Crystal plasticity and experimental studies of nano-indentation of aluminium and copper

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Crystal plasticity and experimental studies of nano-indentation of aluminium and copper

A thesis submitted for the award of the degree of

Doctor of Philosophy

from

UNIVERSITY OF WOLLONGONG

by

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2014
Declaration

I, Mao Liu, declare that this thesis, submitted in fulfilment of the requirements for the award of Doctor of Philosophy, in the School of Mechanical, Materials and Mechatronic Engineering (MMM), University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other academic institution.

Mao Liu
March 2014
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List of Publications during the PhD course


6. **Mao Liu**, Cheng Lu, Anh Kiet Tieu, The effects of different azimuthal orientations of the pyramidal indenter on indentation mechanical behaviours and texture evolution of copper single crystal. (in preparation)

7. **Mao Liu**, Cheng Lu, Anh Kiet Tieu, Crystal plasticity finite element method study of indentation piling-up of single crystal copper. (in preparation)


Abstract

In this thesis the mechanical behaviour and micro-texture evolution during nano-indentation deformation of single crystal aluminium has been numerically and experimentally investigated. The hardening parameters of single crystal copper have been estimated systematically by crystal plasticity finite element method (CPFEM) modelling.

The IBIS/UMIS nano-indentation system has been used to conduct nano-indentation tests on three different oriented samples with (001), (101) and (111) parallel to the sample surface respectively. The orientations of indentation impressions have been measured by electron backscatter diffraction (EBSD) and transferred to CPFEM simulations as inputs. The elastic moduli and indentation hardness on three different surfaces have been determined from the measured indentation load-displacement curves. The cross section of the (111) sample has been observed by transmission electron microscope (TEM) to measure the lattice rotation induced by indentation deformation. A multi-mode Scanning Probe Microscopy (SPM) has been used to measure the piling-up patterns of indent impressions on three different oriented surfaces.

A three-dimensional CPFEM model has been developed to investigate the nano-indentation process. The numerical results of load-displacement, elastic modulus, hardness, piling-up patterns, and indentation induced lattice rotation have been found in good agreements with experimental observations. The effects of the coefficient of friction on texture evolution have also been numerically studied.

The anisotropy of deformation behaviour during nano-indentation of aluminium has been disclosed via piling-up patterns, the out-of-plane surface profiles along the diagonal and vertical cutting plane, slip traces and pole figures of the cross sections along different cutting planes.

The effect of the indenter’s geometry has been investigated by comparing the simulation results of Berkovich indenter and its equivalent conical indenter with a 70.3° semi-apex angle. Piling-up has been found to be independent of the geometry of indenter but dependent of in-plane crystallographic orientations. The geometry of indenter does not
Abstract

affect the location of the maximum strain rate along the diagonal cutting line but it does influence the location of the maximum strain rate along the vertical cutting line. It has also been found that the geometry of indenter plays a very important role in texture evolution during nano-indentation.

Indentation size effect has been observed in the CPFEM simulations. The Mises stress, critical resolved shear stress and the main lattice curvature have been studied. The mechanism responsible for the ISE has been discussed in details.

A three dimensional simulation model has been developed to study the tensile test of polycrystalline aluminium via combination of CPFEM and Voronoi diagram. The numerical true stress-strain has been found in a good agreement with the experimental observation. The correlation between the Berkovich indentation hardness (H) and the yield stress (σ) at the special representative plastic strain of 0.07 has been compared. H=Cσ with C=6.19 has been deduced from the present study. It is believed that strain hardening significantly affects the factor C.

The parameters of the hardening model of single crystal copper have been estimated by comparing the CPFEM simulation results with experimental data. It has been found through simulations that the reference value of slip γ₀ and the reference strain rate ᵇ affect the indentation mechanical behaviour significantly, while the influence of the initial hardening modulus h₀ is insignificant behaviour.
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XVI
Chapter 1 Introduction

1.1 Aims of the present work

Indentation is perhaps the most commonly applied means of testing the mechanical properties of materials. Conventional indentation tests have the length scale of the penetration in microns or millimetres. In the mid-1970s, the indentation technique was applied to measure the hardness of small volumes of material, such as thin film. The length scale of the penetration is usually in nanometre. Therefore, this new technique is called nano-indentation.

Extensive research has been conducted on nano-indentation during last two decades. However, deformation mechanism during the nano-indentation has not been fully understood. In addition, several phenomena relevant to nano-indentation still remain unclear, such as

1) It is often observed in nano-indentation that the indentation hardness, $H$, which is defined as the load on the indenter normalized with the projected contact area of the hardness impression, is dependent of the depth of indentation penetration. This phenomenon is called the indentation size effect. Current explanations on this phenomenon are still controversial.

2) The indentation hardness ($H$) is often related to the yield stress ($\sigma$) of the material by an equation $H=C\sigma$. $C$ is a constant (~3) for a rigid perfectly-plastic material, which only depends on the geometry of the indenters. What is the $C$ value for work-hardened material?

In this study, nano-indentation tests on aluminium single crystal and crystal plasticity finite element method (CPFEM) simulations will be conducted to answer the above questions. The CPFEM model incorporates crystal plasticity constitutive equations (crystallographic slip, lattice rotation, rate-dependent hardening model, etc.) into a finite element framework.

Crystal plasticity modelling of polycrystalline materials during large deformation is a quite complicated system. Its accuracy depends on the following: (a) deformation model;
(b) texture model (crystallographic orientation rotation scheme); (c) work-hardening model; (d) polycrystalline structure. The first two sub-models used in the CPFEM simulation stem from the well-recognized theory. The parameters of hardening model need to be estimated by fitting the experimental results (stress-strain curve or load-displacement curve). However, it is hard to separate the influences of polycrystalline structure and hardening model. Experiments of single crystals provide an opportunity to avoid the effect of polycrystalline structure. The parameters of hardening model for aluminium used in Chapters 5-7 were obtained by fitting the experimental results of compression and tension tests of aluminium single crystals. Unfortunately such information is not always available for most materials. Nano-indentation is a unique technique to achieve this goal. Due to the nature of small scale, most nano-indentation impressions are far from the grain boundary. The grain boundary has a negligible influence on such nano-indentation. Therefore, nano-indentation test results can be used to estimate the parameters of the crystal plasticity hardening model. In this study, the parameters of the hardening model for pure copper will be determined by nano-indentation simulations.

1.2 Structure of the thesis

Chapter 2 introduces the literature review on indentation tests, indentation size effect and FEM simulation of the indentation process.

The experimental process of indentation tests on different oriented Al single crystal samples will be discussed in Chapter 3 which includes the material preparation, experimental facilities and results analysis. All the experimental results will be compared with the simulated ones in Chapter 5 to validate the CPFEM model.

The crystal plasticity constitutive model and numerical implementation will be presented in Chapter 4.

The effects of friction, initial orientation and indenter’s geometry on indentation mechanical behaviour and texture evolution will be discussed in Chapter 5.
The indentation size effects will be simulated and analysed in Chapter 6.

A 3D CPFEM model to simulate the tensile test of polycrystalline aluminium will be developed in Chapter 7. The relationship between nano-indentation hardness and tensile yield stress will be discussed in this chapter.

The simulation of nano-indentation of copper single crystal will be presented in Chapter 8. The parameters of Bassani-Wu hardening model will be estimated by comparing the experimental data with simulation results.

Lastly, the conclusions and recommendations of future work with regards to aforementioned chapters will be given in Chapter 9.
Chapter 2 Literature review

This chapter contains a substantial amount of information on topics considered essential for the understanding of the mechanical behaviour, deformation mechanism, micro-texture evolution and size effect during nano-indentation.

2.1 A brief history of nano-indentation testing

Indentation tests were first performed by a Swedish iron mill’s technical manager Brinell who used spherical balls from hardened steel ball bearings or made of cemented tungsten carbide as indenters to measure the plastic properties of materials in 1900 [1-4]. Brinell’s testing approach is schematically shown in Fig. 2.1(a). Brinell also proposed the following formula to determine the hardness

\[ H_B = \frac{2P}{\pi D \left(D - \sqrt{D^2 - d^2}\right)} \]  

(2.1)

here \( H_B \) is the Brinell hardness, \( P \) is the load, \( D \) is the diameter of the ball indenter and \( d \) is the diameter of residual area of the impression.

Brinell’s work was then followed and improved by Meyer in 1908 [5]. In Meyer’s work, the hardness (H) is calculated by the load (P) divided by the projected area (A), namely

\[ H = \frac{P}{A} \]  

(2.2)

In 1922, the Vickers test was carried out and commercialized by the Firth-Vickers company [6]. A square-based pyramid diamond indenter with 136° semi-angle was used instead of ball indenter, as shown in Fig. 2.1(b). The Vickers hardness (\( H_V \)) is defined as the load divided by the surface area of the impression, namely

\[ H_V = 1.8544 \frac{P}{d_V^2} \]  

(2.3)

where \( d_V \) is the length of diagonal of the surface area.
Fig. 2.1 Commonly used methods of indentation hardness tests: (a) spherical indenter (Brinell and Meyer); (b) diamond pyramid (Vickers). [7]

The Brinell, Meyer and Vickers methods were then widely used in the metallurgical and engineering industries in the early 20th century as indentation tests offered simplicity, low-cost and high speed compared with conventional tensile testing. No special shape or extra fabrication of sample is required for indentation tests except for a simple sample with a flat surface. In addition, several indentation tests could be quickly performed on a small area without destroying the whole sample.

Another significant finding is that the hardness is load-dependent, which was proposed by Meyer through ball indentation experiments on a wide range of metals [7]. For a given ball size, the diameter of the impression after unloading was found to be related to the applied load by the following empirical relationship:

\[
P = C d^n
\]

(2.4)

here \(P\) is the applied load, \(C\) is a constant of proportionality, the exponent \(n\) is the well-known Meyer index and \(d\) is the diameter of the residual area of the impression after unloading.

When the \(n\)-value is less than 2, according to Eqs. (2.2) and (2.4), the hardness increases with decreasing the load. While the \(n\)-value is larger than 2, the hardness decreases with decreasing the load. If the Meyer index equals to 2, the hardness is a constant, namely load-independent. For most metals that can be work hardened by the indentation process, \(n > 2\) [7]. Therefore, the Meyer index has been found to be strongly dependent on the
work hardening of the tested material and to be independent of the size of the ball indenter [8]. Meyer [8] also found the same hardness using balls of different diameters could be obtained only if the indentations were geometrically similar, namely with the same ratio \( d/D \).

Tabor’s work [9] performed in 1948 represents a landmark in the understanding of the indentation process. He qualitatively described the procedure how an indentation by a ball initially led to elastic deformation, then to plastic flow associating with work hardening, and final on removal of the load to elastic recovery. In 1951, Tabor [1] had proven that the indentation hardness \( (H) \) could be related to the yield stress \( (\sigma) \) of the material by an equation based on the theory of indentation of a rigid perfectly-plastic solid, namely

\[
H = C\sigma
\]  

(2.5)

where \( C \) is a constant, which depends on the geometry of the indenters. For the strain-hardened materials and the materials which consequently have no definite yield stress, the stress measured at a representative strain \( \varepsilon_r \) can be used as \( \sigma \) [10]. The value of the representative strain is relevant to the geometry of the indenter. For instance, \( \varepsilon_r \approx 0.08 \) for a Vickers diamond indenter.

Tabor [7] started taking an interest in the indentation response of polymers and of macroscopically brittle materials in the studies of their frictions. In the following couple of years, Tabor and King [11] reported the method of estimating the yield pressure on polyethylene, PMMA (Polymethyl methacrylate), PTFE (Polytetrafluoroethylene), and halocarbon polymer via Vickers hardness measurements. Subsequently, Pascoe and Tabor [12] reported a range of polymers obeyed Meyer’s laws. The single-crystal rock salt was also investigated by King and Tabor [13] with Vickers indentations, by which, they found the values of yield stress from the indentation matched well with the compression experiments.

In 1960s, indentation at high temperature was investigated by Atkins and Tabor [14]. They studied the mechanical properties of single crystals of MgO at temperatures of
600°C to 1700°C via the mutual indentation hardness technique. It was found the short-
time hardness decreased when the temperature increased.

Conventional indentation tests have the length scale of the penetration in microns or millimetres. In the mid-1970s, the indentation technique was applied to measure the hardness of small volumes of material, such as thin film. The length scale of the penetration is usually in nanometre. Therefore, this new technique is called nano-
indentation. Apart from the penetration length scale, the distinguishing feature of nano-
indentation testing is the indirect measurement of the contact area [15]. In conventional
indentation tests, the contact area is directly measured from the residual impression area.
In nano-indentation test, the residual impression area is too small to be directly measured. Therefore, the contact area is determined by the measured penetration depth in nano-indentation.

Since 1980s, especially after 1990s, extensive experimental studies of nano-indentation have been performed on many different types of materials.

2.2 Nano-indentation facilities

The most famous manufacturers of nano-indentation equipment include MTS Systems Corp. (www.mtsnano.com), Micro Materials Ltd. (www.micromaterials.co.uk), CSIRO. (www. csiro.au/hannover/2000/catalog/projects/umis.html), Hysitron Inc. (www.hysitron.com) and CSM Instruments Corp. (www.csm-instruments.com). The iconic products of these manufacturers are shown in Fig. 2.2. The working theories of different representative instruments are given in Fig. 2.3. All of these instruments include three principal parts, namely indenter, load application, and capacitive sensor for measuring the displacements of the indenter. For instance, in Fig. 2.3(a), the load is applied by an electromagnetic coil which is connected to the indenter shaft by a series
of leaf springs. The deflection of the springs is a measure of the load applied to the
indenter, and the displacement usually can be measured by a capacitive sensor.
The indenter is conventionally made of diamond which has been ground to shape and sintered in a stainless steel chuck. The frequently used shapes of indenter are shown in Fig. 2.4(a)-(f) [17]. The conical indenter has a sharp, self-similar geometry. Normally, the cone angle is 60° or 90° and the tip radii are 0.7, 1, 2, 5, 10, 20, 50, 100, 200 µm.
The applications of conical indenter are extensive, including scratch testing, wear testing, nano-scale 3D imaging capturing and tensile and compression tests on MEMS (Microelectromechanical systems). Berkovich indenter is the most frequently used indenter for indentation tests. The most noticeable feature of Berkovich indenter is that it is a three-sided pyramid that can be ground to a point, which makes it easy to maintain a self-similar geometry to micro-scale or nano-scale. The radius is about 150 nm when it is new and will become 250 nm 12 months later. The applications of Berkovich indenter are much more extensive, such as bulk materials tests, thin films tests, polymers tests, scratch testing, wear testing, MEMS tests and in situ imaging. The Vickers indenter is a four-sided pyramid and suitable for measuring mechanical properties on the very small scale, such as nano-scale as the line of conjunction at the tip limits the sharpness of tip for determination of hardness for very shallow indentation. The recommended applications include bulk materials tests, films and foils tests, scratch testing and wear testing.

**Fig. 2.4** Shapes of different indenters: (a) Conical indenter; (b) Berkovich indenter; (c) Vickers indenter; (d) Knoop indenter; (e) Cube-corner indenter; (f) Spherical indenter. [17]

The Knoop indenter is originally designed for hard metals. It is also can be used to probe anisotropy in sample surface. The Cube-corner indenter is a three-sided pyramid with mutually perpendicular faces, which is like the corner of a cube. The sharpness of
the cube corner produces much higher stresses and strains in the area in contact with the indenter, thus makes it capable of producing very small and well-defined cracks around imprint in brittle materials. The toughness at micro-scale or nano-scale can be investigated via these induced fine cracks. Meanwhile, the Cube-corner indenter is fairly fragile and easily broken. The spherical indenter can be used to examine yielding and work hardening theoretically. Moreover, the elastic-plastic transition can also be investigated. The reason is, the contact stresses with spherical indenter are initially small and produce only elastic deformation, and with increasing indentation depth, a transition from elastic to plastic deformation can be captured. The parameters of all of these indenters are listed in Table 2.1.

**Table 2.1** Parameters of different indenters.[18]

<table>
<thead>
<tr>
<th>Indenter type</th>
<th>Projected area (A)</th>
<th>Semi angle (θ)</th>
<th>Effective cone angle (α)</th>
<th>Intercept factor</th>
<th>Geometry correction factor (β)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sphere</td>
<td>( A \approx \pi 2R h_p )</td>
<td>N/A</td>
<td>N/A</td>
<td>0.75</td>
<td>1</td>
</tr>
<tr>
<td>Berkovich</td>
<td>( A = 3h_p \tan^2 \theta )</td>
<td>65.3°</td>
<td>70.2996°</td>
<td>0.75</td>
<td>1.034</td>
</tr>
<tr>
<td>Vickers</td>
<td>( A = 4h_p \tan^2 \theta )</td>
<td>68°</td>
<td>70.32°</td>
<td>0.75</td>
<td>1.012</td>
</tr>
<tr>
<td>Knoop</td>
<td>( A = \frac{2h_p \tan \theta_1 \tan \theta_2}{\tan \theta_1 \tan \theta_2} )</td>
<td>( \theta_1 = 86.25° )</td>
<td>77.64°</td>
<td>0.75</td>
<td>1.012</td>
</tr>
<tr>
<td>Cube-corner</td>
<td>( A = 3h_p \tan^2 \theta )</td>
<td>35.26°</td>
<td>42.28°</td>
<td>0.75</td>
<td>1.034</td>
</tr>
<tr>
<td>Conical</td>
<td>( A = \pi h_p \tan^2 a )</td>
<td>a</td>
<td>a</td>
<td>0.72</td>
<td>1</td>
</tr>
</tbody>
</table>

### 2.3 Application of nano-indentation

In 1992, Oliver and Pharr [19] proposed a method to measure the Young’s modulus based on the indentation load-displacement curve, which was frequently used until now. Kucharski and Mroz [20] presented a new procedure for determining the plastic stress–strain curve by means of a cyclic spherical indentation test in 2007, which constitutes an ground-breaking improvement with respect to the traditional method. Kruzic et al. [21] improved the indentation techniques of evaluating the fracture toughness of biomaterials and hard human bones although accurately measuring the fracture
toughness of brittle materials can be quite challenging. The fracture toughness can be obtained directly from indent crack length measurements, as shown in Fig. 2.5.

![Indentation site on the transverse section of human cortical bone. Images are of (a) as-indented, (b) dehydrated, (c) ESEM (environmental scanning electron microscope) and (d) SEM. The bottom corner of the indentation, (e) shows a crack with l=10μm, which is one of the two cracks emanated from indent corners among 30 corners. High-magnification imaging of the top right corner, (f) revealed that no cracks were generated from this indent corner. [21]](image)

Huber and Tsakmakis [22] proposed that nano-indentation tests can be used to identify effects of kinematic hardening on the material response. They also indicated that the identification may rely on the measurement of the opening of the hysteresis loop produced in the indentation load-displacement curve as shown in Fig. 2.6. It is obvious
that each material has a unique opening diagram indicating the corresponding effect of kinematic hardening.

![Opening of experimental measured hysteresis loops.](image)

**Fig. 2.6** Opening of experimental measured hysteresis loops. [22]

Durban and Masri [23] in 2007 found that the nano-indentation test data with conical indenter over a range of cone angles can be used to reconstruct the axial stress-strain curve. Mata et al. [24] modified the previous hardness formulation within the elastic-plastic transition derived for solids by Hill [25] in 1950 and Marsh [26] in 1964 as it didn’t exhibit strain hardening.

Application of nano-indentation tests to determine the mechanical properties of surface coatings is another milestone. Normally, the investigations of the mechanical properties of the coatings are difficult as the traditional compression and tensile tests are unable to apply well at very small scales. Rodriguez et al. [27] performed depth sensing indentation in plasma sprayed Al\textsubscript{2}O\textsubscript{3}-13%TiO\textsubscript{2} nano-coatings in order to determine the Young’s modulus and hardness. It was found the mechanical properties were dramatically enhanced in the nanostructured coating compared to the conventional one, which is shown in Fig. 2.7.
Chapter 2 Literature review

Fig. 2.7 Young’s modulus and hardness vs. total penetration depth for both coatings. [27]

Thin water film was studied by Opitz et al. [28] in 2003. It was believed that the thin water films which covered most of the micro-scale and nano-scale surfaces could be particularly important for micro electromechanical systems (MEMS) and the upcoming nano electromechanical systems (NEMS), as they played a critical role in defining the micro- and nano-tribological properties of a system. Kim et al. [29] presented a nano-indentation method to measure the Poisson’s ratio of thin films for MEMS applications. In their test, a double-ring shaped sample was designed to conduct the measurement of the Poisson’s ratio as shown in Fig. 2.8. The load-deflection data of the double ring sample after nano-indenter loading was analysed to obtain the Poisson’s ratio. Lou et al. [30] investigated the mechanical behaviour of LIGA (an acronym of the German words “lithographie, galvanoformung, abformung”) nickel MEMS structures which were developed for applications in micro-switches and accelerometers via the nano-indentation method. Both Berkovich and Cube-corner indenter were used to conduct the nano-indentation tests and study the effects of residual indentation depth on the hardness of LIGA Ni MEMS structures between the micro-scale and nano-scales. Almost no apparent size dependence has been found for LIGA Ni films indented by a Berkovich indenter. In contrary, the hardness dramatically increased with decreasing residual indent depth for films indented by the Cube-corner indenter.
Indentation tests were also used to evaluate adhesion strength of the thermal barrier coatings (TBCs) which were used to improve the performance and efficiency of advanced gas turbines [31-35]. Interfacial strength was one of the most important properties in TBCs, and traditional tensile method was found to be restricted due to the size dependence. Yamazaki et al. [36] used the indentation method to investigate the interfacial strength of TBC which was subjected to thermal cycle fatigue. An indent was made directly at the interface on the polished surface of the sample as shown in Fig. 2.9. The crack length and the diagonal length of the indentation were measured using SEM right after indentation as shown in Fig. 2.10. The typical interfacial crack initiated by the indentation test in Fig. 2.10(a) showed the crack mainly propagated in the ceramic top coat near the interface. Even after 500 thermal cycles with the formation and growth of TGO (thermally grown oxide) layer shown in Fig. 2.10(b), the crack propagated only in the ceramic top coat too.
Chapter 2 Literature review

Fig. 2.9 Schematic illustration of the instrumented indentation test equipment. [36]

Fig. 2.10 Typical interfacial cracks initiated by the indentation test: (a) for 0 thermal cycles (As-sprayed); (b) after 500 cycles. [36]

The dependence of nano-indentation piling-up patterns and of micro-textures on the crystallographic orientation was studied by Wang et al. [37] using high purity copper single crystal with three different initial orientations. The indentation tests were performed on a Hysitron nano-indentation setup using a conical indenter to avoid symmetries. The results are shown in Fig. 2.11. Four-, two-, and sixfold symmetrical piling-up patterns were captured on the surface of (001), (011) and (111) initial oriented single crystal, individually, which could be explained in terms of the strong crystallographic anisotropy of the out-of-plane displacements around the indenter.
Fig. 2.11 Experimentally observed contour plots of the pile-up patterns on (a) (001), (b) (011) and (c) (111) oriented copper single crystal surfaces after indentation with a conical indenter. [37]

With the development of focused ion beam milling of site-specific electron transparent foils, the investigation of cross-sections of nano-indentations with the transmission electron microscope (TEM) or electron backscatter diffraction has recently become feasible [38]. Lloyd [39] and his colleagues combined nano-indentation and TEM to survey the deformation behaviour in a range of single crystal materials with different resistances to dislocation flow as shown in Fig. 2.12. The principal deformation models included phase transformation (silicon and germanium), twinning (gallium arsenide and germanium at 400°C), lattice rotations (spinel), shear (spinel), lattice rotations (copper) and lattice rotations and densification (TiN/NbN multilayers). Generally, the residual impresses were sectioned though the tip of the indent with the thin foil normal approximately parallel to either [110] or [100] zone axis.

