

**Micromechanical Studies of Intergranular Strain and
Lattice Misorientation Fields and Comparisons to Advanced
Diffraction Measurements**

A Dissertation
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DEDICATION

This doctoral dissertation is dedicated to my parents, my husband, my parents-in-law, and all beloved ones for offering me the greatest love and the most unconditional supports.

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Abstract

Inhomogeneous deformation fields arising from the grain-grain interactions in polycrystalline materials have been evaluated using a crystal plasticity finite element method and extensively compared to neutron diffraction measurements under fatigue crack growth conditions. The roles of intergranular deformation anisotropy, grain boundary damage, and non-common deformation mechanisms (such as twinning for hexagonal close packed crystals) are systematically evaluated. The lattice misorientation field can be used to determine the intragranular deformation behavior in polycrystals or to describe the deformation inhomogeneity due to dislocation plasticity in single crystals. The study of indentation-induced lattice misorientation fields in single crystals sheds lights on the understanding of the scale-dependent plasticity mechanisms.

A two-scale micromechanical analysis is performed to study the lattice strain distributions near a fatigue crack tip. The experimental finding of vanishing residual intergranular strain in polycrystals as the increase of the fully reversed loading cycles suggests the intergranular damage be the dominant failure mechanism. Our model predictions are compared to in situ neutron diffraction measurements of Ni-based superalloys under fatigue crack growth conditions. Predicted and measured lattice strains in the vicinity of fatigue crack tips illustrate the important roles played by the intergranular damage and the surrounding plasticity in fatigue growth.

Motivated by the synchrotron x-ray measurements of lattice rotation fields in single crystals under indentation, the effect of the orientation of slip systems on the 2D wedge indentation of a model single crystal is investigated. Furthermore, the crystallographic orientations of the indented solids are gradually rotated, resulting

changes of lattice misorientation patterns under the indenter. These 2D simulations, as well as a 3D Berkovich indentation simulation, suggest a kinematic relationship between the lattice misorientation and crystalline slip fields.

Advanced structural materials such as light-weighted materials, nanocrystalline metals/alloys, and hierarchically structured alloys often encounter unconventional deformation mechanisms. The convolution of crystalline slip and deformation twin are considered in the hexagonal close packed polycrystals. Specifically, we have determined the lattice strain distributions near fatigue crack tips in Zircaloy-4, and the role of tensile-twins on intergranular strain evolution in a wrought Mg alloy, which compare favorable to available neutron diffraction measurements.

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Chapter 1

Introduction

Controlling the deformation and failure behavior of materials is an issue of fundamental importance for applications that range from the design and reliability of components for traditional automotive and aerospace industries, to the fabrication and operation of microelectronics and micro-electro-mechanical systems, to the design of mechanically superior nanostructured composites and nanocrystalline materials, and to emerging manufacturing processes such as nano-imprinting. Plasticity is inherently a multi-scale phenomenon: the behavior of individual dislocations is governed by atomic scale processes at length scales of a few nanometers; while deformation, damage initiation and microstructural evolution involve the collective behavior of large number of defects, at length scales of micrometers; the macroscopic material response is controlled by microstructural features up to 100 μm . Phenomenological plasticity constitutive laws have been successfully developed and applied mostly to macroscopic materials phenomena. The development of continuum crystal plasticity provides a critical link from microstructures to overall mechanical behavior, even though the crystal plasticity theory is largely phenomenological (Asaro, 1983). The behavior at the mesoscale – between atoms and the continuum – is governed by a complex interplay between atomic-scale interactions; the nucleation, propagation and interaction of defects; and the material microstructure (van der Giesen and Needleman, 2002). The plasticity models across these scales are usually developed with a comparison to experimental measurements such as the force-displacement curves, hardness, or other macroscopic measurements. The development of predictive

plasticity theories clearly relies on direct quantitative comparisons on microstructural levels.

Intergranular stress/strain fields arise in polycrystalline metals due to the incompatible plastic deformation in of the neighboring grains and can have significant effects on the material mechanical properties. Lattice misorientation is of tremendous interests due to its relationship to the geometrically necessary dislocation (GND) densities, which are found to be one of the keys to the mechanistic origins of the indentation size effect (ISE) (Pharr et al., 2010). Theoretically, the above microstructure-related information could be captured at the grain by modeling a polycrystal grain by grain with each grain governed by the crystal plasticity constitutive law. The comparison between the experimental characterization and computational modeling of inter- and intragranular deformation behavior will enrich our understandings towards the nature of material structure and plastic anisotropy. The mechanisms involved in the current dissertation include lattice strain prediction in low cycle fatigue and fatigue crack growth tests, interpretation of grain-orientation-dependent residual intergranular strain and intergranular damage, lattice misorientation under small-scale indentation.

Fracture and fatigue failures are naturally multiscale problems. The crack tip process zone involves cleavage, void, and grain boundary damage process, which often require atomistic mechanistic studies. On the other hand, the failure resistance is mainly contributed by the plastic dissipation in the vicinity of the crack tip. The plastic zone analysis is usually restricted to post-mortem fractography or indirect measurements of the strain fields. A quantitative study of plastic deformation at the microstructural scales is clearly critical in such failure analyses. Recently a number of nondestructive deformation analysis tools have been developed, notably including the

neutron and synchrotron x-ray diffraction techniques. These methods provide microstructural level strain mapping, which is critical in understanding deformation and failure of traditional and novel structural materials. To better investigate the micromechanical properties around the fatigue crack tip, in our model, a multiscale view of the crack tip plasticity is implemented based on the work of van der Giessen and Needleman (van der Giessen and Needleman, 2002). The crack tip plastic zone in front of the tip and the plastic wake left behind are treated as the plastic mechanisms at the macroscopic scale, which can be reproduced naturally by the irreversible, hysteretic cohesive interface model and the continuum surrounding plasticity without any references to the material microstructure. As we discussed above, crystal plasticity theory is capable to simulate the lattice strain at the grain size level. Thus the key difficulty becomes the quantitative connections between the fatigue macroscale/continuum plastic zone and the model of the lattice strain simulation.

In this Chapter, we first review these advanced diffraction methods and representative results in Section 1.1, and then survey a number of simulation tools that can be used in conjunction with these novel experimental findings in Section 1.2. An outline of the thesis will be given in Section 1.3.

1.1. Experimental motivations

With the establishment of many user-friendly facilities worldwide in the past several decades, neutron and synchrotron X-ray diffraction techniques gain popularity as non-destructive tools to probe the microstructures and mechanical properties of advanced structural materials. Because of the deep penetration capability of neutron beams, neutron diffraction allows bulk measurements with a typical spatial resolution in the order of millimeters. Recently neutron diffraction techniques have been widely

used to study the effects of material microstructure, thermo-mechanical loading conditions, and environment, among many others on the deformation and failure behavior of polycrystals (Clausen et al., 1998; Pang et al., 1998; Garlea et al., 2010; Wong and Dawson, 2010). The neutron powder diffraction has an instrumental resolution of $\Delta d / d = 2.5 \times 10^{-3}$, making it suitable to measure the small shifts in lattice spacing associated with internal strains (Brown et al., 2005). Similar to the X-ray diffraction, the lattice strain can be recorded from the shift of the diffraction peaks, thus corresponding to the elastic lattice distortion of the grains that satisfy the diffraction conditions. The lattice strain relies on the intergranular interactions of the inhomogeneous deformation fields in neighboring grains, so that it is sometimes referred to as Type-II strain. In the elastic deformation stage, the anisotropy in various lattice strains ε_{hkl} is solely determined by the elastic anisotropy and texture. In the plastic deformation stage, deformation mechanisms such as plastic slip and intergranular cracks may contribute to the evolution and anisotropy of lattice strains.

Fig. 1.1 schematically represents the strain measurement by neutron diffraction in two different directions: axial (in-plane) and transverse (through-thickness) of specimen. The incident neutron beam impinges on the sample with an angle rotated from the loading axis and is scattered onto two detector banks situated at $\pm 90^\circ$ relative to the incident beam. The time-of-flight technique utilizes the continuous energy spectrum and pulsed structure of the neutron beam to collect an entire diffraction pattern (effective d-space range from 0.3-4 Å) in each detector panel simultaneously (Brown et al., 2005). Diffraction conditions select the grain with the plane normal along the direction of the diffraction vector and thus the lattice strain for a specific crystallographic plane is calculated by the lattice spacing obtained by

the peak shifting of the diffraction pattern. One of the lattice strain diffraction results from the neutron diffraction technique is shown in Fig. 1.2 (a) and (b) (Garlea, 2008). In the experiments, the microscopic deformation mechanisms under uniaxial and fatigue loading in a Zircaloy-4 Alloy are studied. Zircaloy-4 is a hexagonal structural material with both slip and twin mechanisms observed when deformed plastically. The detailed mechanical properties of Zircaloy-4 will be studied in Chapter 6. It is necessary to distinguish the lattice strain, ε_{hkl} , from the intergranular strain, λ_{hkl} , as shown in Fig 1.2. Intergranular strain is denoted as the deviation of the lattice strain ε_{hkl} from the projected values from the elastic stage. Both the lattice strain and the intergranular strain exhibit significant anisotropy when the plastic stage is reached.

Intergranular strain depends on lattice structures, slip modes, grain texture and morphology, among many others, because it arises in polycrystalline metals and alloys to accommodate the deformation incompatibilities among neighboring grains. Besides, intergranular strain exhibits large anisotropy with respect to the variation of the diffraction vector during neutron diffraction measurements. The directly measured intergranular strain can also be used to construct the Stress Orientation Distribution Function (SODF) to calculate the corresponding intergranular stress (Wang et al., 2003). The residual intergranular stress, which is defined as the internal stress when the external load is completely removed, is investigated by the previous diffraction tests to vary among grains due to the elastic anisotropy caused by the different crystallographic orientations (Dawson et al., 2000). It is believed that the heterogeneous stress and strain developed in a polycrystalline material is critical for the crack initiation (Guiu et al., 1982) because the residual stress combined with the applied stress or together with some unknown defects or poor microstructures can somehow lower the critical stress when failure occurs (Ward-Close et al., 1989). In

reality, failure caused by cyclic loadings with the loading stress lower than the material yield stress is much more common than under any other loading conditions.

A number of low-cycle-fatigue tests on face-centered cubic (FCC) polycrystals (Wang et al., 2003; Huang et al., 2010) have discovered the gradual decrease of the intergranular strains (or equivalently, the gradual change of lattice strains to the projected values from elastic stage) with the increase of fatigue cycles under uniaxial loading condition and load ratio of $R = -1$. The residual lattice strains are vanishing rapidly when intergranular cracks emerge in the late stage of the LCF test, indicating that intergranular damage is the dominant fatigue mechanism. Thus the residual intergranular stress anisotropy can be used to monitor the initiation and propagation of cracks under fatigue. The representative grain-orientation-dependent residual intergranular strains are plotted in Fig. 1.3 with the process of the fatigue test (Wang et al., 2003). The residual intergranular stresses for different grain orientations calculated by the SODF are also shown as a form of inverse pole figures in Fig. 1.4. Further discussions of the grain-orientation-dependent residual intergranular strain/stress will lead to the investigation of the intergranular damage rate, which will be given in details in Chapter 3 and 4.

The ideal fatigue test is under the constant-amplitude cyclic loads, which is however rarely observed in reality. Instead, materials or components are usually exposed to variable-amplitude loading. Thus lots of interests have been given to the study of the fatigue with single or multiple overloads, which is often referred as the study of the overload effects. One famous phenomenon is the crack closure mechanism, sudden changes in the cyclic mechanical-loading patterns results in a significant acceleration and/or retardation of the crack growth as clearly shown in Fig. 1.5 for HASTELLOY C-2000 (Lee, 2008). The change of the crack-growth rate

(da/dN) is plotted as a function of the applied stress intensity factor range (ΔK) under fatigue, fatigue with a single tensile overload and fatigue with a compressive underload conditions respectively. It clearly shows an instantaneous acceleration of crack-growth rate followed by a large retardation period, resulting in a temporary decrease in the crack-propagation rates for the single tensile overload condition. It is believed that the compressive residual strain developed behind of the crack tip is the root cause of the crack growth retardation because it requires a higher crack-opening load to compensate it to make the crack growth. Again, the connection between such a macroscopic loading effect and grain-level damage process cannot be established without a knowledge of inter- and intra- granular deformation behavior.

In this dissertation we will focus on the micromechanical properties of the fatigue crack growth, such as the lattice strain distribution around the crack tip and the intergranular damage phenomenon during fatigue, etc. The materials involved in this study include the FCC structural C2000, hexagonal-close-packed (HCP) structural Zircaloy-4 and AZ31B polycrystalline alloys. Compared to FCC structural materials, investigations for HCP structural materials are much more comprehensive due to the asymmetry of the crystal structural and the deformation twinning, both of which have a particularly important correlation to the material texture. Neutron diffraction technique is capable of the texture measurement for the polycrystalline materials. For the material with initial texture, the peak intensity from the scattered neutron beam is showing different peak intensity from that with a random texture. Thus the full pole figure can be generated by investigating the intensity variation of one family of specific crystallographic planes as a function of sample orientation. Fig. 1.6 shows the texture measured by the neutron diffraction technique for a hot-rolled magnesium alloy AZ31B plate (Brown et al., 2005). AZ31B has a well defined but

simple crystallographic texture. The preferred orientation of grains within the plate has basal poles parallel to the plate normal direction and exhibits a near random orientation of prism poles about the axis.

With all the fascinating facts of neutron scattering, such as the large scattering volume, capable for samples with grain size larger than 80 microns, etc., it does have some drawbacks, for instance, the insensitivity to the presence of geometrically necessary dislocations (GNDs) and geometrically necessary boundaries (GNBs). Instead, X-ray microbeam can map the three-dimensional (3D) spatially resolved dislocation character within a submicron volume. Fig. 1.7 shows the dislocation density as a function of distance from the crack tip immediately after overload by polychromatic X-ray microdiffraction in the study of the fatigue crack deformation behavior of ferrite stainless steel (Barabash et al., 2008).

Another interesting line investigated in the current dissertation is the lattice misorientation in single crystals under indentation, which will be an extended study for the Indentation Size Effect (ISE). ISE denotes the phenomenon that when decreasing the indentation depth the hardness is increasing, especially when the penetration depth is less than micrometers, which is caused by the increases of the yield or flow strength at the small scale (Pharr et al., 2010). ISE can be explained by the concept of GNDs. The flow stress is related to the total dislocation density, which requires the accounts of the density of GNDs and SSDs (Statistically Stored Dislocations). During indentation, the material originally located underneath the indent region must have been pushed into the underneath substrate material as the inelastic deformation is accommodate by the GNDs (Gao and Huang, 2000). Thus GNDs will increase the hardening component when the contact area is smaller. Considering that it is not easy to obtain the information of the dislocation structures

directly, alternatively, X-ray diffraction, backscattered electron diffractions (EBSD) in high-resolution scanning electron microscopes (SEM), convergent beam electron diffraction (CBED) in scanning transmission electron microscopes (STEM) are capable of measuring the local lattice orientations. Lattice misorientation or curvature can be used to evaluate the local GNDs densities (Larson et al., 2007).

Three-dimensional x-ray measurements for the deformation microstructure under microindents in single-crystal copper have been conducted by Yang et al. (2003). In the experiment, point to point, micrometer-resolution x-ray microbeam measurements of local lattice rotations are made for selected positions under 100 mN Berkovich and conical indents in single-crystal copper. The copper crystal is orientated as shown in Fig. 1.8, so that the crystallographic axis $[110]$ is normal to the plane of the figure and axis $[112]$ is along the trace of the surface. The edges of the Berkovich indenter are aligned within 80° of $[110]$ directions in the surface plane of the sample. The nondestructive measurements of local lattice orientations and lattice curvatures can be made as a function of depth for all positions below the indents by rastering the sample beneath the x-ray beam and performing depth scans for micrometer steps. As shown in Fig. 1.8 (a), position A and B are measured with the x-ray incident microbeam. The measured results are shown in Figs. 1.8 (b) and (c) for the lattice misorientation angle as a function of the penetration depth.

1.2. Modeling methodologies

Driven by the development of the diffraction techniques as described in Section 1.1, simulations for material micromechanical behaviors, such as lattice strain, intergranular stress/strain, lattice misorientation, etc, are consequently extensively studied by a variety of simulation methods including the elasto-plastic

self consistent model (EPSC) (Clausen et al., 1998; Agnew et al., 2003; Brown et al., 2005; Garlea, 2008), the dislocation dynamics models (Balint et al., 2006), and the continuum crystal plasticity model (Dawson et al., 2000; Wong et al., 2010).

The EPSC model considers each grain as an elastoplastic anisotropic ellipsoidal inclusion embedded in an elastic-plastic anisotropic homogeneous effective medium (HEM). The HEM is treated as a textured anisotropic aggregate with unknown properties. The stress and strain turn out to be uniform inside the inclusion but different from the macroscopic stress and strain rate. The average stress over grains has to be equal to the macroscopic stress. The interaction between the grain and the HEM is carried out by an elastoplastic Eshelby approach (Hill, 1965). The homogenization of these grains will lead to the HEM properties, hence called self-consistent approach. The model can be used to predict internal strain and texture evolution, residual and thermal strains and to compare with neutron diffraction experiments (Tome et al., 1991; Pang et al., 1999). Fig. 1.9 (a) is the lattice strain evolution for copper polycrystals simulated by Clausen et al. (1998) using the EPSC model and (b) shows the calculation results for Zircalloy-4 by the EPSC model and the neutron diffraction experiment (Garlea, 2008). It is easy to notice that once entering the plastic deformation region, the simulated lattice strain starts to deviate from the neutron results, which might be caused by the inappropriate hardening parameters or other complicated mechanisms involved.

Another unique opportunity to the theoretical study of the intergranular strain/stress is offered by the microstructure- and micromechanics-based crystal plasticity theory. The plasticity behavior reveals different characteristics at different length scales. At the macroscopic length scale, one can use classic J_2 -type plasticity with calibrated hardening law and rate-sensitivity. Such a continuum plasticity theory

is only valid when the deformation is insensitive to the grain length scale, and provides only a phenomenological fitting to the experiments. At the microstructural length scale, plasticity is governed by individual processes that create and multiply dislocations inside the grains, which can be modeled by intensive dislocation dynamics simulation or continuum crystal plasticity theory. Even though some lattice strain or intergranular strain calculations have been done by Dawson et al., using the crystal plasticity finite element simulation (Dawson et al., 2000), it is still challenging to (i) quantitatively characterize and predict the interplay between plasticity and damage evolutions at mesoscopic length scales for fatigue; (ii) predict the Type III strain or intragranular strain for polycrystals; (iii) investigate the ISE by the lattice misorientation calculation; (iv) predict deformation behavior when twinning or other nontrivial deformation mechanisms are involved.

Fatigue life prediction is still an empirical art at present. Following a number of stress intensity factor based models, it is argued that they cannot completely capture the essential physics of fatigue-crack growth. Computationally, the cohesive theories of fracture are used as an alternative approach, which regards the crack failure as the separation of two weakly bounded surfaces. The traction and separation between the surfaces both increase until the separation reaches a critical value, beyond which the traction eventually reduces to zero upon the attainment of the maximum value of the separation, where the failure occurs. A number of phenomenological cohesive zone models have been proposed and can successfully model the monotonic fracture plasticity (Needleman, 1990; Pandolfi and Ortiz, 1988). However these models cannot capture the crack growth behavior due to fatigue loading. Thus a hysteresis, irreversible hysteretic cohesive zone model has been developed by to simulate the dissipative mechanisms (Nguyen et al., 2001). As we

discussed at the beginning of this chapter, the key difficulty of our fatigue damage simulation is how to quantitatively connect the fatigue macroscale/continuum plastic zone and the model of the lattice strain simulation. From the macroscopic point of view, the deformation field near a steady fatigue crack includes a plastic zone in front of the crack tip and a plastic wake behind it, where the cyclic loading and fatigue crack growth lead to a compressive residual strain. We believe that the magnitude, distribution, and history of the residual strain along the crack path depend on the stress multiaxiality, material properties, and history of stress intensity factor and crack growth rate.

The irreversible cohesive zone model with J2-plasticity can faithfully simulate such a fatigue crack growth behavior, which generates the strain/stress distributions and their histories near the tip, which will be passed as inputs to the crystal plasticity model that connects macro-strain and lattice strains.

Another application of the crystal plasticity theory is the lattice misorientation calculation. A number of simulations have been implemented to investigate the ISE by predicting the lattice misorientation fields under indentation, for instance, the discrete dislocation (DD) plasticity simulations and the continuum crystal plasticity simulations. The DD simulations are capable of capturing size dependent hardening but the predicted lattice rotation fields disagree with the experimental measurements (Balint et al., 2006). However the continuum crystal plasticity finite element simulations with the same slip systems specified in the DD simulation can well predict the rotation sectors to the wedge indentation experiments (Pharr et al., 2010). In the current dissertation, we will further examine the lattice misorientation pattern under indentation.

The mechanical behavior of hexagonal materials, such as magnesium alloys, is controlled by the relative strengths and hardening responses of a variety of slip and twinning modes (Agnew et al., 2003). Thus the constitutive modeling of the plastic deformation of hexagonal crystals is much more complicated than that in most cubic structural crystals. More specifically, possible slip systems that could be activated during plastic deformation in hexagonal crystals at room temperature are even difficult to be identified due to the low crystallographic symmetry and the insufficient slip directions. There are some recent development based on either crystal plasticity framework (Staroselsky and Anand, 2003) or EPSC approach (Clausen et al., 1998).

1.3 Outline of this thesis

Based on the above discussion, this dissertation is organized as follows. In Chapter 2, we simulate the evolution of lattice or intergranular strains in FCC polycrystals by a slip based crystal plasticity constitutive. The calculation results are compared to the neutron diffraction data and the self consistent model respectively, which give the better understanding of the material plastic properties at the microstructural length scale. A two-scale model is used to simulate the crack tip growth under fatigue for FCC polycrystals in Chapter 3. An irreversible, hysteretic cohesive interface model is adopted to simulate a steady fatigue crack, which allows us to generate the stress/strain distribution and history near the fatigue crack tip. Such a deformation history will be used as inputs for the micromechanical analysis of lattice strain evolution using the slip-based crystal plasticity model, thus making a mechanics connection between macro- and micro-strains. Experimental measurements were conducted using the neutron diffraction technique for the fatigue test of a Ni-based superalloy compact tension specimen. Predictions from perfect

grain-boundary simulations will be compared with the experimental results. The orientation-dependent-residual lattice strain is studied by our slip-based crystal plasticity model in Chapter 4. The simulation results will be compared with the neutron experimental results to further prove the intergranular damage mechanism responsible for fatigue damage. Besides, the lattice strain distribution around the fatigue crack tip will be recalibrated by taking consideration of the intergranular damage on the crack process zone. In Chapter 5 the lattice misorientation prediction is conducted by the continuum crystal plasticity model in FCC single crystals when the plane strain condition is satisfied. The calculation is also conducted with gradually change the initial lattice orientation. Furthermore the 3D Berkovich indentation is simulated and the results will be compared with the X-ray diffraction results. A constitutive model considering both slip and twinning behavior in the plastic deformation stage in Chapter 6 is used to capture the lattice strain evolution in HCP polycrystals, such as Zirconium and magnesium alloys. The calculation results will be compared with the neutron diffraction data and the EPSC model results. Chapter 7 gives the conclusions of the present work and some suggestions for future work.

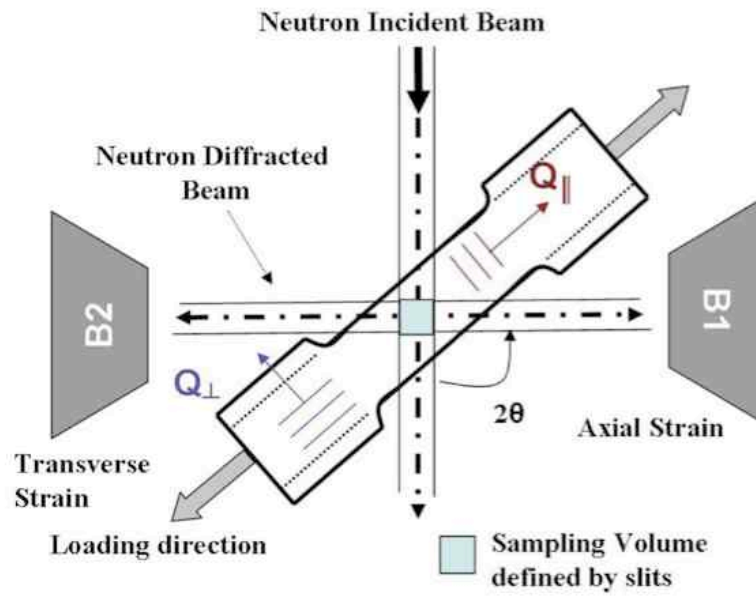


Figure 1.1. Schematic representation of the strain measurement by neutron diffraction technique in two different directions: axial (in-plane) and transverse (through-thickness) of specimen (Garlea, 2008).

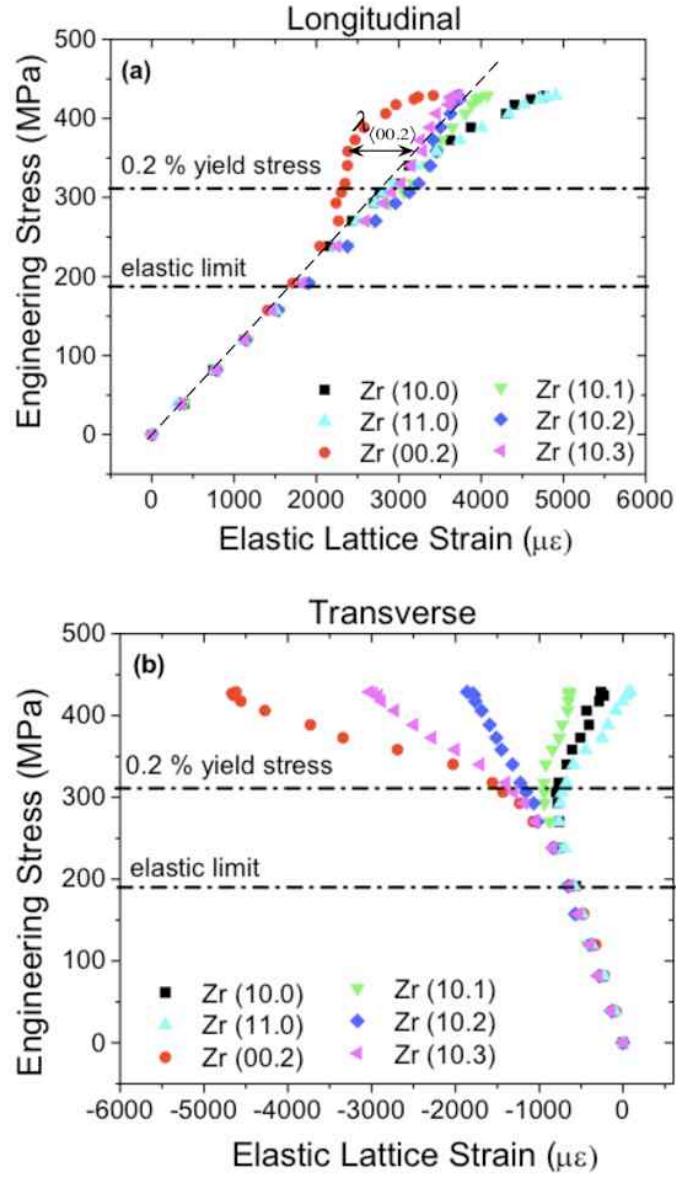


Figure 1.2. The lattice strain evolution under the applied tensile load for (a) longitudinal direction with the intergranular strain λ_{002} indicated in the figure and (b) transverse directions (Garlea, 2008).

