
<td>3.7599</td>
<td>3.7445</td>
<td>3.7055</td>
<td>0.0390</td>
<td>0.0154</td>
<td>(\xi_{10,1}=0.29)</td>
<td>0.21</td>
</tr>
<tr>
<td>15</td>
<td>3.7055</td>
<td>3.6895</td>
<td>3.6603</td>
<td>0.0292</td>
<td>0.0160</td>
<td>(\xi_{15,1}=0.24)</td>
<td>0.16</td>
</tr>
<tr>
<td>20</td>
<td>3.6603</td>
<td>3.6454</td>
<td>3.6244</td>
<td>0.0210</td>
<td>0.0149</td>
<td>(\xi_{20,1}=0.20)</td>
<td>0.12</td>
</tr>
<tr>
<td>25</td>
<td>3.6244</td>
<td>3.6181</td>
<td>3.5907</td>
<td>0.0274</td>
<td>0.0063</td>
<td>(\xi_{25,1}=0.19)</td>
<td>0.15</td>
</tr>
<tr>
<td>30</td>
<td>3.5907</td>
<td>3.5851</td>
<td>3.5602</td>
<td>0.0249</td>
<td>0.0056</td>
<td>(\xi_{30,1}=0.17)</td>
<td>0.14</td>
</tr>
<tr>
<td>35</td>
<td>3.5602</td>
<td>3.5565</td>
<td>3.5287</td>
<td>0.0278</td>
<td>0.0037</td>
<td>(\xi_{35,1}=0.18)</td>
<td>0.16</td>
</tr>
</tbody>
</table>
In view of the strong dependence of fractional loss per impact on the number of impacts, it is appropriate to use the asymptotic values for the determination of the size effect. Unfortunately, the interval between the particle sizes and the number of sizes tested are not sufficiently large to draw a definite conclusion about the size effect. Nevertheless, the asymptotic values of fractional loss per impact are plotted in Fig. 6.5 against the particle size, where the data have been fitted with a straight line by least-squares linear regression. Similar to those of melt-grown crystals in Chapter 5, the results here suggest that the dependence of fractional loss per impact on particle size follows a linear relationship fairly well, and this is in agreement with that predicted by the theory.
Fig. 6.5 A plot of fractional loss per impact as a function of particle size for PDV Type III salt.

6.4 Discussion

Handling losses are an important factor influencing the accuracy of the determination of fractional loss per impact. By improving the experimental procedure, and by careful experimental work, the handling losses have been reduced to 0.04% per impact. Nevertheless, this value amounts to about 15% of the value of the fractional loss per impact. This is significant because it leads to the overlapping of error bars for data points from adjacent particle sizes as shown in Fig. 6.4. To reduce the handling losses further, it is necessary to improve the experimental procedure. Alternatively, for the purpose of the study of size effect, the difference in particle size should be increased to improve the resolution of the measurements when investigating the size effect. However this is not possible for PDV salt as a very small fraction of particles lies above 500 µm.

The morphology of the surfaces of the feed particles is slightly different for different particle size cuts, as observed by incident light optical microscopy. It appears that the 500 µm particles have been subjected to more surface damage than the 355 µm particles during processing. The presence of higher rates of attrition in the early stages of repeated impacts for larger particles could be partly due to this effect,
although the breakdown of multiple crystals is the most significant contributor during this stage. However, the important feature of the results is that the fractional loss per impact has eventually approached a constant rate. It would be of interest to see whether the rate of breakdown remains constant if the number of impacts is further increased, and to compare the results with those of the melt-grown crystals. However, this was not attempted here because the corners and edges of the crystals were so much damaged that the propensity to fragmentation was becoming appreciable.

The process of attrition under investigation here is chipping of the corners and edges of the particles, without leading to the breakage of the mother particles into several fragments. Therefore, careful optical observations on the damaged particles have been carried out after each cycle of impact to ensure that tests are carried out at sufficiently low impact velocity so that no fragmentation takes place. These observations showed this to be the case at the low velocity of 2.7 m s\(^{-1}\) to which the above results relate. Some tests were also carried out initially at a higher velocity of 10 m s\(^{-1}\) on the 500 µm particles, where it was found that a large number of particles fragmented on every impact. At this velocity, the upper limit of fractional loss per impact \(\xi_{n,n}^{(+)}\) for 10 repeated impacts, and the size analysis after 10 impacts are shown in Tables 6.4 and 6.5, respectively. Microscopic observations as shown in Fig. 6.6, confirm that the high value of fractional loss includes particle fragmentation. This has not been accounted for in the theoretical model and therefore the results cannot be used for the verification of the model. Nevertheless, this test has provided data regarding the transition of breakage mechanism from chipping to fragmentation for the PDV salt.

### Table 6.4 Breakage rate of 500 µm PDV Type III salt particles at impact velocity of 10 m s\(^{-1}\) (mass of particles is in gram).

