Characteristic cyclic plastic deformation in ZK60 magnesium alloy

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Abstract: A detailed analysis of the plastic deformation characteristics was performed for an extruded ZK60 magnesium alloy under uniaxial cyclic loading along the extrusion direction. The experiments **used for the analysis** were performed under single-step strain-controlled loading, two-step strain-controlled loading, and stress-controlled loading. An elastic limit with an offset of 10^{-5} plastic strain is used for the demarcation of elastic and elastic-plastic deformation. An inflection point is used to signify a transition of the dominated deformation mechanism from twinning-detwinning to dislocation slips. The macroscopic stress-strain response of the material is intrinsically related to the microstructures of the material during cyclic loading. **The elastic limit range is closely related to the microstructure of the material at the peak stress prior to the loading reversal. If the microstructure at the peak stress displays a strong basal α -texture, yielding is dominantly associated with the activation of basal slips. The elastic limit range to activate basal slips for the ZK60 magnesium alloy under investigation is 100 MPa. If the microstructure at the peak stress contains tension twins, the elastic limit range during subsequent loading reversal reflects the activation stress of detwinning/retwinning process, which can be interpreted as the critical stress to activate the gliding of twin boundaries. The stability of twin boundaries is influenced by twin volume fraction, twin morphology, and cyclic hardening. Dependent on the twin volume fraction and loading history, the elastic limit range varies from 20 MPa to 100 MPa for the material under investigation.**

Keywords: Cyclic deformation, elastic limit, twinning/detwinning, ZK60 magnesium alloy

1. Introduction

Magnesium (Mg) alloy has drawn great attentions due to its high strength-to-weight ratio and its potential applications in transportation vehicles, electronic apparatus, and aircraft industry (Froes, 1994; Mordike and Ebert, 2001). Structural components are often subjected to cyclic loading, and it is important to understand the cyclic plastic deformation of Mg alloys. Despite the significant progress in the study of cyclic deformation and fatigue of Mg alloys, there is still a lack of a quantitative understanding of the cyclic plastic deformation characteristics and the associated micro-mechanisms.

Mg alloy has a hexagonal close packed (hcp) crystal structure and the c/a ratio is 1.624. The most common deformation modes in Mg alloys include basal $\langle a \rangle$ slip, prismatic $\langle a \rangle$ slip, pyramidal $\langle c+a \rangle$ slip, and mechanical twinning. The basal $\langle a \rangle$ slip can be easily activated but it possesses only two independent slip systems and it cannot accommodate the strain along the c -axis. Non-basal $\langle c+a \rangle$ slip and twinning are required to satisfy the Taylor criterion (Taylor, 1938) to accommodate the strain along the c -axis (Agnew and Duygulu, 2005). However, non-basal slips are difficult to be activated owing to the high critical resolved shear stress (CRSS) at room temperature (Agnew et al., 2001). This makes twinning as a critical plastic deformation mode for Mg alloys. Among all the possible twinning systems at room temperature, tension twinning has been reported to be activated most frequently due to its low critical resolved shear stress (Reed-Hill, 1960; Agnew and Duygulu, 2005; Wang et al., 2013b).

Wrought Mg alloys usually have a strong basal texture with the c -axes of most grains aligned perpendicular to the extrusion or rolling direction. Tension twins can be activated under compression along the extrusion or rolling direction but cannot be activated under tension along the same direction. The process results in pronounced tension-compression asymmetry in the deformation of wrought Mg alloys (Ball and Prangnell, 1994). If the load which leads to tension twinning is reversed, the c -axis of tension twin domain can be reoriented to align with that of the matrix domain, which is commonly referred to as “detwinning.” For extruded or rolled Mg alloys loaded in the extrusion or rolled direction, tension twins can be activated in the compression reversal, and detwinning occurs under subsequent tensile loading (Wu et al., 2008b).

In the last decade, a great number of studies were conducted to understand the cyclic deformation behavior and low-cycle fatigue of wrought Mg alloys. The most studied wrought Mg alloys include extruded and rolled AZ31 (Albinmousa et al., 2011a, b; Begum et al., 2009a; Begum et al., 2009b; Chamos et al., 2008; Chen et al., 2012; Duan et al., 2014; Geng et al., 2013; Gu et al., 2014; Hama et al., 2012; Hasegawa et al., 2007; Hazeli et al., 2015; Hong et al., 2011; Huang et al., 2014; Huppmann et al., 2010a; Huppmann et al., 2010b; Hyuk Park et al., 2010; Ishihara et al., 2013; Kang et al., 2014; Kwon et al., 2011; Lee et al., 2015; Li et al., 2016; Lin and Chen, 2008; Lin et al., 2013b; Lin et al., 2013c; Lin et al., 2015; Lv et al., 2009; Lv et al., 2011a; Lv et al., 2011b; Matsuzuki and Horibe, 2009; Muhammad et al., 2015; Ozaki et al., 2014; Park et al., 2010a; Park et al., 2010b; Wu et al., 2010a; Wu et al., 2013; Wu et al., 2012; Wu et al., 2014; Wu et al., 2010b; Xie et al., 2016; Xiong et al., 2012; Yin et al., 2008a; Yin et al., 2008b;

Zhang et al., 2010; Zhang et al., 2011b), extruded AZ61 (Bhuiyan et al., 2008; Jordon et al., 2011; Kleiner and Uggowitzer, 2004; Li et al., 2010; Sajuri et al., 2005; Yu et al., 2011b; Zeng et al., 2009; Zhang et al., 2011a), rolled AZ91 (Chen et al., 2013; Lin et al., 2011; Lin et al., 2013a), extruded ZK60 (Dong et al., 2014; Dong et al., 2015; Qiao et al., 2015; Wu et al., 2008a; Wu et al., 2008b; Xiong and Jiang, 2014; Xiong et al., 2014; Yu et al., 2012), extruded AM30 (Begum et al., 2008; Fan et al., 2009; Jordon et al., 2013; Luo et al., 2010), extruded AM50 (Chen et al., 2007), and extruded AM60 (Akbaripanah et al., 2013). Most of the cyclic deformation experiments were conducted under fully reversed strain-controlled loading. Dislocation slips dominate the cyclic plastic deformation at smaller strain amplitudes while twinning-detwinning plays a dominant role at larger strain amplitudes. It was reported that under fully reversed strain-controlled cyclic deformation of an extruded ZK60, three distinguishable mechanisms of cyclic deformation can be identified at different strain amplitudes in light of the involvement of twinning/detwinning (Xiong et al., 2014): slip dominated, partial twinning-complete detwinning, and twinning-detwinning exhaustion. For the stress-controlled cyclic deformation experiments, the ratcheting behavior of Mg alloys depends greatly upon the applied mean stress (Xiong et al., 2014). It was reported that ratcheting occurred in the extruded AZ31 Mg alloy during the stress-controlled cyclic loading, but different evolution features were observed in the uniaxial ratcheting tests with different mean stresses (Kang et al., 2014; Lin et al., 2013c). This can be attributed to the different plastic deformation mechanisms under different mean stresses and corresponding stress amplitudes. *In situ* neutron diffraction (Wu et al., 2008a; Wu et al., 2008b; Wu et al., 2010a) and optical microscopy (Yu et al., 2011a; Yu et al., 2013) confirmed the participation of twinning/detwinning activities in the cyclic deformation of Mg alloys. Moreover, transmission electron microscopy (TEM) (Guillemer et al., 2011; Morrow et al., 2014) and electron backscatter diffraction (EBSD) (Yin et al., 2008a; Yin et al., 2008b; Dong et al., 2015) were used to characterize the microstructure evolution in the Mg alloys under cyclic loading. It was suggested that cyclic hardening caused by twinning/detwinning and dislocation slips increases twin nucleation sites but inhibits twin growth and twin shrinkage (Dong et al., 2015).

