

**In Situ Diffraction Experiments and Multiscale Mechanics Modeling of
Fatigue Behavior in Mg Alloys**

**A Dissertation Presented for the
Doctor of Philosophy
Degree
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DEDICATION

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

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ABSTRACT

The accelerated adoption of magnesium alloys as structural components in the automobile and aerospace industry is driven by their unique properties of low density, high strength-to-weight ratio, and high specific stiffness. In spite of many potential applications due to their superior mechanical properties, magnesium alloys still find many practical restrictions primarily due to our limited knowledge of their failure mechanisms. In-situ and non-destructive diffraction measurements on the microstructural scales are critical in understanding the fatigue crack behavior, which has greater advantages than the macroscopic measurements based on replica technique and the small-scale mechanical testing that cannot easily provide a complete view on the synergy of many scale-dependent deformation and failure mechanisms.

The primary objective aims to identify the roles of the deformation dynamics during fatigue experiments of Mg alloys from in situ diffraction and micromechanical modeling studies, which allows us to bridge the gap between microscopic failure processes and macroscopic fatigue properties. In the present research, the following tasks have been investigated: (1) Deformation mechanisms of Mg alloys under uniaxial loading condition, which was investigated by the state-of-the-art in situ neutron diffraction measurements at the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL). (2) Plastic anisotropy near the fatigue crack tip, which was studied through the full-field mapping around fatigue cracks by the high-energy synchrotron X-ray diffraction at the Advanced

Photon Source (APS), Argonne National Laboratory (ANL), coupled the cohesive interface model and crystal-plasticity model.

The principal outcome of this research will be an improved understanding of deformation dynamics and fatigue mechanisms at inter- and intra-granular scales, and a predictive capability based on the microstructural level understanding with which materials scientists can improve the practical applications of Mg alloys.

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CHAPTER I

INTRODUCTION

The past achievements of metallurgy and mechanics of materials have delivered great combinations of properties, including high strength and toughness, and degradation resistance under extreme thermal, mechanical, chemical, and irradiation environments. Yet one class of new materials, which is of critical practical importance and not fully understood, is the lightweight metallic alloys. Of particular interests are Mg alloys, which are the lightest among all the commercial structural materials. Mg alloys have been extensively investigated in the past due to their unique properties of low density ($2/3$ that of aluminum and $1/4$ that of iron), high strength-to-weight ratio, and high specific stiffness. Detailed knowledge on the fatigue behavior and lifetime prediction of Mg alloys as structural materials is vital for the use of these materials as structural components. The limited knowledge of in-situ, non-destructive microstructural evaluation is a major roadblock to the technological viability of this class of lightweight materials.

1.1 Motivation and objective

Fracture and fatigue cracks have multiscale characteristics, as shown in Figure 1.1 [1-5]. The successful development of high-toughness metals, ceramics, and composites relies on various mechanisms that resist crack growth or shield the crack from stress singularity at the crack tip. Examples include crack branching, fiber pull-out as a bridging mechanism, phase transformation, and plastic deformation in the surrounding material [6].

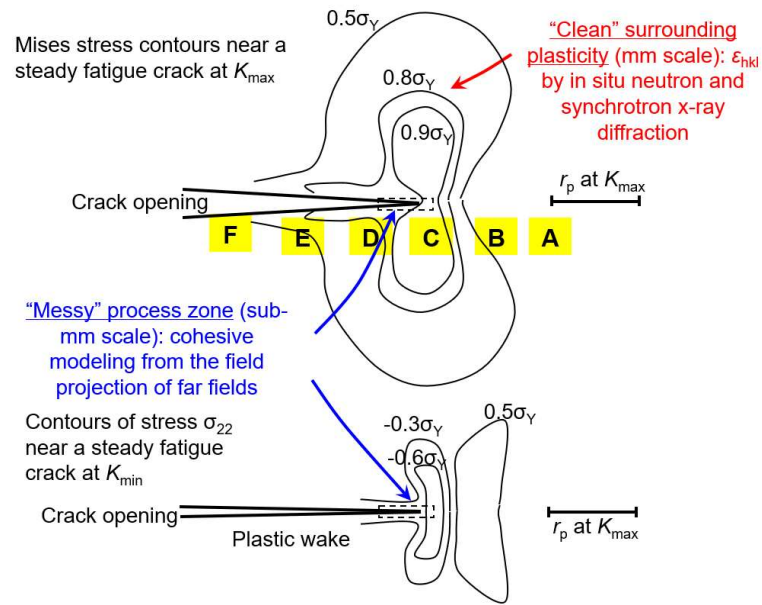


Figure 1.1. Multiscale nature of fatigue crack-tip process zones, where the stress multi-axiality varies significantly from A to F. Contours are based on Mises plasticity, while those for Mg alloys might be very different.

The deformation near the fatigue crack tip can be delineated into a “messy” (largely unclear) process zone comprising the micromechanical fatigue damage mechanisms, and a surrounding “clean” (well-studied) region dominated by continuum plasticity.

First, we note in Figure 1.1 that along the crack path for points A to F, the multiaxial stress states are quite different. Using the J_2 plasticity model as an example [5], the stress multiaxiality becomes increasingly more complex as it transitions from a biaxial tensile stress state in the linear elastic region (e.g., point A), to a triaxial stress state in the plastic zone region (points B and C), and to a biaxial stress state in the plastic wake region (points D, E, and F). Consequently, a material point in the vicinity of a fatigue crack tip will experience a complex history of multiaxial stress states, which has significant implications on the plastic deformation of Mg alloys at the macroscopic scale (e.g., texture design leading to anisotropic plasticity that potentially resists the crack growth in certain directions) and the mesoscale (e.g., deformation of a finite number of grain aggregates around the crack tip to accommodate the multiaxial stress fields). A full-field strain mapping to understand the above complex stress history in the “clean” plastic zone is essential for the fatigue-crack-growth studies in Mg alloys. Second, the “messy” process zone that dictates the damage and irreversible micromechanical mechanisms dominate over length scales that are not amenable to in situ and nondestructive evaluations. A phenomenological cohesive zone approach that helps generate steady fatigue crack models can be used to simulate the failure processes in this “messy” zone.

The proposed research aims to combine theory, modeling, and experiments to investigate the roles of the surrounding plasticity and fracture-process zone in fatigue experiments of Mg alloys. Specifically, we seek to quantify the relationship between the fatigue-growth resistance and the stress multiaxiality that controls the texture evolution and plastic anisotropy.

1.2 Framework

Our main hypotheses on the fatigue behavior include: (1) the deformation near the fatigue crack tip can be delineated into a “messy” process zone comprising the micromechanical fatigue damage mechanisms, and a surrounding “clean” region dominated by continuum plasticity; (2) the “clean” plastic zone requires knowledge of the Mg alloy deformation mechanisms under complex multiaxial stress histories, which can be quantified from in situ neutron and synchrotron X-ray studies; (3) The quantitative characterization of “messy” process zone is extremely challenging and not easily amenable to traditional experimental methods, and cohesive zone failure models are typically used to simulate the failure processes in this “messy” zone. Figure 1.2 outlines the integrated experimental and computational framework. The synergy between experiments and modeling is focused on the following two scientific topics:

- (a) Deformation mechanisms of Mg alloys under uniaxial loading condition, which was investigated by in situ neutron diffraction measurements at the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL):

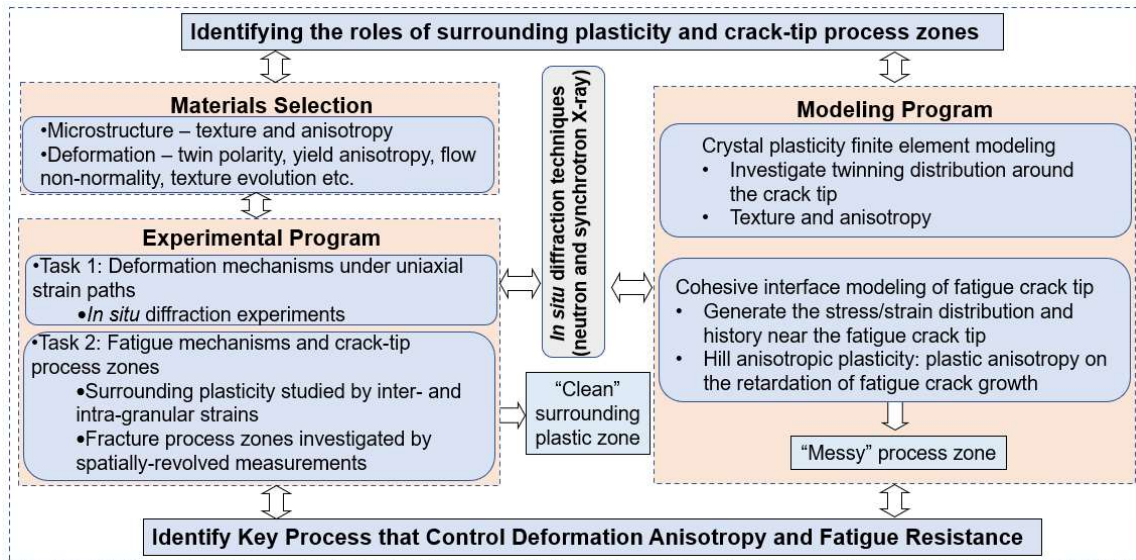


Figure 1.2. Summary of the proposed research.

The yield and flow characteristics of Mg alloys are texture-dependent due to the complicated deformations mechanisms. Neutron beams are highly penetrating so that they can be used as a nondestructive probe in real-time measurement, which can provide the texture evolution and lattice strain evolution at grain levels. The objective of this task is to:

- (1) Investigate the mechanical behavior of Mg alloys under uniaxial loading path, including tension/compression combined with in-situ diffraction experiments.
 - (2) Explore the deformation dynamic during the cyclic loading.
- (b) Plastic anisotropy near the fatigue crack tip during the fatigue-crack-growth test in Mg alloys, which was performed at high-energy synchrotron X-ray diffraction at the Advanced Photon Source (APS), Argonne National Laboratory (ANL):

It has been widely recognized that metallic materials are generally toughened intrinsically by inducing a significant plastic-zone size ahead of the crack tip, and the shape and size of the plastic zone play an important role in influencing the fatigue crack extension. It is thus of great technical importance to determine the effect of microstructure, texture, and slip/twin modes on the development of the plastic zone during the fatigue crack growth, which is termed as the “clean” process zone in this work. The objective of the task is to:

- (1) Investigate fatigue crack growth behavior of Mg alloys by in situ synchrotron X-ray diffraction measurements, and provide a full field mapping near the fatigue crack tip.

- (2) Develop a non-hardening modified crystal-plasticity-based constitutive model accounting for both slip and twins to address the twinning distribution around the crack tip.
- (3) Develop an irreversible cohesive interface model to simulate a steady fatigue crack, and to study the stress/strain distributions near the crack tip for anisotropic plastic solids.

The principal outcome of this research will be an improved understanding of deformation dynamics and fatigue mechanisms at inter- and intra-granular scales in Mg alloys, and a predictive capability based on the microstructural level understanding, which will help materials scientists to improve the practical applications in Mg alloys.

CHAPTER II LITERATURE REVIEW

2.1 Deformation and failure mechanisms in Mg alloys

The plastic deformation of Mg alloys is characterized by a significant tension-compression anisotropy (tension yield strength/compression yield strength, 0.3 ~ 0.6), which originate from the complicated deformation systems in the hexagonal-close-packed (HCP) structure, including a limited number of slip systems and deformation twinning [7-9]. The plastic deformation in Mg alloys at room temperature is typically controlled by the synergy of dislocation slips and deformation twinning. In principle, Mg alloys have five independent slip systems, including basal $\langle a \rangle$ slip system $\{0001\}\langle 11\bar{2}0 \rangle$, prismatic $\langle a \rangle$ slip system $\{1\bar{1}00\}\langle 11\bar{2}0 \rangle$, pyramidal $\langle a \rangle$ slip system $\{10\bar{1}1\}\langle 1\bar{2}10 \rangle$, 1st order pyramidal $\langle c+a \rangle$ slip system $\{10\bar{1}1\}\langle 2\bar{1}\bar{1}\bar{3} \rangle$, and second-order pyramidal $\langle c+a \rangle$ slip system $\{2\bar{1}\bar{1}2\}\langle \bar{2}113 \rangle$ (see Figure 2.1) [10-12]. Among them, the $\langle c+a \rangle$ slip system is the only one that can accommodate the strains along the $\langle c \rangle$ direction. However, it is difficult to activate at room temperature due to the high critical resolved shear stress [13]. These $\langle a \rangle$ -type slip systems on the basal planes in HCP materials cannot satisfy the arbitrary anisotropic deformation beyond the elastic stage, which triggers the onset of mechanical twinning to accommodate the deformation along $\langle c \rangle$ direction to satisfy the Mises criterion [14, 15]. The direction of shear associated with twinning in HCP materials is c/a ration dependent [10, 11], which can result in a strong asymmetry in mechanical response in HCP materials. For example, for materials with $c/a < \sqrt{3}$, the twinning direction is along $\langle \bar{1}011 \rangle$ direction and twin occurs with tension along $\langle c \rangle$ direction; for

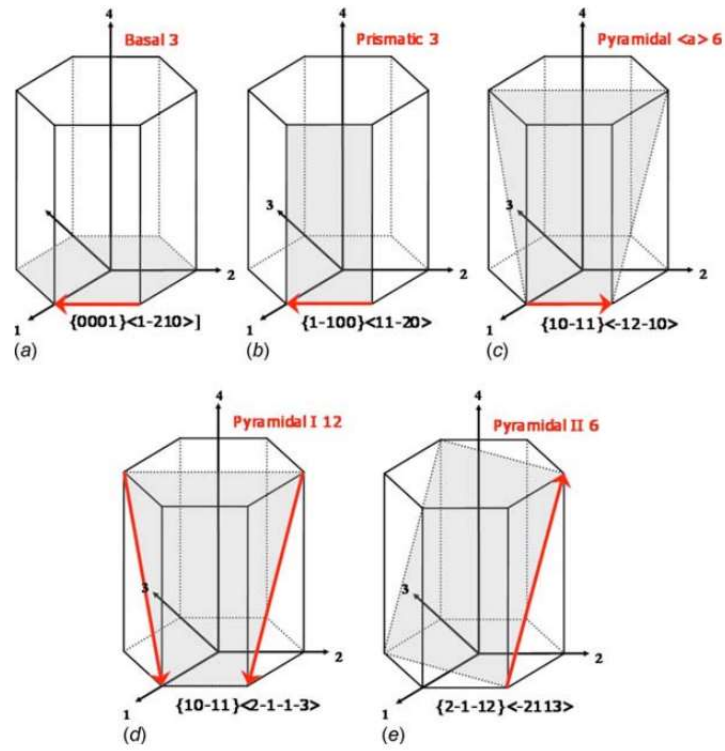


Figure 2.1. Typical slip systems in Mg alloys. [16]

materials with $c/a > \sqrt{3}$, the twinning direction is along $\langle \bar{1}012 \rangle$ direction and twin will be activated by compression along $\langle c \rangle$ direction. As the c/a ratio of Mg is 1.624, which is smaller than the ideal ratio $\sqrt{3}$, the most prominent twinning mode in Mg alloy is the $\{10\bar{1}2\}$ extension twinning (or called tensile twinning), which gives a tensile strain parallel in a direction of shear $[\bar{1}011]$ to the c -axis. The twinning shear value in Mg is 0.129. Due to the polar nature, the extension twinning in Mg alloys can be activated by tension parallel to the c -axis or the compression perpendicular to the c -axis. The occurrence of extension twinning will lead to a significant change of the texture by a sudden reorientation of the domain grains at an angle of $\sim 86.3^\circ$. Detwinning can occur during the subsequent reverse loading [17, 18]. For instance, microstructural evolution during the first fatigue cycle is given in Figure 2.2(b) for a wrought AZ31B Mg alloy [19, 20]. The microstructure of undeformed, twinned, and slightly detwinned samples are shown in these EBSD measurements, corresponding to the three strain levels in Figure 2.2(a) [19]. Neutron and synchrotron X-ray studies have been performed to probe the effects of the initial texture on the above deformation behavior [18, 19, 21-23].

Contraction twinning, including $\{10\bar{1}1\}\langle 10\bar{1}2 \rangle$ and $\{10\bar{1}3\}\langle 10\bar{1}2 \rangle$ twinning systems, can accommodate the compressive strains along the $\langle c \rangle$ direction [24, 25]. Opposite to extension twinning, the contraction twinning can be activated under compression along the c -axis or tension perpendicular to the c -axis, which also results in a lattice rotation, $\sim 56.2^\circ$, towards a more favorable orientation for basal glide [24]. However, it is usually activated at a large strain level. In addition to primary twinning systems in Mg

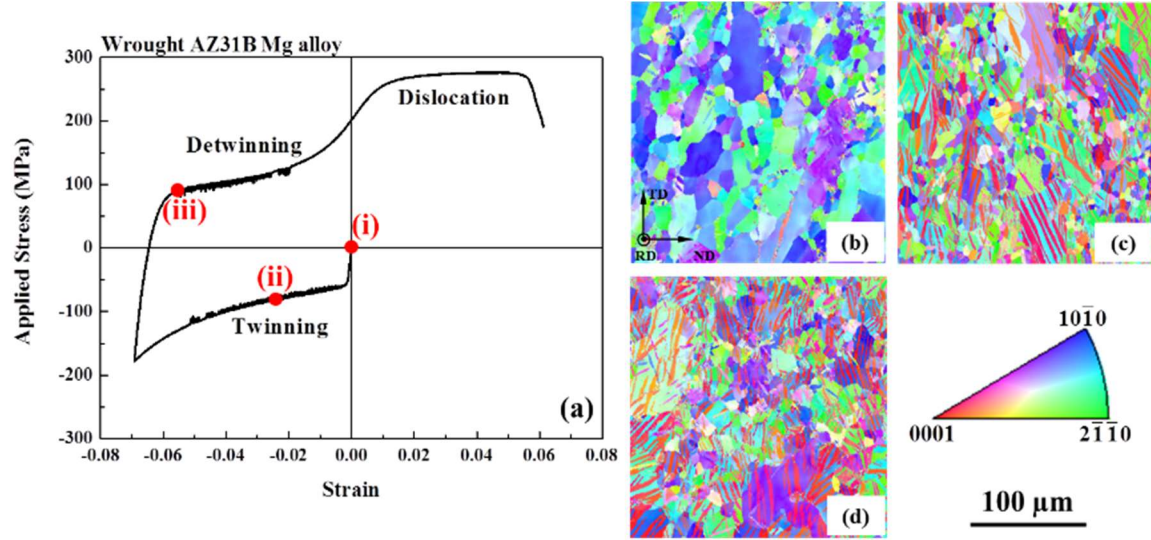


Figure 2.2. (a) Stress-strain curve for a wrought AZ31B Mg alloy. (b)-(d) Inverse pole figures in RD at (i)-(iii) in (a), respectively. [19, 20]

alloys, second twinning may take place in the primary contraction twins, which can be explained by the fact that the stress state that favors the nucleation of contraction twinning also favors the activation of secondary extension twinning within the primary twin domain. the twinning systems and corresponding re-orientation angle in Mg alloys are summarized in Table 2.1, and the grain reorientations due to different deformation twinning in the HCP-structured materials are demonstrated in Figure 2.3.

2.2 Low cycle fatigue behavior in Mg alloys

Structural components are frequently subject to cyclic deformation leading to catastrophic fracture after a certain period of time. Therefore, it is important to investigate the fatigue life of the components and to establish a reliable life prediction model. Fatigue is caused by the accumulation of small-scale defects over a number of loading cycles, including the nucleation and growth of cracks or voids, which eventually lead to failure. The fatigue behavior is strongly dependent on material microstructure (i.e., grains size, textured), specimen surface condition, and deformation history (i.e., strain path). Based on the fatigue life, fatigue can be categorized into low-cycle fatigue (LCF) and high-cycle fatigue (HCF), where the former has a low cycle number, $< 10^4$ or 5×10^4 cycles, and the strain and stress amplitudes usually exceed the elastic limit of a material, the fatigue life of the latter one requires more than 10^4 cycles to failure which implies the stress level is relatively low and the deformation is in the elastic range. For practical application, it is of importance to identify the evolution of plastic deformation mechanisms in Mg alloys during LCF, where plastic deformation occurs. LCF experiments can be performed under

Table 2.1. Deformation twinning systems in HCP-structured Mg alloy. [24]

Type of Twin	Twin Plane	Misorientation Angle
Extension twinning(ET)	$\{10\bar{1}2\}$	86.3°
Contraction twinning (CT1)	$\{10\bar{1}1\}$	56.2°
Contraction twinning (CT2)	$\{10\bar{1}3\}$	64°
Double twinning (DT1)	$\{10\bar{1}1\}\{10\bar{1}2\}$	37.5°
Double twinning (DT2)	$\{10\bar{1}1\}\{10\bar{1}3\}$	22°

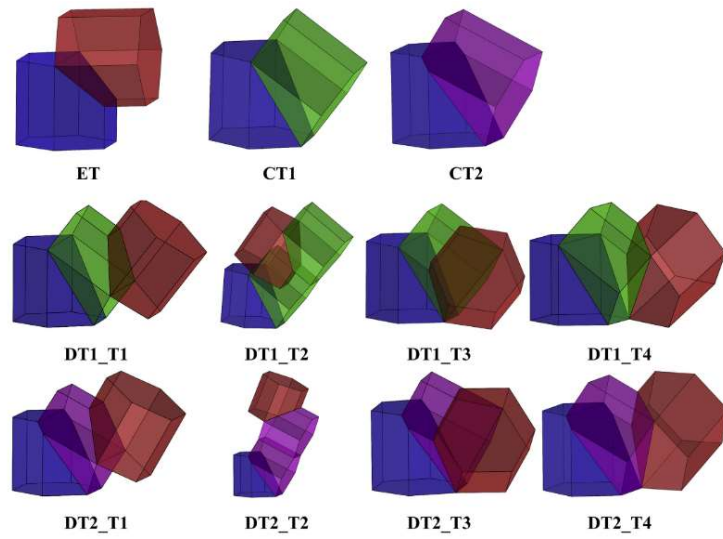


Figure 2.3. Schematics of grain reorientation due to different deformation twinning systems in HCP-structured material. [24]

stress-controlled or strain-controlled conditions but in most cases, strain-controlled conditions are used.

The peak stress is constant during a stress-controlled LCF experiment, while the peak strains during fatigue cycles can be varied due to the material work-hardening/softening behavior in the materials. The wrought Mg alloys are usually highly textured, therefore, the corresponding stress-strain hysteresis curves usually present asymmetric. Figure 2.4(a) exhibited typical hysteresis curves in a stress-controlled LCF test in an extruded AZ31 alloy [26]. The easy yielding in compression by twinning in an extruded AZ31 alloy makes the hysteresis curves asymmetric in the first cycle due to the texture built up by extrusion [26]. And the asymmetric feature gradually disappeared in the hysteresis curve at half-life with the stress-controlled cycling [26]. Chen et al. [27] examined the LCF failure behaviors of a hot-rolled AZ90 Mg alloy under uniaxial asymmetric stress-controlled cyclic experiments at room temperature. They suggested that the cracks initiated from the surface of samples and the kinked residual twins deteriorated the grain boundary. The number of residual twins decreased with the increased stress ratio and decreased peak stress [27].

For the strain-controlled LCF test, the total strain amplitude is constant throughout the fatigue lifetime with various peak stress due to cyclic hardening/softening. Begum et al. [28] investigated the cyclic deformation characteristics and evaluate strain-controlled LCF behavior of a recently developed AM30 extruded Mg alloy. The total strain amplitude can affect the hardening behavior in the sense that the cyclic stress amplitude almost

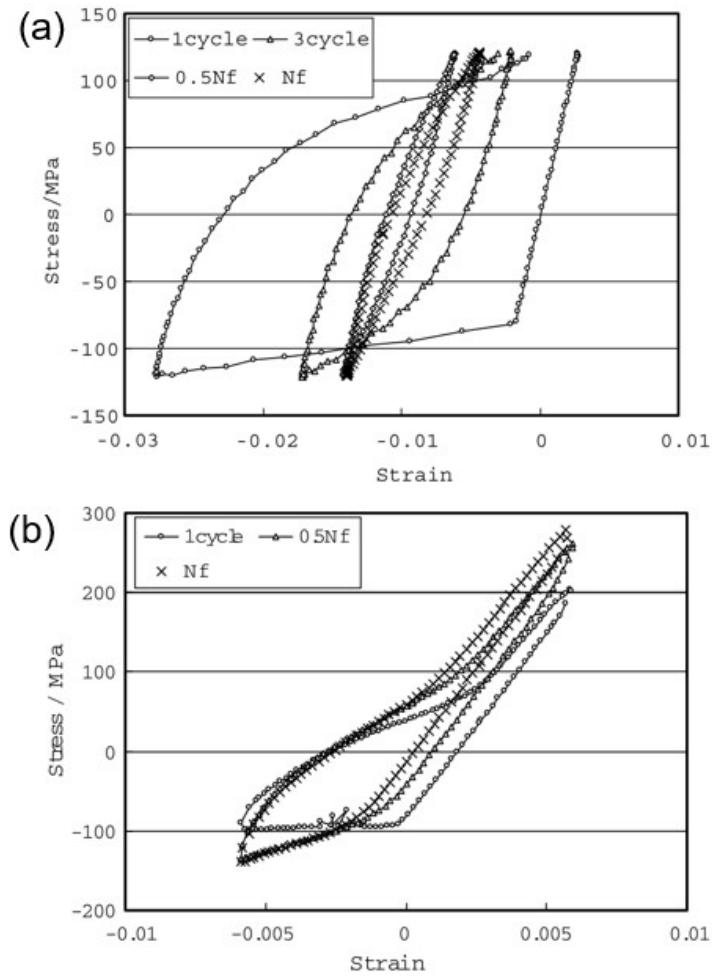


Figure 2.4. Hysteresis curves of representing cycles in a stress-controlled LCF in an extruded AZ31 Mg alloy. [26]

remained constant at low strain amplitudes, while at higher strain amplitudes cyclic hardening occurred until failure in the AM30 alloy [28]. And the cyclic strain-hardening exponent of the alloy was found to be much higher than the monotonic strain-hardening exponent (i.e., 0.33 vs 0.15) obtained at the same strain amplitude, indicating stronger cyclic hardening capacity [28]. The significant asymmetric hysteresis loops during cyclic loading are attributed to the twinning in compression and subsequent detwinning in reverse tension [28]. As shown in Figure 2.4(b), different from the disappearance of asymmetric hysteresis curve at half-life with stress-controlled cycling in AZ31 alloy, the strain-controlled cycling retains the asymmetry till the end and produces tensile mean stresses [26].

In the realm of cumulative fatigue-damage methods, the stress-life approach is usually applied on the analysis of the HCF ($N_f > 10^4$), which creates a plot of the cyclic stress (S) against the cycles to failure (N) on a logarithmic scale. Theoretically, it is usually described by the Basquin equation [29]:

$$\frac{\Delta\sigma}{2E} = \frac{\Delta\varepsilon_e}{2} = \frac{\sigma_f'(2N_f)^b}{E} \quad (2-1)$$

where $\Delta\sigma = \sigma_{\max} - \sigma_{\min}$ is the stress range applied, $\sigma_{\max}$ and $\sigma_{\min}$ are the maximum and minimum stresses, respectively, E is Young's modulus, $2N_f$ is the number of load reversals to failure, σ_f' is the fatigue-strength coefficient, and b is the fatigue-strength exponent. The strain-life approach (the ε -N curve method) is usually applied to assess for the LCF, where a logarithmic plot of the cyclic strain (ε) versus the cycles to failure (N) is used to represent

fatigue data. The Manson-Coffin formula below is frequently used to describe low-cycle fatigue [30, 31]:

$$\frac{\Delta \varepsilon_p}{2} = \varepsilon_f' (2N_f)^c \quad (2-2)$$

where $\Delta \varepsilon_p$ is the plastic strain range, ε_f' is the fatigue-ductility coefficient, and c is the fatigue-ductility exponent. The strain-life curves for Mg alloys are reported to transition from LCF regime to HCF regime smoothly, which can be described well by a relation unifying Equations (2-1) and (2-2) through the decomposition of the total strain into the elastic and plastic components:

$$\frac{\Delta \varepsilon_t}{2} = \frac{\sigma_f' (2N_f)^b}{E} + \varepsilon_f' (2N_f)^c \quad (2-3)$$

Previous studies [32-35] generally employed ex-situ texture measurements using laboratory X-ray and ex-situ microstructural fractography examination. In wrought Mg alloys, twins can be activated in through-thickness tension or in-plane compression, which can be confirmed by postmortem microstructural observations. However, when a deformed sample is unloaded, some new twins will be detwinned. It is almost impossible to know when and where the detwinning process starts during unloading without a real-time measurement technique. Surface measurements, such as the EBSD images in Figure 2.2, have difficulties in deciphering the detwinning behavior [36-38]. In situ neutron and synchrotron X-ray diffraction methods offer us a great opportunity to fill in this knowledge gap by establishing the real-time relationship between microstructures and mechanical properties. Recently, comprehensive studies have been performed to understand the LCF performance of wrought magnesium alloys using the in situ neutron diffraction technique

[18, 21-23, 39, 40]. The plastic deformation of strong textured wrought magnesium alloys at room temperature during cyclic loading is dominated by the alternate twinning and detwinning mechanisms, which contribute to the significant asymmetry in the hysteresis loops.