<table>
<thead>
<tr>
<th>n</th>
<th>(M_f(n))</th>
<th>(M_a(n))</th>
<th>(M_b(n))</th>
<th>(\xi_{n,n}^{(+)}), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.0076</td>
<td>4.9010</td>
<td>3.3240</td>
<td>33.6</td>
</tr>
<tr>
<td>2</td>
<td>3.3240</td>
<td>3.2705</td>
<td>2.7650</td>
<td>16.8</td>
</tr>
<tr>
<td>3</td>
<td>2.7650</td>
<td>2.7342</td>
<td>2.3530</td>
<td>14.9</td>
</tr>
<tr>
<td>4</td>
<td>2.3530</td>
<td>2.3530</td>
<td>2.0443</td>
<td>13.1</td>
</tr>
<tr>
<td>5</td>
<td>2.0443</td>
<td>2.0338</td>
<td>1.7890</td>
<td>12.5</td>
</tr>
<tr>
<td>6</td>
<td>1.7890</td>
<td>1.7657</td>
<td>1.5760</td>
<td>11.9</td>
</tr>
<tr>
<td>7</td>
<td>1.5760</td>
<td>1.5560</td>
<td>1.4025</td>
<td>11.0</td>
</tr>
<tr>
<td>8</td>
<td>1.4025</td>
<td>1.4001</td>
<td>1.2681</td>
<td>9.6</td>
</tr>
<tr>
<td>9</td>
<td>1.2681</td>
<td>1.2670</td>
<td>1.1405</td>
<td>10.1</td>
</tr>
<tr>
<td>10</td>
<td>1.1405</td>
<td>1.1287</td>
<td>1.0453</td>
<td>8.4</td>
</tr>
</tbody>
</table>
Table 6.5  Size analysis of 500 µm PDV Type III salt particles after 10 impacts at velocity of 10 m s⁻¹.

<table>
<thead>
<tr>
<th>Nominal aperture size (µm)</th>
<th>Amount of material remaining on sieve (mass %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>425</td>
<td>92.6</td>
</tr>
<tr>
<td>355</td>
<td>3.8</td>
</tr>
<tr>
<td>300</td>
<td>1.5</td>
</tr>
<tr>
<td>250</td>
<td>1.0</td>
</tr>
<tr>
<td>180</td>
<td>0.7</td>
</tr>
<tr>
<td>125</td>
<td>0.3</td>
</tr>
<tr>
<td>Collector tray</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Fig. 6.6  SEM view of the breakdown of a 500 µm PDV Type III salt particle impacted at 10 m s⁻¹.
Another aspect, which was not addressed in the experimental procedure for the melt-grown crystals, is the attrition due to the action of sieving. This is briefly described below, which is applicable to PDV salt only. The melt-grown particles were sufficiently large that they could be separated manually from the debris, and therefore sieving was not necessary.

A sample of about 2 grams of 500 µm near size particles, which included multiple crystals, was sieved using a 425 µm size mesh. The amount of materials passing through the mesh was recorded every minute. The results are shown in Table 6.6. As it can be seen, the amount of materials which can pass through the sieve gradually decreases with time. Examination by optical microscopy of these particles showed that some of the weak multiple crystals broke down on sieving. This implies that the initial attrition rate reported in Fig. 6.4 may have been affected by sieving. However, when the attrition rate has reached its asymptotic value, the sieving does not greatly influence the measured attrition rate.

Table 6.6 Attrition of initial feed material (500 µm near mesh size) due to sieving as quantified by material passing through a 425 µm sieve.

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Incremental amount of materials passing through the sieve (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0026</td>
</tr>
<tr>
<td>2</td>
<td>0.0024</td>
</tr>
<tr>
<td>3</td>
<td>0.0018</td>
</tr>
<tr>
<td>4</td>
<td>0.0010</td>
</tr>
<tr>
<td>5</td>
<td>0.0012</td>
</tr>
<tr>
<td>6</td>
<td>0.0009</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>0.0002</td>
</tr>
<tr>
<td>22</td>
<td>0.0005</td>
</tr>
<tr>
<td>23</td>
<td>0.0004</td>
</tr>
<tr>
<td>24</td>
<td>0.0003</td>
</tr>
<tr>
<td>25</td>
<td>0.0000</td>
</tr>
<tr>
<td>26</td>
<td>0.0000</td>
</tr>
<tr>
<td>27</td>
<td>0.0000</td>
</tr>
</tbody>
</table>
6.5 Conclusions

Commercial PDV salt contains a large number of multiple crystals. These crystals breakdown easily at low impact velocity by the failure of the inter-crystalline bond without suffering appreciable surface damage, hence making the assessment of attrition rate difficult. However, by using a procedure based on repeated impacts and visual examination by optical microscopy, it was possible to measure the attrition rate as a function of particle size. The dependence of fractional loss per impact on particle size follows closely the theoretical prediction, i.e. a linear relationship. This applies of course only to the chipping mechanism. Fragmentation at high impact velocity above 10 m s$^{-1}$, and the breakage of multiple crystals also prevail in impact attrition of PDV salt. However, these two processes were not investigated. Experience in the chipping range showed that the transition to fragmentation depends on the impact velocity and number of impacts.
A mechanistic model of impact attrition of particulate solids having a semi-brittle failure mode has been developed in this work. This model has been verified for several materials having a wide range of mechanical properties. Various aspects of the model have been addressed in Section 2.3.4. In this chapter, the general features of the model are further discussed.

According to this model, the rate of impact attrition of semi-brittle materials is a function of the attrition propensity parameter $\eta$ which is given by:

$$\eta = \frac{\rho v^2 I H}{K_c \phi^2}$$

Equation 7.1 suggests that $\eta$ is directly proportional to the incident energy, size and hardness of the particles, and inversely proportional to the square of fracture toughness and constraint factor.

The influence of impact velocity and particle size was discussed in details previously in Sections 5.5.2 and 5.5.3. Here, the role of material properties, and in particular that of the constraint factor is further examined. The constraint factor describes the relationship between hardness and yield stress. The reason why the constraint factor appears in eqn 7.1 arises from the formulation of the model, where it has been assumed that the deformation stress and size of the plastic zone of a corner of a particle are related to the hardness (see eqns B.1 in Appendix B and 2.9). The use of hardness rather than yield stress is preferred here because it can more readily be measured than the latter for particulate solids. Furthermore, the use of $\phi$ can account for materials with differing anisotropy and work-hardening rate, for which the use of $Y$ alone is inadequate.