Fig. 2.12 (a) Schematic illustrating how the indents were sectioned in the FIB to allow examination with a TEM; (b) Secondary electron FIB image of the final stages of preparation of a thin foil through a 50 mN indent in silicon. [39]
The indents in (001) silicon at loads of 30 mN and 60 mN are shown in Fig. 2.13. It was found a transformed region was under the area of the Berkovich indenter in both cases, and a crack was originating at the base of the transformed region in the case of 60 mN.

**Fig. 2.13** Bright field images of indents in silicon formed with loads of (a) 30 mN and (b) 60 mN. The structure is observed here after the load has been removed, allowing the high pressure phase to transform back to a mixture of other structures. [40]
Shear bands were captured in spinel crystals, which are shown in Fig. 2.14 and Fig. 2.15. It was found the shear band spacing increased with increasing distance from the indent tip, and the spacing on the steep side of the indent was a little smaller for the large load. Lloyd [40] concluded that the increase of the shear band spacing with distance far away from the indenter tip indicated there was a limit to the amount of displacement occurring through any shear band due to strain hardening. Consequently, a high concentration of shear bands was close to the indenter tip where had the largest vertical
displacements, while a low density of slip bands was sufficient to accommodate the relatively small vertical displacement in the region far away from the indenter tip.

![Diagram showing slip band spacing as a function of distance from the indenter tip](image)

**Fig. 2.15** Slip band spacing in spinel as a function of distance from the indenter tip (see Fig. 2.14b) for the 50 mN and 80 mN indents. The error in measurement of the shear band spacing is approximately 10 nm. [39]

Lattice rotation angles around an axis perpendicular to the [110] zone axis were investigated [39] and are shown in Fig. 2.16. It was found the rotations only occurred in the region immediately below the indent impression. The greatest rotations were quite near the indent tip and the magnitude of rotation angles decreased significantly with the increasing distance from the indent tip along the surface on the shallow side.
Fig. 2.16 (a) Bright field image of a 50 mN indent in spinel with lattice rotations (in degrees) at selected positions indicated. (b) and (c) are dark field images of the surface of the same indent taken using streaks from either side of the 004 reflection. [39]

The indentation-induced plastic zones below indentations in copper single crystals, with depths ranging from 250 nm to 250 µm, were examined by Rester et al. [41] via the implementation of focussed ion beam (FIB) and electron backscatter diffraction (EBSD) techniques. Two or three distinguishable regimes shown in Fig. 2.17 were captured by analysing scanned orientation micrographs. Meanwhile, the changes in the evolution of the microstructure were reflected in the hardness curve shown in Fig. 2.18. Regime α described the impression which is smaller than 300 nm, in which no significant orientation changes are observed by EBSD (Fig. 2.18(a)). Regime β describes the indentation depth between 300 nm and 30 µm, which is characterized by regions having noticeable changes of the orientation (Fig. 2.18(c) and (d)). It was also found that the orientation differences increases with growing indentation depth in this regime. Regime γ is associated with indentation depth larger than 30 µm exhibiting a typical substructure of FCC (face centered cubic) single crystal of pure metals during indentation. Therefore, Rester et al. [41] concluded that the hardness of a material varies with the size of the indent impression and the source size becomes the dominant effect only for very small impressions (ie. in regime α).
Fig. 2.17 Misorientation maps of indentations in copper for loads of 0.5, 1, 10, 300 mN; 10 and 100N. [41]
Fig. 2.18 Logarithmic plot of the hardness versus the indentation depth for loads ranging from 40µN to 100 N. The arrows mark the hardness values of imprints investigated in course of this work. [41]

Zaafarani et al. [42] investigated texture and microstructure below a conical nano-indent in a (111) oriented Cu single crystal using 3D EBSD. The tests were performed using a joint high-resolution field emission scanning electron microscopy/electron backscatter diffraction (EBSD) set-up coupled with serial sectioning in a focused ion beam system in the form of a cross-beam 3D crystal orientation microscope (3D EBSD) as shown in Fig. 2.19.

The EBSD testings conducted in sets of subsequent (11$ar{2}$) cross-section planes exhibited a pronounced deformation-induced 3D patterning of the lattice rotations below and around the indent, which are shown in Fig. 2.20.
Fig. 2.19 (a) Joint high-resolution field emission SEM/EBSD set-up together with a FIB system in the form of a cross-beam 3D crystal orientation microscope for conducting 3D EBSD measurements by serial sectioning (Zeiss). (b) Schematic of the joint FIB/EBSD set-up. (c) Schematic of the FIB sectioning geometry. [42]
Fig. 2.20 Rotation angles and rotation directions in the (11-2) plane. [42]

2.4 Nano-indentation size effect

Over the past several decades, with the development of new technologies, people felt that it was very important to understand how materials perform at small scales because the mechanism properties are significantly different from those at macro-scales. Therefore, a great number of researchers became interested in micro- and nano-scale deformation phenomena which also made them try to look for a new method to examine these physical phenomena at ever-decreasing length scales. In the field of the mechanical behaviour of materials, one of the more interesting small-scale phenomena is an increase in yield or flow strength that is often observed when the size of the test sample is reduced to micro-meter and sub-micrometer dimensions [43]. Pharr et al. [43] believed such size-dependent increases in strength were due to unique deformation phenomena which could be observed only when the sample dimensions approached the
average dislocation spacing and when plastic deformation was controlled by a limited number of defects.

In the metal micro-forming process, grain size, grain orientation and material’s dimension are the important influence factors for the material’s deformation. It is reported that when the dimension of the material is downscaled to a much smaller scale, the mechanical properties are significantly different from those on the macro-scale, which is called ‘size effect’ [44]. Size effect is an interesting and important topic for the development of micro-forming technologies. Recently, numerous experiments have shown that metallic materials display noticeable size effects once the size of non-uniform plastic deformation zone associated their characteristic length size are on the order of microns [44]. Fleck et al. [45] found a dramatic increase of plastic work hardening when the wire’s diameter decreased from 170 µm to 12 µm via doing thin copper wire’s torsion experiments. Stolken and Evans [46] observed that the plastic work hardening increased significantly when the nickel beam thickness was decreased from 50 µm to 12.5 µm while doing a micro-bend test. Gau et al. [47] found that the conventional concept of spring-back cannot be applied on brass sheet metal when its thickness is less than 350 µm by studying the spring-back behaviour of brass in micro-sheet forming. Geiger et al. [48] and Kals and Eckstein [49] have conducted compression tests, tension tests, and bending tests, respectively, in order to study how the material properties change due to size effect. Saotome et al. [50] carried out investigations in micro-deep drawing, and found that the relative punch diameter (punch diameter related to the sheet thickness) has a significant influence on the limit draw ratios (LDR).

The indentation size effect (ISE) is often observed for materials that are indented with geometrically self-similar indenters like pyramidal or conical tips (see Fig. 2.21) [43]. In general, the hardness, H, defined as the load on the indenter normalized with the projected contact area of the hardness impression (see Fig. 2.21), should be independent of the depth of penetration, h. However, over the past 60 years, it was observed that there were significant variations of hardness with respect to penetration depth, especially when the depths decreased to less than a few micro-meters [51-57]. In addition, two types of indentation size effects have been reported. One is the normal
ISE (the tip is pyramidal or conical)—the hardness increases with decreasing penetration depths, according to the expression “smaller is stronger” (see Fig. 2.21). Another one is the reverse ISE (the tip is spherical) [52, 53, 58], which displayed that the hardness decreases with increasing depths. However, for the reverse ISE, it was observed that the hardness also increases with the decreasing of tip radius. The reverse ISE is thought to be derived from testing artefacts such as vibration in the testing system or problems with accurate imaging and measuring the sizes of hardness impressions at dimensions approaching the limits of optical microscopy [43].

![Indentation size effect (ISE) for geometrically self-similar indenters such as a conical or pyramidal tip. [43]](image)

Although classical descriptions of the ISE show a decrease in hardness for increasing indentation depth (Fig. 2.22), recently new experiments [59] have shown that after the initial decrease, hardness increases with increasing indentation depth. After this increase, eventually the hardness decreases with increasing indentation (Fig. 2.23). This phenomenon is very prominent for copper, but not noticeable for aluminium.
Fig. 2.22 Visualization of the ISE: hardness decreases with increasing indentation depth for cold-worked polycrystalline copper. [59]

Fig. 2.23 Visualization of the ISE with the incorporated hardening effect. For $h < h_1$, hardness decreases, for $h_1 < h < h_2$, hardness increases and for $h > h_2$, hardness decreases again. [59]

There are different theories to explain ISE. The most popular theory is based on strain gradient plasticity which is a class of continuum theories aimed to bridge the gap between classical plasticity and dislocation. This theory assumes that the flow stress of metals depend on the density of statistically stored dislocations (SSD) which relate to
effective strain, and the density of the geometrically necessary dislocations (GND) which relate to the strain gradient. Dislocations are generated, moved and stored during the process of plastic deformation. The process of storing dislocation is also a process of strain hardening. It is assumed that dislocations become stored because they either accumulate by randomly trapping each other or they are required for compatible deformation of various parts of the material. When they randomly trap each other, they are often known as the statistically stored dislocation [60], whereas when they are required for the compatibility purpose, they are often called geometrically necessary dislocations and related to the gradient of plastic shear strain in a material [60, 61].

The most famous strain gradient plasticity model for nano-indentation was proposed by Nix and Gao [62] in 1998. It has been assumed that plastic deformation of the surface is accompanied by the generation of dislocation loops below the surface, which are contained in an approximately hemispherical volume below the region in contact, as shown schematically in Fig. 2.24. The deformation is self-similar and the angle (θ) between the conical indentation tip and indented surface remains constant.

![Fig. 2.24 Model of geometrically necessary dislocation for a conical indent: (a) Sample being indented by a rigid conical indenter; (b) Deformation loops created during indentation process. [59]](image)

The angle θ can be calculated by

\[
\tan(\theta) = \frac{h}{a} = \frac{b_G}{L_G}, \quad L_G = \frac{b_G a}{h}
\]

where \( h \) is the residual plastic depth, and \( a \) is the contact radius, and \( b_G \) is Burger’s vector. The number of geometrically necessary dislocation loops is \( h/b_G \). \( S \) is the
spacing between individual slip steps on the indentation surface, as shown in Fig. 2.24. Assume that $\lambda$ is the total length of the injected loops, thus between $r$ and $r+dr$ we have

$$d\lambda = 2\pi r \frac{dr}{s} = 2\pi r \frac{h}{ba} dr$$  \hspace{1cm} (2.7)

which after integration from 0 to $a$ gives the total length of dislocation loops,

$$\lambda = \int_{0}^{a} \frac{h}{ba} 2\pi r dr = \frac{\pi ha}{b}$$  \hspace{1cm} (2.8)

The model assumes that the dislocations are distributed uniformly in a hemispherical volume with the contact radius. Thus, we have $V = 2\pi a^3 / 3$, and therefore the density of geometrically necessary dislocation is

$$\rho_G = \frac{\lambda}{V} = \frac{3}{2bh} \tan^2 \theta$$  \hspace{1cm} (2.9)

Taylor hardening model has been used to find the shear strength which can be used to measure the deformation resistance.

$$\tau = \alpha \mu b \sqrt{\rho_T} = \alpha \mu b \sqrt{\rho_G + \rho_S}$$  \hspace{1cm} (2.10)

where $\tau$ is the resolved shear stress, $\mu$ is the shear modulus, $b$ is the Burgers vector and $\alpha$ is a constant which is usually in the range 0.3-0.6 for FCC metals [63]. Here, they note that $\rho_S$ does not depend on the depth of indentation. Rather it depends on the average strain in the indentation, which is related to the shape of the indenter($\tan(\theta)$). They also assume that the von Mises flow rule applies and that Tabor’ factor of 3 can be used to convert the equivalent flow stress to hardness:

$$\sigma = \sqrt{3} \tau, H = 3 \sigma$$  \hspace{1cm} (2.11)

According to these relations the hardness can be expressed by as

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}}$$  \hspace{1cm} (2.12)

where

$$H_0 = 3\sqrt{3} \alpha \mu b \sqrt{\rho_S}$$  \hspace{1cm} (2.13)
is the macroscopic hardness from the statistically stored dislocations alone, in the absence of any geometrically necessary dislocations, and

\[
h^* = \frac{81}{2} b \alpha^2 \tan^2 \theta \left( \frac{\mu}{H_0} \right)^2
\]  

(2.14)

is a length scale for the depth dependence of hardness.

From Eq. (2.12), it can be seen that the indentation hardness \( H \) is related only to indentation depth \( h \) as \( h^* \) and \( H_0 \) are material constants which can be obtained by fitting the experimental results.

After Nix and Gao [82], many other researchers continue to observe ISE through experiments and simulations. Most of them explained the size effect via the strain gradient theory (Ma and Clarke [64], Fleck et al. [45], Nix and Gao [62], Poole et al. [65], Stelmashenko et al. [66], Gao et al. [67, 68], Acharya and Bassani [69], Huang et al. [70], and Gurtin [71]). They believed that ISE must have a relationship with the strain gradient as the geometrically necessary dislocation density \( \rho_G \) is usually related to an effective strain gradient \( \eta \) as

\[
\rho_G = \frac{2\eta}{b}
\]  

(2.15)

According to Eq. (2.9), strain gradient was inversely proportional to the indentation depth \( h \), which meant strain gradient should be larger at the shallow depth.

However, Demir, Raabe and Zaafarani [72] expressed a different theory about ISE. They thought as \( \rho_G \) is the GND density that is required to accommodate a curvature \( \omega \), the crystallographic misorientation between two neighboring points can be used as an approximate measure for the GNDs as shown in the following Eq. (2.16).

\[
\rho_G = \frac{\omega}{b}
\]  

(2.16)

According to Eqs. (2.15) and (2.16), the size dependence of indentation hardness has been associated with strain gradients which exist in the lattice through GNDs. Thus, these researches decided to directly measure lattice rotations below indents with the aim
of quantifying the density of these defects. For this purpose they performed an experiment using a tomographic high-resolution electron backscatter diffraction orientation microscope in conjunction with a focused ion beam instrument to map the orientation distribution below four nanoindents of different depths. Unfortunately, the experimental result contradicted the commonly expected inverse relationship between the indentation depth and the density of the GNDs. In terms of GND-based strain gradient theories, larger GND densities should appear at shallow indentation depth, not at a deep one. But their experiment showed a opposite trend that the total GND density below the indents reduces with decreasing indentation depth. According to the experimental results, they concluded that the explanation size-dependent material strengthening effects by using average density measure for GNDs was not sufficient to understand the indentation size effect.

Kiener [54] investigated Nix-Gao model with the cross-sectional EBSD method and proposed that Nix-Gao model’s physical basis was still under debate, and its validity cannot be addressed alone with load versus displacement characteristics. There were two assumptions with respect to the Nix-Gao model, hemispherical plastic zone and self-similarity of the evolving deformation structure. However, according to the EBSD observation shown in Fig. 2.17, it is clear that the true plastic deformed zone deviates from the assumption of a half-sphere. Other reports indicated that there were differently shaped deformation areas, depending on the indenter geometry [42, 73, 74]. Regarding the self-similarity, based on the Nix-Gao model, the strain gradient induced by the indenter should be determined solely by the indenter geometry. Therefore, it should be constant for self-similar indenter shapes, which means the observed misorientations should depend only on the indenter angle and not on the indent size. However, the observations for Vickers indents in copper and tungsten displayed that the maximum misorientation was along the indent flanks for different sizes, which was shown as in Fig. 2.25.
Meyer’s law is another widely used method to describe ISE. For the indenters which have ideal geometry, the relationship between the test load and the resultant indentation diagonal length curve could be obtained from [75]

\[ P = C \cdot d^n \]  
\[ (2.17) \]

where \( P \) is the load, \( d \) is the diagonal length of impression, \( C \) is the material/indenter constant and \( n \) is the Meyer index due to the curvature of the curve. Since \( d \) is proportional to the contact depth \( h_c \) which in turn is proportional to the indentation depth \( h \). Eq. (2.17) could be expressed as follows:

\[ P = C' \cdot h^n \]  
\[ (2.18) \]

where \( C' \) is constant and \( h \) is the indentation depth.

Fischer-Cripps [76] mentioned that if the plastic zone was fully developed (beyond elastic-plastic transition point), the load-displacement (P-h) curve of the loading section could be related to the square of the displacement \( (P = C' h^2) \). As for the loading stage of the P-h curve in the elastic-plastic field, Sakai [77] stated that the load is proportional to the square of the indentation depth. According to Eq. (2.17), if \( n=2 \), the materials

---

**Fig. 2.25** Maximum misorientation angle measured along the flanks of the indent for different sized Vickers indents into fcc copper (closed squares) and bcc tungsten (open squares). [90,104]
shows no ISE. But if n<2, the materials shows ISE and this case was confirmed by different materials [78, 79].

Considering aforementioned facts, Ebisu and Horibe [75] analyzed the relationship between the P-h curve and ISE behaviour in their experiments by differentiating the P-h curves of the three samples. They found for single 8Y-FSZ(8 mol%Y₂O₃–ZrO₂ single crystal) in the load range of 200-1900 mN, the index n calculated from the P-h curve was 1.873, which means single 8Y-FSZ showed ISE behaviour. They also found this ISE result agreed well with the experimental results of another report [80] in which the hardness decreased as the indentation increased between the load range of 100-2000 mN. The same procedure was conducted in the 12Ce-TZP P-h curve and they found n=1.808 within the load range 200-1900 mN. It also was consistent with the results (ISE behaviour) from zirconia ceramics [80, 81]. However, for fused quartz, index n=1.961 calculated from P-h curve in the load range of 200-1900 mN meaned that this material showed almost no ISE behaviour [75]. This agreed with Oliver and Pharr’s report [19] which stated that quartz showed very little indentation size effect.

Kolemen [82] conducted the same experiments with superconductors, which showed a apparent ISE. He then concluded that for hard materials like brittle ceramics at low indentation loads, n is significantly less than 2. According to Onitsch [83], n lied between 1 and 1.6 for hard materials and higher than 1.6 for soft materils.

However, Peng et al. [84] pointed out the correlation between n and C seemed to be of little significance for understanding the ISE, as their previous study has showed that the best-fit value of the Meyer’s law coefficient C depended on the unit system used for recording the experimental data and completely different trends of n versus C may be observed in different unit systems [85] as shown in Fig. 2.26
Fig. 2.26 Variation of Meyer's law coefficient C with Meyer's exponent n. Note that completely different trends are observed when different unit systems were used for recording the experimental data. [85]

The first unit system used is P in Newton (N) and d in milli-meter (mm) and the second is P in gram (g) and d in micrometer (mm). As can be seen from Fig. 2.26, completely different trends of n versus A were observed in different unit systems. Similar conclusions were reported by Li and Bradt [86] when they analyzed the experimental data on single crystals. Thus, they concluded that a particular care should be taken when analyzing the microstructural effects on the measured hardness based on Meyer's power law.

It should be noted that only n plays an important role in determining ISE in Meyer's law and it is not necessary to consider the variation of Meyer's law coefficient C. Although numerous researchers used Meyer's law to analyze ISE, all of them just fit the whole P-h curve. In fact, the P-h curve can be fitted separately from the ISE-boundary which is shown as in Fig. 2.27. In the left side of ISE-boundary a n-value smaller than 2 can be obtained, while a n-value of about 2 can be achieved in the right side.
Some other factors may influence the ISE, which include: inadequate measurement capabilities of extremely small indents [87], presence of oxides or chemical contamination on the surface [88], indenter-sample friction [89], and increased dominance of edge effects with shallow indents [64].

Elmustafa and Stone [90] proposed a vast number of hypotheses to explain the ISE, including: friction, and lack of measurement capabilities, and surface layers, oxides, chemical contamination and dislocation mechanisms. To calculate the hardness for the nano-indentation measurements, indents were imaged in calibrated optical and scanning electron microscopes. However, because of the inaccuracies inherent to “optical” method, they did not just rely on them alone. In addition, they used contact stiffness as a method to determine indirectly the projected contact area. Interestingly, the two methods agreed very well.

Elmustafa et al. [90] conducted experiments using alpha brass and aluminium as samples. It has been found that the oxide on a fresh surface of alpha brass is less than 5 nm thick, while the oxide thickness on an aluminium surface is only 1-3 nm [91]. For Elmustafa et al.’s experiment, the smallest indents were approximately 0.3 μm across or 60 times larger in lateral dimensions than the oxide thickness. Therefore, the oxide layer would not greatly affect the hardness.

The indenter piling-up or sinking-in was also seen as a factor that influences ISE. McElhaney et al. [92] did numerous experiments and found that the indenter piling-up
and sinking-in had a huge effect on the micro-indentation hardness. However, after very careful examination, they found the indentation hardness still displayed strong dependence on the penetration depth. This observation displayed that the indenter piling-up and sinking-in cannot explain the depth-dependent indentation hardness alone.

Loading rate was raised as another influence factor for ISE because usually materials tended to display larger a plastic work hardening at a large strain rate (loading rate) [93]. The strain rate in the indented material should be proportional to the ratio \( \frac{\dot{h}}{h} \), where \( \dot{h} \) is the rate of change of the indentation depth and \( h \) is the indentation depth. It is not hard to understand that for a constant rate of indentation depth (\( \dot{h} = \text{constant} \)), the strain rate should be very huge at the initial penetration. However, Lilleodden [94] performed indentation test at a constant ratio \( \frac{\dot{h}}{h} \) and still observed the phenomenon of ISE.

Indenter tip radius was regarded as another factor which affected ISE. However, McElhaney et al. [92] and Huang et al. [95] found that the sharp indenter tip radius less than 100 nm had definitely no influence on the micron and sub-micron scale indentation, which indicated that indenter tip radius cannot be used to explain the ISE observed in the indentation test with sharp indenters.

### 2.5 Simulations of nano-indentation

#### 2.5.1 Conventional finite element method (FEM) simulations

Lee and Kobayashi’s work [96] was the first to conduct the finite element simulation (FEM) of indentation in 1969. Plane strain and axisymmetric flat punch indentation was simulated to study the development of the plastic zone, the load-displacement relationships, and the stress and strain distributions during continued loading, taking into account the changes of the punch friction and sample dimensions. However, problems such as the accuracy of the solutions and the efficiency of the computation still existed. Then three years later, Lee and his colleagues [97] performed another finite element simulation of indentation using ball indenter, and compared all the results with
their own experiments for heat-treated SAE4340 steel. It was found that the simulated load-displacement curve, plastic zone development and indentation pressure were in good agreement with the experimental observations. In addition, by calculating the mean effective strains with FEM, the representative strains defined by Tabor were found to be equal to the mean effective strains of the plastic zone under the indenters shown in Fig. 2.28.