2.3 Fatigue crack growth behavior

Linear elastic fracture mechanics (LEFM), to date, are still the most prevalent approach for a look into the subcritical crack growth in terms of parameters that capture the local conditions at the crack tip, considering the crack length, externally applied load, and geometrical parameters. This method is detailed in the American Society for Testing and Materials (ASTM) E647 [41]. Figure 2.5 represents a schematic diagram for the general nature of fatigue-crack growth in metallic materials and its description using fracture mechanics [42]. The da/dN - ΔK plot is in a sigmoidal shape and bounded by the threshold stress intensity factor range, ΔK_{th} , and the critical cyclic fracture toughness of the material, ΔK_c . In general, the crack-growth rate versus the stress intensity factor curve typically can be divided into regions, as shown in Figure 2.5. In region I, when ΔK is below ΔK_{th} , cracks will not grow, which suggests the existence of a fatigue limit. As long as ΔK approaches ΔK_{th} , the crack growth rate will suddenly increase. The relationship between ΔK_{th} and the fatigue limit, σ_{af} , can be expressed in the following equation [43]:

$$\sigma_{af} = \frac{\Delta\sigma}{2} = \gamma \frac{\Delta K_{th}}{2\sqrt{\pi a}} \quad (2-4)$$

where γ is a scaling factor, a is the crack length. In region II, the crack-growth rate reaches a steady-state value, which follows the Paris' crack-growth law given as [44]:

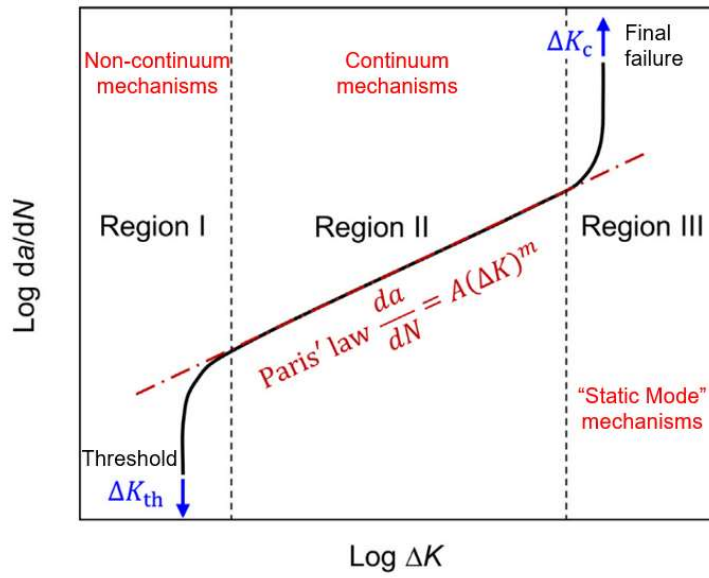


Figure 2.5. Schematic representation of the crack-growth rate as a function of the stress intensity factor range for long cracks, where ΔK_{th} and ΔK_c represent the threshold stress intensity factor range and the critical cyclic fracture toughness, respectively, and region II is described by the Paris' law.[42]

$$\frac{da}{dN} = A(\Delta K)^m \quad (2-5)$$

where A and m are material constants, $\Delta K = Y\Delta\sigma\sqrt{\pi a}$ with Y being a geometry factor depending on the loading and cracked body configuration. In Region III, the crack growth rate will increase dramatically till to fracture.

It is worth noting that the LEFM working well for brittle materials has its limit, i.e., it neglects the underlying competition between the intrinsic damage mechanisms occurring ahead of the crack (i.e., alternating crack-tip blunting and resharpening) that usually promote crack extension ahead of the tip, and the extrinsic shielding mechanisms occurring primarily behind the crack tip that impede fatigue growth (i.e., crack closure and bridging) [45] (Figure 2.6 [6]). In metallic materials, intrinsic damage mechanisms comprise the microcracks or voids initiation, growth, and coalescence in the stress concentration regions; repetitive blunting and resharpening of the crack tip are involved under cyclic loads [46]. The intrinsic mechanisms are an inherent property of the material, which are independent of the crack length or the specimen geometry. Extrinsic shielding mechanisms usually originate from the creation of inelastic zones surrounding the crack wake or form physical contact between the crack surfaces via wedging, bridging, sliding [47]. For ductile metallic materials, the plastic zone ahead of the crack tip induced by the dislocation motion will toughen the materials intrinsically, which may have a more significant influence on the retardation of the crack growth, compared to the crack closure during cyclic loading. The plastic zones are sufficiently large compared to microstructural dimensions.

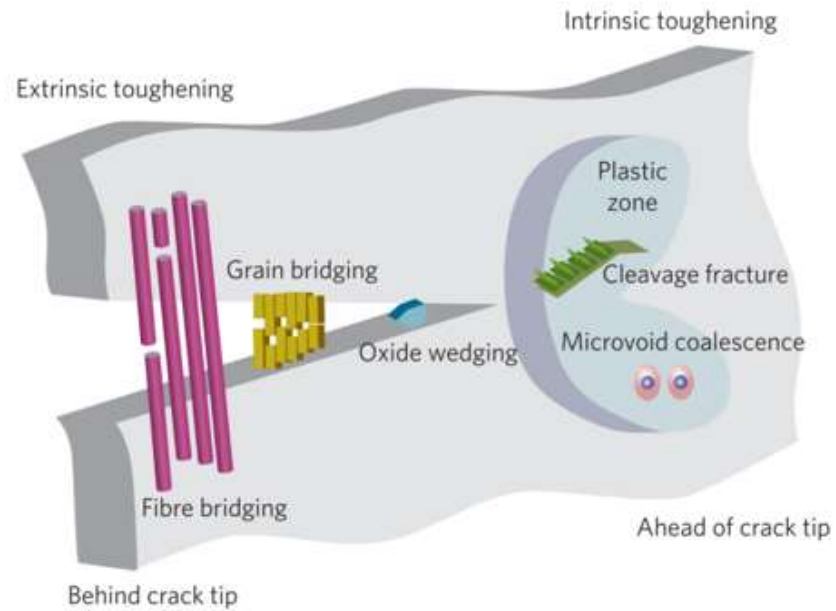


Figure 2.6. Schematic illustration of mutual competition between intrinsic mechanisms of damage/crack advance and extrinsic mechanisms of crack-tip shielding involved in crack growth. [6]

The fatigue crack growth rates in metallic materials depend on numerous factors, such as stress-tensity range, temperature, load ratio R , frequency, environment, reverse compressive stress. The deformation field near a crack tip includes a plastic zone in front of the crack tip and a plastic wake behind the crack tip, and the compressive stress/strain is developed due to prior plastic deformation before unloading. In general, the fatigue crack propagation rate of wrought Mg alloys increases with reducing frequency (or strain rate) and increasing load ratio R , which may be associated with the thickness of oxide films on the fracture surfaces or the crack closure effect [48]. In order to improve fatigue crack retardation, several methods have been applied, i.e., grain refinement, residual compressive stress [49], crack branching [50], fiber pull-out [51], phase transformation [52], and plastic deformation in the surrounding materials [53].

Due to the strong anisotropic mechanical response in Mg alloys [54, 55], the development of the plastic fields surrounding the fatigue-crack tip is markedly sensitive to the loading directions. The complexity of plastic deformation modes (dislocation, twinning, and detwinning) around fatigue crack tip of wrought Mg alloys results in the lack of fundamental understanding of surrounding plasticity. In situ diffraction technique offer us a great opportunity to monitor the microscopic deformation dynamic near a crack tip. In-situ neutron diffraction was used to investigate the twinning and detwinning behavior during fatigue-crack propagation on a wrought magnesium alloy, AZ31B, which suggests the reversible twinning and detwinning are the dominant deformation mechanisms near the crack tip during cyclic loading, and the development of compressive residual stresses

around the crack tip during unloading contributes to the detwinning behavior [56]. Synchrotron X-ray microscopy with high resolution provides a means of shedding new light on crack-tip phenomena allowing the fatigue resistance by plastic deformation in the “Clean” surrounding material to be quantified and fully mapped on the microscale from synchrotron X-ray diffraction [24]. The investigation about the plastic anisotropy, twin polarity, flow non-normality, and texture evolution in Mg alloys under the arbitrary stress multiaxiality, as shown in Figure 1.2 (locations A-F), is especially critical for improving our understanding of the fatigue crack growth behavior.

2.4 Cohesive interface model

Cohesive zone models are well accepted to simulate fracture behaviors of solid structures [57]. A cohesive zone is generally characterized by the relationship between the cohesive-zone tractions in equilibrium with the stress fields of the surrounding body and the cohesive-zone separations compatible with the deformation fields of the surrounding material [58]. The cohesive-zone models with various constitutive laws can be used to describe different nonlinear fracture processes, i.e., void-growth in ductile metals [1], crack bridging in quasi-brittle materials [59], crazing in glassy polymers [60], and interface decohesion between dissimilar materials [61].

In fracture mechanics, the characterization of the relationship between the loading parameter (i.e., stress intensity factor, K) to the crack extension is critical. The stress fields in an annular zone near the crack tip can be described by the product of the applied stress intensity factor, K_{appl} , the inverse square root singularity, $1/\sqrt{2\pi r}$ (with r being the radial

coordinate), and a set of characteristic fields that only depend on the polar angle, θ [5]. The deformation near the fatigue crack tip can be delineated into a “messy” process zone comprising the complicated micromechanical fatigue damage mechanisms, and a surrounding “clean” region dominated by continuum plasticity. The “clean” plastic process zone ahead of the crack tip has been well studied, as rigorous mechanistic analysis can be conducted. The “messy” process zone at the crack tip and inside the plastic process zone, however, are still difficult to quantify. The “messy” process zone dominates over a length scale that is not amenable to in situ and non-destructive evaluations.

A “top-down” approach developed by Hutchinson et al. [62] can be applied to investigate a “clean” plastic zone dominated by K-field by assuming a set of cohesive zone laws that simulate the failure process in the “messy” damage-related process zone during cyclic loading. There exists the main problem of how to develop a phenomenological cohesive interface model that allows the simulation to numerically develop a steady fatigue crack. As we know, a reversible cohesive interface model will only lead to the crack resistance curve, but not propagate the crack, while the addition of irreversibility will result in the plastic shakedown and the crack will stop growing after several cycles under constant K value. To solve this problem, several models have been developed [63-67]. Though introducing a damage variable, defined as the effective surface density of micro-defects, and specifying the evolution of this variable, Roe and Siegmund [66] proposed a cohesive law to describe the energy dissipation processes during cyclic loadings. An irreversible, hysteretic cohesive interface model for fatigue crack simulation in which the hysteresis

describes the gradual damage of the interface strength and dissipation of fracture energy has been developed by Nguyen et al. [68] to realize a steady crack growth.

Wrought Mg alloys with heavy textures will exhibit strong plastic anisotropy due to the polar nature of deformation twins. Consequently, it is anticipated that the fatigue crack growth rate might vary concerning the crack directions. It is essentially critical to investigate the effect of the mechanical anisotropy on the deformation fields near a fatigue crack for Mg alloys. For the textured materials, Hill's plasticity function is usually applied to capture the anisotropic mechanical response. Hill's 1948 yield criterion is a generalization of the von Mises function, which expresses anisotropy in materials with three orthogonal planes of symmetry, as shown in the following equation [69]:

$$f(\sigma) = \sqrt{F(\sigma_{22} - \sigma_{33})^2 + G(\sigma_{33} - \sigma_{11})^2 + H(\sigma_{11} - \sigma_{22})^2 + 2L\sigma_{23}^2 + 2M\sigma_{31}^2 + 2N\sigma_{12}^2} \quad (2-8)$$

where σ_{ij} and τ_{ij} represent the normal and shear stress components, respectively, for the local coordinate system; F, G, H, L, M, and N are constants that collectively represent the state of plastic anisotropy present. For the general stress state, Hill's plasticity is the most widely used criterion both in practice and commercial finite element software.

CHAPTER III
IN SITU MONITORING OF DISLOCATION, TWINNING, AND
DETWINNING MODES IN AN EXTRUDED MAGNESIUM ALLOY
UNDER CYCLIC LOADING CONDITIONS

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Di Xie is responsible for leading the research project, conducting the experiments, analyzing data, and writing the manuscript.

Abstract

This work investigates the microscopic deformation mechanisms of an extruded, precipitation-strengthened AZ80 magnesium (Mg) alloy subjected to strain-controlled low-cycle fatigue using in situ neutron diffraction measurements. Results demonstrate that the plastic deformation during cyclic loading is dominated by the alternating $\{10.2\}$ extension twinning and detwinning mechanisms. The observed deformation mode is strongly texture and precipitate dependent. For the initial texture, the tested material has two major texture components which result in the occurrence of extension twinning during both compression and reverse tension in the first two cycles. The prolonged detwinning process in the following cycles is proposed to relieve the shear stress field of $\{00.2\}$ grains, leading to the disappearance of twinning. The precipitation strengthening results in an increase of the critical resolved shear stress (CRSS) by ~ 33 MPa for the extension twinning in this AZ80

alloy. The synergistic effects of the initial texture, precipitation strengthening, and load sharing of various grain families and phases contribute to the complicated evolution of dominant deformation mechanisms, among which elevated dislocation activities are believed to be responsible for the relatively poor low-cycle-fatigue lifetime when compared to other Mg alloys.

3.1 Introduction

The accelerated adoption of magnesium alloys as structural components in the automobile and aerospace industry is driven by their unique properties of low density, high strength-to-weight ratio, and high specific stiffness [70-72]. Often, these structural components subject to cyclic deformation, usually suffer sudden, catastrophic failures during the service. Therefore, failure lifetime predictions of Mg alloys through a fundamental understanding of their fatigue behavior is vital for their applications as structural alloys. There is an increasing interest in the investigation of the fatigue behavior in Mg alloys [18, 22, 23, 73].

Unlike face-centered-cubic (FCC) or body-centered-cubic (BCC) metal alloys such as Cu and Ni, Mg alloys have high directional anisotropy and hard deformation mode [7, 74], resulting in limited ductility and its application. Although significant advances have been developed in fatigue mechanics, most of the previous studies on fatigue performance of Mg alloys rely on ex-situ mechanical testing, and microstructural characterizations [36, 37, 75, 76]. These ex-situ investigations cannot adequately resolve the relationship between the macroscopic mechanical behavior and microscopic mechanisms activated during cyclic

loading. Recently, comprehensive studies have been performed to understand the low-cycle fatigue (LCF) performance of wrought magnesium alloys using the in-situ neutron-diffraction technique [18, 21-23, 39, 40]. Most of the in-situ work is to identify the deformation mechanism sequence of the most widely used classes of Mg alloys, i.e., Mg-Al-Zn (AZ series) and Mg-Zn (Z series) [18, 23]. The plastic deformation of wrought magnesium alloys at room temperature during cyclic loading is dominated by alternate twinning and detwinning mechanisms [18, 23], which are strongly affected by the initial texture component as well as loading modes [22, 73]. On the other hand, one subject with increasing interest is the effect of precipitation strengthening on the major slip systems, i.e., basal slip and prismatic slip, and extension twinning system in Mg alloys [14, 15, 77-79]. Different types of precipitates are proposed to have distinct effects on the anisotropy by affecting the critical resolved shear stress (CRSS) for different deformation modes in Mg alloys [14, 15, 78]. Robson et al. [15] proposed that the presence of c-axis rod-type precipitates in the Z5 (Mg-5 wt.% Zn) alloy increases asymmetry, while precipitation of the basal plates in AZ91 alloy reduces asymmetry. Whilst those understandings are based on the monotonic loading, to date, the influence of precipitates on the cyclic fatigue behavior and potential activated deformation modes are not well established.

The above issue can be partly understood by the neutron diffraction measurements. The deep penetration capability of neutron beams enables the neutron diffraction technique to be an in-situ, non-destructive probe method to determine the crystal structures, atomic spacing, and other structural information [80, 81]. The shift of the diffraction peak position

that yields the lattice strain is related to the inhomogeneous deformation on the grain scale (or called Type-II strain), while the peak broadening corresponds to the inhomogeneous deformation inside the grains (or called Type-III strain) [81]. In this study, the state-of-the-art in-situ neutron-diffraction measurement is employed to investigate the strain-controlled LCF behavior of the AZ80 alloy strengthened by the plate-type precipitates. The goal of this study is to investigate the effects of deformation history, initial texture, and, in particular, precipitates on plastic deformation during cyclic loading. Given the complicated deformation modes in Mg alloys, attention will also be paid to the information regarding the relative significance of various twinning/detwinning/dislocation plasticity mechanisms during cyclic straining.

3.2 Experimental details

3.2.1 Material

The AZ80 Mg alloy sample used in this study was cut from a commercially extruded flat bar with the T5 temper. The crystallographic texture of the as-extruded plate was determined using the synchrotron X-ray diffraction technique at the Advanced Photon Source (APS), beamline 11-ID-C, at Argonne National Laboratory. Figure 3.1 shows the SEM image for the as-received AZ80 alloy, from which the gray precipitates (β -Mg₁₇Al₁₂) can be observed on the Mg matrix due to the aging treatment and have a common Burgers orientation relationship (OR) $[1-11]_{\beta} // [2-1-10]_{\alpha}$, $(110)_{\beta} // (0001)_{\alpha}$ with the Mg matrix [82, 83]. The content of the β -Mg₁₇Al₁₂ phase has been determined to be about 12.5% in this alloy. A dog-bone shaped sample with gauge dimensions of $\phi 8\text{mm} \times 16\text{ mm}$ was cut with



Figure 3.1. Optical microscopy (OM) image for the initial microstructure of the AZ80 alloy with T5 temper.

the longitudinal axes parallel to the transverse direction (TD) of the flat bar.

3.2.2 In-situ neutron diffraction measurements during LCF

The in-situ neutron-diffraction measurement was performed at the VULCAN Engineering Materials Diffractometer [84, 85], the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL) under a continuous-loading condition. In this study, the fully reversed strain-controlled LCF experiment was conducted at a total strain amplitude of $\pm 1\%$, at room temperature with triangular-loading waveforms. In-situ neutron-diffraction measurements were performed for the 1st, 2nd, 5th, 10th, 20th, 50th, 70th, and 90th fatigue cycles. We adopt strain rates of $7.4 \times 10^{-6} \text{ s}^{-1}$ in the 1st and 2nd cycles and $1.11 \times 10^{-5} \text{ s}^{-1}$ in the remaining cycles where in-situ neutron diffraction measurements were performed. In the other cycles, we adopt a fixed strain rate of $1 \times 10^{-2} \text{ s}^{-1}$ (1 Hz).

The sample was mounted horizontally to the MTS load frame, with the loading direction along the extruded direction (ED) and the radial direction parallel to the ND of the sample, respectively. The angle between the incoming neutron beam and the specimen was 45° . The two stationary detector banks (Bank1 and Bank2) are located at $\pm 90^\circ$ to the incoming beam, simultaneously recording two complete diffraction patterns with diffraction vectors parallel ($Q\parallel$) and perpendicular ($Q\perp$) to the loading direction. Therefore, the diffraction patterns along the axial and radial directions of the sample were recorded by Bank 1 and Bank 2, respectively. The neutron beam slits were 7 mm (horizontal) $\times$ 7 mm (vertical), with a 5 mm receiving collimator, leading to a neutron gauge total volume of 245 mm^3 . The neutron diffraction measurements were conducted under the high-

intensity mode with a chopper frequency of 30 Hz with a center wavelength of 2.7 Å, which corresponds to a lattice d-spacing range of 0.9~3.2 Å. After the measurement, the data reduction was performed, using VULCAN Data Reduction and Interactive Visualization software (VDRIVE) [86]. The neutron diffraction data were chopped into time bins with a time interval of 3 min. The lattice (internal) strains of a given orientation (hk.l) plane to the detector banks can be calculated based on the peak shift between the loaded and load-free patterns, given by:

$$\varepsilon_{hk.l} = \frac{d_{hk.l} - d_{hk.l}^0}{d_{hk.l}^0} \quad (3-1)$$

where $d_{hk.l}$ and $d_{hk.l}^0$ are the plane d-spacings in the loaded and load-free conditions, respectively. The counting time for the undeformed sample was 10 min to reduce the propagated $d_{hk.l}^0$ statistic error.

3.3 Results

3.3.1 Material texture

The crystallographic texture of the extruded plate by synchrotron X-ray diffraction is displayed in Figure 3.2(a). Different from traditional rolling and extruded Mg alloys, the initial texture of the current AZ80 alloy exhibits a combined feature of typical rolling and extrusion textures, with two major texture components, one with the basal plane of the HCP structure parallel to the normal direction (ND) and the other with the basal plane parallel to the transverse direction (TD) of the extruded flat bar. The former texture component is much stronger than the latter one. The neutron diffraction patterns for the

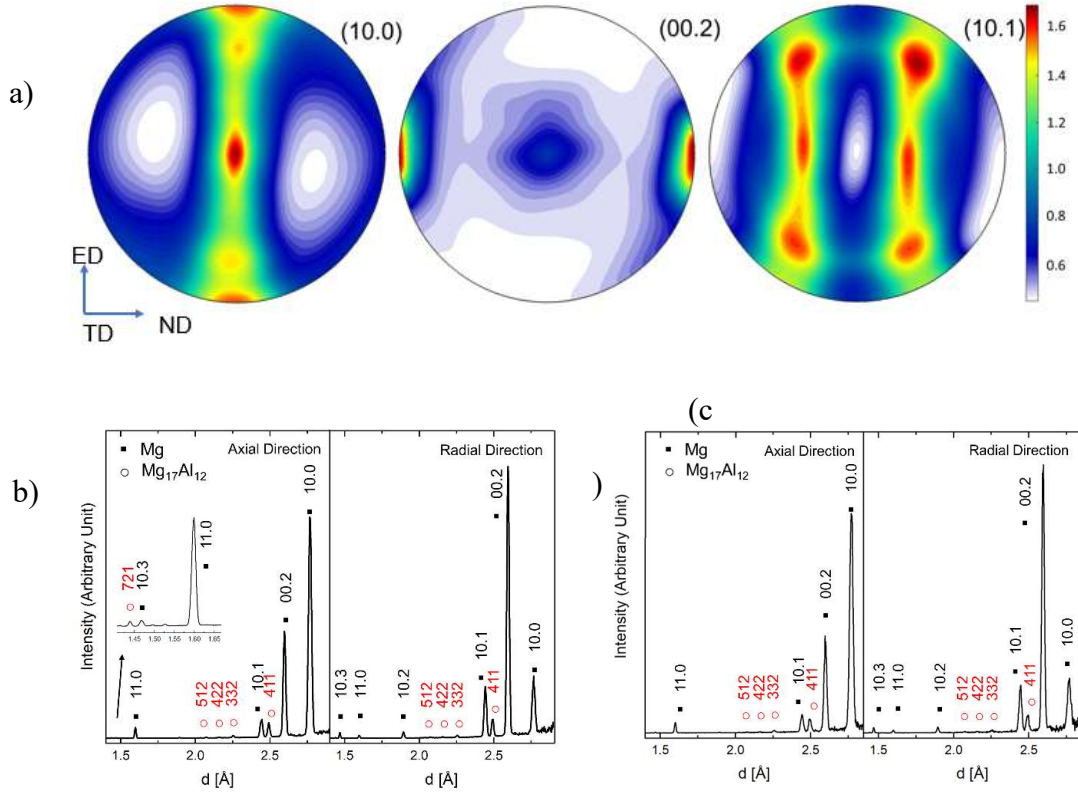


Figure 3.2. (a) The initial texture of the AZ80 alloy measured by ex situ synchrotron X-ray diffraction, which exhibits a combined feature of typical rolling and extrusion textures, with two major texture components, one with the basal plane of the HCP structure parallel to the normal direction (ND) and the other with the basal plane parallel to the transverse direction (TD) of the extruded flat bar, (b) and (c) the neutron diffraction pattern under as-received and complete tensile unloading state in the 70th cycle of the AZ80 alloy, respectively.

initial state and completely tensile unloading state in the 70th cycle are exhibited in Figures 3.2(b) and 3.2(c), respectively, from which two sets of diffraction peaks are shown: the peaks marked with the red circle belong to the β -Mg₁₇Al₁₂ phase, and the others pertain to the Mg matrix. By comparing these two diffraction patterns, no obvious difference in the relative diffraction intensity, especially for {00.2} and {10.0} peaks, are observed. This fact indicates that the texture under the tensile unloading state will recover to the initial state during cyclic straining. According to the polar nature of the twinning, the {00.2} and {10.0} grains in Bank 1 are favorably oriented for the {10.2} extension twinning in tension and compression, respectively, under current loading condition (loading direction along with the TD). It is reasonable to anticipate that the initial texture in this alloy can give rise to a more complicated deformation mode sequence during cyclic loading than that in previous studies [18, 22, 23, 56].

3.3.2 Macroscopic mechanical behavior

The hysteresis loops of the selected fatigue cycles for the in-situ neutron diffraction measurement are plotted in different colors, as shown in Figure 3.3(a). The asymmetric hysteresis loops are primarily ascribed to the formation of the {10.2} extension twins during the compressive loading and detwinning during the subsequently reversed loading [18, 23]. Figure 3.3(b) exhibits the peak stress and 0.2 % proof stress in each fatigue cycle. Slight strain hardening is observed on the tensile peak stress evolution, and the compressive peak stress is almost saturated. Note that the 0.2% proof stress in the reverse tension side

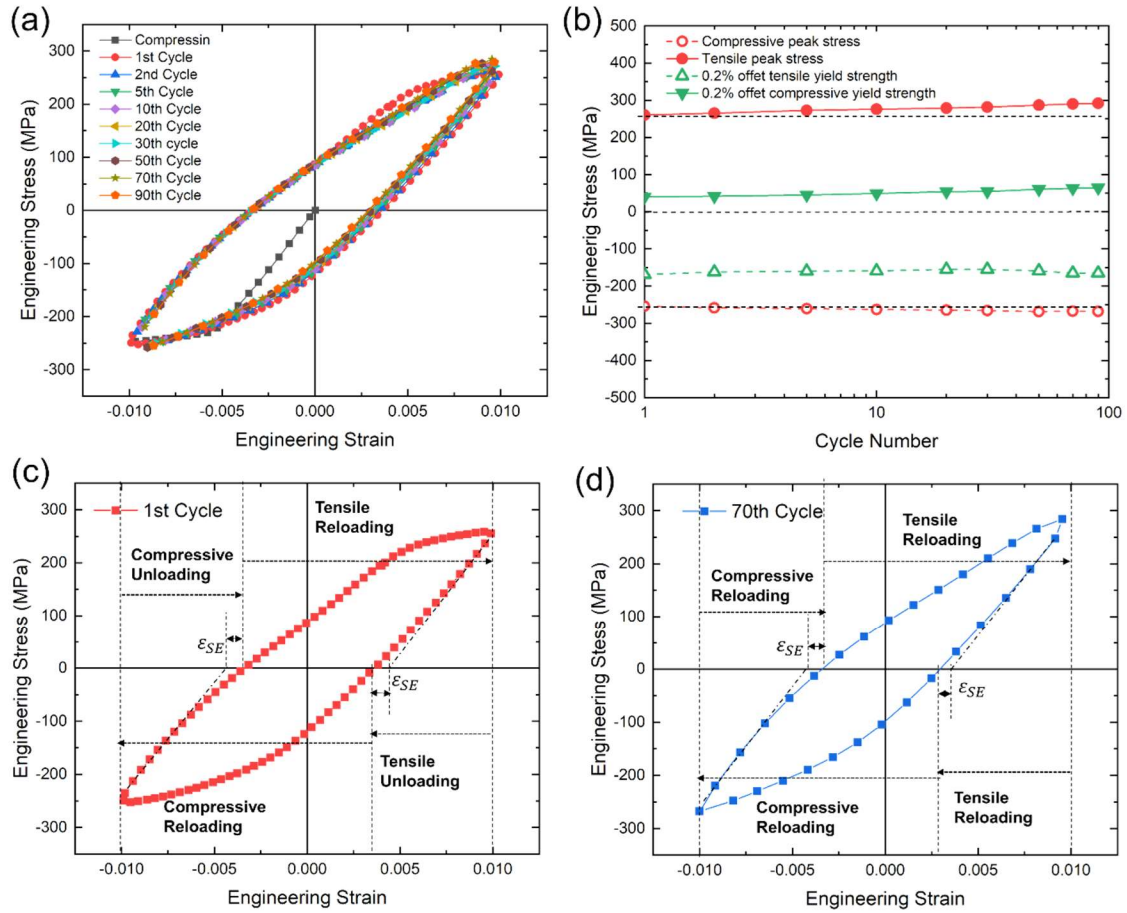


Figure 3.3. (a) Asymmetric hysteresis loops loaded along the prior transverse direction for selected cycles, (b) the peak and 0.2% offset yield stress as a function of fatigue cycles, (c) and (d) the hysteresis loops of the 1st and 70th cycle, respectively, which are divided into four loading stages: compressive loading, compressive unloading, tensile reloading, and tensile unloading.

exhibits a much smaller value than that in the compression side in each fatigue cycle, which can be explained by the early onset of detwinning [18]. The mechanical stress-strain responses in the 1st cycle and 70th cycle are displayed in Figures 3.3(c) and 3.3(d), respectively. Small pseudo-elastic strains are observed upon tensile and compressive unloading, which are reported to be related to the reactivation of basal slip [21, 87]. In particular, upon tensile reloading in the first cycle, right after 0% strain, an inflection with an increased slope (hardening rate) is observed. The different shapes of the hysteresis loop for the 1st and 70th cycles in the tensile side are quite different, as displayed in Figures 3.3(c) and 3.3(d), indicating different deformation mode sequences in these two fatigue cycles.