It is possible to replace the hardness by the yield stress, $Y$, while taking account of work-hardening, as described in Chapter 4. In this case, $\phi$ can also be replaced by its appropriate value given by eqn 4.8, where $\phi$ is related to $Y$, $E$ and the work hardening rate $\Pi'$. The attrition propensity parameter $\eta$ can hence be expressed as:
For elastic-perfectly plastic materials, the rate of work-hardening $\Pi' = 0$, and we get

$$
\eta = \frac{\rho v^2 I}{\frac{2}{3} \left[ 1 + \ln \left( \frac{2E}{3Y} \right) \right] + \frac{2\pi^2}{27} \frac{\Pi'}{Y}}
$$

With the reference to Figs. 4.5 and 4.6, for glasses and polymers, $\frac{E}{Y}$ is in the range of 10-100, and $\eta$ may be given approximately as:

$$
\eta = (0.3 - 0.5) \frac{\rho v^2 I}{K_c^2}
$$

Therefore, for non-work-hardening materials having a semi-brittle failure mode, the major material properties are toughness and yield stress. However, for work-hardening materials, it is more appropriate to use eqns 7.1 or 7.2 because $\phi$ can be much larger than 3.

An alternative view of eqn 7.1 may be obtained, if the constraint factor $\phi$ is replaced by $H/Y$, i.e.:

$$
\eta = \frac{\rho v^2 I}{K_c^2} \frac{1}{H}
$$

The form $\frac{K_c^2}{Y^2}$ is proportional to the plastic zone size, $r_p$, for elastic-perfectly plastic materials (e.g. see Hertzberg, 1989). Therefore, the attrition propensity parameter may be expressed as a function of the plastic zone size:
This implies that the attrition propensity parameter is inversely proportional to the plastic zone size, which is of course expected. Materials having a relatively large plastic zone, show a lesser degree of brittleness, and hence are more reluctant to chipping. However, it should be noted that the plastic zone size \( r_p \) and hardness \( H \) are not independent parameters, and in fact they have an inverse relationship. Therefore, the plastic zone size should not be regarded as an independent variable.

For an approximation based on linear elastic fracture mechanics (see Section 4.5), the critical stress intensity factor \( K_c \) may be related to the fracture surface energy \( \Gamma \) for the case of plain strain by:

\[
\frac{K_c^2}{1 - \nu^2} = 2E\Gamma
\]  

(7.7)

Considering the attrition propensity parameter \( \eta \), it then follows that:

\[
\eta \propto \frac{\rho v^2 l}{\Gamma} \times \frac{H}{E \phi^2}
\]  

(7.8)

where the dimensionless form \( \frac{\rho v^2 l}{\Gamma} \) may be considered as the Weber number, \( w \), as suggested by Kafui and Thornton (1993). The form \( \frac{H}{E \phi^2} \) is attributed to the plastic deformation characteristics.

Kafui and Thornton (1993) have recently investigated the impact damage of agglomerates by computer simulations. In their simulations, spherical agglomerates were made by bringing into contact a large number of primary particles in the range of 992 to 8000. The primary particles in the agglomerate adhered to each other by the surface energy. The agglomerates were then impacted on a wall at various velocities and their breakdown was monitored. As the inter-particle contacts were treated as elastic, and their breakage was assumed to be governed by the surface energy, the
breakdown of the agglomerate should be considered as highly brittle. The extent of damage was quantified by the ratio of the number of broken bonds to the initial number of bonds between the particles prior to impact, and this was defined as the damage ratio, $\Delta$.

Kafui and Thornton analysed their simulation results by the use of dimensional analysis, and found that the extent of damage is described very well by the Weber number. The correlations between the damage ratio and the Weber number for the simulation results of face-centred cubic agglomerates are shown in Figs. 7.1 and 7.2 for the primary particle sizes of 20 $\mu$m and 40 $\mu$m, respectively. The data points in these figures represent different surface energies and impact velocities. The velocity is in the range of 0.1-3.0 m s$^{-1}$. The two agglomerates were made in a similar size of about 0.5 mm. It is therefore clear that the smaller primary particle size leads to higher impact strengths for the same packing structure.

It is very difficult to relate the damage ratio $\Delta$ to the fractional loss per impact $\xi$. Furthermore, the Weber number in our case is in a range, where $10 < \sqrt{\gamma} < 70$, which is quite different from that of Figs. 7.1 and 7.2. It is therefore difficult to make direct comparison for the experimental results. Nevertheless, it is interesting to note that the dimensional analysis of Kafui and Thornton unifies their results for different surface energies and velocities, and this is in accord with the mechanistic model developed here. However, their simulation results show that the agglomerate strength depends on the primary particle size, but this trend could not be accounted for by the Weber number alone! Therefore, Kafui and Thornton fit different empirical equations to the simulation results to express the effect of primary particle size. This is given by:

$$\Delta = \varsigma \left( \sqrt{\frac{\rho v^2 L}{\Gamma}} \right)^\chi = \varsigma (\sqrt{\gamma})^\chi$$  \hspace{1cm} (7.9)

where different values were obtained for the power index $\chi$, depending on the primary particle size. For the agglomerate having a primary particle size of 20 $\mu$m, $\chi = 0.250$, while for the agglomerate having a primary particle size of 40 $\mu$m, $\chi = 0.131$. It is clear that further development based on the micromechanics of damage is required to account for this effect.

Comparing equation 7.9 with 7.8, it is clear that the effect of plastic deformation has not been taken into account in the model of Kafui and Thornton, and therefore their model is only applicable to ideal brittle materials in its present form.
Fig. 7.1 Correlation between $\Delta$ and $\sqrt{w}$ for an agglomerate having a primary particle size of 20 µm. $\Gamma = (*) 0.2, (\circ) 0.4, (\circ) 1.0, (\star) 2.0, (o) 4.0$ J/m². (After Kafui and Thornton, 1993).