![Fig. 2.28 Strain contours and elastic-plastic boundaries (— FEM; ---- experiment). [97]](image)

Bhattacharya and Nix [98] performed elasto plastic FEM simulations of nano-indentation using conical indenter to study the elastic and plastic properties of materials on a sub-micro scale under the conditions of frictionless and completely adhesive contact. The simulated load-displacement curves for nickel and silicon were compared with experimental results as shown in Fig. 2.29 and Fig. 2.30, respectively. It was concluded that the FEM is suitable to simulate nano-indentation behaviour at a sub-
micro scale for different types of materials. Bhattacharya and Nix [99] then investigated the relationship between Young’s modulus and yield strength, and concluded from FEM simulation that the shapes of the plastic zones for an elastic-plastic bulk material under a conical indenter depend strongly on the ratio $E/\sigma_y$ (Young’s modulus / yield stress) with a fixed indenter angle as shown in Fig. 2.31.

Fig. 2.29 Comparison between the results from the present FEM analysis and those from Pethica et al. [100] on indentation of nickel. [98]

Fig. 2.30 Comparison between the results from the present FEM analysis and those from Pethica et al. [100] on indentation of silicon. [98]
Subsequently, FEM nano-indentation simulation of thin films [101-104], stress distribution [105-107], hardness [108-110], friction effects [103, 111], brittle cracking behaviour [112-114] and coatings [115, 116] were extensively developed.

Most of the researchers thought the conventional plasticity theory cannot be used to explain ISE because its constitutive models possess no intrinsic (internal) material lengths. However, Storakers et al. [117] did observe the reversed ISE through simulation by using a parabola-shaped indenter.

### 2.5.2 Crystal plasticity FEM simulation

The evolution of crystallographic texture and grain lattice rotation under the indentation has not been well understood. This work must be done through the crystal plasticity based simulation.

Casals and Forest [118] investigated the anisotropy in the contact response of FCC and HCP (hexagonal closest-packed) single crystal via simulating spherical indentation experiments of bulk single crystals and thin films on hard substrates. Their simulations predicted that the plastic zone beneath the indenter preferentially grew along the slip system directions as shown in Fig. 2.32 and Fig. 2.33. Consequently, in coated thin film...
systems, a prominent localization of plastic deformation occurred at those specific regions where slip system directions intersected the substrate. Meanwhile, these specific areas were prone to crack nucleation in terms of accumulative plastic damage. Therefore, the identification of these areas was meaningful for the prediction of potential delamination and failure of the coatings. Casals et al. [119] also used three-dimensional crystal plasticity finite element simulations to examine Vickers and Berkovich indentation experiments of strain-hardened copper. The results showed that the simulation was in a good agreement with experimental observations with respect to hardness, load-displacement curves, material piling-up and sinking-in development at the contact boundary.

![Fig. 2.32 Details of the indentation-induced plastic zone in the simulations concerning FCC copper crystals. (a), (c) and (e) correspond to the (001), (011) and (111) indented](image)

Fig. 2.32 Details of the indentation-induced plastic zone in the simulations concerning FCC copper crystals. (a), (c) and (e) correspond to the (001), (011) and (111) indented
planes of bulk crystals. (b), (d) and (f) correspond to their thin film counterparts. Plastic zone is assessed by considering the total accumulated plastic strain variable. White arrows point to the specific locations where high plastic strain localization occurs within the coating substrate interface. Penetration depth in the figures is \( h_a = 3.5\mu m \). [118]

Alcala et al. [120] analysed Vickers and Berkovich indentation behaviour via extensive crystal plasticity finite element simulation by recourse to the Bassani and Wu hardening model for pure FCC crystals. The simulated results have been used to illustrate the impact of the crystallographic orientation, as shown in Fig. 2.34. It was clear that the irregular appearance of pyramidal indentations was governed by the crystallography of FCC crystals on the indented surface.

![Fig. 2.33 Details of the indentation-induced plastic zone in the simulations concerning HCP zinc crystals. (a) and (c) correspond to the basal and prismatic indented planes of bulk crystals. (b) and (d) correspond to their thin film counterparts. White arrows point to the specific locations where high plastic strain localization occurs within the coating substrate interface. Penetration depth in the figures is \( h_a = 3.5\mu m \). [118]](image-url)
Fig. 2.34 Imprint morphologies for Vickers indentation in the (0 01) plane for different orientations (rotations) of the tip. (a) The slip directions at the surface coincide with the edges of the indenter (arrows indicate development of pincushion effects). (b) The slip directions coincide with the sides of the indenter. (c) Intermediate orientation to those described in (a) and (b). [120]

Liu et al. [121] implemented crystal plasticity constitutive model initially developed by Peirce et al. [122] in a finite element code Abaqus/Explicit to study the material behaviour of nano-indentation on (001) oriented surface of single crystal copper. All of the appropriate meso-plastic parameters used in the hardening model was determined by fitting the simulated load–displacement curves to the experimental data. Their studies demonstrated that the combined nano-indentation/CPFEM simulation approach for
determining mesoplastic model parameters works reasonably well from micro level to the macro level as shown in Fig. 2.35. They also investigated the orientation effects in nano-indentation of single crystal copper [123]. Simulated load-displacement curves were found to be in agreement with those from experimental tests as shown in Fig. 2.36. Meanwhile, two-, three-, and four-fold symmetric piling-up patterns were observed on (011), (111), and (100) oriented surface with respect to CPFEM simulation. The anisotropic nature of the surface topographies around the imprints in different crystallographic orientations of the single crystal copper samples then were related to the active slip systems and local texture variations. Wang et al. [37] had similar observations while performing a 3D elastic-viscoplastic crystal plasticity finite element method simulation to study the dependence of nano-indentation piling-up patterns. Their simulation showed that the piling-up patterns on the surface of (001)-, (011)- and (111)-oriented single crystal copper had four-, two-, and sixfold symmetry, respectively. All the simulated piling-up patterns were in agreement with those from the experiments. The explanations of the anisotropic surface profiles were also related to the active slip systems and local texture variations.

Fig. 2.35 (a) Results of FEM simulations showing the variation of the indentation force, F with indent diameter, 2a for different indenter radii, R; (b) Results of indentation experiments showing the variation of the indentation force, F with indent diameter, 2a for different indenter radii, R. [121]
Zaafarani et al. [42] carried out the 3D elastic–viscoplastic crystal plasticity finite element simulations with the same geometry of indenter and boundary conditions as those from experiments. Their CPFEM simulations predicted a similar pattern for the absolute orientation changes as the experiments as shown in Fig. 2.37. However, it was found that the simulations over-emphasized the magnitude of the rotation field tangent to the indenter relative to that directly below the indenter tip. The reason was then found to be due to edge effects at the contact zone and milling-induced curvature caused by ion beam so that no complete EBSD mapping could be made up to the actual contact interface [124].
Eidel [125] simulated pyramidal micro-indentation on the (001) surface of Ni-base superalloy single crystal with three different azimuthal orientations of the pyramidal indenter. The numerical piling-up patterns were compared with the experimental results. It was found that the resultant material piling-up was insensitive to different azimuthal orientations of the pyramidal indenter as shown in Fig. 2.38. The reason could be due to the piling-up formation determined by crystallographic processes rather than by the stress distribution pattern, induced under the non-isotropic pyramidal indenter. He then also found that the piling-up was independent of the indenter shape (sphere or pyramid) and the elastic anisotropy, which further confirmed that only the geometry of the slip systems in the (001) oriented crystal governed piling-up, whereas stress concentrations introduced by different indenter shapes, by the azimuthal orientation of a pyramidal indenter and also by the characteristics of the elasticity law, had no significant influence.
Fig. 2.38 Pyramidal indentation experiments into (001) fcc CMSX-4. Left: experiment (SEM); right: simulation with isolines of height, uz (µm), for azimuthal orientation angle θ = 0°, 22.5°, 45° in row 1–3. In the coordinate system, X-, Y-, Z-axes each represent h001i directions. The white stains in the experimental indentation craters are debris from sputtering. [125]
2.6 Summary

Nano-indentation is the most popular method to investigate the mechanical properties of materials in micro- or nano-scale. The main advantage of this technology is its convenience and applicable on a very small sample which normal tensile test cannot be applied. A vast number of experiments have been conducted to investigate different characters during indentation deformation, including hardness, Young’s modulus, load-displacement curve, ISE (indentation size effect), piling-up and sinking in, cracks, texture evolution and lattice rotation and so on. Meanwhile, a wide range of materials were studied, such as metals, ceramics, rubbers, human bones, coatings, etc.

Indentation size effect has been extensively studied by numerous researchers. Among all of these researchers, Nix and Gao has proposed a strain gradient model which was believed to be the best way to simulate and explain the ISE in the past. However, it has been found that the main assumptions for that model are in conflict with the experimental observations. Other potential influence factors, such as inadequate measurement capabilities of extremetly small indents, presence of oxides or chemical contamination on the surface, indenter-sample friction, increased dominance of edge effects with shallow indents and tip radius have been eventually proven to be ineffective in determining the ISE.

The conventional FEM has been used to study the indentation process to predict hardness, stress distribution, friction effects, Young’s Modulus and load-displacement curves, and brittle cracking behaviour and so on. However, the anisotropy of materials could not be taken into account in the conventional FEM. Crystal plasticity FEM (CPFEM) has then been used to simulate the texture evolution, lattice rotation, initial orientation effects etc.
Chapter 3 Experimental investigation of nano-indentation of aluminium single crystal

This chapter aims to carry out experimental studies of mechanical properties of nano-indentation, including load-displacement curve, Young’s modulus, hardness and surface profiles, on aluminium single crystal samples with different initial orientations. The experimental observations will be compared with the simulated results.

3.1 Material and sample preparations

The material used in the nano-indentation tests was Al single crystal with a purity of 99.9999% wt.%, provided by MaTecK. The detailed information of the raw materials is shown in Table 3.1.

<table>
<thead>
<tr>
<th>Crystal structure</th>
<th>Production method</th>
<th>Sample size</th>
<th>Orientation</th>
<th>Orientation accuracy</th>
<th>Roughness of surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>FCC</td>
<td>Bridgman</td>
<td>Dia.15mm</td>
<td>(100),(101),(111)</td>
<td>&lt;0.1°</td>
<td>&lt;10nm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Thickness 2mm</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The Al single crystal disk samples with three different orientations were prepared for the nano-indentation tests. The diameter and thickness of the samples were 15 mm and 2 mm, respectively. Three orientations are (100), (101) and (111) parallel to the sample surface. All the samples were electro-polished before indentation. The roughness of polished surface is less than 10 nm, measured by an atomic force microscope (AFM). The Electron Backscattering Diffraction (EBSD) technique was used to measure the distribution of the crystallographic orientation after the nano-indentation tests.

3.2 Experimental instruments

3.2.1 IBIS/UMIS nano-indentation system

The indentation tests were carried out using IBIS/UMIS nano-indentation system which was manufactured by CSIRO Division of Applied Physics in Sydney, Australia. Fig. 3.1
shows a photo of the IBIS/UMIS nano-indentation system in the tribology laboratory at the University of Wollongong. The IBIS/UMIS nano-indentation system can continuously record load verse depth curves with a resolution of 75 nN in load and 0.05 nm in depth. The system can provide a wide load range from 100 µN up to 500 mN.

Three-sided diamond Berkovich indenter with a radius of 200 nm was used. The area function of the indenter and the machine compliance has been calibrated on a standard sample of fused silicon from 1 mN to 400 mN before the nano-indentation tests. All nano-indentation tests were conducted in air at room temperature and during night time in order to get rid of the thermal drift and other effects. 200 data points were recorded automatically for the indentation load and displacement during loading and unloading process, individually.

![IBIS/UMIS nano-indentation system](image)

**Fig. 3.1** IBIS/UMIS nano-indentation system.

### 3.2.2 Scanning electron microscope (SEM) JEOL JSM-7001F

The JEOL JSM-7001F was used to measure the crystallographic orientation. It is a 30 kV analytical thermal field emission gun scanning electron microscope (FEG SEM) capable of 3 nm spatial resolution. It is well suited for the detailed characterisation of conductive/coated samples. The large area EDS detector enables rapid quantitative microanalysis and elemental mapping while the high resolution EBSD system enables
orientation mapping in scanning and transmission modes. The system is shown as in Fig. 3.2.

**Fig. 3.2** JEOL JSM-7001F Basic system.

### 3.2.3 Focus ion beam microscope-XT Nova Nanolab 200

The XT Nova NanoLab 200 was used to polish the cross-sections of indents for further TEM observations. The system combines a dual beam high resolution fociussed ion beam (FIB) and a high resolution scanning electron microscope. It is equipped with platinum deposition system, an iodine enhanced etching and a SiOx deposition. The FIB uses a fine, energetic beam of gallium ions that scan over the surface of a sample. At high beam currents the gallium beam rapidly sputters away the sample surface which allows subsurface cross-sections to be prepared. If the beam current is reduced, the secondary electrons, or secondary ions emitted from the sample surface, can be detected and used to form high resolution images. The FIB is therefore widely used in the examination of materials such as semiconductors, solar cells, thin films, where structural information about the subsurface is required. The FIB can be used for nano-milling very thin (~100 nm) sections, suitable for TEM examination, of traditionally challenging materials such as semiconductors, internally oxidized steels and powder
materials. During the FIB cross sectioning of indents on Al single crystals, an accelerating voltage of 30 KV and high ion currents were used, and lower currents of 1 nA and 0.5 nA for nano-milling. Fig. 3.3 shows a photo of the XT Nova Nanolab 200 system.

![XT Nova Nanolab 200 system](image1.jpg)

**Fig. 3.3** XT Nova Nanolab 200 system.

### 3.2.4 Transmission electron microscope- Phillips CM200

A Philips CM200 field emission gun transmission electron microscope (FEG-TEM) equipped with an energy dispersive X-ray spectroscopy (EDAX) system was used to investigate the lattice rotation angles of FIBed cross-sections of the indents comparing with undeformed regions of Al single crystals. The Philips CM200 field emission gun transmission electron microscope allows very high resolution images to be obtained from thin, electron transparent sections of materials. Ultimately, this microscope allows individual atoms to be imaged. The TEM allows not only structural information to be obtained, but also crystallographic studies of materials are routinely possible. This microscope has an EDAX energy dispersive x-ray spectroscopy system interfaced to it, which can allow chemical analysis from regions as small as 10 nm in diameter to be obtained. In addition, it has a SIS CCD camera for direct recording of digital images. An accelerating voltage of 200 KV was used in this study. The whole TEM system is shown as in Fig. 3.4.
Fig. 3.4 Philips CM200 TEM system.

3.2.5 Atomic force microscope

A multi-mode scanning probe microscopy (SPM) from Digital Instruments operating in contact AFM mode (Nanoscope IIIA AFM) as shown in Fig. 3.5 was used to obtain piling-up patterns of the indented surfaces of Al single crystals. The AFM has a lateral resolution of 1-5 nm and a vertical resolution of 0.08 nm.

The V-shaped nano-probe cantilevers were made of silicon nitride (Si₃N₄) with a nominal tip radius of 20-60 nm. The lengths of cantilevers were 100 µm and 200 µm. The tip is a four sided pyramid with a 35° angles between every two sides. The tip with 0.12 m/s spring constant was used to scan the indenter surfaces of Al single crystals. The Digital Nanoscope software version 5.12b was used to analyse the surface profiles of the indented samples [126].
Chapter 3 Experimental investigation of nano-indentation of aluminium single crystal

3.3 Experimental procedure of nano-indentation

Each electro-polished sample was mounted separately on the smooth surface of an aluminium cylinder with a thin layer of epoxy glue. A 6 x 6 indentation matrixes were conducted on each individual sample. All the indents were located in the middle of the sample and far away from the edge of the sample in order to avoid the effects by the edge. Meanwhile, the distance between each indent was set as 200 µm which was more than 10 times the indent impression diameter to avoid the mutual interaction. After the nano-indentation tests, the EBSD was used to scan the indented surface to obtain the accurate orientation of the indent. During the scanning, the indented surface was set as perpendicular to the normal direction (ND) and one edge of one chosen indent was set as parallel to the rolling direction (RD). The Euler angles from the EBSD measurements then were transferred to Miller index which could be substituted into crystal plasticity simulation. AFM also was used to obtain the surface topographies and the piling-up profiles by scanning the indented surfaces. (101) initial oriented sample was chosen to do the FIB test in order to obtain the cross-sectional sample of the indent. TEM tests were then conducted on the FIBed sample to analyse the lattice rotation angles using selected area diffraction patterns. All the experimental data are then compared with the simulation results in Chapter 5.

Fig. 3.5 The Multi-Mode Scanning Probe Microscopy (SPM).
3.4 Results

Fig. 3.6 shows the experimental load-displacement curves for three different samples.

![Fig. 3.6 Experimental load-displacement curves for single crystal aluminium: (a) (001) surface; (b) (101) surface; (c) (111) surface.](image)

Elastic modulus was calculated based on the curve of Fig. 3.6 by using the following equations proposed by Oliver and Pharr [19, 127]

\[
h_i = h_{\text{max}} - k \frac{P_{\text{max}}}{S}, k = 0.75, S = \left( \frac{dP}{dh} \right)_{h=h_{\text{max}}} \tag{4.1}
\]

\[
A_c = 3\sqrt{3}h_c^2 \tan^2 65.3 = 24.56h_c^2, H = \frac{P}{A_c} \tag{4.2}
\]

\[
E_r = \frac{dP}{dh} 2h_c \frac{1}{\beta} \sqrt{\frac{\pi}{24.5}}, \beta = 1.034 \tag{4.3}
\]

\[
\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i} \tag{4.4}
\]
where $h_c$ represents the contact depth, the factor $k$ depends upon the indenter. $A_c$ is the contact area. For the spherical indenter (including Berkovich) $k=0.75$. For the conical indenter $k=0.72$. $S$ denotes the slope of the unloading curve. $\beta$ is relative to the indenter, and it equals 1.00, 1.034, or 1.012 for the conical, Berkovich, or Vickers indenter individually. $E_r$ represents the residual elastic modulus, $E_i$ represents elastic modulus of the diamond indenter, and $E$ is elastic modulus of the measured sample. $v$ is the Poisson’s ratio. Poisson’s ratio for aluminium is 0.32-0.36, but 0.347 is commonly used. $E_i=1141 \text{ GPa}$ and $v_i=0.07$ was used in this study [19].

![Graph](image)

**Fig. 3.7** Experimental results for single crystal aluminium: (a) Hardness; (b) Elastic modulus.

Fig. 3.7 shows the average experimental hardness and elastic modulus based on all of the valid results of three single crystals. The indentation elastic modulus for the (001), (101) and (111) crystals are 63.18 GPa, 71.79 GPa and 75.09 GPa, respectively. The elastic modulus values of the (111) crystal is ~15.9% larger than that of the (001) crystal. Wang and Lu [128] found the difference in measured indentation modulus between the (111) and (001) Cu crystals is ~20%. The standard elastic modulus of Al single crystal along different orientations can be calculated by the following equations [129].
$S_{11} = (C_{11} + C_{12})/\{(C_{11} - C_{12})(C_{11} + 2C_{12})\}$  \hspace{1cm} (4.5)

$S_{12} = -C_{12}/\{(C_{11} - C_{12})(C_{11} + 2C_{12})\}$  \hspace{1cm} (4.6)

$S_{44} = 1/C_{44}$  \hspace{1cm} (4.7)

$\frac{1}{E_{ijk}} = S_{11} - 2[S_{11} - S_{12}] - \frac{1}{2}S_{44}(l^2m^2 + m^2n^2 + l^2n^2)$  \hspace{1cm} (4.8)

where, $E_{ijk}$ is the elastic modulus in the $[i\ j\ k]$ crystallographic direction, l, m and n are the direction cosines of the $[i\ j\ k]$ direction, $C_{11}$, $C_{12}$ and $C_{44}$ are the elastic constant values. It has been known that $C_{11}=112$ GPa, $C_{12}=66$ GPa and $C_{44}=28$ GPa [130]. Therefore, the standard elastic modulus calculated by Eq. (4.8) for three tested orientation are $E_{001}=63.14$ GPa, $E_{101}=71.56$ GPa and $E_{111}=74.98$ GPa, respectively. It is clear that the measured elastic moduli are in good agreement with standard values[131].

In addition to the elastic modulus anisotropy, the hardness anisotropy is also observed by our experiments. The reason has been attributed to the fact that the resolved shear stress needed for the motivation of slide systems on different crystallographic planes is different [132-134]. The hardness values from indentation tests for (001), (101) and (111) crystals are 0.3665 GPa, 0.3573 GPa and 0.3991 GPa, respectively. The hardness values of the (101) and (001) crystals are much lower than that of the (111) crystal.
Fig. 3.8 AFM images of the indent impressions made on a single crystal aluminium work piece with a Berkovich indenter (tip radius 200nm) at different crystallographic orientations: (a) (001) surface; (b) (101) surface; (c) (111) surface.

Fig. 3.8 (a)-(c) shows AFM images of the indent with a Berkovich indenter on three different crystals. The bright colour represents the piling-up. The figure displaces the fourfold symmetry of the height profile for the (001) crystal, the twofold symmetry for the (101) crystal and the threefold symmetry for the (111) crystal. Flom and Komanduri [135] who did the indentation tests on the (011) and (111) surfaces of single crystal aluminium with a sapphire stylus indenter found the same phenomenon. Hollatz et al. [136] found a fourfold, twofold, and threefold symmetry on the (001), (011), and (111) initial oriented surface of NiAl single crystal, respectively. Liu et al. [15] found a fourfold, twofold, and threefold symmetry on the (001), (011), and (111) initial oriented surface of copper single crystal. However, the indenters used in their researches were spherical indenters.
Fig. 3.9 shows the selected indent for EBSD scanning. The indented surface was set perpendicular to the ND and one edge of the indent was set parallel to the RD.

![Selected indent for EBSD scanning](image)

**Fig. 3.9** The selected indent for EBSD scanning.

Fig. 3.10 shows the IPF (Inverse Pole Figure) mapping of the selected indents on three different initial oriented surfaces. The measured orientation at the location far from the indent was used in the CPFEM simulations as the initial orientation.

![EBSD IPF mapping](image)

**Fig. 3.10** EBSD IPF mapping of the selected indent on different initial oriented surface: (a) (001) surface; (b) (101) surface; (c) (111) surface.
**Fig. 3.11** FIB micrographs of a 10mN indent on the (101) surface before lift-out.

Fig. 3.11 shows the cross-section through the centre of a 10 mN indentation impression on the (101) surface before lift-out, which was fabricated using a focused ion beam workstation (XT Nova Nanolab 200). The centre of the impression was marked first in order to obtain cross-section which proceed right though the middle of the indent and then a tungsten layer with a thickness of about 500 nm was deposited on the surface to avoid the ion damage.