In addition to the commonly observed features of cyclic deformation in wrought Mg alloys, a nonlinear deformation phenomenon is always perceived when Mg alloys are unloaded from a peak stress to zero stress in the cyclic loading-unloading or cyclic tension-compression experiments. This nonlinear feature is regularly referred to as inelastic or pseudoelastic behavior in the loading-unloading experiments (Cáceres et al., 2003; Lee and Gharghour, 2013). Investigations by experimental (Cáceres et al., 2003; Lee and Gharghour, 2013; Li and Enoki, 2007, 2008; Mann et al., 2007; Muránsky et al., 2009) and modeling (Hama et al., 2013; Hama and Takuda, 2011; Wang et al., 2013a; Wang et al., 2013b) efforts have been made to understand the inelastic behavior occurring in the cyclic loading-unloading experiments. Both the experimental results (Lee and Gharghour, 2013) and simulation results (Wang et al., 2013a) reveal that the inelastic behavior is more significant in cyclic compression-unloading than that in cyclic tension-unloading for wrought Mg alloys. This can be attributed to the different deformation modes under operation: reversible twinning-detwinning in cyclic

compression-unloading, and dislocation slips in cyclic tension-unloading. As further experimental evidence, *in situ* neutron diffraction (Muránsky et al., 2009; Wu et al., 2008a), *in situ* acoustic emission (Li and Enoki, 2007, 2008), and *in situ* optical microscopy (Cáceres et al., 2003; Mann et al., 2007) provide direct and indirect observations of detwinning events during unloading. Most investigations regarding the pseudoelastic behavior in Mg alloys are carried out in the cyclic loading-unloading experiments. The detailed characteristics of inelastic behavior associated with **reversed loading** from the peak stress during cyclic deformation have not been well analyzed.

Although extensive work has been conducted on the cyclic plasticity of Mg alloys, critical phenomena such as the Bauschinger effect, the inflection point in the stress-strain hysteresis loop, and the inelastic behavior associated with unloading from the peak stress/strain, remain uncovered. Accurate modeling of cyclic plasticity of Mg alloys requires a comprehensive understanding of these cyclic plastic deformation phenomena. The current work is a detailed analysis of the characteristic cyclic plastic deformation and the associated mechanisms of a typical wrought Mg alloy under uniaxial loading. **The experiments were performed under three types of loading conditions: single-step strain-controlled loading, two-step strain-controlled loading, and stress-controlled loading. Among the experiments used for the detailed analysis of the stress-strain relationships, the single-step strain-controlled cyclic loading experiments were taken from a previous publication (Dong et al., 2014). A major objective of the current work is to understand the cyclic plastic deformation characteristics of a Mg alloy with the help of twinning-detwinning microstructures.** The results will provide a guideline for the development of cyclic plasticity constitutive models for Mg alloys.

2. Material and experiment

2.1. Material and specimen

An as-extruded ZK60 Mg alloy with a composition of Mg-6.0%Zn-0.5%Zr (wt%) was studied. The material used in the current work is identical to that investigated in a previous publication (Dong et al., 2014). The microstructure consists of a mixture of small and large elongated grains. On the plane perpendicular to extrusion direction (ED), small recrystallized equiaxed grains have an average size of 10 μm and large unrecrystallized grains can have a size over 50 μm . No initial twins were identified in the as-extruded grain structure. The as-extruded ZK60 exhibits a typical basal texture with the *c*-axes of most of grains perpendicular to the extrusion direction.

The dog-bone shaped testing specimen has a gage length of 15 mm and a diameter of 10 mm within the gage section. The loading axis of the specimen is aligned with the extrusion direction. Before mechanical testing, the specimen surface within the gage section was polished using silicon carbide papers with grit numbers ranging from 250 to 1500. Quasi-static mechanical properties and original microstructures were reported in an earlier publication (Dong et al., 2014).

2.2 Experiments and microscopic observations

All the monotonic and cyclic loading experiments were conducted in ambient air using a servo-hydraulic tension-torsion load frame which has a capacity of ± 222 kN in the axial load. Three loading conditions were adopted for the cyclic tension-compression experiment: (1) single-step fully reversed strain-controlled loading, (2) two-step fully reversed strain-controlled loading and, (3) stress-controlled loading. For the strain-controlled experiments, an extensometer with a gage length of 12.7 mm and a range of $\pm 10\%$ was used to measure the axial strain. The strain amplitude ranges from 4% to 0.32%, and the loading frequency ranges from 0.06 Hz to 5 Hz dependent on the strain amplitude. For the monotonic and stress-controlled cyclic loading experiments, a clip-on extensometer with a gage length of 12.7 mm and a range of $\pm 40\%$ was used for the strain measurement. Unless otherwise specified, the true stress and the true strain are reported and discussed through the current work. For the strain-controlled experiments, the engineering strain was controlled. In the stress-controlled experiments, the load instead of the true stress was controlled. The true stress and true strain are reported after correction of the cross-section area assuming a plastic incompressibility during plastic deformation.

The microstructure of the specimens after monotonic and cyclic loading was examined by EBSD. A cylindrical sample was cut from the gage section of the testing specimen for EBSD scans. The observing plane was perpendicular to the extrusion direction. Automatic EBSD scans were performed with TSL data acquisition software on an area of $200\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}$ with a step size of $0.6\text{ }\mu\text{m}$. The EBSD data was analyzed using TSL OIM analysis software.

3. Experimental results and discussion

3.1. Deformation and microstructure evolution under monotonic compression

Fig. 1 presents the stress-strain curves of as-extruded ZK60 obtained from monotonic tension and monotonic compression experiments along ED. The compressive stress and strain are reported in their absolute values. The evolution of microstructure and twin volume fraction (TVF) under monotonic compression measured by EBSD are shown in Fig. 1 as well. The Mg alloy exhibits a pronounced tension-compression asymmetry due to the strong basal texture formed by the extrusion process. The elastic limits under both monotonic tension and monotonic compression were identified to be 100 MPa (Dong et al., 2014). When the stress exceeds the elastic limit, the linear elastic deformation stage terminates and the material yields. The microyielding plastic deformation can be attributed to the activation of basal slips in favorably orientated grains.

Fig. 1. Monotonic stress-strain curves under tension and compression, and twin volume fraction (TVF) evolution under monotonic compression.

Pronounced deformation tension-compression asymmetry occurs in the material. Under monotonic compression, bulk tension twins nucleate and propagate when the stress reaches 134 MPa. It was suggested that twin always nucleates at grain boundary and rapidly propagates on the twinning plane along the twinning shear direction until it reaches the other side of the grain boundary (Wu et al., 2015). The nucleation and propagation of tension twins can lead to stress relaxation, resulting in a very low strain hardening rate in the stress-strain curve (Wu et al., 2015; Yu et al., 2011a). The EBSD scans shown in Fig. 1 indicate that when the compression strain is 0.01, the twin bands are very slim, implying that these freshly nucleated twin bands have grown insignificantly. As a result, the TVF at 0.01 compression strain is only 6%. As the compressive strain increases, the narrow twin bands are thickened by twin growth. Moreover, basal slips can be activated and multiply in the twins, which contributes to the increasing strain hardening rate in the stress-strain curve. When the compression strain reaches 0.04, the TVF is 29%, and the twin bands are obviously thickened by twin growth as compared to those observed at 0.01 compression strain. When the compression strain reaches 0.06, most of the matrix has been twinned, and the corresponding TVF is 79%. With further increase in the compression strain, the twinning process is exhausted and non-basal slips occur (Ma et al., 2011; Ma et al., 2012). The change in the compressive stress-strain curve from a concave-up shape to a concave-down shape signifies the exhaustion of twinning process. The exhaustion of twinning process can be further verified from the near identical TVFs at the compressive strains of 0.10 and 0.15. At the later stage of monotonic compression deformation, compression twins and double twins can be observed, which can be ascribed to the favorability of *c*-axis compression in the twin domain and the increased localized stresses (Yu et al., 2015).

3.2. Cyclic deformation under fully reversed strain-controlled loading

Fig. 2 presents the stress-strain hysteresis loops obtained from single-step fully reversed strain-controlled experiments of ZK60 with three representative strain amplitudes: 4%, 1%, and 0.4%. All the strain-controlled experiments started with a tensile load. For a selected amplitude, the stress-strain hysteresis loops of the first ten loading cycles and the cycles approximately corresponding to a quarter, a half, and three quarters of fatigue life are presented in order to show the evolution of the stress-strain hysteresis loops with respect to loading cycles. The stress-strain hysteresis loops corresponding to half fatigue lives are taken to be the stabilized hysteresis loops.

(a)

(b)

(c)

Fig. 2. Stress-strain hysteresis loops of three representative strain amplitudes under fully reversed strain-controlled loading; (a) $\Delta\epsilon/2=4.0\%$; (b) $\Delta\epsilon/2=1.0\%$; (c) $\Delta\epsilon/2=0.4\%$.