Fig. 7.2 Correlation between $\Delta$ and $\sqrt{w}$ for an agglomerate having a primary particle size of 40 µm. $\Gamma = (*) 0.2, (\star) 0.4, (\circ) 1.0, (\star) 2.0, (o) 4.0$ J/m². (After Kafui and Thornton, 1993).
Chapter 8

Conclusions

In this work a combined theoretical and experimental approach was adopted in the analysis and development of a model of impact attrition of particulate solids. The aim of this work was to develop a mechanistic model of impact attrition, which could take account of material properties, and hence have a predictive capability. The main conclusions are summarized below.

Theoretical

The mechanism of impact attrition of interest here is the chipping process, where the material loss occurs from the corners and edges of the particles by the propagation of subsurface lateral cracks. The chipping process is one of the major damage mechanisms in attrition of particulate solids, as demonstrated by high speed photography and scanning electron microscopy. Furthermore, the test materials which were chosen for the work had a semi-brittle failure mode. In this case, considerable plastic deformation occurs prior to the formation of the lateral cracks. The residual tensile stresses which are imposed by the relaxation of deformed materials around the plastic deformation zone during the unloading stage are responsible for the propagation of lateral cracks.

The model of impact attrition developed here is based on indentation fracture mechanics of subsurface lateral cracks. It is considered that the volume of material bounded between these cracks and the free surface easily detaches to form the attrition debris. The ratio of this volume to the volume of the particle is defined as the fractional loss. The damage volume can be estimated from the depth and length of the subsurface lateral cracks, which in turn are related to the impact velocity and material properties through the indentation fracture mechanics. The development of the model has led to the description of the fractional loss per impact as a function of a dimensionless parameter which is considered to represent the attrition propensity of particulate solids. This parameter accounts for material physical properties (i.e. density and size) and mechanical properties (i.e. hardness, toughness and constraint factor) as well as impact velocity by the following equation:
It can be seen that $\eta$ is proportional to the incident kinetic energy $\rho v^2$, particle size and material hardness.

Characterization of impact damage was carried out to provide a solid ground for the theoretical work. Confocal laser scanning microscopy (CLSM), which is a rapid and non-destructive technique, was used for the characterization of subsurface lateral cracks. The results for the ionic crystals tested here have shown that the power index for the dependence of lateral crack length on impact velocity is about 0.75, which is in good agreement with the theory. The depths of these cracks are comparable with the size of impact impression which is agreement with the theoretical work by Chiang et al. (1982). In addition, the contact time obtained from the high speed photographs, albeit not very accurate, agrees reasonably well with the elastic-plastic impact theory. The dependence of the size of impression on impact velocity to the power of 0.5, also based on the above theory, has also been confirmed.

In conclusion, it appears that the model developed in this work is the first mechanistic model which can describe impact attrition of particulate solids. Previous work reported in the literature is either empirical or simply records of experimental data.

**Experimental**

The experimental work on impact attrition of particulate solids is based on the study of single particle impact testing of ionic crystals of MgO, NaCl and KCl. Both melt-grown crystals and commercially produced solution-grown crystals were used. By choosing high purity melt-grown single crystals of MgO, NaCl and KCl as test materials, the hardness varies by a factor of about 60 and the toughness by a factor of about 7, and therefore these materials can be regarded as representing a very wide range of semi-brittle materials. The experimental results of fractional loss per impact have shown that the dependence of attrition rate on material properties, impact velocity and particle size follows that predicted by the model.

It is commonly assumed in the empirical models of attrition and comminution that the process is of a first order rate. The experimental results here clearly show that this

\[ \eta = \frac{\rho v^2}{K_c^2 c H^2} \]
assumption is of limited validity. For the range of conditions tested here, MgO follows approximately a first order rate, while NaCl and KCl follow higher order rates. It is suggested that this is due to the work-hardening effect, where on each impact the hardness increases. This trend is implicitly predicted by the attrition model, where $\xi \propto H$. However, the experimental determination of the variation of hardness with the number of impacts is complex and will require extensive effort on surface hardness testing on micro and nano scales.

The effect of particle size on attrition rate was investigated. A linear relationship between fractional loss per impact and particle size was predicted by the model, and this was verified for both melt-grown crystals in size range of 2-5 mm and solution-grown salt crystals in the range of 300-500 μm.

The effect of impact velocity was also investigated. It has been shown that there is a threshold impact velocity for the transition of breakage mechanism from chipping to fragmentation. The tests were carried out below this velocity. For the chipping mechanism, the power index for the dependence of attrition rate on impact velocity varies in the range of 2 to 2.5, depending on the number of impacts. The lower limit is considered to be related to that predicted by the theory.

**Application**

The model of impact attrition of single particles, developed in this work, has provided a base for the analysis of attrition of particulate solids in process equipment. It has recently been applied to the analysis of attrition in the jetting region of fluidised beds (Ghadiri et al., 1992b and 1994). Considering that particle attrition in the jetting region of a fluidised bed is as a result of inter-particle collisions, Ghadiri et al. coupled the single particle attrition with a hydrodynamic model of fluid and particle motion in the jet region. This enabled the prediction of the dependence of attrition on orifice gas velocity for the jet region of a fluidised bed, which was in a good agreement with the experimental work. A similar approach can be used in the analysis of attrition in various items of process equipment such as pneumatic conveying lines, cyclones and centrifuges.
A simple mechanistic model of impact attrition of particulate solids, having a semi-brittle failure mode, has been developed in this work based on indentation fracture mechanics. This model has been verified for a number of ionic crystals, which are commonly classified as semi-brittle materials. It is however necessary to extend the approach of single particle impact attrition test to other types of material, such as highly brittle materials (e.g. silica, glass and alumina extrudate, etc.) and ductile materials such as polymers.