3.3.3 Microscopic response by neutron diffraction

3.3.3.1 Diffraction intensity evolution

The diffraction intensity evolutions of certain $hk.l$ s in both the axial and radial directions in the selected fatigue cycles are presented in Figures 3.4(a) and 3.4(b), respectively. The occurrence of $\{10.2\}$ extension twinning leads to a sudden re-orientation of grains at about 86.3° , which converts the $\{00.2\}$ grains into $\{10.0\}$ grains during loading in tension, and vice versa in compression in Bank 1 [18, 23]. Consequently, the diffraction intensity variations in twin-related grains can be used to identify the $\{10.2\}$ extension twinning process [18, 22, 23, 56]. It should be pointed out that the $\{11.0\}$ and $\{10.3\}$ grains are also in $\{10.2\}$ extension twin-related orientations [88]. Upon initial compression, $\{10.2\}$ extension twinning initiates at a strain of $\sim -0.5\%$, where the diffraction intensity of the

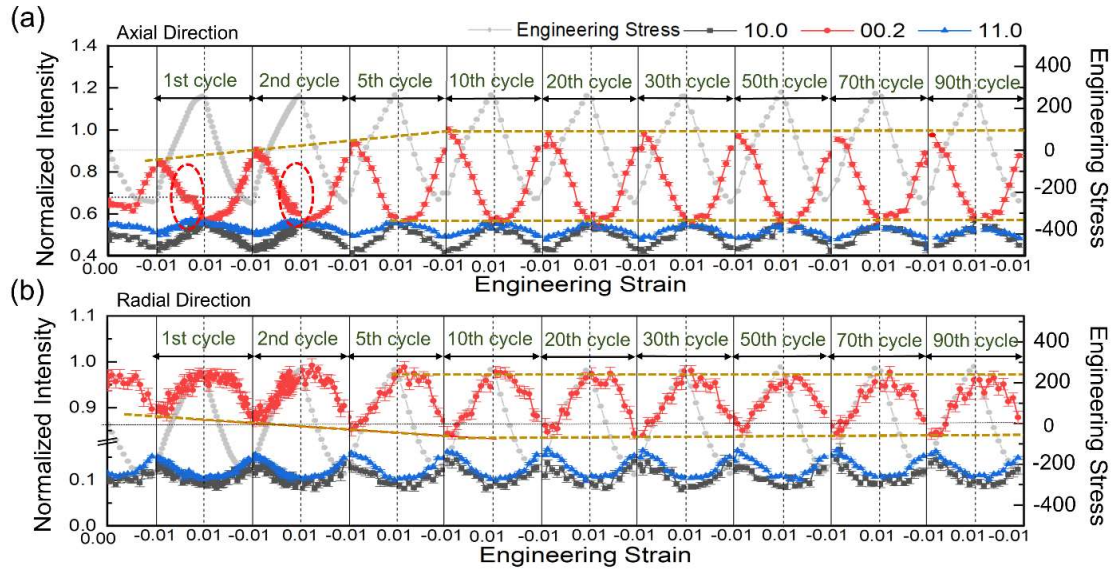


Figure 3.4. The diffraction intensity evolution of certain hkl 's in selected fatigue cycles for (a) the axial detector bank, and (b) the radial detector bank.

$\{00.2\}$ grains starts to increase in Bank 1 and decrease in Bank 2. While during reverse tension, the diffraction intensity of $\{00.2\}$ gradually decreases, and the diffraction intensity of the $\{10.0\}$ grains increases in Bank 2, which indicates the detwinning behavior [18]. The cyclic increase-decrease (or decrease-increase) sequence in the diffraction intensity of $\{00.2\}$ (or $\{10.0\}$) grains manifests that the alternative twinning and detwinning deformation occur during the compression-tension sequence in each fatigue cycle. It can be seen that the diffraction intensity of the $\{00.2\}$ grains in Bank 1 at the minimum strain, -1%, increases progressively in the first 5 cycles before saturating. This suggests that a progressively increasing number of $\{10.0\}$ grains are involved in the $\{10.2\}$ extension twinning during the first 5 cycles, and then cease changing in the following fatigue cycles [22]. It is worth noting that the significantly increased diffraction intensity in the $\{00.2\}$ grains at a strain of -1% in the 1st cycle is partly due to the different compressive strain levels, i.e., -1% for the initial compression, and $\sim -1.3\%$ for the first cycle [23]. Nevertheless, different from previous reports [18, 22, 23, 56], two plateau values appear on the diffraction intensity evolution curve in the first two cycles under reverse tension, noted with a red dash cycle in Figure 3.4(a), which will be illustrated in the following.

Figure 3.5 shows the diffraction intensity evolution of $\{00.2\}$ grains in Bank 1 as a function of engineering stress during cyclic loading. “T”, “D”, “DT”, and “TDD” refer to twinning, dislocation, detwinning, and transition from detwinning to dislocation, respectively. In this paper, “T1” and “T2” refer to the $\{10.2\}$ extension twinning in the parent grains, whose original c-axis is perpendicular and parallel to the loading direction,

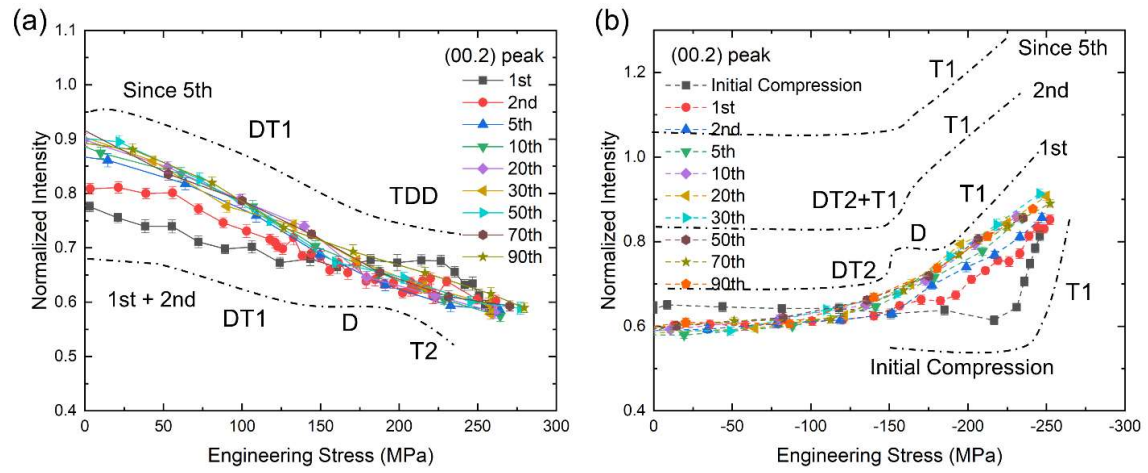


Figure 3.5. The diffraction intensity variation of {00.2} grains as a function of engineering stress in selected fatigue cycles (a) during tension, and (b) during compression in the axial detector bank.

respectively. “DT1” and “DT2” refer to the detwinning process from “T1” and “T2” “child” grains, respectively. The slopes of the diffraction intensity with applied stress curves can provide the information regarding the deformation modes in wrought Mg alloys [22, 23]: in Bank 1, the increase of $\{00.2\}$ diffraction intensity under compression is related to the twinning/detwinning process, the decrease of $\{00.2\}$ diffraction intensity upon tension corresponds to the detwinning/twinning process, and the plateau regime of $\{00.2\}$ diffraction intensity with stress associates with the dislocation-controlled mode. The general trends of these curves are represented by the dashed lines in Figure 3.5.

As shown in Figure 3.5(a), the $\{00.2\}$ diffraction intensity under tension in the first two cycles decreases initially, but gradually reaches a constant value, and subsequently decreases sharply again. According to the initial texture, the decrease in $\{00.2\}$ diffraction intensity can be related to the detwinning in the “child” twinned $\{00.2\}$ grains or the twinning in the “parent” $\{00.2\}$ grains. The constant regime is related to the dislocation-controlled mode. The detwinning process is reported to initiate more easily than the twinning process [18], presumably because detwinning is a mechanism of shrinking existing twins, which does not require twin nucleation [74]. Therefore, the first decrease region is related to the detwinning (DT1) mode, and the second one corresponds to the twinning (T2) mode. Consequently, the plastic deformation during tension in the first 2 cycles is thus controlled by the detwinning (DT1) mode, dislocation (D) mode, and twinning (T2) mode. The same diffraction intensity level in the constant region upon tension in the 1st cycle with that in the undeformed state further, seen in Figure 3.5(a),

illustrates that the detwinning (DT1) is fully exhausted. Since the 5th cycle, the $\{00.2\}$ diffraction intensity initially undergoes a rapidly decreased region and then a slowly decreased region by the end of tension that corresponds to the transition for detwinning to dislocation (TDD) mode where the detwinning is almost exhausted and dislocation rules over instead of the activation of twinning (T2). Therefore, the plastic deformation since the 5th cycle is dominated by the detwinning (DT1) mode and then transition from detwinning to dislocation (TDD) mode. The prolonged detwinning process in the following cycles suggests that the range of the detwinning-controlled (DT1) region strongly depends on how many grains are involved in the prior twinning process in each fatigue cycle.

Figure 3.5(b) shows the diffraction intensity evolution of $\{00.2\}$ grains upon compression. During the initial compression, the plastic deformation is dominated by the extension twinning (T1) process. The twinned $\{00.2\}$ grains formed in the initial compression process, however, are not enough for the consumption of the subsequent detwinning process, leading to the premature end of the detwinning mechanism and the beginning of the dislocation-controlled mode, as displayed in Figure 3.5(a). The $\{00.2\}$ diffraction intensity shows a trend of initial increase, then becomes stable, and followed by another increase during compression in the 1st cycle, similar to the analysis of the deformation mechanism during tension in the first two cycles, which correspond to the detwinning (DT2) mode, dislocation (D) mode after fully detwinning (DT2), and twinning (T1) mode, respectively. The $\{00.2\}$ diffraction intensity undergoes a rapid increase to a slow increase during compression in the 2nd cycle. The initial rapidly increased region is

proposed to be related to the combination of detwinning mode (DT2) and extension twinning (T1) mode, and the slowly increased region corresponds to the sole extension twinning (T2) mode due to the exhaustion of detwinning (DT2). From the 5th cycle, the plastic deformation is quite identical to the initial compression, solely controlled by twinning (T1) mode, as displayed in Figure 3.5(b). Obviously, the twinning (T1) process is the dominant deformation mechanism upon compression.

3.3.3.2 Lattice strain evolutions

The lattice strain evolutions of $\{10.0\}$, $\{10.3\}$, $\{00.2\}$ and $\{11.0\}$ grains of the Mg, and $\{411\}$ grains of the β -Mg₁₇Al₁₂ phase in the initial compression, 1st cycle, and 70th cycle are shown in Figure 3.6. The inflection points in the lattice strain with applied stress curves can be attributed to the activation of specific deformation mechanisms. In general, the lattice strain of given grains groups diverging vertically suggests that these “soft” grain groups begin to accommodate plastically, and simultaneously the “hard” grain groups start to share the more elastic load, causing a divergence with a small slope. The lattice strain variations can be understood by considering the Schmid factors for the basal slip and $\{10.2\}$ extension twinning for the given set of grain orientations [88]. The different deformation-controlled regions are separated by the square-dashed lines and the elastic line for Mg alloys or the $\{411\}$ grains corresponds to the dash-dotted line in Figure 3.6.

During initial compression, it is clear from Figure 3.6(a) that all the grain orientations have a similar slope in the initial elastic deformation owing to the near-elastic isotropy of Mg and its alloys [18]. The $\{10.1\}$ and $\{10.3\}$ grains yield first with the

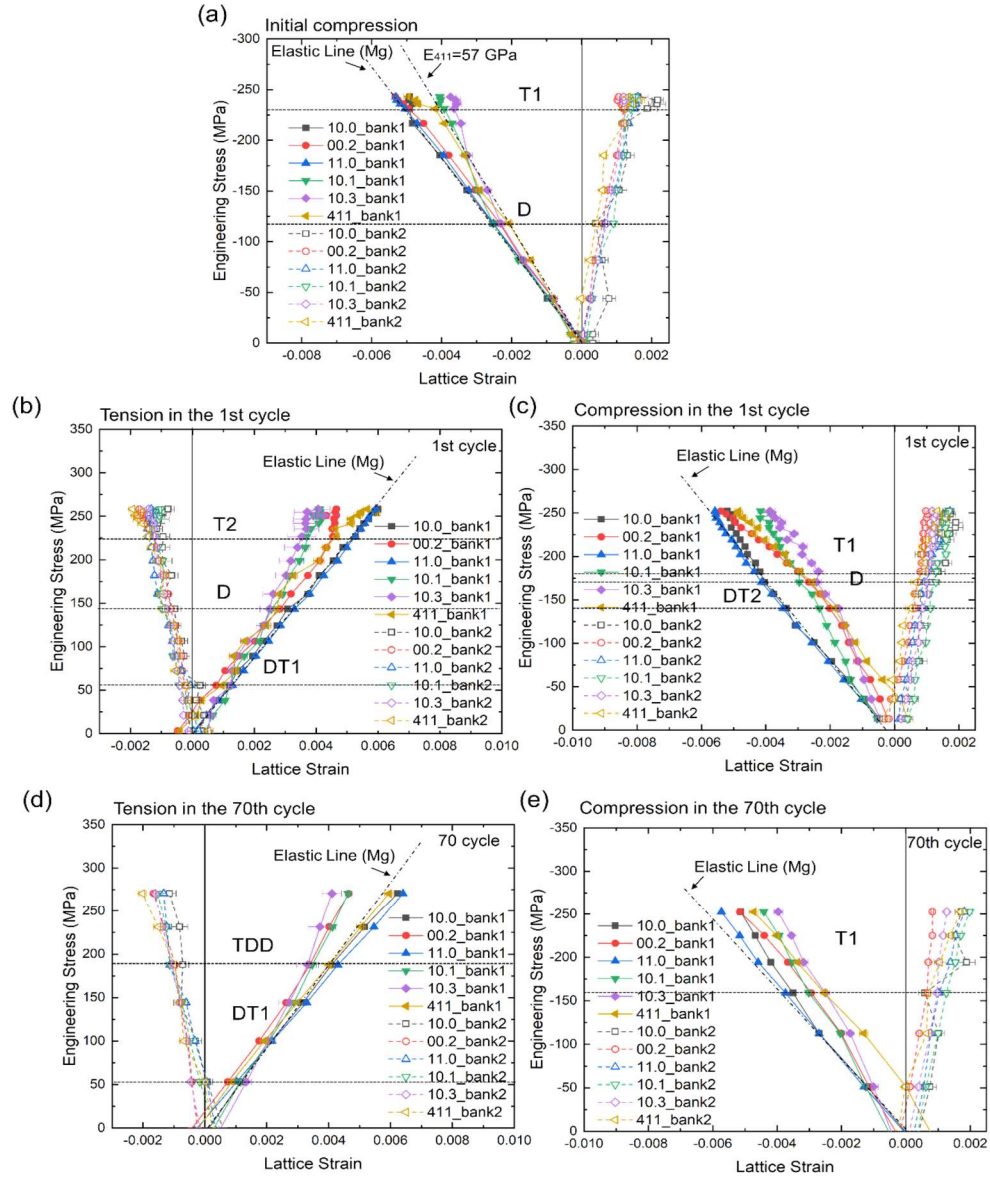


Figure 3.6. Lattice strain evolution of certain interested hk.l.s during (a) the initial compression, (b) tension and (c) compression in the 1st cycle, and (d) tension and (e) the compression in the 70th cycle.

corresponding curve diverging from the linear response due to the activation of basal $\langle a \rangle$ slip at relatively low stress [89]. Correspondingly, other hard grains begin to accommodate more load with a smaller slope. All the lattice elastic strain curves show a strong inflection at the macroscopic yield stress of ~ 228 MPa, where the $\{10.2\}$ extension twinning is activated. Once the twinning is activated, the $\{00.2\}$ and $\{10.3\}$ grains in the Bank 1 begin to accommodate more loads with a decreased slope, while the $\{10.0\}$ and $\{11.0\}$ grains are relaxed which depart from the linear response. Kada et al. [90] suggested that the β - $\text{Mg}_{17}\text{Al}_{12}$ phase has a hard Young's modulus of 69 GPa along (100) direction and a soft Young's modulus of 52 GPa along (111) direction, whilst Gharghouri et al. [8] reported a high Young's modulus of 87 GPa along (111) direction. In this study, Young's modulus for the $\{411\}$ grains is determined to be ~ 57 MPa, which is among the range of the elastic moduli of the β - $\text{Mg}_{17}\text{Al}_{12}$ phase suggested by Kada et al. [90]. The departure from the elastic response is noticed at ~ 118 MPa in the $\{411\}$ grains, where the basal slip activates. With further increase in the applied stress, the lattice strain of $\{411\}$ grains varies almost linearly with stress until an increased rate of the lattice strain accumulation is observed after yielding. At the macroscopic yield point, forward stress of ~ 30 MPa is accumulated in the $\{411\}$ grains.

As for the 1st cycle, upon reverse tensile loading [Figure 3.6(b)], the lattice strain responses of certain grain groups in Bank 1 during different deformation mode regions are distinct. During the detwinning-controlled (DT1) region where the $\{10.0\}$ and $\{10.1\}$ diffraction intensity increase, and $\{00.2\}$ diffraction intensity decreases, the $\{10.0\}$ and

$\{10.1\}$ grains share more loads, and the $\{00.2\}$ grains yield. During the dislocation-controlled (D) region where the $\{00.2\}$ diffraction intensity keeps constant in Figure 3.5(a), the $\{10.1\}$ and $\{10.3\}$ grains yield immediately. With the increase of stress, the sample then suddenly undergoes a twinning-dominated (T2) region where the $\{00.2\}$ diffraction intensity decreases dramatically and the lattice strains of the $\{00.2\}$ and $\{10.3\}$ grains diverges vertically because these grains are relaxed relative to the stress field in the surrounding grains. During compression in the 1st cycle in Figure 3.6(c), the general trend of lattice strain evolution is identical to that in the initial compression except for the occurrence of detwinning-controlled (DT2) mode. Once detwinning (DT2) is active, the $\{00.2\}$ and $\{10.3\}$ grains begin to accommodate more loads with lower slopes, coupled with the $\{00.2\}$ diffraction intensity decreasing [Figure 3.5(b)]. It is noted that the $\{411\}$ grains share a larger portion of loads since the activation of detwinning (DT2).

The lattice strain evolutions of certain grain orientations in the 70th cycle are shown in Figures 3.6 (d) and 3.6(e). During tension, the plastic deformation is controlled by detwinning-controlled (DT1) mode and transitions from detwinning to dislocation-controlled (TDD) mode, sequentially. The $\{10.1\}$ and $\{10.3\}$ grains that favor basal slip begin to yield at ~ 190 MPa due to the activation of TDD mode, where a large portion of “child” twinned grains is consumed by the detwinning process and dislocation motion become more active. During compression, the general trend of lattice strain evolution is identical to that in the initial compression in Figure 3.6(a).

3.4 Discussion

3.4.1 Deformation mechanisms

The deformation modes during plastic deformation have been divided by the peak stress, 0.2 proof stress, and transition stress in Figure 3.7, in which the T1, T2, DT1, DT2, D, and TDD-controlled regions are distinguished by different patterns. Apparently, the complicated deformation mode sequence can be seen in the first two cycles, which are closely related to the initial texture with two major texture components in this AZ80 alloy. While the deformation mode sequence in the following cycles becomes similar to that of Mg alloys with simple rolling or extruded texture [18, 23].

The tension following compressive deformation occurs predominantly by detwinning initially. This is consistent with previous reports in the literature [74]. The detwinning process is significantly influenced by the twinning processes that take place during prior compression. The occurrence of dislocation-dominated (D) deformation region in the first two fatigue cycles can be explained by the relatively early exhaustion of the detwinning process, as the inadequate twinned grains formed in the previous compression have been completely consumed by the detwinning before the strain reaches +1% in the first two cycles. Nevertheless, the sufficient twinned grains for consumption by detwinning lead to the disappearance of dislocation-controlled (D) mode in the following cycles. The dislocation (D) mode, activated after detwinning, may correspond to the activation of basal $\langle a \rangle$ -type slips, non-basal $\langle a \rangle$ -type slips (i.e., prismatic), or $\langle c + a \rangle$ -type slips. As the CRSS for extension twinning is reported between the non-basal slip and basal $\langle a \rangle$ slip [89, 91], the dislocation (D) mode here therefore is ascribed to the basal $\langle a \rangle$

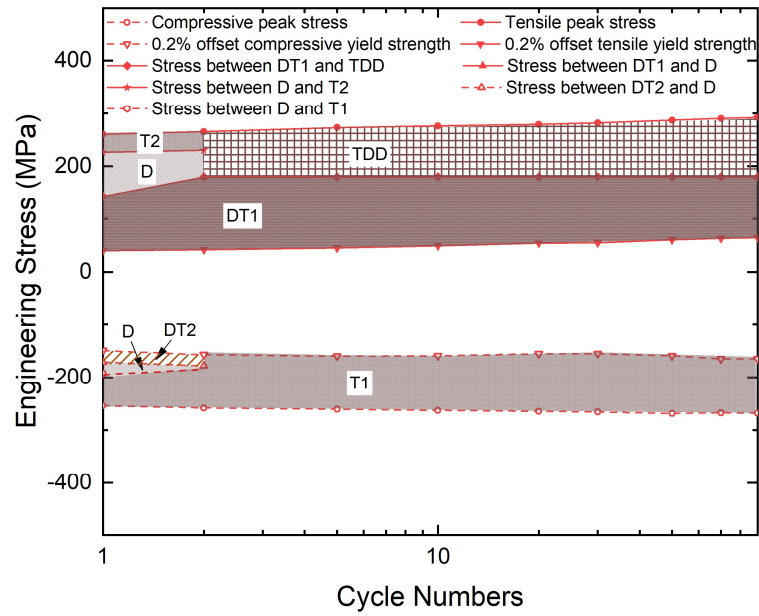


Figure 3.7. The peak stresses, 0.2% proof stresses, stresses at the dividing points between twinning/dislocation, detwinning/dislocation, and detwinning/transition region, and deformation modes as a function of the number of cycles.

slip in the soft grains (i.e., $\{10.2\}$, $\{11.2\}$, and $\{10.1\}$ grains). In this region, the rapid strain-hardening behavior exhibited in the macroscopic response in this region [Figure 3.3(c)] can be explained by the composite effect in the sense that only a small number of grains undergo plastic deformation and the rest remain elastic [21, 92]. The onset of detwinning (DT1) at a relatively low-stress level and dislocation-controlled (D) deformation at a relatively high-stress level produces an S-shaped hardening curve [Figure 3.3(c)].

The compressive deformation is predominantly controlled by twinning (T1) in the whole fatigue cycles. The detwinning mode (DT2) in the first two cycles arises from the detwinning process in the $\{10.0\}$ twinned grains that are obtained in the prior tension. Similarly, the basal $\langle a \rangle$ slip is proposed to be the dominant deformation mechanism in the dislocation-controlled (D) region.

It is interesting to note that, although the texture after total tensile unloading is identical to that in the initial state [Figure 3.2], the extension twinning-controlled (T2) mode only occurs in the first 2 cycles. This fact can be explained by that the CRSS for twinning is not reached during tension in the following cycles — assuming that twinning is controlled by a CRSS. In fact, by comparing Figures 3.6(b) and 3.6(d), it can be seen that the accumulated lattice strain in $\{00.2\}$ grains at the end of the tension in the 70th cycle is still less than that for the twinning onset, ~ 0.046 , in the 1st cycle. This trend makes sense since when detwinning (DT1) is the deformation mechanism, the lattice strain of the “soft” $\{00.2\}$ grains diverges vertically, leading to the slower lattice strain building up. As

more and more grains are involved in the twinning (T1) and sequent detwinning (DT1) process in the first 5 cycles, a large portion of shear stress fields in the {00.2} grains are relieved by the enlarged detwinning process (DT1) during tension, resulting in the insufficient accumulated shear stress for the onset of extension twinning (T2). Therefore, the disappearance of the twinning (T2) mode in the following cycles can be explained by the fact that the CRSS for twinning is not reached due to the prolonged detwinning (DT1) process. Consequently, it can be concluded that the deformation mode sequence strongly depends upon deformation history and texture during cyclic loading for Mg alloys.

3.4.2 Precipitation hardening effect on twinning

In the polycrystalline case, a number of attempts have been made to increase the CRSS for twinning. It is well-accepted that the activation of twinning is controlled by a CRSS, which can be estimated by the Schmid law under a uniaxial loading condition, described by [18]:

$$\tau_{CRSS} = \sigma_c \cos\lambda \cos\phi \quad (3-2)$$

where λ and ϕ are the angles between the loading axis and the twin plane normal and twin direction, τ_{CRSS} is the CRSS, and σ_c is the critical applied stress. Using Eq. (3-2), a CRSS of $\sim 91 \pm 5$ MPa is yielded for {10.2} extension twinning in this AZ80 alloy with a Schmid factor of ~ 0.40 . The CRSS obtained here is quite close to that in the Mg-7.7 at.% Al alloy (65-75 MPa) [17]. Wu et al. [18] reported a CRSS value of 15 MPa in the AZ31 alloy. This fact suggests that the value of CRSS is related to the precipitates, due to the strengthening effect of precipitates on the activation of twinning. The twinning-controlled

compressive yield stress of a solution-treated AZ80 alloy with a similar grain size is reported to be ~ 145 MPa [93]. According to Eq. (3-2), an estimated CRSS increment of ~ 33 MPa can be calculated attributed to the precipitation strengthening effect, which is comparable with that in the AZ91 alloy after and before aging treatment [90, 94]. However, the correction should be made by considering the loss of solute strengthening despite the negligible change in the grain structure and texture caused by aging can be ignored. For the AZ series Mg alloys, a reduction of ~ 10 MPa in the CRSS increment is reported [15]. The β -Mg₁₇Al₁₂ phase deforms elastically under cyclic loading, which cannot be cut by twins or dislocations [15]. Robson et al. [78] proposed that twins will engulf some of the precipitates during propagation when the precipitates are small with a fraction volume between 2–10% ($\sim 12.5\%$ in the current case). This will introduce a strain incompatibility at the particle-matrix interface with a maximum value of $\sim 6.5\%$ [95]. The back-stress arising from the strain incompatibility can result in the strengthening effect in twinning [15], although it can be reduced by the local plastic deformation [95].

3.4.3 Evolution of load sharing

During the twinning/detwinning dominated deformation under compression, the $\{00.2\}$ twinned grains in the axial direction tended to yield with a smaller slope of the lattice stress/strain curve than the calculated linear elastic response, 48 GPa [92]. This can be explained by the fact that although the newly formed $\{00.2\}$ “child” grains are relaxed relative to the matrix due to the small c/a ($< \sqrt{3}$) ratio of magnesium, the existing $\{00.2\}$ grains have to accumulate strain more rapidly than the aggregate as a whole because they

are in a “hard” orientation for both the twinning and basal slip [92]. Therefore, the lattice strain builds up faster than linear elasticity due to load sharing with the existing $\{00.2\}$ grains and surrounding grains. During the twinning/detwinning-dominated deformation region upon tension, loads transfer from the “soft” $\{00.2\}$ grains for twinning and detwinning to surrounding grains. The work-hardening effect in the twinning and detwinning-dominated regions is limited, and the transition from detwinning to the dislocation-dominated (TDD) deformation and dislocation-dominated (D) regions exhibit relatively large hardening response [22].

As the low volume fraction of precipitates in heat-treatable Mg alloys, reports on the load sharing effect of precipitates during cyclic loading are limited [90]. In the present case, however, a load-sharing effect of the precipitates is suggested. Once the activation of plastic deformation, the β -Mg₁₇Al₁₂ phase begins to accommodate a larger portion of the loads. However, Bhattacharyya et al. [96] suggested that the load sharing provided a limited strengthening effect due to the modest phase fractions and stiffness difference between the matrix and precipitates. The fractured surface after LCF is shown in Figure 3.8, which consists primarily of ductile dimples within the grains formed as a consequence of microvoid coalescence [97]. The fatigue striations can be observed in Figure 3.8(b), which are typical characteristics of fatigue crack propagation in ductile materials. To moderate the strain discontinuity between the elastically deformed unsheared precipitates and the Mg matrix, plastic relaxation effects, i.e., dislocation pileup, develop around these precipitates [95]. The local stress concentration near the weak-bonded particle-matrix

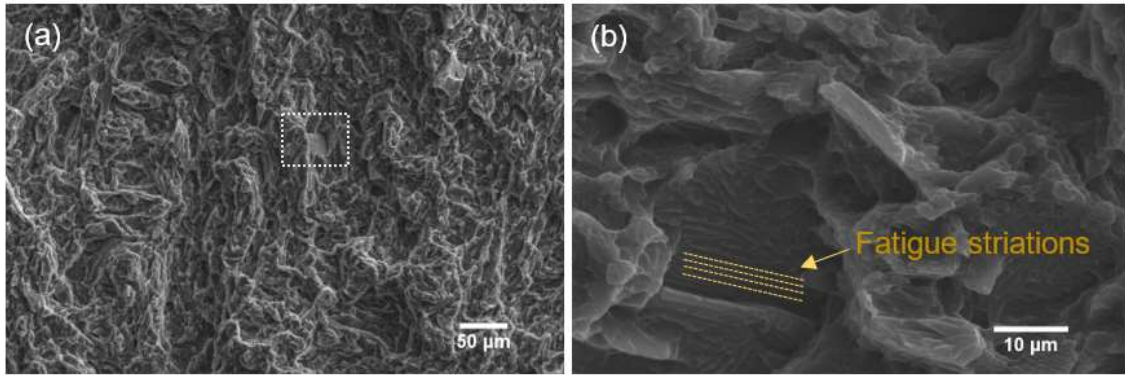


Figure 3.8. (a) Scanning electron microscopy (SEM) micrographs showing the fracture surfaces after low-cycle fatigue (LCF) (1% strain amplitude), and (b) is the magnified images taken from the regions surrounded by white dashed line in Figure 3.8 (a).

interface facilitates the microvoid creation and their coalescence forming dimples, which offers a great opportunity for the formation and propagation of cracks and degrades fatigue life [97].

3.4.4 *Iso-strain model*

To explain the evolution of the orientation-dependent lattice strains through the elastic-plastic transition regime and the macroscopic stress level at which yielding begins on average, the iso-strain model was applied [98]. The deformation systems of materials are specified by the crystal structures. For the Mg alloys with an HCP crystal structure, the plastic deformation is usually controlled by the basal slip, prismatic slip, pyramidal slip, and twinning [21]. In our case, according to the above analysis, only basal slip and extension twinning are considered. As the elastic deformation in Mg alloys is quite isotropic [18], the Hooke's law for an isotropic material (or an elastically isotropic aggregate) can be used directly,

$$E = \frac{\sigma}{\varepsilon} \quad (3-3)$$

where the σ is true stress, ε is the true strain, and E is Young's modulus. Consider a number of $\{10.0\}$, $\{00.2\}$, $\{10.1\}$, $\{11.0\}$, and $\{10.3\}$ grains all of which under the same strain, as schematically displayed in Figure 3.9(a) and distinguished by different colors.