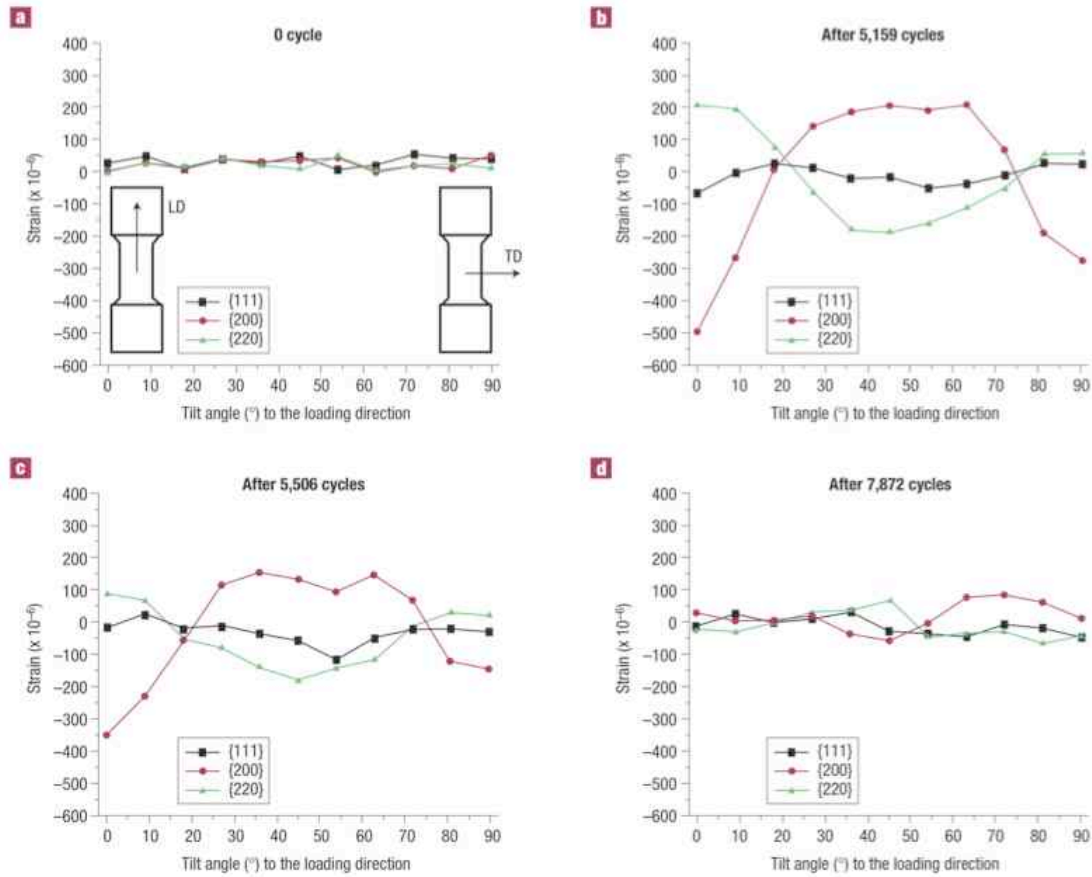


Figure 1.3. Lattice strain distribution in the 316 stainless steel obtained with {111}, {200}, and {220} reflections as a function of tilt angle relative to the loading direction at different stages of fatigue. (a) 0 cycle or virgin sample; (b) 5159 cycles when microcracks start to appear on the surface; (c) 5506 cycles after cracks start to propagate into the bulk; (d) 7872 cycles after fatigue failure is reached (Wang et al., 2003).

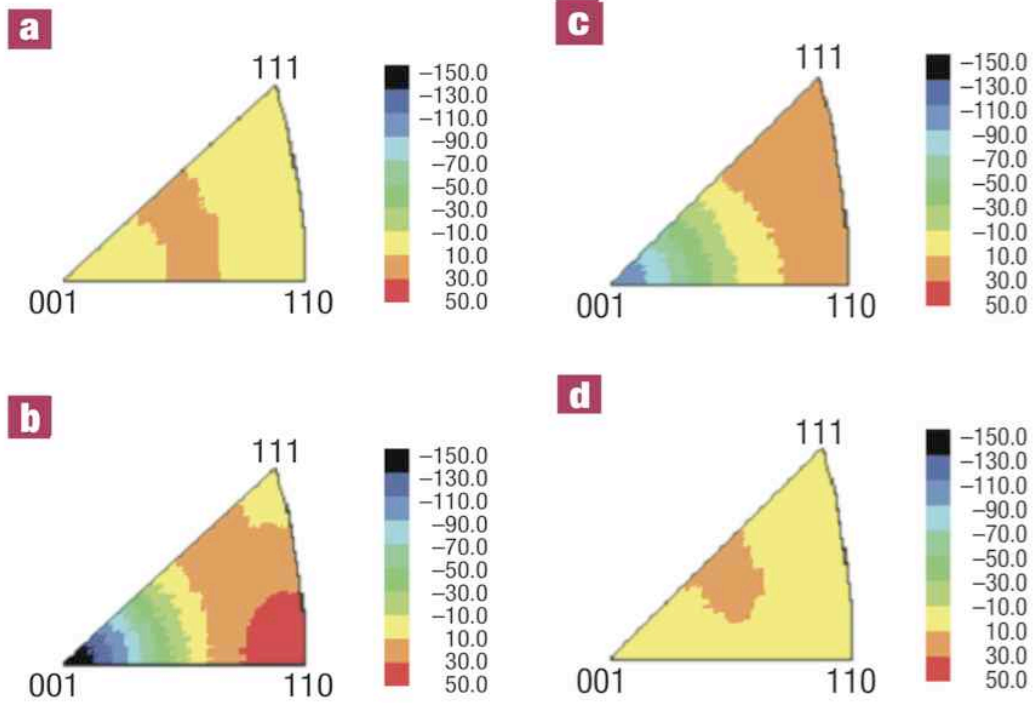


Figure 1.4. Distribution of residual stresses for various grain orientations in the cyclically deformed 316 stainless steel presented in the form of inverse pole figures. (a) after 0 cycles; (b) after 5159 cycles; (c) after 5506 cycles; (d) after 7872 cycles. The unit of stress is MPa (Wang et al., 2003).

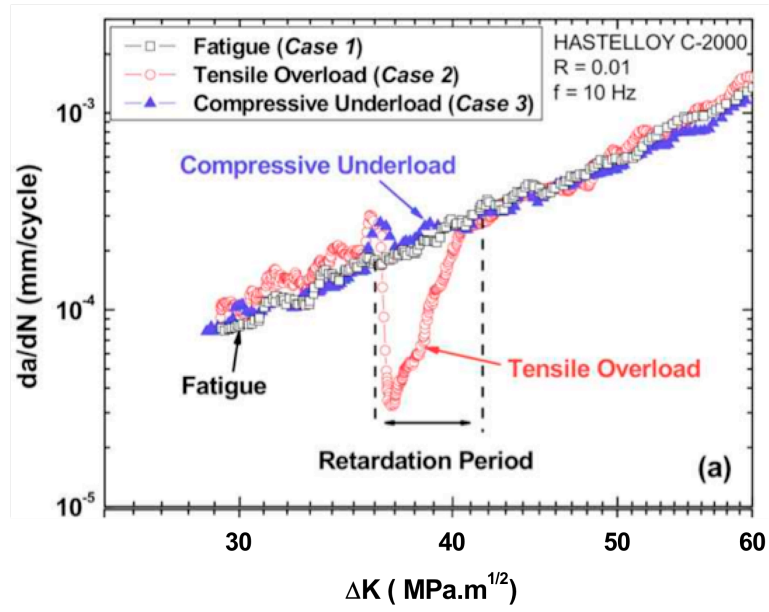


Figure 1.5. The change in the crack-growth rate (da/dN) as a function of the applied stress-intensity-factor range (Lee, 2009).

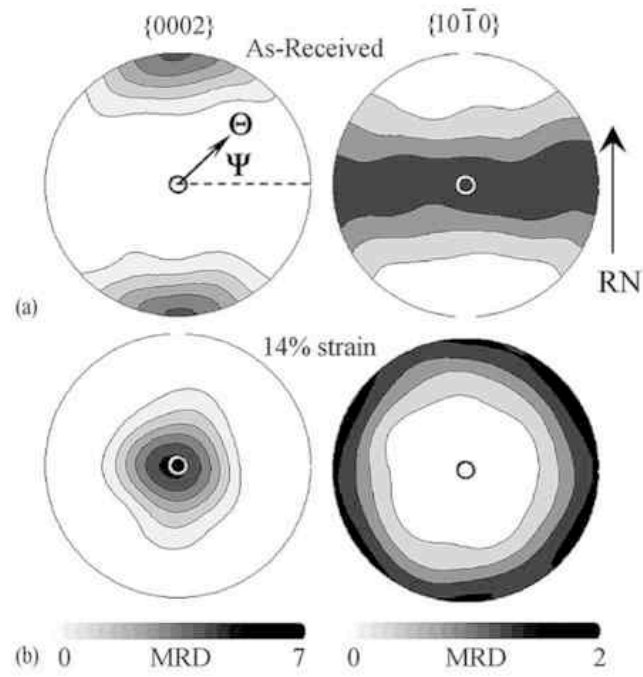


Figure 1.6. Measured texture of rolled magnesium plate (a) as-received, (b) after 14% in-plane compression (Brown et al., 2005).

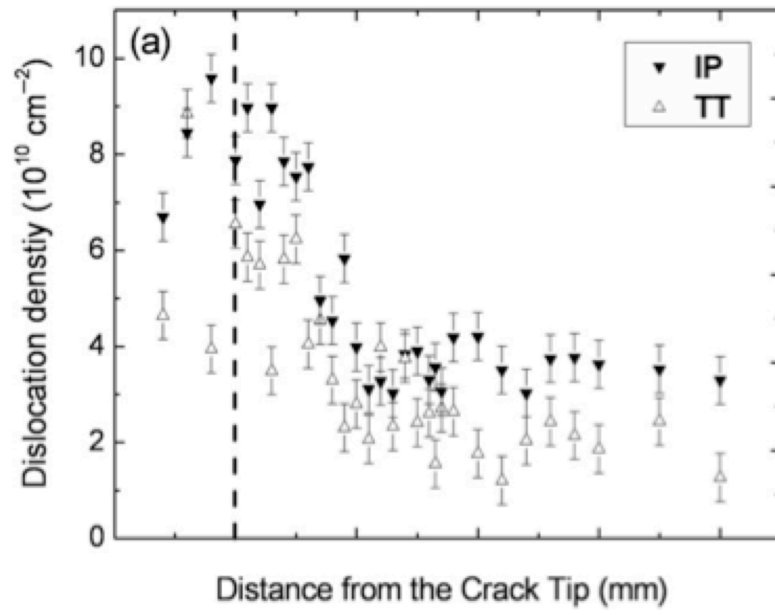


Figure 1.7. Dislocation density distribution for IP and TT directions as a function of distance from the crack tip immediately after fatigue overload (Barabash et al., 2008).

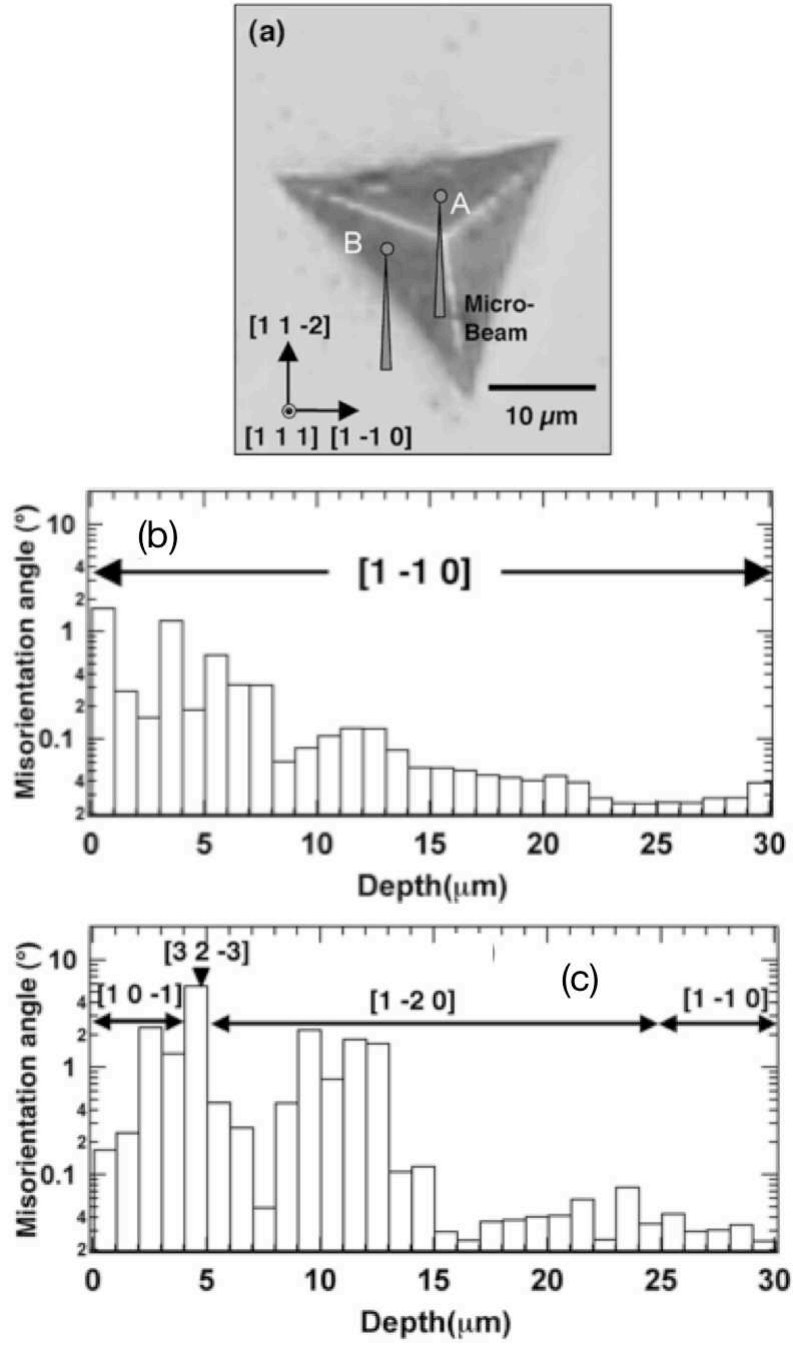


Figure 1.8. (a) Optical image of a 100-mN Berkovich indentation in a single-crystal Cu sample. The arrows at position A and B indicate the microbeam direction and entry points (Yang et al., 2003). The beams are at 45° incident to the surface. Log plot of local misorientation angles for position (b) A and (c) B as a function of penetration depth with the rotation axis labeled $[1\ -1\ 0]$.

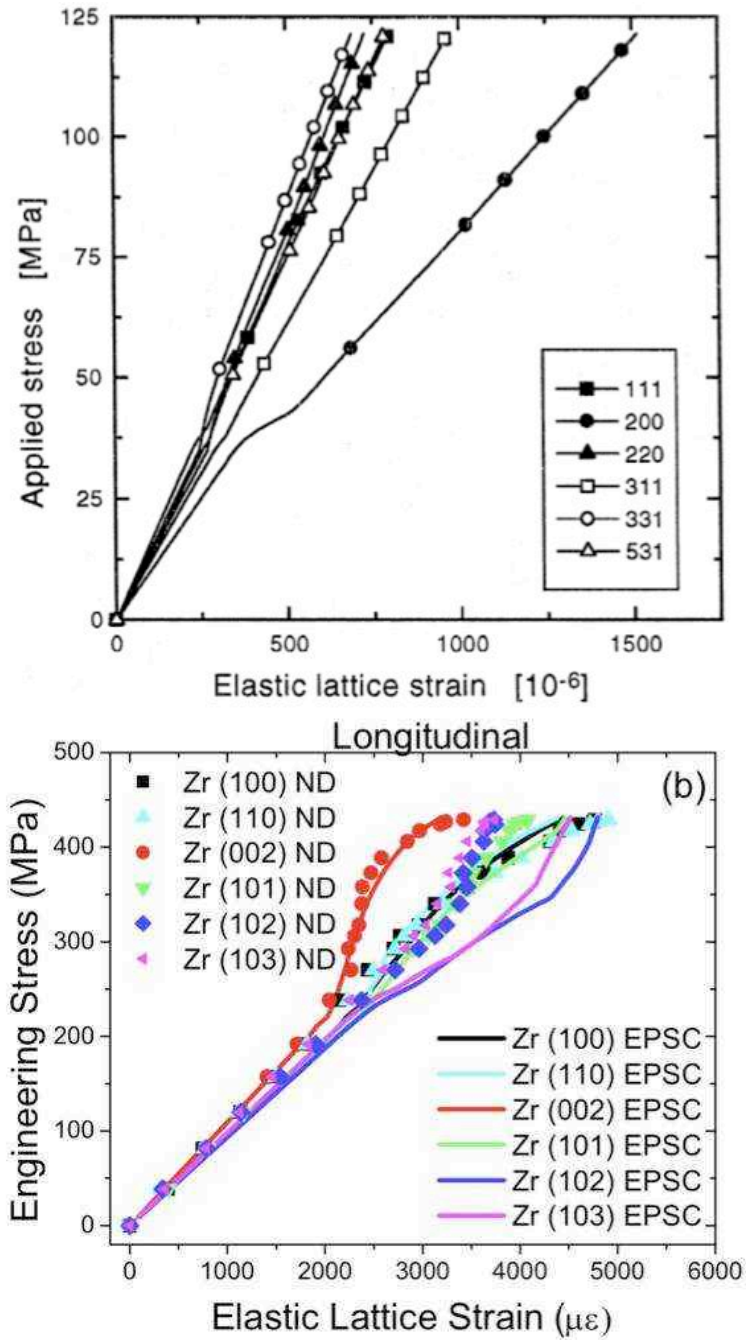


Figure 1.9. Neutron diffraction data and the EPSC calculation: (a) lattice strain evolution for copper polycrystals (Clausen, et al., 1998); (b) considering the combination of slip systems and tensile twinning for the lattice strain evolution in Zircalloy-4 under applied load in longitudinal direction for the basal, prism, and pyramidal planes (Garlea, 2008).

Chapter 2

Crystal plasticity analysis of lattice and intergranular strains in polycrystals

2.1. Lattice and intergranular strains

Lattice strains or intergranular strains are the results of the plastic incompatibility among neighboring grains caused by different grain orientations in polycrystalline materials. Polycrystals are regarded as the aggregates of a number of single crystalline grains with each of them governed by their unique crystalline orientations associated with activated slip systems. When deformed there should be no overlap or void generated between neighboring grains causing the grain-to-grain interactions. Lattice strain ε_{hkl} is defined as the change of the lattice spacing divided by the original value, while the intergranular strain λ_{hkl} is the deviation of the lattice strain from the projected values from the elastic stage as shown in Fig. 1.2. Lattice strain anisotropy in the elastic deformation stage is solely determined by the elastic anisotropy and texture, while when entering the plastic stage, they are controlled by a number factors associated with the crystalline slip, deformation twinning and many other mechanisms. Thus the study of lattice or intergranular strains leads to the investigation of the material plastic properties, which, by their nature, in most cases arise from the deformation slip or twinning, alter the micro structural state of a metal, and, in so doing, modify its derivative mechanical properties.

On the other hand, the residual intergranular stress, which is defined as the internal stress existing even when the applied load is completely removed, can also be

calculated from the lattice or intergranular strains. For instance, the Stress Orientation Distribution Function (SODF) is constructed on the base of the lattice strain information (Wang et al., 2002). The residual stress arises naturally when a polycrystal is unloaded following plastic straining, whether or not the macroscopic elastoplastic deformation is uniform.

Thus to better understand the properties of the residual intergranular stress and the material micromechanical properties in plastic region, in this chapter we will focus on the lattice or intergranular strains evolution and anisotropy behavior under uniaxial loadings by neutron diffraction technique and the crystal plasticity modeling.

2.2. Simulation procedure

In this chapter, the lattice strain evolution is calculated from a crystal plasticity model. We follow the works by Peirce et al. (1982) and Bower and Wininger (2004), and modify the ABAQUS user-defined material (UMAT) subroutine by Huang (1991). From the kinematics point of view as schematically shown in Fig. 2.1, the total deformation gradient, $F_{ij} = \partial x_i / \partial X_j$, with current and original coordinates being x_i and X_i , respectively, can be represented as a multiplicative decomposition:

$$F_{ij} = F_{ik}^e F_{kj}^p, \quad (2.1)$$

where the plastic part, F_{ij}^p , arises from the crystalline slip on a given set of slip systems,

$$\dot{F}_{ik}^p F_{kj}^{p-1} = \sum_{\alpha=1}^{NSLIP} \dot{\gamma}^{(\alpha)} s_i^{(\alpha)} m_j^{(\alpha)}, \quad (2.2)$$

$s_i^{(\alpha)}$, $m_i^{(\alpha)}$, and $\dot{\gamma}^{(\alpha)}$ are the slip direction, the slip plane normal, and the slip strain rate of the α -th slip system, respectively, and $NSLIP$ is the total number of slip systems. The elastic deformation gradient, F_{ij}^e , describes the lattice stretching, rotation, and rigid body motion. The elastic constitutive relationship is given by

$$T_{ij} = C_{ijkl} E_{kl}^e, \quad (2.3)$$

where the material stress tensor T_{ij} relates to the Cauchy stress σ_{ij} by $J\sigma_{ij} = F_{ik}^e T_{kl} F_{jl}^e$,

$J = \det(F^e)$, and the elastic Lagrange-Green strain is $E_{ij}^e = \frac{1}{2}(F_{ki}^e F_{kj}^e - \delta_{ij})$.

We use the power-law flow rule and the Peirce-Asaro-Needleman hardening law, as given by

$$\dot{\gamma}^{(\alpha)} = \dot{\gamma}_0 \left| \frac{\tau^{(\alpha)}}{\tau_{flow}^{(\alpha)}} \right|^n \text{sgn}(\tau^{(\alpha)}), \quad (2.4)$$

$$\dot{\tau}_{flow}^{(\alpha)} = \sum_{\beta} h_{\alpha\beta} |\dot{\gamma}^{(\beta)}|, \quad (2.5)$$

where $\dot{\gamma}_0^{(\alpha)}$ is a characteristic strain rate, $\tau^{(\alpha)} = \sum_{\beta} h_{\alpha\beta} |\dot{\gamma}^{(\beta)}|$ and $\tau_{flow}^{(\alpha)}$ are the resolved shear stress and the flow strength of the α -th slip system, respectively, n is the stress exponent, and $h_{\alpha\beta}$ are hardening moduli. The self-hardening modulus is

$$h_{\alpha\alpha} = h(\gamma) = h_0 \text{sech}^2 \left| \frac{h_0 \gamma}{\tau_s - \tau_0} \right|, \quad (2.6)$$

where $\gamma = \int_0^t \sum_{\alpha} |\dot{\gamma}^{(\alpha)}| dt$, h_0 is the initial hardening modulus, τ_0 is the initial slip strength, and τ_s is the saturation slip strength. No summation on α is assumed in Eq. (2.6). The latent hardening moduli are given by

$$h_{\alpha\beta} = h(\gamma) \left[q + (1-q) \delta_{\alpha\beta} \right], \quad (2.7)$$

when $\alpha \neq \beta$.

The polycrystal aggregate in Fig. 2.2(a) consists of 3,375 cubic grains, each having the same crystal plasticity parameters but different crystallographic orientations. The extraction of lattice strains, ε_{hkl} , is specified as follows. We select a subset of grains whose $\langle hkl \rangle$ directions are parallel to (or within a small tolerance angle from) the diffraction vector, $\mathbf{q}$, which is consistent with the diffraction condition in diffraction method as shown in Fig. 1.1. The tolerance, i.e., the maximum misalignment between $\langle hkl \rangle$ and $\mathbf{q}$, is chosen in our simulations to ensure that a large number of grains (usually about 1~2% of the total grains) can be selected. This generally leads to a choice of $\pm 5^\circ$, in agreement with the self-consistent model (Clausen et al., 1998). Fig. 2.2(a) displays the σ_{22} contours when the applied stress is 400 MPa and Fig. 2.2(b) shows the statistical fluctuations of the deformation fields in the selected $\{100\}$ grains. Material parameters used in these calculations will be discussed shortly. The lattice strain, ε_{hkl} , is a volume average of the projected elastic strain, as given by

$$\varepsilon_{hkl} = \frac{\sum_{N=1}^{NGRAIN} \int \varepsilon_{ij}^{elastic} q_i q_j d\Omega_N}{\sum_{N=1}^{NGRAIN} \int d\Omega_N}, \quad (2.8)$$

where $d\Omega_N$ is the differential volume of the N -th grain, and $NGRAIN$ is the total number of grains selected as in Fig. 2.2(b).

For the comparison with the neutron diffraction and other simulation methods, we choose copper and a Ni-based superalloy (HASTELLOY C-22) as the test materials. The development of elastic lattice strains during uniaxial loading for selected families of grains with specific orientations for copper polycrystal was modeled using the self-consistent scheme (Clausen et al., 1997). The comparison between their results and crystal plasticity model in this work are shown in Fig. 2.4(a). Material parameters involved in the crystal plasticity theory as given in Table 2.1(a) are similar to the self-consistent model parameters used in Clausen's calculation. The lattice strain anisotropy in the elastic stage is solely determined by the anisotropy in the elastic constants since the polycrystal aggregate considered here does not have a texture. As mentioned before, the lattice strain is primarily governed by the short-range interactions among neighboring grains. Thus, provided with elastic properties, slip systems (e.g., 12 fcc slip systems in our material), and material microstructure, we can use the Taylor model to understand the “splitting” behavior of lattice strains beyond the yield point. In the Taylor model, all the grains are assumed to have the same strains as the macroscopic aggregate. Therefore, the first grain to yield will be the one with the lowest strength-to-stiffness ratio, $r_s^{hkl} = \sigma_Y^{hkl} / E_{hkl}$, where σ_Y^{hkl} is the yield stress if the sample is uniaxially tested along $\langle hkl \rangle$ direction and thus is reciprocal to the Schmid factor (Wong and Dawson, 2010). As shown in Table 2.2, r_s^{100} is the largest, so that when yield is reached, ε_{110} and ε_{111} will cease to increase while the “hard” $\{100\}$ grains will carry the applied load and their further deformation will decrease the slope of $\sigma \sim \varepsilon_{100}$ curve.

Another material examined in this study is the nickel-based superalloy (HASTELLOY C-22HS with Ni-21Cr-17Mo in the weight percentage). This alloy has been extensively used under corrosive environments and where high strength is demanded. The alloy is annealed at 1080° C to minimize the formation of precipitates and to achieve a single-phase, face-centered cubic (FCC) alloy, as characterized by transmission-electron-microscopy (TEM) (Lu et al., 2007). The average grain size of this alloy is about 90 μm (Huang et al, 2008). The tensile specimens were selected according to the standard ASTM/E 606 and then in-situ neutron measurements were used to monitor the monotonic-tension experiments (Huang et al., 2008). The specimen structure and the experimental sketch are shown in Figs. 2.3 (a), (b) and (c).

Simulation for the lattice strain evolution for C-22HS is quite similar to that for copper. Material parameters involved in the crystal plasticity theory, as given in Table 2.1, are calibrated by comparing the predicted and measured lattice strains in the monotonic loading on C-22HS, as shown in Fig. 2.4 (b). Elastic constants, c_{11} , c_{12} and c_{44} , are directly referred to Huang’s work (Huang et al., 2008). As we discussed in the above, the strength-to-stiffness ratio, which are listed in Table 2.2, also works for understanding the “splitting” behavior when yielding occurs. After yielding, “hard” $\{100\}$ grains will continue carrying the applied load and the slope of $\sigma \sim \epsilon_{100}$ curve will decrease when they keep on deforming, which are similar to the behavior of copper because of the largest strength-to-stiffness ratio for $\{100\}$ grains.