The base of the model of impact attrition is the indentation fracture mechanics of subsurface lateral cracks. However, the characterization of the length and depth of these cracks is largely semi-empirical. Furthermore, the mechanism of formation of subsurface lateral cracks is generally less understood than other types of crack, although it is widely accepted that these cracks are formed by residual stresses arising from plastic flow. These difficulties are due to the complexity of the stress and strain fields produced by indentation. Considerable efforts (e.g. Chiang et al., 1982) have been made to model the stress field produced by indentation. However, the analyses are far from rigorous. For instance, elastic and plastic anisotropy is not considered, and the effect of work-hardening is oversimplified. Furthermore, considering the unloading stage in which the residual stress distribution is of interest for the formation of lateral cracks, the stress distribution is generally obtained by a simple superposition of an elastic system on the stresses at the end of plastic loading. This elastic system is provided by a distribution of surface normal traction that is equal and opposite to the distribution of the contact pressure. The influences of primary cracking on the calculated stress field and on the subsequent cracking have been ignored in the analysis. Therefore, further theoretical investigation of the stress field in the elastic-plastic indentation is required with the aim of identifying the mechanisms of formation of subsurface lateral cracks, and hence defining the fracture parameters which could describe the characteristics of these cracks.

A linear dependence of attrition rate on particle size has been predicted by the theoretical model. It is however believed that there is a lower limit of the particle size for the validity of this dependence. This is because the development of subsurface
lateral cracks occurs at certain threshold conditions, depending on the material properties and the impact velocity. It is therefore necessary in the future to develop a model to describe the critical particle size as a function of material properties and impact conditions.

This work has provided a method for characterizing the impact attrition of single particles under well-defined conditions. It should be possible to extend the above approach to describe the abrasive wear of particulate solids, where material loss is caused by sliding and rolling of particles on each other under a prevailing inter-particle force. For materials with a semi-brittle failure mode, the wear is caused essentially by the formation of subsurface lateral cracks. Therefore, with some modifications in the formulation of the fractional loss parameter, the wear of particulate solids can also be modelled using the above approach.

Attrition under consideration in this work is that which relates to the chipping process. At sufficiently high velocities, significant fragmentation can occur by the propagation of cleavage and radial cracks. It is therefore necessary in the future to clarify the role of cleavage and radial cracks to fragmentation. Using this information, it should be possible to quantify the threshold conditions for the transition from chipping to fragmentation based on the fracture mechanics. This should have important implications for comminution processes. Furthermore, for comminution processes the size distribution of the product is of the major interest. It is therefore necessary to develop a model describing the breakage and selection functions as addressed earlier in Section 1.2.1, hence providing a mechanistic model of the fragmentation process.

As addressed earlier in Chapter 1, in process equipment for handling particulate solids, attrition occurs due to particle-particle collisions as well as particle-wall collisions. It is therefore necessary to apply the model of impact attrition of single particles to predict the attrition rate in various items of process equipment using assembly calculations. The application of the Discrete Element Analysis (see e.g. Thornton and Yin, 1991; Potapov et al., 1992) has been shown to be a powerful technique for describing the motion of granular materials and the interactions of particles. It should therefore be possible to develop fully mechanistic and rigorous models of attrition of bulk particulate solids based on single particle attrition behaviour when coupled with the Discrete Element Analysis.
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Appendix A Elastic Constants of Ionic Crystals

For crystalline materials the elastic constants are not isotropic. This means that the
strain generated by a particular stress depends on the direction of that applied stress.
For cubic crystal structures such as NaCl, KCl and MgO which are of interest here,
three independent elastic constants are required to describe the constitutive equations
by which strains and stresses are related (see Nye, 1957), i.e. \( s_{11} \), \( s_{12} \) and \( s_{44} \):

\[
\begin{align*}
\varepsilon_1 &= s_{11} \sigma_1 + s_{12} \sigma_2 + s_{12} \sigma_3 \\
\varepsilon_2 &= s_{12} \sigma_1 + s_{11} \sigma_2 + s_{12} \sigma_3 \\
\varepsilon_3 &= s_{12} \sigma_1 + s_{12} \sigma_2 + s_{11} \sigma_3 \\
\varepsilon_4 &= s_{44} \sigma_4 \\
\varepsilon_5 &= s_{44} \sigma_5 \\
\varepsilon_6 &= s_{44} \sigma_6
\end{align*}
\]

where \( \varepsilon_i \) (i = 1-6) are strains, \( \sigma_i \) (i = 1-6) are stresses and \( s_{11} \), \( s_{12} \) and \( s_{44} \) are
independent compliance constants. The stiffness constants, \( c_{ij} \), can be expressed in
terms of \( s_{ij} \) as:

\[
\begin{align*}
c_{11} &= (s_{11} + s_{12})/(s_{11} - s_{12})(s_{11} + 2s_{12}) \\
c_{12} &= (-s_{12})/(s_{11} - s_{12})(s_{11} + 2s_{12}) \\
c_{44} &= 1/s_{44}
\end{align*}
\] (A.2)