The Schmid factors, $m^{hk.l}$, of basal slip and extension twinning for certain grain orientations under uniaxial loading condition are listed in Table 3.1. The calculation process of the Schmid factor is in detail in the Appendix. The directional strength measure, $S^{hk.l}$, for a certain grain orientation, $hk.l$, under uniaxial loading is therefore defined as:

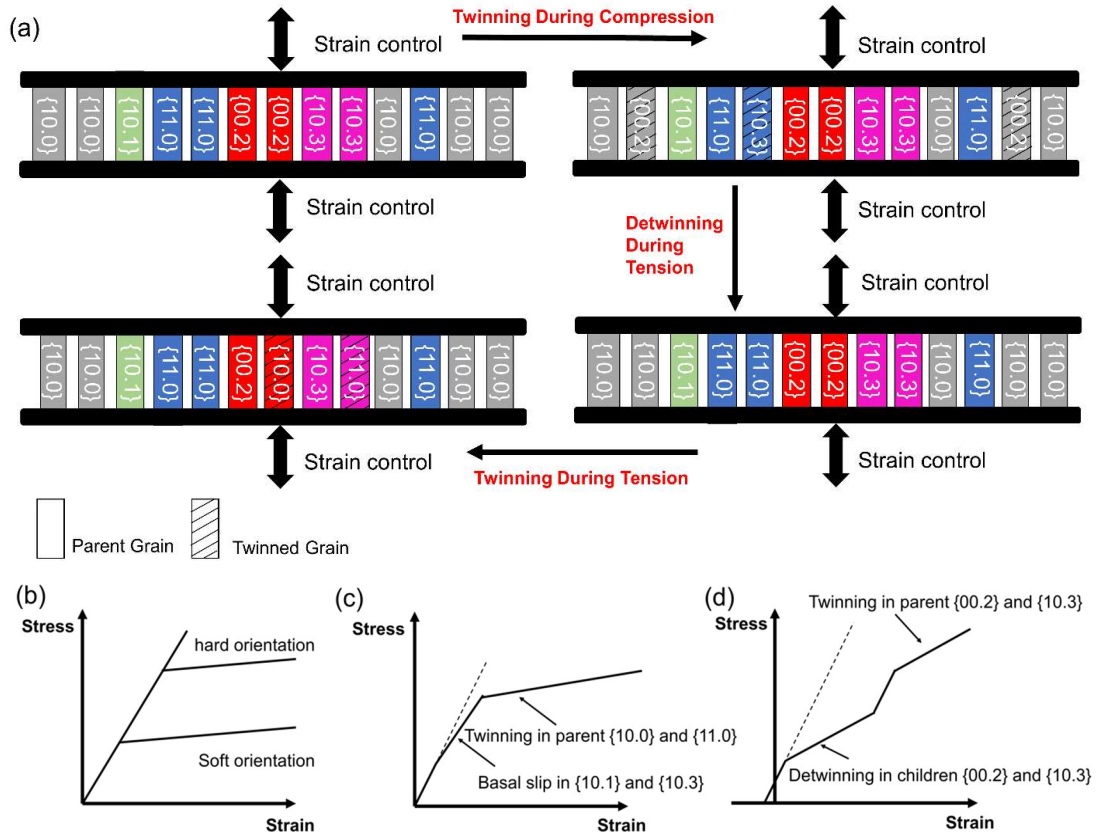


Figure 3.9. (a) A schematic pattern for the evolution of grain orientations during a compression-tension loading sequence, distinguished by different colors, loaded in the uniaxial condition between rigid plates, which results in an iso-strain condition for these grains, (b) individual stress-strain response of “soft” and “hard” grains for a certain deformation mode, (c) combined stress-strain response of during initial compression, and (d) combined stress-strain response during reverse tension in the first cycle.

Table 3.1. The Schmid factors, $m^{hk,l}$, of basal slip and extension twinning in Mg alloys for certain grain families under uniaxial loading condition.

Deformation mode	Reflection	Compression					Tension				
		$10\bar{1}0$	$10\bar{1}1$	$11\bar{2}0$	0002	$10\bar{1}3$	$10\bar{1}0$	$10\bar{1}1$	$11\bar{2}0$	0002	$10\bar{1}3$
Extension Twinning/ Detwinning	$(10\bar{1}2)[\bar{1}011]$	0.499	0.305	0.374	-0.499	0.190	-0.499	-0.305	-0.374	0.499	0.190
	$(01\bar{1}2)[0\bar{1}11]$	0.125	0	0.374	-0.499	0.309	-0.125	0	-0.374	0.499	0.309
	$(\bar{1}102)[\bar{1}\bar{1}01]$	0.125	-0.027	0	-0.499	0.338	-0.125	0.027	0	0.499	0.338
	$(\bar{1}012)[10\bar{1}1]$	0.499	0.251	0.374	-0.499	0.248	-0.499	-0.251	-0.374	0.499	0.248
	$(0\bar{1}12)[01\bar{1}1]$	0.125	-0.027	0.374	-0.499	0.338	-0.125	0.027	-0.374	0.499	0.338
	$(\bar{1}\bar{1}02)[\bar{1}\bar{1}01]$	0.125	0	0	-0.499	0.309	-0.125	0	0	0.499	0.309
	MAX	0.499	0.305	0.374	-0.499	0.190	-0.125	0.027	0	0.499	0.338
Basal <a> Slip	$(0001)[2\bar{1}\bar{1}0]$	0	-0.360	0	0	0.389	0	0.360	0	0	0.389
	$(0001)[\bar{1}2\bar{1}0]$	0	0	0	0	0	0	0	0	0	0
	$(0001)[\bar{1}120]$	0	0.360	0	0	0.389	0	-0.360	0	0	-0.389
	MAX	0	0.360	0	0	0.389	0	0.360	0	0	0.389

$$S^{hk.l} = \frac{1}{m^{hk.l}} \quad (3-4)$$

For each deformation mode, the values of $S^{hk.l}$ for different grain orientations calculated based on the maximum value of the Schmid factor are listed in Table 3.2. Generally, for a specific deformation mode, the high values of $S^{hk.l}$ suggest the “hard” grain orientations that are not favorably oriented for slip or twinning and are associated with high strength; the low values of $S^{hk.l}$, conversely, correspond to the “soft” grain orientations associated with low strength and earlier yield. It is worth mentioning that the grain orientations with the negative $S^{hk.l}$ values are unable to respond to the corresponding deformation modes under current load conditions, which belong to “hard” orientations. Consequently, under iso-strain condition, the soft grains with small $S^{hk.l}$ values of a certain deformation mode yield first, as shown in Figure 3.9(b).

From Table 3.2, it is clear to see that the $\{10.1\}$ and $\{10.3\}$ grains have much smaller $S^{hk.l}$ values compared with other orientations under both compression and tension for basal slip, suggesting that these grains are in “soft” orientations for basal dislocations. The deformation mode with relatively smaller CRSS will initiate earlier and the CRSS for basal slip is the lowest in Mg alloys [21, 89]. This explains the initial inflection in the $\{10.1\}$ and $\{10.3\}$ lattice strain in Figure 3.6(a). For the extension twinning mode under compression, the ranking from soft to hard orientations is $\{10.0\}$, $\{11.0\}$, $\{10.1\}$, $\{00.2\}$ and $\{10.3\}$, sequentially. Therefore, the divergences in the lattice strains of the $\{10.0\}$ and $\{11.0\}$ grains are observed in the extension twinning controlled region in Figure 3.6(a). The formation of the “child” twinned grains with $\{00.2\}$ and $\{10.3\}$ orientations related to

Table 3.2 Directional strength measures, $S^{hk,l}$, of for major slips and extension twinning in Mg alloys for certain grain families under uniaxial loading condition.

Deformation mode	Reflection	Compression					Tension				
		$10\bar{1}0$	$10\bar{1}1$	$11\bar{2}0$	0002	$10\bar{1}3$	$10\bar{1}0$	$10\bar{1}1$	$11\bar{2}0$	0002	$10\bar{1}3$
Extension Twinning/ Detwinning	$\{10\bar{1}2\}$ $<10\bar{1}1>$	2	3.27	2.67	-2	-5.26	-8	37.04	∞	2	2.96
Basal $<a>$ Slip	$\{0001\}$ $<12\bar{1}0>$	∞	2.78	∞	∞	2.57	∞	2.78	∞	∞	2.57

the twinning process is schematically shown in Figure 3.9(a). The combined load-displacement response under initial compression is presented in Figure 3.9(c). Once the basal slips are active in the $\{10.1\}$ and $\{10.3\}$ grains, the slope of the load-displacement curve tends to decrease slightly due to the small amount of $\{10.1\}$ and $\{10.3\}$ grains. After the activation of extension twinning in the $\{10.0\}$ and $\{11.0\}$ grains, the slope of the load-displacement curve decreases dramatically.

During the reverse tension in the first cycle, the $\{00.2\}$ and $\{10.3\}$ grains are in “soft” orientations for both the detwinning and twinning. The detwinning process with relatively low CRSS causes the initial inflection in the lattice strain of the “child” twinned $\{00.2\}$ and $\{10.3\}$ grains [18, 74], and the lattice strain divergence in these grains at relatively high stress is due to the onset of twinning, as displayed in Figure 3.6(b). The change in the grain orientations for detwinning and twinning during tension is also schematically shown in Figure 3.9(a). It should be pointed out that the basal slips in the $\{10.1\}$ and $\{10.3\}$ grains can even initiate during the previous compressive unloading process [21, 87] and keep active in the whole reverse tension. The combined load-displacement response during reverse tension in the first cycle is exhibited in Figure 3.9(d). The slope of the load-displacement curve dramatically decreases corresponding to the activation of the detwinning and twinning process, respectively, and recovers in the basal slip-dominated region. There is no essential difference between Figures 3.6(b) and 3.6(d), and between 3.6(c) and 3.6(f), which suggests that the local environment of twinning, detwinning, and retwinning is essentially the same.

3.5 Conclusions

In-situ neutron diffraction was employed in the current study to investigate the deformation mechanisms of an extruded AZ80 Mg alloy at the grain level during the fully reversed strain-controlled LCF. Major conclusions can be drawn based on the present research, as described below:

- (1) The initial texture and deformation history played an important role in the mechanical behavior of Mg alloys under a uniaxial loading condition. The occurrence of extension twinning during both compression and reverse tension in the first two cycles was ascribed to the initial texture with two major texture components in this alloy.
- (2) Upon compression, plastic deformation was mainly dominated by twinning with a limited basal slip for compatibility. More and more grains were involved in the twinning and the following detwinning process in the first 5 cycles. The CRSS for $\{10.2\}$ extension twinning was determined to be $\sim 91 \pm 5$ MPa in the AZ80 alloy. A CRSS increment of ~ 33 MPa for twinning was proposed due to precipitation strengthening.
- (3) Upon tension following compression, the deformation relies on detwinning (DT1) rather than extension twinning (T2). The extension twinning mode (T2) only occurred in the first 2 cycles upon reverse tension, presumably because the higher degree of the detwinning process (DT1) in the following cycles relieved the shear stress field of $\{00.2\}$ grains so that the CRSS for extension twinning is not reached.
- (4) The lattice strains evolved nonlinearly during unloading and reloading, revealing that load redistributions continuously occur between “soft” and “hard” grain orientations, related to the deformation mode. The loading sharing effect of the strengthening

precipitates was observed that the elastically deformed precipitates tend to convey more loads during plastic deformation.

APPENDIX

The Schmid factors can be calculated by $m = \cos\lambda\cos\varphi$ with λ and ϕ being the angles between the loading directions and the slip (or twinning) plane normal and slip (or twinning) direction, respectively [47,48]. For a specific deformation system, i.e., $\{hkil\}\langle uv\bar{t}w\rangle$, the slip or twinning plane normal is reassigned by [47,48]:

$$[h_1, k_1, i_1, l_1] = [h, k, i, \frac{3}{2}l(\frac{a}{c})^2] \quad A(3-1)$$

where a and c are the lattice constants, given the c/a axial ratio as 1.624. The cosine functions, $\cos\lambda$ and $\cos\varphi$, can be calculated by:

$$\cos\varphi(\lambda) = \frac{u_1u_2+v_1v_2+\frac{1}{2}(u_1v_2+u_2v_1)+\frac{1}{3}\omega_1\omega_2(\frac{c}{a})^2}{\sqrt{u_1^2+v_1^2+u_1v_1+\frac{1}{3}\omega_1^2(\frac{c}{a})^2}\times\sqrt{u_2^2+v_2^2+u_2v_2+\frac{1}{3}\omega_2^2(\frac{c}{a})^2}} \quad A(3-2)$$

where $[u_1, v_1, t_1, w_1]$ is the new slip (twinning) plane normal or slip (twinning) direction, while $[u_2, v_2, t_2, w_2]$ is the loading direction. For a specific grain orientation, $\{hkil\}$, the loading direction $[u_2, v_2, t_2, w_2]$ can be given as $[h, k, i, \frac{3}{2}l(\frac{a}{c})^2]$ by applying Eq. A(3-1).

CHAPTER IV
MICROMECHANICAL ORIGIN OF THE ENHANCED DUCTILITY
IN TWINLESS DUPLEX MG-LI ALLOY

A version of this chapter was originally published by Di Xie, Mingyu Fan, Huck Beng Chew, Peter K. Liaw, Hongbin Bei, Zhongwu Zhang, and Yanfei Gao:

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Di Xie is responsible for leading the research project, conducting the experiments, analyzing data, and writing the manuscript.

Abstract

Unlike many Mg alloys that exhibit limited ductility, duplex Mg–Li alloys possess enhanced ductility but the underlying mechanisms are unclear. Using real-time in situ neutron diffraction measurements, we show that the underlying deformation modes in this duplex microstructure include an early yield of body-centered-cubic (BCC) β -Li phase and the later elevated hardening in this phase at large macroscopic plastic strain, and the sequential activation of basal and pyramidal $\langle a \rangle$ slip systems in hexagonal-close-packed (HCP) α -Mg phase. The latter relieves the Mises constraint for deformation compatibility, and the successive yielding sequence promotes the overall work hardening rate, both of which are beneficial for ductility enhancement. No obvious twinning activities were found and correspondingly the hysteresis loops were symmetric upon a full loading cycle within $\pm 1\%$ applied strain.

4.1 Introduction

Magnesium alloys are low-density alloys of interest to the automotive and aerospace industries due to the ever-increasing demand for weight reduction, but their widespread adoption has been hindered by their limited ductility [70, 72, 99]. It has now been established that the primary deformation modes of commercial Mg alloys at room temperature comprise $\{0002\}\langle 1\bar{2}10\rangle$ basal slip and $\{10\bar{1}2\}$ extension twin [18, 100]. The limited number of independent slip systems (less than five) cannot satisfy the Mises constraint on deformation compatibility, which is a major reason for the limited ductility of Mg alloys. Among numerous metallurgical and mechanical studies in ductility enhancement, a particular balance of ductility and strength was recently observed in duplex Mg-Li alloys, suitable for many promising applications [101-103].

To date, it remains unclear whether the ductility enhancement of duplex Mg-Li alloys is related to the Mises constraint, or the composite structure comprising of α -Mg and soft β -Li phases, or anything that ex situ characterization cannot derive. *First*, regarding the former, it has been postulated that the addition of Li reduces the c/a axial ratio of the α -Mg phase, which hinders the activation of $\{10\bar{1}2\}$ extension twin [104] while favoring the activation of non-basal slip systems [13, 105-108]. Hence, an increasing focus is placed on elucidating the role of these non-basal slip systems in accommodating strain incompatibility in the HCP α -Mg phase. Some studies reported that the activities of pyramidal slips were significantly enhanced in Mg-Li alloys [13, 106], and the competition between basal and pyramidal slip systems worked in concert to limit the activities of

prismatic slip [107]. In contrast, other studies suggested that prismatic $\langle a \rangle$ slip was more important than pyramidal slip at the onset of plastic deformation at room temperature [108], and the activity of prismatic slip was improved with the increase of test temperature, which might hinder the activity of pyramidal slip in Mg-Li alloys [105]. *Second*, the macroscopic ductility is often dictated by the onset of necking, which is very sensitive to the work-hardening rate. Therefore, ductility enhancement strategies often involve a composite structure of soft and hard phases, such as ferrite-martensite dual-phase steels, or the addition of transformation-induced plasticity (TRIP) effects, such as the TRIP steels with retained austenite [109]. It is anticipated that the soft BCC β -Li phase can accommodate the deformation anisotropy and thus incompatibility in the hard HCP α -Mg phase, and also provides enhanced work-hardening rate due to the successive yield sequence just like in the dual-phase steel. However, the extent of such load partitioning is still unknown and cannot be obtained from ex situ investigations. Therefore, we perform a quantitative investigation into the deformation mechanisms of Mg-Li alloys using in situ neutron diffraction technique [84, 85, 110, 111]. The employment of real-time in situ neutron diffraction technique hence allows us to non-destructively determine the lattice strain behaviors of such duplex alloys during a compression-tension-compression sequence, thus revealing the micromechanical origin for the ductility enhancement.

4.2 Experimental Procedures

A hot-rolled Mg-8Li-3Al-3Ho alloy was studied in this work, which was fabricated by pure elements (purity 99.9 wt.%) of Mg, Li, Al, and master alloy of Mg-30%Ho in a

vacuum induction furnace under inert argon gas protection. The chemical composition is 8.26 wt.% Li, 3.02 wt.%, 2.98 wt.%, and balanced Mg. Hot rolling was conducted on the as-cast sample at 320 °C with a total thickness reduction of 50% through 5 passes with 10% thickness reduction per pass [112]. Microstructural characterization of the hot-rolled samples was performed by optical microscopy (OM). The specimen for OM was prepared by mechanical polishing and etching using a solution consisting of 5 ml acetic acid, 100 ml ethanol, and 10 ml water. The initial crystallographic texture was measured by synchrotron X-ray diffraction technique at the beamline 11-ID-C, the Advanced Photon Source (APS), Argonne National Laboratory (ANL).

In situ neutron-diffraction measurements were performed at the VULCAN Engineering Materials Diffractometer, the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL) under a continuous-loading condition. A cylindrical dog-bone-shaped sample with gauge dimensions of $\phi 8\text{mm} \times 16\text{ mm}$ was cut with the longitudinal axes parallel to the rolling direction (RD). During the neutron measurement, the sample was first loaded to a strain of -1% and then reversed tension to a strain of +1%, followed by another compression to a strain of -1% at room temperature with a strain rate of $7.4 \times 10^{-6} \text{ s}^{-1}$. The tested sample was mounted horizontally to the MTS load frame, with the loading direction along the rolling direction (RD) and the radial direction parallel to the normal direction (ND) of the sample, respectively. The angle between the incoming neutron beam and the specimen was 45°. The diffraction patterns with diffraction vectors

parallel (Q||) and perpendicular (Q⊥) were recorded by two stationary detector banks. The neutron measurement setup is schematically shown in Figure 4.1. The neutron beam slits were 7 mm (horizontal) × 7 mm (vertical), with a 5 mm receiving collimator, leading to a neutron gauge total volume of 245 mm³. The neutron diffraction measurements were conducted under the high-intensity mode with a chopper frequency of 30 Hz with a center wavelength of 2.7 Å, which corresponds to a lattice d-spacing range of 0.9~3.2 Å. VULCAN Data Reduction and Interactive Visualization software (VDRIVE) was employed to obtain the data points and analyze the diffraction data.

The lattice strains of a given orientation (hkl) plane in the detector banks can be calculated based on the peak shift between the loaded and load-free patterns, given by:

$$\varepsilon_{hk,l} = \frac{d_{hk,l} - d_{hk,l}^0}{d_{hk,l}^0} \quad (4-1)$$

where $d_{hk,l}$ and $d_{hk,l}^0$ are the plane d-spacings in the loaded and load-free conditions, respectively. Full-pattern Rietveld refinements via GSAS-II were conducted to obtain the lattice parameters and volume fractions of the two phases. The Rietveld strains of a given phase are estimated based on the lattice parameter shift between the loaded and load-free patterns, given by:

$$\varepsilon_{Rietveld} = \frac{a - a_0}{a} \quad (4-2)$$

where a and a_0 are the lattice parameter in the loaded and load-free conditions for a certain phase, respectively.

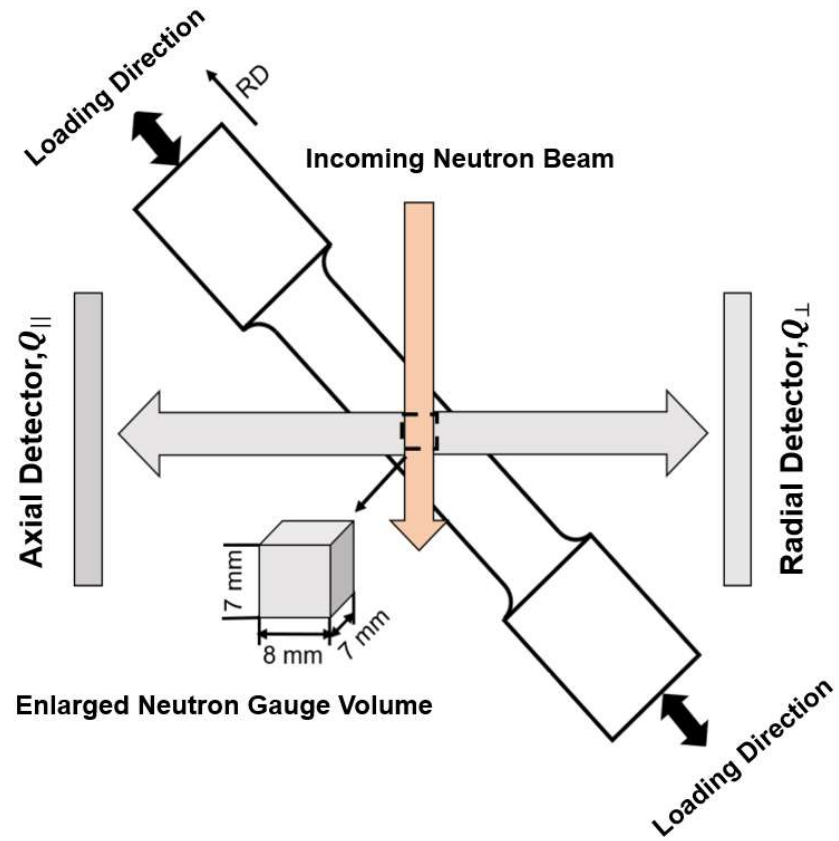


Figure 4.1. Neutron diffraction experimental setup at VULCAN (Spallation Neutron Source, Oak Ridge National Laboratory). The loading axis is parallel to the rolling direction of samples.

4.3 Experimental Results and Discussion

The initial microstructure of the hot-rolled Mg-8Li-3Al-3Ho alloy on the TD-ND plane (TD: transverse direction; ND: normal direction) is shown in the inset of Figure 4.2(a). The white “brick-shaped” phase is uniformly distributed within the dark phase. Combined with the EDS mapping results in Figures 4.3(a) and 4.3(b), the “brick-shaped” phase is determined to be the α -Mg phase, which distributes in the β -Li phase matrix. In addition to the peaks of these two phases, additional diffraction peaks of the intermetallic compounds, Al_2Ho , MgAlLi_2 , and AlLi , are observed [112], according to the synchrotron X-ray diffraction pattern of this alloy in Figure 4.2(a). As illustrated in Figure 4.3(c), Al elements are detected in the particles located in the β -Li phase and between the α -Mg phase and β -Li phase. Considering the invisibility of Li element in EDS, these particles are AlLi phase or MgAlLi_2 phase. Ho element distributes evenly in the matrix of the alloy and no obvious aggregation of Ho is observed in Figure 4.3(d). This may be that the Al_2Ho phase is the very fine precipitates that cannot be observed in the EDS mapping, while can be detected by the high-resolution synchrotron X-ray diffraction technique. The volume fraction is determined to be $\sim 42.4\%$ for the α -Mg phase and $\sim 57.6\%$ for the β -Li phase via Rietveld refinement. The lattice parameters of the α -Mg phase and β -Li phase are also extracted from Rietveld refinement of the synchrotron X-ray diffraction pattern, given by $a=3.18494 \text{ \AA}$ and $c=5.11645 \text{ \AA}$ for α -Mg phase and by $a=3.52064 \text{ \AA}$ for β -Li phase. The c/a ratio is calculated to be 1.6065 for the HCP phase, which is consistent with previous

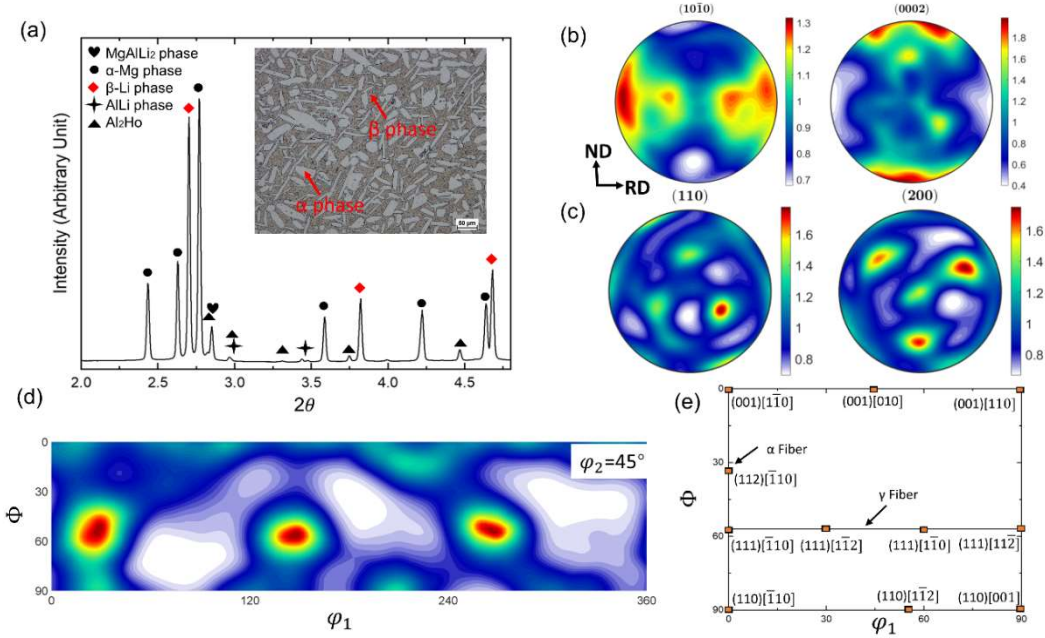


Figure 4.2. (a) Synchrotron X-ray diffraction pattern and optical microscope (OM) image of the duplex Mg-8Li-3Al-3Ho alloy on the TD-ND plane, (b) $(10\bar{1}0)$ and (0002) pole figures of the α -Mg phase, (c) (110) and (200) pole figures of the β -Li phase measured by the synchrotron X-ray diffraction, (d) the ODF for the BCC β -Li phase in $\phi_2 = 45^\circ$, and (e) schematic figure of typical texture component in BCC phase in $\phi_2 = 45^\circ$.

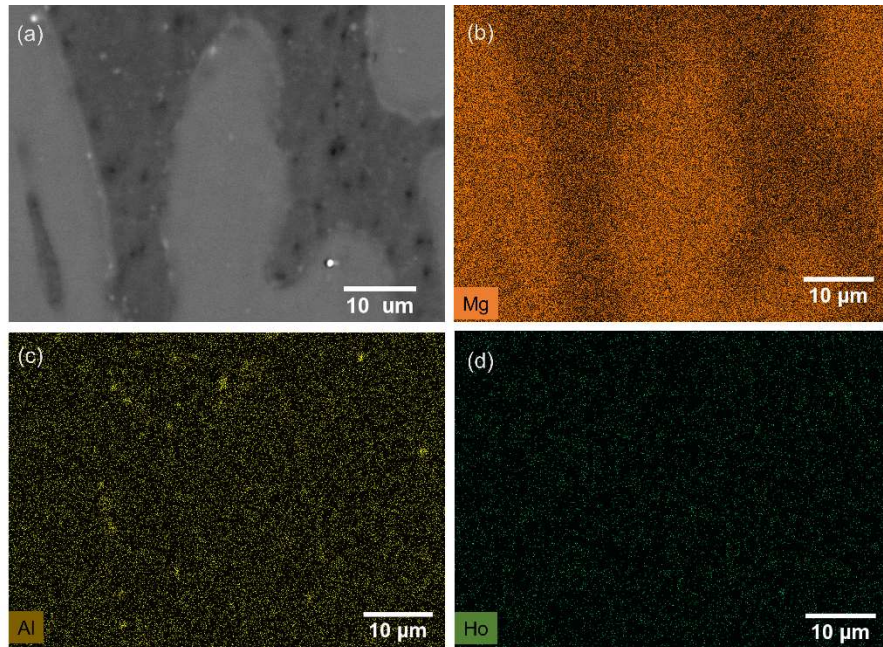


Figure 4.3. (a) BSE-SEM image, and the corresponding elemental mapping results: (b) Mg, (c) Al, and (d) Ho of the Mg-8Li-3Al-3Ho alloy.

studies as the c/a ratio of the HCP phase was greatly reduced due to Li addition [13]. Figures 4.2(b) and 4.2(c) present, respectively, the $(10\bar{1}0)$ and (0002) pole figures of the α -Mg phase, and (110) and (200) pole figures of the β -Li phase measured by the synchrotron X-ray diffraction, where pole intensities are represented in terms of multiples of a random distribution (mrd). The α -Mg phase exhibits a combination of a strong rolling texture with (0002) basal plane perpendicular to ND and a weak extruded texture with the basal planes parallel to the rolling direction. For the BCC β -Li phase, the orientation distribution function (ODF) in the 45° sections of φ_2 is shown in Figure 4.2(d). Figure 4.2(e) is a schematic figure to illustrate the common texture component in BCC structure at $\varphi_2=45^\circ$ section of the ODF [113]. Comparing Figures 4.2(d) and 4.2(e), we can see that the main hot rolling texture of the β -Li phase is the $(111)[1\bar{2}1]$ component that aligns on the γ -fiber.