2.3. Numerical example

In this section a detailed procedure on the use of crystal plasticity model and finite element simulations is provided. The crystal plasticity model in Section 2.2 has been implement as a User-defined Materials (UMAT) subroutine into the commercial

software, ABAQUS (Huang, 1990). The parameters needed for such a model include elastic and plastic parameters, slip system information, and crystallographic orientations. The related part in a representative input file is shown below.

```
*SOLID SECTION, ELSET=ONE, MATERIAL=CRYSTAL
*MATERIAL, NAME=CRYSTAL
*USER MATERIAL, CONSTANTS=160, UNSYMM
** All the constants below must be real numbers!
168400., 121400., 75400.,
** c11 , c12 , c44 , (elastic constants of copper crystal) in unit: MPa , MPa , MPa ,
0. ,
** constants only used for an elastic orthotropic or anisotropic material in unit: MPa ,
0. ,
** constants only used for an elastic anisotropic material in unit: MPa ,
** The elastic constants above are relative to crystal axes, where 1 -- [100], 2 -- [010], 3 -- [001] .
** These elastic constants are arranged in the following order: eight constants each line (data card)
** (1) isotropic:
** E , Nu (Young's modulus and Poisson's ratio)
** 0.
** 0.
** (2) cubic:
** c11 , c12 , c44
** 0.
** 0.
** (3) orthotropic:
** D1111, D1122, D2222, D1133, D2233, D3333, D1212, D1313,
** D2233
** 0.
** (4) anisotropic:
** D1111, D1122, D2222, D1133, D2233, D3333, D1112, D2212,
** D3312, D1212, D1113, D2213, D3313, D1213, D1313, D1123,
** D2223, D3323, D1223, D1323, D2323
3. ,
** number of sets of slip systems
1. , 1. , 1. , 1. , 1. , 0. ,
** normal to slip plane , slip direction , of the 1st set
1. , 1. , 1. , 1. , 2. , 2. ,
** normal to slip plane , slip direction , of the 2nd set
0.
** normal to slip plane , slip direction , of the 3rd set
-1. , 0. , 1. , 0. , 0. , 1. ,
** direction in local system , global system , of the 1st vector (the first vector to determine
crystal ** orientation in global system)
0. , 1. , 0. , 0. , 1. , 0. ,
** direction in local system , global system , of the 2nd vector (the second vector to determine
** crystal orientation in global system)
** constraint: The angle between two non-parallel vectors in the local and global systems should be
the same. The relative difference must be less than 0.1%.
10. , .001 ,
** n , adot , of 1st set of slip systems
** --- , 1/sec ,
** (power hardening exponent and hardening coefficient)
** gammadot = adot * ( tau / g ) ** n
** Users who want to use their own constitutive relation may change the function subprograms F and
** DFDX called by the subroutine
** STRAINRATE and provide the necessary data (no more than 8) in the above line (data card).
```

```

10. , .001 ,
** n , adot , of 2nd set of slip systems
** --- , 1/sec ,
0.
** n , adot , of 3rd set of slip systems
** -- , 1/sec ,
541.5 , 109.5 , 60.8 ,
** h0 , taus , tau0 , of 1st set of slip systems
** MPa , MPa , MPa ,
** (initial hardening modulus, saturation stress and initial critical
** resolved shear stress)
**  $H = H_0 * \{ \text{sech} [ H_0 * \gamma / (\text{taus} - \text{tau0} ) ] \} ** 2$ 
** Users who want to use their own self-hardening law may change the
** function subprogram HSELF called by the subroutine LATENTHARDEN
** and provide the necessary data (no more than 8) in the above line
** (data card).
1. , 1. ,
** q , q1 , Latent hardening of 1st set of slip systems
** -- , -- ,
** (ratios of latent to self-hardening in the same and different sets
** of slip systems)
** Users who want to use their own latent-hardening may change the
** function subprogram HLATNT called by the subroutine LATENTHARDEN
** and provide the additional data (beyond the self-hardening data,
** no more than 8) in the above line (data card).
541.5 , 109.5 , 60.8 ,
** h0 , taus , tau0 , of 2nd set of slip systems
** MPa , MPa , Mpa ,
1. , 1. ,
** q , q1 , of 2nd set of slip systems
0.
** h0 , taus , tau0 , of 3rd set of slip systems
** MPa , MPa , MPa ,
0.
** q , q1 , of 3rd set of slip systems
.5 , 1. ,
** THETA , NLGEOM ,
** THETA: implicit integration parameter, between 0 and 1
** NLGEOM: parameter determining whether finite deformation of single
** crystal is considered
** NLGEOM=0. --- small deformation
** otherwise --- finite rotation and finite strain, Users must
** declare "NLGEOM" in the input file, at the *STEP card
1. , 10. , 1.E-5 ,
** ITRATN , ITRMAX , GAMERR ,
** ITRATN: parameter determining whether iteration method is used to
** solve increments of stresses and state variables in terms of
** strain increments
** ITRATN=0. --- no iteration
** otherwise --- iteration
** ITRMAX: maximum number of iterations
** GAMERR: absolute error of shear strains in slip systems
* DEPVAR
245
** number of state dependent variables, must be larger than (or equal to) ten times total number of slip
** systems in all sets, plus five, plus the additional number of state variables users introduced for their
own ** single crystal model, For example, {110}<111> has twelve slip systems. There are
12*10+5=113 state ** dependent variables.

```

2.4. Summary

To summarize, a rate-dependent single crystal plasticity model is used to simulate the lattice strain evolutions in a model polycrystal with cubic grains. In the elastic stage, the anisotropy in lattice strain is solely determined by the elastic constants and material microstructure. In the plastic stage, the anisotropy in lattice strain is primarily governed by strength-to-stiffness ratios of the slip systems, as shown in our results for copper and nickel. Our detailed microstructure-based simulation agrees well with the self-consistent viscoplastic constitutive law (Clausen et al., 1998) and the direct neutron diffraction experimental data (Huang et al., 2008).

Table 2.1. Crystal plasticity parameters for copper & a Ni-based superalloy
(HASTELLOY C-22HS) polycrystal.

	c_{11} (GPa)	c_{12} (GPa)	c_{44} (GPa)	n	q	h_0 (MPa)	τ_s (MPa)	τ_0 (MPa)
Copper	168.4	121.4	75.4	50	1.0	120	20	15
C-22HS	303	210	106	50	1.0	550	750	120

Table 2.2. Directional Young's modulus and strength-to-stiffness ratio for copper & a Ni-based superalloy (HASTELLOY C-22HS) polycrystal.

	E_{100} (GPa)	E_{110} (GPa)	E_{111} (GPa)	r_s^{110} / r_s^{100}	r_s^{111} / r_s^{100}
Copper	101.5	138.7	158.0	0.75	0.95
C-22HS alloy	131.1	216.8	277.3	0.60	0.74

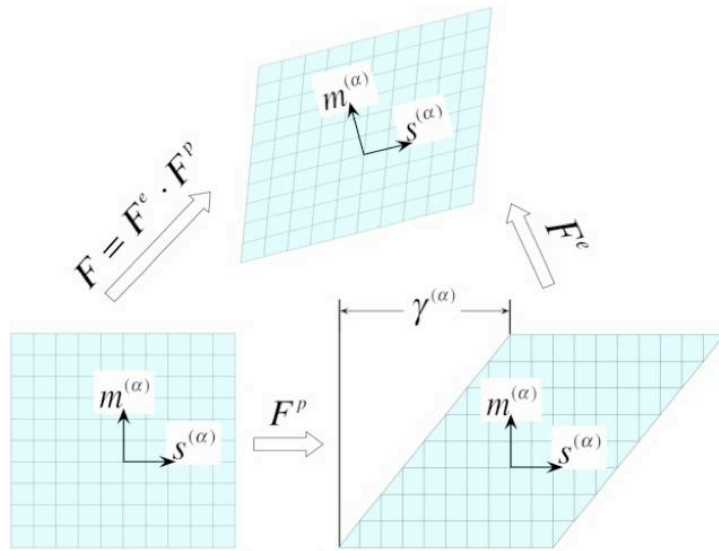


Figure 2.1. The total deformation gradient is decomposed to the elastic part and the plastic part. The elastic part is from the crystal lattice stretching, rotation and rigid body motion; the plastic part is from the crystalline slip.

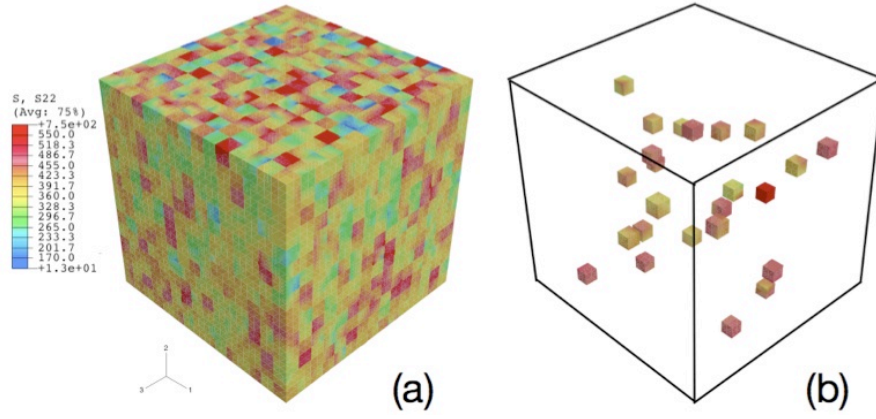


Figure 2.2. (a) Contours of σ_{22} for a model polycrystal with 3,375 cubic grains under an applied uniaxial stress of $\sigma_{22}^{applied} = 400$ MPa. The cubic grains have the same crystal plasticity parameters as in Table 1, but they are assigned with random crystallographic orientations. (b) Selected grains with the $\{100\}$ planes satisfying the diffraction condition, i.e., the angle made between $\langle 100 \rangle$ direction and the diffraction vector is within a tolerance (being 5° here). The average of $\epsilon_{22}^{elastic}$ upon all these grains gives ϵ_{100} .

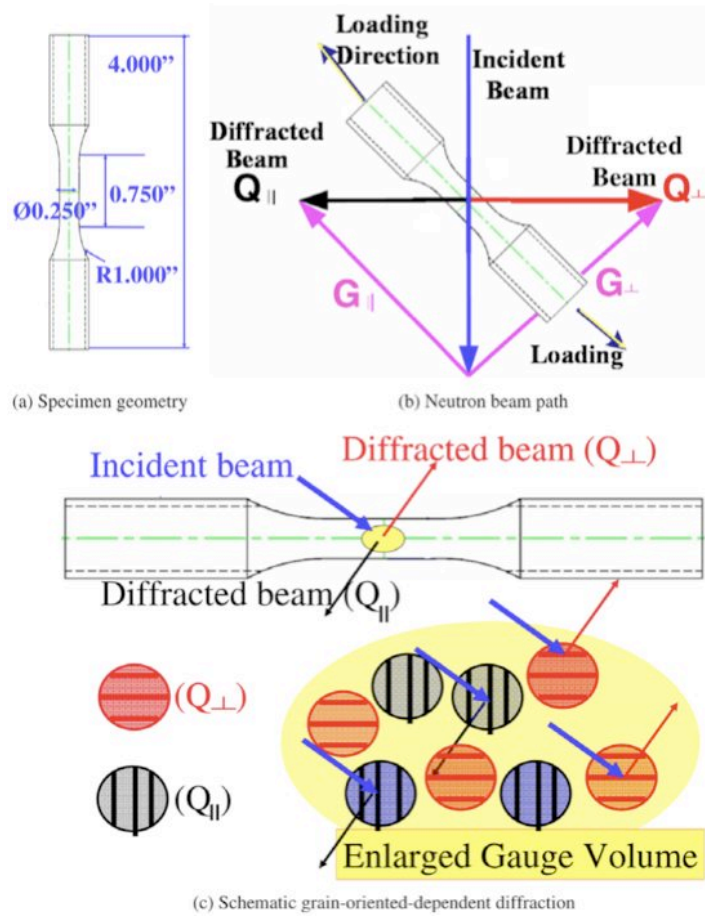


Figure 2.3. (a) The geometry of the specimen. (b) A schematic view of the neutron beam path for measurement. (c) The schematic plot shows that only the grains satisfying Bragg's condition diffract the incident neutrons to the detector banks. The grains with $\{hkl\}$ planes along loading direction ($Q_{\parallel}$) are shown in gray. The grains with $\{hkl\}$ planes in the transverse direction ($Q_{\perp}$) are shown in red (Huang et al., 2008).

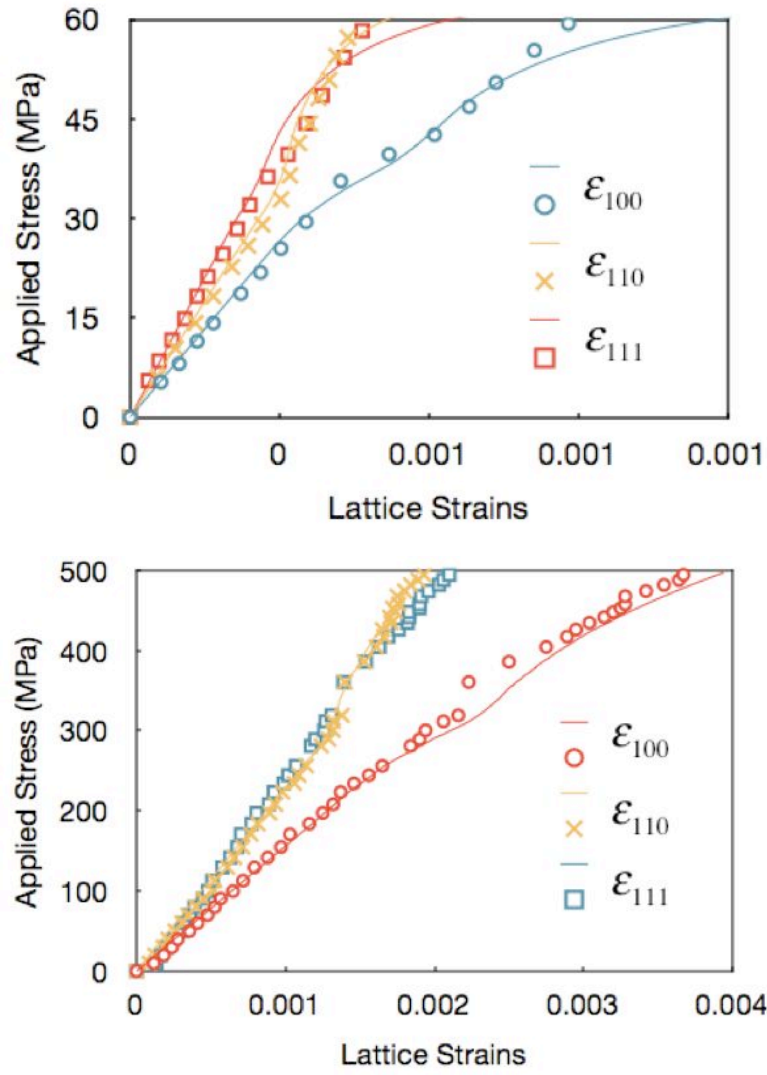


Figure 2.4. Lattice strain evolution under uniaxial loading (a) for copper polycrystal and the comparison with self-consistent model (solid lines) by Clausen et al. (1998); (b) for C-22HS polycrystal and the comparison with the neutron diffraction strain measurements (symbols) by Huang et al. (2008)

Chapter 3

Fatigue analysis in polycrystals

3.1. Continuum analysis of stress fields in the vicinity of crack tip

From the macroscopic point of view, the deformation field near a steady fatigue crack includes a plastic zone in front of the crack tip and a plastic wake behind it, where the cyclic loading and fatigue crack growth leading to a compressive residual strain. The magnitude, distribution, and history of the residual strain along the crack path depend on the stress multi-axiality, material properties, and history of stress intensity factor and crack growth rate. Since the fatigue process zone critically depends on this macroscopic residual strain history and the damage behavior on the microscopic grain level, an *in situ*, full-field, non-destructive measurement of lattice strain (which relies on the intergranular interactions of the inhomogeneous deformation fields in neighboring grains) by neutron diffraction techniques will reveal unprecedented information on the fatigue growth mechanics.

Fig. 3.1 is an idealized illustration of the important length scales involved for fracture analysis in a relatively ductile polycrystalline metal (van der Giessen and Needleman, 2003). The involved length scales include the macroscale to the atomic scale and a number of microstructure length scales in between. Based on the multiscale view of the crack tip plasticity as shown in Fig. 3.1, a numerical tractable formulation to simulate the plastic field of the crack tip is by the decoupling of scales including the continuum plasticity simulations of fatigue behavior and the crystal plasticity simulations of the microstructure and lattice strain. To this end, a two-scale

model is developed in this chapter to study the lattice strain evolution near fatigue crack tips in FCC polycrystalline materials. An irreversible, hysteretic cohesive interface model is adopted to simulate a steady fatigue crack, which allows us to generate the stress/strain distribution and history near the fatigue crack tip. Such a deformation history will be used as inputs for the micromechanical analysis of lattice strain evolution using the slip-based crystal plasticity model, thus making a mechanics connection between macro- and micro-strains. Experimental measurements were conducted using the neutron diffraction technique for the fatigue test of a Ni-based superalloy compact tension specimen.

A phenomenological cohesive interface model is developed in this work in order to realize a steady fatigue crack numerically and to determine the resulting stress multiaxiality distribution and history. A typical cohesive interface model is specified by a set of traction-separation constitutive laws (Needleman, 1987; Xu and Needleman, 1994). A reversible cohesive interface model will only lead to the crack resistance curve, but not propagate the crack. On the other hand, the addition of irreversibility will lead to plastic shakedown and the crack will stop growing after several cycles under constant amplitude K loads. Therefore, as suggested by Nguyen et al. (2001), an irreversible, hysteretic cohesive interface model is needed for fatigue crack simulation in which the hysteresis denotes the gradual damage of the interface strength and dissipation of fracture energy. Several variant models have been developed recently (Yang et al., 2001; Deshpande et al., 2001; Roe and Siegmund, 2003; Yang et al., 2004; Maiti and Geubelle, 2005). For instance, Yang et al. (2001) used different unloading and reloading paths in the traction-separation law. Deshpande et al. (2001) modeled irreversibility in the crack opening displacement due to the formation of an oxide layer. Roe and Siegmund (2003) proposed a cohesive

law to describe the energy dissipation processes during cyclic loadings by introducing a damage variable defined as the effective surface density of microdefects and by specifying the evolution of this variable. Our model in this paper resembles that in Nguyen et al. (2001) and Serebrinsky and Ortiz (2005). As will be discussed in Section 3.3, we carefully choose the cohesive interface properties so that the lattice strain profile outside the crack bridging zone will be insensitive to the choice of cohesive interface model.

The stress history from the macroscopic fatigue simulations will be used as inputs for the microscopic crystal plasticity simulations as discussed in the previous chapter. Clearly, our two-scale model (i.e., macroscopic fatigue simulation and microscopic lattice strain study) is only valid when a separation of length scales exists. The plastic zone sizes ($\sim 4\text{ mm}$) in our experiments are indeed much larger than grain size ($\sim 90\text{ }\mu\text{m}$). When these scales are not far apart, an explicit simulation of grain-level deformation fields in the presence of macroscopic strain gradients is needed as in, e.g., the fretting contact fatigue problem in polycrystals by Goh et al. (2001) in which only a few grains exist under the contact zone.

3.2. Fatigue and neutron diffraction experiments

Experimental measurements were conducted using the neutron diffraction technique for the fatigue test of a Ni-based superalloy compact tension (CT) specimen. Ni-based superalloys such as the HASTELLOY series have been widely used in aerospace applications such as the gas turbine engines because of their oxidation resistance and high temperature strength. Extensive studies have been performed on the low cycle fatigue and fatigue crack growth behavior of these types of alloys (Huang et al., 2010; Lee et al., 2011). In this work, fatigue tests and neutron

diffraction measurements were performed on an HASTELLOY C2000 alloy (56%Ni-23%Cr-16%Mo, in weight percent). This material has a single-phase face-centered-cubic (FCC) structure, yield strength of 393 MPa, ultimate tensile strength of 731 MPa, Young's modulus of 207 GPa, no preferred texture, and average grain size of about 90 μm .

The CT specimen, prepared according to the American Society for Testing and Materials (ASTM) Standards E647-99, has a notch length of 10.16 mm, a width of 50.8 mm, and a thickness of 6.35 mm as shown in Fig. 3.2. The crack growth experiments were conducted using a computer controlled Material Test System (MTS) servo-hydraulic machine. Prior to the crack growth tests, the CT specimens were pre-cracked to a crack length of 1.27 mm, and then the crack growth experiments were performed under a constant-load-range control mode with a frequency of 10 Hz and a load ratio R of 0.01 ($R = P_{\min}/P_{\max}$, where $P_{\min}$ and $P_{\max}$ are the applied minimum (89 N) and maximum (8,880 N) loads, respectively). The crack length was measured by a crack-opening-displacement (COD) gauge using the compliance method. The location of the crack tip was also confirmed using a scanning electron microscope (SEM). For the setup of neutron strain mapping, the crack tip location identified by SEM was marked on the surface of the sample, and the marker was tracked using a set of theodolites during neutron experiments. When the crack length reached 16 mm (already in the Paris power law regime), a single tensile overload ($P_{\text{overload}} = 1.5P_{\max}$) was applied, and then the constant-amplitude fatigue experiment was resumed.

In situ neutron diffraction experiments under the above-mentioned fatigue test were carried out using the time-of-flight (TOF) neutron diffractometer, ENGIN-X at

the ISIS facility, STFC Rutherford Appleton Laboratory, UK (Daymond and Priesmeyer, 2002). The specimen is aligned in a load frame with the loading axis oriented 45° relative to the incident neutron beam as shown in Fig. 3.2. The entire diffraction pattern is recorded in two stationary detector banks centered on diffraction angles of $2\theta = \pm 90^\circ$. Thus, the diffraction vectors are parallel to the in-plane (parallel to the loading direction) and through-thickness directions of the specimen. The through-thickness results are not discussed in this work and thus not depicted in Fig. 1.

The incident beam is defined by 2 mm horizontal and 1 mm vertical slits, and the diffracted beams are collimated using 2 mm radial collimators, resulting in the gauge volume shown in Fig. 3.2. The collected diffraction patterns were analyzed by both the Rietveld refinement method (Rietveld, 1969) and the single peak fitting method (Von Dreele et al., 1982) using the General Structure Analysis System (GSAS) (Larson and Von Dreele, 2004). The diffraction peaks are a result of the collective response of the grains whose $\{hkl\}$ crystalline planes are perpendicular to the diffraction vector. The Rietveld method fits to the entire diffraction pattern, thus giving an average and approximate measure of the macroscopic elastic strain as shown in Fig. 3.3(b). Fitting individual peaks gives the lattice strains as shown in Fig. 3.3(a), which result from the grain-level inhomogeneous deformation fields. The stress-free reference lattice parameters are measured away from the crack tip at a corner of each CT specimen. A total of 19 points are measured as a function of the distance from the crack tip along the direction of crack growth. Holding time during the neutron diffraction data acquisition period will not change the deformation behavior since creep is negligible.

The spatially resolved lattice strain distribution as a function of the distance from the crack tip is shown in Fig. 3.3 at $P_{\min}$ and P_{overload} . Data under $P_{\max}$ were not collected, but our previous measurements on 316 stainless steel suggest the lattice strain distributions under P_{overload} or $P_{\max}$ are very similar except for the magnitude difference (Barabash et al., 2008). The length is normalized by the plastic zone size at $P_{\max}$, which is about 4.3 mm as calculated by

$$r_p = \frac{1}{3\pi} \left(\frac{K_{\max}}{\sigma_y} \right)^2, \quad (3.1)$$

where $K_{\max}$ is the stress intensity factor at the maximum load and σ_y is the yield strength of the material. The calculation of the stress intensity factor for the CT specimen can be found in the ASTM standards. The lattice strain profile in Fig. 3.3(a) indicates that, for both the minimum load and overload, there is a cross-over between lattice strains ε_{100} and ε_{111} . At the minimum load, compressive lattice strains were observed in the plastic wake with the maximum absolute value of about 600 $\mu\epsilon$ (where $\mu\epsilon=10^{-6}$) at about $-0.2r_p$, while the maximum tensile strains located in the plastic zone are about 300 $\mu\epsilon$ at $0.8r_p$. At the overload, the largest tensile strain of about 1,980 $\mu\epsilon$ is observed for ε_{100} at about $0.1r_p$. Results obtained from Rietveld method are shown in Fig. 3.3(b), which approximately corresponds to the macroscopic strain component, $\varepsilon_{22}^{\text{elastic}}$, and displays a transition from compressive to tensile behavior as one traverses from the plastic wake to the crack front.

It should be noted that the measurement of macroscopic strains does not provide a direct reference to the microscopic fatigue mechanisms, while lattice strain mapping around the crack tip provides grain-level information at locations with

different fatigue and stress history. The Rietveld method fits the entire diffraction pattern by considering the crystallographic space group of the material. Two samples having the same macroscopic elastic strain, $\varepsilon_{22}^{elastic}$, may not have the same lattice strains, ε_{hkl} . Consequently, we need to know the history of macroscopic stress/strain fields as well as how such macroscopic deformation fields are accommodated on microscopic grain levels.

3.3. Continuum fatigue simulation based on the hysteretic, irreversible cohesive intergace model

The initiation and growth of fatigue crack are modeled by a top-down approach, including embedded cohesive zone and surrounding plastic deformation. The cohesive zone at the crack tip is modeled by constitutive equations relating the tractions acting on the two bonded solids to the separation between them. Since there is at present no fundamental basis for choosing the form of cohesive relation, the objective of this section is to find a cohesive interface model that can lead to the development of a steady fatigue crack so that the stress history near the fatigue tip can be faithfully reproduced. Numerical artifacts arising from this line of thought will be discussed shortly.

Following the pioneering work by Needleman (1987), a number of cohesive models have been proposed, in most of which the unloading from and subsequent reloading towards the monotonic envelope extends to the origin on the traction-separation plot (Camacho and Ortiz, 1996; de-Andrés et al., 1999). The mere introduction of this irreversibility, however, will eventually lead to plastic shakedown; the crack arrests after a finite number of cycles. Following Nguyen et al. (2001), the introduction of an irreversible cohesive law with unloading–reloading

hysteresis gives a phenomenological means to accumulate crack tip damage and thus to allow a steady crack growth and the development of a plastic wake when the applied stress intensity factor is in a range that is smaller than the intrinsic fracture toughness. Some early attempts on using this approach have been reported previously (Barabash et al., 2008).

As shown in Fig. 3.4(a), during monotonic loading, the traction-separation relationship is specified as

$$\frac{T_n}{\sigma_{\max}} = \frac{\Delta_n}{\delta_n} \exp\left(1 - \frac{\Delta_n}{\delta_n}\right), \quad (3.2)$$

where T_n and Δ_n represent the traction and separation in the cohesive interface model, respectively, $\sigma_{\max}$ is the interface strength, and δ_n is a characteristic length scale. The unloading-reloading hysteresis is introduced by considering the unloading and reloading stiffnesses separately, given by

$$\dot{T}_n = \begin{cases} K^- \dot{\Delta}_n, & \dot{\Delta}_n < 0 \\ K^+ \dot{\Delta}_n, & \dot{\Delta}_n > 0 \end{cases}, \quad (3.3)$$

where K^- and K^+ represent unloading stiffness and reloading stiffness, respectively. The above relationship is shown schematically in Fig. 3.4(a). The unloading stiffness, K^- , being a constant, is solely determined by the unloading point:

$$K^- = \frac{T_n^{\text{unload}}}{\Delta_n^{\text{unload}}}, \quad (3.4)$$

The reloading stiffness evolves during unloading and reloading processes according to

$$\dot{K}^+ = \begin{cases} -K^+ \frac{\dot{\Delta}_n}{\delta_f}, & \dot{\Delta}_n > 0 \\ \left(K^+ - K^-\right) \frac{\dot{\Delta}_n}{\delta_a}, & \dot{\Delta}_n < 0 \end{cases}, \quad (3.5)$$

where δ_f and δ_a are the length parameters characterizing the damage behavior. Clearly, during reloading, K^+ is being damaged with the associated length scale δ_f (empirically, $\delta_f \gg \delta_n$ in order to prevent rapid softening). During unloading, K^+ increases and gradually approaches K^- (empirically, $\delta_a \ll \delta_n$ in order to assure $K^+ \rightarrow K^-$ rapidly).

Treating the cohesive interface as an ad hoc continuum, we can write down the following principle of virtual work,

$$\int_V \sigma_{ij} \delta \varepsilon_{ij} dV + \int_{\Gamma_{int}} T_\alpha \delta \Delta_\alpha dA = \int_{\Gamma_{ext}} t_i^* \delta u_i dA \quad (3.6)$$

where t^* is the applied traction at the external surface Γ_{ext} . Such a formulation allows us to implement the cohesive constitutive law into the commercial finite element package, ABAQUS, through a user-defined element (UEL) subroutine. The cohesive parameters, σ_{max} , δ_n , δ_f , δ_a , and ζ_n , are passed from the input file to the UEL subroutine. Parameters including Δ_n^{unload} , T_n^{unload} , K^- , and K^+ are calculated and saved as the state variables in the UEL subroutine. The implicit integration scheme of these constitutive equations can be found in Section 3.4. Additionally, we note that the cohesive zone model can successfully simulate the growth of a long-pre-existing crack, but faces the snap-back instability problem in crack initiation. In an implicit finite element formulation (which uses Newton–Raphson iteration to solve the non-

linear equilibrium equations), the radius of convergence of the Newton–Raphson scheme reduces to zero at the point of instability. This convergence problem can be avoided by adding a term, $\sigma_{\max} \zeta_n \frac{d}{dt} \left(\frac{\Delta_n}{\delta_n} \right)$, to T_n as updated in the Appendix, where ζ_n is a viscosity-like parameter that governs the viscosity energy dissipation under normal loading (Gao and Bower, 2004; Xia et al., 2007). A detailed discussion on the appropriate choice of ζ_n can be found in these references. Here in order to minimize the additional rate dependence due to the viscous term, we choose $\zeta_n V / \delta_n \approx 10^{-3}$ where V is chosen as the magnitude of the material velocity at r_p .