At a large strain amplitude of 4%, twinning is exhausted in the compressive reversal and detwinning of all the previously twinned area (detwinning completion) is observable in the tensile reversal. Non-basal slips occur in both tensile and compressive reversals during the later deformation stage. Both the tensile and compressive reversals show a sigmoidal shape. With increasing number of loading cycles, the shape of the stress-strain hysteresis loop tends to appear more and more symmetric. For the strain amplitude of 1%, twinning is not exhausted in the compressive reversal and detwinning is completed in the tensile reversal. The so-called “partial twinning-complete detwinning” process (Xiong et al., 2014) dominates the cyclic plastic deformation at the immediate strain amplitude (1%). Only the tensile reversal displays a sigmoidal shape and the stress-strain hysteresis loops exhibit typical asymmetric shapes throughout the whole cyclic loading. For the strain amplitude of 0.4%, “partial twinning-complete detwinning” process dominates the plastic deformation during the initial stage of the cyclic deformation, which results in an asymmetric stress-strain hysteresis loop shape. As the number of loading cycles increases, the twinning-detwinning activity gradually decreases and the stress-strain hysteresis loops tend to be more and more symmetric. Dislocation slips rather than twinning/detwinning dominate the plastic deformation. Accordingly, both the tensile reversal and the compressive reversal exhibit a concave-down shape.

Fig. 3. Schematic stress-plastic strain hysteresis loop where the terminology used to describe cyclic plasticity of magnesium alloy are illustrated.

To facilitate the discussion of cyclic plasticity in Mg alloy, Fig. 3 illustrates the schematic stress-plastic strain hysteresis loop where the terminology used in this paper is denoted. The stress-plastic strain hysteresis loop is obtained from the stress-strain hysteresis loop. The plastic strain is defined as follows:

$$\varepsilon_p = \varepsilon - \frac{\sigma}{E} \quad (1)$$

where,

$$E = E_0 (1 - \sigma/b) \quad (2)$$

In Eq.(1), ε_p is the plastic strain. ε and σ are the true strain and true stress, respectively. E is the elasticity modulus. Nonlinear elasticity is considered. In Eq. (2), E_0 is the elasticity modulus when the stress is zero. b is a material constant to account for the nonlinear elasticity of the material. A simple linear dependence of the elasticity modulus on the normal stress is adopted for considering the nonlinear elasticity of the material. Equation (2) is similar to that used for a steel (Sommer et al., 1991). For the as-extruded ZK60 alloy used in the present investigation, $E_0=44.3$ GPa and $b = 20,000$ MPa provide a good description of the nonlinear elasticity.

It should be noticed that nonlinear elasticity of Mg alloys has never been explored and the cyclic plastic deformation results in the current work are only marginally influenced if nonlinear elasticity is not considered. The reasons to consider non-linear elasticity of Mg alloys are twofold. First, the micro-yielding stresses under tension and under compression should be theoretically equal, but they are slightly different if a constant elasticity modulus is used. A

nonlinear elasticity consideration can nicely correlate the experiments. Second, since nonlinear elasticity is obvious in high strength steels, it is a logical plausibility that nonlinear elasticity also exists in Mg alloys in a similar fashion. The adoption of a simple linear relationship between the elasticity modulus and the normal or hydraulic stress is a natural first approximation. The selection of $b=20,000$ MPa was based on the assumption that the elastic deformation before microyielding is identical under both tension and compression.

A careful observation of the stress-plastic strain hysteresis loops will help understand the characteristics of the cyclic deformation. As shown in Fig. 3, the stress-plastic strain hysteresis loop is divided into two parts: the upper branch (red color) and the lower branch (blue color). The presentation uses the stress range versus the plastic strain range, where the stress range is presented in linear scale and the plastic strain range is plotted in linear scale or logarithmic scale. The x- and y-axes of the coordinate system represent the plastic strain range and stress range, respectively. For the upper branch, the lower tip (compressive peak) of the stress-plastic strain hysteresis loop is selected as the origin of the $\Delta\sigma$ - $\Delta\varepsilon_p$ coordinate system, and for the lower branch, the upper tip (tensile peak) of the stress-plastic strain hysteresis loop is selected as the origin of the $\Delta\sigma$ - $\Delta\varepsilon_p$ coordinate system. In this way, both the upper branch and the lower branch of the stress-plastic strain hysteresis loop can be plotted and compared in the same $\Delta\sigma$ - $\Delta\varepsilon_p$ coordinate system. The $\Delta\sigma$ - $\Delta\varepsilon_p$ curves provide characteristic information about cyclic plasticity of the material. To be specific, the “*elastic limit range*” and “*inflection strain range*” will be defined here. An offset of 10^{-5} plastic strain is chosen to demarcate the elastic and elastic-plastic deformation. In a stress-plastic strain hysteresis loop, the elastic limit range is defined as the stress difference between the tip of the loop (or the tensile/compressive peak) and the point corresponding to the 10^{-5} plastic-strain offset. The elastic limit ranges of the upper branch and the lower branch are denoted by $\Delta\sigma_{EU}$ and $\Delta\sigma_{EL}$, respectively. As shown in Fig. 3, the inflection point of the $\Delta\sigma$ - $\Delta\varepsilon_p$ curve can be identified as the point where the second derivative of the curve equals to zero. The inflection strain range is defined as the plastic strain range between the inflection point and the origin of the $\Delta\sigma$ - $\Delta\varepsilon_p$ coordinate system. The inflection strain ranges are referred to as EDT for the upper branch, and ET for the lower branch (Fig. 3).

3.3. Single-step fully reversed strain-controlled cyclic loading

Fig. 4 shows the $\Delta\sigma$ - $\Delta\varepsilon_p$ curves of the selected loading cycles at two representative strain amplitudes: 1% (Fig. 4a) and 0.4% (Fig. 4b). Both the experiments started with a tension load and the first reversal in an experiment was excluded in the $\Delta\sigma$ - $\Delta\varepsilon_p$ curves. Therefore, the lower branch of cycle 1 shown in Fig. 4 represents the stress-strain response from the maximum stress/strain after the first loading reversal. The upper branch of cycle 1 in Fig. 4 is the stress-strain curve from the first minimum stress/strain. When the strain amplitude is 1%, Fig. 4a reveals that the upper branches and the lower branches display asymmetric shapes. The upper branches exhibit a sigmoidal shape. Results shown in Fig. 4a suggest that for the first loading cycle at a strain amplitude of 1%, the elastic limit range of the upper branch ($\Delta\sigma_{EU}$) is 23 MPa, while the elastic limit range of the lower branch ($\Delta\sigma_{EL}$) is 101 MPa. With increasing

number of the loading cycles, $\Delta\sigma_{EU}$ increases slightly. When the number of the loading cycles reaches 400 (half fatigue life), $\Delta\sigma_{EU}$ is 40 MPa. For the lower branch, the elastic limit range, $\Delta\sigma_{EL}$, keeps at approximately 100 MPa from the first loading cycle to the 400th loading cycle.

(a)

(b)

Fig. 4. Stress range versus plastic strain range for the selective loading cycles: (a) $\Delta\epsilon/2=1.0\%$; (b) $\Delta\epsilon/2=0.4\%$.

When the strain amplitude is 0.4% (Fig. 4b), the upper branches and the lower branches display a significant asymmetry in shape in the initial stage of the cyclic deformation from cycle 1 to cycle 10. The shape of the stress-plastic strain hysteresis loop becomes more and more symmetric as the loading cycles increase.

Fig. 5. Variations of the elastic limit ranges ($\Delta\sigma_{EU}$ and $\Delta\sigma_{EL}$) with respect to the increasing number of loading cycles at four representative strain amplitudes.

To better understand the dependence of the elastic limit range on the loading direction and the applied strain amplitude, Fig. 5 presents the elastic limit ranges as a function of the number of loading cycles for four representative strain amplitudes: 4%, 1%, 0.4%, and 0.32%. At a strain amplitude of 4%, $\Delta\sigma_{EU}$ for the first loading cycle is 43 MPa. It is noted that during the initial few loading cycles (less than six cycles) twinning exhaustion is not reached under the compressive reversal. As the number of loading cycle increases and exceeds six, twinning exhaustion occurs. Almost all of the matrix at the minimum strain is twinned and the material exhibits a *c*-texture, where most of the *c*-axes in the material align with the extrusion direction. At the maximum strain, the twinned matrix is completely detwinned and the material shows a typical basal *a*-texture. Subsequently, a fully symmetric stress-strain hysteresis loop is obtained. $\Delta\sigma_{EU}$ is increased to approximately 100 MPa when the loading cycle reaches six and it does not vary with the increasing number of loading cycles. $\Delta\sigma_{EL}$ is approximately 100 MPa irrespective of loading cycles.