Young's modulus and shear modulus are dependent on the crystallographic direction
<\( hkl \)> as defined by Miller indices:

\[
\begin{align*}
1/E_{hkl} &= s_{11} - 2 \left( s_{11} - s_{12} - 0.5 s_{44} \right) \left( l_1^2 l_2^2 + l_2^2 l_3^2 + l_3^2 l_1^2 \right) \\
1/G_{hkl} &= s_{44} + 4 \left( s_{11} - s_{12} - 0.5 s_{44} \right) \left( l_1^2 + l_2^2 + l_3^2 \right)
\end{align*}
\] (A.3) (A.4)

where \( l_1 \), \( l_2 \) and \( l_3 \) are the direction cosines relative to the <100> axes of the cube.
The second term is zero for strain along <100>, and has a maximum value of \( 1/3 \) in
the <111> direction.
For the strain along the cubic direction \(<100>\), eqns A.3 and A.4 can be simplified to the following forms:

\[
E_{100} = \frac{1}{s_{11}} = \frac{(c_{11} + 2c_{12})(c_{11} - c_{12})}{(c_{11} + c_{12})} \quad (A.5)
\]

\[
G_{100} = \frac{1}{s_{44}} = c_{44} \quad (A.6)
\]

For strain along \(<111>\) direction, Young's modulus is given by:

\[
\frac{1}{E_{111}} = s_{11} - \frac{2}{3} \left[ \left( s_{11} - s_{12} \right) - \frac{1}{2} s_{44} \right] \quad (A.7)
\]

It should be noted that Young's modulus has a maximum value of \(E_{100}\) and a minimum of \(E_{111}\) for conditions when \(s_{44} > 2(s_{11} - s_{12})\), as in the case of NaCl and KCl; on the other hand, if \(s_{44} < 2(s_{11} - s_{12})\) as in the case of MgO crystals, \(E_{100}\) is the minimum value and \(E_{111}\) is the maximum.

Another elastic constant of interest here is Poisson's ratio, which is defined as the ratio of transverse strain to axial strain caused by axial stress. For the simple case of the cubic direction \(<100>\), the Poisson's ratio can be deduced from eqn A.1 by substituting \(\sigma_i (i = 2-6) = 0\), and,

\[
\varepsilon_1 = s_{11} \sigma_1 \\
\varepsilon_2 = s_{12} \sigma_1
\]

hence Poisson's ratio \(\nu\) can be expressed in the form of:

\[
\nu = \frac{-\varepsilon_2}{\varepsilon_1} = \frac{s_{12}}{s_{11}} = \frac{c_{12}}{c_{11} + c_{12}} \quad (A.8)
\]

The elastic constants of ionic crystals have been measured by the use of static, resonance and ultrasonic techniques (Hart, 1968). The static method determines the elastic constants from bending and torsion experiments. The elastic constants of crystals are commonly obtained from their mechanical resonance frequencies or the velocity of ultrasonic waves travelling in specified directions through the crystals.

The numerical values of elastic stiffness for simple ionic crystals have been given by Sprackling (1976), and are shown in Table A.1. The compliance coefficients can be
calculated from eqn A.2, and are also shown in Table A.1. The engineering elastic constants, i.e. Young's moduli and shear moduli for different crystallographic directions, and Poisson's ratio have been obtained by the use of eqns A.3, A.4 and A.8. The numerical values of these elastic constants for NaCl, KCl and MgO crystals are shown in Table A.2.

Table A.1 Elastic constants for MgO, NaCl and KCl single crystals.

<table>
<thead>
<tr>
<th></th>
<th>Elastic Stiffness (GPa)</th>
<th>Compliance coefficient (m² TN⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>c₁₁</td>
<td>c₁₂</td>
</tr>
<tr>
<td>MgO</td>
<td>289.2</td>
<td>87.9</td>
</tr>
<tr>
<td>NaCl</td>
<td>49.3</td>
<td>13.0</td>
</tr>
<tr>
<td>KCl</td>
<td>40.9</td>
<td>7.1</td>
</tr>
</tbody>
</table>

Table A.2 Engineering elastic constants for MgO, NaCl and KCl single crystals.

<table>
<thead>
<tr>
<th></th>
<th>Young's modulus (GPa)</th>
<th>Shear modulus (GPa)</th>
<th>Poisson's ratio v</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>E₁₀₀</td>
<td>E₁₁₀</td>
<td>E₁₁₁</td>
</tr>
<tr>
<td>MgO</td>
<td>248.2</td>
<td>316.2</td>
<td>348.0</td>
</tr>
<tr>
<td>NaCl</td>
<td>43.9</td>
<td>35.0</td>
<td>32.8</td>
</tr>
<tr>
<td>KCl</td>
<td>38.8</td>
<td>19.7</td>
<td>17.0</td>
</tr>
</tbody>
</table>
Appendix B  Quasi-static Compression of Corners of Test Materials

There is a need to establish a relationship between the hardness and the flow stress of a corner of particles for modelling the impact damage (see Chapter 2). It is however difficult to measure the flow stress of a corner of a particle under impact conditions. It was therefore decided to determine the flow stress of a corner of a particle, under quasi-static compression, and to compare the results with hardness which is also measured under quasi-static conditions. The work described here is based on the experimental data reported by Ghadiri et al. (1990). The case of NaCl crystals is analysed here. Results of other test materials can be found elsewhere (Ghadiri and Zhang, 1992).

B1 Material, sample preparation and test procedure

Ghadiri et al. (1990) used 2 mm cubes of melt-grown NaCl crystals in their tests. They used two crystal forms: freshly cleaved form as well as chemically polished and annealed form. Chemical polishing removes the surface damage caused by cleaving. Annealing was used to release the residual stresses introduced during cleavage. Annealing of NaCl crystals was carried out in an oven controlled by a temperature controller/programmer. The specimens were heated up to 620 °C at the maximum possible rate, kept at this temperature for 10 hours, and subsequently, cooled down to 40 °C at a rate of 10 °C per hour.