The macroscopic stress-strain response for this alloy is exhibited in Figure 4.4(a). Different from the reported asymmetric hysteresis loops in the AZ and ZK series Mg alloys, the hysteresis loop is symmetric in this duplex Mg-Li alloy. It is known that the asymmetric loop in the commercial Mg alloys is ascribed to the activation of detwinning during reverse deformation. The lack of such asymmetry indicates no obvious deformation twinning occurs in this duplex Mg-Li alloy and dislocation slips are the primary deformation mode for the entire strain path. This is also confirmed by the OM image on the TD-ND plane of the deformed sample after a 1% compressive strain, given in Figure 4.5, in which no twin is observed. Like in most polycrystalline metals, the Bauschinger effect is observed in this

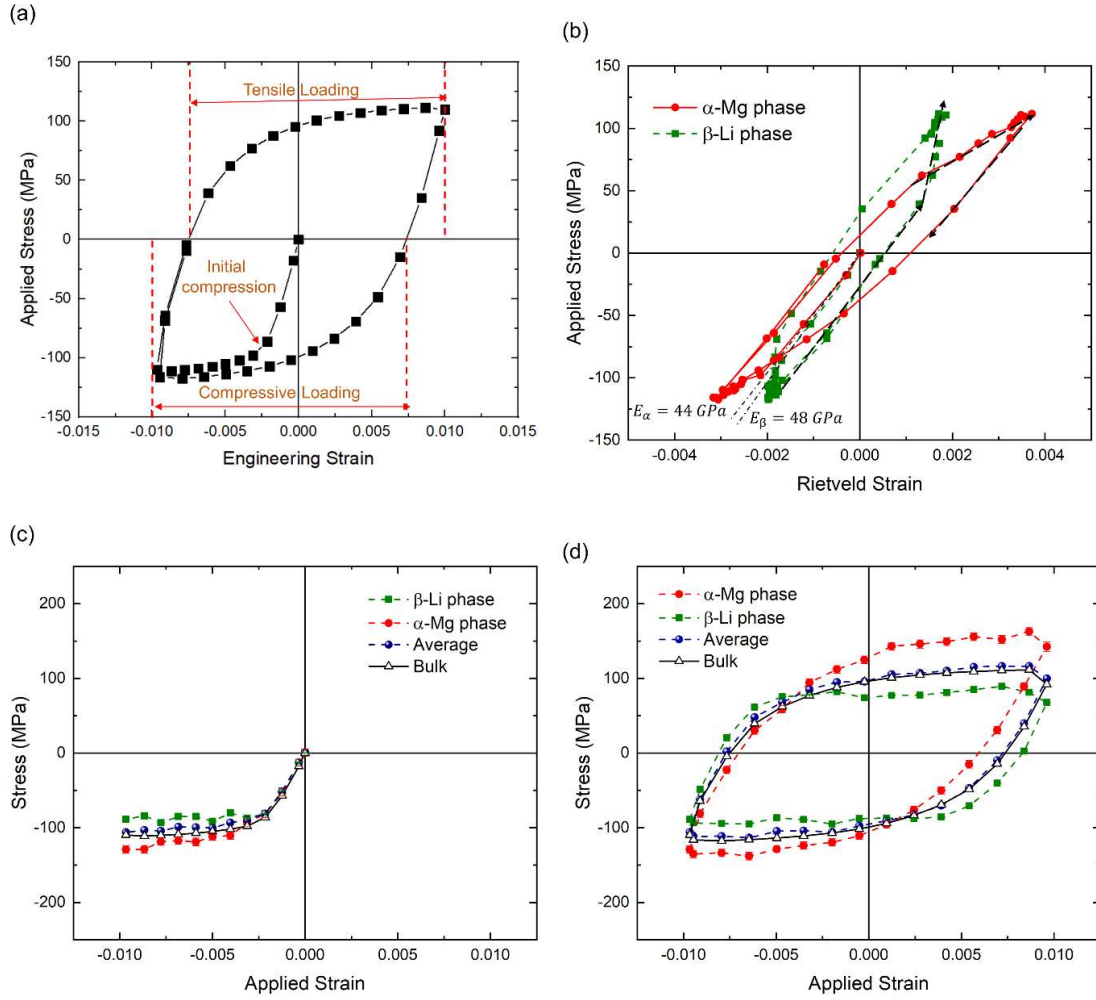


Figure 4.4. (a) The macroscopic stress-strain response for the Mg-8Li-3Al-3Ho alloy, (b) hysteresis loops (applied stress versus Rietveld strain) for the α -Mg phase and β -Li phase along the axial direction. The black arrows indicate the lattice-strain evolution direction for these two phases, where the lattice strains of the α -Mg phase evolve clockwise and those of the β -Li phase evolve counterclockwise. Comparison amongst the measured bulk stress, phase-specific stress, and average stress in the initial compression (c) and the reverse tension and followed compression (d) are plotted.



Figure 4.5. The optical microscope (OM) image of the deformed sample after 1% compressive strain on the TD-ND plane.

alloy, i.e., the reverse tensile yield strength is reduced compared to the initial compressive yield strength. The Rietveld analysis allows us to separate the mechanical responses of the two phases. Figure 4.4(b) shows the hysteresis loops (applied stress vs. Rietveld strain) for the α -Mg phase and β -Li phase along the axial direction. We can see that these loops are again symmetric, and the one for the β -Li phase yields at a relatively small load level, compared to that for the α -Mg phase, as derived from the observation of strain splitting in the initial compressive loading. Also as a consequence of the strain splitting, the Rietveld strain evolution paths as a function of applied stress in the α -Mg phase and β -Li phase are different in the sense that Rietveld strains in the former (hard phase) evolve clockwise, and anticlockwise in the latter (soft phase).

Phase-specific stress can be derived from Hooke's law, $\sigma_{phase} = E_{phase} \varepsilon_{phase}$, where σ_{phase} is the phase average stress, ε_{phase} is the phase average elastic strain obtained via Rietveld refinements, and E_{phase} represents the macroscopic Young's modulus of the individual phase. E_{phase} here is estimated by fitting the relationship between applied stress and Rietveld strain in the uniaxial initial compression: ~ 44 GPa for α -Mg phase and ~ 48 GPa for β -Li phase, indicated by the dashed lines in Figure 4.4(b) as will be explained shortly. The phase stress vs. applied strain for these two phases are displayed in Figures 4.4(c) and 4.4(d). The α -Mg phase begins to carry more stress with relatively large stress amplitude than the β -Li phase when the sample yields. Almost no strain hardening can be seen in the β -Li phase that accommodates plastic deformation at an early stage. The average estimates of the bulk stress states can be obtained from the rule of mixtures: $\sigma_{ave} = \sigma_{\alpha} f_{\alpha} +$

$\sigma_\beta(1 - f_\alpha)$ [114], in which σ_{ave} is the estimated average bulk stress and f_α is the volume fraction of α -Mg phase ($\sim 42.4\%$ in this case), as also displayed in Figures 4.4(c) and 4.4(d). The average stress determined from the rule of mixtures is in good agreement with the measured bulk value. It should be pointed out that the phase stress versus applied strain curves are not the true stress-strain data for these individual phases, as their strains are also different. Therefore, the hardening behavior of individual phases needs to be extracted from lattice strain data next.

The lattice strain evolutions of $\{10\bar{1}0\}$, $\{10\bar{1}1\}$, $\{11\bar{2}0\}$, and $\{0002\}$ grains of the α -Mg phase, and $\{110\}$ and $\{200\}$ grains of the β -Li phase in the axial direction and radial direction are shown in Figure 4.6. The lattice strain variations are related to the activation of specific deformation mechanisms, where the lattice strain of a given grain group diverging vertically indicates that this “soft” grain group has yielded and accommodated plastic deformation, and simultaneously other “hard” grain families tend to share the more elastic load, resulting in divergence with a small slope. The sample initially undergoes compressive elastic deformation and the lattice strains increase linearly with the macroscopic applied stress, as presented in Figure 4.6(a). For the α -Mg phase, the first inflection is observed in the $\{10\bar{1}0\}$ grains at ~ 58 MPa, where the corresponding curve diverges from the linear response with an increasing slope. As the $\{10\bar{1}0\}$ grains are favorably oriented for the activation of basal $\langle a \rangle$ slip with a Schmid factor of $m=0.362$ in this duplex Mg alloy ($c/a=1.6065$), it can thus be deduced that the first inflection is associated with the activation of basal slip, and the other subset of grains share a large

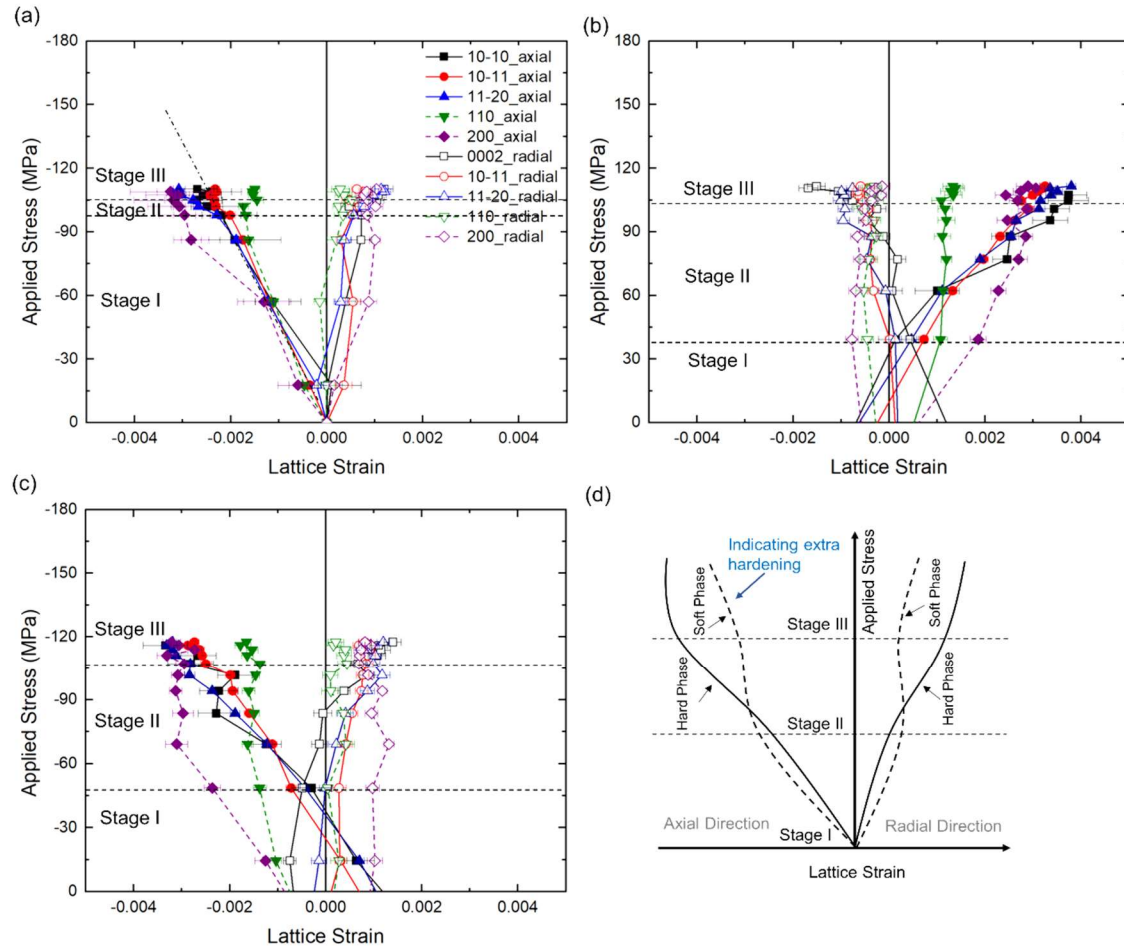


Figure 4.6. The lattice strain evolution of certain hkl's during (a) initial compression, (b) reverse tension, (c) following compression, in the axial detector bank and the radial detector bank, and (d) schematic figure for lattice strain partitioning in the duplex Mg-Li alloy.

portion of the load and their associated slopes decrease. The corresponding critical resolved shear stress (CRSS) value is 21 ± 4 MPa. The yielding in the $\{10\bar{1}1\}$ grains, however, has a limited effect on the overall deformation due to the small volume fraction in this strongly textured alloy [18]. Hence, the macroscopic stress-strain curve still behaves like elastic. With further compression, the second inflection is noticed at the macroscopic compressive yield stress of ~ 98 MPa, where the $\{110\}$ and $\{200\}$ grains yield and the corresponding lattice strain diverge vertically, and other grains tend to share more loads with smaller slopes. This fact suggests the macro-yield of this alloy is strongly associated with the micro-yield of the “soft” β -Li phase. When the sample is loaded to ~ 105 MPa, the $\{10\bar{1}1\}$ grains of the α -Mg phase yield with an increased slope in their corresponding curve, and the $\{110\}$ and $\{200\}$ grains of the β -Li phase depart to the left with decreased slopes. The $\{10\bar{1}1\}$ grains not only favor the activation of basal $\langle a \rangle$ slip but also favor the activation of pyramidal $\langle a \rangle$ slip with $m=0.467$. Therefore, the last inflection in $\{10\bar{1}1\}$ grains may be associated with the pyramidal $\langle a \rangle$ slip. The corresponding CRSS value of 49 ± 3 MPa for pyramidal $\langle a \rangle$ slip is much smaller than that in the AZ or ZK series Mg alloys.

The lattice strain evolution of the α -Mg phase and β -Li phase in the reverse tension and following compression is similar to the initial compression, as shown in Figures 4.6(b) and 4.6(c). The main difference lies in the relatively small stress levels where the $\{110\}$ and $\{200\}$ grains of the β -Li phase depart from the elastic linear response in the reverse tension and the following compression compared to initial compression. The expansion of Stage II arises from the Bauschinger effect in that the reverse tensile yield strength is

reduced compared to the initial compressive yield strength. The earlier yielding of the β -Li phase at ~ 40 MPa leads to the earlier occurrence of the macroscopic yielding in this duplex alloy in the following deformation. We can also notice that after total compressive/tensile unloading, the $\{110\}$ and $\{200\}$ grains of the β -Li phase have large tensile/compressive residual strains, and the other subset grains of the α -Mg phase, conversely, exhibit compressive/tensile residual strains. This behavior can be understood largely in terms of the elastic load transfer between soft- and hard-phase that relatively small lattice strain builds up in the soft β -Li phase compared to the hard α -Mg phase during the previous deformation. The lattice strain partitioning in the duplex Mg-Li alloy is schematically shown in Figure 4.6(d), including three distinct stages. In Stage I (below macroscopic yield), both phases are mostly elastic although some certain degree of basal $\langle a \rangle$ slip can be detected as evidenced by the lattice-strain deflection of $\{10\bar{1}0\}$ grains at ~ 58 MPa in Figure 4.6(a). In Stage II (from macroscopic yield to moderately above), the apparent strain splitting is mainly governed by the load partitioning between the soft β -Li phase and hard α -Mg phase. Such a composite behavior gives a higher work-hardening rate than a single-phase α -Mg alone. In Stage III (at large macroscopic plastic strain), the lattice strain splitting behavior turns into an opposite trend than that in Stage II. That is, the lattice strains for the β -Li phase increase more rapidly to higher absolute values, while those for the α -Mg may cease the increase in their absolute values. This can be rationalized by the elevated strain hardening rate in the BCC β -Li phase in this stage, which again contributes

to the macroscopic work-hardening rate. Subtle features like this cannot be obtained from macroscopic test results.

To gain further knowledge on slip behaviors of Mg-Li duplex phases, we examined the evolution of the dislocation density of certain diffraction planes. Based on the theory developed by Ungár et al. [115], the relationship between dislocation density of a specific lattice plane (hkl) and its full width at half maximum (FWHM) of the diffraction peak can be described by the following proportional relation:

$$\rho \sim \frac{FWHM_G^2, hkl}{d_{hkl}^2 b^2 C_r} \quad (4-3)$$

where ρ is the total density of randomly distributed dislocations, hkl is the Miller index, $FWHM_G$ is the Gaussian component of the full width at half maximum of the peak, d is the lattice plane spacing, b is the magnitude of the Burgers vector, and C_r is the contrast factor associated with the dislocation character (edge or screw). Figure 4.7 shows the FWHM values of the $\{10\bar{1}1\}$ and $\{11\bar{2}0\}$ diffraction planes in the α -Mg phase, and $\{110\}$ diffraction planes in the β -Li phase as a function of applied engineering strain. During initial compression, the FWHM values gradually increase, and almost stay stable in following compressive unloading. During the reverse tension, the FWHM values undergo a decrease-increase sequence, then followed by a decrease during tensile unloading. Then during the following compression, the trend of FWHM is the same as the initial compression. To eliminate the influence of the applied load, the FWHM values under total compressive unloading are taken into consideration. It is clear to notice that the $\{10\bar{1}1\}$ and $\{110\}$ peaks under total compressive unloading gradually broaden, which suggest the

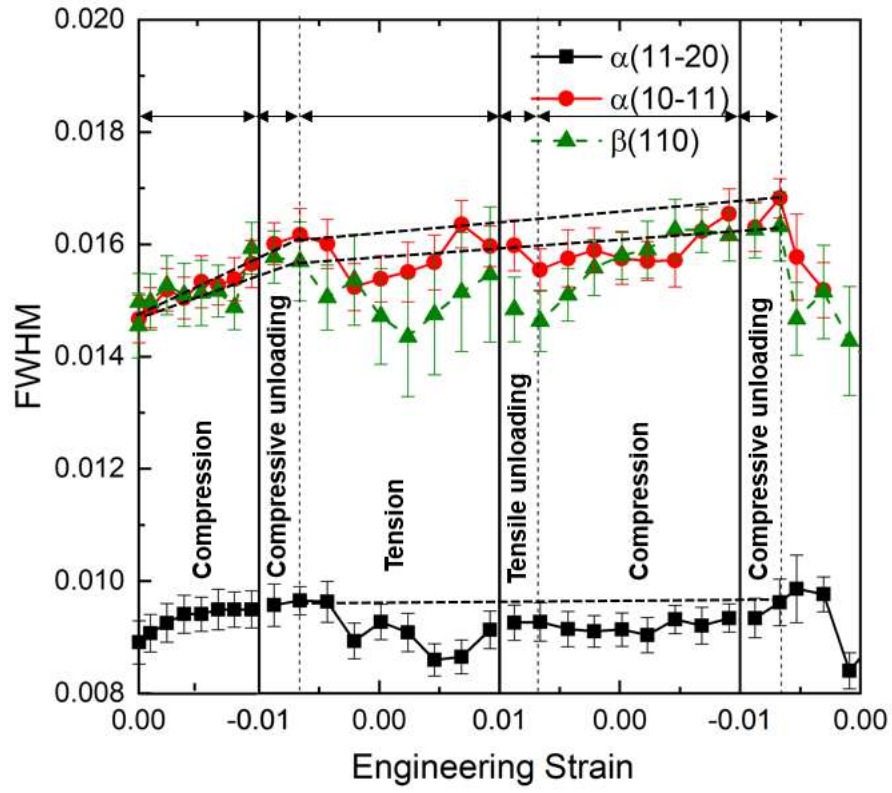


Figure 4.7. Broadening of $\{10\bar{1}1\}$ and $\{11\bar{2}0\}$ diffraction planes in the α -Mg phase, and $\{110\}$ diffraction planes in the β -Li phase as a function of engineering strain.

accumulation of dislocation, while no obvious change in $\{11\bar{2}0\}$ peak can be observed, as displayed in Figure 4.7. For α -Mg phase, the pyramidal $\langle a \rangle$ slipping on $\{10\bar{1}1\}$ plane, therefore, has a high priority in deformation, compared with the prismatic slip on the $\{11\bar{2}0\}$ plane in this duplex Mg-Li alloy. This is consistent with the report by Guo et al. that prismatic slip is not favored to active due to the competition from basal slip and pyramidal slip [107]. For BCC β -Li phase, the significant increase of $\{110\}$ FWHM right before the start of unloading indicates the elevated dislocation activities and thus work hardening rate in correspondence to the above discussion for Stage III.

4.4 Conclusions

The real-time in situ neutron diffraction under a continuous loading condition is performed on a Mg-8Li-3Al-3Ho alloy under a fully reversed strain cycling experiment at room temperature. Individual crystallographic orientations for the α -Mg phase and β -Li phase are obtained using the synchrotron X-ray diffraction. The plastic deformation in the α -Mg phase is accommodated by the basal $\langle a \rangle$ slip and pyramidal $\langle a \rangle$ slip, while the CRSS of the latter is ~ 2.3 times larger than that of the former. On the other hand, the soft β -Li phase exhibits a high work-hardening rate at a large macroscopic plastic strain. The yield sequence and strain splitting behavior amongst various slip systems in the α -Mg phase and different deformation stages of the β -Li phase give rise to the micromechanical origin of an elevated work-hardening rate of the duplex alloy, thus delaying necking and promoting tensile ductility.

CHAPTER V
PLASTIC ANISOTROPY AND TWIN DISTRIBUTIONS NEAR THE
FATIGUE CRACK TIP OF TEXTURED MG ALLOYS FROM IN-
SITU SYNCHROTRON X-RAY DIFFRACTION MEASUREMENTS
AND MULTISCALE MECHANICS MODELING

This article hasn't been published anywhere, nor will it be before I turn in the final version of my ETD, so I didn't include a publication statement.

Abstract

In spite of many potential applications due to their superior mechanical properties, magnesium alloys still find many practical restrictions primarily due to our limited knowledge of their failure mechanisms. In-situ and non-destructive strain measurements on the microstructural scales are critical in understanding the fatigue crack behavior, which has greater advantages than the macroscopic measurements based on replica technique and the small-scale mechanical testing that cannot easily provide a complete view on the synergy of many scale-dependent deformation and failure mechanisms. This work exploits the state-of-the-art synchrotron X-ray diffraction technique to attain in-situ strain mapping results, with high spatial resolution, near a fatigue crack tip in the highly textured ZK60 Mg alloy. The accurate measurements in the plastic zone are compared to a micromechanical modeling framework, in which the steady fatigue crack is simulated by an irreversible, hysteretic cohesive interface model and the surrounding plasticity by Hill anisotropic plasticity. The general agreement in the anisotropic deformation fields is reached down to half of the plastic zone size towards the crack tip, while the discrepancy right at the crack tip can be used to develop an inverse model to extract the material-specific fatigue damage evolution in our future work. It is surprisingly noted that twin activities are localized in a narrow band from the tip to the wake of the fatigue crack, irrespective of the crack growth direction of this textured alloy. Such twin-crack interactions are understood

here by our crystal plasticity finite element simulations with several representative crystallographic orientations.

5.1 Introduction

In the past several decades, significant advances have been developed in the studies of structural alloys, which are at the very heart of modern energy and transportation systems, with the help of modern material testing and characterization techniques across many length scales. Simultaneously, the development of new engineering designs and applications brings tremendous new research opportunities and challenges. Of particular importance is the growing interest in understanding the deformation and failure mechanisms of Mg alloys due to their rapid utilization in the automotive and aerospace industries. Due to the low-symmetric nature of their hexagonal-close-packed (HCP) structure, Mg alloys have desirable combined properties of low density, high strength-to-weight ratio, and high specific stiffness. Most prior studies have focused on the interplay between the texture and complex slip/twin systems [116-119], the development of new Mg alloys with improved ductility [9, 102], and the use of advanced diffraction techniques to identify the deformation mechanisms under simple loading conditions [20, 21, 23, 120, 121]. Under uniaxial loading conditions, the deformation of most Mg alloys at room temperature is mainly dominated by basal $\langle a \rangle$ slip, prismatic $\langle a \rangle$ slip, and $\{10\bar{1}2\}$ extension twins. However, the slip and twin behaviors under multiaxial loading conditions are quite sensitive to the material texture. For example, Koike et al. [3] reported that the anomalous formation of extension twinning under tensile loading parallel to TD (transverse

direction) or RD (rolling direction) in basal textured Mg alloys to accommodate strain incompatibility between soft grains (which deform by strong basal slip activity) and neighboring hard grains. Using in-situ neutron diffraction and electron backscatter diffraction (EBSD) techniques, Sofinowski et al. [121] found that different pre-strains or strain path changes can lead to a variety of complex deformation modes, some of which may be beneficial in activating multivariant secondary twinning and improving the ductility limit.

Interplays amongst microstructure, texture, and various slip/twin systems have been fairly understood in the above test conditions where deformation fields are nominally uniform, but it is not entirely so for their roles in the fracture and fatigue behavior of Mg alloys. We now discuss fracture and fatigue studies of Mg alloys from these two lines of thought.

First, it has been widely recognized that metallic materials are generally toughened intrinsically by inducing a significant plastic-zone size ahead of the crack tip, and the shape and size of the plastic zone play an important role in influencing the fatigue crack extension. It is thus of great technical importance to determine the effect of microstructure, texture, and slip/twin modes on the development of the plastic zone during the fatigue crack growth, which is termed as the “clean” process zone in this work. Earlier reports reveal that the specimen orientation has a significant influence on the fatigue crack growth rate and crack path [122]. Han et al. [123] reported that more twins are observed in the transverse direction (TD) specimens (loading axis perpendicular to the extruded direction) than in the extruded

direction specimens (loading axis parallel to the extruded direction). Kaushik et al. [124, 125] have performed Mode-I fracture tests on magnesium single crystals using three notched three-point-bend (TPB) specimens with different orientations. Although extension twins were observed in these three samples, the patterns of twin development for the samples are different. Twins initially formed near the specimen edge ahead of the notch root and then extended toward it with the increase of load for the sample with c-axis oriented along the notch direction, while twins spread out from the notch root in the other two samples with basal plane aligning with notch direction. However, most previous studies, are limited to surface-based characterizations of cracks [126, 127], and the lack of non-destructive bulk measurements unavoidably cast limitation on the correlation between micromechanism and fatigue performance. In recent two decades, a number of studies have employed the modern diffraction techniques (neutron and synchrotron X-ray diffractions, to be specific) to obtain an in-situ, non-destructive mapping of the entire elastic strain fields near the fatigue crack tip, e.g., the study of overload effects on the fatigue crack growth behavior in commercial stainless steels or aluminum alloys [128-132] and the lattice strain distribution near the fatigue crack tip of HCP zirconium alloys [133]. Here in this work, we will aim to report a full field mapping in a heavily textured Mg alloy with various fatigue crack growth directions, and thus provide an experimental set of information for the deconvolution of texture and deformation-mode effects in our fundamental understanding of the plastic process zone.

Second, the quantitative characterization of damage processes right at the fatigue crack tip, termed as the “messy” process zone in this work here, is extremely challenging and not easily amenable to traditional experimental methods. Based on the synchrotron X-ray tomography, Withers [134] has summarized the latest progress on the direct observation of the crack growth front and other defects such as voids on the micrometer resolution. Such measurements for the fatigue crack growth behavior of Mg alloys are limited. One successful example can be found in the work of King et al. [135], which carefully traced the interactions between crack front and grain boundaries, and therefore enriched the previous modeling work by Zhai et al. [30] on the fatigue crack path selection in Mg alloys. However, these studies cannot determine the crack resistance, or provide insightful strategies in microstructural and texture design for the control of the fatigue crack growth rate. A statistically homogenized view of these damage processes is thus needed to connect to the plasticity-induced resistance to crack growth. In this work, our attempt along this line will be restricted to a phenomenological cohesive zone approach that helps generate a steady fatigue crack. As will be shown later in this paper, the simulated clean process zone due to plastic deformation agrees very well with experimental measurements of the chosen textured Mg alloy, while the differences on the messy process zone could be used as inputs for an inverse analysis [136, 137] to extract a cohesive interface relationship that represents the actual damage processes. The latter will be presented in our subsequent work.

In this paper, synchrotron X-ray diffraction (S-XRD) measurements are employed to investigate the deformation fields around a crack tip in a ZK60 Mg alloy sample with strong texture and plastic anisotropy. The high penetration depth of S-XRD for most materials combined with the excellent brilliance and high spatial resolution leads it unique in studying the relative contributions of twinning and dislocation slip to the crack tip plasticity by mapping internal strains and texture variations during in-situ loading [133, 134]. During the fatigue crack growth, we note that the stress multiaxiality becomes increasingly more complex along the crack path [130]. Consequently, a material point in the vicinity of a fatigue crack tip will experience a complex history of multiaxial stress states, which has significant implications on the plastic deformation of Mg alloys at the macroscopic scale (i.e., texture design leading to anisotropic plasticity that potentially resists the crack growth much more in some certain directions) and the mesoscale (i.e., deformation of a finite number of grain aggregates around the crack tip to accommodate the multiaxial stress fields). A full-field strain mapping to understand the above complex stress history in the plastic zone is essential for the fatigue-crack-growth studies in Mg alloys. The small plastic zone around the crack tip requires a small beam size with a high spatial resolution to resolve the difference between the plastic zone near the crack tip and the surrounding area – this is ideal for S-XRD experiments. Furthermore, a simulated steady fatigue crack will be developed by an irreversible, hysteretic cohesive interface model to study the stress/strain distributions near the crack tip for the anisotropic plastic material used in this study, and the crystal plasticity finite element method (CPFEM) will

be used to understand the twin distributions that are found to surprisingly localize in the plastic wake.

5.2 Experimental procedures

5.2.1 Materials

The ZK60 Mg alloy with the T5 temper is used in this work. This material is fabricated by an extrusion process with a width of 279.4 mm and a thickness of 76.2 mm. The S-XRD technique was utilized to measure the initial crystallographic texture and lattice strain distributions of ZK60 Mg alloy at the beamline 11-ID-C, the Advanced Photon Source (APS), Argonne National Laboratory (ANL). The wavelength employed during the S-XRD measurement was 0.1173 Å and the sample to detector distance was 1800 mm. The texture data was collected by mounting the pin sample on a rotary stage and by rotating from 0° to 180° with a 30° step size, obtaining full pole coverage, as schematically shown in Figure 5.1(a). The Debye-Scherrer rings were converted into diffraction patterns using the Fit2D software, and then the Material Analysis Using Diffraction (MAUD) software was used to analyze these diffraction data and get the raw pole figure data by performing Rietveld refinement [138]. MTEX quantitative texture analysis software [139] was employed to plot the (10 $\bar{1}$ 0), (0002), and (10 $\bar{1}$ 1) pole figures (PF) of the ZK60 alloy, as displayed in Figure 5.1(b), where pole intensities are represented in terms of multiples of a random distribution (mrd). By checking the orientation distribution function (ODF) in the [0, 10°, 20°, 30°, 50°, and 60°] sections of φ_2 in Figure 5.1(c), this alloy exhibits a combination of strong {0001}<10 $\bar{1}$ 0> rolling texture and weak

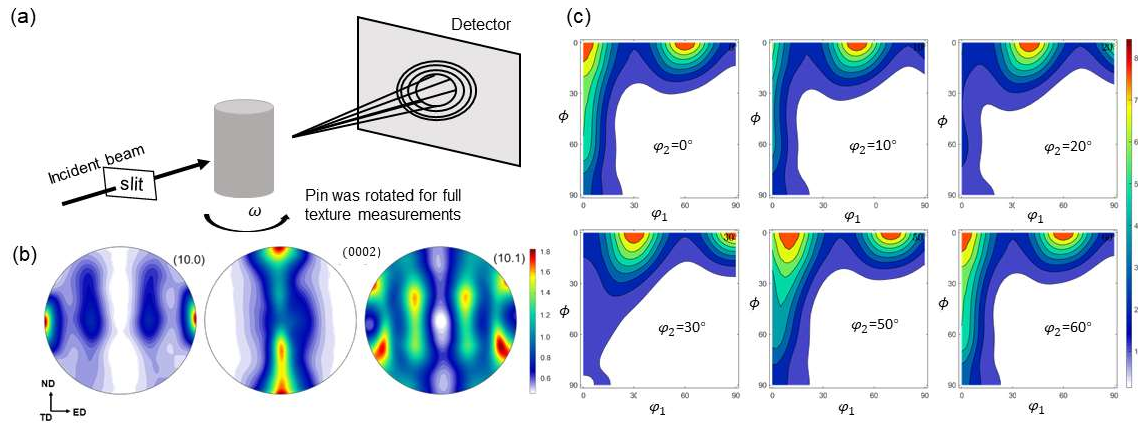


Figure 5.1. (a) A schematic of the diffraction measurement set-up and ω corresponds to the rotation axis for the texture measurement using S-XRD, (b) $(10\bar{1}0)$, (0002) , and $(10\bar{1}1)$ pole figures, where pole intensities are represented in terms of multiples of a random distribution (mrd), (c) orientation distribution function (ODF) in the $[0, 10^\circ, 20^\circ, 30^\circ, 50^\circ, \text{ and } 60^\circ]$ sections of ϕ_2 .