The fatigue crack is simulated by the above irreversible, hysteretic cohesive interface model with the model configuration and representative mesh shown in Figs. 3.4(c) and 3.4(d). We need to ensure that there are at least four or five elements to resolve the crack bridging zone. The size of the semi-circular specimen is much larger than the plastic zone size, e.g., $100 r_p$, in order to ensure a valid K annulus. The K field is applied on the external boundary,

$$\begin{Bmatrix} u_1 \\ u_2 \end{Bmatrix} = \frac{K}{2E} \sqrt{\frac{r}{2\pi}} \begin{Bmatrix} (1+\nu)[(2\kappa-1)\cos(\theta/2) - \cos(3\theta/2)] \\ (1+\nu)[(2\kappa+1)\sin(\theta/2) - \sin(3\theta/2)] \end{Bmatrix}, \quad (3.7)$$

where $\kappa = 3 - 4\nu$, and r and θ are radial coordinates. The prescribed cohesive interface elements are embedded along the crack plane. The surrounding plastic field is governed by the continuum Mises plasticity following the stress-strain behavior shown in Fig. 3.4(b), which is extracted from the experimental results. The material constants obtained are $E = 207$ GPa, $\sigma_y = 393$ MPa, $\nu = 0.33$, and strain hardening exponent, $N = 0.05$. It is known that the hardening law used in the cyclic plasticity

model may dramatically change the residual stress development near the fatigue crack tip (Jiang et al., 2005), but for simplicity, we adopt the isotropic hardening plasticity model without any cyclic hardening/softening in this work.

The phenomenological cohesive interface model can faithfully reproduce a steady fatigue crack if

- (i) a plastic wake will emerge and be larger than the plastic zone size;
- (ii) the crack increment in each cycle is much smaller than the plastic zone and crack bridging zone;
- (iii) the crack bridging zone size is smaller than the plastic zone size.

Note that typically we have a crack bridging size of $r_{cz} \approx 0.2 \delta_n E / \sigma_{\max}$. Once these three criteria are met, the fatigue crack grows steadily and the strain distribution near the fatigue crack tip will be insensitive to the cohesive parameters. The most critical cohesive parameters are the interface strength $\sigma_{\max}$ and fracture energy Γ . As thoroughly studied by Tvergaard and Hutchinson (1992), a crack under monotonic loading shall not grow with a large $\sigma_{\max}$ and will grow too rapidly with a small $\sigma_{\max}$. A choice of $\sigma_{\max} \approx 3.0\sigma_y$ is preferred when $N = 0.05$. According to Eq. (3.5), we choose $\delta_f \gg \delta_n$ to prevent a rapid softening of K^+ , and, $\delta_a \ll \delta_n$ to ensure that K^+ quickly approaches K^- during unloading. Adjusting these parameters will change the crack growth rate. Note that the crack growth rate shall not affect our strain distributions as will be clear soon. Based on the above discussion, two groups of cohesive parameters are chosen as listed in Table 3.1. Next we discuss the effects of these parameters on the numerical results.

Contours of $\varepsilon_{22}^{elastic}$ and σ_{22} are plotted in Fig. 3.5 near the crack tip at the minimum load for the first group of cohesive parameters after a steady fatigue crack has been realized. The crack tip location is determined when the crack opening displacement reaches δ_n , which can be clearly seen from these plots. Since the plastic zone size will not be affected by the choice of cohesive parameters as long as the crack bridging zone size is much smaller than the plastic zone, using the second group of cohesive parameters in Table 1 will not change the distributions as observed in Fig. 3.5. Similar to the experimental observations, a compressive residual stress is developed in the region of $-\infty < x - x_{tip} < \sim 0.1r_p$, and a tensile residual stress is developed in the plastic zone. The tensile stress in front of the crack tip acts as the driving force for crack growth, while the compressive stress at behind – a crack closure effect – resists crack growth.

Although the crack tip process zone is treated phenomenologically by a cohesive interface model, the plastic process zone that is also responsible for fatigue crack growth is determined accurately. The stress history of an arbitrarily chosen point on the crack plane is plotted in Figs. 3.6 and 3.7 against the number of cycles. This arbitrary point is in fact a rectangle of 1 mm×2 mm, and the reported stress is averaged in this area (see the gauge volume in Fig. 1). As the crack propagates, the point of interest approaches the crack tip and coincides with the crack tip at a certain number of cycles as denoted by vertical dashed lines on these plots. The second group of parameters has larger interface strength, so it will take more cycles before the fatigue crack begins to grow. The crack growth rate can be estimated from the number of cycles between the maximum and minimum σ_{22} values in Figs. 3.6(b) and 3.7(b). It can be shown that the second group of cohesive parameters leads to a

smaller crack bridging zone size and a slightly larger fatigue crack growth rate than the first group does. Strain histories in Figs. 3.6 and 3.7 are almost identical if we change the abscissa to the distance of point of interest to the crack tip. In the early stage when the point of interest is far away from the crack tip, the elastic K field is reproduced. Near the crack tip, we will see a large degree of stress multiaxiality and particularly a large triaxial stress state at $\sim 0.2r_p$. All stress components become negative and reach asymptotic limits at $\sim r_p$ away from the crack tip and in the plastic wake.

3.4. Integration scheme for the irreversible, hysteretic cohesive interface model

In order to implement the irreversible, hysteretic cohesive interface model in Eqs. (3.2)-(3.5) into ABAQUS using the user-define element (UEL) subroutine, we need to determine the interface traction and state variables at $t + \Delta t$, as well as the material tangent, provided with the traction and state variables at t and the separation increment $\Delta_n^{t+\Delta t}$. Interface opening and closing need to be dealt with separately.

During crack opening (i.e., $\dot{\Delta}_n > 0$), the stiffness K^+ decreases, and a choice of $\delta_f \gg \delta_n$ will prevent a rapid softening. An implicit integration scheme is written as

$$T_n^{t+\Delta t} = T_n^t + K_{t+\Delta t}^+ (\Delta_n^{t+\Delta t} - \Delta_n^t), \quad (3.8)$$

and from Eq. (3.5),

$$K_{t+\Delta t}^+ = \frac{K_t^+}{1 + (\Delta_n^{t+\Delta t} - \Delta_n^t) / \delta_f}. \quad (3.9)$$

Differentiating the above two equations, we get

$$dT_n^{t+\Delta t} = (\Delta_n^{t+\Delta t} - \Delta_n^t) dK_{t+\Delta t}^+ + K_{t+\Delta t}^+ d\Delta_n^{t+\Delta t}, \quad (4.0)$$

$$(\delta_f + \Delta_n^{t+\Delta t} - \Delta_n^t) dK_{t+\Delta t}^+ + K_{t+\Delta t}^+ d\Delta_n^{t+\Delta t} = 0. \quad (4.1)$$

Consequently, the material tangent is given by

$$\left. \frac{\partial T_n^{t+\Delta t}}{\partial \Delta_n^{t+\Delta t}} \right|_{\Delta_n > 0} = K_{t+\Delta t}^+ \left[1 - \frac{\Delta_n^{t+\Delta t} - \Delta_n^t}{\delta_f + \Delta_n^{t+\Delta t} - \Delta_n^t} \right]. \quad (4.2)$$

During crack closing (i.e., $\dot{\Delta}_n < 0$), the stiffness K^+ gradually approaches K^- , and a choice of $\delta_a \ll \delta_n$ will rapidly bring K^+ to K^- . Since K^- in Eq. (3.4) does not change, we write

$$T_n^{t+\Delta t} = T_n^t + K^- (\Delta_n^{t+\Delta t} - \Delta_n^t), \quad (4.3)$$

so that the material tangent is simply

$$\left. \frac{\partial T_n^{t+\Delta t}}{\partial \Delta_n^{t+\Delta t}} \right|_{\dot{\Delta}_n < 0} = K^-. \quad (4.4)$$

The update of the stiffness K^+ can be given by an implicit scheme for Eq. (3.5):

$$K_{t+\Delta t}^+ = \frac{K_t^+ \delta_a - K^- (\Delta_n^{t+\Delta t} - \Delta_n^t)}{\delta_a - (\Delta_n^{t+\Delta t} - \Delta_n^t)}. \quad (4.5)$$

3.5. Lattice strain distribution near fatigue cracks without consideration of the intergranular damage

Another part of the two-scale model is the crystal plasticity model as we described in the previous chapter. Material parameters involved in the crystal plasticity theory, as given in Table 3.2, are calibrated by comparing the predicted and measured lattice strains in a full loading cycle on the HASTELLOY C2000, as shown in Fig. 3.8(a). Elastic constants, c_{11} , c_{12} and c_{44} , are accurately determined from a least squares fitting of the predicted to the measured lattice strains in the elastic stage (Thomas et al., 1998). Simulations in Fig. 3.8(a) indicate that after a full load cycle, ε_{100} becomes negative and the other two are small and positive. The lattice strain splitting will lead to an accurate choice of the initial slip strength, while the hardening moduli cannot be determined accurately since the applied stress is not far beyond the yield stress in our experimental observations. Additionally, the residual lattice strains may be sensitive to the choice of cyclic hardening laws, but this line has not been explored in the present study. The sign and magnitude of the residual lattice strain after a full load cycle are significantly affected by the stress multi-axiality. In Fig. 3.8(b), a triaxial stress state of $\sigma_{11} = \sigma_{33} = 0.4\sigma_{22}$, as suggested from the stress state in Figs. 3.6 and 3.7, is applied and the results are different from that in Fig. 3.8(a). For a comparison purpose, elastic simulations under uniaxial loading are conducted and results in Fig. 3.8(c) display the elastic anisotropy. These results are intended to mimic the weak-grain-boundary result, as will be discussed in the following.

Combining the stress histories in Figs. 3.6 and 3.7 and the crystal plasticity simulations in Figs. 3.8, we can now generate the lattice strain distributions near a fatigue crack tip. Results are given in Figs. 3.9 and 3.10 for the first and second group

of cohesive parameters in Table 3.1, respectively. Lattice strains are plotted against the distance from the crack tip normalized by the plastic zone size, being about 1.25 mm in this case with the maximum applied stress intensity factor of $K_{\max}=34.8 \text{ MPa}\sqrt{m}$. Results in Figs. 3.9 and 3.10 are almost the same, confirming the notion that the calculated lattice strain distributions are insensitive to the choice of cohesive parameters, if the crack bridging zone is considerably smaller than the plastic zone. Similar to the experimental observation in Fig. 3.3, $\epsilon_{100} > \epsilon_{111}$ ahead of the crack tip, which can be explained by the corresponding positive stress state and the lattice strain evolution suggested in Figs. 3.8(a) and 3.8(b). In the plastic wake, $\epsilon_{100} < \epsilon_{111}$ because of the corresponding compressive stress state. A hindsight is that the present stress state alone cannot determine a correct lattice strain distribution – the entire stress history (or equivalently, strain path) is needed because of the inelastic deformation near the crack tip. However a further comparison between the predicted and measured lattice strain distributions finds a disagreement on the cross-over location (i.e., where ϵ_{100} and ϵ_{111} are about the same). The cross over occurs at $\sim -0.1r_p$ at maximum load and at $\sim 0.1r_p$ at minimum load, while these locations in simulation results are translated by an amount of about $0.2r_p$ to the plastic wake. It is believed that this feature is not an artifact of our numerical simulation since the crack bridging zone is intentionally kept small and simulations using two different groups of cohesive parameters show the same observations. In other words, the cohesive interface model is merely a numerical tool to realize fatigue crack growth. Therefore, the discrepancy suggests a fatigue mechanism within $(-0.1r_p, 0.1r_p)$ which is not captured by the plastic process zone. We will fully discuss about this discrepancy in the next chapter.

3.6. Summary

The ability to quantitatively predict the interplay between plasticity and damage evolution at microscopic length scales is a critical step towards developing mechanism-based models for deformation and failure properties of advanced structural materials. Deformation behavior near a fatigue crack tip is studied experimentally by spatially resolved, *in situ* measurements of lattice strains using neutron diffraction technique for HASTELLOY C2000 alloy, as well as computationally by macroscopic fatigue simulation based on a hysteretic, irreversible cohesive interface model and by microscopic crystal plasticity simulation. The stress multiaxiality history near fatigue cracks as calculated from macroscopic simulations are employed as inputs for the microscopic simulations, thus enabling a quantitative investigation of the development of grain-level deformation fields in the vicinity of a fatigue crack tip.

- From a multiscale point of view, the deformation behavior near fracture or fatigue crack tips consists of a well-defined K field, a plastic zone and wake, and a “messy” tip process zone. Our cohesive interface model does not really capture the exact physical processes in the tip process zone, but it allows us to develop a steady fatigue crack so that the role of the “clean” surrounding plastic zone/wake can be quantified.
- An explicit simulation of a polycrystal aggregate using the crystal plasticity model conveniently predicts the lattice strain evolution as a function of applied stress, microstructure, and material properties. The microstructure-based simulation method is more convenient than the self-consistent model especially in studying the roles of nonconventional deformation mechanisms such as grain boundary plasticity.

- Lattice strain distributions in the vicinity of the fatigue crack tip are very sensitive to the history of stress and its multiaxiality. The stress state can be varied by the load pattern (e.g., mode mixity, overload, etc.) and material properties (e.g., cyclic hardening behavior). The lattice strains are largely dependent on the slip system and material microstructure. Therefore, neutron or synchrotron x-ray diffraction studies near fatigue crack tips can lead to salient understandings of material deformation behavior.
- Predictions from perfect grain-boundary simulations exhibit the same lattice strain distributions as in neutron diffraction measurements, except for discrepancies near the crack tip within about one tenth of the plastic zone size. Specifically, the $\{100\}$ lattice strain is generally larger (smaller) than the $\{111\}$ lattice strain in front of (behind) the crack tip in simulations. The cross-over, however, occurs on or ahead of the crack tip in experiments, rather than in the plastic wake observed in simulations, which is regarded caused by other mechanisms to be discussed in Chapter 4.

Table 3.1 Two groups of cohesive interface parameters in the fatigue crack simulation.

Group	$\sigma_{\max}$ (MPa)	δ_n (μm)	δ_f (μm)	δ_a (μm)	r_b (mm)	r_p (mm)	Γ (J/m ²)
#1	980	0.9	22.5	0.09	0.04	1.25	440
#2	1,225	0.72	18.0	0.072	0.024	1.25	440

Table 3.2 Material parameters used in the crystal plasticity model for HASTELLOY
C2000.

	c_{11} (GPa)	c_{12} (GPa)	c_{44} (GPa)	n	h_0 (MPa)	τ_0 (MPa)	τ_s (MPa)	q
C2000 alloy	254.4	177.0	120.5	50	550	110	750	1.0

Table 3.3 Directional modulus and strength-to-stiffness ratio for HASTELLOY
C2000.

	E_{100} (GPa)	E_{110} (GPa)	E_{111} (GPa)	r_s^{110} / r_s^{100}	r_s^{111} / r_s^{100}
C2000 alloy	109.2	209.4	301.7	0.52	0.54

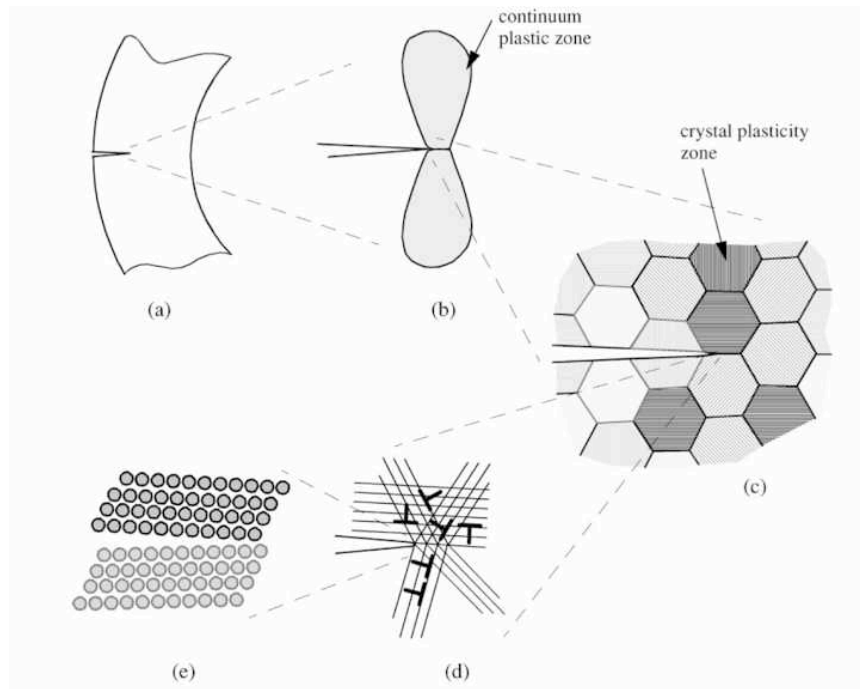


Figure 3.1. Various relevant scales that may determine the response of a crack in a macroscopic component. (a) The component scale; (b) the plastic zone governed by macroscopic continuum plastic flow; (c) the grain scale in a polycrystalline metal; (d) the scale of discrete slip planes and of individual dislocations; and (e) the atomic scale (van der Giessen and Needleman, 2002).

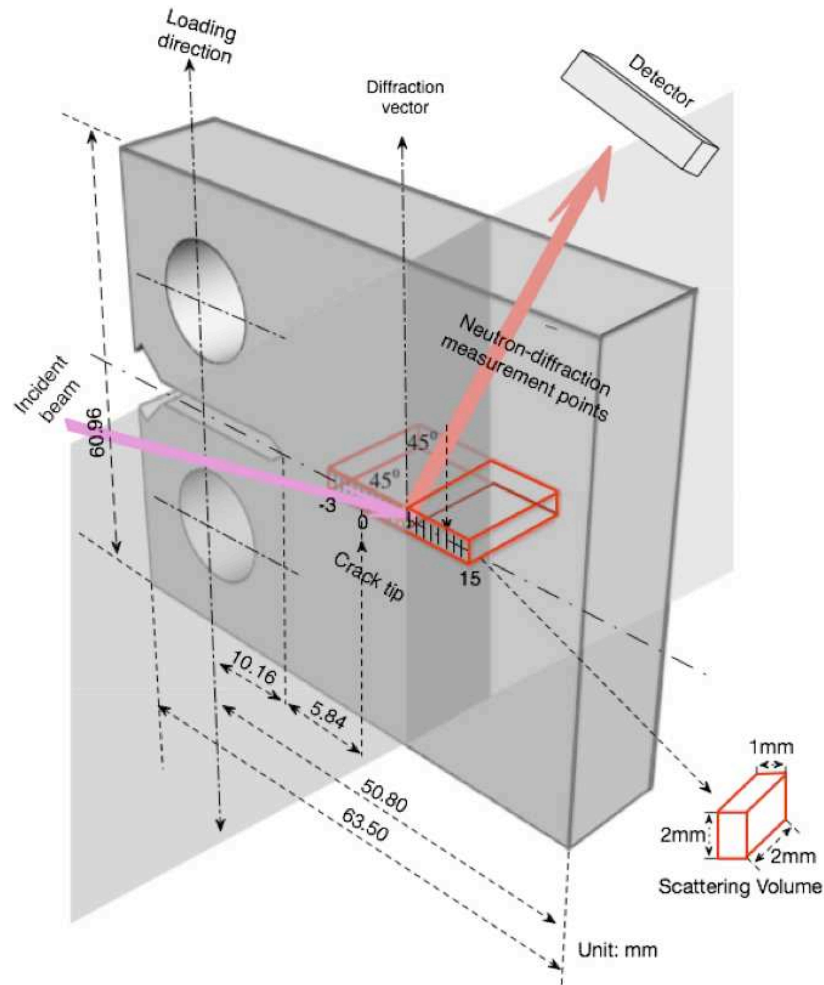


Figure 3.2. Schematic illustration of the compact tension (CT) specimen for HASTELLOY C-2000 (56%Ni-23%Cr-16%Mo, in weight percent) and the neutron diffraction experiment. The incident neutron beam is lying in the plane perpendicular to the specimen plane and at 45° to the loading direction. Thus the diffraction vector is parallel to the loading direction.

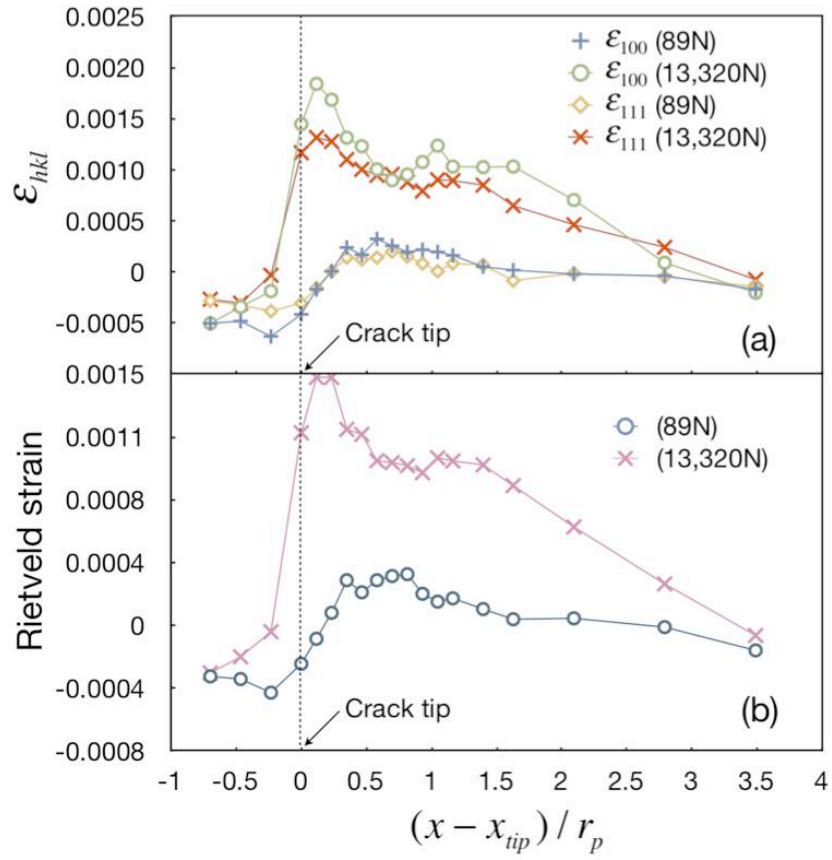


Figure 3.3. (a) Lattice strains, ϵ_{100} and ϵ_{111} , and (b) Rietveld strain (approximately $\epsilon_{22}^{elastic}$) are plotted against the distance along the crack plane, as normalized by the plastic zone size r_p . The top curves are measured at the overload and the bottom ones at the minimum load.

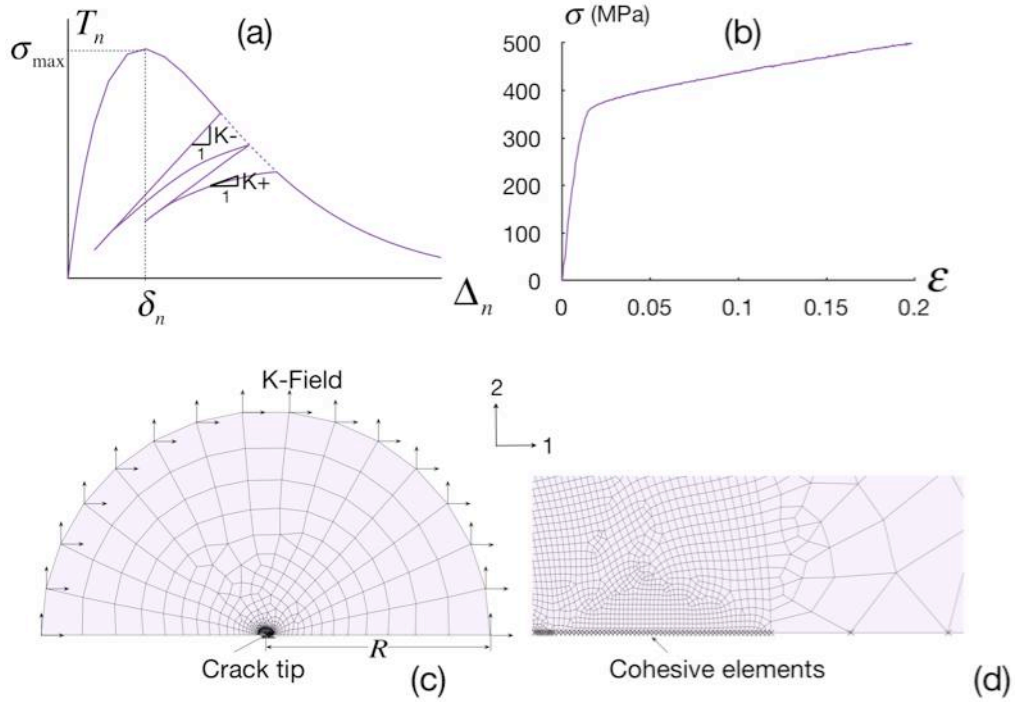


Figure 3.4. (a) The traction-separation ($T_n \sim \Delta_n$) relationship in the irreversible, hysteretic cohesive interface model. The unloading process corresponds to a straight line towards the coordinate origin, and the loading process exhibits a gradual softening behavior. (b) The measured stress-strain curve for HASTELLOY C2000. (c) A semi-circular model with a K field specified at the far away boundary and with cohesive elements along the crack plane. (d) Close-up view of the cohesive elements on the crack path.

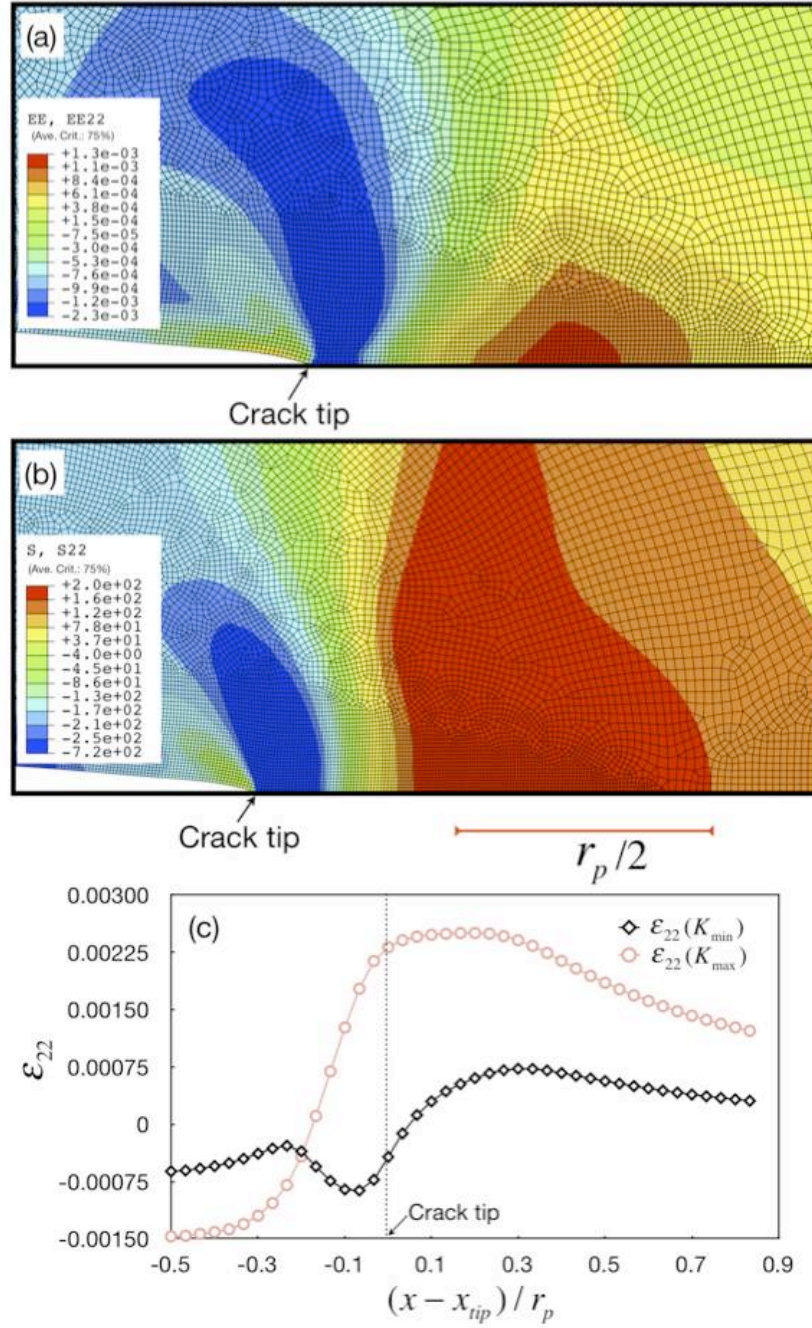


Figure 3.5. Simulated distributions of (a) $\epsilon_{22}^{elastic}$ and (b) σ_{22} near the crack tip when a steady fatigue crack is developed ($N=60$ for this case). The elastic strain exhibits a compressive-to-tensile transition as one traverses from the plastic wake to the crack front.