**Fig. 3.12** Cross-sectional TEM micrographs of 10mN indent on the (101) surface, taken with a 11̅T two beam condition.
Fig. 3.12 presents the bright field image of a 10 mN indenter with marked number from 1 to 8. The number marked in the image represents the positions where the selected area aperture was positioned. SAED was also performed on the cross-sectional TEM sample at the marked positions. The results are shown as Fig. 3.13. Lattice rotation angles along the X axis (RD) then can be determined by comparing the SAED patterns from deformed area marked number 2 to 8 with undeformed area marked number 1. The results are shown in Table 3.2.

Fig. 3.13 Selected area diffraction patterns which correspond to the regions marked from number 1 to 8.
Table 3.2 Lattice rotation angles correspond to the regions marked from number 1 to 8.

<table>
<thead>
<tr>
<th>No.</th>
<th>Rotation angles</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0°</td>
<td>-1°</td>
</tr>
<tr>
<td>2</td>
<td>-9°</td>
<td>-12°</td>
</tr>
<tr>
<td>3</td>
<td>-7°</td>
<td>5°</td>
</tr>
<tr>
<td>4</td>
<td>4°</td>
<td>3°</td>
</tr>
</tbody>
</table>

In Table 3.2 the positive value means the counter-clockwise (CCW) rotation from the undeformed orientation, while the negative value means the clockwise (CW).

3.5 Summary

This chapter can be summarized as follow:

1. The nano-indentation tests were performed on the initial (001), (101) and (111) oriented surfaces of single crystal aluminium by using IBIS/UMIS nano-indentation system.

2. AFM tests were carried out on the indented surfaces and displaced the fourfold symmetry of the height profile for the (001) oriented crystal, the twofold symmetry for the (101) oriented crystal and the threefold symmetry for the (111) oriented crystal.

3. Cross sectional TEM sample corresponding to the indent impression on the (101) initial oriented surface was fabricated using FIB, and SAED was then performed to analyze the lattice rotation angle induced by indentation.
Chapter 4 Crystal plasticity constitutive model and numerical implementation

This chapter presents the basic explanations of crystal plasticity theory and the implementation of the crystal plasticity constitutive models into a commercial FEM framework. The developed crystal plasticity FEM model in this chapter is aiming to simulate the non-uniform deformation behaviour, in-grain texture formation and evolution of aluminium single crystal during nano-indentation.

As a general scheme of notation, boldface lowercase letters (e.g., \( \mathbf{a} \)) represents vectors in the subsequent context, while boldface capital letters (e.g., \( \mathbf{A} \)) represents tensor and matrices. Cartesian components of vectors and tensors are written as, \( a_i \) and \( A_{ij} \), respectively. All inner products are indicated by a single dot and the tensor product is indicated as “\( \otimes \)”. The superscript “-1” of a matrix (e.g., \( \mathbf{A}^{-1} \)) indicates the inverse and the superscript “T” of a matrix ((e.g., \( \mathbf{A}^T \)) denotes the transposition of this matrix. The summation convention is used for Latin indices but summations over crystallographic slip systems are indicated explicitly. Time derivatives are denoted by superposed dots.

4.1 Crystal plasticity theory

The first finite element (FE) simulation was performed by Courant in 1943 [137] while the first CPFE simulations were performed by Peirce, Asaro and Needleman in 1982 [122] to numerically analyze the non-uniform and localized deformations of ductile single crystals which subject to tensile loading. This research then was followed by Harren et al. [138, 139] who applied CPFE simulation on poly-crystal analysis via a 2D setup. 3D setup then was investigated by Becker et al. [140, 141] who successfully simulated texture evolution of the channel-die compression of both aluminium single crystal and poly-crystal. Currently the CPFEM as a multi-physics framework is extensively used in both microscopic and macroscopic scales. For instance, indentation [37, 42, 59, 120, 121, 123, 124, 142, 143], pillar compressing [144-146], the prediction of local lattice curvatures and mechanical size effects [59, 93] all belong to microscopic scale. For macroscopic applications, ECAP [130, 147], HPT [148, 149], rolling [150-152] and crack [153, 154] are most popular.
4.1.1 Kinematical theory

The quantitative description of kinematical theory for the mechanics of elastic-plastic deformation of crystals was derived from the early work of Taylor [155] and Hill [156], who indicated that material flows through the crystal lattice via dislocations gliding along the corresponding slip systems. The constitutive models used here followed the earlier work done by Asaro [157], Asaro and Rice [158], Si [23] and Huynh [150, 159].

According to the crystal plasticity theory, the crystalline material under load undergoes crystallographic slip due to dislocation motion on the active slip systems and elastic deformation including stretching and rotating of the crystal lattice [122, 150, 157, 159-162], which leads to a hypothesis that the crystallographic may be supposed to firstly occur from the reference configuration to the intermediate configuration, followed by the elastic stretching and rotation from the intermediate configuration to the current configuration. Therefore, three configurations have been proposed to describe the deformation process, which is shown as in Fig. 4.1. The initial configuration corresponds to the original un-deformed state of the element and the current configuration to the deformed state of the element. The intermediate configuration is obtained from the current configuration when relaxing the lattice elastic stretching and rotation. The total deformation gradient \( F \) can be decomposed into two different components, namely \( F^* \) and \( F^p \) as

![Diagram showing decomposition of the total deformation gradient](image)

**Fig. 4.1** Decomposition of the total deformation gradient \( F \) into elastic deformation component \( F^* \) and plastic deformation component \( F^p \).
\[
F = \frac{\partial \mathbf{x}}{\partial \mathbf{X}} = \mathbf{F}^* \cdot \mathbf{F}^P 
\]  
(4.1)

Where \( \mathbf{X} \) represents the position of material points in the reference configuration and \( \mathbf{x} \) denotes the position of material points in the current configuration. \( \mathbf{F} \) is the total deformation gradient, \( \mathbf{F}^* \) describes the elastic deformation gradient including the stretching and rotation of the crystal lattice, and \( \mathbf{F}^P \) indicates crystallographic slip on the slip system, which can be written as

\[
\mathbf{F}^P = \sum_{\alpha=1}^{N} \mathbf{F}^{(\alpha)P} = \sum_{\alpha=1}^{N} \mathbf{I} + \gamma^{(\alpha)} \left( \mathbf{s}_0^{(\alpha)} \otimes \mathbf{m}_0^{(\alpha)} \right) 
\]  
(4.2)

Where \( \mathbf{F}^{(\alpha)P} \) is the contribution of the \( \alpha \)-th slip system to \( \mathbf{F}^P \), \( \gamma^{(\alpha)} \) is the shear strain of \( \alpha \)-th slip system, \( \otimes \) indicates the dyadic product of two tensors or vectors, \( \mathbf{I} \) is a second-order unit tensor and \( N \) is the number of activated slip systems.

The Green strain tensor can be written as

\[
\mathbf{E} = \frac{1}{2} (\mathbf{F}^T \mathbf{F} - \mathbf{I}) 
\]  
(4.3)

In the initial configuration, the crystal slip system \( \alpha \) consist of a slip direction vector \( \mathbf{s}_0^{(\alpha)} \) lying in a slip plane with normal vector of \( \mathbf{m}_0^{(\alpha)} \). Both \( \mathbf{s}_0^{(\alpha)} \) and \( \mathbf{m}_0^{(\alpha)} \) are unit vectors and should satisfy the orthogonal relationship, namely.

\[
\mathbf{s}_0^{(\alpha)} \cdot \mathbf{m}_0^{(\alpha)} = 0 
\]  
(4.4)

Both \( \mathbf{s}_0^{(\alpha)} \) and \( \mathbf{m}_0^{(\alpha)} \) will not be changed during crystallographic slip from the initial configuration to the intermediate configuration but converted with the lattice when the lattice is stretched and rotated from the intermediate to the current configuration. The slip direction vector \( \mathbf{s}_0^{(\alpha)} \) can be given in the deformed configuration by

\[
\mathbf{s}^{(\alpha)} = \mathbf{F}^{(\alpha)} \cdot \mathbf{s}_0^{(\alpha)} = \mathbf{F}^* \left( \sum_{\alpha=1}^{N} \mathbf{I} + \gamma^{(\alpha)} \left( \mathbf{s}_0^{(\alpha)} \otimes \mathbf{m}_0^{(\alpha)} \right) \right) \mathbf{s}_0^{(\alpha)} = \mathbf{F}^* \cdot \mathbf{s}_0^{(\alpha)} 
\]  
(4.5)

The normal to the slip plane \( \mathbf{m}_0^{(\alpha)} \) after deformation can be written as

\[
\mathbf{m}^{(\alpha)} = \mathbf{s}^{(\alpha)-1} = \left( \mathbf{F}^* \cdot \mathbf{s}_0^{(\alpha)} \right)^{-1} = \mathbf{m}_0^{(\alpha)} \cdot \mathbf{F}^*^{-1} 
\]  
(4.6)
It should be noted that \( s^{(\alpha)} \) and \( m^{(\alpha)} \) are not in general unit vectors, but remain orthogonal.

\[
s^{(\alpha)} \cdot m^{(\alpha)} = 0 \quad (4.7)
\]

The current velocity gradient is evaluated from the deformation gradient by

\[
L = \frac{\partial v}{\partial x} = \frac{\partial v}{\partial X} = \dot{F}F^{-1} = L^* + L^P
\quad (4.8)
\]

\[
L^* = \dot{F}^* \cdot F^* \quad (4.9)
\]

\[
L^P = F^* \cdot \dot{F}^P \cdot F^P \quad (4.10)
\]

where \( v \) is the velocity of the material point in the current configuration, \( \dot{F} \) represents a time derivative of \( F \), \( L^* \) is the elastic component of the velocity gradient and \( L^P \) is the plastic component of the velocity gradient.

From Eq. (4.2) \( L^P \) can be derived as

\[
L^P = \sum_{\alpha=1}^{N} \dot{\gamma}^{(\alpha)} s^{(\alpha)} \otimes m^{(\alpha)} \quad (4.11)
\]

It is obviously it is related to the shear strain rate \( \dot{\gamma}^{(\alpha)} \) caused by the plastic slip in the \( \alpha \)-th slip system.

The velocity gradient \( L \) can be uniquely decomposed into a symmetric part \( D \) (stretching rate tensor) and a skew-symmetric \( \Omega \) (spin tensor) part as

\[
L = D + \Omega
\quad (4.12)
\]

\[
D = \frac{1}{2} (L + L^T)
\quad (4.13)
\]

\[
\Omega = \frac{1}{2} (L - L^T)
\quad (4.14)
\]

\( D \) is also in general called the rate of deformation. Both tensors \( D \) and \( \Omega \) can also be decomposed into the elastic stretching and lattice rotation component (\( D^* \) and \( \Omega^* \)) and the plastic component (\( D^p \) and \( \Omega^p \)) as follows,

\[
D = D^* + D^p
\quad (4.15)
\]

\[
\Omega = \Omega^* + \Omega^p
\quad (4.16)
\]
and then $D^p$ in Eq. (4.15) can be expressed as

$$D^p = \frac{1}{2} \left( L^p + L^{pT} \right) = \sum_{\alpha=1}^{N} p^{(\alpha)} \dot{\gamma}^{(\alpha)}$$  \hspace{1cm} (4.17)

where $p^{(\alpha)}$ can be defined as

$$p^{(\alpha)} = \frac{1}{2} \left( s^{(\alpha)} \otimes m^{(\alpha)} + m^{(\alpha)} \otimes s^{(\alpha)} \right)$$  \hspace{1cm} (4.18)

Similar to $D^p$, the tensor $\Omega^p$ in Eq. (4.16) can also be expressed as

$$\Omega^p = \sum_{\alpha=1}^{N} w^{(\alpha)} \dot{\gamma}^{(\alpha)}$$  \hspace{1cm} (4.19)

where $q^{(\alpha)}$ can be defined as

$$w^{(\alpha)} = \frac{1}{2} \left( s^{(\alpha)} \otimes m^{(\alpha)} - m^{(\alpha)} \otimes s^{(\alpha)} \right)$$  \hspace{1cm} (4.20)

$s^{(\alpha)} \otimes m^{(\alpha)}$ is Schmid factor. $p^{(\alpha)}$ and $w^{(\alpha)}$ are symmetric and asymmetric part of the Schmid factor, respectively. In the crystalline coordinate system, for an FCC crystal, $s^{(\alpha)}$ and $m^{(\alpha)}$ are given by

$$s^{(1-3)} = \frac{1}{\sqrt{3}} (1,1,1), \; s^{(4-6)} = \frac{1}{\sqrt{3}} (-1,1,1), \; s^{(7-9)} = \frac{1}{\sqrt{3}} (1,-1,1), \; s^{(10-12)} = \frac{1}{\sqrt{3}} (1,1,-1)$$  \hspace{1cm} (4.21)

$$m^{(1)} = \frac{1}{\sqrt{2}} (0,-1,1), \; m^{(2)} = \frac{1}{\sqrt{2}} (1,0,-1), \; m^{(3)} = \frac{1}{\sqrt{2}} (-1,1,0), \; m^{(4)} = \frac{1}{\sqrt{2}} (1,0,1),$$

$$m^{(5)} = \frac{1}{\sqrt{2}} (1,1,0), \; m^{(6)} = \frac{1}{\sqrt{2}} (0,-1,1), \; m^{(7)} = \frac{1}{\sqrt{2}} (0,1,1), \; m^{(8)} = \frac{1}{\sqrt{2}} (1,1,0),$$

$$m^{(9)} = \frac{1}{\sqrt{2}} (1,0,-1), \; m^{(10)} = \frac{1}{\sqrt{2}} (0,1,1), \; m^{(11)} = \frac{1}{\sqrt{2}} (1,0,1), \; m^{(12)} = \frac{1}{\sqrt{2}} (-1,1,0)$$  \hspace{1cm} (4.22)

The derivatives of Eq. (4.5) and (4.6) yield

$$\dot{s}^{(\alpha)} = L^* s^{(\alpha)}$$  \hspace{1cm} (4.23)

$$\dot{m}^{(\alpha)} = -m^{(\alpha)} L^*$$  \hspace{1cm} (4.24)

By differentiating Eq. (4.3), the rate of change of Green’s Lagrangian strain which refers to the reference configuration can be written as

$$\dot{E} = \frac{1}{2} \left( F^T F + F^T F \right) = \frac{1}{2} F^T \left[ (F^T F)^T + (F^T F) \right] F = F^T D F$$  \hspace{1cm} (4.25)

The polar decomposition theory can be used to decompose the deformation gradient $F$ in Eq. (4.1) as

$$F = RU$$  \hspace{1cm} (4.26)
where $R$ is the orthogonal rotation tensor and $U$ is the right stretch tensor which is also a positively defined symmetric tensor. And they satisfy the condition of

\[
R^T = R^{-1} \quad (4.27)
\]
\[
U = U^T \quad (4.28)
\]

Substituting Eq. (4.26) into Eq. (4.8) yields

\[
L = \hat{\Phi}^{-1} = \hat{R}R^{-1} + R\hat{U}U^{-1}R^{-1} \quad (4.29)
\]

Namely,

\[
L' = \hat{R}R^{-1} \quad (4.30)
\]
\[
L^p = R\hat{U}U^{-1}R^{-1} \quad (4.31)
\]

$\dot{R}$ is the time derivative of the orthogonal rotation tensor.

Substituting Eq. (4.16), (4.30) and (4.31) into Eq. (4.14), the asymmetric part $\Omega$ of the velocity gradient can be written as

\[
\Omega = \frac{1}{2}(\hat{R}R^{-1} - R\hat{R}^{-1}) + \frac{1}{2}R(\hat{U}U^{-1} - U^{-1}\hat{U})R^T \quad (4.32)
\]

The derivative of $R^{-1} = I$ yields

\[
\dot{R}R^{-1} = -R\dot{R}^{-1} \quad (4.33)
\]

Supposing $U$ in the reference configuration is a unit tensor, namely

\[
U = \begin{bmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix} \quad (4.34)
\]

then it is easy to obtain

\[
\dot{U}U^{-1} = \dot{U}U^{-1} \quad (4.35)
\]

Therefore, $\Omega$ in Eqs. (4.14) and (4.16) can finally be expressed as

\[
\Omega = \dot{R}R^{-1} \quad (4.36)
\]

and then $R$ can be expressed by

\[
R = I - \left( I + \frac{1}{2}\Omega \Delta t \right) \left( I - \frac{1}{2}\Omega \Delta t \right)^{-1} \quad (4.37)
\]
where, $\Delta t$ is the time increment. Accordingly, Eq. (4.32) can also be expressed as

$$\Omega = \frac{2}{\Delta t} (R - I)(R - I)^{-1} \tag{4.38}$$

### 4.1.2 Constitutive law

Assuming that $t_0$ is the Kirchhoff stress in the reference configuration at the time $t+\Delta t$, it is also the Kirchhoff stress in the current configuration at the time $t$. According to the description in Section 4.1.1 and Section 4.1.2, deformation occurs first by crystallographic slip from the reference configuration to the intermediate configuration, and then the lattice stretching and rotation from the intermediate configuration to the current configuration. It is assumed that the change in stress caused by the slip and lattice stretching is $t_0\Delta t$, where $\dot{t}_0$ is the stress rate in the reference configuration. The stress $(t_0 + \dot{t}_0\Delta t)$ will be rotated to the current configuration. The rotation tensor is $R$. The Kirchhoff stress $t$ in the current configuration can be written as

$$t = R(t_0 + \dot{t}_0\Delta t)R^T \tag{4.39}$$

Taking the time derivative of Eq. (4.39) yields

$$\dot{t} = RT_0R^T + R(t_0 + \dot{t}_0\Delta t)R^T + R(t_0 + \dot{t}_0\Delta t)R^T = RT_0R^T + \Omega - \dot{t} \tag{4.40}$$

where $\dot{t}$ is the material rate of Kirchhoff stress. $RT_0R^T$ is defined as the Jaumann rate of Kirchhoff stress $\nabla t$ on axes that rotate with the material. Therefore, Eq. (4.40) can be written as

$$\nabla t = \dot{t} - \Omega t + \dot{t} \tag{4.41}$$

If deformation from the intermediate to the current configuration alone is taken into account then Eq. (4.41) can be rewritten as

$$\nabla t = t - \Omega t + \dot{t}_1 \tag{4.42}$$

where $\nabla t$ is the Jaumann rate of Kirchhoff stress on axes that rotate with the lattice and $\dot{t}_1$ is the rate of the Kirchhoff stress in the intermediate configuration.

The difference between Eqs. (4.42) and (4.41) can be obtained as

$$\nabla t - \nabla t = \sum_{\alpha=1}^{N} \beta^{(e)}(e)\gamma^{(e)}(\alpha) \tag{4.43}$$
where $\beta^{(\alpha)}$ is defined by

$$\beta^{(\alpha)} = w^{(\alpha)} t - tw^{(\alpha)}$$  \hspace{1cm} (4.44)

The lattice is elastically stretched along the lattice axis. The lattice stretching can be described in the lattice coordinate system by

$$\dot{t}_L = C_0 : D_L$$  \hspace{1cm} (4.45)

where $\dot{t}_L$ is the material rate of the Kirchhoff stress in the lattice coordinate system, $D_L$ is the rate of the elastic stretching in the lattice coordinate system, $C_0$ is the elastic moduli tensor.

Provided the rotation tensor between the lattice coordinate system and the current configuration is $R_L$, the elastic deformation rate $D^*$ in the current configuration can be linked to $D_L$ by the following equation

$$D^* = R_L D_t R_L^T$$  \hspace{1cm} (4.46)

The rate of Kirchhoff stress in the intermediate configuration can be expressed by

$$\dot{t}_L = R_J^T R_L \dot{t}_L R_L^T R$$  \hspace{1cm} (4.47)

Therefore, according to Eqs. (4.27) and (4.42), the Jaumann rate $\nabla t^*$ in Eq. (4.42) can be written as

$$\nabla t^* = R_L \dot{t}_L R_L^T$$  \hspace{1cm} (4.48)

Eqs. (4.46) and (4.48) can be rewritten as

$$D_L = (R_L^T \otimes R_L^T) : D^*$$  \hspace{1cm} (4.49)

$$\dot{t}_L = (R_L^T \otimes R_L^T) : \nabla t^*$$  \hspace{1cm} (4.50)

substituting Eqs. (4.49) and (4.50) into Eq. (4.45) yields

$$\nabla t^* = C : D^*$$  \hspace{1cm} (4.51)

$$C = (R_L \otimes R_L) \cdot C_0 \cdot (R_L^T \otimes R_L^T)$$  \hspace{1cm} (4.52)

where $C$ is the tensor of the elastic modulus defined by
For an FCC crystal, just three parameters \( c_{11}, c_{12}, c_{44} \) are needed. Substituting Eqs. (4.17) and (4.51) into Eq. (4.43), we can have

\[
\frac{\nabla}{\tau} = C : D - \sum_{\alpha=1}^{N} (C : P^{(\alpha)} + \beta^{(\alpha)}) \dot{y}^{(\alpha)}
\]  

(4.54)

It was assumed that crystallographic slip is the only plastic deformation mechanism. The resolved shear stress on each slip system can be used as the vital variable to evaluate plastic flow. The resolved shear stress \( \tau^{(\alpha)} \) can be calculated by

\[
\tau^{(\alpha)} = P^{(\alpha)} : t
\]  

(4.55)

Taking the time derivative gives

\[
\dot{\tau}^{(\alpha)} = (C : P^{(\alpha)} + \beta^{(\alpha)}) : (D - \sum_{\beta=1}^{N} P^{(\beta)} \dot{y}^{(\beta)})
\]  

(4.56)

The relationship of the Cauchy stress \( \sigma \) and the Kirchhoff stress is

\[
t = J \sigma
\]  

(4.57)

And \( J \) is defined as

\[
J = \frac{1}{|t|}
\]  

(4.58)

Therefore, the constitutive law based on Cauchy stress can be expressed as

\[
\sigma = R \left( \sigma_0 \left( 1 + \text{tr}(D) \right) \right) R^T + \frac{\nabla}{\sigma_0} \Delta t
\]  

(4.59)

\[
\frac{\nabla}{\sigma} = C : D - \text{tr}(D) \sigma - \sum_{\alpha=1}^{N} (C : P^{(\alpha)} + \Omega^{(\alpha)} \sigma - \sigma \Omega^{(\alpha)}) \dot{y}^{(\alpha)}
\]  

(4.60)

\[
\dot{\tau}^{(\alpha)} = (C : P^{(\alpha)} + W^{(\alpha)} \sigma - \sigma W^{(\alpha)}) : (D - \sum_{\beta=1}^{N} P^{(\beta)} \dot{y}^{(\beta)})
\]  

(4.61)

where \( \frac{\nabla}{\sigma} \) is the Jaumann rate of Cauchy stress on axes rotating with the material.
4.1.3 Hardening model

In this study, the strain rate-dependent hardening model with the power law is employed, which relates the resolved shear stress \( \tau^{(\alpha)} \) to the shear strain rate \( \dot{\gamma}^{(\alpha)} \) on a slip system \( \alpha \) by a power law [163] given in Eq. (4.62) and (4.63). The slip at a slip system also obeys the Schmidt law, which states that slip begins when the resolved shear stress reaches a critical value.

\[
\dot{\gamma}^{(\alpha)} = \dot{\gamma}_0^{(\alpha)} \text{sgn} \left( \frac{\tau^{(\alpha)}}{\tau_c^{(\alpha)}} \right)^n \quad \text{For} \quad |\tau^{(\alpha)}| \geq \tau_c^{(\alpha)}
\]  
\[
\dot{\gamma}^{(\alpha)} = 0 \quad \text{For} \quad |\tau^{(\alpha)}| < \tau_c^{(\alpha)}
\]

\[
\text{sgn}(x) = \begin{cases} 
-1, & x < 1 \\
1, & x \geq 1 
\end{cases}
\]

where \( \dot{\gamma}_0^{(\alpha)} \) is the reference (initial) value of the shear strain rate and it is a constant for all the slip systems. \( n \) is the strain rate sensitive exponent. As \( n \to \infty \), the plastic constitutive formulation formally becomes rate-independent. Both \( \dot{\gamma}_0^{(\alpha)} \) and \( n \) are the material parameters. \( \tau_c^{(\alpha)} \) is the flow stress which is also known as critical resolved shear stress of the slip system \( \alpha \). According to Eq. (4.62) and (4.63), the shear strain rate in each slip system should be unique and non-vanishing unless the resolved shear stress on that system is identically zero. The critical resolved shear stress \( \tau_c^{(\alpha)} \) for each slip system increases with the procedure of plastic deformation for the work hardening. Kumar and Yang [164] proposed the hardening curve needs to be determined for each possible slip system so that \( \tau_c^{(\alpha)} \) can be expressed as a function of the plastic shear in that slip system \( \tau_c^{(\alpha)} = g(\gamma^\alpha) \). Moreover, when more than one slip system is activated, hardening in each slip system is a function of slip on all the active slip systems.