$\langle 10\bar{1}0 \rangle$ fiber. The grain size of this Mg alloy is about $7 \pm 4 \mu\text{m}$. Uniaxial dog-bone tensile samples were cut on the ED-ND plane with the longitudinal direction parallel to ED, ND, and 45° between the ED and ND in Figure 5.2(a). The uniaxial stress-strain curves are shown in Figure 5.2(b), which yield a 0.2% offset strength ~ 260 MPa parallel to the ED, ~ 145 MPa parallel to the ND, and ~ 153 MPa parallel to the 45° direction between the ED and ND.

5.2.2 Fatigue-crack-growth test

To analyze the effect of texture on the fatigue crack propagation behavior of Mg alloys, a series of compact-tension (CT) specimens, prepared according to the American Society of Testing and Materials (ASTM) Standards E647-99 [41] with the geometry shown in Figure 5.3(a), were cut on the extruded direction (ED)-normal direction (ND) plane with the notch direction parallel to ED, ND, and the 45° between the ED and ND, as in Figure 5.3(b). They are denoted as 0° sample, 45° sample, and 90° sample, respectively. Actual fatigue-crack growth tests were performed under cyclic loading at $f = 10 \text{ Hz}$ at a constant load ratio $R = 0.1$ at room temperature using a Materials Testing System (MTS) 810 servo-hydraulic machine. The CT specimens were pre-cracked to a crack length of 7 mm with a K (stress-intensity factor, $K_{\text{max}}=10 \text{ MPa}\sqrt{\text{m}}$, $K_{\text{min}}=1 \text{ MPa}\sqrt{\text{m}}$) control mode and then further crack to 9 mm under a constant-load ΔP control mode [$\Delta P = P_{\text{max}} - P_{\text{min}}$, where P_{max} and P_{min} are the applied maximum (650 N) and minimum (65 N) loads, respectively]. The crack length was measured by a crack-opening-displacement (COD)

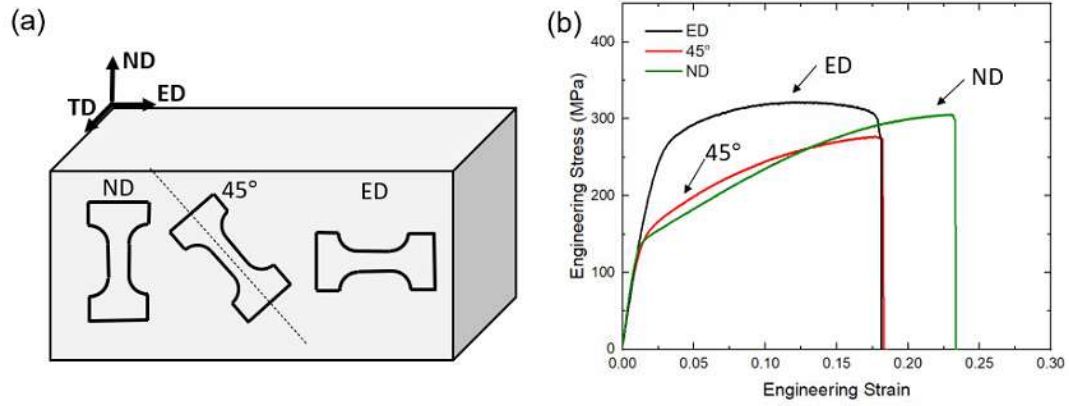


Figure 5.2. (a) Three tension samples of extruded ZK60 Mg alloy, oriented differently with respect to the extrusion direction, and (b) stress-strain curves for the extruded ZK60 Mg alloy with different loading orientations.

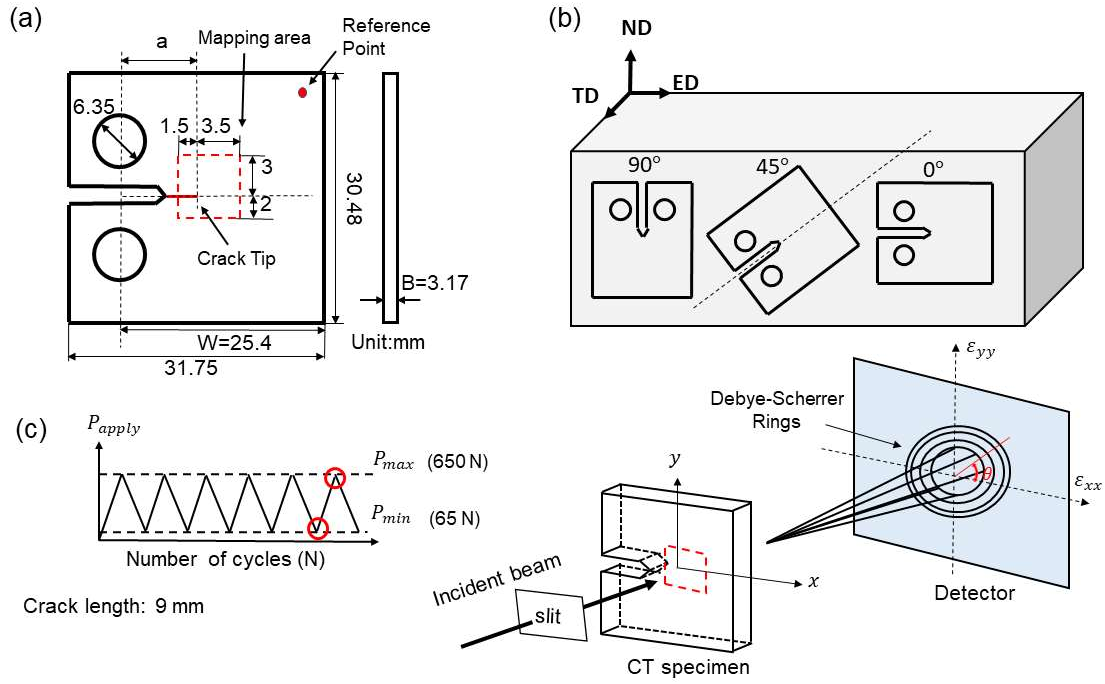


Figure 5.3. (a) CT specimen geometry and schematic of the strain mapping area in the red dashed square, (b) three compact tension (CT) specimens of extruded ZK60 Mg alloy, oriented differently with respect to the extrusion direction, and (c) a schematic of the diffraction measurement setup for strain mapping measurement using S-XRD, where x presents the crack growth direction, and y presents the crack opening direction.

gauge [41], and then further confirmed by scanning electron microscopy (SEM). The stress-intensity factor (K) can be obtained by the following equation [41]:

$$K = \frac{P(2+\alpha)}{B\sqrt{W}(1-\alpha)^{1.5}} (0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4) \quad (5-1)$$

where P is applied load, B is the thickness, W is the width, and α is the ratio of crack length and width. The cracked CT samples were then taken for the in-situ S-XRD strain mapping.

5.2.3 In-situ S-XRD measurement

In-situ S-XRD measurement of the strain field around the crack tip with a crack length of 9 mm was performed at two load levels, $P_{\min}$ (65 N) and $P_{\max}$ (650 N). The setup of the strain mapping is schematically shown in Figure 5.3(c), and the strain mapping area is pointed out by the red dashed square in Figure 5.3(a), which, horizontally, covers from -1.5 mm to +3.5 mm, and, vertically, from -2 mm to +3 mm by setting the crack tip to be the reference point (0, 0). To accurately evaluate the strain, the beam size of $100 \mu\text{m} \times 100 \mu\text{m}$ with a step of $100 \mu\text{m}$ was used in this measurement. For each load level, we held the sample and conducted the strain mapping. The holding time during the S-XRD data acquisition period did not affect the deformation behavior since creep was negligible at room temperature. The General Structure Analysis System (GSAS) II software [140] was used to reduce the diffraction rings and obtain the diffraction patterns by azimuthally averaging the ring pattern over $\pm 5^\circ$ with respect to a series of θ values ($\theta = 0^\circ, 45^\circ$, and 90°). θ is the angle as defined in Figure 5.3(c). The lattice strains were calculated according to the following equation:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad (5-2)$$

where d and d_{hkl}^0 are the strained and unstrained d_{hkl} spacings for a specific grain group, respectively. The d spacings were determined by the Rietveld-fitting for these three samples in this work. The unstrained d_0 was measured at the edge of each CT sample, marked with a red dot in Figure 5.3(a). According to Poulsen et al. [141], the strain tensor (ε_{ij}) can be determined by fitting the angular variation of ε_i to the following expression:

$$\varepsilon_i = \varepsilon_{xx}\cos^2(\theta_i) + 2\varepsilon_{xy}\cos(\theta_i)\sin(\theta_i) + \varepsilon_{yy}\sin^2(\theta_i) \quad (5-3)$$

where ε_{xx} , ε_{yy} and ε_{xy} represent the strain along the crack growth direction, crack opening direction, and shear strain, respectively.

5.2.4 Finite element simulations

A cohesive interface model is used to develop a steady fatigue crack to investigate the stress/strain distributions near the crack tip for ZK60 Mg alloy with strong plastic anisotropy. A typical interface constitutive law relates the tractions acting on two-bonded interfaces to the separation between them. In order to realize steady fatigue crack numerically, an irreversible, hysteretic cohesive interface model is introduced based on Nguyen et al. [68] and further modified in [4, 5], which can phenomenologically accumulate crack tip damage and dissipate fracture energy during the fatigue cycles. The relationship between the normal traction T_n and the normal separation Δ_n is given by the following equation:

$$\frac{T_n}{\sigma_{\max}} = \frac{\Delta_n}{\delta_n} \exp\left(1 - \frac{\Delta_n}{\delta_n}\right) \quad (5-4)$$

where T_n and Δ_n represent the interface traction and interface separation, respectively, $\sigma_{\max}$ is the interface strength, and δ_n is a characteristic length scale. Then the unloading-

reloading hysteresis is introduced by considering the unloading stiffness, K^- , and the reloading stiffness, K^+ , separately,

$$\dot{T}_n = \begin{cases} K^- \dot{\Delta}_n, & \dot{\Delta}_n < 0 \\ K^+ \dot{\Delta}_n, & \dot{\Delta}_n > 0 \end{cases} \quad (5-5)$$

$$K^- = \frac{T_n^{\text{unload}}}{\Delta_n^{\text{unload}}} \quad (5-6)$$

$$\dot{K}^+ = \begin{cases} (K^+ - K^-) \dot{\Delta}_n / \delta_a, & \dot{\Delta}_n < 0 \\ -K^+ \dot{\Delta}_n / \delta_f, & \dot{\Delta}_n > 0 \end{cases} \quad (5-7)$$

where δ_a and δ_f are length parameters that are associated with the damage behavior during the unloading and reloading process, and T_n^{unload} and Δ_n^{unload} are the normal traction and separation at the unloading point, resulting in a constant K^- at each unloading point. The above relationship is schematically shown in Figure 5.4(a). The interface model in Eqs. (5-4 – 5-7) has been implemented as a user-defined element (UEL) subroutine in the commercial finite element (FE) software, ABAQUS [142]. One hidden issue in cohesive one simulations is the snap-back instability when the calculated crack bridging size is significantly smaller than the crack size. The resulting numerical divergence problem can be avoided by introducing a fictitious viscosity to the above Eq. (5-4); details on this procedure can be found in [143].

In the finite element simulation setup, a standard CT specimen adopted the same geometry in Figure 5.3(a) is used to simulate the fatigue crack, which is subjected to cyclic loading of 65-650 N ($P_{\text{max}}/P_{\text{min}} = 0.1$), as schematically shown in Figure 5.4(b). The representative mesh of the CT sample is shown in Figures 5.4(c) and 5.4(d), where the cohesive interface element is introduced along the crack growth plane while the

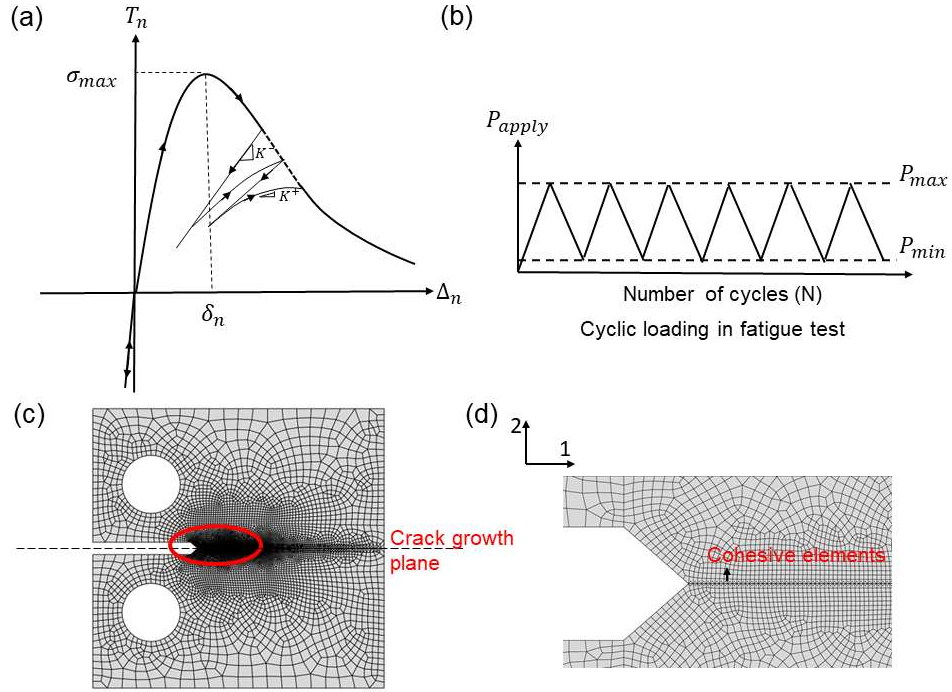


Figure 5.4. (a) Traction-separation (T_n - Δ_n) relationship in the irreversible, hysteretic cohesive interface model, where the unloading process corresponds to a straight line extending through the coordinate origin, and the loading process exhibits a gradual softening behavior, (b) The applied cyclic loading levels with a ratio of $P_{min}/P_{max} = 0.1$, which result in the alternating stress intensity factors, K_{min} and K_{max} , (c) Cohesive interface simulations of a CT specimen (The geometric sizes are taken from Figure 3), and (d) Enlarged view of the cohesive element on the crack tip.

surrounding medium is elasto-plastic. The element size should be smaller than the crack bridging size (roughly scaled as $E\sigma_{max}/\delta_n$).

In order to address the twinning distribution around the crack tip of these CT samples, especially in the plastic zone area, a crystal plasticity finite element method (CPFEM) accounting for both slip and twin, following the work of Staroselsky and Anand [144], is employed in this study. The detailed constitutive laws for this model are described in the Supplementary material. The total slip and twinning systems considered in the constitutive model are listed in Table 5.1. The above constitutive equations are implemented into the finite element software ABAQUS explicit VUMAT. The detailed procedure of the crystal plasticity theory and its constitutive equations can be found in the work of Staroselsky and Anand [144].

5.3 Results

5.3.1 Fatigue-crack propagation behaviors

Figure 5.5 presents the fatigue-crack-growth rate (da/dN) as a function of ΔK for the 0° sample, 45° sample, and 90° sample. The fatigue crack growth characteristics of the three samples are broadly similar to each other in that the crack-growth rate increased linearly with increasing ΔK in the steady fatigue crack growth region, which follows the Paris law. However, when the steady fatigue crack propagation is achieved, the 90° sample presents the largest crack-growth rate, and the 0° sample exhibits the smallest crack-growth rate. The values of the threshold stress intensity factor are approximately the same for the 45° sample and the 90° sample, while a relatively large value is observed for the 0° sample.

Table 5.1. Slip and twin systems in the HCP crystal plasticity finite element model.

Deformation system	System
Basal $\langle a \rangle$ slip	$\{0001\} \langle 11\bar{2}0 \rangle$
Prismatic $\langle a \rangle$ slip	$\{1\bar{1}00\} \langle 11\bar{2}0 \rangle$
Pyramidal $\langle a \rangle$ slip	$\{1\bar{1}01\} \langle 11\bar{2}0 \rangle$
Pyramidal $\langle a+c \rangle$ slip	$\{11\bar{2}2\} \langle \bar{1}\bar{1}23 \rangle$
Extension Twinning	$\{10\bar{1}2\} \langle \bar{1}011 \rangle$
Compression Twinning	$\{\bar{1}011\} \langle \bar{1}012 \rangle$

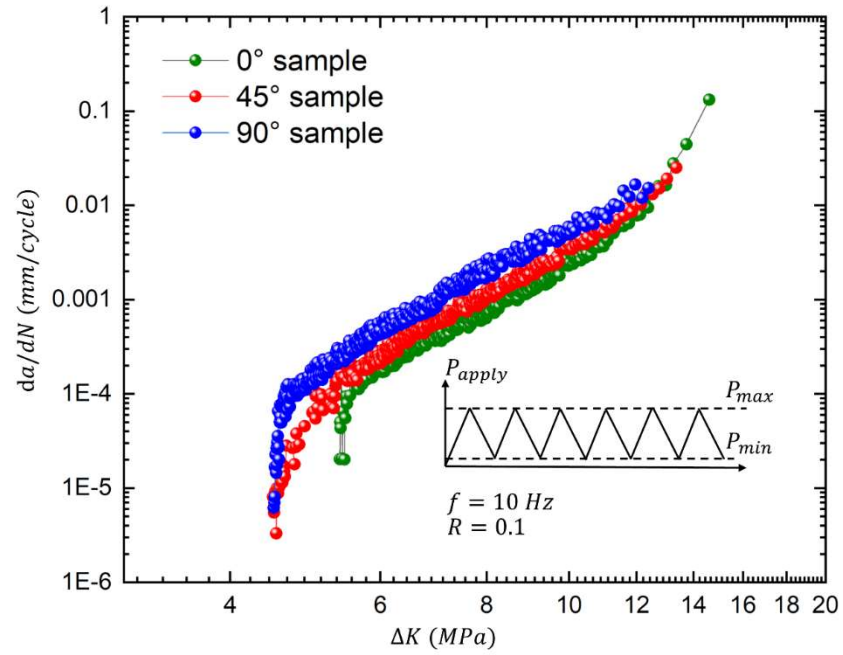


Figure 5.5. Fatigue crack growth rate (da/dN) with applied stress intensity factor (ΔK) for the 0° sample, 45° sample, and 90° sample.

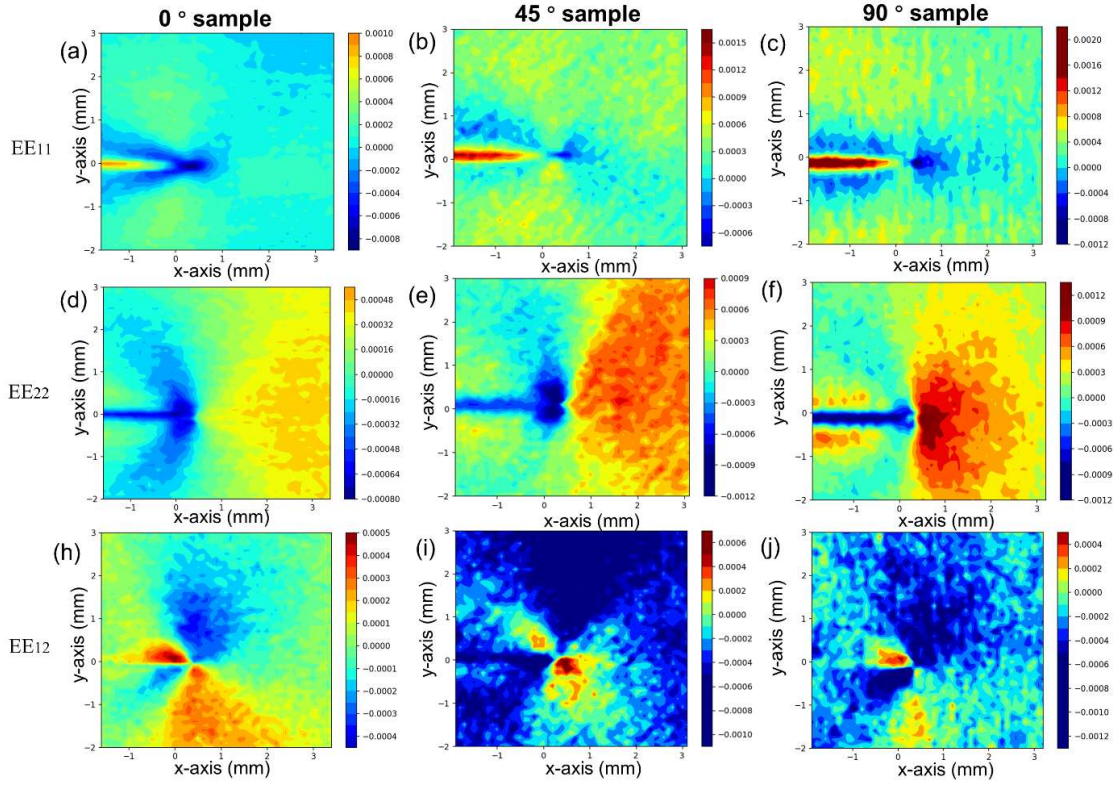
Hence, specimen orientation has a significant influence on crack growth, especially in the threshold value. Such orientation dependence in the fatigue-crack-growth rate has been reported in Mg alloys [122]. However, further investigation on reasonable factors affecting its fracture resistance has not yet been extensively examined.

5.3.2 Lattice strain and stress fields and their evolution near a fatigue crack tip

The spatially resolved lattice strain mapping results along the crack opening direction (EE₂₂), the crack growth direction (EE₁₁), and the shear strain (EE₁₂) as a function of the distance from the crack tip for the 0° sample, 45° sample, and 90° sample with a crack length of ~ 9 mm at K_{min} and K_{max} are shown in Figure 5.6 and Figure 5.7, respectively. It is important to note that the lattice strains measured by diffraction techniques directly are elastic strains on a certain set of grains that satisfy the diffraction conditions, and the lattice strain values here represent the average values over the gauge volume of 100×100×3170 μm³. The plastic zone size (R_p) can be estimated by the following equation:

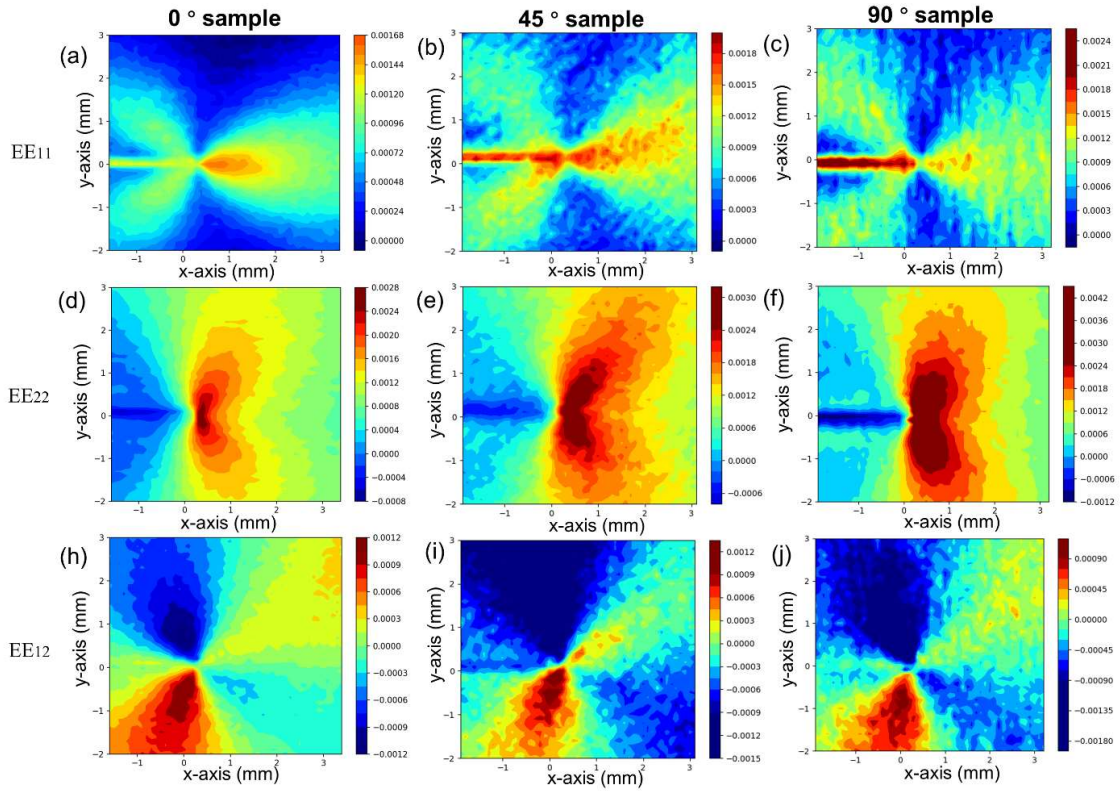
$$R_p = \frac{1}{2\pi} \left(\frac{K_I}{\sigma_Y} \right)^2 \quad (5-8)$$

where σ_Y is the yield stress (~ 260 MPa along with ED, ~ 145 MPa along with ND, and ~ 153 MPa along 45° between the ED and ND), and K is the stress intensity factor, calculated according to Eq. (5-1), which yields a value of ~ 8 MPa√m at P_{max} and ~ 0.8 MPa√m at P_{min} for these three samples with a crack length of 9 mm. Therefore, according to Eq. (5-8), the 90° sample has a relatively small plastic zone at K_{max}, compared with that in the 0° sample, and the 45° sample. Given the 100 μm×100 μm beam size and 100 μm step size in



@ $K_{\min} = 0.8 \text{ MPa}\sqrt{m}$

Figure 5.6. Lattice strain contours measured as a function of distance from the crack tip for the 0° sample, 45° sample, and 90° sample at $K_{\min}$: (a - c) strain along the crack growth direction (EE_{11}); (d - f) strain along the crack opening direction (EE_{22}); (h - j) shear strain around the crack tip (EE_{12}). The (0, 0) coordinate indicates the approximate position of the crack tip.



@ $K_{\max} = 8 \text{ MPa}\sqrt{m}$

Figure 5.7. Lattice strain contours measured as a function of distance from the crack tip for the 0° sample, 45° sample, and 90° sample at $K_{\max}$: (a - c) strain along the crack growth direction (EE_{11}); (d - f) strain along the crack opening direction (EE_{22}); (h - j) shear strain around the crack tip (EE_{12}). The (0, 0) coordinate indicates the approximate position of the crack tip.

the diffraction mapping, the diffraction measurement can capture the millimeter-sized plastic zone ahead of the crack tip at $K_{\max}$.

The lattice strains in these three samples are examined with particular emphasis on the similarities and differences between the contours of lattice strain caused by the plastic anisotropy in Mg alloy. The general characteristics in the lattice strain field evolution are similar in these three samples except for some small differences. Compressive residual strain fields are observed at the crack tip in both the EE_{11} contour and EE_{22} contour in these three samples at $K_{\min}$ in Figure 5.6. The compressive lattice strain, EE_{22} , along the crack path transitions into a tensile zone ahead of the crack tip at $K_{\min}$, for example, ~ 0.3 mm in front of the crack tip in the 0° sample. While the tensile lattice strain, EE_{11} , along the crack path transitions into a compressive zone behind the crack tip at $K_{\min}$. As one might expect, with the increase of load level from $P_{\min}$ (or $K_{\min}$) to $P_{\max}$ (or $K_{\max}$), the magnitude of lattice strains (EE_{11} , EE_{22} , and EE_{12}), especially ahead of the crack tip, is significantly increased due to the higher stress concentration, as shown in Figure 5.7. A tensile lattice strain field, EE_{22} , is developed ahead of the crack tip, while a compressive lattice strain field is still observed behind the crack tip at $K_{\max}$, and the position of the peak EE_{22} is observed ahead of the crack tip.

Although the general trends of the lattice strain fields are similar in these three samples, there are some differences. From Figure 5.6, we can see for the tensile strain fields in both the contours of EE_{11} and EE_{22} at $K_{\min}$, the largest tensile strain magnitude is observed in the 90° sample, the moderate one in the 45° sample, and the least one in the 0°

sample. Given the same applied $K_{\max}$ in Figure 5.7, the 90° sample loaded along ED exhibits the largest lattice strain field, EE_{22} , ahead of the crack tip, compared to the 0° sample loaded along with ND and the 45° sample loaded along 45° direction between the ED and ND. This can be explained by the largest 0.2% offset strength parallel to ED, resulting in a relatively large elastic strain field in the 90° sample. Distinct to the symmetric lattice strain fields (EE_{11} and EE_{22}) on the crack growth plane in the 0° sample and the 90° sample, the asymmetric shape is observed in the 45° sample, as displayed in Figure 5.6-5.7. Both the strain component fields, EE_{11} and EE_{22} , around the crack tip are asymmetric on the crack growth plane in the 45° sample at $K_{\max}$ and $K_{\min}$. These differences are principally caused by the anisotropic mechanical response in the ZK60 Mg alloy.