When the strain amplitude is 0.40%, the elastic limit range is 20 MPa in the upper branch for the first loading cycle and the value is 102 MPa in the lower branch. $\Delta\sigma_{EU}$ increases with the number of loading cycles and $\Delta\sigma_{EL}$ does not change with the number of loading cycles. When the number of loading cycles reaches 1,600, $\Delta\sigma_{EU}$ reaches 97 MPa which is practically identical to $\Delta\sigma_{EL}$. When the number of loading cycles is higher than 1600, $\Delta\sigma_{EU}$ keeps a constant value of approximately 100 MPa irrespective of the number of loading cycles.

When the strain amplitude is 0.32%, the minimum stress during cyclic deformation does not reach the twinning stress of the material, and macro twinning-detwinning process does not occur in the plastic deformation. Dislocation slips, mainly basal slips, dominate the plastic deformation. The stress-strain hysteresis loops exhibit nearly symmetric shape. The elastic limit ranges for both upper branches and lower branches are approximately 100 MPa, and they

do not change with the number of loading cycles.

To understand the dependence of the elastic limit on the applied strain amplitude, Fig. 6 shows the $\Delta\sigma$ - $\Delta\epsilon_p$ curves obtained from stabilized stress-plastic strain hysteresis loops at the selected strain amplitudes and Fig. 7 presents the variation of the elastic limit ranges with the applied strain amplitudes under fully reversed strain-controlled loading. Stabilized stress-strain hysteresis loops were taken at approximately half fatigue lives. The elastic limit ranges of the first loading cycle and the cycle corresponding to a half of fatigue life are plotted in Fig. 7.

Fig. 6. Stress range-plastic strain range relationship of the stabilized stress-strain hysteresis loops at selective strain amplitudes: (a) upper branches; (b) lower branches.

Fig. 7. Relationship between the elastic limit range and the applied strain amplitudes under fully reversed strain-controlled loading.

From Fig. 6b and Fig. 7, it can be seen that $\Delta\sigma_{EL}$ (the elastic limit range of the lower branches) is approximately 100 MPa for the material under investigation irrespective of the strain amplitudes and loading cycles. For the upper branch, the dependence of $\Delta\sigma_{EU}$ on the strain amplitude exhibits different features at the first loading cycle and at the half-fatigue life. For the first loading cycle, $\Delta\sigma_{EU}$ is approximately 100 MPa when the strain amplitude is 0.32%. As the strain amplitude increases to 0.4%, $\Delta\sigma_{EU}$ drops to 20 MPa. With increasing strain amplitudes, $\Delta\sigma_{EU}$ gradually increases to approximately 43 MPa at a strain amplitude of 4%. At half fatigue life, $\Delta\sigma_{EU}$ is 100 MPa when the strain amplitude is below 0.4%. When the strain amplitudes are larger than 0.4%, $\Delta\sigma_{EU}$ first decreases and then increases with the increasing strain amplitudes. The relationship between the elastic limit range of the upper branch and the applied strain amplitude displays a check-marker shape, having a maximum of 100 MPa and a minimum of 32 MPa. The minimum $\Delta\sigma_{EU}$ at half fatigue life corresponds to a strain amplitude of 0.6%.

The observed constant $\Delta\sigma_{EL}$ value of 100 MPa is independent of the strain amplitudes and loading cycles for the material under investigation. This is attributed to the activation of basal slips. When the strain amplitude is below 0.4%, such as 0.32% in Figs. 6 and 7, plastic deformation is dominated by basal slips without involving sustainable twinning-detwinning. The original extruded Mg is a typical basal α -texture and the texture does not change during cyclic deformation when the strain amplitude is below 0.4%. When the strain amplitude is larger than 0.4%, twinning-detwinning occurs. Under fully reversed strain-controlled cyclic loading, detwinning is always completed at the end of the tensile reversal and only few residual twins can be detected even at a very large strain amplitude of 4% at the maximum strain of a loading cycle (Dong et al., 2015). Therefore, for any specimens conducted in the single-step strain-controlled experiments in the present work, the texture at the maximum strain of a loading cycle retains a

strong basal a -texture. It was reported that in the cyclic tension-unloading experiments, the non-linear behavior during unloading from the tensile peak stress is attributed to basal slips (Lee and Gharghour, 2013; Wang et al., 2013a). Similarly, in the present study, when the material is unloaded and reloaded from the maximum stress/strain, yielding corresponds to the activation of basal slips due to the a -texture at the tensile peak stress. In brief, $\Delta\sigma_{EL}$ reflects the onset of basal slips of the basal-textured state of material and is a material constant.

Fig. 8. EBSD scans after cyclic deformation unloaded from the minimum strain: (a) 0.4% after first loading cycle, (b) 1% after first loading cycle, (c) 2% after first loading cycle, (d) 4% after first loading cycle, (e) 0.4% at half fatigue life, (f) 1% at half fatigue life, (g) 2% at half fatigue

life, (h) 4% at half fatigue life.

In contrast to the lower branch of the stress-strain hysteresis loop, the elastic limit of the upper branch reflects the activation of either basal slips or detwinning, dependent on the strain amplitude. When the strain amplitude is 0.32%, no bulk twinning-detwinning occurs under cyclic loading. The elastic limit range is 100 MPa, which is the activation stress range for basal slips. When the strain amplitude is equal to or larger than 0.4%, the elastic limit is strongly associated with the detwinning process. Detwinning can be activated during unloading from the peak compressive stress in the cyclic loading-unloading experiments (Cáceres et al., 2003; Li and Enoki, 2007, 2008; Mann et al., 2007; Muránsky et al., 2009; Wu et al., 2008a). The driving force of the detwinning process during unloading can be attributed to the internal tensile stresses in certain grains which result from significant twinning activity prior to unloading (Muránsky et al., 2009; Wu et al., 2008a). In order to investigate the mechanism of the evolution of elastic limit ranges in the upper branches at different strain amplitudes, EBSD scans were conducted on the testing specimens unloaded from the minimum stress after the first cycle and after half-fatigue life at different strain amplitudes, and the results are shown in Fig. 8. Although detwinning could be activated during unloading (Wu et al., 2008a; Yin et al., 2008b), experimental results (Wu et al., 2010a; Yin et al., 2008b) reveal that the twin volume fraction (TVF) at the minimum stress and that at zero stress after unloading from the minimum peak shows insignificant change. This suggests that the EBSD microstructure scanned on the unloaded samples can reasonably represent the microstructure at the minimum stress in a loading cycle.

For the first loading cycle, it can be seen from Figs. 8a, b, c, and d that TVF at the minimum stress/strain increases with increasing strain amplitudes. Fig. 8a demonstrates that tension twin can occur in the compression reversal at a small strain amplitude of 0.4%. The involvement of twinning-detwinning activity results in a dramatic drop in the elastic limit range of the upper branch ($\Delta\sigma_{EU}$) from 100 MPa at a strain amplitude of 0.32% to 20 MPa for 0.4% strain amplitude. $\Delta\sigma_{EU}$ at the first loading cycle for 0.4% strain amplitude reflects the activation of detwinning process during the reversed loading process. When the strain amplitude increases from 0.4% to 1%, the TVF at the minimum stress of the first loading cycle slightly increases from 0.3% to 6.4%. Most of the twin bands at the 1% strain amplitude are very slim, indicating these twin bands have experienced limited twin growth. $\Delta\sigma_{EU}$ increases slightly from 20 MPa to 23 MPa as the strain amplitude increases from 0.4% to 1%. When the strain amplitude is larger than 1%, the TVF increases significantly with the increasing strain amplitudes. From Figs. 8b, c and d, it can be seen that when the strain amplitude increases from 1% to 4%, the TVF at the minimum strain increases from 6.4% to 73.1%. Correspondingly, the twin bands become wider, resulting in a multiplication of twin boundaries. The slips activated in the twins can interact with twin boundaries. Slip dislocations and twin dislocations pile up and pin at the twin boundaries. These pinned dislocations at twin boundaries decrease the mobility of twin boundaries and make the formed twin bands more stable. Such a process makes the detwinning process more difficult in the subsequent loading cycles. Consequently, during reversed loading

from the compressive peak stress, the detwinning process requires a higher driving force to shrink the twinned area when a greater amount of twin bands with wider shape exist at a larger strain amplitude. Macroscopically, $\Delta\sigma_{EU}$ shows an obvious increase from 23 MPa to 43 MPa when the strain amplitude increases from 1% to 4%, as contrast to nearly constant $\Delta\sigma_{EU}$ when the strain amplitude varies from 0.4% to 1%. As a conclusion, when the strain amplitudes range from 0.4% to 4%, for the first loading cycle where no cyclic hardening effect is observed, the elastic limit range of the upper branch ($\Delta\sigma_{EU}$) predominantly depends on the TVF and the twin morphology at the compressive peak stress. The value of $\Delta\sigma_{EU}$ reflects the driving force to activate detwinning.