Therefore, for a strain rate dependent model, a linear hardening is assumed and the rate of change of the critical resolved shear stress is expressed as [165]

\[
\dot{\tau}_c^{(\alpha)} = \sum_{\beta=1}^{N} h_{\alpha\beta} \dot{\gamma}^{(\beta)}
\]
Different hardening models have been proposed and developed since the evolution of crystal plasticity theory. However, the biggest discrepancy is the definition of hardening moduli $h_{\alpha\beta}$. It has been discussed and studied extensively by Taylor [166], Kocks and Bron [167], Nakada and Keh [168], Jackson and Basiniski [169], Havner and Shalaby [170], Hutchinson [171] and Asaro [172], Bassani and Wu [173].

Wu et al. [173] proposed the revolution of the resolved shear stress in a slip system is with respect to the shear strain as a three-stage process, as shown in Fig. 4.2. In stage I, the hardening rate is low and is almost a constant. Slips just occur on only one slip system and crystal undergoes little strain hardening. At this stage, dislocation density is also low, most dislocations escape from the crystal to the surface. Hence, this stage is called easy glide stage. In stage II, strain hardening occurs rapidly with high, constant hardening rate. It is nearly independent of temperature or strain rate. At the same time, slips occur more than one set of planes and the dislocation density is much higher than that in stage I. Meanwhile, dislocation tangles begin to develop. In stage III, the hardening rate decreases continuously until fracture occurs and it is very sensitive to temperature and strain. This stage is also called dynamic recovery stage.

![Fig. 4.2](image)

**Fig. 4.2** A typical curve of resolved shear stress versus shear strain in a slip system with three-stage hardening for an FCC single crystal. (Point “A” denotes where secondary slip commences). [174]

Before Bassani and Wu [173], it was assumed that the self-hardening modulus was either a constant or a simple function that monotonically decreased with shear strain.
Unfortunately, this assumption obviously could not capture the shear stress-shear strain curve in Fig. 4.2. However, Bassani and Wu [173] incorporated the history of slip into the development of a new hardening law. Finally, the new founded hardening modulus was able to capture the transition of stages of hardening (Fig. 4.2) and the orientation dependence of hardening. The self-hardening and latent hardening moduli are shown in Eq. (4.66). Therefore, Bassani and Wu model [173] was believed to be the best way to study the interactions between the slip systems in FCC crystals and also the best one which considers the latent hardening. In this study, we choose Bassani and Wu model [173] as our hardening model.

\[
h_{\alpha\alpha} = \left( h_0 - h_s \right) \text{sech}^2 \left( \frac{(h_0 - h_s)\gamma^{(\alpha)}}{\tau_1 - \tau_0} \right) + h_s \left[ 1 + \sum_{\beta=1}^{N} f_{\alpha\beta} \tanh \left( \frac{\gamma^{(\beta)}}{\gamma_0} \right) \right]
\]  

(4.66a)

\[
h_{\alpha\beta} = q h_{\alpha\alpha}, \alpha \neq \beta
\]  

(4.66b)

where \( h_{\alpha\beta} \) is the hardening modulus including the self-hardening of each system (\( \alpha=\beta \)) and latent hardening (\( \alpha\neq\beta \)); \( q \) is the latent hardening parameter; \( \gamma_0 \) is the reference value of slip; \( \gamma \) is the shear strain; \( \tau_0 \) the initial critical resolved shear stress; \( \tau_s \) (or \( \tau_1 \)) is the breakthrough stress where large plastic flow initiates or we can call it saturation stress; \( h_0 \) is the hardening modulus just after initial yield; \( h_s \) is the hardening modulus during easy glide and \( f_{\alpha\beta} \) represents the magnitude of the strength of a particular slip interaction between two slip systems \( \alpha \) and \( \beta \). The factors \( f_{\alpha\beta} \) depend on the geometric relation between two slip systems, which denote the hardening matrix of the dislocation interaction defined by Bassani and Wu [173]. There are five constants for \( f_{\alpha\beta} \), namely \( a_1 \) (no junction), \( a_2 \) (Hirth lock), \( a_3 \) (coplanar junction), \( a_4 \) (glissile junction) and \( a_5 \) (sessile junction) as shown in Table 4.1.
Table 4.1 Hardening matrix of dislocation interaction.\[123, 173, 174]\n
<table>
<thead>
<tr>
<th></th>
<th>1</th>
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<th>4</th>
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<td>G</td>
<td>G</td>
<td>H</td>
<td>C</td>
<td>C</td>
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</table>

The factors $f_{\alpha\beta}$ are given in terms of five constants [173]:

(i) $a_1$ (no junction): the resultant Burgers vector from slip systems $\alpha$ and $\beta$ is parallel to the original one;

(ii) $a_2$ (Hirth lock): the resultant Burgers vector is not energetically admissible;

(iii) $a_3$ (coplanar junction): the resultant Burgers vector is on the same slip plane as the original ones;

(iv) $a_4$ (glissile junction): the resultant Burgers vector is energetically admissible and on one of the two slip planes;

(v) $a_5$ (sessile junction): the resultant Burgers vector is energetically admissible but not on either of the two slip planes;

The most important feature of the Bassani and Wu model [173] is that it considers the interaction between different slip systems on the hardening because of the slips of all systems.
4.2 Development of crystal plasticity FEM model

4.2.1 Fundamental equations of FEM

It is clear that the equilibrium for problems neglecting the body forces can be expressed by the virtual work principle in rate form

\[ \int_V \sigma : \delta \mathbf{D} \, dV = \int_S \mathbf{f} : \delta \mathbf{v} \, dS \]  

(4.67)

where \( V \) is the volume of the solid body in the current configuration; \( S \) is the bounding surface of the volume \( V \); \( \sigma \) is the Cauchy stress; \( \mathbf{f} \) is the surface traction per unit of the current area; \( \delta \mathbf{v} \) is the kinematically admissible virtual velocity field and \( \delta \mathbf{D} \) is the virtual form of the rate of deformation.

According to the Finite Element Method theory, the solid body is divided into \( n \) elements, where each element is associated with \( m \) nodal points. The velocity field in each element is interpolated by interpolation functions \( N \) (shape functions), which link the velocity field \( \mathbf{v} \) to the nodal velocities \( \mathbf{v}^n \) as follows,

\[ \mathbf{v} = N \mathbf{v}^n \]  

(4.68)

The deformation rate \( \mathbf{D} \) and spin tensor \( \Omega \) can be expressed as

\[ \mathbf{D} = \mathbf{B} \mathbf{v}^n \]  

(4.69)

\[ \Omega = \mathbf{G} \mathbf{v}^n \]  

(4.70)

\( \mathbf{B} \) and \( \mathbf{G} \) are the symmetric part and the skew part of the coefficient matrix of velocity gradient, respectively.

Therefore, the equilibrium equation (Eq. (4.67)) can be rewritten as

\[ \int_V \mathbf{B}^T \sigma \, dV : \delta \mathbf{v}^n = \int_S \mathbf{N}^T \mathbf{f} \, dS : \delta \mathbf{v}^n \]  

(4.71)

Since \( \delta \mathbf{v}^n \) is arbitrary, it also can be rewritten as

\[ \int_V \mathbf{B}^T \sigma \, dV = \int_S \mathbf{N}^T \mathbf{f} \, dS \]  

(4.72)

The non-linear Eqs. (4.71) and (4.72) are functions of the nodal velocities \( \mathbf{v}^n \). They form the basis of the displacement-based finite element method so that Eq. (4.72) can be rewritten as
The Newton algorithm is generally used to solve non-linear equations (Eq. (4.73)). An iteration process is performed. In the (i+1)th iteration step the nodal velocities are updated by

\[ \mathbf{v}_{i+1}^n = \mathbf{v}_i^n - K^{-1} \mathbf{F}(\mathbf{v}_i^n) \]  
\[ K = F'(\mathbf{v}_i^n) \]  

where \( \mathbf{v}_i^n \) and \( \mathbf{v}_{i+1}^n \) are the nodal velocities at iteration steps ith and (i+1)th, respectively; \( K \) is the Jacobian matrix, which is the derivative of \( \mathbf{F}(\mathbf{v}_i^n) \) with respect to \( \mathbf{v}_i^n \) at \( \mathbf{v}_i^n = \mathbf{v}_i^n \).

To solve Eq. (4.73) \( K \) must be developed. It can be expressed by

\[ K = \int_V \mathbf{B}^T \frac{d\sigma}{dv_n} d\mathbf{V} - \int_S \mathbf{N}^T T d\mathbf{S} \]  

Accordingly, the constitutive relation for the rate-dependent materials can be described in incremental form as follows

\[ \frac{\mathbf{\nabla}}{\sigma} = \mathbf{H} : \mathbf{D} \]  

\( \mathbf{H} \) is the fourth-order hardening parameter tensor. The derivative of the Cauchy stress with respect to \( \mathbf{v}_i^n \) can be calculated as follows

\[ \frac{d\sigma}{dv_n} = \frac{d\mathbf{\nabla}}{dv_n} + \frac{d\mathbf{\Omega}}{dv_n} \sigma - \sigma \frac{d\mathbf{\Omega}}{dv_n} \]  

Substituting Eqs. (4.69), (4.70) and (4.77) into Eq. (4.78) yields

\[ \frac{d\sigma}{dv_n} = \mathbf{H} : \mathbf{B} + \mathbf{G} \sigma - \sigma \mathbf{G} \]  

The Jacobian matrix (\( K \)) can be expressed as

\[ K = \int_V \mathbf{B}^T (\mathbf{G} \sigma - \sigma \mathbf{G} + \mathbf{H} : \mathbf{B}) d\mathbf{V} - \int_S \mathbf{N}^T T d\mathbf{S} \]  

The hardening parameter tensor \( \mathbf{H} \) needs to be calculated based on the constitutive law to determine \( K \).
4.2.2 Numerical integration of crystal plasticity theory

Based on the theory presented in the aforementioned section, the crystal plasticity constitutive model is implemented in a commercial finite element code ABAQUS/Standard using a user-defined subroutine UMAT, where the material properties can be defined and the data can be exchanged with ABAQUS [175]. The subroutine performs two functions. First of all, it updates the stresses and the solution dependent state variables to their values at the end of the increment. Second of all, it provides the material Jacobian matrix for the constitutive model [176]. The stability and rate of convergence are significantly influenced by the material constitutive model. To explain arbitrary crystal orientations, two coordinate systems are introduced in the calculation, which are sample (global) coordinate and crystalline (local) coordinate systems, respectively. The global coordinate system is set the same as the reference configuration and the local coordinate system is aligned with the crystal lattice and always rotates in the same way as the lattice.

The user should provide the main subroutine UMAT with the following seven groups of data in the ABAQUS input file [176]:

(i) Elastic moduli of materials;

(ii) Number of sets of potentially activated slip systems for specified materials;

(iii) Initial crystallographic orientations in the sample (global) coordinate system;

(iv) Shear strain rate \( \dot{\gamma} \) dependence on resolved shear stress and current strength;

(v) Self and latent hardening moduli;

(vi) Forward gradient time integration parameter (\( \theta \)) and the parameter NLGEOM which determines whether the small deformation theory or the theory of finite strain and finite rotation is used in the analysis;

(vii) Parameters for the iteration method;

Two time integration schemes are used in the UMAT. The first one solves the linear equations among the increments of stresses, strains and state variables such as shear
strain, resolved shear stresses, and current strengths in slip systems. The stresses and state variables are evaluated at the start of the time increment \( t \). Then, a Newton-Rhapson iterative method is used as the second scheme to solve the nonlinear incremental equations, in which the stresses and state variables are evaluated at the end of the time increment \( t+\Delta t \).

The main subroutine is divided into eight subroutines to achieve the calculation step by step. When the UMAT is first called by main program in ABAQUS, the step will be initialized and the initial orientation of a crystal in the sample system will be determined. Meanwhile, slip systems in the same set for a crystal will be generated in the reference state. The slip rates in all slip system then will be calculated right at the start of the increment. Subsequently, the hardening matrix and the current strength in all slip systems at the reference state will be generated.

Accordingly, all the variables and Jacobian matrix can be obtained by the related equations shown in section 4.1 by computing. The incremental stress is computed and the transformation to the sample coordinate system is performed. Finally, all the values of state variables will be updated and returned to the main program in ABAQUS. If the Jacobian matrix converges, then the displacement will be increased by an increment and computation will proceed to the next step. Otherwise, the time increment will be estimated and iterated. This procedure will be repeated and ultimately terminated until the deformation is finished.

### 4.2.3 Crystal plasticity parameters

The parameters used for the hardening model, as described in Eq. (4.62) to Eq. (4.66) are shown in Table 4.2. All of these parameters were evaluated by fitting the simulated stress-strain curve with the experimental results of single crystal aluminium under plane strain compression [177, 178].

Franciosi et al. [179] and Lu et al. [130] have reported the factor \( f_{\alpha\beta} \) for aluminium in Eq. (4.66) can be chosen as:

\[
\begin{align*}
\alpha_1 &= \alpha_2 = \alpha_3 = 1.75 \\
\alpha_4 &= 2
\end{align*}
\]
Aluminium has an FCC structure with elastic moduli $C_{11} = 112,000\text{MPa}$, $C_{12} = 66,000\text{MPa}$ and $C_{44} = 28,000\text{MPa}$. There is one set of slip systems, $(111)<110>$ which including 12 different slip systems shown as in Table 4.3. In the deformed single crystal aluminium, slips occur on the $(111)$ planes and in the $<110>$ directions. All of aforementioned parameters have been validated in the simulations of rolling, tensile and ECAP deformation [130, 150, 153]. These parameters characterize the properties of a high purity aluminium single crystal and will be used throughout the simulations in this study.

**Table 4.2** Parameters in the Bassani-Wu hardening model. [173]

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<th>$h_s$, MPa</th>
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**Table 4.3** Notation of the slip systems for the FCC materials considered in this study.

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<th>Number</th>
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<th>Slip plane</th>
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<td>(-111)</td>
<td>[101]</td>
</tr>
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<td>[101]</td>
</tr>
<tr>
<td>12</td>
<td>d3</td>
<td>(11-1)</td>
<td>[-110]</td>
</tr>
</tbody>
</table>
4.3 Summary

In this chapter, the basic crystal plasticity theory and Bassani-Wu hardening model used in this study have been extensively introduced. The procedure of implementing the crystal plasticity constitutive model into a commercial finite element code ABAQUS/Standard has also been described. All the parameters in the CPFEM model and the Bassani-Wu hardening law for the aluminium single crystal has been determined by fitting the simulated stress-strain curve with the experimental results or reported results directly.
Chapter 5 CPFEM simulations of nano-indentation of aluminium

In this chapter, three-dimensional crystal plasticity FEM simulations of the nano-indentation process of aluminium single crystal has been carried out. In order to validate the developed model, the simulated load-displacement curve and Young’s modulus has been compared with the experimental data. The effects of friction, initial orientation and indenter geometry on the indentation behaviour have been discussed in detail.

5.1 Three-dimensional CPFEM model

A three dimensional model has been developed to simulate the deformation behaviour of nano-indentation, as shown in Fig. 5.1.

Fig. 5.1 3-D Nano-indentation model setup.

The indenter used in this chapter is the Berkovich indenter with a radius of 200 nanometres, as shown in Fig. 5.2. Sakharova et al. [180] used this indenter in their simulation. However, the indenter was modelled without considering the round tip. Antunes et al. [181, 182] found that the imperfection of the Vickers indenter did influence the accuracy of the indentation tests to some extent.
Fig. 5.2 3D indenter: (a) Berkovich indenter and (b). Round tip of Berkovich indenter

The simulated sample of single crystal aluminium consisted of 17040 eight-node brick elements and 18352 nodes with reduced integration (element id: C3D8R) to ensure that the mesh was fine enough, especially the area that makes contact with the indenter. The total number of nodes and elements are about 7 times more than those used in most published papers [121, 123]. The depth of indentation in this study is also larger (1000 nm) than that in the published literatures (300nm) [121, 123]. The X, Y, and Z coordinates represent the rolling direction (RD), the transverse direction (TD), and the normal direction (ND), respectively. The Euler angle measured in the experiments can be transferred to Miller index by using Eq. (5.1). The calculated Miller index ([2 3 -16], [11 -2 1] and [-1 -6 -1] parallel to the X, Y and Z axes, respectively) was then implemented into the input file of CPFEM simulation.

\[
\begin{pmatrix}
u & r & h \\
v & s & k \\
w & t & l
\end{pmatrix}
\begin{pmatrix}
\cos \varphi_1 \cos \varphi_2 - \sin \varphi_1 \sin \varphi_2 \cos \varphi \\
- \cos \varphi_1 \sin \varphi_2 - \sin \varphi_1 \cos \varphi_2 \cos \varphi \\
\sin \varphi_1 \sin \varphi
\end{pmatrix}
\begin{pmatrix}
\sin \varphi_1 \cos \varphi_2 + \cos \varphi_1 \sin \varphi_2 \cos \varphi \\
- \sin \varphi_1 \sin \varphi_2 + \cos \varphi_1 \cos \varphi_2 \cos \varphi \\
- \cos \varphi_1 \sin \varphi
\end{pmatrix}
\begin{pmatrix}
\sin \varphi_2 \sin \varphi \\
\cos \varphi_2 \sin \varphi \\
\cos \varphi
\end{pmatrix}
\] (5.1)

The dimensions of the workpiece in the FEM model are 40 µm × 40 µm × 20 µm. The height of the simulated sample is much larger than the maximum depth of indentation of 1 µm. This satisfies the 10% rule that the sample should be at least 10 times thicker than the depth of indentation in order to avoid the influence from the boundary condition [17].
All the nodes on the bottom surface and four surrounding surfaces of the sample were constrained along three normal directions. A fixed time step increment 0.01 s is chosen and a total time step increments of 24,062 in the simulation are used, including contact, loading, and unloading.

5.2 Validation

The developed CPFEM model has been applied to simulate the experimental cases described in Chapter 3. Two typical experimental load-displacement curves were chosen from all valid results of each single crystal described in Chapter 3 to validate the CPFEM model.

![Fig. 5.3](image)

**Fig. 5.3** Comparisons between numerical and experimental load-displacement curves for single crystal aluminium: (a) (001) surface; (b) (101) surface; (c) (111) surface.

Fig. 5.3 shows the comparisons between numerical and experimental load-displacement curves for single crystal aluminium on three different initial oriented surfaces. It can be seen that the simulation results are in good agreement with the experimental data. To
the best knowledge of the authors, no satisfactory agreements between numerical and experimental load-displacement curves on single crystal aluminium on all three orientations have been reported previously, especially when the Berkovich indenter is used. Liu et al. [15] compared the experimental results with the simulated load-displacement curves on (001), (011) and (111) oriented single crystal copper using ABAQUS with a user-defined material subroutine VUMAT. However, the indenter used in the research was spherical. Wang et al. [37] implemented the constitutive model for single crystalline copper and the implicit time-integration procedure proposed by Kalidindi et al. [183] into the commercial finite element code MARC by means of the user defined material subroutine HYPELA2 to do the nano-indentation simulation and indicated the experimental and simulated load-displacement curves were generally very difficult to compare so that they can only present the comparison of the piling-up patterns.

![Fig. 5.4 Comparisons between numerical and experimental results for single crystal aluminium: (a) Hardness; (b) Young’s modulus.](image)

Fig. 5.4 shows the comparison of hardness and Young’s modulus between numerical and experimental results. There are excellent agreement between simulations and experiments.
To compare the simulated rotation of the crystallographic orientation during indentation process with the experimental observation, the misorientation of each node relative to the initial orientation is partitioned into three components representing the rotation angles around the X (RD), Y (TD) and Z (ND) axes, individually. The method was proposed by Wert et al. [184]. Contour maps of crystallite rotation angles around X axis are shown as in Fig. 5.5. The value of each marked point in Fig. 5.5 is listed in Table 5.1. It agrees well with the experimental results, which demonstrated that the CPFEM model developed in this study can predict accurate micro-textures deformed by indentation. Both numerical and experimental rotation angles were measured along X axis but were observed from opposite directions so that the experimental value is opposite from the numerical one.

Fig. 5.5 Distributions of rotation angles of the cross-section along.
Table 5.1 Lattice rotation angles correspond to the regions marked from number 1 to 8 for both simulation and experiment.

<table>
<thead>
<tr>
<th>No.</th>
<th>Simulated rotation angles</th>
<th>Reference</th>
<th>1°</th>
<th>10°</th>
<th>12.5°</th>
<th>7°</th>
<th>-5°</th>
<th>-3.5°</th>
<th>-3.5°</th>
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<tr>
<td></td>
<td></td>
<td>0°</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>No.</th>
<th>Experimental rotation angles</th>
<th>Reference</th>
<th>-1°</th>
<th>-9°</th>
<th>-12°</th>
<th>-7°</th>
<th>5°</th>
<th>4°</th>
<th>3°</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0°</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The developed CPFEM model has also been used to simulate the nano-indentation test conducted by Kuo and Huang [131] in 2010 using a Berkovich diamond tip on a single crystal of 99.9999% pure aluminium with an initial orientation [Euler angle: (265°, 102°, 190°)]. In the experiments, the indented surface was grinded with 2000 grit SiC abrasive paper first and then polished with 0.5 mm diamond paste. The electropolishing process was also conducted to avoid the undesirable effects caused by mechanical hardening on the sample during sample preparation. Nano-indentation was carried out using the MTS System Nano Indenter XP with the continuous stiffness monitoring (CSM) model and the typical load-displacement curve obtained from Berkovich indentation was shown as Fig. 5.6. The measured Young’s modulus is in the range of 67.2–69.5 GPa. The comparison between numerical result and experiment observation is shown as Fig. 5.6. The result shows that the simulated results agree well with that from the experiment.