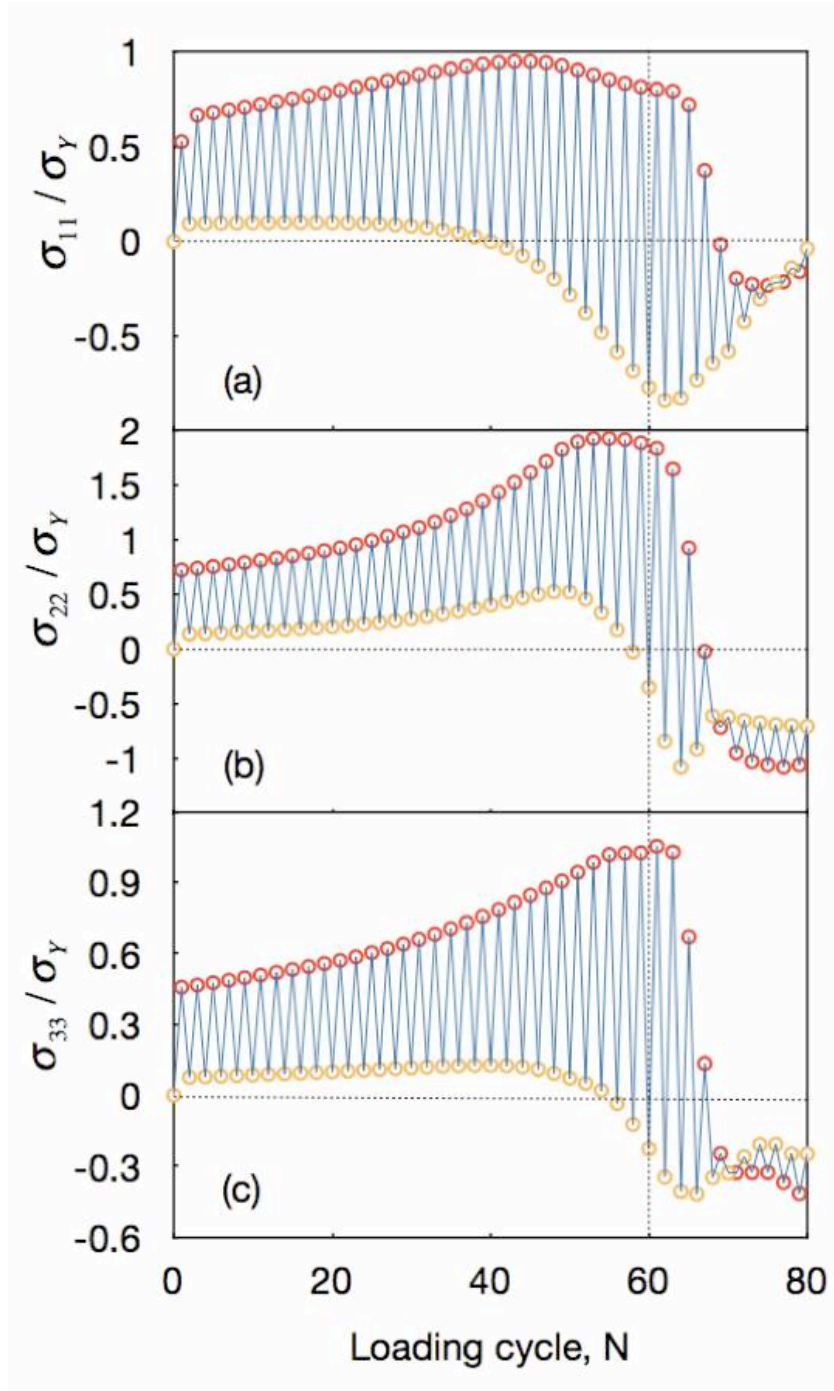


Figure 3.6. Multiaxial stress history as a function of the number of fatigue cycles for the first group of parameters in Table 1. These results correspond to the average stress in a $2\text{mm} \times 1\text{mm}$ rectangle with center located at a certain position on the crack plane. When $N=60$ (i.e., the dashed vertical line), the point of interest corresponds to the crack tip in Fig. 3.4.

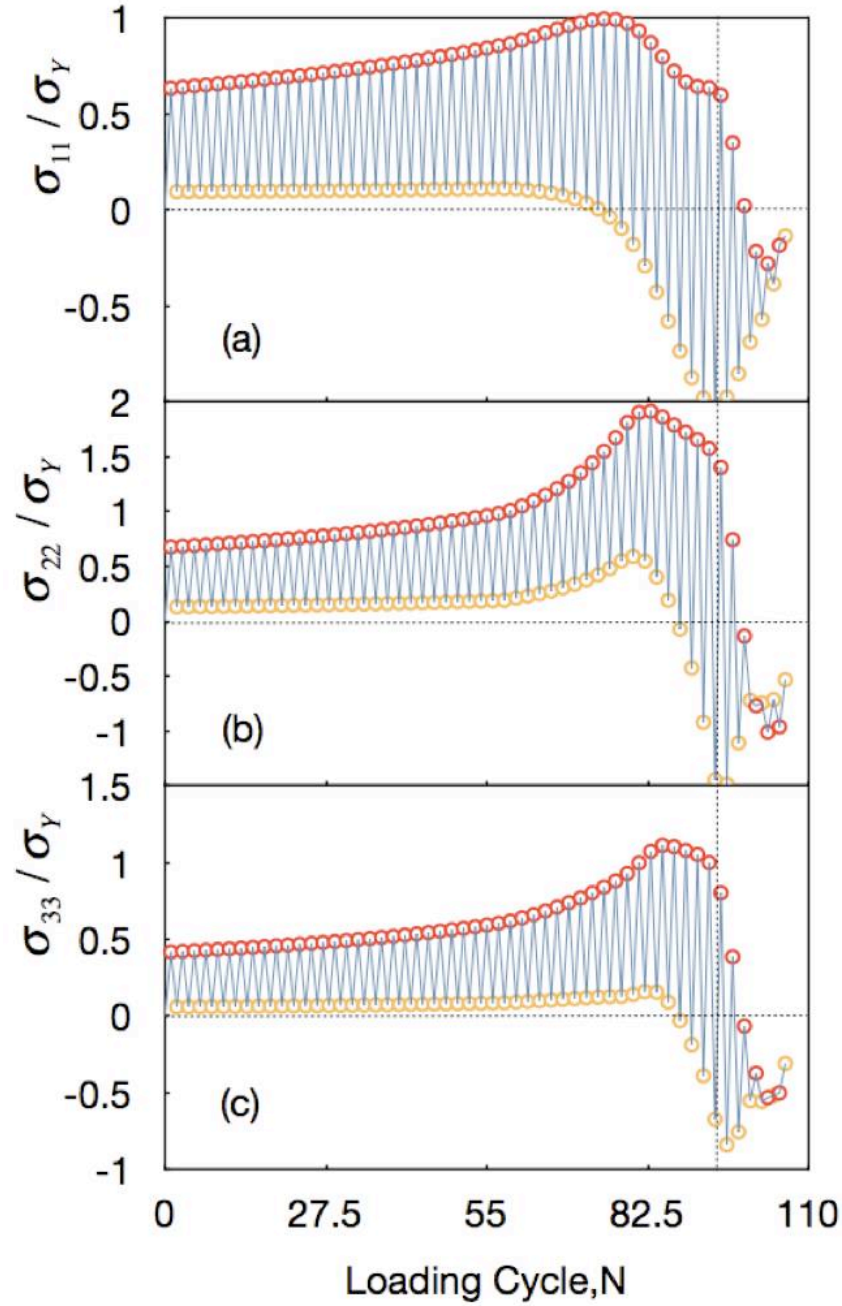


Figure 3.7. Multiaxial stress history as a function of the number of fatigue cycles for the second group of parameters in Table 1. These results correspond to averaged stress in a $2mm \times 1mm$ rectangle with center located at a certain position on the crack plane. When $N=90$ (i.e., the dashed vertical line), the point of interest corresponds to the crack tip.

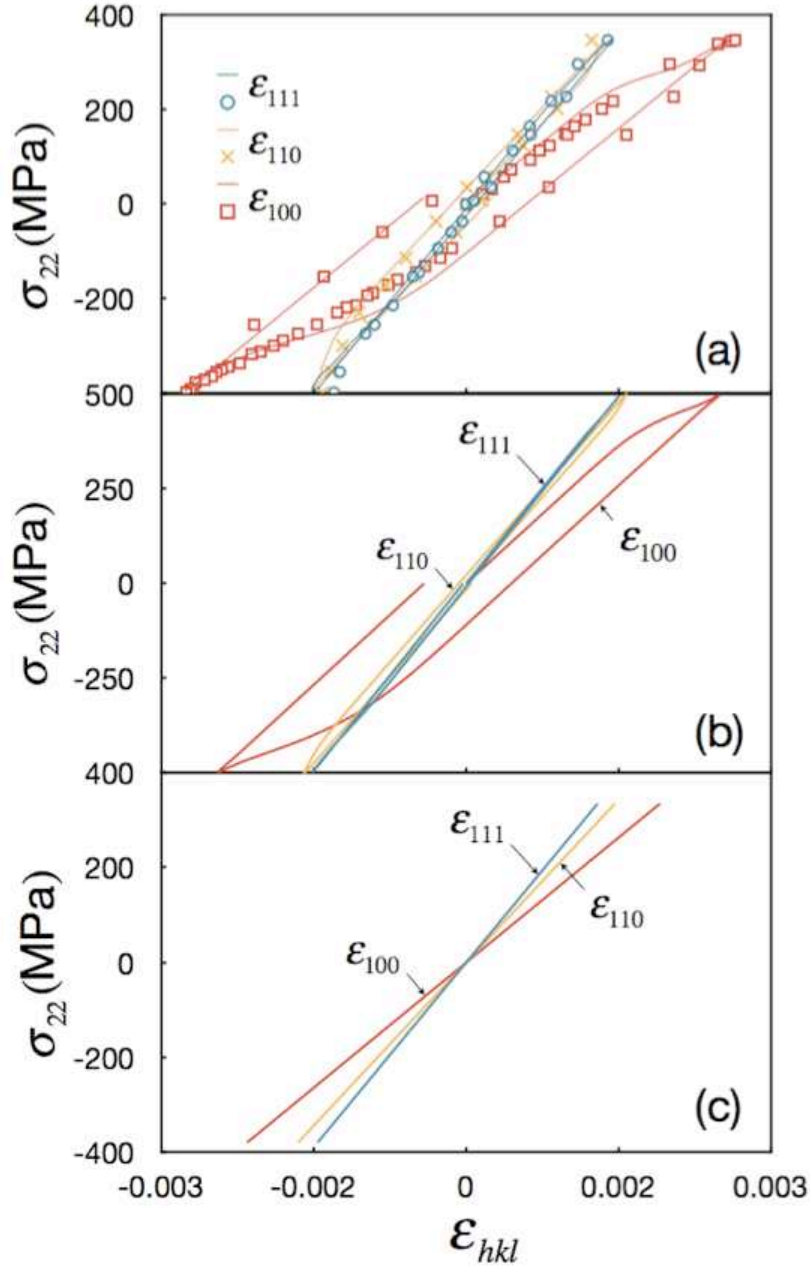


Figure 3.8. Lattice strain evolutions in the model polycrystal under various multiaxial stress states. (a) $\sigma_{11} = \sigma_{33} = 0$. Discrete markers are neutron diffraction measurements for HASTELLOY C2000 under a full cycle of fatigue loading. ϵ_{100} gives the largest absolute value due to the largest strength-to-stiffness ratio in $\langle 100 \rangle$. (b) $\sigma_{11} = \sigma_{33} = 0.4\sigma_{22}$. (c) Lattice strain evolution due to elastic anisotropy (under $\sigma_{11} = \sigma_{33} = 0$ condition) when slip plasticity is turned off.

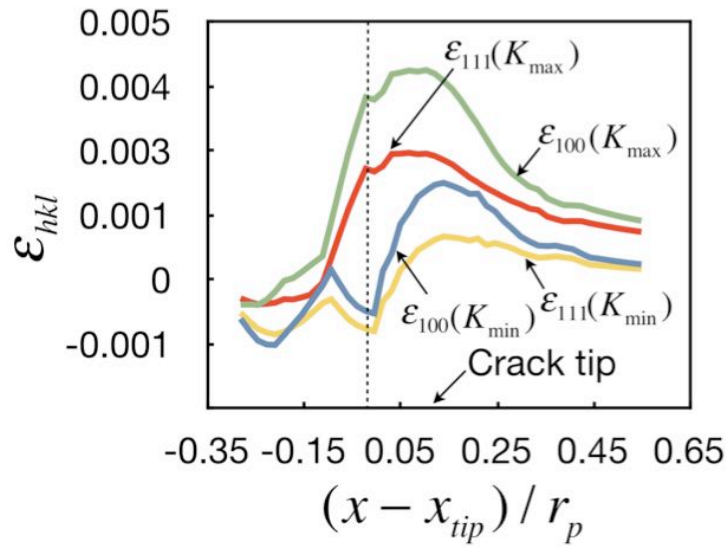


Figure 3.9. Predicted lattice strain profiles along the crack plane using the first group of cohesive parameters in Table 3.1. Crystal plasticity simulations with perfect grain boundaries.

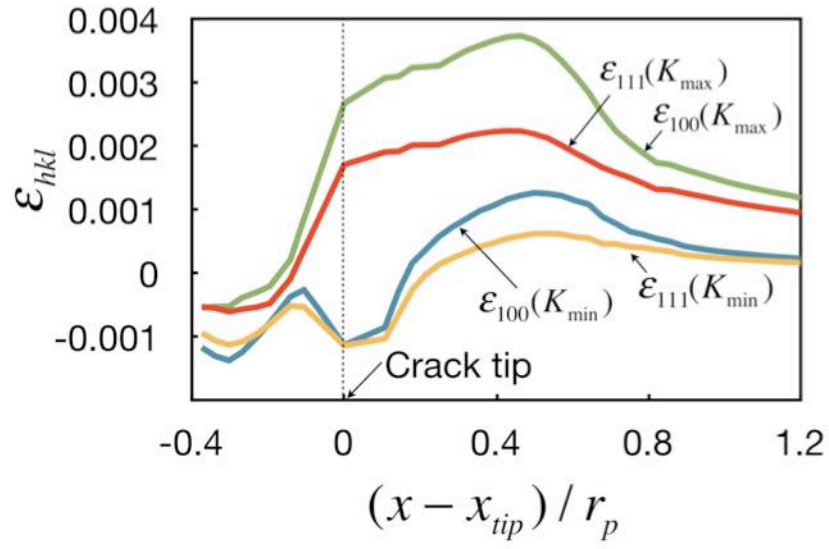


Figure 3.10. Predicted lattice strain profiles along the crack plane using the second group of cohesive parameters in Table 3.1. Crystal plasticity simulations with perfect grain boundaries.

Chapter 4

Effects of intergranular damage

4.1. Grain-orientation-dependent intergranular stress

Following the fatigue crack analysis in Chapter 3, the crossover locations on lattice strain curves from our simulation results are different from those in the experimental results. We believe that it is not due to the artifact of the model. According to the existing documents about the fatigue crack which indicate that intergranular damage happens when the microcracks start to initiate, we will further explore the correlation between the intergranular damage and the fatigue crack growth in this chapter. One of the indirect evidence of the intergranular damage can be found in Wang et al. (2003), which correlates the intergranular damage to the grain orientation dependent residual lattice strain anisotropy. Thus in this chapter, the grain-orientation-dependent residual lattice strain will first be analyzed.

As we discussed before, the intergranular strain arises to accommodate the deformation incompatibility of neighboring grains caused by distinctive Schmid factor of each single grain. Residual intergranular strain was believed to be related to the crack initiation (Guiu et al., 1982; Ward-Close et al., 1989). On the other hand, similar to the neutron diffraction experiment, the finite element modeling and the self-consistent modeling are both devoted to simulate the residual strain and stress at the grain size level and hence to compare with the neutron diffraction measurement for a better understanding of the essence of the micromechanical mechanisms.

The development of the lattice strains perpendicular or parallel to the tensile axis during uniaxial loading for selected families of grains with specific crystallographic orientations has been intensively studied by the above simulation methods for FCC polycrystalline material (Clausen and Lorentzen, 1997; Clausen et al., 1998; Dawson, 2000; Dawson et al., 2000), as well as in Chapter 2. However only few models can be found that deal with the anisotropy of the grain-orientation-dependent lattice strain because of the complexity due to the gradual change of the angle between the diffraction vector and the loading direction. In this chapter we construct a tetragonal specimen with cubic grains with each grain assigned a set of crystal plasticity parameters and randomly generated orientation. Following the procedure of crystal plasticity calculation introduced in the previous chapters, we plot the simulation results as well as the neutron diffraction results for FCC Steel polycrystal as shown in Fig. 4.1. The residual intergranular strains are plotted as a function of the tilt angle θ with representing (a) the VULCAN results and (b) the simulation results. By comparing residual lattice strain ϵ_{100} , ϵ_{110} and ϵ_{111} from both the two methods, nearly the same trends of the intergranular strains on the three crystallographic planes are achieved. During the change of θ , residual lattice strain ϵ_{100} changes its sign from negative to positive and to negative again and its curve exhibits an approximately symmetric shape with respect to $\theta=45^\circ$. Residual lattice strains ϵ_{110} and ϵ_{111} both resemble ϵ_{100} , possessing the symmetric shape except for the blurry due to the small values. The reason that they behave such an orientation dependent property will be discussed with details later. The model construction is shown in Fig. 4.2 (a) by the stress contours. The material constants chosen from the FCC stainless steel are: $C_{11}=204.6\text{GPa}$, $C_{12}=137.7\text{GPa}$, $C_{44}=126.2\text{GPa}$, $n=10$, $\gamma_0=0.001$, $h_0=205\text{MPa}$, $\tau_0=87\text{MPa}$, $\tau_s=140\text{MPa}$ and $q=1.0$. The lengths of the

specimen we construct are $a_3=4a_1=4a_2=4a$, where 3-direction is the loading direction, and the top surface is the loading surface. The boundary conditions we apply here are: $u_3=0$ on the bottom surface and $u_3=0.005a$ on the top surface, as shown in Fig. 4.1. Figs. 2 (b) to (h) represent stress contour with different grains selected according to the diffraction condition when calculating lattice strains for different crystallographic plains. Approximately 2.5% to 4.5 % grains are selected in these cases.

In Fig. 4.1, the residual lattice strain anisotropy arises largely after a full cycle, which, similar to the discussion in Chapter 2, is believed to be caused by the crystallographic orientation dependent strength to stiffness ratio, which is defined as $r_s = \sigma_{\langle hkl \rangle} / E_{\langle hkl \rangle}$ and is introduced to describe the lattice strain anisotropy after yielding, where $\sigma_{\langle hkl \rangle}$ and $E_{\langle hkl \rangle}$ denote the yield strength and the elastic modulus along the crystallographic direction of the $\{hkl\}$ plane normal. After yielding, the splitting behavior in the stress versus lattice strain curves is caused by the different yielding point of the $\{hkl\}$ plane, which is determined by the crystallographic orientation dependent r_s . The lower the r_s value, the earlier yielding occurs, which is corresponding to the “go up” behavior of the curve due to the lattice strain ceases increasing after yielding and hence leading to a positive residual lattice strain after a full cycle. Similarly, the one with the higher r_s value will end up with a negative residual lattice strain after a full cycle. The above theory assists to understand the anisotropy in Fig. 4.1.

4.2. Taylor analysis

To better explain the residual lattice strain anisotropy, we construct a Taylor model, which indicates that grain-scale mechanisms, such as residual intergranular strains, are essentially governed by the strain fields that must be compatible over

grain scales (Raabe et al., 2001). Two cuboid single grain specimens are established to calculate lattice strains ϵ_{100} and ϵ_{110} . For each specimen, the orientation is set to ensure that $\{100\}$ or $\{110\}$ plane normal is along the direction of the diffraction vector, which rotates from the loading direction gradually by angle θ . The macroscopic strain histories obtained from the previous model in Fig. 4.2 are used as inputs for the single-grain Taylor model. The residual lattice strains obtained by this method are shown in Fig. 4.3 as a function of θ . Due to the uncertainty of the orientations of the other two directions perpendicular to $\{100\}$ or $\{110\}$ plane normal, we randomly choose 10 cases and the lattice strains are calculated by taking the average. In Fig. 4.3, when $\theta=0^\circ$, ϵ_{100} yields later and exhibits the positive residual lattice strain but ϵ_{110} has a negative value. When $\theta=10^\circ$, both ϵ_{100} and ϵ_{110} keep the same signs with those when $\theta=0^\circ$ but with a slight decreasing of the absolute value. When $\theta=40^\circ$, both ϵ_{100} and ϵ_{110} change their signs and ϵ_{100} exhibits the positive value while ϵ_{110} exhibits the negative value. The above behaviors from the Taylor model agree well with the results from Fig. 4.1.

Residual lattice strains are calculated for several more crystallographic orientations to construct the inverse pole figure in Fig. 4.4 (a). Comparing to the literature measurement (Wang et al., 2003), our lattice strain contour distribution is very similar to their residual-stress inverse pole figure from the neutron experiments. Grain boundary voids are often found to be nucleated at impinging slip traces or steps (Lim and Raj, 1984). As a consequence, an indirect measure of void growth rate is the cumulative shear strain rate. The distribution of cumulative shear strain along loading direction is shown in Fig. 4.4 (b) in the form of inverse pole figure for various grain orientations. The maximum value was observed to occur on somewhere between

direction of $\langle 110 \rangle$ and $\langle 111 \rangle$, while the minimum was on direction $\langle 100 \rangle$. This suggests that the residual lattice strain for $\langle 100 \rangle$ vanishes in a smaller rate than that for $\langle 110 \rangle$ or $\langle 111 \rangle$. The same tendency was reported by Wang et al. (2003), where residual lattice strain ϵ_{100} decreases 30% while ϵ_{110} decreases 50% when intergranular damage happens. Hence it is believed that the intergranular damage happens firstly on the largest cumulative plastic shear strain rate location.

4.3. Intergranular damage involved lattice strain prediction around fatigue crack tip

As the discussion in Chapter 3, we mention that the cross-over discrepancy is not an artifact of our numerical simulation since the crack bridging zone is intentionally kept small and simulations using two different groups of cohesive parameters show the same observations. In other words, the cohesive interface model is merely a numerical tool to realize fatigue crack growth. Therefore, the discrepancy suggests a fatigue mechanism within $(-0.1r_p, 0.1r_p)$ which is not captured by the plastic process zone. Based on the analysis in this chapter, we believe that the discrepancy is due to the intergranular damage, which is not considered by the perfect grain boundary simulation in Chapter 3.

Analysis in the preceding section helps understand the connection between lattice strain and fatigue damage mechanisms. The absolute values of residual lattice strains after full load cycles are found to decrease with the increase of fatigue cycles under uniaxial loading condition and load ratio of $R = -1$. These residual lattice strains at the first full cycle can be faithfully reproduced by our crystal plasticity simulations (e.g., results in Fig. 3.7(a)). But the gradual decrease of residual lattice strain is out of the question in our perfect-grain-boundary simulations. These

experiments further discover the correlation between the vanishing residual lattice strain and the appearance of intergranular cracks, suggesting the intergranular damage be the fatigue mechanism. In the context of our fatigue crack growth simulations, to incorporate the above mechanism into our simulations in Figs. 3.8 and 3.9, we use the elastic polycrystal model in Fig. 3.7(c), since the simulation results also exhibit vanishing residual lattice strains which are similar to the observation of vanishing residual lattice strains as intergranular damage proceeds. Results are given in Figs. 4.5 and 4.6 for the first and second group of cohesive parameters in Table 3.1, respectively. The cross-over locations are now shifted to the locations that have been observed experimentally in Fig. 3.2.

The role of intergranular damage can be further supported by previous synchrotron X-ray microbeam studies of a fatigue crack in a similar Ni-based HYSATELLOY superalloy (Barabash et al., 2008). In the plastic wake, numerous small voids and small cracks running approximately perpendicular to the main crack direction were observed in a width of roughly the grain size. Diffraction studies in this regime showed significant streaking on the Laue pattern, which indicate large strain gradients and large densities of geometrically necessary dislocations (Barabash et al., 2010). These findings suggest that severe intragranular deformation (Type-III strain) is a precursor of the intergranular damage.

Damage initiation along the grain boundaries are often observed in fatigue crack growth tests of polycrystals. For example, at low to intermediate plastic strain amplitude, the impingement of persistent slip bands at grain boundaries may initiate micro-cracks (Suresh, 1998). If the intergranular damage and the residual lattice strain are mechanistically related, the lattice strain evolution should display a strong orientation dependence since the intergranular damage is often caused by the plastic

incompatibility at highly misoriented grain boundaries. An intragranular strain analysis based on synchrotron X-ray diffraction studies is better suited along these lines because of the gauge volume size limitation in neutron beam. From the modeling point of view, an explicit coupling between grain-interior crystal plasticity and grain-boundary damage model is needed, which is beyond the scope of this work.

4.4. Summary

In summary, we have developed a microscopic study, which is based on the crystal plasticity theory, to elucidate the grain-orientation-dependent lattice strain anisotropy of FCC structural superalloys during cyclic loadings. Stimulated by the previous work, the grain-orientation-dependent residual lattice strain anisotropy is important to the failure process during fatigue loading by neutron diffraction method. In this work, from the simulation point of view, we give a theoretical explanation about the anisotropy property indicating that it is determined by the strength-to-stiffness ratio of the specific crystallographic plane being considered.

In comparison to the neutron diffraction experimental result, the crystal plasticity model agrees very well with it. Besides, the Taylor model in this study allows us to further explore that the crystallographic plane with higher residual intergranular strain rate is weaker than others.

By taking consideration of the intergranular damage, which leads to vanishing intergranular strains as damage proceeds, we find a significantly improved agreement between predicted and measured lattice strains inside the fatigue process zone. Consequently, our synergistic modeling/experimental study suggests that the intergranular damage near the fatigue crack tip where a large degree of stress multiaxiality is present be responsible for fatigue crack growth.

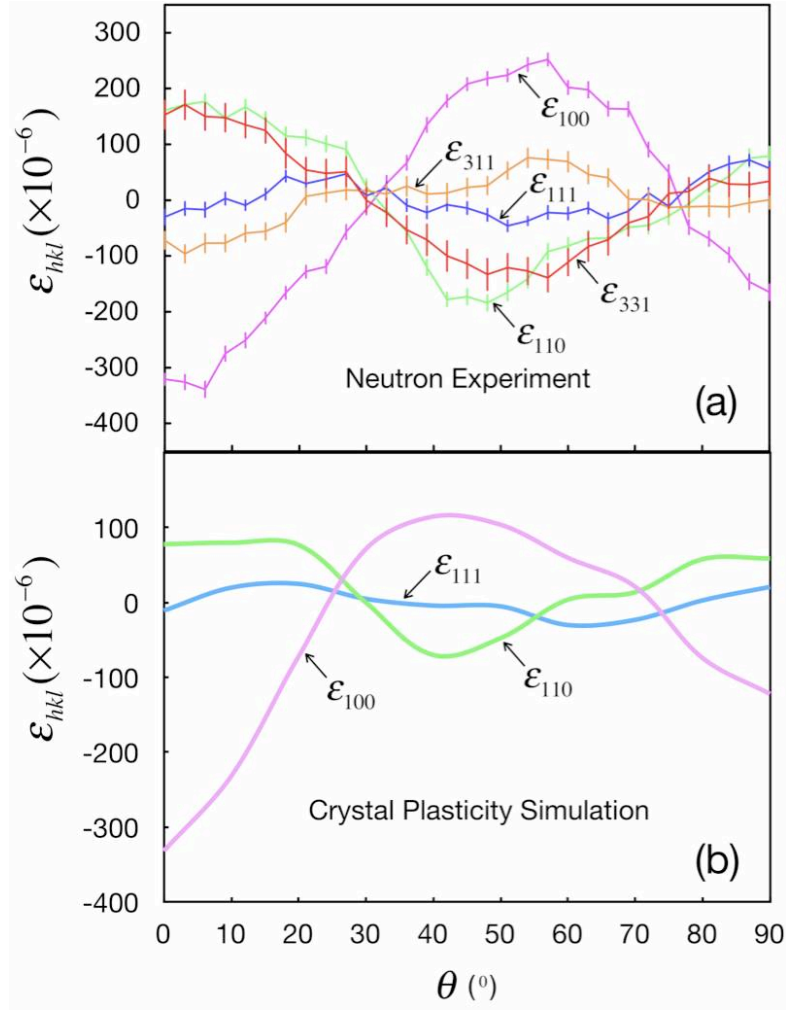


Figure 4.1. Residual lattice strains at the end of a fully reversed loading cycle with respect to the angle θ formed between the uniaxial loading axis and the diffraction vector for FCC stainless steel: (a) neutron diffraction experiments; (b) crystal plasticity simulations.