According to the ASTM E399-90 for linear-elastic plane-strain fracture toughness [145], a crack in a fracture mechanics specimen is likely to undergo plane strain conditions if its thickness ($B=3.17 \text{ mm}$) is greater than $2.5 (K/\sigma_y)^2$, which gives for the 0° sample $B > 7.6 \text{ mm}$, for the 45° sample $B > 6.8 \text{ mm}$, and for the 90° sample $B > 2.3 \text{ mm}$ at $P_{\max}$. These thicknesses will be a little bit smaller for the fatigue sample considering the effective stress intensity (K_{Ie}). At first glance, the 0° sample and the 45° sample are under a plane-stress condition, while the 90° sample is in a plane-strain condition at $P_{\max}$; however, all these samples at $P_{\max}$ are in the plane-stress condition, which will be verified by the cohesive interface model in Section 5.3.5.1. This is due to the plastic deformation near the crack tip, where the linear elastic fracture mechanics (LEFM) does not work anymore in that region. And the stress field at $P_{\min}$ is calculated using a plane-strain approximation for these

samples. As the elastic mechanical response of Mg alloys is nearly isotropic [18, 100], the relationship between the stress and strain can be expressed as the generalized Hooke's Law,

$$\sigma_i = \frac{E}{1+\nu} [\varepsilon_i + \frac{\nu}{(1-2\nu)} (\varepsilon_x + \varepsilon_y + \varepsilon_z)] \quad (5-9)$$

where E ($= 50$ GPa) is the young's modulus, and ν ($= 0.3$) is Poisson's ratio. For the plane-stress state where the $\sigma_{zz} = \sigma_{xz} = \sigma_{yz} = 0$, Eq. (5-9) can be re-written as a matrix equation:

$$\begin{Bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{xy} \end{Bmatrix} = \frac{E}{1-\nu^2} \begin{bmatrix} 1 & \nu & 0 \\ \nu & 1 & 0 \\ 0 & 0 & 1-\nu \end{bmatrix} \begin{Bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{xy} \end{Bmatrix} \quad (5-10)$$

For the plane-strain state where the $\varepsilon_{zz} = \varepsilon_{xz} = \varepsilon_{yz} = 0$, Eq. (5-9) can be re-written as a matrix equation:

$$\begin{Bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{xy} \end{Bmatrix} = \frac{E}{(1+\nu)(1-2\nu)} \begin{bmatrix} 1-\nu & \nu & 0 \\ \nu & 1-\nu & 0 \\ 0 & 0 & 1-2\nu \end{bmatrix} \begin{Bmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{xy} \end{Bmatrix} \quad (5-11)$$

$$\sigma_{zz} = \frac{E\nu}{(1+\nu)(1-2\nu)} (\varepsilon_{xx} + \varepsilon_{yy}) \quad (5-12)$$

The calculated stress distributions converted from the measured lattice strain for these three samples are shown in Figures 5.8-5.9. All three specimens display analogous tendencies of the stress field evolution along the crack path. Reverse plastic zones with a triaxial compressive stress state ($\sigma_{11} \approx \sigma_{22} \approx 2\sigma_{33}$) in the crack tip are developed in these samples at $K_{\min}$, as displayed in Figure 5.8. Our results are consistent with those recorded by Lee et al. [131] using neutron diffraction for 316 steel that a residual reverse plastic zone is observed at the crack tip. The compressive zones at the crack tip are developed as a result of the prior plastic deformation before unloading. It can be evidently seen from Figure 5.9 that the stress multiaxiality near the crack tip transitions from a biaxial tension-

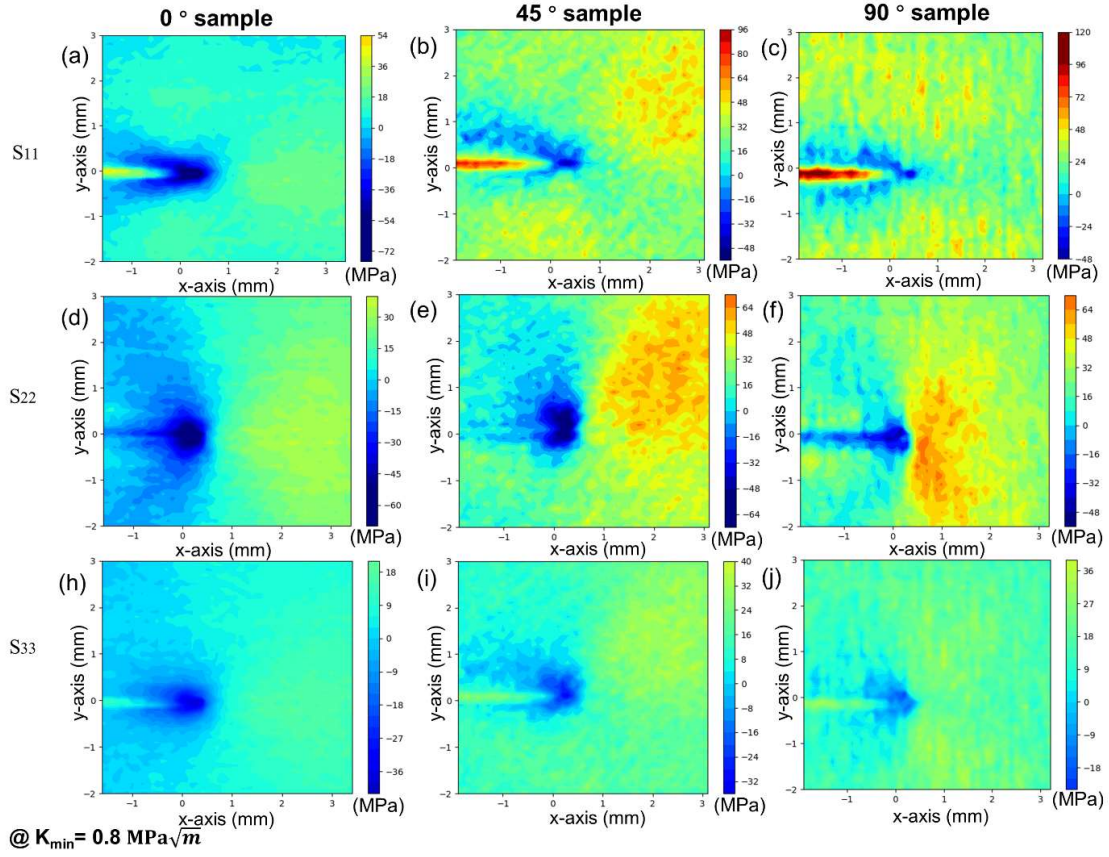


Figure 5.8. Stress contours converted from the measured lattice strains as a function of distance from the crack tip for the 0° sample, 45° sample, and 90° sample at $K_{\min}$: (a - c) strain along the crack growth direction (S11); (d - f) strain along the crack opening direction (S22); (h - j) normal to the plate direction (S33). The (0, 0) coordinate indicates the approximate position of the crack tip.

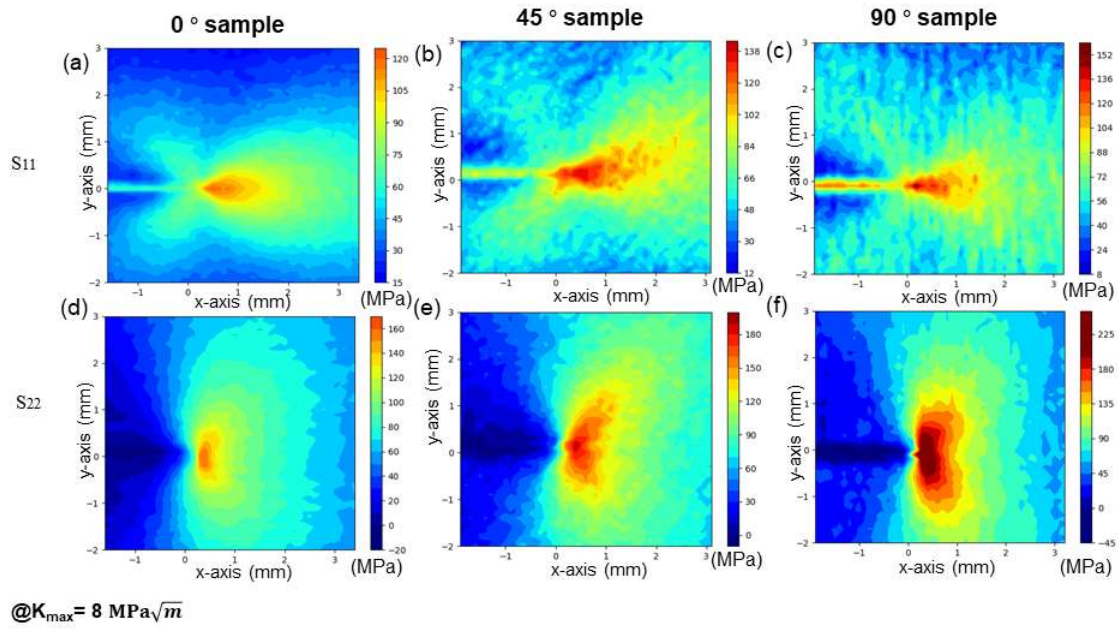


Figure 5.9. Stress contours converted from the measured lattice strains as a function of distance from the crack tip for the 0° sample, 45° sample, and 90° sample at $K_{\max}$: (a - c) strain along the crack growth direction (S_{11}); (d - f) strain along the crack opening direction (S_{22}). The (0, 0) coordinate indicates the approximate position of the crack tip.

compression stress state ($\sigma_{11} \approx -2\sigma_{22}$) in the plastic wake region, and to a biaxial tensile stress state ($\sigma_{11} < \sigma_{22}$) in the plastic zone region, and to a biaxial tensile stress state in the linear elastic region at $K_{\max}$. A schematic illustration of the deformation fields near a steady fatigue crack tip with Mises plasticity is shown in Figure 5.10. Clearly, the increase of the applied stress intensity factor to $K_{\max}$ leads to a tensile plastic deformation region in front of the fatigue crack tip, and the unloading from $K_{\max}$ to $K_{\min}$ results in a reverse compressive residual stress zone at the crack tip. The tensile stress ahead of the crack tip acts as the driving force for crack propagation, while the compressive stress behind the crack tip serves as a crack closure effect that resists crack growth.

There are also some differences between the stress distribution due to the strong plastic anisotropy in the ZK60 Mg alloy. Similar to the asymmetric contour of lattice strain in the 45° sample, the contours of stress components (σ_{11} and σ_{22}) also exhibit asymmetry on the crack-growth plane. The contour of the σ_{22} in the 90° sample presents the largest peak stress ahead of the crack tip compared to the other two samples at $K_{\max}$, which may contribute to the highest fatigue crack propagation rate. Comparing the compressive stress fields around the crack tip at $K_{\min}$, relatively larger reverse compressive stresses are distributed in a broader zone size in the 0° sample, followed by the 45° sample. The larger reverse plastic zone with higher stress magnitude is more important than crack closure in influencing crack growth [130, 146], which contributes to the greater crack-growth retardation in the 0° sample.

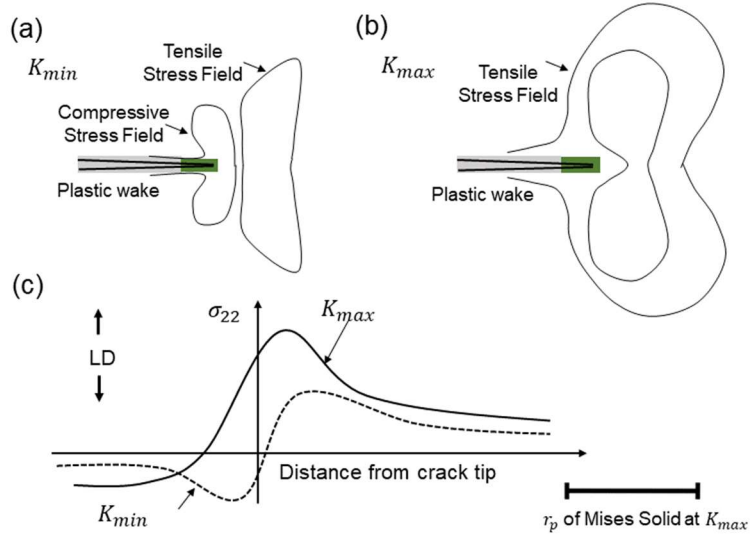


Figure 5.10. Schematic illustration of the contours of stress σ_{22} near a steady fatigue crack tip using a Mises plastic model at K_{max} and K_{min} , respectively. (c) Stress σ_{22} evolution along the crack path at K_{min} and K_{max} .

5.3.4 Diffraction intensity and twin evolution near a fatigue crack tip

Extension twinning as the most important twinning system has been extensively investigated by diffraction techniques in the past decade [17, 18, 92]. The occurrence of extension twinning can be easily captured by the variation of peak intensity in twin-related crystallographic orientation. i.e., the twin-related (0002) and (10 $\bar{1}$ 0) orientations in Mg alloys [24, 40, 56, 147]. If extension twinning occurs, the (0002) peaks would appear at a relative $\sim 90^\circ$ to the initial (0002) orientation angles due to the reorientation angle of extension twinning at 86.3° [148], as schematically illustrated in Figure 5.11(d). The diffraction patterns discussed here are obtained by azimuthally averaging the ring pattern over $\pm 5^\circ$ along the initial ED, that is $\theta = 0^\circ$ (x-direction), 90° (y-direction), and 135° on the Debye-Scherrer rings for the 0° sample, the 90° sample, and the 45° sample, respectively, as shown in Figure 5.11(a-c), so that there should be no obvious (0002) peaks appear in the diffraction patterns before deformation based on the initial texture. Although there may have other twinning systems that occur during the cyclic loading, i.e., contraction twinning system that usually occurs at very large strain amplitude with small fraction [149], only extension twinning is considered in this work.

In order to provide a direct picture about the twinning process that is related to the intensity change in the twin-related (0002) and (10 $\bar{1}$ 0) peaks, the diffraction patterns of the reference point (red point in Figure 5.3) and crack tip point at $P_{\max}$ for these three samples are shown in Figure 5.11. No obvious (0002) peaks in the diffraction patterns for the reference points are observed as expected. While the (0002) peaks appear with the consumption of (10 $\bar{1}$ 0) peak in the diffraction patterns for the crack tips at $P_{\max}$, which

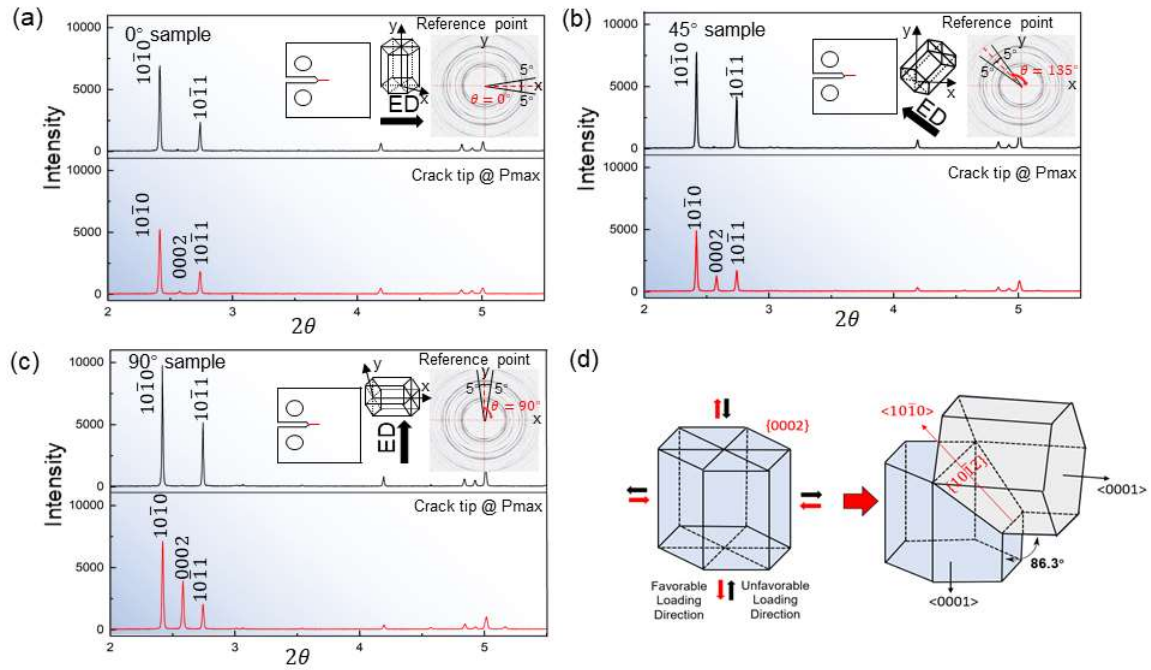


Figure 5.11. Diffraction patterns at the reference point and the crack tip at K_{max} for (a) the 0° sample, (b) the 45° sample, and (c) the 90° sample. The intensity is obtained by azimuthally averaging the ring pattern over $\pm 5^\circ$ with respect to the θ value of 0° , 90° , and 135° on the Debye-Scherrer rings for the 0° sample, the 90° sample, and the 45° sample, respectively.

suggests the activation of extension twinning. Taking the initial strong rolling texture component of this alloy into consideration, one may expect to observe extension twinning in the 0° sample (loading along with ND) and 45° sample (loading along 45° between ED and ND) to accommodate the strain along the loading direction (LD), while twinning cannot be activated in the 90° sample (loading along with the ED) due to the far-field tension. This is, however, not the experimental finding. The intensity increment of {0002} peak is the largest in the 90° sample, the least in the 0° sample.

Figures 5.12(a-f) depict the contours of $I_{xx}\{0002\}$ for the 0° sample, and $I_{yy}\{0002\}$ for the 90° sample, and $I_{135^\circ}\{0002\}$ for the 45° sample at $P_{\max}$ and $P_{\min}$, respectively. It is interesting to note that $I\{0002\}$ with relatively increased values compared with the weak surroundings only appears in a $\sim 200\text{-}300\ \mu\text{m}$ wide band along the crack-growth path at the plastic zone region and behind the crack tip at both $K_{\max}$ and $K_{\min}$ in these three samples. This suggests the activation of extension twins in the vicinity of fatigue crack surfaces and the formation of a “twin band” in these three samples. Meanwhile, the $I\{0002\}$ on the “twin band”, as expected, rises with the increase of applied load from $K_{\min}$ to $K_{\max}$ (or the decreases with the unloading from $K_{\max}$ to $K_{\min}$), suggesting the occurrence of the alternate twinning-detwinning process during the cyclic loading-unloading sequence.

Compared to surrounding materials, the increased magnitude of $I\{0002\}$ on the “twin band” from the large to small order under a far-field tensile stress state is 90° sample > 5° sample > 0° sample. This fact suggests that more grains are involved in twinning in the 90° sample and 45° sample, contrary to one’s expectation, compared to the 0° sample,

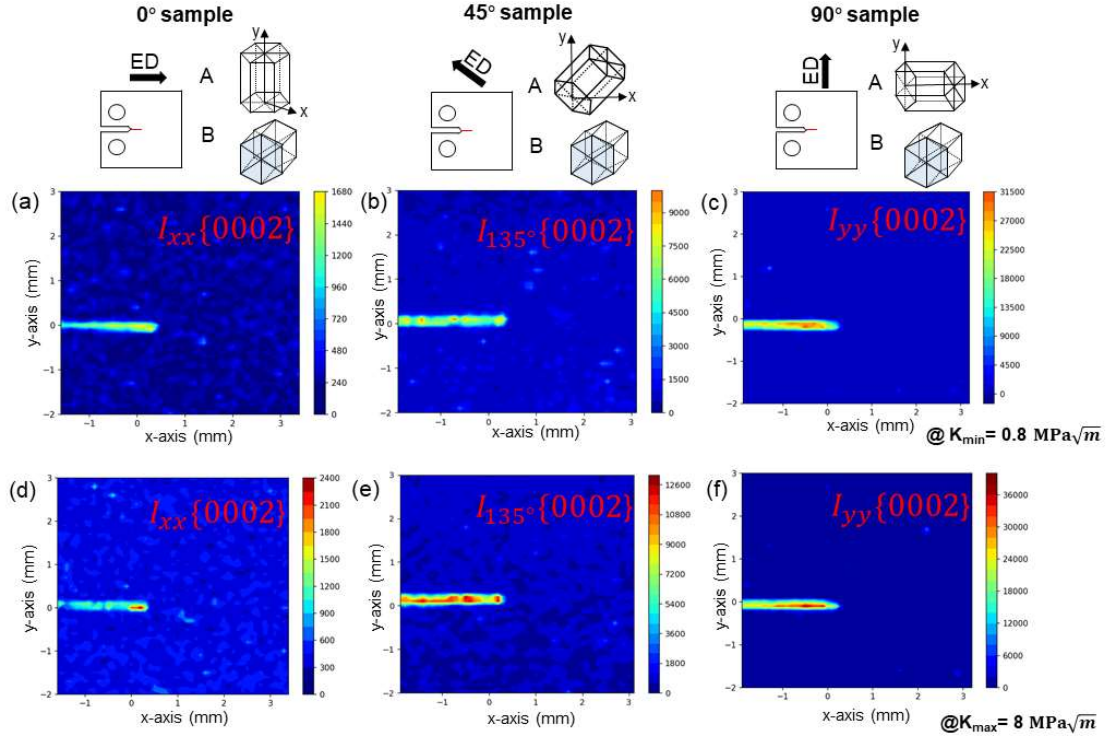


Figure 5.12. (0002) intensity evolution with an arbitrary unit at $K_{\min}$ and $K_{\max}$ for the 0° sample (a) and (d), the 45° sample (b) and (e), and the 90° sample (c) and (f). The (0, 0) coordinate indicates the approximate position of the crack tip. The intensity is obtained by azimuthally averaging the ring pattern over $\pm 5^\circ$ with respect to the θ value of 0° , 90° , and 135° on the Debye-Scherrer rings for the 0° sample, the 90° sample, and the 45° sample, respectively.

which is also confirmed by the optical microscope OM image of the deformed samples after S-XRD mapping (Figure 5.A1 in Appendix). The $I\{0002\}$ behind the crack tip along the “twin band” in the 0° sample and 45° sample can be a result of the accumulation of residual twins formed in the plastic zone region. While the greater $I\{0002\}$ behind the crack tip in the 90° sample along the “twin band” cannot solely result from the accumulation of the residual twins at the crack tip. The onset of extension twinning under uniaxial load is generally characterized by critical shear stress (CRSS) [17, 40], whereas the twinning activities under a complex stress state are far more complicated. Based on the initial texture in Figure 5.1(c), one may argue that the unexpected intensity variation in these samples can be related to the weak fiber texture, in addition to the strong rolling texture. Two reasons are proposed here to deny this argument. In order to make our discussion more concisely, two extreme cases for grain orientations are considered here: c -axis parallel to ND (denoted as A-grain, representing the strong rolling texture) and c -axis along with TD (denoted as B-grain, representing one component of the weak fiber texture) in these three samples, as schematically shown in Figure 5.12. First, the fraction of the B-grain component is much smaller than of the A-grain component according to the weak extruded texture and strong rolling texture, which means the intensity difference caused by the B-grain component in these samples is negligible. Secondly, as all three samples have the same weak B-grain component, it is reasonable to speculate that the difference in the fatigue performance and the microstructural evolution in these three samples mainly result from the A-grains. And the twinning/detwinning behavior in A-grains is the primary

contribution promoting the difference of the “twin band”. To illustrate the twinning process in the samples with different orientations, a simplified crystal plasticity finite element simulation will be applied to investigate the twin distributions in Section 5.3.5.2.

5.3.5 Finite element simulation

5.3.5.1 Cohesive interface model for fatigue crack growth simulations

To capture the anisotropic mechanical response in the ZK60 alloy, the Hill anisotropic plasticity is adopted for the clean process zone. The quadratic Hill yield criterion is a simple extension of the Mises function to describe the anisotropic plastic deformation with three orthogonal planes of symmetry, given by a quadratic form [69]:

$$f(\sigma) = \sqrt{F(\sigma_{22} - \sigma_{33})^2 + G(\sigma_{33} - \sigma_{11})^2 + H(\sigma_{11} - \sigma_{22})^2 + 2L\sigma_{23}^2 + 2M\sigma_{31}^2 + 2N\sigma_{12}^2} \quad (5-13)$$

where σ_{ij} and τ_{ij} represent the normal and shear stress components, respectively; F, G, H, L, M, and N are constants that represent the state of plastic anisotropy.

In ABAQUS, plastic anisotropy is typically represented employing the anisotropic yield stress ratios, R_{ij} ($= \sigma_{ij}/\sigma^0$) [142], which represents the yield stress ratios in the normal (R_{11} , R_{22} , and R_{33}) and shear (R_{12} , R_{13} , and R_{23}) directions. The values of these constants are defined by the following relations:

$$F = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{22}^2} + \frac{1}{\bar{\sigma}_{33}^2} - \frac{1}{\bar{\sigma}_{11}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{22}^2} + \frac{1}{R_{33}^2} - \frac{1}{R_{11}^2} \right) \quad (5-14)$$

$$G = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{33}^2} + \frac{1}{\bar{\sigma}_{11}^2} - \frac{1}{\bar{\sigma}_{22}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{33}^2} + \frac{1}{R_{11}^2} - \frac{1}{R_{22}^2} \right) \quad (5-15)$$

$$H = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{11}^2} + \frac{1}{\bar{\sigma}_{22}^2} - \frac{1}{\bar{\sigma}_{33}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{11}^2} + \frac{1}{R_{22}^2} - \frac{1}{R_{33}^2} \right) \quad (5-16)$$

$$L = \frac{3}{2} \left(\frac{\tau^0}{\bar{\sigma}_{23}^2} \right)^2 = \frac{3}{2R_{23}^2} \quad (5-17)$$

$$M = \frac{3}{2} \left(\frac{\tau^0}{\bar{\sigma}_{13}^2} \right)^2 = \frac{3}{2R_{13}^2} \quad (5-18)$$

$$N = \frac{3}{2} \left(\frac{\tau^0}{\bar{\sigma}_{12}^2} \right)^2 = \frac{3}{2R_{12}^2} \quad (5-19)$$

where σ^0 is the user-defined reference yield stress and τ^0 is defined as $\sigma^0/\sqrt{3}$. The material properties of the ZK60 alloy in the cohesive interface model are assumed to be elastic and plastic with young's modulus $E = 50$ GPa, Poisson's ratio $\nu = 0.3$, yield strength $\sigma_y = 260$ MPa (LD parallel to ED), and strain hardening exponent, $N = 0.15$. And the plastic anisotropy characterized by Hill's potential function in ABAQUS is given in terms of anisotropic yield stress ratios R_{ij} , which can be evaluated by the uniaxial tensile yield strength loading in 0° (ED), 45° (45° between ED and ND), 90° (ND), and transverse direction (TD), according to following equations. The mathematic derivation process is elaborated in Ref [150]. Therefore, Eq. (5-14 – 5-19) can be rewritten as:

$$F = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{90^\circ}^2} + \frac{1}{\bar{\sigma}_{TD}^2} - \frac{1}{\bar{\sigma}_{0^\circ}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{22}^2} + \frac{1}{R_{33}^2} - \frac{1}{R_{11}^2} \right) \quad (5-14-1)$$

$$G = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{TD}^2} + \frac{1}{\bar{\sigma}_{0^\circ}^2} - \frac{1}{\bar{\sigma}_{45^\circ}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{33}^2} + \frac{1}{R_{11}^2} - \frac{1}{R_{22}^2} \right) \quad (5-15-1)$$

$$H = \frac{(\sigma^0)^2}{2} \left(\frac{1}{\bar{\sigma}_{0^\circ}^2} + \frac{1}{\bar{\sigma}_{90^\circ}^2} - \frac{1}{\bar{\sigma}_{TD}^2} \right) = \frac{1}{2} \left(\frac{1}{R_{11}^2} + \frac{1}{R_{22}^2} - \frac{1}{R_{33}^2} \right) \quad (5-16-1)$$

$$N = (\sigma^0)^2 \left(\frac{2}{\bar{\sigma}_{45^\circ}^2} - \frac{1}{2\bar{\sigma}_{TD}^2} \right) = \frac{3}{2R_{12}^2} \quad (5-19-1)$$

Through-thickness anisotropic parameters related to shear L and M are assumed to be identical to the anisotropic parameters of N . By assuming the $R_{11} = 1$, the anisotropic parameters R_{ij} in the yield function are listed in Table 5.2.

Table 5.2. Anisotropic parameters in the Hill yield function in the Abaqus modeling for ZK60 alloy.

R_{11}	R_{22}	R_{33}	R_{12}	R_{13}	R_{23}
1.0	0.558	0.588	0.596	0.596	0.596

The interface parameters are $\sigma_{\max} = 820$ MPa, $\delta_n = 1$ μm , $\delta_a/\delta_n = 0.4$, and $\delta_f/\delta_n = 10$, since a large δ_f/δ_n will prevent a rapid softening of K^+ to slow down the crack growth rate, and a small δ_a/δ_n will ensure that K^+ approaches K^- rapidly during unloading. As illustrated by Tvergaard and Hutchison [1], the choice of $\sigma_{\max}/\sigma_y$ will significantly affect the propagation of crack in the sense that the crack under monotonic loading will not propagate with a large $\sigma_{\max}$ and will grow too fast with a small $\sigma_{\max}$ without forming a fully developed plastic zone, and $\sigma_{\max}/\sigma_y \approx 3$ is usually suggested. Zheng et al. [4] found the choice of cohesive parameters shall not affect the plastic zone size and strain history under a cyclic loading but affect the crack growth rate when the crack bridging zone size is much smaller than the plastic zone. For simplicity, a plastic anisotropic hardening model without any cyclic hardening/softening is considered in this work. From the perspective of illustrating the effect of the plastic anisotropy on the lattice strain fields, local coordinate systems have been defined by rotating 0° , 45° , and 90° , respectively, about the 3-axis for defining the material properties, which are used to achieve the three same cases in our S-XRD mapping.