![Fig. 5.6 Simulated and experimental load-displacement curves for aluminium.](image-url)


5.3 Influence of friction between indenter and sample

The coefficient of friction ($\mu$) between highly polished metallic surfaces and diamond lies between 0.1 and 0.15, as proposed by Tabor in 1951 [185]. Johnson et al. [186] were the first group to research the effects of friction during indentation by recourse to the theory of the slip-line field by rigid wedges. It was found that the hardness increased by around 20% comparing to the frictionless ones. However, the results were of limited applicability to conventional pyramidal indentation of strain hardening materials. Since then, researchers have paid more attention to this topic. Normal inelastic contact between spherical bodies was examined theoretically and numerically by Carlsson [187]. The effect of Coulomb friction was analysed in some detail, by regarding global variables such as the mean contact pressure and contact area, as well as any field variables. It has been concluded that the effects of friction are essentially negligible except where cracks begin and then grow, in which case the stress and strain fields can be substantially influenced by friction. Moreover, the effects of friction are relatively independent of the actual value of the coefficient of friction. In Carlsson’s research [8] it was proven that when $\mu > 0.4$ there was no essential difference in the simulation results assuming a finite value of the coefficient of friction. Mesarovic et al. [188] performed a numerical study of the normal indentation of an elastic-plastic half-space by a rigid sphere, and the friction conditions were frictionless and sticking. The results showed that friction strongly affected the strain field beneath the indenter and had a quantitative effect on the size of the contact, as a function of indent depth. However, it was also reported that the contact stiffness was almost the same for sticking and for frictionless indentation within a similarity regime. Mata et al. [185] conducted the experimental and simulated researches on the role of friction on sharp indentation. Their analyses showed that friction caused more material to pile up around the contact area, while materials that develop moderate piling-up or sinking-in are less sensitive to friction.

Although the aforementioned researchers did extensive studies on the effect of friction on materials deformation during indentation, the evolution of the microstructure of materials was never investigated. In this section, the CPFEM model is used to study the effects of friction for Kuo and Huang’s experiment [131].
The Coulomb friction model was chosen for the contact between the indenter and the sample in the simulation [147]. Four different coefficients of friction (\(\mu = 0, 0.1, 0.2, \) and \(0.4\)) were used in the simulation to investigate the effect of friction on the evolution of texture and mechanical behaviour, such as the shape of the P-h curves, the hardness, the profile of surface deformation (piling-up and sinking-in), lattice rotation, and the pole figure.

Fig. 5.7 shows the comparison between the experimental load-displacement curve [131] and the simulated ones with different coefficients of friction. It has been found that the coefficients of friction does not significantly affect the load-displacement curve, which is consistent to the observation of Liu et al. [121].

![Simulated and experimental load-displacement curves for aluminium: (a) 1000 nm and (b) 2000 nm.](image)

Young’s moduli were calculated from the curves of Fig. 5.7(a) and plotted against the coefficient of friction in Fig. 5.8. It is clear that Young’s modulus decreases with an increasing coefficient of friction. Young’s modulus varies from 69.17 GPa to 67.55 GPa when the coefficient of friction changes from 0 to 0.4. The results matched the Young’s modulus of the aluminium single crystal of 70.4 GPa in Ref. [189] and 67.2-69.5 GPa in Ref. [131].
Chapter 5 CPFEM simulations of nano-indentation of aluminium

**Fig. 5.8** Young’s modulus vs. coefficient of friction.

**Fig. 5.9** Distribution of out-of-plane displacement after unloading with COF=0.

Fig. 5.9 shows the contour of out-of-plane displacement of the top sample surface after unloading for $\mu=0$. The positive values mean the displacement along the penetration direction, while negative values indicate the piling-up. It can be seen that there are four piling-up regions. Two red lines (diagonal line and vertical line) are marked in Fig. 5.9. They will be called Line A and Line B, respectively, in the following text. The detailed analysis will be given along these two lines.
Fig. 5.10(a) shows the simulated surface profile after unloading along Line A for different coefficients of friction. Fig. 5.10(b) gives the details of one of the piling-up regions in Fig. 5.10(a). The results indicate that piling-up decreases as the coefficient of friction increases, which is consistent to Ref. [121]. Mata et al. [185] believed that the decreasing of piling-up is because of the friction which opposes slip of the elements at the indenter’s face. Fig. 5.10(a) also shows that the depth of indentation is almost constant and independent of the coefficient of friction. Liu et al. [121] believed that this is the reason why the load-displacement curve is not affected by the coefficient of friction.

![Comparison of surface profiles](image)

**Fig. 5.10** Comparisons of the piling-up and sinking-in for different coefficients of friction: (a) piling-up curve; (b) magnification from the red circle; (c) sinking-in curve and (d) magnification from the red circle.

Eq. (5.2) can be used to describe the surface profile [190]:

\[ \sqrt{\alpha} = \frac{h_r}{h_{max}} \]  

(5.2)
Where $h_r$ is the residual depth after the unloading step, and can be read directly from the loading-displacement curve. In general, if $0.875 < \sqrt{\alpha} \leq 1$ then piling up occurs but if $0 < \sqrt{\alpha} \leq 0.875$, it means sinking-in will occur. There will be no piling-up or sinking-in when $\sqrt{\alpha} = 0.875$, where the true contact area $A$ and the apparent contact area $A_c$ are equal. Fig. 5.11 shows $\sqrt{\alpha}$ versus coefficient of friction. It is obvious that $\sqrt{\alpha}$ increases with an increasing coefficient of friction in the first place, but then the value of $\sqrt{\alpha}$ almost keeps constant after the coefficient of friction reaches 0.2, which agrees with the results from the ref. [121]. They found that as the coefficient of friction reaches 0.4, any further increase in the coefficient of friction does not cause an additional reduction in the material piling-up. It is also clear the deviation of piling-up between $\mu=0.2$ and $\mu=0.4$ is quite small according to Fig. 5.10(b). $h_c/h_{\text{max}}$ was also used to determine piling-up or sinking-in, but recently this method has been proven to be not good enough to explain the surface profile [191].

![Graph](image)

**Fig. 5.11** Calculation results as obtained with Berkovich indenter of single crystal aluminium: $\sqrt{\alpha}$.

Fig. 5.10(c) shows the simulated surface profile after unloading along Line B marked in Fig. 5.9 for different coefficients of friction. The region circled by the red line in Fig. 5.10(c) has been enlarged in Fig. 5.10(d). The results from Fig. 5.10(d) show that there is a mainly sinking-in outside one corner of indent followed by a small piling-up. Interestingly, for all of the sinking-ins, they decrease as the coefficient of friction increases. Peralta [192] found the same phenomenon by using Vickers indenter. They
reported sinking-in and piling-up could be present along the same crystallographic direction (<100> direction of indent).

For further analysis of the relationship between piling-up and coefficient of friction, the equivalent strain rate and von Mises stress were captured and investigated.

![Fig. 5.12 Distribution of equivalent strain rate after unloading: (a) $\mu=0$; (b) $\mu=0.1$; (c) $\mu=0.2$ and (d) $\mu=0.4$.](image)

Fig. 5.12 shows the distribution of equivalent strain rate $\dot{\varepsilon}$ after unloading on the through-thickness cross section cut along Line B of the indented sample. The arrow on each figure of Fig. 5.12 shows the evolution of equivalent strain rate from $\mu=0$ to $\mu=0.4$. It is clear the plastic deformation zone along the facet of indent becomes smaller and smaller when the coefficient of friction increases. It indicates the deformation becomes localized when the coefficient of friction increases. This results in the decrease of the sinking-in.
Fig. 5.13 Distribution of von Mises stress after unloading: (a) μ=0; (b) μ=0.1 and (c) μ=0.4.

Fig. 5.13 shows the distribution of von Mises stress after unloading on the same cross section as in Fig. 5.12 for the coefficient of friction varying from 0 to 0.4. The maximum von Mises stress for the frictionless condition is concentrated at the contact surface between the indenter and the sample, whereas on the frictional surfaces it is located at the inner region of the sample towards the edge where the indenter touches. This means the maximum von Mises stress flows from the surface of the sample to the inner part of the sample, which indicates that a frictional indentation incurs a comparatively larger plastic deformation in the contact surface area and squeezes out a bigger piling-up or at least decreases the sinking-in.

Fig. 5.14 shows the hardness as a function of the coefficient of friction. The hardness ranges from about 263 MPa to 273 MPa. In Almasri and Voyiadjis’s experiments [59, 193], the hardness of 99.9999% pure aluminium is around 280 MPa, which is slightly higher than our simulated results. They used poly-crystal aluminium and the initial orientation is not the same as that used in the present simulation. According to our
simulation, the hardness slightly increases when the coefficient of friction increases. But, the variation of hardness is insignificant.

**Fig. 5.14** Hardness for different COFs.

**Fig. 5.15**  
(a) original pole figure before deformation; (b) $\mu=0$; (c) $\mu=0.1$; (d) $\mu=0.2$ and (e) $\mu=0.4$.  

Fig. 5.15 shows the $\{110\}$ pole figures for the region under the left side of the indenter. Fig. 5.15(a) is the original pole figure before deformation. Fig. 5.15(b)-(e) denotes the pole figures after deformation. The pole figure without friction looks more scattered while those with friction become concentrated. Aluminium has 12 slip systems which
could be seen in Table 4.3 in Chapter 4. When it is deforming, some slip systems will be activated. For an activated slip system, the normal of a slip plane tend to rotate towards the loading direction (tension) or away from the loading direction (compression), as shown in Fig. 5.16. Ultimately, this leads to an evolution of orientation.

![Diagram](image)

**Fig. 5.16** Rotation of the crystal orientation in: (a) original (b) tension and (c) compression.

![Diagram](image)

**Fig. 5.17** Lattice rotation angle for different coefficients of friction: (a) \(\mu=0\); (b) \(\mu=0.1\); (c) \(\mu=0.2\) and (d) \(\mu=0.4\).
Fig. 5.17 shows the lattice rotation angles around the X axis (RD) after unloading for different coefficients of friction. The cross section is cut out along Line B marked in Fig. 5.9. In general, the lattice rotation has three components, including the angles around the X, Y, and Z axis respectively. The way to calculate the lattice rotation angles is based on the method proposed by Wert et al. [184]. According to Fig. 5.17(a)-(d), it seems there is just a slight difference induced by the coefficient of friction. In order to get more detailed information about the effect of the coefficient of friction on the lattice rotation, the rotation angles along red line marked in Fig. 5.17(a) are shown in Fig. 5.18.

![Graphs showing lattice rotation angles](image)

**Fig. 5.18** Crystal rotation angles around the three axes as a function of friction in the indentation sample: (a) Rotation angles around RD; (b) Rotation angles around TD and (c) Rotation angles around ND.

There are two peaks in each of Figs. 5.18(a)-(c). All three rotation angle component significantly changes from one peak to another. In Figs. 5.18(a) and 5.18(b), the rotation angles around RD and TD increase when the coefficient of friction increases at the left peak, whereas Fig. 5.18(c) shows the rotation angle around ND decrease as the
coefficient of friction increases at the left peak. At the right peaks, Figs. 5.18(a) and 5.18(c) show that the rotation angles decrease as the coefficient of friction increases while they increase in Fig. 5.18(b).

![Histograms and Laplace distribution of rotation angles around different axes: (a) RD, (b) TD and (c) ND.](image)

Fig. 5.19 Histograms and Laplace distribution of rotation angles around different axes: (a) RD, (b) TD and (c) ND.

Fig. 5.19 shows the histograms and Laplace distribution of rotation angles around different axes. The calculation method is based on the following equation:

$$ y = \frac{1}{2b} \exp \left( -\frac{|x - \mu|}{b} \right) $$

(5.3)

where \( b \) denotes standard deviation and \( \mu \) is the mean value. According to Fig. 5.19(a) and c, \( \mu \) is close to 0, which means that the rotation of crystal during deformation is almost symmetrical.

Fig. 5.20 shows the effect of the coefficient of friction on the standard deviation of rotation angles. The standard deviation of rotation angles around RD and ND are larger
than those around TD, but almost no effect on the standard deviation of the rotation angle around TD as the coefficient of friction increases. The standard deviation of rotation angles around ND increases when the coefficient of friction increases, while those around the RD increase first and then decrease.

![Graph showing standard deviation of rotation angles as a function of the coefficient of friction](image)

**Fig. 5.20** Standard deviation of rotation angles as a function of the coefficient of friction.

### 5.4 Influence of initial orientation

The anisotropy of the materials deformation has attracted a great interest of researchers. Nano-indentation is a good way to study this topic. Wang and Raabe [37] investigated the dependence of nano-indentation piling-up patterns and of micro-textures on the crystallographic orientation using high purity copper single crystals. It was found that the piling-up patterns on the surfaces of (0 0 1), (0 1 1) and (1 1 1) oriented single crystals have four-, two-, and six-fold symmetry, respectively and the different piling-up patterns could be explained in terms of the strong crystallographic anisotropy of the out-of-plane displacements around the indents. Flom and Komanduri [135] conducted indentation and sliding experiments on some FCC, BCC, and HCP structured single crystals (copper, aluminium, iron, zinc, and cadmium) and polycrystalline (iron, tin, aluminium oxide and magnesium meta-aluminate) materials to investigate the anisotropic behaviour of these materials in indentation and sliding. Strong anisotropic
effects were revealed during indentation and sliding with different orientations. Kiely and Houston [133] quantitatively investigated the mechanical properties for the (111), (001), and (110) surfaces of Au single crystals to analyse the effect of anisotropy. The results showed the differences in indentation behaviour on different initially orientated surface. Lim and Chaudhri [194] conducted micro-indentation hardness tests with high-purity poly-crystals and single crystals of copper and aluminium at room temperature using a Vickers and spherical indenters. However, the results showed a negligible effect of crystal orientation and indenter size effects on the measured hardness of copper and aluminium. In this section, the CPFEM model was used to investigate the influence of the initial orientation on the indentation process. In the model, the Cube, Goss and Brass orientation are set as the initial orientations, namely the (001) , (011) , and (111) slip planes parallel to the Z axis individually, and the [100] , [100] , and [110] slip directions parallel to the X axis , respectively.

Fig. 5.21 shows the simulated contour plots of the out-of-plane displacement profiles on the surfaces of the (001), (011) and (111) single crystals. It is obvious that the piling-up patterns of Al single crystal are strongly dependent on the orientation of crystal. The figure displaces the fourfold symmetry of the height profile for the (001) crystal, the twofold symmetry for the (011) crystal and the threefold symmetry for the (111) crystal. The simulated results agree well with the experimental observation reported in Chapter 3.

To further analyse the piling-up patterns, the slip traces on the different initially oriented surfaces after unloading are plotted in Fig. 5.22. Three slip systems with larger magnitudes of accumulative shear strain are chosen and displaced for each FEM element. The lines represent the lines of intersection between the slip planes and the indented surface. The black, blue, and red colours indicates the first largest magnitude, the second largest magnitude and the third largest magnitude of the accumulative shear strain, respectively. The length of the straight line represents the relative magnitude of the accumulative shear strain and the direction infers the orientation of the slip trace.
Fig. 5.21 Simulated contour plots of the out-of-planes displacement profiles on (a) (001), (b) (011) and (c) (111) oriented aluminium single crystal surfaces.
Fig. 5.22 Simulated slip traces plots of the initial oriented surface on (a) (001), (b) (011) and (c) (111) oriented aluminium single crystal surfaces.
The inset on the top right corner of Fig. 5.22(a) shows that the orientations of the slip traces are along two vertical directions. The resultant effect makes the material flow along a 45° angle between two vertical intersections. The same thing happens on the right-hand side edge of the impression. This is the reason why four-fold symmetry emerges on the (001) initially oriented surface. With the indented (011) surface, the material may flow along a certain angle between two cross slip traces, so there is a twofold symmetry on the surface. With the indented (111) surface, the inset on the top right corner of Fig. 5.22(c) shows the two activated slip systems (black and blue lines) slip along one direction. Another red line is very short compared to the black and blue lines and it could be ignored. This also happens on the right-hand side edge of the impression and the downside of the impression. The material may flow along these three directions and form a three-fold symmetry.

Fig. 5.23 shows the simulated surface profiles of the (001), (011) and (111) crystals after unloading along Line A marked in Fig. 5.21(a). The results indicate that the (001) surface has the largest piling-up and the (111) surface has the smallest piling-up. This is because all the activated slip systems on the indented (111) surface slip along in the same direction, while the activated slip systems on the indented (011) and (001) surfaces slip along different directions and the angle between different slip traces on the (001) surface are larger than that on the (011) surface. This is the reason why the (001) crystal has the biggest piling-up pattern.

Fig. 5.24 shows the simulated surface profiles of the (001), (011), and (111) crystals after unloading along Line B marked in Fig. 5.21(a). The results are different from those of Fig. 5.23. It is clear here that only the (111) crystal has the piling-up. However, sinking-in emerges on the surfaces of the (001) and (011) crystals.
Fig. 5.23 Comparisons of the surface profile along the diagonal cutting line for different oriented surfaces: (a) (001) surface; (b) (011) surface; (c) (111) surface.

The insets on the bottom right hand corner of Figs. 5.22(a)-(c) show details of how this happens. The inset on the bottom right hand corner of Fig. 5.22(a) shows the slip traces parallel with the downside edge of the impression, which means that the material flows away along this direction to both the left and right sides of the model, and leaves it with a sinking-in. The inset on the bottom right hand corner of Fig. 5.22(b) shows the same thing, but here the difference is that the material flows away along a certain degree to both the left and right hand sides of the model, which is why the sinking-in in (011) indented surface is smaller than in (001) indented surface. The inset on the bottom right hand corner of Fig. 5.22(c) shows that the slip traces are almost vertical to the bottom edge of the impress, and obviously, the piling-up is still there.
Fig. 5.24 Comparisons of the surface profile along the vertical cutting line for different oriented surfaces: (a) (001) surface; (b) (011) surface; (c) (111) surface.

Fig. 5.25 Comparisons of the \{111\} pole figures with different oriented surface on the diagonal cutting plane after unloading: (a) (001) surface; (b) (011) surface; (c) (111) surface.

Fig. 5.26 Comparisons of the \{111\} pole figures with different oriented surface on the vertical cutting plane after unloading: (a) (001) surface; (b) (011) surface; (c) (111) surface.
Fig. 5.25 and Fig. 5.26 show the \{111\} pole figures of different crystals on Line A and Line B, respectively. It can be seen that the orientation mostly rotates around TD and the direction of rotation is opposite for Line A and Line B. In general, several slip systems can be activated while the crystal is deformed. The normal of the slip plane rotates towards the loading direction (tension) or away the loading direction (compress) depending on the loading direction, leading to different evolutions of the orientation.

5.5 Influence of the indenter’s geometry

The Berkovich indenter is frequently used to measure the hardness and Young’s modulus of bulk materials and thin materials. Conical indenters with the various semi-angles are also commonly used [180, 195]. It is interesting to know if both indenters could give the similar results. Bhattacharya and Nix [98, 99] firstly used the conical indent with a 68° semi-apex angle to conduct a 2D simulation in order to avoid elastic singularity at edges of Berkovich indenter during 3D simulation, although this effect was not noticeable. Sun [196] replaced the Berkovich indenter by a conical indenter with a 70.3° semi-apex angle, based on the rule that it has the same project area as the Berkovich indenter. Bolshakov [197] also used a conical indenter with a 70.3° semi-apex angle to simulate the indentation on an aluminium alloy surface and compared the numerical results with the experimental data performed via the Berkovich indenter. It has been found that the load-displacement curves were generally the same. However, Sakharova et al. [180] carried out three-dimensional numerical simulations of Berkovich, Vickers and conical indenter hardness tests to investigate the influence of indenter geometry on indentation test results of bulk and composite film/substrate materials. They found that the differentiation between the results obtained with the three indenters was material sensitive for bulk materials. Specifically, distinct mechanical behaviours were observed for composite materials depending on the indenters’ geometry.

In this section, the CPFEM model is used to study the texture evolution of single crystal aluminium during indentation between a conical indenter and the Berkovich indenter. The indenters used in this section are Berkovich and cone (70.3° semi-apex angle) with a radius of 200 nanometres, respectively. It is shown in Fig. 5.27.
Chapter 5 CPFEM simulations of nano-indentation of aluminium

![3D Indenter Diagram](image)

**Fig. 5.27** 3D indenter: (a) and (b) Berkovich indenter; (c) and (d) conical indenter with a 70.3° semi apex angle.

![Load-Displacement Curve](image)

**Fig. 5.28** Comparison of the load-displacement curves between the Berkovich indenter and the conical indenter.

The initial orientations which are same as used by Kuo and Huang [131] have been used in the CPFEM simulations. The results are shown as in Fig. 5.28. It is apparent that the indenter geometry influences the P-h curve of single crystals. To investigate the reason,
the surface profile and micro-texture evolution was analysed to provide more detailed information.

Fig. 5.29 Simulated contour plots of the out-of-planes displacement profiles with different geometry of indenter: (a) Berkovich indenter; (b) Conical indenter.

Fig. 5.29 shows the simulated contour plots of the out-of-plane displacement profiles on the \(\overline{161}\) oriented surface with two different indenters. Fig. 5.29(b) displaces a twofold symmetry of the height profile for the initial oriented crystal. Fig. 5.29(a) looks similar as Fig. 5.29(b), but just not perfectly symmetrical. From the study, the indenter geometry does not affect the out-of-plane profile significantly. Peralta et al. [192] found sinking-in and piling-up behaviour depend on in-plane crystallographic orientations rather than the orientation of the indenter. In this study, it also indicates piling-up only depends on in-plane crystallographic orientations rather than the geometry of indenter.
Fig. 5.30 Comparisons of the surface profile along the diagonal cutting line for different indenter.

Fig. 5.30 shows the simulated surface profile on the $\overline{\text{ Observatory}}$ surface after unloading along the diagonal cutting line of the impress on the Fig. 5.29(b). The result indicates the piling-up is also almost symmetrical for the conical indenter. However, for the Berkovich indenter, the piling-up generated by one surface of the indenter is obvious higher than that generated by one edge. That means that, although the geometry of indenters does not affect the piling-up and sinking-in pattern significantly, it still can change the magnitude of the piling-up.

Fig. 5.31(a) and Fig. 5.31(b) display the distribution of equivalent strain rate $\dot{\varepsilon}$ after unloading on the cross section cutting along the vertical cutting line in Fig. 5.29(b) of the indented work piece. The maximum value of equivalent plastic strain rate is higher for the Berkovich indentation (3.220E-15 s$^{-1}$) than for conical (2.400E-15 s$^{-1}$). The maximum strain rate is located at the surface in the edge regions of indentation for the Berkovich indenter. However, for the conical indenter, the maximum strain rate is just located under the surface. Similar to the prediction by Sakharova [180], the presence of edges in the indenter geometry can influence the plastic strain under the indentation.