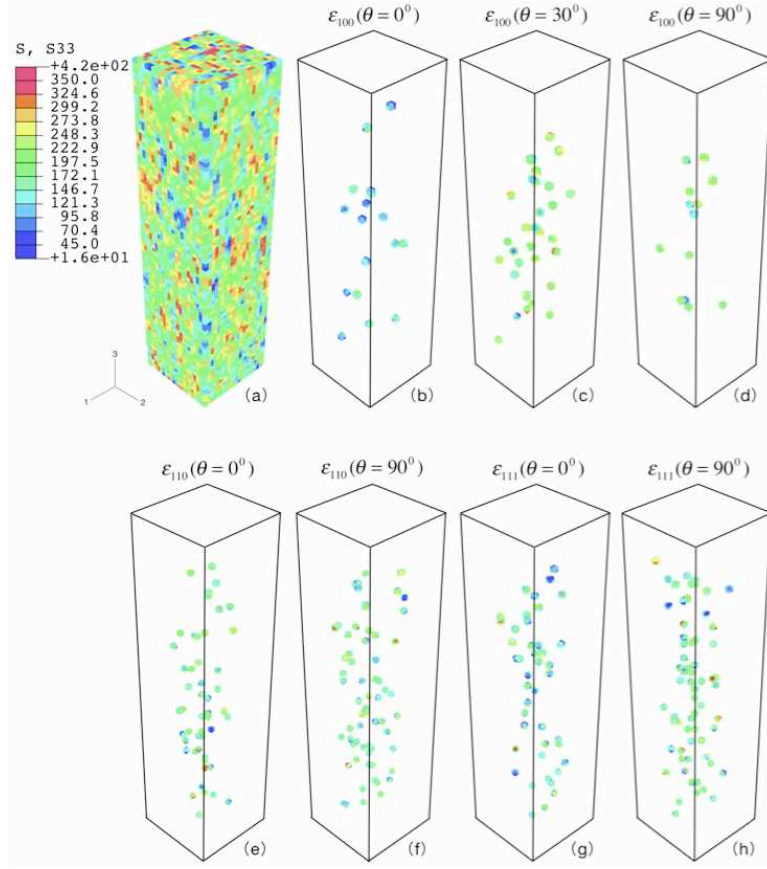


Figure 4.2. A polycrystal is numerically constructed by cubic grains with each grain assigned with the same set of crystal plasticity parameters but with random crystallographic orientations. Contours of normal stress along the loading axis at the end of a full loading cycle on the entire specimen in (a) and on the selected grains (i.e., their hkl directions are parallel to the diffraction vector direction θ) in (b)-(h).

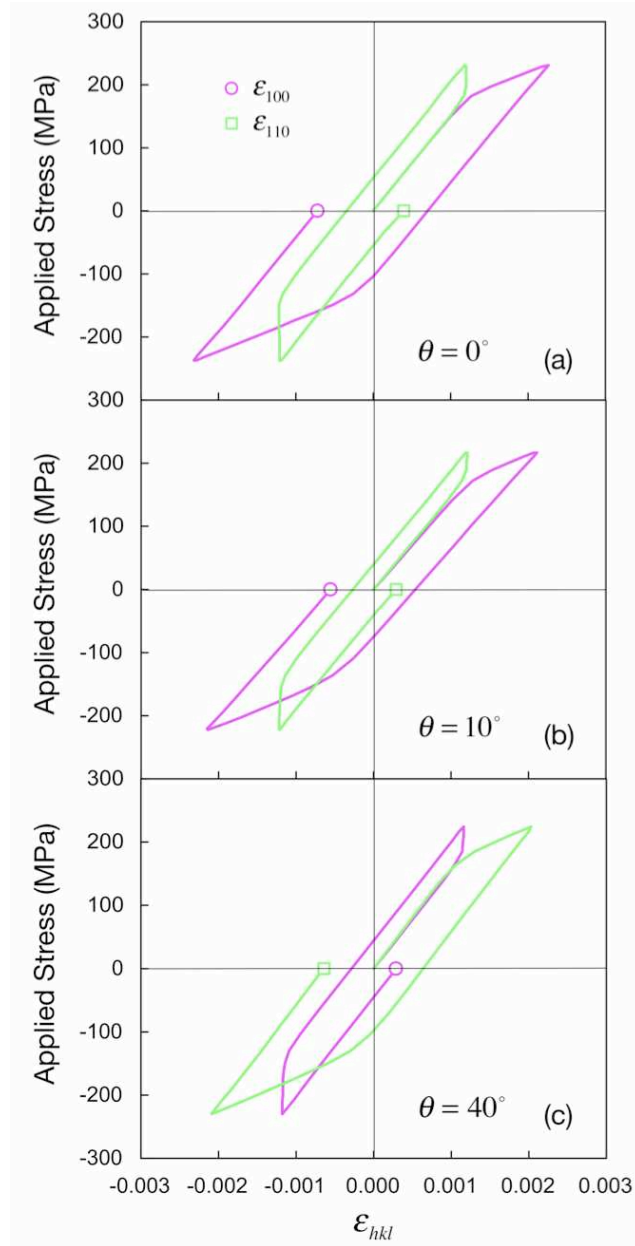


Figure 4.3. The applied stress versus lattice strain curves from our Taylor model with varying angle θ . See text for model construction.

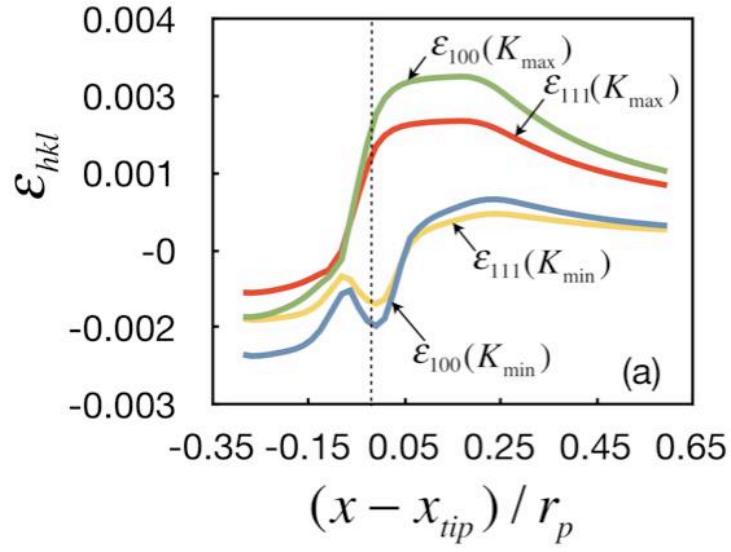


Figure 4.5. Predicted lattice strain profiles along the crack plane using the first group of cohesive parameters in Table 3.1. The slip plasticity is turned off to mimic the intergranular damage. The major difference between Fig. 3.8 and this figure lies on the $\varepsilon_{111} \sim \varepsilon_{100}$ cross-over location.

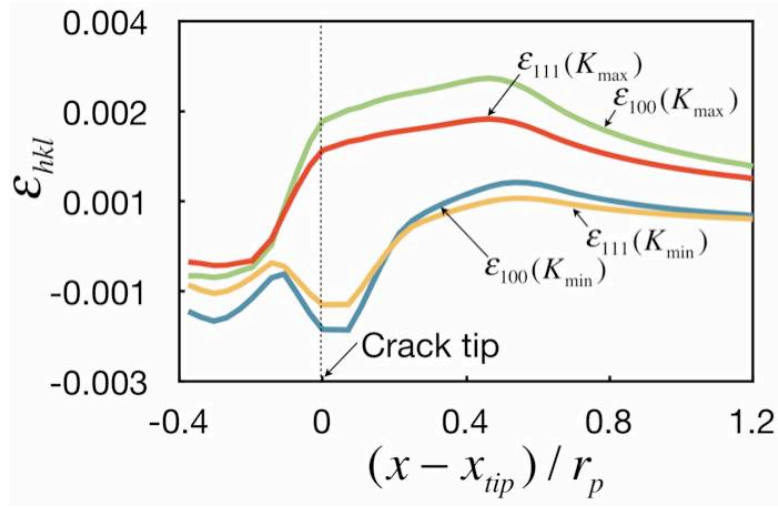


Figure 4.6. Predicted lattice strain profiles along the crack plane using the second group of cohesive parameters in Table 3.1. The slip plasticity is turned off to mimic the intergranular damage. The major difference between Fig. 3.9 and this figure lies on the $\epsilon_{111} \sim \epsilon_{100}$ cross-over location.

Chapter 5

Kinematic relationships between lattice misorientation and crystalline slip fields in wedge indentation

5.1. Indentation size effects and lattice misorientation fields

In the past several decades, great interests in the small-scale deformation behavior have been gained since the plasticity mechanisms might be quite different when the specimen size is reduced to less than several micrometers, which is known as the size effect. The new imaging and characterization techniques provide the effective way to examine these size effect phenomena. One interesting phenomenon that has been observed by the indentation technique is that the mechanical hardness exhibiting significant size-dependent behavior when the indentation depth approaches the micro- and nano-scales (Gerberich et al., 2002; Bull, 2003).

Experimentally, indentation size effects (ISE) on carefully prepared single crystals suggest that the underlying mechanisms are related to the geometrically necessary dislocations (GNDs) (Gao and Huang, 2003; Pharr et al., 2010; Kiener et al., 2009), which are introduced to ensure compatible deformation fields. The conventional plasticity cannot be used to predict the size effect because of the lack of a material length scale (Nye, 1953; Ashby, 1970; Gurtin, 2000). During indentation, the material lying underneath the indenter will be pushed aside and into the substrate material by dislocation-accommodated material flow. One application based on the GND theory is the Nix-Gao Model (Nix and Gao, 1998). The material hardness at small scales is finally related to the GND density by the Taylor relation and a

sequence of mathematical calculations. The hardness increases at small depths because the GND density is inversely proportional to the depth and rises dramatically and without bound when the contact is small. The prediction of the small-scale material hardness for the single crystal materials under berkovich indenter by Nix-Gao Model agrees well with the indentation tests. A mechanism-based strain gradient plasticity theory has been proposed by Gao and Huang (Gao et al., 1999; Huang et al., 2000) to link the microscale statistically stored dislocation and geometrically necessary dislocation to the mesoscale plastic strain and strain gradient fields.

Even though the observation of the dislocation microstructure may provide the valuable information for understanding the ISE behavior, it is not easy due to the complexity of the techniques required to image dislocations at the related scales. Since the last decades, the diffraction technique has been popular due to its capability to investigate the material microstructure, including the grain level strain and the lattice misorientation. The latter represents the crystal lattice curvature, which can be used to determine the density of the GND during indentation (Larson et al., 2008; Larson et al., 2007). A few experimental techniques have been developed to investigate the indentation-induced dislocation microstructure on or less than μm , such as electron back-scatter diffraction (EBSD) technique (Kiener et al., 2009; Kysar et al., 2007) and the synchrotron x-ray measurements (Wang et al., 2004; Larson et al., 2007; Feng et al., 2008). A newly developed 3D x-ray structural microscopy technique can measure the lattice misorientation fields with spatial resolution up to sub-micron length scale (Wang et al., 2004; Larson et al., 2007).

In this chapter, the effect of the orientation of slip systems on the 2D wedge indentation of a model single crystal is investigated. The crystal is of the FCC

structure with crystallographic axes [101] and [010] lying in the x_1 and x_2 directions under global coordinates, which ensures a plane strain deformation field when indenting along $-x_2$ direction. Continuum crystal plasticity theory with rotation angles calculated by the polar decomposition method is used to predict the lattice misorientation field under the indenter. Furthermore, the crystalline solid is gradually rotated about the x_2 axis, resulting changes of lattice misorientation patterns under the indenter. These 2D simulations, as well as a 3D Berkovich indentation simulation, suggest a kinematic relationship between the lattice misorientation and crystalline slip fields.

5.2. Indentation-induced lattice rotation fields

The lattice curvature field (i.e., the gradient of lattice rotation), together with the elastic strain field, can be used to evaluate the dislocation density tensor, which is of critical importance in understanding the dislocation microstructure at mesoscopic length scales (Larson et al., 2007). The confined volumes of nanoindentation deformation provide highly instrumented and controlled defect distributions. To this end, crystal plasticity simulations have been performed for wedge and Berkovich indentation, as described below.

Under finite deformation condition, the multiplicative decomposition of the deformation gradient $\mathbf{F}$ gives $\mathbf{F} = \mathbf{F}^e \mathbf{F}^p$, where plastic deformation due to crystalline slip is described by $\mathbf{F}^p$ (Bower and Wininger, 2004). The plastic rate of deformation is

$$\dot{\mathbf{F}}^p \mathbf{F}^{p-1} = \sum_{\alpha} \dot{\gamma}^{(\alpha)} \mathbf{s}^{(\alpha)} \otimes \mathbf{m}^{(\alpha)}, \quad (5.1)$$

The lattice rotation field, θ_i , can be calculated from the rotation tensor, $\mathbf{R}^e$, by

$$R_{ij}^e = \delta_{ij} \cos \theta - e_{ijk} w_k \sin \theta + w_i w_j (1 - \cos \theta), \quad (5.2)$$

where δ_{ij} is Kronecker delta, e_{ijk} is the permutation tensor, and $\theta_i = \theta w_i$ with a unit vector w_i . The rotation tensor can be determined by

$$\mathbf{F}^e = \mathbf{R}^e \mathbf{U}^e = \mathbf{R}^e \sqrt{\mathbf{I} + 2\mathbf{E}}, \quad (5.3)$$

where $\mathbf{U}^e$ is the right-hand stretch tensor, $\mathbf{I}$ is unit tensor, and $\mathbf{E}$ is the Lagrange-Green strain tensor. The crystal plasticity finite element method in Chapter 2 has been expanded to include the above calculations (Gao et al., 2011).

Wedge indentation on single crystal copper is performed. The indenter is assumed to be frictionless and rigid with an angle of $\alpha = 10^\circ$ as shown in Fig. 5.1. The faraway boundary is clamped. The material parameters include the Young's modulus $E=70\text{GPa}$, Poisson's ratio $\nu=0.33$, stress exponent $n=10$ (as in the power-law creep), reference strain rate $\dot{\gamma}_0 = 0.001\text{s}^{-1}$, initial slip strength $\tau_0 = 50\text{MPa}$, and latent hardening parameter $q=1$. No strain hardening is prescribed. These parameters have been explained in Chapter 2.

The original orientation is shown in Fig. 5.1, which satisfies the pseudo plane strain condition with the assumption that shears in the same slip system can be regarded as vectors and follow the vector calculus (Rice, 1987). Consequently the 12 slip systems are combined to 3 as shown in Fig. 5.2(c). Fig. 5.2(a) represents an FCC lattice structure with 12 slip systems marked in the form of NM [hkl] (Mth slip system with slip direction in [hkl] following the sequence in Fig. 5.2(b)). Thus the three pseudo slip directions $[\bar{1}21]$, $[121]$ and $[101]$ in plane (101) as shown in Fig. 5.2(c) can be formulated individually by the following combinations:

(i) The equal amounts of N1 and N3 slips in the same slip plane (111) make the effective slip direction $[1\bar{2}1]$ in plane (101);

(ii) Similar to (i), N7 and N8 slips make pseudo slip direction $[121]$;

(iii) N4 and N11 slips make pseudo slip direction $[101]$.

When loading in plane (101) along direction $[010]$, the above three combinations are the only activated pseudo slip systems when deformation achieves plastic region. Plastic strains in other slip systems or other combinations are found to be zero under the plane strain mode.

5.3. Simulation results

The lattice rotation field with rotation axis $[1\bar{1}0]$ is plotted in Fig 5.3. The lattice rotation pattern forms distinct sectors with clockwise and counter-clockwise rotations. This compares favorably to the discrete dislocation simulation (Pharr et al., 2010) and a number of EBSD experimental observations (Kiener et al., 2009).

Analyses based on the knowledge of crystallographic slip are considered to understand the rotation pattern. Consider the single slip plastic deformation of a single crystal under uniaxial tension test as shown in Fig. 5.4(a). If the loading grips are constrained along the loading axis, the single crystal should rotate as a consequence of directional preference of the slip field. As the previous discussion of the three equivalent slip systems in plane (101) specified by Rice (1987), we plot the shear strain on these three equivalent slip systems (i), (ii) and (iii) as shown in Fig. 5.5 (a)-(c) with the discrete sectors. We believe that the lattice misorientation is determined by the indentation direction and the slip system. To this end, more simulations have been performed to systematically investigate the dependence of

lattice rotation patterns on the orientation of slip system. In order to ensure the plane strain condition, we gradually rotate the crystalline solid counterclockwise about the out of plane direction by an angle ψ . Fig. 5.6 shows the results for several values of ψ . It shows that the rotation pattern becomes symmetric again for $\psi = 90^\circ$ as shown in Fig. 5.6(c). In this case, the combined slip direction $[1\bar{2}1]$ and $[121]$ are lying symmetrically with $[101]$ lying in the $-x_2$ direction.

Fig. 5.7 shows the variation of schmid factor in the standard stereographic triangle for an FCC crystal, which contains all the possible orientations of the specimen. It also shows the path traced by the specimen axis during deformation, where A represents the direction vector of the specimen axis in Fig. 5.4(a). The FCC lattice possesses twelve crystallographic slip systems of the type slip plane $\{111\}$, slip direction $\langle 110 \rangle$ although the resolved shear stress is different on each system. The four slip planes are named according to their function. The primary slip plane $H = (11\bar{1})$ in Fig. 5.7, together with the slip direction $g = [101]$ forms the slip system with the largest Schmid factor, operative at the onset of plastic deformation. At the symmetry boundary $[100]$ - $[111]$ the stress on the conjugate system $D = (1\bar{1}1)$, $[110]$ is the same as on the primary system and two systems operate alternately. $Q = (1\bar{1}\bar{1})$ is the cross-slip plane, which contains the same slip direction $g = [101]$ as the primary slip plane. The stress axis position on the projection tends to move toward the active slip direction. As a result, slip will be instituted on this slip system, called the conjugate slip system, and the stress axis will now move along a path toward the $[110]$ slip direction. Theoretically, while this shift in the slip system should occur once the stress axis crosses the boundary separating the two stereographic triangles, there is always some degree of overshoot before the conjugate slip system takes over. The

conjugate slip system will bring the stress axis back into the original stereographic triangle where the primary slip system is again favored. This oscillation of the stress axis will be repeated a number of times, with the result that the axis eventually reaches the $[211]$ direction, a direction that lies on the same great circle as the conjugate and primary slip directions and the midway between them. This is stable end orientation for the crystal, and once it has obtained this orientation, further deformation will not change the orientation of the crystal relative to the tensile stress axis.

5.4. Comparison between 2D discrete dislocation and crystal plasticity simulations

Besides, based on the dislocation dynamics principles, in the dislocation discrete simulations, dislocations can be either randomly placed in the initial microstructure or created by the activation of dislocation sources. Such dislocation discrete model can also be used to capture the lattice misorientation behavior during indentation. For instance, the wedge indentation simulated by the dislocation dynamics simulations by Balint et al. (2006) is shown in Fig. 5.8 (a). The misorientation contour is shown in Fig. 5.8 (b), which shows the discrete regions separated by well-defined boundaries. Comparing with Fig. 5.3 from our finite element simulations, the subsurface rotation sectors are distinctly different. In the dislocation model, there is only one boundary separating positive and negative rotations. While, in the continuum crystal plasticity model, there are four distinct sectors with clockwise and counter-clockwise rotations, which is more similar to the wedge indentation experiments by Kysar et al (Kysar et al., 2007). Further studies are required to resolve such discrepancies.

5.5. Simulations of 3D Berkovich indentation

We extend our method to the 3D case. The Berkovich indentation-induced lattice misorientation field is also simulated by the continuum crystal plasticity model. The problem that we are interested in is on a particular plane of the crystal structure, which ensures a comparison of the 3D Berkovich indentation to the 2D wedge indentation. The Berkovich indenter is placed with the indenter ledge lying on the plane spanned by $[111]$ and $[1\bar{2}1]$ as shown in Fig. 5.9(a), which is referred to as in Fig. 1.8 by Yang et al. (2003). A corresponding wedge indentation, which is supposed to provide the same effect as the 3D case, is shown in Fig. 5.9(b), which shows that by setting $\psi = \beta$ in the 2D model, we can get the orientation pattern on plane (-101) with the indentation direction $[\bar{1}\bar{1}\bar{1}]$. The geometry gives $\beta = \tan^{-1} \sqrt{2} = 54.7^\circ$.

Simulation results are presented in Fig. 5.10. For the Berkovich indentation, Fig. 5.10 (a) views the cross-sectional $(\bar{1}01)$ plane at the contact center. Note that the pyramidal surface is on the left, which is intended to be simulated by the right side surface of the asymmetric wedge indenter in Fig. 5.10 (b). Thus the two images in Fig. 5.10 should be compared after a mirror reflection as well as by an opposite change of color code.

As expected, these orientation patterns are similar at the contact center, and are different at the contact shoulders where the 3D contact field dominates. Both simulations demonstrate the formation of four sectors with clockwise and counter-clockwise rotation about the out-of-plane direction. These results are amenable to

comparisons to experimental observations that are available in literature (Larson et al., 2009).

5.6. Summary

In summary, the procedure to determine the lattice rotation field from deformation gradient is presented and the misorientation patterns with varying initial crystallographic orientations are analyzed numerically by finite element simulations. These simulations suggest a kinematic relationship between the lattice misorientations and slip fields, while more detailed comparisons to experiments are needed.

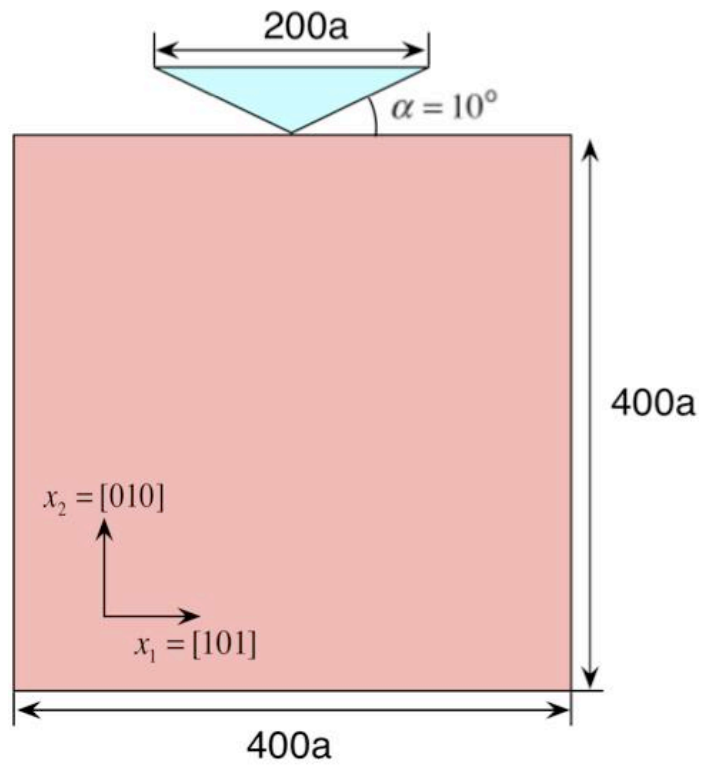


Figure 5.1. Schematics of the wedge indentation on a model FCC single crystal.

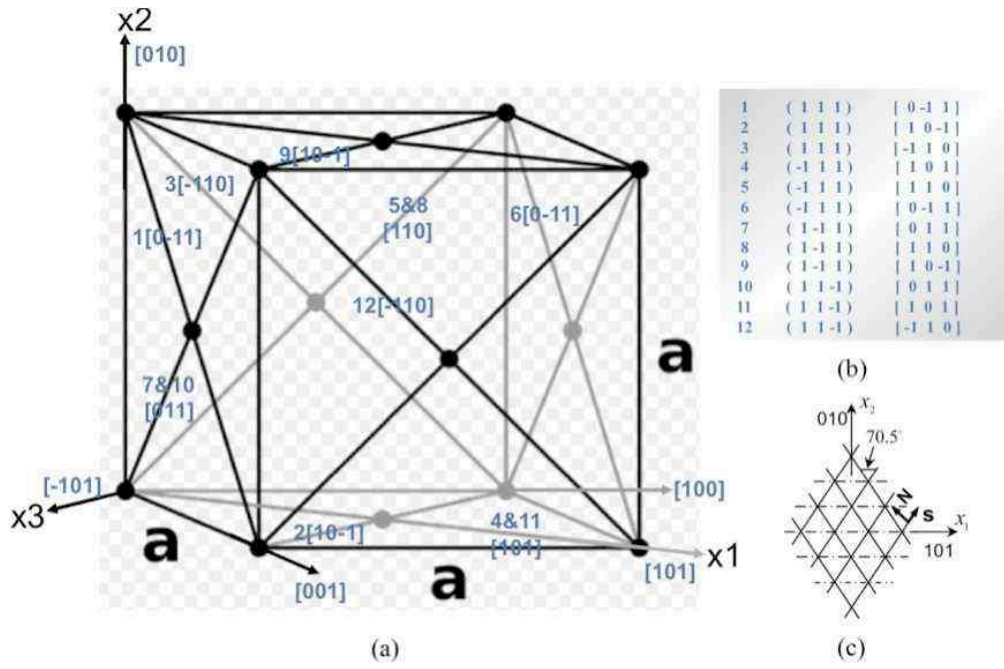


Figure 5.2. (a) FCC lattice structure with 12 slip systems; (b) sequence of 12 slip systems used in our simulation; (c) 3 equivalent slip directions under plane strain condition in Fig. 5.1. The construction follows Rice (1987).

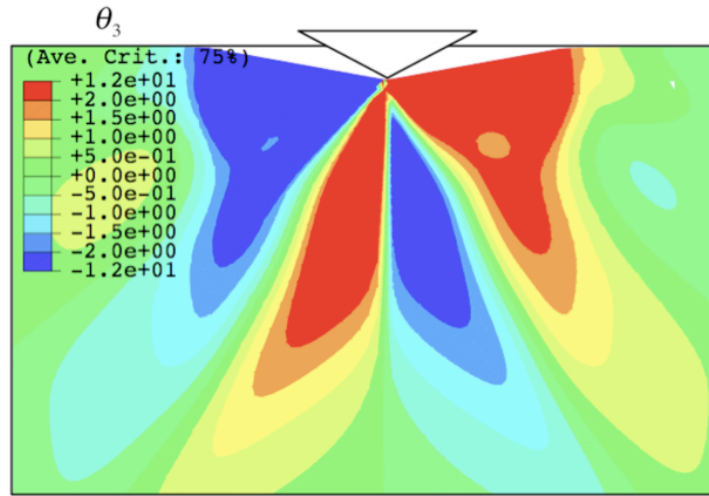
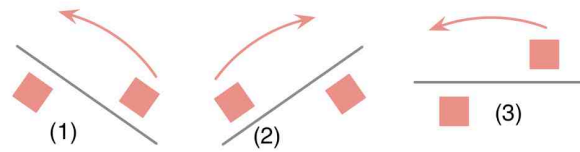
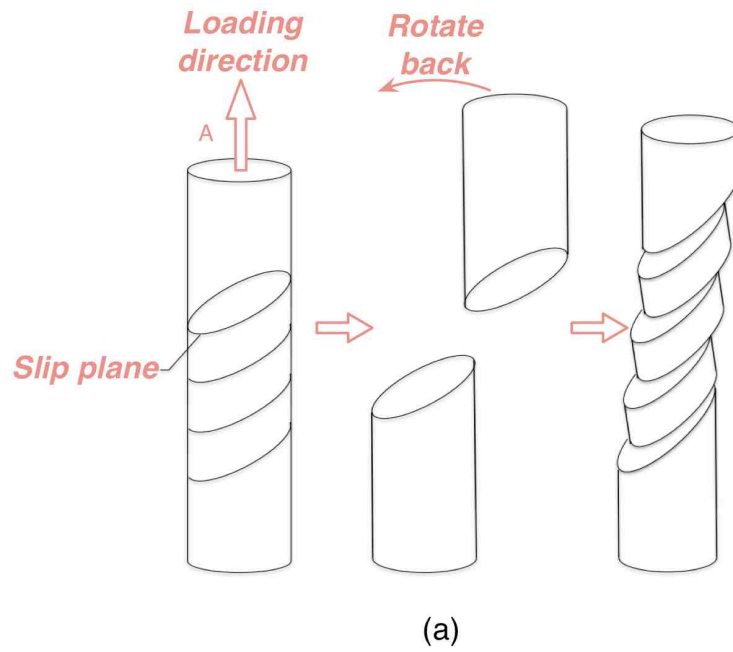


Figure 5.3. Lattice misorientation pattern for a model FCC single crystal under wedge indentation. The crystallographic orientations are chosen to ensure a plane strain deformation condition.



(1): $[0-11](111)+[-110](111)=[1-21](101)$
 Positive slip strain=Positive misorientation about x3
 (2): $[011](-111)+[110](1-11)=[121](101)$
 Positive slip strain=Negative misorientation about x3
 (3): $[101](-111)+[101](11-1)=[101](101)$
 Positive slip strain=Positive misorientation about x3

Figure 5.4. (a) For fixed-grip uniaxial tests, a single slip system will lead to rotation of lattice. (b) Plastic slip and the corresponding lattice misorientation induced by slip in 3 equivalent slip systems.