Figs. 8e, f, g, and h present the EBSD scans of the samples unloaded from the peak compressive stress at different strain amplitudes at half fatigue lives. At a strain amplitude of 0.4%, small amount of irregular-shaped tension twin fragments can be detected (Fig. 8e). These trivial residual twins might be generated in the early loading cycles and are retained in the later stage of the cyclic deformation. During the early stage of cyclic deformation, twinning–detwinning is effectively operated. The elastic limit ranges of the upper branch, which signify the activation of the detwinning process, can be very low, as evidenced by approximately 20 MPa for the first loading cycle (Fig. 7). As the number of loading cycles increases, the activity of twinning–detwinning process gradually diminishes (Dong et al., 2014) and the elastic limit range of the upper branch increases. When the number of loading cycles reaches half of the fatigue life, the elastic limit range is 100 MPa (see Fig. 4b, Fig. 5 and Fig. 7), which indicates that the dominating cyclic deformation mechanism has changed from twinning–detwinning to basal slips.

When the strain amplitude is larger than 0.4%, the twinning–detwinning process does not diminish at the half-life loading cycle and its role on cyclic deformation becomes more and more important as the strain amplitude increases. As shown in Fig. 7, when the strain amplitude increases from 0.4% to 0.6%, $\Delta\sigma_{EU}$ shows a decreasing trend. This signifies a transition of the yielding mechanism during **reversed loading** from compressive peak stress at the half-life cycle from basal slips to detwinning. At the strain amplitude of 0.6%, a minimum value of 32 MPa for $\Delta\sigma_{EU}$ is found. When the strain amplitude is further increased from 0.6% and up to 4%, the $\Delta\sigma_{EU}$ increases again. Similar to the first loading cycle, such an increase in $\Delta\sigma_{EU}$ implies the increasing driving force of detwinning as the strain amplitude is increased. It should be noticed that when the strain amplitude is 4%, $\Delta\sigma_{EU}$ is approximately 100 MPa which is identical to the elastic limit range of the lower branch, $\Delta\sigma_{EL}$.

From Fig. 8f, g, and h, it can be seen that the TVF at the minimum strain at half-life increases as the strain amplitude is increased. Moreover, at strain amplitudes of 1% (Fig. 8f) and 2% (Fig. 8g), the twin bands at half fatigue lives appear thinner than those observed at the first loading cycle. This can be attributed to the cyclic hardening effect caused by the accumulation of dislocations during cyclic deformation (Guillemer et al., 2011). The gliding dislocations due to basal/non-basal slips and the residual twin dislocations after repeated twinning–detwinning process can multiply, accumulate, and might be pinned at the grain boundaries and twin boundaries (Guillemer et al., 2011; Morrow et al., 2014). Certain

irreversible dislocation substructures can stabilize twin boundaries, making the twin growth more difficult (Dong et al., 2015). As a result, the twin bands shown in Figs. 8f and g are much thinner than those observed in Figs. 8b and c. Furthermore, the dislocation substructures generated during cyclic deformation can retard the twin boundary shrinkage, which makes the detwinning process more difficult (Dong et al., 2015). Therefore, the detwinning process during **reversed loading** from the compressive peak stress requires a higher driving force, which is manifested as a higher $\Delta\sigma_{EU}$ at the half fatigue life as compared to that at the first loading cycle, as shown in Fig. 7. When the strain amplitude is 4%, TVF is 94.4% and the twinning exhaustion occurs near the end of the compressive reversal. Most of the matrix has been twinned at the minimum strain (Fig. 8h). The microstructure exhibits a *c*-texture. Moreover, cyclic hardening due to repeated twinning-detwinning makes the detwinning process more difficult from the compressive peak stress during **reversed loading**. At a strain amplitude of 4%, the elastic limit range for the upper branch ($\Delta\sigma_{EU}$) at half fatigue life is 100 MPa, indicating the activation of basal slips during **reversed loading**.

3.4. Inflection point in the stress-plastic strain hysteresis loop

In the current work, the inflection point is defined as the turning point in the stress-plastic strain hysteresis loop where the continuous decreasing slope changes to an increasing slope as the plastic flow proceeds. Mathematically, the point is corresponding to $d^2\sigma/d^2\varepsilon_p = 0$ as shown in Fig. 3. The inflection point reflects a transition of the dominant plastic deformation mechanism from twinning/detwinning to dislocation slips. It should be noticed that twinning or detwinning can continue even after the inflection point. For example, the inflection point on the monotonic compression curve is depicted as the red solid circular point in Fig. 1. The inserted EBSD scans suggest that further twinning occurs after the inflection point.

The circular points indicated in Fig. 4a are the inflection points corresponding to the selective loading cycles at $\Delta\varepsilon/2 = 1.0\%$. The plastic strain range between the inflection point and the origin of the coordinate system is referred to as the inflection strain range. The value of the inflection strain range represents the plastic strain range where twinning/detwinning dominates the plastic deformation. It can be seen that for both upper and lower branches, the inflection strain increases in the first ten loading cycles. When the number of the loading cycles is larger than ten, the inflection strain range decreases as the number of the loading cycles increases. It has been mentioned that with the increasing number of loading cycles, cyclic hardening due to the accumulation of dislocations makes the twinning/detwinning more difficult. As a result, the material's capacity to twin and to detwin is decreased and a shortened inflection strain ranges are resulted in the compressive and tensile reversals, respectively.

Fig. 9 shows the inflection points on the stabilized stress-plastic strain hysteresis loops at different strain amplitudes and Fig. 10 presents the variation of inflection strain ranges as a function of the applied strain amplitude. It can be seen that both EDT (inflection strain range for the upper branch) and ET (inflection strain ranges for the lower branch) increase with the increasing strain amplitudes. With a larger strain amplitude under fully reversed loading, more tension twins are activated in the compression reversal and henceforth, more tension twins are

detwinned during the tensile reversal. Therefore, twinning/detwinning dominates the plastic deformation to a prolonged extent at larger strain amplitudes, resulting in larger EDT and ET. It is noticed in Fig. 10 that EDT approximately equals to ET at a given strain amplitude, confirming that the cyclic stability is reached.

Fig. 9. Inflection points on the stabilized stress-plastic strain hysteresis loops at different strain amplitudes.

Fig. 10. Inflection strain ranges as a function of the applied strain amplitude under fully reversed strain-controlled loading.

3.5. High-low two-step fully reversed strain-controlled cyclic loading

With the disclosure of the elastic limit range evolution and its mechanism from the single-step fully reversed strain-controlled experiments, it would be of interest to explore whether similar phenomena exist in the material subjected to two-step cyclic loading.

Fig. 11. High-low two-step strain-controlled experiments.

In a high–low loading sequence, a specimen is first subjected to cyclic loading at a high constant strain (or stress) amplitude for a certain number of loading cycles followed by cyclic loading at a lower strain (or stress) amplitude. In the current work, the following high–low two-step sequence loading experiments were conducted:

Step 1: cyclic tension-compression at $\Delta\epsilon/2 = 4\%$ for five loading cycles, then unloaded at C6.0 or C6.1, where the first index “x” in notation “Cx.y” denotes the number of loading cycle at which the specimen has been loaded and the second index “y” denotes the specific position in the stress-strain hysteresis loop. For the second index “y,” “0” represents the position where the strain is zero on the upper branch of the stress-strain curve, and “1” denotes the position of the maximum strain in the stress-strain hysteresis loop (refer to Fig. 11).

Step 2: cyclic tension-compression started from C6.0 or C6.1 with $\Delta\epsilon/2 = 0.7\%$, 0.5% , and 0.35% until final fatigue fracture.

Fig. 11 displays the stress-strain hysteresis loops obtained from the high–low two-step sequence loading experiments, in which the specimens were subjected to cyclic tension-compression at $\Delta\epsilon/2 = 4\%$ for five loading cycles and subsequently to tension-compression with $\Delta\epsilon/2 = 0.7\%$. In order to show the evolution of the stress-strain response, the stress-strain hysteresis loops of the first ten loading cycles and the loading cycles approximately corresponding to a quarter, a half, and three quarters of fatigue life are presented. When the second step starts from C6.0, the shapes of the upper and lower branches of a stress-strain hysteresis loop are practically identical. When the second step starts from C6.1, asymmetric stress-strain loops can be observed. As the number of loading cycles increases, the shapes of the stress-strain hysteresis loops keep almost unchanged. The symmetric/asymmetric stress-strain hysteresis loops of the second step loading experiment can be ascribed to the different microstructures at C6.0 and C6.1 resulted from the cyclic deformation at the end of the first loading step.