However, Fig. 5.31(c) and Fig. 5.31(d) shows there is almost no difference in the maximum strain rate location for two different indenters along the diagonal cutting line. This can be attributed to the fact that the direction of the diagonal cross section is the same as the direction along which the material wells up. Along these directions, the
strain rate will only depend on the in-plane crystallographic orientations rather than the geometry of the indenter. In general, the shape of the plastic deformation zone generated by Berkovich indenter is different from that generated by the conical indenter. The equivalent strain rate distribution is shallower and less ‘spherical’ for the Berkovich indenter than for the conical one, as can be seen in Fig. 5.31(a) and Fig. 5.31(b).

![Fig. 5.31 Distribution of equivalent strain rate after unloading](image)

The simulated and measured pole figures for the different locations of workpiece were selected to study texture evolution during the indentation process. The pole figures from the same position, with different indenters, were also compared with each other. Fig. 5.32(a) shows the initial orientation of the workpiece, and that any other pole figures from the deformed parts of the workpiece can be compared with it to investigate the variation. Fig. 5.32(b) shows pole figure of a piece of small central area which just contacts the Berkovich indenter tip. Fig. 5.32(e) shows the same central area which contacts a conical indenter tip. It is obvious that both pole figures do not change a lot.
comparing with Fig. 5.32(a). Fig. 5.32(c) and Fig. 5.32(f) display pole figures of the cross-section of the workpiece along the vertical cutting line for both indenters. It is clear that initial pole figure of both cross-sections rotate along X (RD), Y (TD) and Z (ND) axes, respectively. For the Berkovich indenter, it is can be seen in Fig. 5.32(c) that initial pole figure rotate along both X (RD) and Y (TD) axes clockwise and counter-clockwise, respectively. For X axis rotation, the counter-clockwise direction is slightly prominent. But, for Y axis rotation, the clockwise direction is much more prominent. Comparing with the Berkovich indenter, Fig. 5.32(f) shows rotations of initial pole figure along X (RD) and Y (TD) have the same tendency, namely clockwise and counter-clockwise respectively. Both clockwise and counter-clockwise rotations along X (RD) axis are almost the same and very small. However, counter-clockwise rotation is slightly prominent when rotations are along the Y (TD) axis. The biggest difference between Fig. 5.32(c) and Fig. 5.32(f) is the rotation along the Z (ND) axis.

For the Berkovich indenter, it is can be seen in Fig. 5.32(c) that the rotation is mainly clockwise. Fig. 5.32(f) shows there are both clockwise and counter-clockwise rotations for the conical indenter.

Fig. 5.32(d) and Fig. 5.32(g) denote pole figures of cross-section of the workpiece along diagonal cutting line for the Berkovich and conical indenter, individually. Fig. 5.32(d) shows, for the Berkovich indenter, the counter-clockwise rotation is noticeable along the X (RD) axis and clockwise rotation is noticeable along the Y (TD) axis. For the conical indenter, Fig. 5.32(g) shows the rotation along the X (RD) is clockwise and counter-clockwise respectively and the amplitudes are almost same. The rotation along the Y (TD) has the same tendency. Fig. 5.32(d) and Fig. 5.32(g) also shows the rotation along the Z (ND) axis is clockwise and counter-clockwise for both indenters.
To analyse the rotation of the crystallographic orientation during indentation process, the misorientation of each node relative to the initial orientation is partitioned into three components representing the rotation angles around the X (RD), Y (TD) and Z (ND) axes, individually. The method was proposed by Wert et al. [184]. Contour maps of crystallite rotation angles around X, Y and Z axes are shown in Fig. 5.33(a)-(c) for...
indented cross-section which is cut along vertical cutting line of workpiece. The indenter used here is Berkovich. The positive value represents the counter-clockwise rotation and the negative value represents the clockwise rotation. Fig. 5.33(d) shows the rotations angles along the red line marked in Fig. 5.33(a). Fig 5.33(a)-(c) shows the rotation angles of the area just under the tip of the indent are very small, which matches with the observation from Fig. 5.32(b). It is also can be seen from Fig. 5.33(a) that counter-clockwise rotation slightly dominates. Fig. 5.33(b) shows clockwise rotation is much more prominent. Fig. 5.33(c) shows most of rotation angles are counter-clockwise. Rotation angles from Fig. 5.33(b)-(c) also match with the results from Fig. 5.32(b).

**Fig. 5.33** Distributions of rotation angles of the cross-section along vertical cutting line in the work piece indented by Berkovich indenter: (a) contour map of rotation angle around X axis; (b) contour map of rotation angle around Y axis; (c) contour map of rotation angle around Z axis; (d) rotation angles along red line marked in (a).

Fig. 5.34(a)-(c) shows the contour maps of crystallite rotation angles around the X, Y and Z axes for indented cross-section which is along the same vertical cutting line of work piece. The indenter used here is conical indenter. It can be seen in Fig. 5.34(a) that both clockwise and counter clockwise rotations exist in each side of the indent. The
distribution of rotation angles in Fig. 5.34(a) matches well with the result in Ref. [42, 124] which also reported the clockwise and counter-clockwise rotation could emerge in each side of indent by conical indenter. This is different from the observation of indent by the Berkovich indenter in Fig. 5.33(a). Fig. 5.34(b) and Fig. 5.34(c) show there are both clockwise and counter clockwise rotations around the Y and Z axes, and the counter clockwise rotations are slightly dominant. The difference between Fig. 5.34(d) and Fig. 5.33(d) is also noticeable. Fig. 5.33(d) shows the amplitude of counter-clockwise rotation is larger than clockwise rotation along the X axis, and the tendency is opposite when it rotates along the Y axis. However, in Fig. 5.34(d) it is seen the amplitudes of both clockwise and counter-clockwise rotations along the X and Y axes are almost the same. It also shows mainly counter-clockwise rotation occurs in Fig. 5.33(d) along the Z axis, but both clockwise and counter-clockwise rotations exist in Fig. 5.34(d).

**Fig. 5.34** Distributions of rotation angles of the cross-section along vertical cutting line in the work piece indented by conical indenter: (a) contour map of rotation angle around X axis; (b) contour map of rotation angle around Y axis; (c) contour map of rotation angle around Z axis; (d) rotation angles along red line marked in (a).
Fig. 5.35(a)-(c) shows the contour maps of crystallite rotation angles around the X, Y and Z axes for the Berkovich indented cross-section cut along diagonal cutting line of work piece. There is a difference between Fig. 5.35(a) and Fig. 5.33(a) as Fig. 5.35(a) shows both clockwise and counter-clockwise rotations occur in each side of indent, although both Fig. 5.35(a) and Fig. 5.33(a) are from the Berkovich indented work piece. It seems the rotation angles around the Y axes in Fig. 5.35(b) have the same tendency as those along the vertical cutting line in Fig. 5.33(b). Fig. 5.35(c) shows both clockwise and counter-clockwise rotations exist, which is also quite different from that along vertical cutting line. Fig. 5.35(d) shows it almost has the same tendency as those in Fig. 5.33(d) except the rotation angle along the Z axis.

![Fig. 5.35](image)

**Fig. 5.35** Distributions of rotation angles of the cross-section along diagonal cutting line in the work piece indented by Berkovich indenter: (a) contour map of rotation angle around X axis; (b) contour map of rotation angle around Y axis; (c) contour map of rotation angle around Z axis; (d) rotation angles along red line marked in (a).

Fig. 5.36(a)-(c) shows the contour maps of crystallite rotation angles around the X, Y and Z axes for Cone indented cross-section cut along diagonal cutting line of the workpiece. The amplitude of rotation angle in Fig. 5.36(a)-(c) has the same tendency as
those in Fig. 5.34(a)-(c). Fig. 5.36(d) also has the same tendency as that in Fig. 5.34(d). It means the conical indenter will not affect deformation induced rotation angles a lot along different orientation. However, for the Berkovich indenter, along different orientations, the deformation induced rotation angles will be affected a lot. Fig. 5.36(a) also shows the clockwise rotations mainly stay on the surface of the indent while the counter-clockwise rotations obviously stay beneath the indent, which is different from the distribution of rotation angles in Fig. 5.34(a) deformed by the same conical indenter. It is also different from those in Fig. 5.35(a) deformed by the Berkovich indenter. This means the geometry of the indenter will affect the distribution of rotation angles induced by plastic deformation during indentation.

![Fig. 5.36 Distributions of rotation angles of the cross-section along diagonal cutting line in the work piece indented by conical indenter: (a) contour map of rotation angle around X axis; (b) contour map of rotation angle around Y axis; (c) contour map of rotation angle around Z axis; (d) rotation angles along red line marked in (a).]
Chapter 5 CPFEM simulations of nano-indentation of aluminium

5.6 Summary

In this chapter, the influence factors on the deformation behaviour and texture evolution during the indentation process have been studied systematically. The following summaries can be obtained:

1. The load-displacement curves with different coefficient of friction have been analyzed and compared with the experimental results. All the calculated values of Young’s modulus agree well with the real material and all the load-displacement curves match those observed in the experiments. According to the simulation results, the coefficient of friction does not significantly affect the load-displacement curves. Although Young’s modulus decreases as the coefficient of friction increases, all of the values are still in the narrow range. The piling-up was captured from the numerical result and it decreases as the coefficient of friction increases. Sinking-in profile was obtained as well, and it increases with the coefficient of friction. The indentation hardness with different coefficients of friction was investigated and it was found that its effect was very small.

2. Pole figures after unloading were investigated from the simulated results, and it is clear that the {110} pole figures become more concentrated as the coefficient of friction increases. Lattice rotation angles at a fixed zone were investigated. The results show that the rotation angles around the RD and ND vary significantly at the right peaks as the coefficient of friction increases. However, almost nothing affects the standard deviation of rotation angles around the TD as the coefficient of friction increases. The standard deviation of rotation angles around the ND increases when the coefficient of friction increases, while those around the RD increase first and then decreases.

3. Simulations of indentation on the (001), (011) and (111) orientated surfaces showed fourfold, twofold, and threefold piling-up patterns, respectively. The out-of-plane surface profiles along the diagonal and vertical cutting planes showed that the (001) initial orientated surface had the largest piling-up, and the (111) orientated surface had the smallest piling-up along the diagonal cutting plane. However, along the vertical cutting plane, only the (111) orientated surface had the piling-up. Sinking-in instead of piling-up emerged on the (001) and (011) orientated surfaces. The detailed explanations were provided by analyzing the slip
traces on different initially oriented surfaces. Pole figures of the cross sections along different cutting planes showed that the direction of rotation is opposite each other when a different cutting plane is chosen.

4. The simulated load-displacement curves on the (1̅1̅1̅), (011) and (124) orientated surfaces by the conical indenter with a 70.3° semi-apex angle have been compared with that by the Berkovich indenter and they do not match with each other. It also shows that piling-up patterns only depend on in-plane crystallographic orientations rather than the geometry of indenter. However, the simulated piling-up figures indicate that although the geometry of indenter does not affect the piling-up pattern, it can change the magnitude of the piling-up. For vertical cut cross-section, the maximum strain rate is located at the surface in the edge regions of indentation by the Berkovich indenter, but, for the conical indenter, the maximum strain rate is just located under the surface. There is almost no difference in the location of the maximum strain rate for two different indenters along the diagonal cutting line as the strain rate may only depends on in-plane crystallographic orientations rather than the geometry of indenter along the direction where piling-up emerges.

5. A detailed analysis has been successfully carried out to investigate the pole figure evolution during the indentation process. It has been found that the geometry of indenter plays a very important role in texture evolution. The pole figures do not change significantly at the small central area just contact both indenters tip. For vertical cut cross-section, the counter-clockwise direction is slightly prominent along the X axis and clockwise direction is much more prominent along the Y axis for the Berkovich indenter. In contrast, both clockwise and counter-clockwise rotations along the X axis are almost the same and counter-clockwise direction is slightly prominent along the Y axis for conical indenter. However, for the rotation direction along the Z axis, the rotation is mainly counter-clockwise for the Berkovich indenter but both clockwise and counter-clockwise for the conical indenter. For the diagonal cut cross-section, the counter-clockwise rotation is noticeable along the X axis and clockwise rotation is noticeable along the Y axis for the Berkovich indenter, but the amplitude of both clockwise and counter-clockwise rotation along individual the X and Y axes are almost the same for the conical indenter.
6. The geometry of the indenter significantly affects the distribution of rotation angles beneath the indent. The simulated rotation angles indicate the conical indenter does not affect the amplitude of deformation induced rotation angles along different orientations. But, for the Berkovich indenter, along different orientations, the deformation induced rotation angles are affected significantly.
Chapter 6 Indentation size effect

Since the 1950s [198], a great number of researchers have found that the microscopic mechanical properties of materials are significantly different to those of bulk materials at the macro-scale. For example, the measured indentation hardness of metallic materials increases sharply as the penetration depth decreases at the nano-scale. This phenomenon was named “indentation size effect (ISE)”. The earlier study of ISE was done by Mott [198] and then numerous other researches followed his study. For example, Gane and Cox [199] reported that hardness could be increased by a factor of three by decreasing the contact diameter from $10^4$ to $10^2$ nm for an Au single crystal. Stelmashenko et al. [66] and Ma and Clark [200] found that hardness increase greatly with the depths less than 1 $\mu$m. Fleck and Hutchinson [201, 202] believed classical plasticity theory cannot predict ISE as its constitutive model did not include intrinsic (internal) length scale. Therefore, they developed a strain gradient plasticity model which included material length scale which is thought of as an internal material length related to storage of geometrically necessary dislocations. They also found this internal material length is approximately 4 microns for copper. Subsequently, by considering the geometrically necessary dislocations (GND), Nix and Gao [62] developed a mechanism-based model which agrees well with experiments. This model then was further modified by Gao et al. [67, 68] and Huang et al. [70]. It was believed, the GNDs would accumulate underneath an indenter during the deformation process. Meanwhile, these dislocations could provide work hardening to the material and therefore increase the indentation hardness.

In this chapter, the CPFEM model has been developed to predict the ISE.

6.1 CPFEM model

The CPFEM model used in this chapter is the same as that mentioned in Section 5.1. The simulations were carried on the Cube oriented surface of single crystal aluminium with a Conical indenter. The indentation hardness can be calculated according the following equations.
Chapter 6 Indentation size effect

\[ H = \frac{P}{A_c} \]  

(6.1)

\[ A_c = 3\sqrt{3}h_c^2 \tan^2 65.3 = 24.56h_c^2 \]  

(6.2)

\[ h_c = h_{max} - k \frac{P_{max}}{S}, k = 0.75, S = \left( \frac{dP}{dh} \right)_{h=h_{max}} \]  

(6.3)

where \( H \) is the indentation hardness, \( A_c \) is the contact area between indenter and deformed surface, \( h_c \) is the contact depth.

The simulated results have been compared with the experimental data obtained by Voyiadjis and Peter [59] in 2009 who conducted the nano-indentation tests on the polished surface of a 99.9999% purity poly-crystal aluminium work piece. The conical indenter with a 70.3° semi apex angle has been used in the simulations. Two simulations have been performed. The maximum indentation depths in two simulations are 2000 nm and 110nm, respectively. The simulated results are shown as Fig. 6.1. The figure shows that both simulated and experimental results have the same tendency.

![Fig. 6.1](image)

**Fig. 6.1** Comparison of numerical (Conical indenter) and experimental indentation hardness-displacement curve.
6.2 Results and discussion

![Simulated load-displacement curve using conical indenter: (a) full loading/unloading curve for the maximum indentation depth (d) of 2000 nm; (b) the enlarged loading curve for the range of the indentation depth from 0 nm to 110 nm.](image1)

Fig. 6.2 Simulated load-displacement curve using conical indenter: (a) full loading/unloading curve for the maximum indentation depth (d) of 2000 nm; (b) the enlarged loading curve for the range of the indentation depth from 0 nm to 110 nm.

![Simulated load-displacement curve using conical indenter with full loading/unloading curve for the maximum indentation depth d of 110 nm.](image2)

Fig. 6.3 Simulated load-displacement curve using conical indenter with full loading/unloading curve for the maximum indentation depth d of 110 nm.

Fig. 6.2 shows the simulated load-displacement curve using the conical indenter on the Cube initial oriented surface. Fig. 6.2(a) gives the full loading/unloading curve for the maximum indentation depth d of 2000 nm, while Fig. 6.2(b) shows the enlarged loading curve for a range of the indentation depths from 0 nm to 110 nm. It can be seen that for the lower indentation depth (d<110nm), the loading curve is convex, while it is concave for the higher indentation depth (d≥110nm).
In order to analyse the indentation size effect, the loading curve in the red rectangle marked in Fig. 6.2 is curve-fitted by a power relationship. The result can be expressed by

\[ P = 0.00781xd^{0.50628} \quad (0 < d < 110\text{nm}) \] (6.4)

where \( P \) is the indentation load and \( d \) is the penetration depth. As the slope of unloading curve is very sharp (shown in Fig. 6.3), the contact depth \( h_c \) can be replaced by displacement \( d \). Substituting Eq. (6.4) into Eq. (6.1), it is easy to obtain

\[ H = \frac{P}{A_c} = \frac{3.2 \times 10^{-4}}{d^{1.49372}} \] (6.5)

It can be known from Eq. (6.5) that when the penetration depth \( d \) increases from 0 to around 110 nm the indentation hardness \( H \) decreases significantly.

The loading part corresponding to the larger indentation depth (\( d \geq 110\text{nm} \)) can be regressed by the following expression:

\[ P = 1.016 \times 10^{-5}xd^{1.921} \] (6.6)

Likewise, the hardness can be expressed by

\[ H = \frac{P}{A_c} = \frac{4.137 \times 10^{-7}}{d^{0.079}} \] (6.7)

As the exponent 0.079 is close to 0, the indentation hardness will nearly remain constant as the indentation depth changes.

![Fig. 6.4 Simulated indentation hardness-displacement curve with three characteristic points.](image)
Fig. 6.4 shows the simulated indentation hardness-displacement curve with three characteristic stages (Stages A, B and C) during the indentation process. Stages A, B and C will be used to further understand ISE. The distribution maps of Mises stress, critical resolved shear stress and strain gradient at three different stages will be analysed.

Fig. 6.5 The distribution of Mises stress and critical resolved shear stress at different stage during indentation: (a) stage A; (b) stage B; (c) stage C.

Fig. 6.5 shows the distribution map of Mises stress and critical resolved shear stress at different stages during indentation. It is clear both the maximum Mises stress and critical resolved shear stress are located right under the indenter at Stage A. However,
both the maximum Mises stress and critical resolved shear stress start to split at Stage B. Finally, the maximum Mises stress and critical resolved shear stress move to the side of the indenter.

Fig. 6.6 Stress for a selected element under the indenter tip.

Fig. 6.6 shows the stress for a selected element under the indenter tip. It is clear the stresses increase quickly at the beginning of indentation and then remain constant. Fig. 6.7 shows shear strain rates of 12 slip systems for the same selected element below the indenter tip. 4 slip systems were activated at the beginning of the indentation.

According to the hardening model, $\dot{\gamma}^{(\alpha)} = \dot{\gamma}_0^{(\alpha)} \text{sgn}(\tau^{(\alpha)}) \frac{\tau^{(\alpha)}}{\dot{\gamma}_0^{(\alpha)}},$ when the resolved shear stress on the primary slip system is much larger than the critical shear stress, the shear strain rate has a relatively larger value. This larger shear stress also results in larger resolved shear stress on other slip system, which finally activates another slip system at same time. Once another slip system is activated, the higher shear stress occurs on the secondary slip system because of the result of very high initial hardening rate when these secondary systems are activated. Or in other words, the flow stress on the secondary slip system becomes higher than the one of the primary slip system because
of the high hardening rate. The larger flow stress $\sigma$ correlates with indentation hardness in the formation of $H = C\sigma$ which will help to explain why the hardness is so big at the beginning of the indentation.

![Graph showing shear strain rates of 12 slip systems for a selected element below the indenter tip.]

**Fig. 6.7** Shear strain rates of 12 slip systems for a selected element below the indenter tip.

It is well known that GND density is meaningful for quantitatively characterizing strain gradient which is also determined by crystallographic orientation gradient. It is clear the crystallographic orientation gradient is the most important factor when investigating the strain gradient which is used to popularly explain indentation size effect. Arsenlis and Parks [203] and Sun et al. [204] studied the crystallographic orientation dependence of GND before.

The lattice rotation angle with respect to $x(x_1)$, $y(x_2)$ and $z(x_3)$ coordinates is defined as $\omega_1$, $\omega_2$ and $\omega_3$, respectively. The relationship between the global $x(x_1)$, $y(x_2)$ and $z(x_3)$ coordinates and the local $x'(x_1')$, $y'(x_2')$ and $z'(x_3')$ coordinates are shown in Fig. 6.8.
Fig. 6.8 Schematic drawing shows crystal lattice curvature, global and local coordinate systems. [205]

The crystal lattice curvature tensor, \( k_{ij} \), defined by Nye [61] is

\[
k = \begin{pmatrix}
    k_{11} & k_{12} & k_{13} \\
    k_{21} & k_{22} & k_{23} \\
    k_{31} & k_{32} & k_{33}
\end{pmatrix}
\] (6.8)

The component can be expressed by [45, 206, 207]

\[
k_{ij} = \frac{\partial \omega_i}{\partial x_j} \approx \frac{\Delta \omega}{\Delta x}
\] (6.9)

where, \( \frac{\partial \omega}{\partial x} \) is the strain gradient, and \( \Delta x \) is the transition distance along \( x \) axis. \( \Delta \omega \) represents the local lattice rotation with respect to the undeformed lattice, which is shown in Fig. 6.9 as follows
Due to plane strain conditions with y-axis perpendicular to the (010) plane in our 3D indentation model, it is clear $\omega_1 = 0$, $\omega_3 = 0$, and $\omega_2 = \omega_y$. $\omega_y$ represents the in-plane rotation angle which can be calculated based on the method reported by Wert et al. [184]. For the plane strain deformation state, only two of the nine components of $k_{ij}$ are not equal to zero and they are

$$k_{21} = \frac{\partial \omega_2}{\partial x_1} = \frac{\partial \omega_2}{\partial x} \quad (6.10)$$

$$k_{23} = \frac{\partial \omega_2}{\partial x_3} = \frac{\partial \omega_2}{\partial z} \quad (6.11)$$

According to Eqs. (6.10) and (6.11), the value of lattice curvature $k_{21}$ and $k_{23}$ can be determined by numerical differentiation of the crystal lattice rotation angles in the global coordinate system. In this simulation, $k_{21}$ is the domain lattice curvature as the indentation direction parallels with the $z(x_3)$ axis.

The information of global coordinate systems of each node in our model was extracted before and after deformation to calculate the lattice rotation angles. Then, the values of lattice curvature $k_{21}$ form Stage A to Stage C during indentation were converted to contour maps which are shown as follows.
Fig. 6.10 The distribution of lattice curvature $k_{21}$ at different stage during indentation: (a) stage A; (b) stage B; (c) stage C.