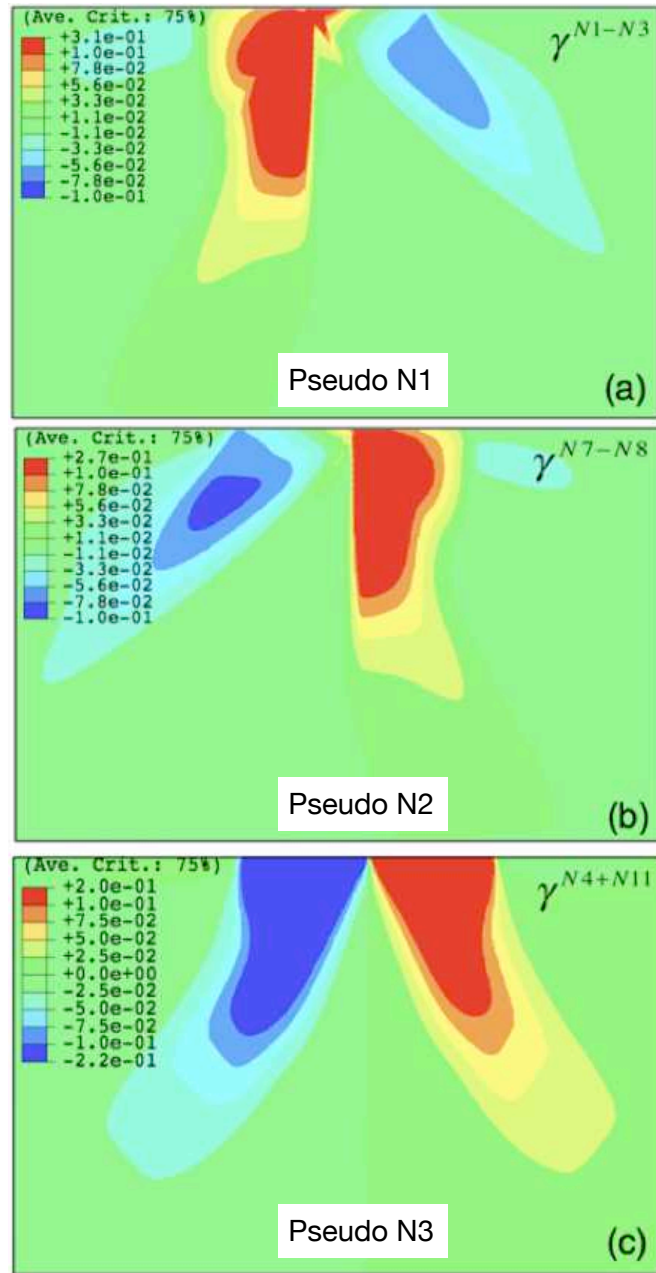


Figure 5.5. Slip strains on the three equivalent slip systems in (a)-(c).

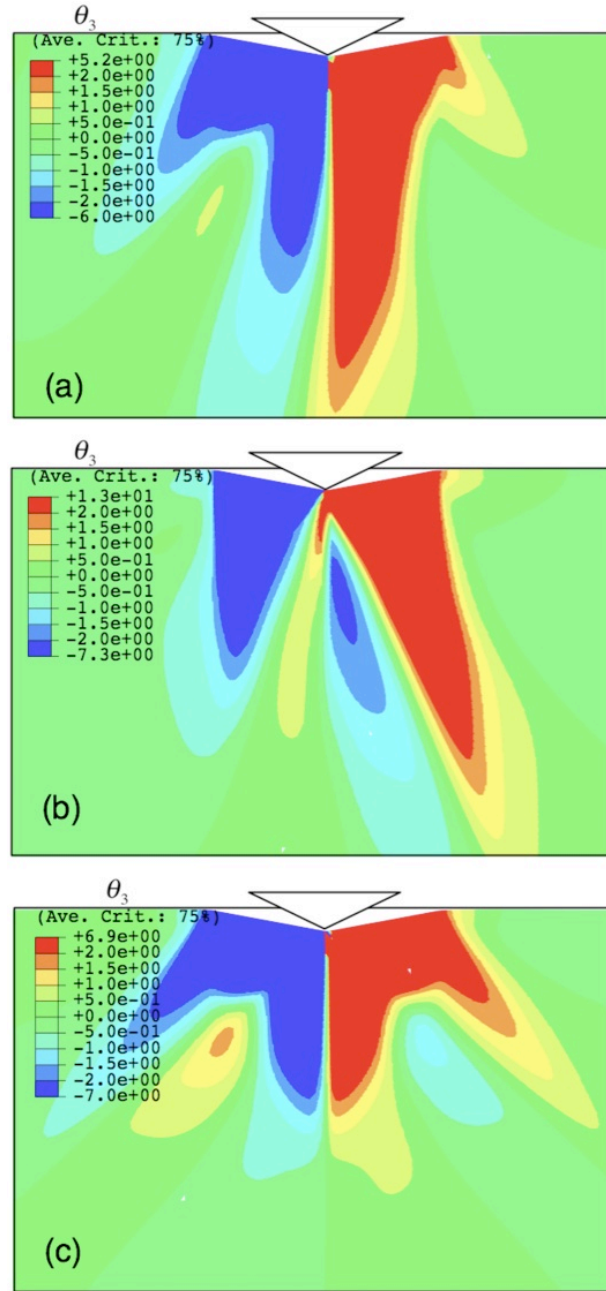


Figure 5.6. Lattice misorientation patterns for the wedge indentation on a model single crystal with (a) $\psi = 30^\circ$, (b) $\psi = 60^\circ$, and (c) $\psi = 90^\circ$. ψ is the lattice rotation angle as described on page 88.

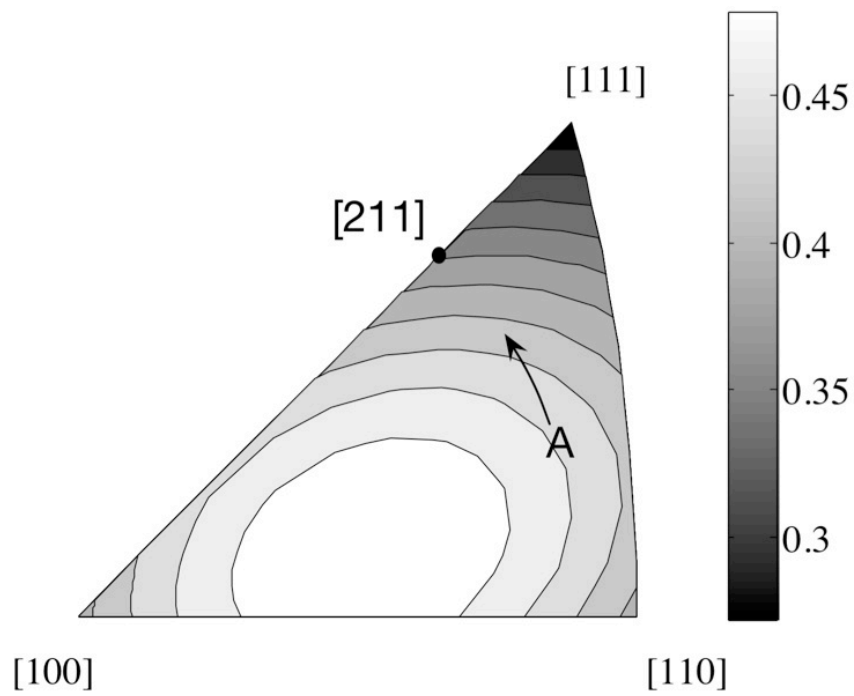


Figure 5.7. Slip systems in the FCC lattice and path traced by the crystal axis in a tensile test shown in the standard triangle of the stereographic projection. Lines with equal Schmid factors are shown for the primary slip system. Refer to Fig. 5.4 (a) for the origin of lattice rotation.

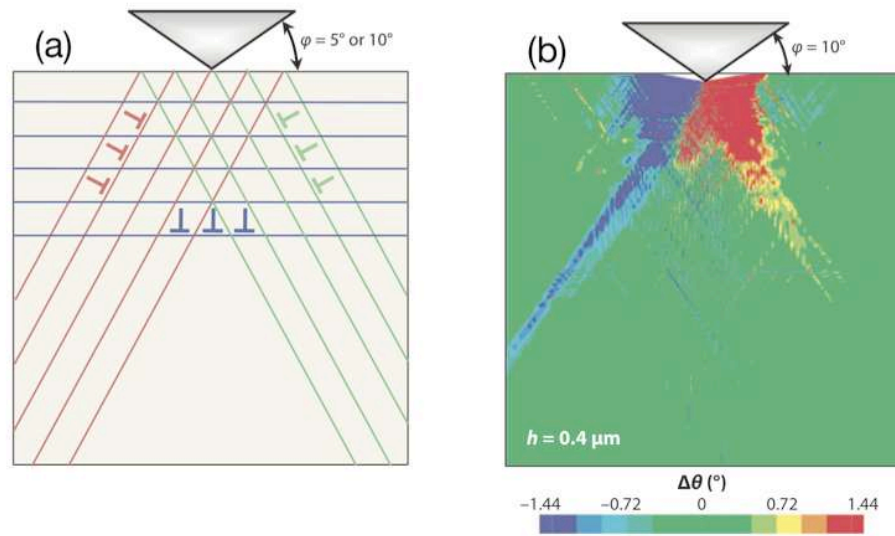


Figure 5.8. Results of two-dimensional dislocation dynamics simulations by Balint et al. (2006). (a) The simulation geometry for wedge indentation, showing the three independent slip systems. (b) The equilibrium distribution of dislocations for indentation with a 170° wedge, color-coded according to the three different slip systems.

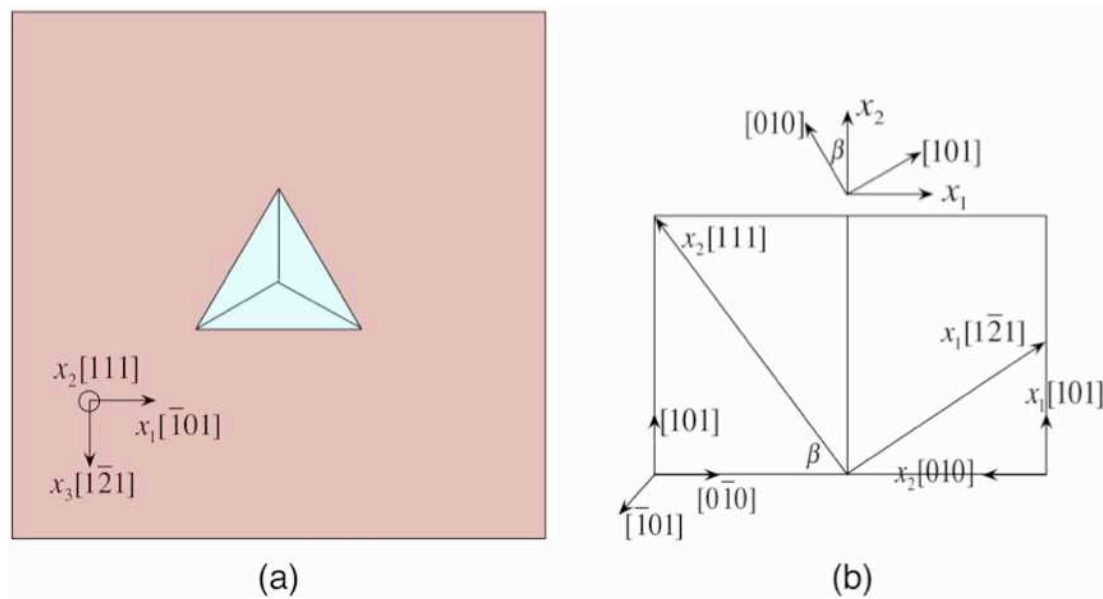


Figure 5.9. The top surface of the 3D simulation under Berkovich indentation. The indent direction is along $-x_2$ direction.

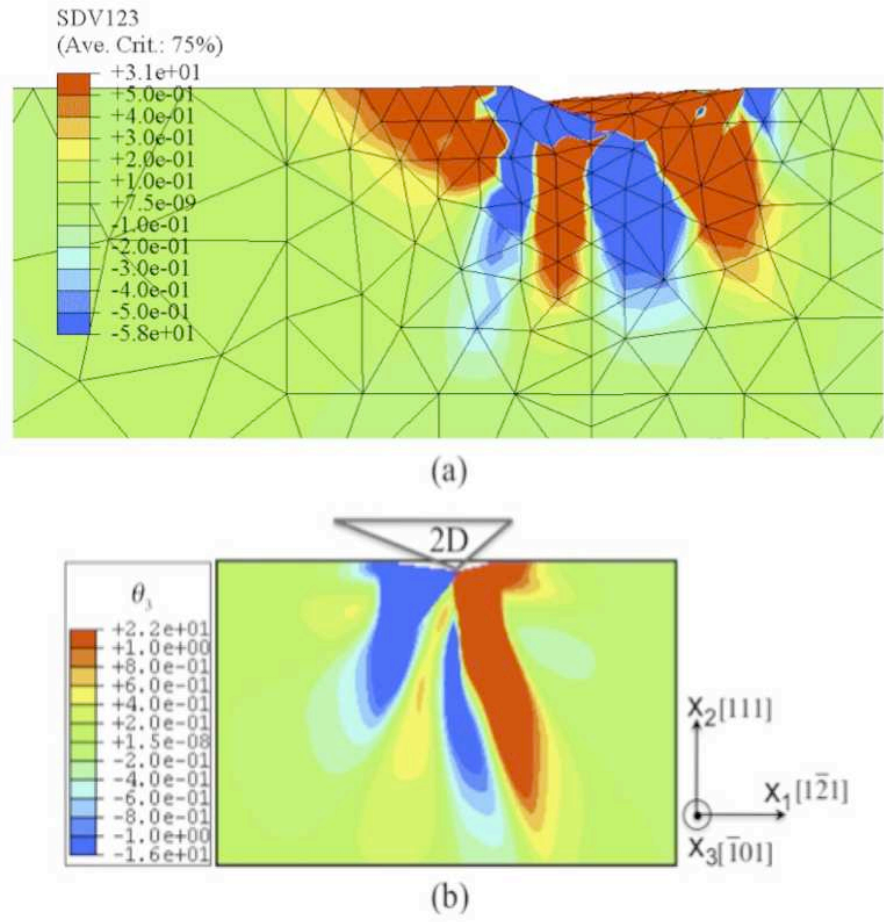


Figure 5.10. Lattice rotation patterns with rotations about $[-101]$ directions for (a) a 3D Berkovich indentation and (b) a 2D asymmetric wedge indentation.

Chapter 6

Effects of non-common slips and twins

6.1. Deformation mechanisms in HCP materials

The inelastic deformation mechanisms of hexagonal close-packed (HCP) crystals at low homologous temperatures are typically controlled by the synergy of crystallographic slip and deformation twinning. The basal plane is always the closest packed among all crystallographic planes in HCP materials. The lattice ratio c/a , where a is interatomic distance on the basal plane in any of the three $\langle 11\bar{2}0 \rangle$ directions and c is the distance between the first and the third layers, is normally considered to control the deformation behavior of HCP materials. The ideal c/a ratio, calculated on the basis of a hard sphere model is 1.633. However, in real materials it varies from 1.567 for beryllium to 1.886 for cadmium. The axial ratios for magnesium and zirconium alloys, which are of interests and will be discussed with more details in this chapter, are 1.624 and 1.593 respectively. The higher c/a ratio the more close-packed of the basal planes than other alternative planes. Thus the smaller c/a ratio leads the easier non-basal slip systems (Staroselsky and Anand, 2003). The possibly slip and twinning systems for a hexagonal material include basal slip $\{0001\}\langle 11\bar{2}0 \rangle$, pyramidal $\langle a+c \rangle$ slip $\{11\bar{2}2\}\langle \bar{1}\bar{1}23 \rangle$, pyramidal $\langle a \rangle$ slip $\{1\bar{1}01\}\langle 11\bar{2}0 \rangle$, prism $\langle a \rangle$ slip $\{1\bar{1}00\}\langle 11\bar{2}0 \rangle$, second order pyramidal $\langle a+c \rangle$ slip $\{11\bar{2}2\}\langle \bar{1}\bar{1}23 \rangle$ as shown in Fig. 6.1. Tensile twinning $\{10\bar{1}2\}\langle \bar{1}011 \rangle$ and compressive twinning $\{10\bar{1}1\}\langle \bar{1}012 \rangle$ may also occur in reality. We can clearly see that the slip

directions among three slip systems are all in direction $\langle 11\bar{2}0 \rangle$, which indicates the inadequate independent slip systems in HCP materials. Therefore the slip systems in HCP materials cannot satisfy the arbitrary anisotropic deformation beyond the elastic stage. As a result, twinning is an alternative way to allow plastic deformation along c direction. The shear direction in twinning is also determined by the c/a ratio (Yoo, 1981; Hosford, 1993). For example, materials with $c/a < \sqrt{3}$, the twinning direction is $\langle \bar{1}011 \rangle$, and twinning happens under tension along c direction. On the other hand with $c/a > \sqrt{3}$, twinning shear is in direction $\langle \bar{1}012 \rangle$ and twinning is under compression along c direction.

These deformation mechanisms make the inelastic deformation behavior of HCP structural materials very complicated, and thus it necessarily requires the investigation on the length scale associated with slip/twinning. In this chapter, we present a systematic study on mechanical properties of HCP materials in the plastic stage by neutron diffraction internal strain measurements and the crystal plasticity finite element method. Neutron diffraction technique have been used for HCP materials to probe the grain-level plastic anisotropy, especially the internal strains such as type II strains (intergranular strains) and texture evolutions (Tome et al., 2002; Agnew et al., 2002; Agnew et al., 2003; Wu et al., 2008).

From the modeling point of view, except for the explicit finite element methods, the self-consistent model is also a powerful tool. The Visco Plastic Self-Consistent (VPSC) model was developed by Turner and Tome in 1994 following the scheme of Hutchinson in 1976 (Turner and Tome, 1994; Hutchinson, 1976). The hardening behavior in the EPSC model is described by the extended voce law:

$$\tau^s = \tau_0^s + (\tau_1^s + \theta_1^s \Gamma)[1 - \exp(-\theta_0^s \Gamma / \tau_1^s)], \quad (6.1)$$

where τ^s is the instantaneous CRSS, τ_0^s and $\tau_0^s + \tau_1^s$ are the initial and final back-extrapolated CRSSs, respectively. θ_0^s and θ_1^s are the initial and asymptotic hardening rates, Γ is the accumulated plastic shear in the grain. The model can be used to predict internal strain and texture evolution, residual and thermal strains and to compare with neutron diffraction experiments. It has been recently extended to typical HCP materials such as magnesium and zirconium alloys (Clausen et al., 1998).

A crystal-plasticity-based constitutive model accounting for both slip and twin has been developed by Staroselsky and Anand (2003). We made slight modifications of their VUMAT program for lattice strain analysis and applied to magnesium alloy AZ31B and zirconium alloy Zircaloy-4 (Zr-4). Predictions by the explicit finite element simulations are compared to the neutron diffraction internal strain measurements results and the EPSC model results to further explore the deformation mechanisms for hexagonal structural materials.

6.2. Constitutive law for HCP materials

Following the work of Staroselsky and Anand (2003), the plastic deformation at the length scale associated with slip and twinning is highly inhomogeneous and it is obviously unnecessary (probably impossible) to take consideration of the fine structure to the level of the dislocation loops, slip bands and twin lamella of the crystal. Alternatively, similar to the composite mechanics, Staroselsky and Anand (2003) introduced a representative volume element (RVE) model, which is the smallest volume element containing enough dislocation loops and twins to result in

the smooth plastic deformation response process. The volume fraction of twins for the i th twin system in such a RVE is f^i , which satisfies $f^i > 0$ and $\sum_i f^i \leq 1$ and no detwinning is assumed here. The kinematics in classic crystal plastic model in Chapter 2 needs to be added with the contribution to the plastic strain rate by the twins, which are treated as phase transformation strains multiplied with the corresponding volume fraction.

The total slip and twinning systems considered in constitutive model are listed as in Table 6.1 (Staroselsky, 1998). The above constitutive equations are implemented into the finite element software ABAQUS explicit VUMAT. The detailed procedure can be referred to the work of Staroselsky and Anand (2003). In this chapter, this constitutive VUMAT is modified to calculate the lattice strain evolution for Zr-4 and AZ31B respectively. A cubic specimen with 729 elements, which represent 729 grains, is set up to represent the polycrystalline aggregates. Each element is assigned with three Euler angles to prescribe the crystallographic orientation. The diffraction condition, which is similar to the neutron diffraction internal strain measurements, can be found with details in Chapter 2.

6.3. Deformation mechanisms of Zr-4 alloy

6.3.1. Background

Zircaloy-4 alloy (Zr-4) is a HCP structural zirconium alloy, which has been studied since several decades ago due to its wide applications in the nuclear industry. The nominal chemical composition (weight percent, wt.%) of the starting Zr-4 is 1.5 Sn, 0.22 Fe, 0.12 Cr, 0.13 O, and balance Zr (Garlea, 2008). It is being used extensively as the nuclear fuel sheathing material and fuel pressure tubes (Garlea et

al, 2010). The elastic, plastic and thermal anisotropic properties of Zr-4 lead to the non-negligible residual stresses when subjected to thermal or mechanical loading conditions because of the interactions between grains with distinctive crystallographic orientations. The studies of residual stresses in Zr-4 alloys are reported elsewhere (Carr et al., 2004; Elena et al., 2010; Carr et al., 2006). The studies show that the internal/intergranular stresses may highly impact the material performances under severe pressure or temperature or irradiation conditions. Thus it becomes clearly important to understand the evolution of the internal stresses/strains especially from the mechanical point of view. Due to the asymmetry of HCP structure, there are possibly 5 slip systems and 2 twinning systems that may or may not be activated.

As we mentioned before, neutron or X-ray diffraction techniques are extensively used for the Type II or Type III strain measurements (Holden et al., 2002; Garlea et al., 2010; Garlea et al., 2007) because of the high penetration and non-destructive characteristics of incident beams. As we discussed before that the mechanical properties of HCP materials are strongly affected by the c/a ratio, that is, the larger the c/a value, the more closed packed on basal plane, and thus the easier the basal slip occurs. The c/a ratio for Zr-4 is 1.593, which is relatively low compared to other HCP materials meaning that non-basal slip, such as prism or pyramidal slips, happens easily. Some evidences also show the comparably information of the governing slip modes in Zr-4 by neutron diffraction experiments. During plastic deformation of zirconium alloy, the prism $\langle a \rangle$ slip is shown to be the most easily activated, followed by pyramidal $\langle a+c \rangle$ slip and then tensile twinning (Akhtar, 1973; Ballinger et al., 1984). Strong evidence is found for tensile twinning in tensile tests and in compression tests in some typical directions for the textured polycrystalline zirconium alloy (Daymond, 2007). The basal slip mode in zirconium

or its alloys is still not clear in literature and in most cases the pyramidal $\langle a \rangle$ slip is always considered instead of basal slip (Pang et al., 1999; Francillette et al., 1998).

The EPSC model has been extensively used to predict the evolution of lattice strain and texture in zirconium based alloys. The evolution of internal stresses in Zircaloy-2 with rod texture under thermal or stress conditions was simulated by the EPSC model (Turner and Tome, 1994). Besides the documented evidence of the prism slip and pyramidal $\langle a + c \rangle$ slip, as well as tensile twinning as we discussed before, there is little evidence on whether the basal slip occurs or not at the room temperature or whether pyramidal slip is an alternate mechanism. However the inclusion of the basal slip in the EPSC model has been found to improve the ability to reproduce experimental macroscopic flow curves and bulk texture development caused by plastic deformation (Pang et al., 1999; Francillette et al., 1997; Francillette et al., 1998). While on the other hand, the pyramidal $\langle a \rangle$ slip was preferred as an alternative to basal slip to better fit the experimental data (Philippe et al., 1988; Tome et al., 1991; Philippe et al., 1988).

In this study, the crystal plasticity constitutive law together with the explicit finite element simulation method is applied to probe the lattice strain evolution and the lattice strain distribution around the fatigue crack tip for Zr-4 alloy, which are compared with the neutron diffraction experimental results. It is indicated that the Zr-4 alloy has the random texture and the mean grain size of about 700 micrometers. The systematic parameter study to improve the fitting to the experimental data can also extends our understandings of the plastic deformation mechanisms in Zr-4 at room temperature.

6.3.2. Lattice strain evolution during uniaxial loading

A cubic specimen consisting of 729 elements with each element represents a single grain is constructed to calculate the lattice strain evolution in Zr-4 alloy under both uniaxial compression and fatigue loadings. Each element/grain deforms according to the constitutive law prescribed in Section 6.2. Similar to the diffraction method, the diffraction conditions assist to select contribution grains and the volume average is made among those selected grains. The detailed procedure for the lattice strain calculations can be found in Chapter 2.

Garlea (2008) has explored a large number of possible deformation systems and hardening coefficients in the EPSC model to improve the reproduction of the neutron diffraction results. Among them, three most reasonable cases are listed. For case 1, basal slip, pyramidal $\langle a + c \rangle$ slip, pyramidal $\langle a \rangle$ slip, prism $\langle a \rangle$ slip and second order pyramidal $\langle a + c \rangle$ slip, with the corresponding slip planes and slip directions specified in Fig.6.1, are chosen to be activated during the deformation beyond yielding. For case 2 and case 3, besides the slight change of the hardening parameters and the negligible of the second order pyramidal $\langle a + c \rangle$ slip, tensile twin is also considered to positively affect the inelastic deformation mechanisms. The final volume fractions of tensile twin for case 2 and case 3 are 3% and 12% respectively. The detailed calculation results and the fitting by the EPSC model can be found in Garlea (2008).

The calculation results by our explicit finite element method and the fittings to the neutron data by Garlea are shown in Fig.6.2, 6.3 and 6.4 for the three cases correspondingly. The basic constants for Zr-4 are: Young's modulus $E=100$ GPa,

0.2% yield stress $\sigma_y = 318\text{MPa}$ (Garlea, 2008), Elastic constants $C_{11} = 143.5\text{MPa}$, $C_{12} = 72.5\text{MPa}$, $C_{13} = 65.4\text{MPa}$, $C_{33} = 164.9\text{MPa}$, $C_{44} = 32.1\text{MPa}$ (Pang et al, 1999), The initial yield stresses and the saturated stresses for the slip/twin systems used in the three cases are listed in Table 6.2, which are the same with the EPSC model except for that no hardening is considered in our simulation (the results supports that the non-hardening model can also well capture the plastic mechanisms in this case). The plane normal index used in the figures is different from the 4-index scheme and the comparisons between these two index schemes are shown in Table 6.3. It is evident that our results are comparable to the EPSC model. Good fittings can be achieved in the elastic regime, while when entering the deep plastic regime, the deviation from the neutron diffraction results become severely apparent for all the 6 plots in the 3 cases as shown in Fig.6.2, 6.3 and 6.4. The slightly change of the parameters and the activation of the tensile twin system cannot effectively improve the fitting, which requires the systematical and theoretical study to understand the deformation mechanisms among a number of the slip/twin modes combinations.

Here we refer to the strength to stiffness ratio, which can be found in details in Chapter 4, to explain the plastic anisotropy for Zr-4. The typical standing point is that the smaller strength to stiffness ratio, the earlier yield occurs, which means the lattice versus applied stress curve goes up beyond the yielding point, just like lattice strain ε_{001} . The stiffness along several directions that we are interested in are calculated directly from the neutron experimental data. They are: $E_{001} = 121.2\text{GPa}$, $E_{100} = 108.0\text{GPa}$, $E_{110} = 106.1\text{GPa}$, $E_{101} = 101.5\text{GPa}$, $E_{102} = 100.9\text{GPa}$, $E_{103} = 104.4\text{GPa}$. From the neutron diffraction experimental data, we easily obtain

that lattice strain ε_{001} has a smaller strength-to-stiffness ratio so that it yields first and thus we believe that the basal slip is not the easiest.

On the other hand, the basal, pyramidal $\langle a \rangle$ and prism $\langle a \rangle$ slips cannot accommodate any strains along the c-direction because Schmid factor along c-direction is zero. However the fact that the plastic deformation of ε_{001} from the neutron data is clearly observed, which is the deformation along the normal to the basal plane or the c-direction, requires the activation of the pyramidal $\langle a + c \rangle$ slip or the tensile twinning. Based on the analysis above, we try a number of parameters and the best fitting is obtained as shown in Fig.6.5. The parameters we use are listed in Table 6.4.