It should be noticed that C6.0 started with a zero strain after termination of the first step loading at zero strain. The difference between the stress at zero strain at the end of the first loading step and that at the start of the second loading step was due to stress relaxation. There was 50 seconds between the termination of the first loading step and the start of the second loading step. The stress relaxation occurring between two loading steps is a common phenomenon that can be observed even in steels which do not display significant time-dependent behavior. The stress relaxation may have a slight influence on the deformation of the few loading reversals in subsequent loading step.

Fig. 12. Stress range-plastic strain range for the second step started from C6.0 in the two-step loading experiment.

Fig. 13. Variations of the elastic limit ranges with respect to the increasing loading cycles for the

second step started from C6.0 in the two-step loading experiment.

In order to study the evolution of the elastic limit range during high-low two-step loading experiments, the experimental result from the second-step cyclic loading with a strain amplitude of 0.7% started from C6.0 is selected for a detailed analysis. Figs. 12 and 13 present the $\Delta\sigma$ - $\Delta\varepsilon_p$ curves and the variations of the elastic limit ranges with the increasing loading cycles, respectively. It can be seen that the elastic limit ranges for both upper branches and lower branches are approximately 85 MPa, and they do not change with the number of loading cycles. Unlike the single-step strain-controlled experiment with an identical strain amplitude of 0.7% which displays stress-strain asymmetry, the upper and lower branches of the stress-plastic strain hysteresis loops for the second step of the two-step experiment are almost identical (Fig. 12). This indicates that the cyclic plastic deformation mechanism under single step loading at $\Delta\varepsilon/2=0.7$ is different from that loaded at the same strain amplitude but with a preloading history at $\Delta\varepsilon/2=4\%$. For the single-step cyclic loading, partial twinning-complete detwinning process dominates the cyclic plastic deformation, which results in an asymmetric stress-strain hysteresis loop. The near-symmetric stress-strain hysteresis loops at $\Delta\varepsilon/2=0.7\%$ with prior loading history are due to the microstructures resulted from the first loading step, which will be discussed in the following paragraph.

Fig. 14. Microstructures measured by EBSD for ZK60 specimens having experienced cyclic tension-compression started from C6.0 at $\Delta\varepsilon/2=0.7\%$ in the second step for 1,500 loading cycles. The specimens were unloaded at: (a) minimum strain, (b) maximum strain.

For the second step cyclic loading at $\Delta\varepsilon/2=0.7\%$ started from C6.0, it was reported (Dong et al., 2015) that the resulted microstructure at C6.0 (after five tension-compression loading cycles at $\Delta\varepsilon/2=4\%$) consists of 43% twins and 57% basal textured matrix. In order to study the microstructure evolution during the second loading step, microstructures were measured by EBSD for two specimens having experienced cyclic tension-compression at $\Delta\varepsilon/2=0.7\%$ in the second loading step for 1,500 loading cycles (Fig. 14). One specimen was unloaded at the

minimum strain (Fig. 14a) and the other one was unloaded at the maximum strain (Fig. 14b). The TVFs in Fig. 14a and Fig. 14b are ~59% and ~49%, respectively. It should be noticed that the TVF at C6.0 after five loading cycles in the first loading step is very similar to the TVFs shown in Figs. 14a and b, where approximately half of the material is basal-textured matrix and the other half are *c*-textured twins. Therefore, under the compressive reversal in the second step at $\Delta\epsilon/2=0.7\%$ started from C6.0, the matrix domain undergoes twinning while the twin domain can be literally regarded to experience “detwinning.” Since the strain amplitude (0.7%) is small, a slight increase (and decrease) in the twin (and matrix) volume fraction is produced, which results in insignificant changes of the twin and matrix volumes, i.e., the volume fractions of twin and matrix remain almost unchanged (see Figs. 14a and b). If the loading direction is reversed to tension, the same process is repeated: half of the material is subjected to twinning while the other half undergoes detwinning. Therefore, the microstructural change under either tensile or compressive reversal involves simultaneous twinning and detwinning for approximately half of the material. When the strain amplitude is small, such as 0.7%, the compressive reversal and the tensile reversal exhibit almost identical mechanical response (see Fig. 12). As a consequence, the stress-strain hysteresis loops display nearly a symmetric shape, as being clearly evidenced in Fig. 11.

It has been revealed that the elastic limit range of 100 MPa during **reversed loading** from a detwinning-completed *a*-textured material is dominantly associated with basal slips. For the current second-step cyclic loading, the elastic limit ranges for both upper branches and lower branches are approximately 87 MPa. This indicates that yielding during **reversed loading** from the tensile and compressive peaks involves twinning and detwinning processes, respectively. As shown in Fig. 14, the material at the maximum stress and minimum stress contains approximately equal amount of deformation twins. The existence of deformation twins signifies the presence of twinning dislocations pinned at grain boundaries at the maximum and minimum stresses. This further infers that the critical driving forces to forward (retwinning) and reverse (detwinning) the motion of pinned twinning dislocations dominate the microyielding at the lower branch and upper branch, respectively. Since the TVF does not change significantly at the maximum and minimum stresses due to a small strain amplitude of 0.7% in the second-step loading, an approximately equal driving force to forward and reverse the pinned twinning dislocations is expected to be resulted, which might account for the nearly identical elastic limit range at both the lower and upper branches. Moreover, the significant cyclic hardening resulted from the first step loaded at a large strain amplitude (4%) might be responsible for the substantial increase in the elastic limit ranges in the second-step loading experiment.

Fig. 15. Stabilized stress range-plastic strain range at selective strain amplitudes for the second loading steps started from C6.0 in the two-step loading experiments.

Fig. 16. Variations of the elastic limit ranges with respect to the applied strain amplitudes for the second loading steps started from C6.0 in the two-step loading experiments.

Fig. 15 presents the stress range-plastic strain range relationship obtained from the stabilized stress-strain hysteresis loops at the selective strain amplitudes for the second loading steps started from C6.0 in the two-step loading experiments. Fig. 16 shows the variations of the elastic limit ranges as a function of the applied strain amplitude. It can be seen that the upper branch and lower branch of the stress-plastic strain hysteresis loop exhibit near symmetric shape for all of the selected strain amplitudes ranging from 0.35% to 0.7% for the second step started from C6.0. The elastic limit ranges are approximately 85 MPa for both upper branch and lower branch irrespective of the applied strain amplitudes.

The experimental results shown in Figs. 13 and 16 reveal an interesting phenomenon: the elastic limit ranges for both upper branch and lower branch in the second loading step started from C6.0 retains at ~87 MPa regardless of the applied strain amplitudes (0.7%, 0.5%, and 0.35%) and the loading cycles. It suggests the phenomenon should be related to the TVF (~50%) and the significant cyclic hardening resulted from first loading step with a large strain amplitude of 4%. In the second loading step, the strain amplitudes, ranging from 0.35% to 0.7%, are significantly smaller than 4% at the first loading step. It has been mentioned that the TVFs after 1,500 loading cycles at $\Delta\epsilon/2=0.7\%$ in the second step (Fig. 14) are similar to the TVF after five loading cycles at $\Delta\epsilon/2=4\%$ in a single step loading (Dong et al., 2015). In other words, the second loading step at $\Delta\epsilon/2=0.7\%$ produces a marginal change in the TVF (~50%) generated in the first loading cycle because of the much smaller strain amplitude. For the same reason, the TVF (~50%) resulted from the first loading cycle should remain unchanged as well during the second loading steps where even smaller strain amplitudes of 0.5% and 0.35% are applied. This marginal change of TVF during the second loading step due to the small applied strain amplitude explains why the elastic limit range is independent of the applied strain amplitude (0.7%, 0.5%, and 0.35%) and the loading cycles as shown in Figs. 13 and 16.

Fig. 17. $\Delta\sigma$ - $\Delta\epsilon^p$ curves for the second loading step started from C6.1 in the two-step loading experiment.

Fig. 18. Variations of the elastic limit ranges with respect to the loading cycles for the second step started from C6.1 in the two-step loading experiment.