Compression tests were carried out using a standard mechanical test machine, J.J. Lloyd compression testing unit. The test arrangement is shown schematically in Fig. B.1. The specimen was mounted on an anvil and immobilised by an epoxy 'Araldite' adhesive. The specimen was carefully orientated with axis as near vertical as possible. The loads and crosshead speeds were in the range 6-264 N and 0.2-550 mm/min, respectively, giving strain rates in the range of 0.001-1 s⁻¹. The results at low strain rates were not significantly different from those at higher strain rates. Therefore, only the results at the low strain rates are reported here. In all the tests, the specimens were compressed against a glass slide, thus ensuring the elastic response of the contact surface. The test arrangement does not allow the contact area to be viewed during the tests, so that all that could be recorded in an experiment were the applied load and deflection. A large number of specimens were therefore compressed at various degrees of deflection. The specimens were then examined by optical and scanning electron microscopy. The contact area was measured by automatic image analysis after removing the specimen from the anvil.
B2 Results and discussion

The loading conditions and experimental results of the tests carried out on the freshly cleaved but unannealed NaCl crystals are summarized in Table B.1. The scanning electron micrographs of the samples after compression are shown in Fig. B.2.

B2.1 Plastic deformation and mean contact pressure

The occurrence of plastic deformation for NaCl crystals is evident at different levels of peak load (see e.g. Figs. B.2 and B.3). As a corner of crystal is loaded, it deforms plastically because its tip radius is below the critical elastic/plastic limit as discussed in Chapter 2. As the load is increased the plastic deformation zone becomes larger and the contact area increases in order to support the load. The contact face is flat and optically smooth. This feature enables a good measurement of the contact area.

The flow stress of a corner can be obtained from the plastically deformed area and the applied force for a number of cases in the early stages of plastic deformation. The results are summarized in Figs. B.4 and B.5 for both freshly cleaved and chemically polished and annealed NaCl crystals. It can be seen that the flow stress is approximately independent of the load. The mean flow stresses for freshly cleaved NaCl and chemically polished and annealed NaCl are about 0.17 GPa and 0.20 GPa, respectively. It is not clear why the flow stress for the freshly cleaved specimens is lower than those of the chemically polished and annealed crystals. However, the mean flow stresses are comparable with the Vickers diamond hardness of 0.19 GPa. This suggests that the mean contact pressure for the plastic regime may be estimated from hardness according to the following relationship:

\[ p = \frac{F}{A} \equiv H \] (B.1)
B2.2 Crack morphology and path

Scanning electron micrographs (SEM) in Figs. B.2 and B.3 show a pattern of cracks similar to the quasi-static indentation fracture of a flat face by a sharp indenter, i.e. the formation of \(\{110\}\langle1\bar{1}0\rangle\) cracks. In fact, as it appears, these cracks are better observed in the compression of a corner than in the indentation of a flat face because the crack plane includes a particle edge so that the line of the crack on the edge can also be seen (as shown in Fig. B.2(b)). Based on the mechanism of formation of these cracks, i.e. dislocation glide on the complementary \(\{110\}_{45}\) slip planes, leading to fracture on \(\{110\}_{90}\) planes, Ghadiri et al. (1990) proposed a pattern of cracks as shown in Fig. B.6. In addition to this pattern, there are a few cracks (as shown in Fig. B.2(b)) similar to the \(\{110\}_{90}\) cracks but slightly displaced from the diagonal plane so that the \(\{110\}\) plane cuts the \(\{100\}\) face at a small distance away from the edge as illustrated in Fig. B.7. Examination of many specimens indicates that the displacement may be an artifact of not being able to orientate the crystal with the load acting exactly perpendicular to the \(\{111\}\) plane. This produces more deformation on one or two faces than on the third face, causing a displacement of the crack plane.

No subsurface lateral crack is formed here, as in indentation fracture of NaCl crystals (see Section 4.5). This is considered to be influenced by material hardness. Formation of subsurface lateral cracks has been observed for MgO under quasi-static conditions (Ghadiri and Zhang, 1992), and also in NaCl and KCl under impact conditions. In both cases, the hardness is much higher than that of the case considered here.

In summary, the flow stress of a corner of NaCl crystals under quasi-static compression is constant and close to the Vickers indentation hardness as the compression load is varied. Therefore, the use of hardness in representing the flow stress of a corner in the development of the model of impact attrition (see Chapter 2) is justified.
Fig. B.1 Schematic diagram of the testing arrangement.

Table B.1 Experimental results for freshly cleaved NaCl crystals under quasi-static conditions.

<table>
<thead>
<tr>
<th>Test No</th>
<th>Load (N)</th>
<th>Deflection (µm)</th>
<th>Crosshead speed (mm/min)</th>
<th>Plastic deformation area (mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.6</td>
<td>98</td>
<td>0.2</td>
<td>0.0586</td>
</tr>
<tr>
<td>2</td>
<td>10.1</td>
<td>140</td>
<td>0.3</td>
<td>0.0555</td>
</tr>
<tr>
<td>3</td>
<td>6.0</td>
<td>79</td>
<td>0.35</td>
<td>0.0371</td>
</tr>
<tr>
<td>4</td>
<td>15.7</td>
<td>175</td>
<td>0.35</td>
<td>0.0942</td>
</tr>
<tr>
<td>5</td>
<td>7.8</td>
<td>107</td>
<td>0.4</td>
<td>0.0441</td>
</tr>
<tr>
<td>6</td>
<td>7.8</td>
<td>111</td>
<td>0.5</td>
<td>0.0545</td>
</tr>
<tr>
<td>7</td>
<td>15.3</td>
<td>172</td>
<td>0.55</td>
<td>0.0932</td>
</tr>
<tr>
<td>8</td>
<td>19.6</td>
<td>152</td>
<td>0.55</td>
<td>0.1055</td>
</tr>
<tr>
<td>9</td>
<td>18.9</td>
<td>212</td>
<td>0.6</td>
<td>0.1050</td>
</tr>
<tr>
<td>10</td>
<td>25.7</td>
<td>274</td>
<td>0.7</td>
<td>0.1545</td>
</tr>
<tr>
<td>11</td>
<td>38.0</td>
<td>272</td>
<td>0.8</td>
<td>0.2335</td>
</tr>
<tr>
<td>12</td>
<td>35.4</td>
<td>393</td>
<td>1.0</td>
<td>0.2430</td>
</tr>
<tr>
<td>13</td>
<td>49.4</td>
<td>250</td>
<td>1.0</td>
<td>0.3425</td>
</tr>
<tr>
<td>14</td>
<td>69.3</td>
<td>494</td>
<td>1.25</td>
<td>0.4380</td>
</tr>
</tbody>
</table>
Fig. B.2 Compression of a corner of freshly cleaved NaCl crystals:
(a) at $F = 7.8$ N and (b) at $F = 69.3$ N.
Fig. B.3 Compression of a corner of chemically polished and annealed NaCl crystals: (a) at $F = 31$ N and (b) at $F = 264$ N.
Fig. B.4 Flow stress at various loads for freshly cleaved NaCl crystals under quasi-static compression of a corner.