6.3.3. Lattice strain distribution around fatigue crack tip

The mode I fatigue study was conducted by Garlea et al. (2007). The CT specimens were pre-cracked and fatigued using a servo-hydraulic Material Test System (MTS) testing machine, which is interfaced with a Teststar IIs controller. The details can be found in Garlea (2008). The specimen was pre-cracked under a constant stress-intensity-factor range, ΔK , to the crack length, $a=11.43\text{mm}$. The fatigue crack growth tests were performed under ΔK control model, with a frequency of 10 Hz and a constant load ratio, $R=P_{\min}/P_{\max}=0.1$ at ambient temperature. Crack length was measurement by the crack-opening-displacement (COD) gauge using the unloading-compliance method (Chen et al., 2003). The $P_{\max}$ was chosen to be 4,000 N to the crack length of $a=22.86\text{mm}$. Spatially-resolved strain measurements were performed on the compact tension specimen using the Spectrometer for Materials Research at Temperature and Stress (SMARTS) instrument at the Los Alamos National

Laboratory. The two detector banks at SMARTS allow the measurements of the in-plane (IP) and transverse (TT) lattice strain according to the diffraction vectors parallel and perpendicular to the loading direction. In this section, for simplicity, only the IP lattice strain is being discussed and calculated to compare with the neutron data. Strain scanning was conducted along the crack, starting from 4 mm behind of the crack tip to 20 mm in front of the crack tip. The lattice strain distribution around the crack tip is shown in Fig.6.6 (a). The plastic zone is within a single grain, which means the size is smaller than 700 micrometers. Lattice strains ϵ_{002} , ϵ_{100} and ϵ_{102} are plotted respectively as the function of the distance from the crack tip. The lattice strain along the basal normal direction is shown clearly always at the bottom among the three except for a small region behind the crack tip, where the plastic wake takes the charge and the region far away in front of the crack tip. In the vicinity of the crack tip, ϵ_{102} appears to be slightly larger than ϵ_{100} .

Numerically, similar to Chapter 3, a two-scale model is developed to study the lattice strain evolution near fatigue crack tips for Zr-4 materials. An irreversible, hysteretic cohesive interface model is adopted to simulate a steady fatigue crack, which allows us to generate the stress/strain distribution and history near the fatigue crack tip. Such a deformation history is used as inputs for the micromechanical analysis of lattice strain evolution using the slip and twin-based constitutive model, thus making a mechanistic connection between macro- and micro-strains. Please refer to Chapter 3 for the detailed procedures and interpretations. The predict lattice strain are plotted against the distance from the crack tip normalized by the plastic zone size as shown in Fig.6.6 (b). Comparing Fig.6.6 (a) and (b), similar trends among the three particular lattice strain curves are observed. In the vicinity of the crack tip, lattice strain ϵ_{102} becomes slightly larger than lattice strain ϵ_{100} . Lattice strain ϵ_{001} is always

at the bottom except in the plastic wake region behind of the crack tip and at far away in front of the tip, which is somehow consistent with the earlier yield property of ϵ_{001} during uniaxial loading as shown in Fig. 6.5.

6.4. Deformation mechanics of magnesium alloy AZ31B

Magnesium alloys have been intensively studied recently due to its light weight, high strength, recyclability and its electromagnetic shielding property (Mordike and Ebert, 2001). Magnesium alloys thus have a wide use for automobile and electrical devices. In this section, an initially-textured magnesium alloy AZ31B (86.486% Mg, 2.798% Al, 0.715% Zn, Balance, Mn, Fe, in wt.%) is studied by the prescribed constitutive law. It has been shown that even though basal slip dominates in plastic deformation of magnesium, prism $\langle a \rangle$ slip and pyramidal $\langle a \rangle$ slip are also observed at the room temperature (Burke and Hibbard, 1952). Similar to Zr-4 alloy, It is impossible to accommodate strains along the c-axis, thus the pyramidal $\langle a + c \rangle$ slip and the tensile twinning can help alleviate the problem and thus play important roles in the deformation mechanism of magnesium alloys (Agnew et al., 2002; Agnew et al., 2001). One interesting observation that the stress-strain curves in tension and compression obtained from specimens made from extruded magnesium rods are quite asymmetry also supports the activated deformation twinning, Since a compressive stress applied nearly parallel to the basal planes favors twinning, whereas a similarly-directed tensile stress cannot induce such twins (Reed-Hill, 1973). The deformation of Mg alloys has been studied extensively using either finite-element methods (Staroselsky and Anand, 2003; Graff et al., 2007; Prakash et al., 2009) or self-consistent models (Agnew and Duygulu, 2005; Turner and Tome, 1994; Agnew et al., 2003), which show that the stress-strain response and texture evolution of Mg alloys

can be reasonably predicted. In this section, we focus our study on the lattice strain evolution of AZ31B under uniaxial loading to compare with the existing EPSC model.

The parameters listed in Table 6.5 are consistent with the work of Agnew et al. (2003). Only the basal and prism $\langle a \rangle$ slip and the tensile twinning are activated. The preliminary lattice strain results are shown in Fig. 6.7 (a), which are quite similar to the EPSC model results shown in Fig. 6.7 (b). To this end, we have shown the capability to predict the lattice strain evolution of magnesium alloy AZ31B and the comparison to the EPSC model.

6.5 Summary

To summarize, the deformation mechanism for HCP polycrystals, Zr-4 alloy and AZ31B alloy, are examined by the crystal plasticity based constitutive model. For Zr-4 alloy, by comparing with the EPSC model and the neutron diffraction data, we find by activating basal, pyramidal $\langle a+c \rangle$, pyramidal $\langle a \rangle$ and prism $\langle a \rangle$ slip and the tensile twin, the simulation results agree well with the experimental results. The basal plane normal direction in Zr-4 has a higher stiffness, which might be the reason of the smaller strength-to-stiffness ratio and thus leads to yield earlier than others. Besides the uniaxial loading condition, fatigue crack growth for Zr-4 alloy has also been considered. The lattice strain distribution around the fatigue crack tip is calibrated by the two-scale model as we described in Chapter 3 in details, which also qualifiedly agrees well with the neutron experimental results.

In our preliminary calculation results for magnesium alloy AZ31B, the lattice strain evolution under uniaxial loading condition is considered and plotted out for the initial textured AZ31B by using the similar activated slip and twinning modes and the

corresponding hardening parameters in the EPSC model in Agnew et al. (2003)

Quantitatively our results agree well with their EPSC results.

Table 6.1 Slip and twinning systems in HCP materials.

Type of systems	System
Basal $\langle a \rangle$ slip	$\{0001\} \langle 11\bar{2}0 \rangle$
Pyramidal $\langle a+c \rangle$ slip	$\{11\bar{2}2\} \langle \bar{1}\bar{1}23 \rangle$
Pyramidal $\langle a \rangle$ slip	$\{1\bar{1}01\} \langle 11\bar{2}0 \rangle$
Prism $\langle a \rangle$ slip	$\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$
Tension twinning	$\{10\bar{1}2\} \langle \bar{1}011 \rangle$
Compression twinning	$\{10\bar{1}1\} \langle \bar{1}012 \rangle$

Table 6.2. Three sets of hardening parameters used in the hcp crystal plasticity model.

Deformation system	Case-1 τ_0 (MPa)	Case-1 τ_s (MPa)	Case-2 τ_0 (MPa)	Case-2 τ_s (MPa)	Case-3 τ_0 (MPa)	Case-3 τ_s (MPa)
Basal	215	225	200	230	210	240
Pyramidal <a+c>	230	240	250	260	260	270
Pyramidal <a>	110	140	110	150	120	160
Prism <a>	110	120	110	120	110	120
Tensile twin	n/a	n/a	220	230	220	220

Table 6.3 Comparison of different notations for slip and twining systems in h.c.p.
materials.

	Basal	Pyramidal <a+c>	Pyramidal <a>	Prism <a>	Second order pyramidal <a+c>
Miller-bravais	{0001}	{10 $\bar{1}$ 2}	{10 $\bar{1}$ 1}	{10 $\bar{1}$ 0}	{11 $\bar{2}$ 2}
Miller index	{001}	{102}	{101}	{100}	{103}

Table 6.4 Parameters calibrated by the best fitting to the neutron data.

	Basal <a> slip MPa	Pyramidal <a+c> slip MPa	Pyramidal <a> slip MPa	Prism <a> slip MPa	Tension twinning MPa
τ_0	200	200	190	110	110
τ_s	220	240	200	150	110

Table 6.5 Fitting parameters that are consistent with Agnew's EPSC model
(2003).

	Basal <a> slip MPa	Pyramidal <a+c> slip MPa	Pyramidal <a> slip MPa	Prism <a> slip MPa	Tension twinning MPa
τ_0	0.78	n/a	n/a	55	30
τ_s	0.78	n/a	n/a	80	30

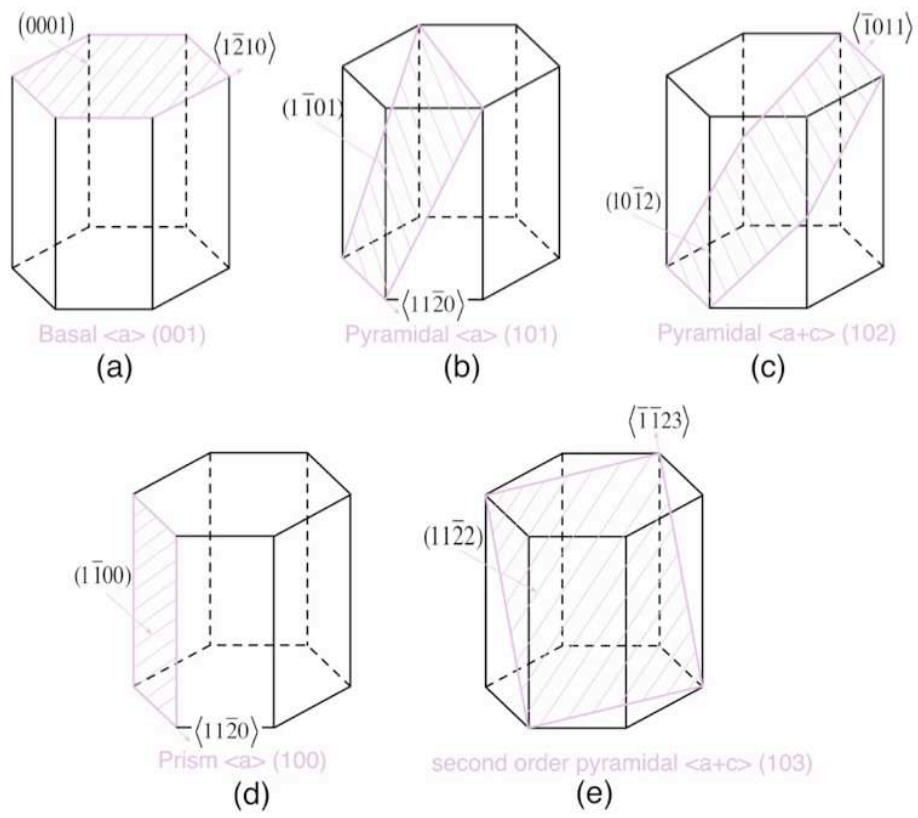


Figure 6.1. Possible slip systems in hexagonal structural materials.

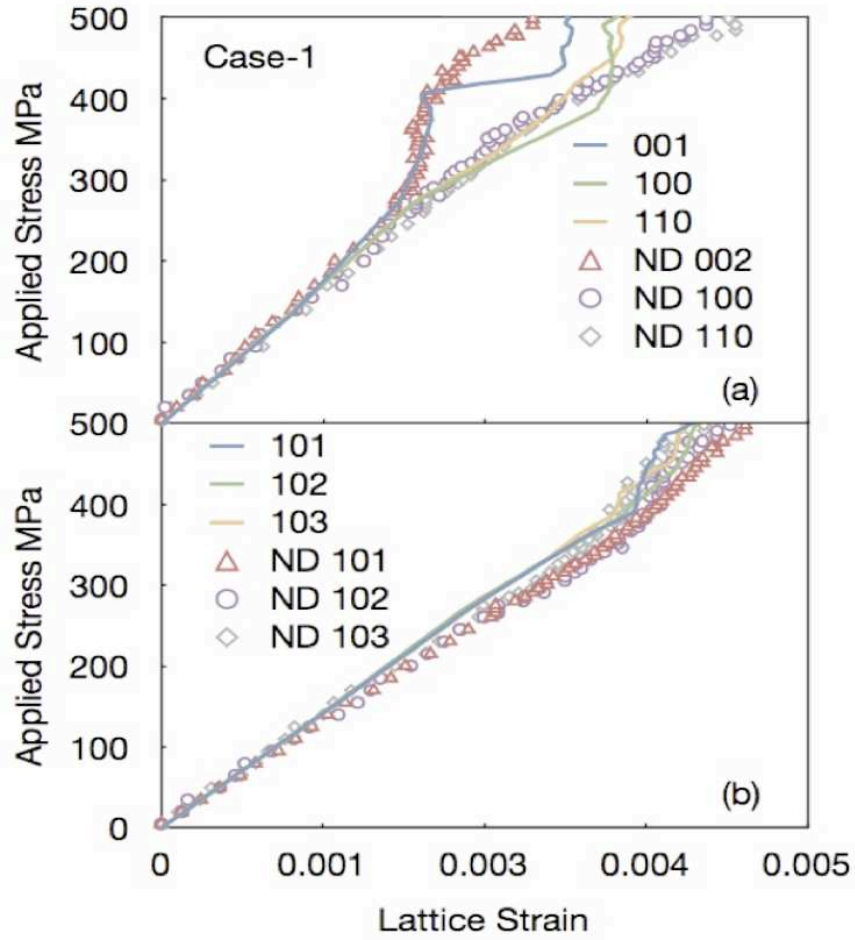


Figure 6.2. Lattice strain evolution and comparison to the neutron diffraction data with the hardening parameters (Case 1).

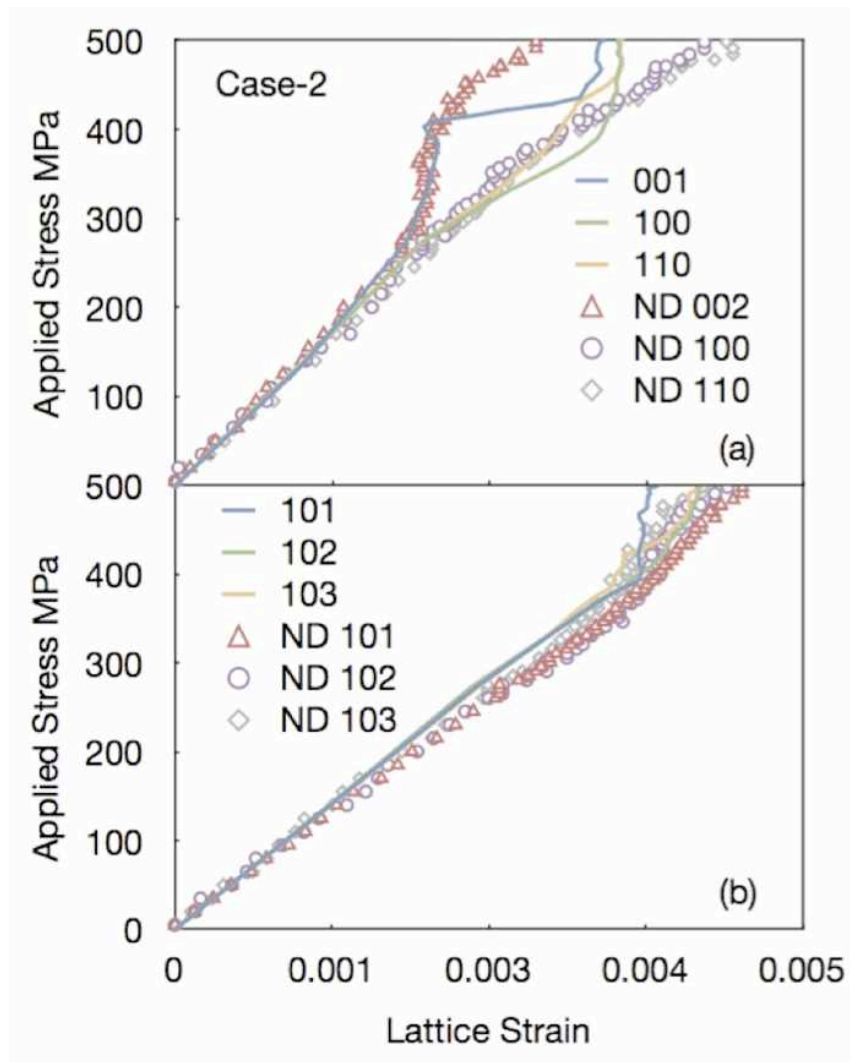


Figure 6.3. Lattice strain evolution and comparison to the neutron diffraction data with the hardening parameters (Case 2).

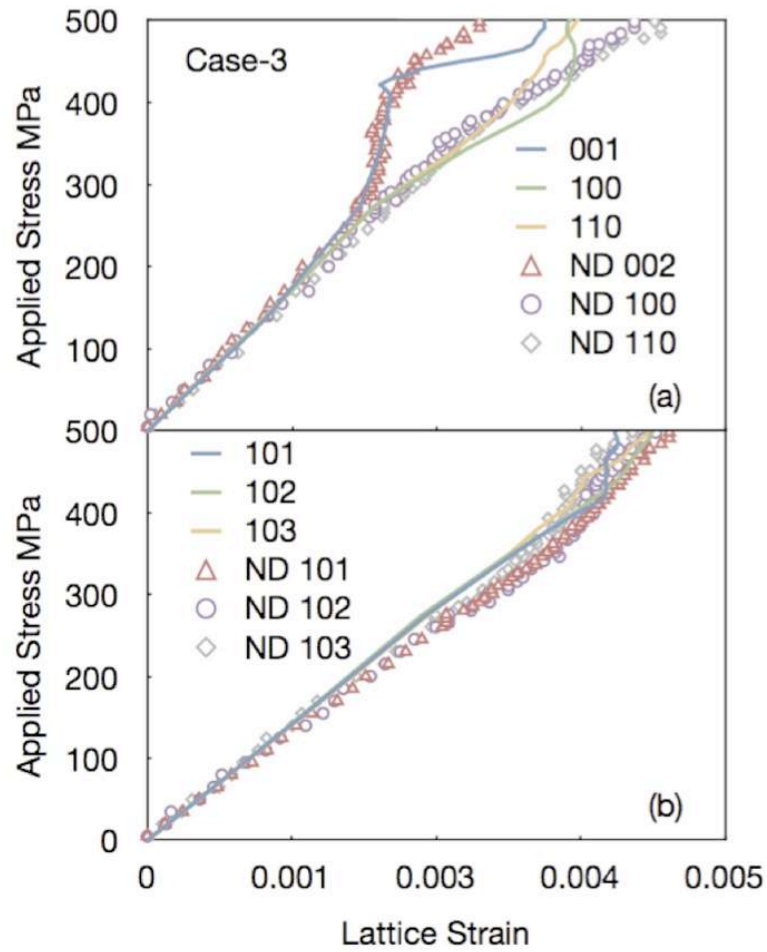


Figure 6.4. Lattice strain evolution and comparison to the neutron diffraction data with the hardening parameters (Case 3).

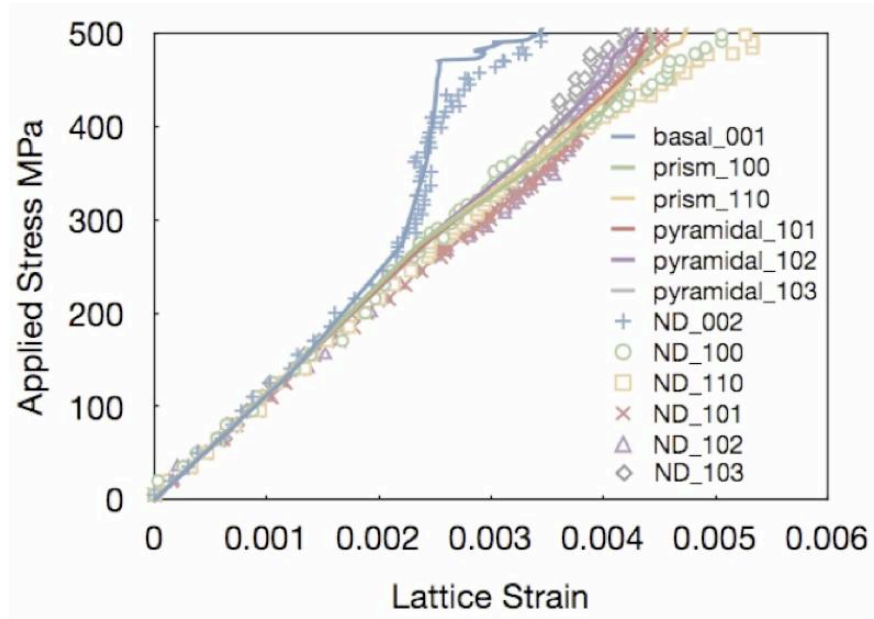


Figure 6.5. Lattice strain evolution during uniaxial loading for Zr-4. The hardening parameters we used here for the slip/twin system are calibrated by the best fitting to the neutron data.

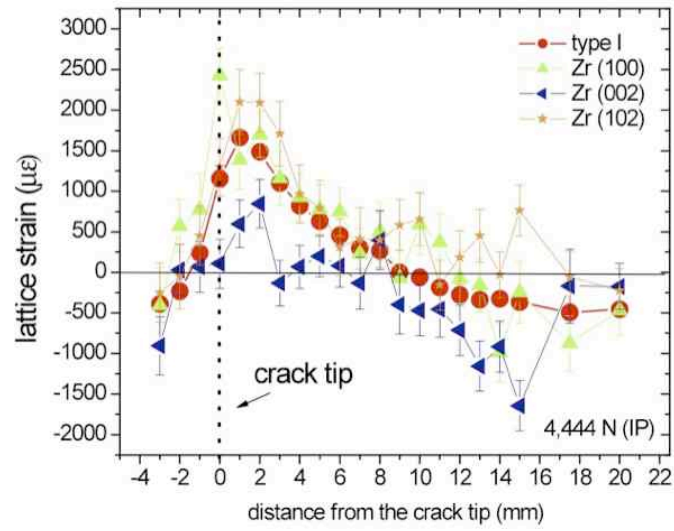
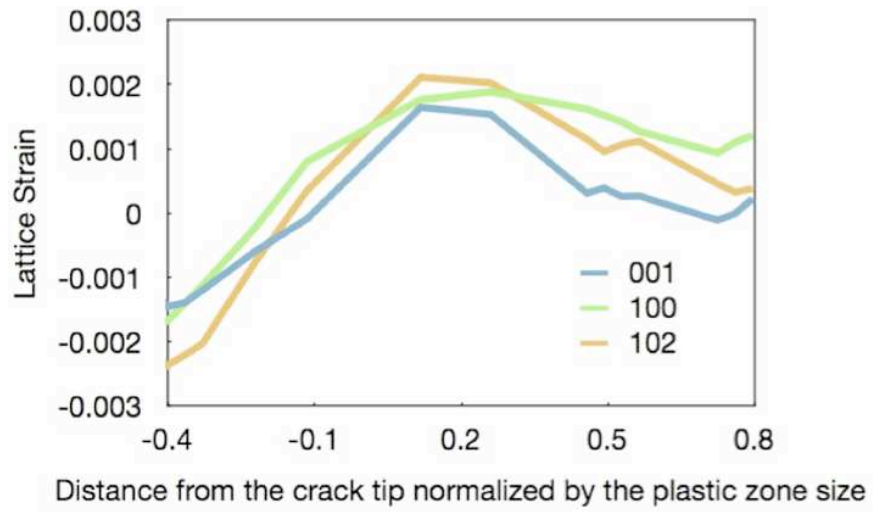


Figure 6.6. Lattice strain distribution around the fatigue crack tip. (a) From the finite element simulation; (b) from neutron diffraction experiments.

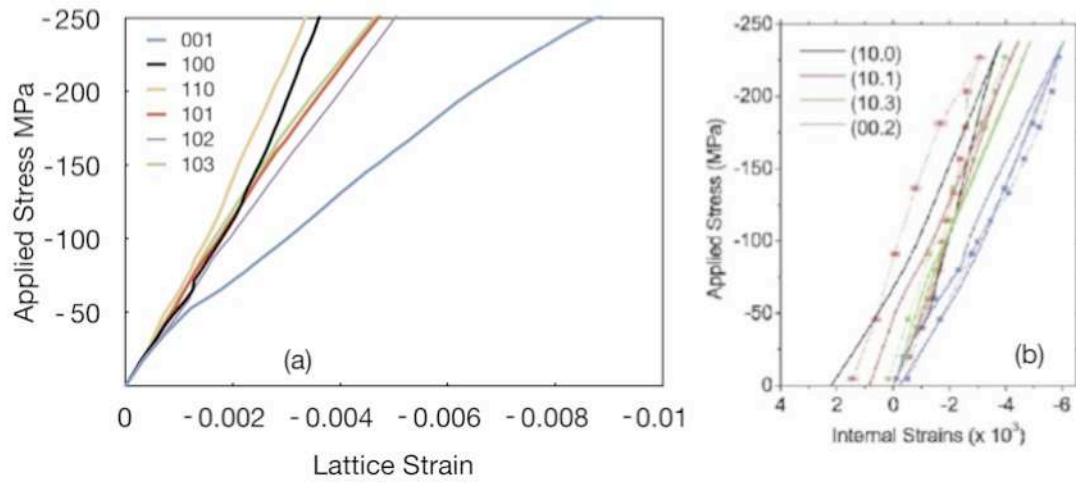


Figure 6.7. Lattice strain evolution during uniaxial compression test: (a) finite element simulation results in this work; (b) predictions by the EPSC model from Agnew et al. (2003).

Chapter 7

Conclusions and suggestions for future work

This thesis has performed theoretical and computational studies on crystal plastic deformation behavior at microstructure length scales and compared extensively to advanced diffraction measurements.

[1]. Deformation behavior near a fatigue crack tip is studied experimentally by spatially resolved, *in situ* measurements of lattice strains using neutron diffraction technique for HASTELLOY C2000 alloy, as well as computationally by macroscopic fatigue simulation based on a hysteretic, irreversible cohesive interface model and by microscopic crystal plasticity simulation. Lattice strain distributions in the vicinity of the fatigue crack tip are very sensitive to the history of stress and its multiaxiality. The stress state can be varied by the load pattern (e.g., mode mixity, overload, etc.) and material properties (e.g., cyclic hardening behavior). The lattice strains are largely dependent on the slip system and material microstructure. Therefore, neutron or synchrotron x-ray diffraction studies near fatigue crack tips can lead to salient understandings of material deformation behavior. By comparing the neutron diffraction experimental results (with the presence of a real, “messy” tip process zone) to the numerical simulations (where the cohesive zone serves as an artificial tip process zone), we find that the real tip process zone leads to vanishing intergranular strains and thus is governed by the intergranular damage. This is further supported by previous synchrotron x-ray studies, which found high density of geometrically necessary dislocations, severe intragranular deformation, and micro-cracks near the fracture surface.

[2]. We have developed a microscopic study, which is based on the crystal plasticity theory, to elucidate the grain-orientation-dependent intergranular strain anisotropy of FCC structural superalloys during cyclic loadings. Stimulated by the previous work, the grain-orientation-dependent residual lattice strain anisotropy can be an important symbol of the failure process during fatigue loadings instrumentally by neutron diffraction technique. This work, on the other hand, from the simulation point of view, gave a theoretical explanation about the anisotropy behavior, which has been proved to be determined by the strength-to-stiffness ratio of the specific crystallographic plane being considered.

[3]. The procedure to determine the lattice rotation field from deformation gradient is presented and the misorientation patterns with varying initial crystallographic orientations are analyzed numerically by finite element simulations. These simulations suggest a kinematic relationship between the lattice misorientations and slip fields, while more detailed comparisons to experiments are needed.

[4]. The deformation mechanism for HCP polycrystals, Zr-4 alloy and AZ31B alloy, are examined by the crystal plasticity based constitutive model. The model can nicely predict the lattice strain evolution under both uniaxial and fatigue loading conditions. For Zr-4 alloy by activating basal, pyramidal $\langle a+c \rangle$, pyramidal $\langle a \rangle$ and prism $\langle a \rangle$ slip and the tensile twin, the calculating results fit the neutron diffraction experimental results fairly well. However, we believe it is just one of the possible fitting parameters, which can lead to the agreement with the neutron diffraction experimental results.

[5]. Magnesium alloy AZ31B agree quantitatively well with the available EPSC results in literature.

The future direction along this line would be the fatigue behavior of textured Magnesium alloy, the lightest alloy with high strength, which is highly interested by the aerospace or auto industry. As we know that textured magnesium alloy exhibits yield anisotropy and asymmetry, Hill's model is ineffective in this case, because it was meant for cubic metals and thus was represented by the even function of stresses, which cannot describe the yield asymmetry. Thus we will develop a user material subroutine to describe the macroscopic anisotropic yield criterion based on Barlat's work in 2004 (Cauzacu and Barlat, 2004), which involves a material parameter expressed in terms of both the tensile and compressive yield stresses. We believe that the anisotropic and asymmetric yield criterion can faithfully reproduce the surrounding plastic deformation behavior induced by the fatigue crack growth in wrought magnesium alloys.

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Vita

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