In addition to the second step started from C6.0, high-low two-step experiments were also conducted with the second step started from C6.1. Fig. 17 presents the $\Delta\sigma$ - $\Delta\varepsilon^p$ curves and Fig. 18 shows the variations of the elastic limit ranges with respect to the loading cycles for the second step at $\Delta\varepsilon/2=0.7\%$ started from C6.1 in the two-step experiment. It can be seen that the upper branch and lower branch for a given loading cycle do not coincide, indicating an asymmetric shape of the stress-plastic strain hysteresis loop. The elastic limit ranges for the upper branches and lower branches are approximately 100 MPa and 68 MPa, respectively. It is noticed that $\Delta\sigma_{EU}$ and $\Delta\sigma_{EL}$ do not change with the increasing loading cycles. The microstructure at C6.1 after five tension-compression loading cycles at $\Delta\varepsilon/2=4\%$ exhibits a strong basal α -texture (Dong et al., 2015). At the maximum strain/stress in the first loading step, detwinning has been completed and few residual tension twins can be found. The microstructure at C6.1 is similar to the initial microstructure of the extruded ZK60, both displaying a strong basal α -texture. Therefore, similar to the single-step strain-controlled cyclic loading experiment with a strain amplitude of 0.7%, microyielding mechanism during **reversed loading** for the second step (started from C6.1) can be interpreted as basal slips for the lower branch and detwinning for the upper branch. The much higher $\Delta\sigma_{EU}$ for the high-low two-step experiment compared to that exhibited in the single-step experiment with an identical strain amplitude can also be attributed to the enhanced driving force to activate detwinning produced by the cyclic hardening in the prior cyclic loading history.

Fig. 19. $\Delta\sigma$ - $\Delta\varepsilon^p$ curves obtained from the stabilized stress-strain hysteresis loops for the second loading step at different strain amplitudes (0.7%, 0.5%, and 0.35%) with the second step starting from C6.1 in the two-step loading experiment.

Fig. 19 presents the $\Delta\sigma$ - $\Delta\varepsilon^p$ curves obtained from the stabilized stress-strain hysteresis loops of the selected strain amplitudes for the second step started from C6.1 in the two-step loading experiment. Fig. 20 plots the variations of the elastic limit ranges with respect to the applied strain amplitudes for the second loadings step. It can be seen from Fig. 19 that when the strain amplitude is 0.7%, the upper branches and the lower branches of the stress-plastic strain hysteresis loops exhibit obvious asymmetric shapes. The elastic limit ranges for the upper and lower branches are 68 MPa and 100 MPa, respectively. It can be readily derived that partial twinning-detwinning process dominates the cyclic deformation during the second step at the strain amplitude of 0.7%. As the strain amplitude decreases, the dissimilarity of the upper branch and lower branch decreases. When the strain amplitude is 0.35%, the upper and lower branches tend to be identical, resulting in a symmetric stress-plastic strain hysteresis loop with both $\Delta\sigma_{EU}$ and $\Delta\sigma_{EL}$ being approximately 100 MPa. With a lower strain amplitude in the second loading step, the cyclic plastic deformation is dominated by dislocation slips. In brief, when the strain amplitude decreases from 0.7% to 0.35%, the cyclic plastic deformation mechanism shows a transition from partial twinning-detwinning to dislocation slips, resulting in a more symmetric stress-plastic strain hysteresis loop.

Fig. 20. Variations of the elastic limit ranges with respect to the applied strain amplitudes for the second step starting from C6.1 in the two-step loading experiment.

3.6. Stress-controlled cyclic loading

Fig. 21 shows the cyclic stress–strain hysteresis loops obtained from the stress-controlled experiments where the applied load has a stress amplitude of 200 MPa and a compressive mean stress of -50 MPa. For the first ten loading cycles, twinning occurs in the compressive reversals and detwinning occurs in the tensile reversals. The stress-strain hysteresis loops exhibit a pronounced asymmetry shape. The maximum strain, $\varepsilon_{\max}$, and the minimum strain, $\varepsilon_{\min}$,

evolve in an opposite direction, resulting in a reduced width of the stress-strain hysteresis loop.

The shape of the hysteresis loops becomes less asymmetric as the loading cycles increase. The strain range of the stress–strain hysteresis loop decreases and the shape of the hysteresis loops becomes more and more symmetric as the loading cycles are increased. The stress-strain hysteresis loops evolves from an asymmetric shape to a near symmetric shape. The observed cyclic hardening reflects a transition of cyclic plastic deformation mechanism that is inherently caused by the microstructure evolution during the stress-controlled cyclic loading.

Fig. 21. Stress-strain hysteresis loops obtained from the stress-controlled experiment.

Fig. 22. Stress range-plastic strain range curves for stress-controlled cyclic loading

Fig. 22 shows the stress range-plastic strain range curves for the selected loading cycles obtained from the stress-controlled experiment. A glance of the results shown in Fig. 22 reveals that the shape of the upper and lower branches gradually evolves to be symmetric as the loading cycle is increased. When the number of loading cycles is 2,100, the upper branch and the lower branch are almost identical.

Fig. 23. Variations of the elastic limit ranges with respect to the loading cycles for the stress-controlled experiment.

Variations of elastic limit ranges with respect to the loading cycles are shown in Fig. 23. $\Delta\sigma_{EU}$ is practically unchanged with increasing loading cycles while $\Delta\sigma_{EL}$ increases as the loading cycle is increased, changing from a value of 20 MPa at $N=1$ cycle to 48 MPa at $N=2100$ cycles where N denotes the number of loading cycles. After $N=2,100$ cycles, $\Delta\sigma_{EL}$ saturates and becomes identical to $\Delta\sigma_{EU}$.

Similar to the strain-controlled experiments, the elastic limit ranges are directly related to the microstructures at the stress/strain peaks where **reversed loading** was started. Fig. 24 shows the microstructures measured by EBSD on the samples unloaded from different positions of the selected loading cycles in the stress-controlled experiment. During the initial compressive phase, bulk tension twins are nucleated when the stress reaches the twinning stress (134 MPa) of the material. As the compressive stress increases, the twinning process proceeds with continued twin nucleation and growth. When the stress reaches the minimum stress ($\sigma=-250$ MPa), the corresponding strain is -6.6%. The TVF at this point is approximately 77% (Fig. 24a). When **unloaded and reloaded** from the minimum stress, detwinning occurs and the elastic limit range is identified to be 46 MPa, as shown in Figs. 22 and 23. During the **reversed tensile loading**, detwinning dominates the plastic deformation. When the stress reaches the maximum stress, the upper branch of the stress-strain hysteresis loop for the first loading cycle shows a concave-down shape. This is different from the sigmoidal shape exhibited in the tensile reversal from the fully reversed strain-controlled experiment. No inflection point can be detected in the tensile reversal in the current stress-controlled experiment. Fig. 24b clearly shows that the detwinning process is not completed at the maximum stress. The residual twins shown in Fig. 24b exhibit shrunk twin thickness as compared to the twins fully developed at the minimum stress shown in Fig. 24a. The TVF decreases from 77% to 46%.

Fig. 24. EBSD scans for the stress-controlled experiment ($\Delta\sigma/2=200$ MPa, $\sigma_m=-50$ MPa): (a) at the minimum strain of the first loading cycle, (b) at the maximum strain of the first loading cycle, (c) at the minimum strain of the 10th cycle, (d) at the maximum strain of the 10th cycle, (e) at the minimum strain of the 2,100th cycle, (f) at the maximum strain of 2,100th cycle.

When unloaded from the maximum stress after the first loading cycle, yielding can be associated to the occurrence of further twinning (not basal slips) from the microstructure consisting of 46% twins and 54% matrix. It should be reiterated that the elastic limit range associated with twinning/detwinning strongly depends on the TVF and twin morphology for the first few loading cycles. The elastic limit range of the lower branch of the stress-strain hysteresis loop is approximately 20 MPa, smaller than 46 MPa of the upper branch. The difference between the elastic limit range at the lower branch and that at the upper branch might be due to the lower driving force responsible for further twinning (including retwinning and fresh twinning) during **reversed loading** from tensile peak stress. As the number of loading cycles increases, twinning exhaustion was approached at the minimum stress of a loading cycle. In the meantime, as the strain range of the tensile reversal is shortened during cyclic hardening in the stress-controlled experiment, less twins can be detwinned during the tensile reversal. As shown

in Figs. 24c and d for the 10th cycle, the TVFs at the minimum stress and the maximum stress are 79% and 64%, respectively. Twinning-detwinning process still occurs but the difference between the TVFs at the minimum stress and the maximum stress is much smaller as compared to that exhibited at the first loading cycle. Therefore, the difference between the elastic limit range in the upper branch and that in the lower branch for the 10th cycle is reduced. As shown in Figs. 22 and 23, the value of $\Delta\sigma_{EU}-\Delta\sigma_{EL}$ is 26 MPa at the first loading cycle but it reduces to 18 MPa at the 10th loading cycle. When the number of loading cycles reaches 2,100, the TVFs at the minimum stress and the maximum stress are practically the same, being approximately 78%. Interestingly, the elastic limit ranges for both the upper and lower branches are practically identical. The decreasing difference between the elastic limit range in the upper branch and that in the lower branch with respect to the increasing of loading cycles is believed to be related to the cyclic hardening associated with reduced twinning-detwinning activities. Further investigations are needed.