The contours of elastic strain, EE_{11} , EE_{22} , for the 90° sample from our simulation under plane-strain and plane-stress conditions are shown in Figure 5.13. By comparing the amplitude and distribution of the strain, we can see the plane-stress results agree well with the experimental data, while the plane-strain results underestimated the crack tip strains. This indicates that the samples were apparently sufficiently thin to achieve a near-true plane-stress condition at $K_{\max}$.

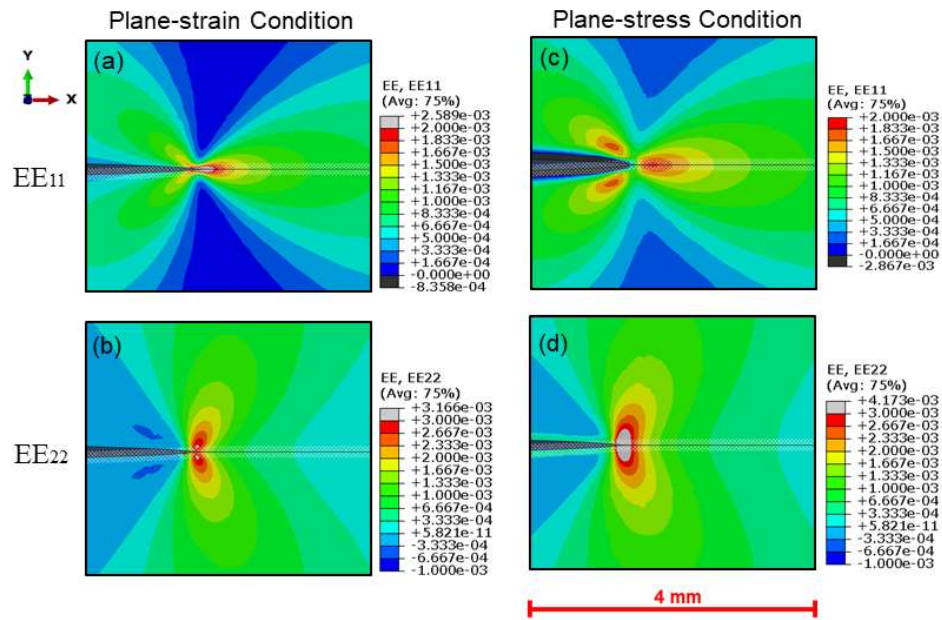


Figure 5.13. Contours of elastic strains along the crack growth direction, EE_{11} , and the crack opening direction, EE_{22} for 90° sample (a – b) under the plane-strain condition from FE simulation; (c – d) under the plane-stress condition from FE simulation. The deformation field is magnified by a factor of 10.

Contours of the elastic strain components (EE_{11} , EE_{22} , and EE_{12}) and stress components (S_{11} , and S_{22}) near the crack tip at the $K_{\max}$ with a crack length of 9 mm under plane-stress condition for the above three cases are plotted in Figure 5.14 and Figure 5.15, respectively. The crack tip location is determined when the crack opening displacement reaches δ_n . Obviously, the shapes of these contours are consistent with our experimental results. A squid-shaped tensile elastic strain field, EE_{11} , is developed along the crack growth path; a dumbbell-shaped tensile elastic strain field, EE_{22} , is observed ahead of the fatigue crack tip with a compressive strain field, EE_{22} , behind the crack tip; and a butterfly-shaped elastic strain field, EE_{12} , is noticed around the crack tip. Similar to our experimental results, the 90° sample exhibits the largest S_{22}/EE_{22} fields, and the asymmetric character of the lattice strain/stress fields in the 45° sample is also captured by the cohesive interface model by applying Hill's anisotropic plasticity model. The peak stress/strain amplitude matches our S-XRD experimental results very well. However, the tensile strain field, EE_{11} , in the plastic wake region is not captured by the simulation. This discrepancy can be related to the limit of our model, where the deformation twinning and texture evolution induced by the twinning process are not considered.

It should be noted again that the employed cohesive interface model is of a phenomenological nature with the sole purpose being to generate a steady fatigue crack. To achieve this objective, we need to ensure that the crack bridging zone size and the crack increment per cycle are significantly smaller than the plastic zone size. To this end, we can

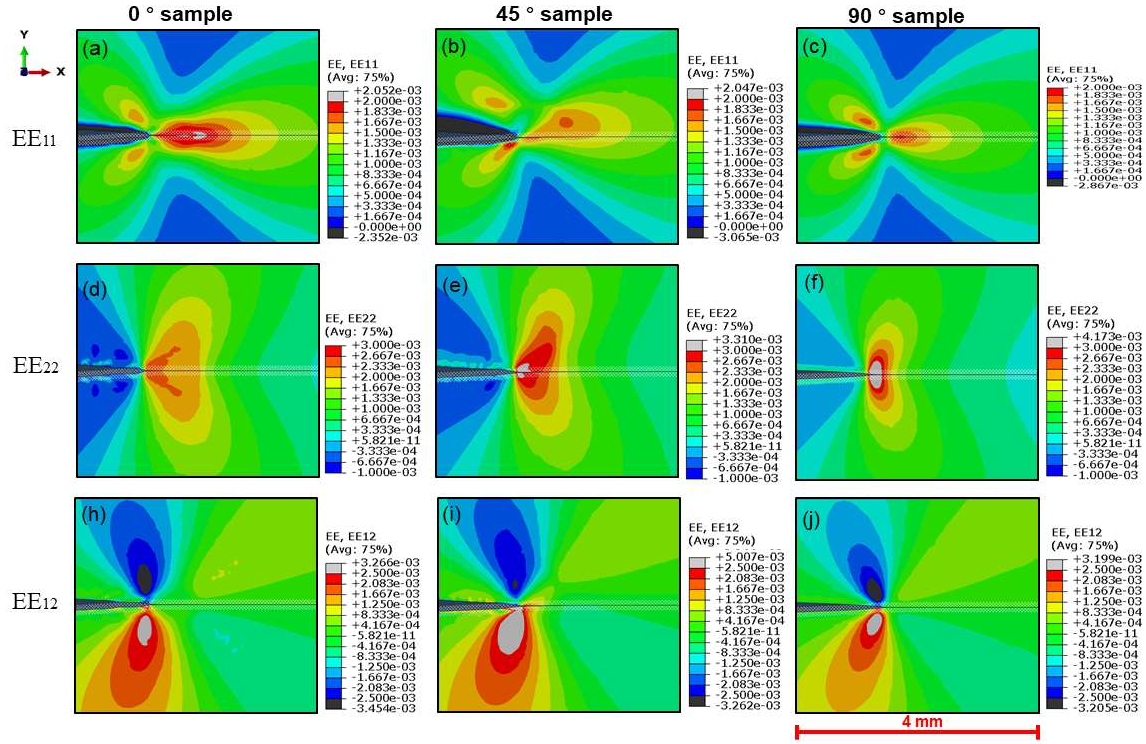


Figure 5.14. (a - c) Contours of elastic strains along the crack growth direction, EE_{11} ; (d - f) contours of elastic strains along the crack opening direction, EE_{22} ; and (h - j) contours of shear strain, EE_{12} , at K_{max} under plane-stress condition for the 0° sample, 45° sample, and 90° sample from FE simulation. The deformation field is magnified by a factor of 10.

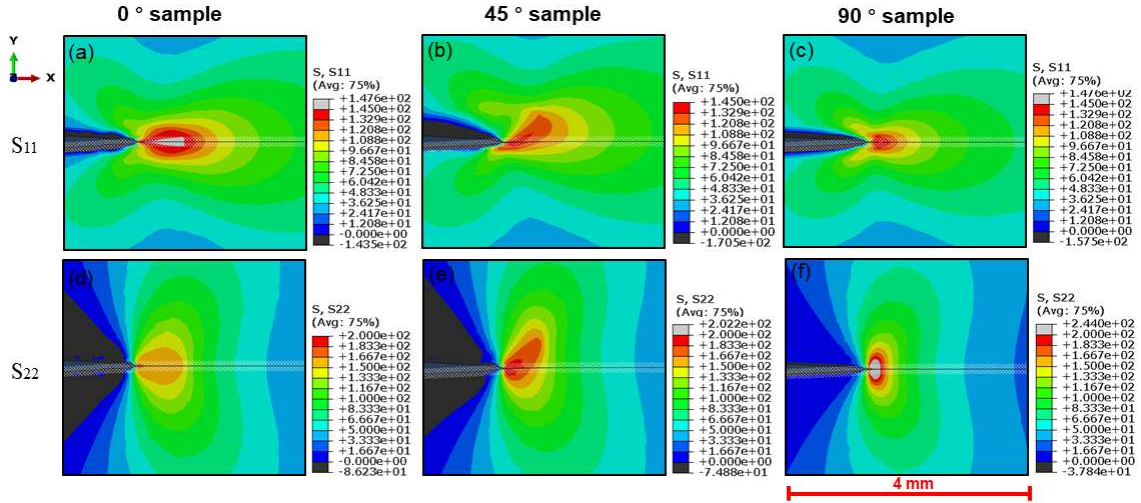


Figure 5.15. (a - c) Contours of stress along the crack growth direction, S_{11} ; and (d - f) contours of stress along the crack opening direction, S_{22} , at $K_{\max}$ under plane-stress condition for the 0° sample, 45° sample, and 90° sample from FE simulation. The deformation field is magnified by a factor of 10.

easily adjust the parameters used in the plasticity model to attain a good agreement between the simulated and measured “clean” plastic zones in Figure 5.7 and Figure 5.15 even for this heavily textured and plastically anisotropic material, but the “messy” process zone at the crack tip (within half of R_p) shows some noticeable discrepancies. This difference can actually be used as inputs for an inverse problem to extract a mechanism-based cohesive zone law, but this will be left for future work.

5.3.5.2 Crystal plasticity finite element simulations of twin distributions

In ductile materials, the propagation of fatigue crack during the cyclic loading involves the progressively plastic blunting of the crack tip on loading and resharpening on unloading [45]. The crack tip in this simulation, therefore, can be simplified as a notch instead of a sharp crack based on the plastic zone size. A 2-D finite element analysis on a standard CT sample with a long crack (crack length = 9 mm, crack root radius = 100 μm) is considered under a monotonic loading to P_{max} . Fatigue loading conditions are not computed here as detwinning is not included in the above crystal plasticity finite element constitutive model. As will be shown shortly, this limitation does not prevent us from understanding the texture dependence of twin distributions.

The finite element mesh for simulation is shown in Figure 5.16, with the same geometry of the CT sample in Figure 5.3(a). As previously mentioned, the difference of the “twin band” is primarily dominated by the twinning/detwinning behavior in A-grains in these samples, the grain orientations in the simulation are consistent with the A-grains in these samples. Based on the initial strong $\{0001\}\langle 10\bar{1}0 \rangle$ rolling texture, for simplicity,

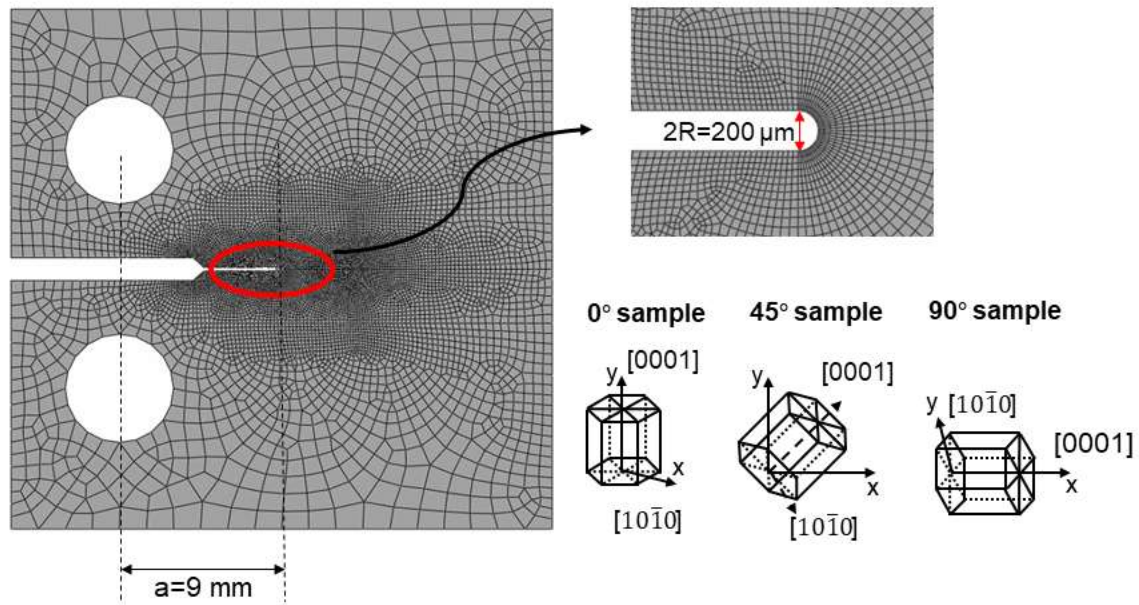


Figure 5.16. The crystal plasticity finite element setup for a notched CT specimen to mimic the blunted fatigue crack tip. The geometric sizes are taken from Figure 5.3.

the CT specimen is assumed to be a single crystal, where the crack growth direction is along $[10\bar{1}0]$ -direction, the crack opening direction is along the $[0001]$ -direction in the 0° sample; compared to the 0° sample, the crystal lattice rotate 45° clockwise on the $[10\bar{1}0]$ - $[0001]$ plane in the 45° sample; the crack growth direction is along the $[0001]$ -direction, the crack opening direction is along $[10\bar{1}0]$ -direction in the 90° sample. For all three samples, the normal to the flat surface of the notch is aligned along the $[\bar{1}2\bar{1}0]$ -direction. In our calculations, the elastic constants of the ZK60 Mg alloy is given as $C_{11} = 59.3$ GPa; $C_{12} = 25.7$ GPa; $C_{13} = 21.4$ GPa; $C_{33} = 61.5$ GPa; $C_{55} = 16.4$ GPa [151]. The hardening parameters used in the HCP crystal plasticity model are estimated from [18], as listed in Table 5.3, where only basal slip, prismatic slip, and extension twinning are considered. And the parameters for the twinned fraction are given by $s_{th} = 170$ MPa; $s = 220$ MPa; $\dot{\epsilon}_0 = 0.001s^{-1}$; $m = 0.07$.

The contours of the extension twinning volume fraction of the three samples are shown in Figure 5.17 at a load level of $P = 650$ N. The extension twinning volume fraction, f_T , as summed from all possible variants, are distinctively different in these three cases. The extension twinning predominantly occurs in a small region ahead of the notch tip for the 0° sample, which has a symmetric shape on the crack growth plane. The calculation results by our explicit finite element method are qualitatively the same as the 3D finite element simulations by Kaushik et al. [125] that the extension twin distributions are near the notch root. However, unlike the 0° sample, the contour of extension twinning volume fraction is asymmetric on the crack growth plane in the 45° sample. The extension twinning

Table 5.3. The initial and final critical resolve shear stresses used in the HCP crystal plasticity finite element model, as estimated from Wu et al. [18]

	Basal < <i>a</i> > slip	Prismatic < <i>a</i> > slip	Pyramidal < <i>a</i> > slip	Pyramidal < <i>a+c</i> > slip	Extension Twinning	Compression Twinning
τ_0	8	113 MPa	n/a	n/a	58 MPa	n/a
τ_s	8	113 MPa	n/a	n/a	58 MPa	n/a

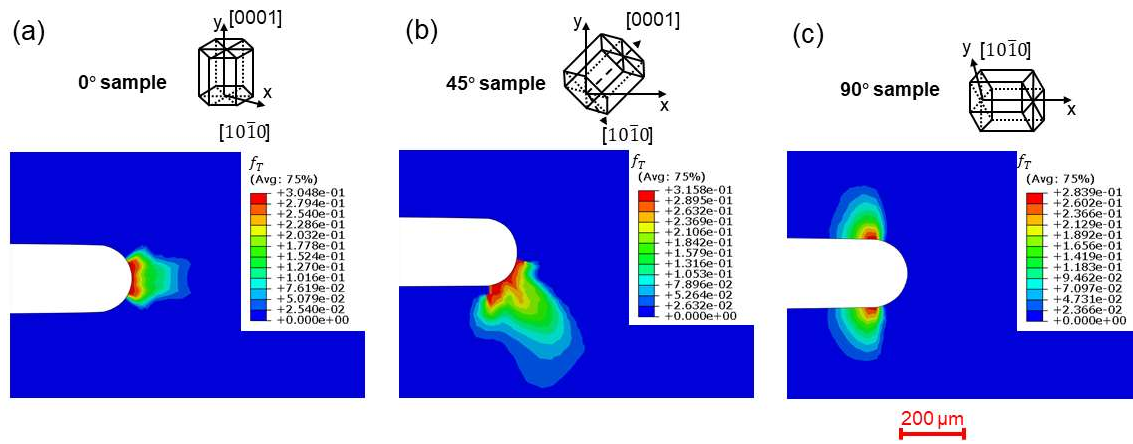


Figure 5.17. Contours of the extension twin fraction near the crack root for (a) 0° sample, (b) 45° sample, and (c) 90° sample from FE simulation.

activity occurs prevailing in the lower left quarter of the notch, which is associated with the strong plastic anisotropic response in this ZK60 Mg alloy. Whereas in the 90° sample, in contrast to the 0° and 45° samples, the activation of extension twins spread in the vicinity of fatigue crack surfaces behind the notch tip instead of the notch root.

Clearly, the distribution of extension twinning is strongly dependent on crystallographic orientations, which shows very good agreement with the S-XRD experimental results in Figure 5.12. We can also notice that the region near the notch root experiences high levels of twin volume f_T in all three samples. As noted above, extension twinning occurs around the crack root in all three samples with far-field tensile stress. It is reasonable to anticipate the formation of a narrow “twin band” along the crack growth path during the progressive extension of the fatigue crack in Figure 5.12, due to the accumulation of residual extension twins formed around the crack root.

5.4 Discussion

In our direct strain measurement by in-situ S-XRD measurement, the stress state at the crack tip is not uniaxial, but a non-equal biaxial tension, as displayed in Figure 5.9. This biaxial tension at the crack tip facilitates the occurrence of extension twinning in these three samples. This is consistent with previous reports [152, 153]. Cheng et al. [152] reported that biaxial tension can increase the difference in the Schmid factor for the six twin variants and make Schmid law more efficient, and twin variant selection and volume fraction are highly dependent on the directional strength ratio. Xia et al. [153] suggested that the activity of extension twins was increased under the biaxial compressive stress state,

compared with the uniaxial stress state, since the equivalent yield strength under a biaxial compressive stress state was lower than that under a uniaxial stress state.

The relatively higher $I\{0002\}$ in the 90° sample behind the crack tip can be attributed to the non-equal biaxial compression-tension stress/strain state behind the crack tip, where tensile stress σ_{11} along the c -axis and compressive stress σ_{22} perpendicular to the c -axis of the A-grain favor the activation of extension twinning in these A-grains. Our FEM result for the twin distribution also predicts that the extension twinning predominantly occurs behind the crack tip, as shown in Figure 5.17. The synergy analysis of the stress and intensity evolution along the “twin band” indicates the activation of extension twinning depends on the collective of local stress state and texture [122, 154].

In terms of the relatively low threshold value and high crack growth rate under the same ΔK value in the 90° sample and 45° sample, three affected factors are proposed here. Firstly, as shown in Figure 5.7 and Figure 5.9, with the same K value, the 90° sample has the largest stress/strain fields ahead of the crack tip at $K_{\max}$, followed by the 45° sample. The difference between the magnitude of stress/strain at the crack tip due to the sample orientations shall act as an important factor that affects the fatigue crack growth rate, as high stress/strain level at the crack tip facilitates the damage initiation and crack propagation, resulting in the high fatigue crack growth rate. Secondly, the reverse plastic zone with greater zone size and magnitude is developed in the 0° sample at $K_{\min}$, as displayed in Figure 5.8, compared with the other two samples. Analogous to the overload effect on the fatigue resistance [155, 156], the relatively large compressive stress fields can

significantly decrease the effective crack-opening load and cause the crack-growth retardation in the 0° sample. Thirdly, the accumulated deformation twins would facilitate the initiation and the early growth of fatigue crack [157]. The fatigue deformation of Mg alloys is strongly affected by the interaction between deformation twin and dislocation slip [158]. The high deformation incompatibility between matrix and twin will impede dislocation movement, and result in high local stress [159]. Cracks prefer to initiate and extend along the twin boundaries [32, 159, 160]. More twins that are observed in the 90° sample and 45° sample during the fatigue crack growth result in more cyclic deformation irreversibility and cumulative fatigue damage, which explains the relatively low threshold value for crack propagation and worse fatigue performance [123]. In this regard, deformation twinning appears to be detrimental to fatigue property. Our experimental results here can be useful for further simulation, such as the finite element method that considers the twin/detwin process to determine the effect of sample orientation on the twin evolution, stress intensity factors, crack-propagation path, and even the fatigue lifetime.

5.5 Summary

The deformation fields near a crack tip in a ZK60 Mg alloy with different orientations are measured using in-situ S-XRD, coupled with the cohesive interface and crystal-plasticity finite element simulations. Three CT samples with crystallographic orientations referred to as 0°, 45°, and 90° samples are chosen for this study. Key experimental observations and theoretical interpretations are given below:

- (1) The lattice strain fields near a fatigue crack tip are successfully obtained by the in-situ S-XRD technique. The shapes of lattice strain fields and stress fields exhibit strong dependences on the plastic anisotropy in the ZK60 alloy.
- (2) An irreversible, hysteretic cohesive interface model is used in finite element simulations to develop a steady fatigue crack for the anisotropic solids. Using the Hill anisotropic plasticity, our numerical simulations illustrate the development of plastic wake behind the crack and elastic strain field around the crack tip, together with the strong dependence on the plastic anisotropy.
- (3) Extension twinning is observed in the vicinity of the fatigue crack surface and form a “twin band” along the crack growth path in these three samples. The activation of extension twinning is both texture and local stress-state dependent. To our surprise, more grains are involved in the twinning process in the 90° sample with the basal plane aligning the far-field tensile loading direction, and only a few twins are observed in the 0° sample with the *c*-axis along the loading direction.
- (4) A CPFEM model for Mg alloy is employed to simulate the distribution of extension twinning near a crack root, which illustrates the occurrence of extension twinning primarily in a small region surrounding the notch tip. The numerical modeling results partly explain the observation of the “twin band” along the crack growth path.
- (5) Compared with the 0° sample, the collective effect of high stress/strain fields at $K_{\max}$, lower reverse compressive stress field distribution at $K_{\min}$, and more deformation twins involved in the crack extension in the 90° and 45° sample contributes to more cyclic

deformation irreversibility and cumulative fatigue damage, leading to the weak fatigue resistance.

APPENDIX

5.A1 Twin Observation near a crack tip

In order to further confirm the distribution of twins near a fatigue crack tip, optical microscopic (OM) micrographs are captured around the crack tip for these three samples. From Figure 5.A1, we can see that a large volume of twins spread in the 90° sample, a relatively small number of twins in the 45° sample, a few twins in the 0° sample, which are consistent with the intensity analysis. We can also note that deformation twins are mainly developed in the large matrix colony where the Mg matrix exhibits close crystal orientation and provides the beneficial crystallographic spaces for twin growth, and the twin growth is restricted by the grain boundary or colony boundary, as seen in Figure 5.A1.

5.A2 Crystal Plasticity Finite Element Model

In the crystal plasticity finite element method, the volume fraction of twins for the i -th twin system is a field parameter, denoted as f^i , which satisfies $f^i \geq 0$, with $\xi = \sum_i f^i \leq 1$ ($i = 1, 2, \dots, N_t$, where N_t is the total number of twin systems).

The constitutive relationship between stress and strain in a metallic single crystal is taken as a linear relation as follows:

$$\mathbf{T}^e = \mathbf{C}[\mathbf{E}^e] \quad \text{A(5-1)}$$

where $\mathbf{C}$ is a fourth-order anisotropic elasticity tensor, $\mathbf{E}^e$ and $\mathbf{T}^e$ are the Green elastic strain measure and the symmetric second Piola–Kirchhoff stress tensor related to the relaxed configuration, given in terms of the elastic deformation gradient $\mathbf{F}^e$,

$$\mathbf{E}^e = (1/2)\{\mathbf{F}^{eT}\mathbf{F}^e - \mathbf{1}\} \quad \text{A(5-2)}$$

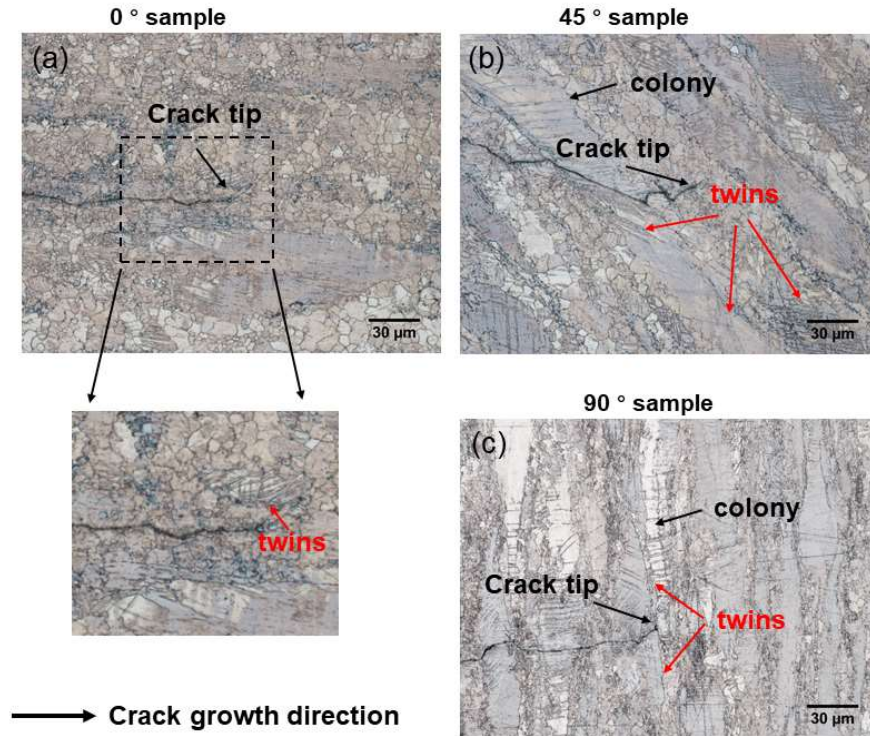


Figure 5.A1. Optical micrographs around the crack tip for (a) the 0° sample, (b) 45° sample, and (c) 90° sample after S-XRD mapping. Red arrows point out the twins and colony, and black arrows point out the crack.

$$\mathbf{T}^e = (\det \mathbf{F}^e) \mathbf{F}^{e-1} \mathbf{T} \mathbf{F}^{e-T} \quad \text{A(5-3)}$$

The Schmid tensors for the i -th slip/twin system, $\mathbf{S}_0^i$, are described in terms of a unit normal $\mathbf{n}_0^i$ to the slip/twin plane and a unit vector $\mathbf{m}_0^i$ denoting the slip/twin direction,

$$\mathbf{S}_0^i = \mathbf{m}_0^i \otimes \mathbf{n}_0^i \quad \text{A(5-4)}$$

The conditions for slip and twinning are restricted by the resolved shear stress, $\mathbf{T}^i$, and slip/twin system deformation resistances, s^i , on the i -th slip/twin system:

$$\mathbf{T}^i = (\mathbf{C}^e \mathbf{T}^e) \cdot \mathbf{S}_0^i \quad \text{A(5-5)}$$

$$\phi^i = |\tau^i| - s^i \leq 0 \quad \text{A(5-6)}$$

The flow rule described by the evolution of the plastic deformation gradient due to crystallographic slip and twinning is taken as:

$$\dot{\mathbf{F}}^p = \mathbf{L}^p \mathbf{F}^p \quad \text{A(5-7)}$$

In this equation, $\mathbf{L}^p$ is the sum of the shearing rates on all the slip and twin systems, given by:

$$\mathbf{L}^p = \sum_i \dot{\gamma}^i \text{sign}(\tau^i) \mathbf{S}_0^i \quad \text{A(5-8)}$$

And the shearing rates are restricted as follows:

$$\dot{\gamma}^i \geq 0, \text{ and } \dot{\gamma}^i \phi^i = 0, \text{ if } \phi^i = 0. \quad \text{A(5-9)}$$

For the twinning plasticity, a composite sum is assumed in which the twinned fraction deforms by a Mises plasticity, and the untwinned fraction assumes Eq. A(5-8). Thus, slip bands and twin lamella of the crystal are not explicitly considered in this type of model. The flow rule is now written as

$$\mathbf{L}^p = (1 - \xi) \sum_i \dot{\gamma}^i \text{sign}(\tau^i) \mathbf{S}_0^i + \xi \mathbf{M} \quad \text{A(5-10)}$$

$$\mathbf{M} = \dot{\varepsilon}_0 \left\{ \frac{\bar{\sigma} - s_{th}}{s} \right\}^{\frac{1}{m}} \left(\frac{3\mathbf{T}^{e'}}{2\bar{\sigma}} \right) \quad \text{A(5-11)}$$

$$\bar{\sigma} = \sqrt{\frac{3}{2} \mathbf{T}^{e'} \cdot \mathbf{T}^{e'}} \quad \text{A(5-12)}$$

where s is a strength measure, s_{th} is a threshold value, and $\mathbf{T}^{e'}$ is the deviatoric part of $\mathbf{T}^e$.

Note that the “isotropic term” is activated only if the equivalent tensile stress $\bar{\sigma}$ in a grain that exceeds the threshold, s_{th} , which is used to bound the stress levels.

CONCLUSION

Structural alloys are at the very heart of modern life and are materials that underpin most of our energy and transportation systems. Our limited knowledge of materials failure mechanisms places the ultimate restrictions on the technological viability of Mg alloys. The present research on micromechanical modeling and in situ diffraction studies enhances the fundamental knowledge of Mg alloys by connecting the macroscopic behavior with microscopic mechanisms. Specifically, an unprecedented focus is placed on elucidating (1) the complex multiaxial stress history in the “clean” plastic zone, and (2) a phenomenological cohesive zone law in the “messy” process zone to simulate the fatigue crack growth even for the heavily textured and plastically anisotropic material. The modeling/experimental synergy allows us to achieve goals that would be difficult to reach separately.

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