Fig. B.5 Flow stress at various loads for chemically polished and annealed NaCl crystals under quasi-static compression of a corner.
PLASTICALLY DEFORMED ZONE

CRACK PLANES \{110\}, 45° WITH RESPECT TO \{100\} PLANE

Fig. B.6 Schematic diagram of the crack morphology and path in quasi-static compression of a corner of ionic crystals of rocksalt structure.

Fig. B.7 Schematic diagram of a slightly displaced \{110\}\langle110\rangle crack.
Appendix C Specific Attrition Rate and Fractional Loss per Impact

Two methods have been proposed in Section 5.4 to estimate the material loss for a repeated impact process. One is the specific attrition rate, $s$, following the concept of a first order attrition rate as outlined in Chapter 1, and the other is the fractional loss per impact, $\xi$. Vervoorn and Austin (1990) have shown that the equation defining a first order rate for a repeated impact attrition process is similar to that of a continuous process given by eqn 1.1. If $M(0)$ is the mass of a sample of mono-size particles undergoing attrition, and $M(N)$ is the mass of surviving mother particles after $N$ impacts, then for the specific attrition rate, it follows:

$$\frac{dM(N)}{dN} = s \cdot M(N) \quad (C.1)$$

After integration, it yields:

$$s = -\frac{1}{N} \ln \frac{M(N)}{M(0)} \quad (C.2)$$

The fractional loss per impact is defined as the following:

$$\xi_{0,N} = \frac{1}{N} \frac{M(0) - M(N)}{M(0)} \quad (C.3)$$

In this work the fractional loss per impact has mainly been adopted for the assessment of attrition, in view of its direct relevance to the theoretical model and the invalidity of the first order attrition process for NaCl and KCl crystals. However, for a low attrition rate process, the specific attrition rate, $s$, is approximately equal to the fractional loss per impact $\xi_{0,N}$. This is shown as follows.

Let $$\frac{M(0) - M(N)}{M(0)} = p_N \quad (p_N < 1)$$

Then $$s = -\frac{1}{N} \cdot \ln(1 - p_N) = \frac{1}{N} \cdot \ln \frac{1}{1 - p_N}, \quad \text{and} \quad \xi_{0,N} = \frac{1}{N} \cdot p_N$$

Using Taylor expansion:
\[
\frac{1}{1-p_N} = 1 + p_N + p_N^2 + \cdots + p_N^m + \cdots
\]

and

\[
\ln(1+p_N) = p_N - \frac{p_N^2}{2} + \frac{p_N^3}{3} - \cdots + (-1)^{m+1} \frac{p_N^m}{m} + \cdots
\]

when \( p_N < 0.1 \), which is equivalent to the condition of \( \xi < 5 \times 10^{-3} \) with a total impact number of \( N = 20 \), then

\[
\frac{1}{1-p_N} = 1 + p_N \quad \text{and} \quad \ln(1+p_N) = p_N
\]

Therefore, the following approximation can be obtained:

\[
s = \frac{1}{N} \ln(1+p_N) \approx \xi_{0,N} \quad \text{(C.4)}
\]

**Systematic error of fractional loss per impact**

The fractional loss per impact is usually very small in the chipping process, e.g. in the order of \( 1 \times 10^{-4} \). It is necessary in this case to consider the systematic error involved in the determination of the fractional loss per impact. Using eqn C.3, the error can be calculated as follows:

\[
|\delta \xi_{0,N}| = \frac{1}{N} \left| \delta \left( \frac{M(N)}{M(0)} \right) \right|
\]

\[
= \frac{1}{N} \frac{M(N)}{M(0)} \sqrt{\left( \frac{\delta M(N)}{M(N)} \right)^2 + \left( \frac{\delta M(0)}{M(0)} \right)^2} \quad \text{(C.5)}
\]

As \( \delta M(N) = \delta M(0) \), it then follows:

\[
|\delta \xi_{0,N}| = \frac{1}{N} \frac{M(N)}{M(0)} \sqrt{\left( \frac{1}{M(N)} \right)^2 + \left( \frac{1}{M(0)} \right)^2} \cdot |\delta M(N)| \quad \text{(C.6)}
\]

As \( \frac{M(N)}{M(0)} \approx 1 \), eqn C.6 can be simplified as:

\[
|\delta \xi_{0,N}| \approx \frac{1}{N} \frac{\sqrt{2}}{M(0)} \cdot |\delta M(N)| \quad \text{(C.7)}
\]