Despite similar TVFs at the minimum stress and maximum stress at the 2,100th loading cycle, it is believed that the twinning-detwinning process is effectively operated. This hypothesis can be confirmed from the much lower elastic limit range of 48 MPa as compared to the activation stress for basal slips, known to be approximately 100 MPa. As the number of loading cycles increases, the plastic strain amplitudes decrease obviously from 2.53% at the first loading cycle to 0.29% at N=2,100 cycles, indicating significant cyclic hardening. A small difference between TVF at the minimum stress and that at the maximum stress can still be detected by EBSD measurement. The experimental observation further confirms that twinning-detwinning occurs persistently during the entire loading history but its activity at the later stage of cyclic loading is reduced significantly.

4. Further discussions

4.1. Elastic limit and yielding

Elastic limit and yielding in material deformation are theoretically identical but practically ambiguous dependent on the applications. Theoretically speaking, yielding or elastic limit is the point where plastic deformation is about to occur with further loading. In practical applications, however, the identification of the elastic limit can be difficult due to the limit of accuracy in the load and strain measurements. An offset of plastic strain is often used to define the yielding point, such as 0.2% offset which is commonly used in engineering design applications. There is no widely acceptable definition of elastic limit in cyclic plasticity experiments.

For Mg alloy, both dislocation slips and twinning/detwinning play important roles in cyclic plasticity. For example, the extruded Mg subjected to monotonic compression undergoes a series of microscopic processes, including microyielding due to basal slips, twinning, and non-basal slips. Upon the reversal from the prior loading, the physical processes underlying plastic deformation can be changed completely. Therefore, the identification of the specific microyielding micro-mechanisms under different loading paths requires a reliable and consistent analysis of the elastic limit. In the current discussion, the stress corresponding to a small plastic

strain offset (10^{-5}) is chosen as the elastic limit. Such an elastic limit criterion enables an accurate capture of microyielding regardless of the loading histories.

4.2. Plastic deformation mechanism during unloading

Based on the experimental results obtained from single-step strain-controlled, two-step strain-controlled, and stress-controlled experiments, it can be concluded that the reported elastic limit range depends on the microstructure at the peak stress. If the microstructure at the peak stress displays a strong basal α -texture, yielding during **reversed loading** is dominantly associated with the activation of basal slips, which is macroscopically manifested as an elastic limit range of 100 MPa. If the microstructure at the peak stress contains tension twins, the elastic limit range during **reversed loading** reflects the activation stress of detwinning process. The activation of detwinning can be driven by the internal stresses generated during the prior twinning process. For example, when loaded from the compressive peak stress in the single-step fully reversed strain-controlled experiments, tensile internal stress generated by twinning in the prior compression reversal may exist in the twinned grains (Muránsky et al., 2009) even when the applied external stress is still compressive. Such internal tensile stresses can act as the driving force of the detwinning process during **reversed loading**.

The activation of detwinning involves the gliding of twin boundaries. Therefore, the elastic limit range also reflects the driving force to activate gliding of twin boundaries. The stability of twin boundaries is influenced by twin volume fraction, twin morphology, and the degree of cyclic hardening, all of which being strongly dependent on the cyclic loading history. During the initial stage of the cyclic deformation, such as the first loading cycle, the effect of cyclic hardening is minor. The mobility of the twin boundaries is mainly affected by the TVF and twin morphology. For instance, the TVF at the peak compressive stress increases with the applied strain. The twin bands become much wider as well (see Fig. 8). Correspondingly, more dislocations can be pinned at twin boundaries as the strain amplitude is increased. As a result, the elastic limit range in the upper branch of the stress-strain hysteresis loop at the first loading cycle increases as the applied strain amplitude increases (Fig. 7). This phenomenon can also be observed from the stress-controlled experiments. At the first loading cycle of the stress-controlled experiment, the TVF at the compressive peak stress is 77% (Fig. 24a) and the TVF at the tensile peak stress is 46% with the twin bands displaying a thinner morphology (Fig. 24b). Consistently, the elastic limit range for the lower branch (20 MPa) is significantly lower than that for the upper branch (46 MPa).

4.3. Plasticity models for Mg alloys

Material deformation models are used to describe the stress-strain response when the material is subjected to external loading. In the framework of cyclic plasticity, the conventional macroscopic phenomenological models, such as Armstrong-Frederick model (Armstrong and Frederick, 1965), Chaboche model (Chaboche, 1986), and Ohno-Wang model (Ohno and Wang, 1993), were constructed from macroscopic cyclic deformation experiments. Although the phenomenological models are able to describe cyclic stress-strain response to some extent, the microscopic physical mechanisms were usually not considered in these models. Additionally,

due to the adoption of the isotropic von-Mises yielding surface, such models can be used only for the isotropic materials. With the initial texture, magnesium alloys usually exhibit strong anisotropic mechanical behavior. Therefore, such macroscopic phenomenological models cannot be directly applied to describe the cyclic plasticity of Mg alloys.

In the recent years, crystal plasticity models were developed to simulate the deformation behavior of Mg alloys (Benedetti et al., 2016; Guillemer et al., 2011; Hama and Takuda, 2011; Hama et al., 2016; Lebensohn and Tome, 1993). The physical-mechanism based crystal plasticity models attempt to take account of the essential plastic deformation mechanisms (including dislocation slips, twinning and detwinning processes) in Mg. Some of the crystal plasticity based constitutive models have been established to describe the uniaxial ratchetting (Yu et al., 2014) and dynamic stress-strain responses (Xie et al., 2016) of polycrystalline magnesium alloys. Very recently, elastic viscoplastic self-consistent (EVPSC) models that incorporates the mechanism of twinning and de-twinning (TDT) have been applied to investigate the cyclic deformation of Mg alloys (Qiao et al., 2015; Wang et al., 2013a; Wang et al., 2013b). The stress-strain response, texture evolution, lattice-strain evolution during cyclic deformation can be simulated for Mg alloys to some extent. However, accurate modeling of cyclic plasticity of Mg alloys is still a challenging task, especially for simulating the evolving cyclic deformation with applied loading cycles. This challenge lies in a lack of the detailed analysis of the characteristic cyclic plasticity for Mg alloy. The experimental results obtained in current work uncover characteristic cyclic plasticity deformation for Mg alloy. The macroscopic features and the associated micro-mechanisms are essential and should be accounted for in developing advanced physics-based cyclic plasticity models for Mg alloys.

5. Conclusions

A detailed analysis of the plastic deformation characteristics was performed for an extruded magnesium alloy ZK60 under uniaxial cyclic loading. The experiments were performed under single-step strain-controlled loading, two-step strain-controlled loading, and stress-controlled loading. An elastic limit range was defined to correspond to a small plastic strain of 10^{-5} . The following conclusions can be drawn from the experimental study:

1. Under cyclic tension-compression, the elastic limit range is closely related to the microstructure of the material at the peak stress prior to the loading reversal. If the microstructure at the peak stress displays a strong basal a -texture, yielding is dominantly associated with the activation of basal slips. The elastic limit range to activate basal slips for the ZK60 magnesium alloy under investigation is 100 MPa.
2. If the microstructure at the peak stress contains tension twins, the elastic limit range during subsequent loading reversal reflects the activation of gliding of twin boundaries for the twinning/detwinning process. Dependent on the twin volume fraction and loading history, the elastic limit range varies from 20 MPa to 100 MPa for the material under investigation.
3. The inflection point in the stress-plastic strain hysteresis loop signifies a transition of the dominant plastic deformation mechanism from twinning/detwinning to dislocation slips. The inflection strain range is a measure of plastic strain range from the starting point of a

loading reversal to the inflection point in the stress-plastic strain hysteresis loop. Under fully reversed strain-controlled loading, the inflection strain range is practically identical for both tension and compression reversals in a loading cycle and it is proportional to the applied strain amplitude.

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