It is obvious the main lattice curvature $k_{21}$ decreases with the increasing of the indentation depth from Stage A to Stage C, and the difference between Stage A and Stage B is much significant than that between Stage B and Stage C.
6.3 Summary

This chapter can be summarized as follows:

1. The load-displacement curve has been divided into two separate parts and fitted by two power relationships. It has been found that hardness significantly decreases with the indentation depth, while it remains nearly constant after the indentation depth exceeds a certain value.

2. The distribution of Mises stress and critical resolved shear stress has been investigated during three different stages of the indentation process. It has been found both the maximum Mises stress and critical resolved shear stress are located right under the indenter at Stage A, and then they start to split at Stage B. Eventually, the maximum Mises stress and critical resolved shear stress move to the side of the indenter.

3. The lattice curvature has been studied and simulated during three different stages of indentation process. It has been found the main lattice curvature $k_{21}$ decreases with the indentation depth from Stage A to Stage C, and the difference between Stage A and Stage B is much significant than that between Stage B and Stage C.
Chapter 7 Relationship between hardness and yield stress

The objective of this chapter is to investigate the relationship between indentation hardness and tensile yield stress for pure aluminium using the CPFEM simulations.

7.1 Simulation of tensile test of polycrystalline aluminium

The simulated tensile test sample was a round bar with the length of 4 mm and the diameter of 0.5 mm, as shown in Fig. 7.1. The total number of elements is 5550. During the simulation the displacement along the X axis at the cross section of X=0 is constrained and a constant speed of 0.001125 mm/s along the X axis has been applied to the cross section of X=L, where L is the length of the sample.

![Fig. 7.1 The 3D CPFEM tensile test model.](image)

The Voronoi diagram is the most popular method to generate polycrystalline material structure [209] in the present. In this study, the 3D Voronoi diagram has been used to generate a number of three-dimensional cells. Each Voronoi cell corresponds to one seed and the number of seeds will be controlled to determine the average size of the cells. The generated Voronoi cells are then assigned with different crystallographic orientations and implemented into the CPFEM model. In the CPFEM simulation each Voronoi cell represents a virtual grain. The detailed description for constructing polycrystal CPFEM model has been given in Ref. [159]. In this study 135 grains were generated and the grain size was approximately 200 µm as shown in Fig. 7.2. Different colours indicate different crystallographic orientations in Fig. 7.2. The grain size used in
the present simulation is close to that measured from the annealed commercially pure aluminium EN AW1050 in Ref. [210].

![3D CPFEM poly-crystal tensile test model](image)

**Fig. 7.2** The 3D CPFEM poly-crystal tensile test model.

In order to validate the poly-crystal simulation model developed in the present study, an aluminium tensile experiment performed by Matteis et al. [211] has been simulated. The material used in their tensile test was commercially pure aluminium alloy EN AW1050 subjected to four annealing cycles. The measured stress-strain curve is shown in Fig. 7.3.

### 7.2 Results and discussion

![Comparison between experiment and simulation of tensile test of pure aluminium](image)

**Fig. 7.3** Comparison between experiment and simulation of tensile test of pure aluminium. [211]
Fig. 7.3 shows comparison between experiment and simulation of tensile test of pure aluminium. It is clear the numerical result of the CPFEM model agrees well with that from the experiment.

According to Tabor’s research [1], the relationship between indentation hardness and yield stress of metal material can be expressed as the following equation

\[ H = C\sigma \]  \hspace{1cm} (7.1)

where \( \sigma \) is the uniaxial yield stress and \( H \) is the indentation hardness. The factor \( C \) is termed as elastic constraint factor and has a value of approximately 3 for metals with a strain hardening exponent \( n \) equalling zero [212]. The yield stress value in Eq. 7.1 corresponds to the plastic strain that is unique to the hardness test performed, or more specifically, to the geometry of the indenter tip. In the case of diamond pyramid hardness (DPH) via Vickers, the flow stress corresponds to a plastic strain of 0.08 which is defined as the representative plastic strain [213]. Jayaraman et al. [214] determined a representative plastic strain of 0.07 and 0.225 for Berkovich and Cube-corner indenters, respectively.

However, Marcinkowski et al. [215] reported annealed Fe-Cr alloys exhibiting some strain hardening had \( H = 5\sigma \). Speich and Warlimont [216] found \( H = 4\sigma \) for some low carbon martensites and Fe-Ni alloys.

In the present study, the indentation hardness of aluminium single crystals with different initial orientations was measured between 350 MPa and 400 MPa as shown in Table 7.1. The average hardness is 371.5 MPa

It can be obtained from Fig. 7.3 that the simulated yield stress is about 60Mpa at the true plastic strain of 0.07. This gives the C value of 6.19, which is much larger than 3. The strain hardening exponent at the true strain of 0.07 is 0.284. This means the strain hardening significantly affects the factor \( C \).
Table 7.1 Indentation hardness of aluminium single crystals with different initial orientations.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Hardness (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(001)</td>
<td>362.4</td>
</tr>
<tr>
<td>(101)</td>
<td>354.3</td>
</tr>
<tr>
<td>(111)</td>
<td>397.9</td>
</tr>
</tbody>
</table>

Fig. 7.4 shows the distribution of Mises stress at the true strain of 0.25 on the outside surface of the model. It is clear that the higher Mises stresses are located around the grain boundaries, which can be seen as marked by A, B, C and D in Fig. 7.4. Fig. 7.5 shows the distribution of Mises stress on the cross-sectional surface of the model and the higher stress has also been concentrated around the grain boundaries.

Fig. 7.4 The distribution of Mises stress on the outside surface of 3D poly-crystal model.

Fig. 7.5 The distribution of Mises stress on cross-sectional surface of 3D poly-crystal model.

The shear strain rates of both indentation and tensile test simulations are investigated to analyse the activation behaviour of slip systems during two different deformation
processes. The notation of slip systems for FCC metals was shown in Table 4.3 of Chapter 4. Fig. 7.6 shows the shear strain rates of nano-indentation simulation with three different initial orientations. 12 slip systems are activated for both the (001) and (101) oriented single crystal, while 11 slip systems are activated for the (111) oriented single crystal and just 8 slip system is inactivated. Fig. 7.7 shows the shear strain rates of three grains with three different initial orientations during the tensile deformation. 4 slip systems are inactivated for the (001) initially oriented grain and they are a₁, b₃, c₁ and d₃ respectively. a₂, b₁, b₂, b₃, c₃ and d₁ remain inactivated during the tensile deformation for the (101) initially oriented grain. For the (111) initially oriented grain, 6 slip systems remain inactivated and they are a₁, a₂, a₂, b₃, c₃ and d₃ respectively.

It is clear that more slip systems are activated during the nano-indentation process than the tensile test. The interaction of activated slip systems would result in the strain hardening. This is the reason why the C factor is much larger than 3 for pure aluminium.

Fig. 7.6 Shear strain rates of nano-indentation simulation with three different initial orientations: (a) (001) surface; (b) (101) surface; (c) (111) surface.
Chapter 7 Relationship between hardness and yield stress

Fig. 7.7 Shear strain rates of tensile test simulation with three different initial orientations: (a) (001) grain; (b) (101) grain; (c) (111) grain.

7.3 Summary

In this chapter, the relationship between the indentation hardness and the yield stress of annealed pure aluminium has been studied. The following summaries can be obtained:

1. The 3D poly-crystal tensile test CPFEM model has been developed. It is found the simulated true stress-strain curve is in good agreement with the experimental observation.
2. The correlation between the indentation hardness (H) and the tensile yield stress (σ) at the true strain of 0.07 for pure aluminium has been studied based on the simulated indentation hardness and the true stress-strain curve. It is found that $H = 6.19\sigma$. This result indicates that the strain hardening significantly affects the elastic constraint factor $C$.
3. It has been found that more slip systems are activated during the nano-indentation process than the tensile deformation. The interaction of activated slip
systems results in the strain hardening. This is the reason why the C factor is much larger than 3 for annealed pure aluminium.
Chapter 8 Estimation of work hardening parameters for pure copper

Crystal plasticity modelling of polycrystalline materials during large deformation is a quite complicated system. Its accuracy depends on the following: (a) deformation model; (b) texture model (crystallographic orientation rotation scheme); (c) work-hardening model; and (d) polycrystalline structure. The first two sub-models used in the CPFEM simulation stem from the well-recognized theory. There are several candidates for the third sub-model, such as Asaro model [217], Bassani and Wu model [218] and dislocation-based models [219]. The parameters of hardening model need to be estimated by fitting the experimental results (stress-strain curve or load-displacement curve). However, it is hard to separate the influences of polycrystalline structure and the hardening model.

Experiments of single crystals provide an opportunity to avoid the effect of polycrystalline structure. The parameters of hardening model for aluminium used in Chapters 5-7 were obtained by fitting the experimental results of compression and tension tests of aluminium single crystals. Unfortunately such information is not always available for most materials. Nano-indentation is a unique technique to achieve this goal. Due to the nature of small scale, most nano-indentation impressions are far from the grain boundary. The grain boundary has a negligible influence on such nano-indentation. Therefore, nano-indentation test results can be used to estimate the parameters of the crystal plasticity hardening model.

In this chapter, the parameters of the hardening model for pure copper will be determined by nano-indentation simulations.

8.1 CPFEM implementation

The CPFEM model used in this chapter is the same as Chapter 5. The differences are the parameters of Bassani-Wu hardening model [218].

A three dimensional model was built to describe the behaviour of nano-indentation as shown in Fig. 8.1.
The indenter is a Berkovich indenter with a 200 nm radius round tip as shown in Fig. 8.1(d). 13024 eight-node brick elements and 14463 nodes with reduced integration (element id: C3D8R) are used in the CPFEM model. A refined mesh was generated in the contact area (Fig. 8.1(b)) directly underneath the indenter in order to obtain the most accurate contact solution while a coarser mesh was created in the rest region to reduce the total number of the elements in the model and to keep the computer time within limits. The importance of having an appropriate mesh density in the contact area has been proposed in Ref. [121, 123]. The size of the smallest element was about 100 nm in all three directions. The X, Y and Z coordinates represent the rolling direction (RD), transverse direction (TD) and normal direction (ND), respectively. In this model, the specific Goss orientation from a Ref. [31] is set as the initial orientation, namely the
(011) slip planes parallel to the Z axis and the [100] slip direction parallels to the X axis, respectfully.

The dimensions of the sample simulated in the CPFEM model are 60 µm × 60 µm × 30 µm. The height of the sample is much larger than the maximum indentation depth (1µm), which satisfies the condition that the thickness of sample should be at least 10 times more than indentation depth in order to avoid the influence from the boundary.

The single crystal copper is conducted to investigate the micro-scale behaviour under nano-indentation. All nodes on the bottom surface and four surrounding surfaces of workpiece are constrained along three axes considering the real indentation condition (the metal sample is mounted inside a piece of resin and just the top surface is free). Liu et al.’s [121] studies showed that both the indentation depth and the load-displacement curve are not affected by the coefficient of friction. Therefore, a frictionless contact pair is defined by two contact surfaces with associated nodes between the indenter and workpiece. The time step increment is also set for the convergence of modelling. In this study, same as in Chapter 4, a fixed time step increment 0.01s is chosen and a total time step increments of 24,062 in the simulation are used, including contact, loading, and unloading. However, the calculation time is significant reduced from 360h to 120h by using symmetrical mesh.

Bassani and Wu [174], and Huang [220] have reported the factor $f_{\text{def}}$ for copper can be chosen as: $a_1 = a_2 = a_3 = 8$, $a_4 = 15$ and $a_5 = 20$, and three elastic moduli are $C_{11} = 168,400$ MPa, $C_{12} = 121,400$ MPa, $C_{44} = 75,400$ MPa. Other parameters will be optimized in the following sections.
8.2 Effect of work hardening parameters on indentation mechanical behaviours

Fig. 8.2 Effects of the rate sensitivity exponent (n) on the indentation mechanical behaviour.

Fig. 8.2(a) shows the effects of rate sensitivity exponent (n) on the load-indentation depth curve. The n value varies from 1 to 5. All the other parameters are listed in Table 8.1. The load-displacement curve change significantly when the n value decreases from 2 to 1, while it just slightly changes when the n value increases to 5. Fig. 8.2(b) and (c) show that both the simulated Young’s modulus and indentation hardness decrease with the n value. The differences in Young’s modulus and indentation hardness between n=1 and n=2 are approximately 19% and 20%, respectively. However, the differences in Young’s modulus and indentation hardness between n=2 and n=5 are just 11% and 3%.
Table 8.1 Parameters in the constitutive model.

<table>
<thead>
<tr>
<th>$\dot{\gamma}_0$, 1/s</th>
<th>$h_0$, MPa</th>
<th>$h_s$, MPa</th>
<th>$\tau_1$, MPa</th>
<th>$\tau_0$, MPa</th>
<th>$\gamma_0$</th>
<th>$q$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0001</td>
<td>90</td>
<td>1.5</td>
<td>1.3</td>
<td>1</td>
<td>0.001</td>
<td>1</td>
</tr>
</tbody>
</table>

Fig. 8.3 Effect of the reference value of slip $\gamma_0$ on the indentation mechanical behaviour.

Fig. 8.3 shows the effect of the reference value of slip $\gamma_0$ on the indentation mechanical behaviour. $\gamma_0$ varies from 0.0001 to 0.01. It is obvious that $\gamma_0$ affects the load-displacement curve significantly. The maximum load at the indentation depth of 1000 nm increases with the decrease of $\gamma_0$. It can be seen from Figs. 8.3(b) and 8.3(c) that the Young’s modulus and indentation hardness decrease with $\gamma_0$. The differences in Young’s modulus and indentation hardness between $\gamma_0 = 0.0001$ and $\gamma_0 = 0.01$ are approximately 46% and 112%.
Fig. 8.4 Effect of the reference strain rate $\dot{\varepsilon}$ on the indentation mechanical behaviour.

Fig. 8.4 shows the effect of the reference strain rate $\dot{\varepsilon}$ on the indentation mechanical behaviour. $\dot{\varepsilon}$ changes from 0.0001 to 0.01. The load-displacement curve quickly moves down when $\dot{\varepsilon}$ increases from 0.0001 to 0.001, but it has just a slight change while it increases from 0.001 to 0.01. The Young’s modulus and indentation hardness decreases by 45% and 46% when $\dot{\varepsilon}$ increases from 0.0001 and 0.001. However, the differences in Young’s modulus and indentation hardness between $\dot{\varepsilon} = 0.001$ and $\dot{\varepsilon} = 0.01$ are just 21% and 4%.
Fig. 8.5 Effect of the initial hardening modulus $h_0$ on the indentation mechanical behaviour.

Fig. 8.5 depicts the effect of the initial hardening modulus $h_0$ on the indentation mechanical behaviour for the range of $h_0=80$~100MPa. It can be seen that the effect of $h_0$ is not significant. It is hard to see the difference in the load-displacement curve in Fig. 8.5(a). Although both Young’s modulus and indentation hardness increase with $h_0$, the change is quite small. The differences in Young’s modulus and indentation hardness for the range of $h_0=80$~100MPa are less than 1%. 
Fig. 8.6 Effect of the ratio of latent over self-hardening moduli \( q \) on the indentation mechanical behaviour.

Fig. 8.6 shows the effect of the ratio of latent over self-hardening moduli (\( q \)) on the indentation mechanical behaviour. \( q \) varies from 0 to 1.4. The load-indentation depth curve significantly moves up when \( q \) increases from 0 to 1. The effect of \( q \) is relatively small when \( q \) changes from 1 to 1.4. The differences in Young’s modulus and indentation hardness between \( q=0 \) and \( q=1 \) are about 29% and 54%, respectively, while they are 3% and 10%, respectively, when \( q \) changes from 1 to 1.4.
Chapter 8 Estimation of work hardening parameters for pure copper

Fig. 8.7 The effect of the initial yield stress $\tau_0$ on the indentation mechanical behaviour.

Fig. 8.7 shows the effect of the initial yield stress $\tau_0$ on the indentation mechanical behaviour for the range of $\tau_0=0.9$~1.1. It can be seen that the load-indentation depth curve moves down as $\tau_0$ increases. The Young’s modulus and indentation hardness decreases by 13% and 36%, respectively, when $\tau_0$ varies from 0.9 to 1.1.
Fig. 8.8 Effect of the stage I stress $\tau_i$ on the indentation mechanical behaviour.

Fig. 8.8 shows the effect of the stage I stress $\tau_i$ on the indentation mechanical behaviour for the range of $\tau_i=1.2-1.4$. The load monotonically increases with $\tau_i$ at the same indentation depth. The Young’s modulus and indentation hardness increases when $\tau_i$ increases. They change by 14% and 36%, respectively, when $\tau_i$ increases from 1.2 to 1.4.

**8.3 Determination of the work hardening parameters**

To validate our numerical results and hardening parameters, the load-displacement curve and indentation hardness were compared with the experimental results. Wei et al. [221] performed the nano-indentation test on the (110) initial oriented surface of the single crystal Cu to study the load-displacement curve. The surface was ground and polished with number 600-1200 sand sheets smoothly and then was polished via diamond paste with 0.1-nm average particle size. Chemical methods were used also to remove the remainders on the surface and eliminate residual stress within the surface layer. Finally, the nano-indentation tests were conducted on the test instrument MTS-
Nanoindenter II using the Berkovich indenter. Meanwhile, McElhaney et al. [59, 92] conducted the nano-indentation test on the surface of the strain-hardened polycrystal Cu with the Berkovich indenter. The strain-hardened sample was polycrystalline with a grain size that was large compared to the size of the large indentations, so of the indentations were made in single grains. As the initial orientation of the indented surface almost does not affect the result of the hardness according to the results from Chapter 6, it is possible to compare the experimental hardness-displacement curve with our simulated one.

Fig. 8.9 Comparisons between numerical and experimental load-displacement curves for single crystal copper.

Simulations with different sets of parameters have been performed. The total number of simulations is 16. By comparing with the experimental results, the parameters listed in Table 8.2 have been found to be the best set of work hardening parameters. Comparisons between the experimental results and simulation results by the optimum parameters are shown in Fig. 8.2. It can be seen that there is a very good agreement between them.

Table 8.2 Parameters in the constitutive model.

<table>
<thead>
<tr>
<th>n</th>
<th>$\dot{\gamma}_0$, 1/s</th>
<th>$h_0$, MPa</th>
<th>$h_s$, MPa</th>
<th>$\tau_1$, MPa</th>
<th>$\tau_0$, MPa</th>
<th>$\gamma_0$</th>
<th>q</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.0001</td>
<td>90</td>
<td>1.5</td>
<td>1.3</td>
<td>1</td>
<td>0.001</td>
<td>1</td>
</tr>
</tbody>
</table>
8.4 Summary

In this chapter, the effects of Bassani-Wu hardening model parameters on nanoindentation deformation behaviour of copper single crystals have been studied in detail. Among all these parameters, the changes of the reference value of slip $\gamma_0$ and the reference strain rate $\dot{\varepsilon}$ affect the indentation mechanical behaviour most significantly, but the changes of the initial hardening modulus $h_0$ affect the indentation mechanical behaviour most insignificantly. The optimum work hardening parameters for pure copper have been estimated via comparing the simulated load-displacement curve and with the experimental one.
Chapter 9  Conclusions and future work

This study aims to numerically and experimentally investigate mechanical behaviour and micro-texture evolution during nano-indentation deformation of aluminium and to estimate the work hardening parameters for pure copper. The general conclusions can be outlined as follows.

1) The indentation tests have been conducted by IBIS/UMIS nano-indentation system on the three different oriented samples with (001), (101) and (111) parallel to the normal of the sample surfaces respectively. The elastic moduli and indentation hardness have been calculated from the measured indentation load-displacement curves. Piling-up patterns have been observed by AFM and lattice rotation angles have been investigated by FIB and TEM.

2) A three-dimensional crystal plasticity FEM model has been developed to investigate the nano-indentation process. The simulation results of the load-displacement curve, elastic modulus, hardness, piling-up pattern, and indentation induced lattice rotation agree very well with the experimental observations.

3) It has been found through simulations that the load-displacement curves are independent of the coefficient of friction. Young’s modulus and indentation hardness are influenced by the coefficient of friction slightly and it has been demonstrated that the coefficient of friction can be neglected when considering the indentation hardness or the size effect. Crystallographic texture becomes more concentrated in pole figure as the coefficient of friction increases. The lattice rotation angles around the RD and ND has been found to be affected significantly with the coefficient of friction. The coefficient of friction does not affect the standard deviation of rotation angles around the TD although the standard deviation of rotation angles around both ND and RD has been influenced.

4) The simulations of indentation on the (001), (011) and (111) orientated surfaces show fourfold, twofold, and threefold piling-up patterns, respectively. The deformation anisotropy has been disclosed via piling-up pattern, out-of-plane surface profiles along the diagonal and vertical cutting
plane, slip traces and pole figures of the cross sections along different cutting planes.

5) The simulated load-displacement curve by the conical indenter with 70.3° semi-apex angle has been compared with that by the Berkovich indenter. They show different results. Piling-up has been found to depend on in-plane crystallographic orientations rather than the geometry of indenter. The geometry of indenter does not affect the location of the maximum strain rate along the diagonal cutting line but it does influence the location of the maximum strain rate along the vertical cutting line. It has been found that the geometry of indenter plays a very important role in texture evolution.

6) Indentation size effect has been observed by the CPFEM simulations. It has been seen that the maximum Mises stress and critical resolved shear stress are located right under the indenter at the shallow depth. They then start to split with the increasing of the indentation depth. The maximum Mises stress and critical resolved shear stress finally move to the edges of the indenter. The main lattice curvature $k_{21}$ decreases with the indentation depth. This is the major reason responsible for the indentation size effect.

7) A three dimensional poly-crystal CPFEM model has been developed to simulate the tensile deformation of pure aluminium. The simulated true stress-strain curve is in a good agreement with the experimental observation. The correlation between the indentation hardness (H) and the tensile yield stress ($\sigma$) at the true strain of 0.07 aluminium is investigated. $H=C\sigma$ with the factor C of 6.19 has been deduced. It has been found that the strain hardening significantly affects the factor C.

8) The parameters of the hardening model have been estimated by comparing the CPFEM simulation results with experimental data for pure copper. It has been found through simulations that the reference value of slip $\gamma_0$ and the reference strain rate $\dot{\varepsilon}$ affect the indentation mechanical behaviour significantly, while the influence of the initial hardening modulus $h_0$ is insignificant.

The recommendations for future work can be outlined as follows.
1) Since indentation size effect correlates to geometrically necessary dislocation, it is better to combine CPFEM simulation together with MD simulation to investigate the dislocation evolution during indentation deformation in small scale.

2) The CPFEM model developed in the current work has been proven to be suitable to predict nano-indentation mechanical behaviour and texture evolution for single crystal aluminium and copper. However, the simulation ignores the influences from grain boundaries. It is strongly recommended to create 3D poly-crystal nano-indentation models or bi-crystal nano-indentation models to investigate the effects of grain boundaries.

3) Since CPFEM model is suitable to investigate the indentation deformation of FCC single crystal, it will be interesting to estimate the indentation behaviour of BCC and HCP single crystal.
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