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Electron backscatter diffraction observations of twinning-detwinning evolution in a magnesium alloy subjected to large strain amplitude cyclic loading

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Abstract: An extruded ZK60 magnesium alloy was subjected to fully-reversed strain-controlled cyclic loading at a strain amplitude of 4.0% in the extrusion direction in ambient air. Electron backscatter diffraction (EBSD) analyses were conducted on samples taken from companion specimens terminated at different loading cycles to study the twinning-detwinning process and the evolution of the twin structures at different stages of cyclic deformation. It is observed that the twin nucleation sites are increased whereas twin growth/shrinkage is inhibited due to repeated twinning-detwinning. The enhanced twin nucleation sites are responsible for the observed increase in the number of twin lamellae and the increased twin volume fraction with loading cycles. Cyclic loading enhances formation of compression and double twins which do not result in immediate fracture of the material. With increasing number of loading cycles, more and larger sized residual tension twin lamellae can be detected by EBSD, but the total volume fraction of the residual twins is trivial.

Keywords: Cyclic deformation, twinning-detwinning, magnesium alloy

1. Introduction

Due to the low density and high strength-to-weight ratio, magnesium (Mg) alloys are becoming attractive engineering materials for structural components [1]. Engineering

components are often subjected to cyclic loading and therefore, cyclic deformation and fatigue of Mg alloys are important design considerations. With a hexagonal close-packed (HCP) crystal structure, Mg has a limited number of slip systems. Mechanical twinning is the major difference from a conventional metal and plays an important role in deformation and failure in Mg. Despite early recognition of the deformation phenomenon, studies of the cyclic plastic deformation and the associated micro-mechanisms in Mg are very limited. Common twinning modes include $\{10\text{-}12\}$ tension twinning, $\{10\text{-}11\}$ compression twinning, and $\{10\text{-}11\}$ - $\{10\text{-}12\}$ double twinning [2-4]. $\{10\text{-}12\}$ tension twinning can be activated by tension along the c -axis or compression perpendicular to the c -axis. The “ c -axis” denotes the basis vector c in the hexagonal lattice system. $\{10\text{-}11\}$ compression twinning, on the contrary, can accommodate compression along the c -axis. Secondary $\{10\text{-}12\}$ tension twinning can be activated within the primary $\{10\text{-}11\}$ compression twins to form $\{10\text{-}11\}$ - $\{10\text{-}12\}$ double twins. It is suggested that the formation of double twins in polycrystalline Mg alloys under large tensile straining ($\sim 22\%$ in Ref. [5]) can cause an immediate material fracture due to the severe slip localization within these twins [5,6]. Koike et al. [7] experimentally found double twins in a specimen after fatigue failure. However, the observation of compression/double twinning during cyclic deformation prior to fatigue fracture was not reported.

For as-extruded Mg alloys with a strong initial basal texture, $\{10\text{-}12\}$ tension twins can be activated under compression along the extrusion direction and detwinning occurs under the subsequent tensile loading. Twinning-detwinning plays an important role in the cyclic deformation and fatigue of Mg alloys. Work [8-18] was done on the microstructure evolution during the cyclic deformation of Mg alloys. Most studies focused on the microstructural change during the first loading cycle [14,15]. Little work has been done on the microstructure evolution during cyclic deformation. Moreover, existing work was mainly conducted at a relatively small strain amplitude [8,9,13,14,15] where the stress-strain hysteresis loops were asymmetric. Very limited work has been done on the microstructure evolution under cyclic loading at a large strain amplitude that exhibits a near-symmetric stress-strain hysteresis loop [20]. Furthermore, investigations emphasizing the microstructural characterization were mainly conducted by neutron diffraction [8,9,11,12] and optical microscopy [13-18]. Neutron diffraction characterizes the twinning-detwinning process in a material aggregate containing a large number of grains without details of local twin structures [11,12]. Optical microscopy can provide morphology change during twinning-detwinning but is unable to determine the crystal orientation [16]. Limited work has been done on the microstructure evolution in Mg alloys during cyclic deformation measured by electron backscatter diffraction (EBSD) [19,21]. The twinning/detwinning structures characterized by EBSD in prior work were mainly associated with different positions in a single loading cycle. The evolution of twinning structures with

respect to the increasing loading cycles is still unknown. In the current study, evolution of local twinning/detwinning structures, such as twin type, twin morphology, and twin volume fraction, at different stages of cyclic deformation is investigated by EBSD in detail.

2. Material and experiment

In this paper, a hot extruded ZK60 Mg alloy with a composition of Mg-6.0%Zn-0.5%Zr was studied. The initial material exhibits a twin-free microstructure which consists of a mixture of small equiaxed grains (5-25 μm) and large elongated grains (larger than 50 μm). The Mg alloy displays a typical basal texture [24]. Dog-bone shaped cylindrical specimens with a diameter of 10 mm within a 15 mm gage section were machined from the extruded bar along the extrusion direction. The specimens were subjected to fully-reversed strain-controlled tension-compression at a strain amplitude of 4%. To study the microstructure evolution during cyclic deformation, experiments of companion specimens were terminated at different positions in the stress-strain hysteresis loops of the first, sixth, and 21st loading cycles. Each companion specimen was unloaded to zero stress from the designed point on the stress-strain hysteresis loop. It is assumed that minimal or no twinning or detwinning occurs during such unloading. A cylindrical sample was cut from the gage section of each tested specimen for EBSD characterization. The observing plane was perpendicular to the extrusion direction. Basic mechanical properties and original microstructures of the ZK60 Mg alloy were reported in a separate paper [24].

3. Results and discussion

Fig. 1 presents the microstructure evolution measured by EBSD of the extruded ZK60 alloy under cyclic deformation at a strain amplitude of 4%. Companion specimens were unloaded at the positions of minimum strain (C1.1, C2.1 and C6.1), zero strain in the tensile reversal (C1.2 and C6.2), maximum strain (C1.3, C6.3 and C21.3), and zero strain in the compressive reversal (C1.4 and C6.4), respectively, at different loading cycles. The first index “x” in notation “Cx.y” indicates the number of loading cycles that the specimen has been loaded and the second index “y” denotes the specific position in the stress-strain hysteresis loop. All the experiments started with compressive loading reversal. Fig. 2 shows the tension twin volume fraction (TVF) developed in the material as a function of loading cycles. The TVF is calculated from an EBSD scan area of 200 μm ×200 μm in each sample. Two or more scans having the same area size were measured by EBSD in the same sample. It is confirmed that the TVF acquired from an area of 200 μm ×200 μm can statistically represent the TVF in the current material. Also, it should be mentioned that the high-magnified EBSD area in Fig. 1 is only used to show the featured local microstructure at the specific loading position.

When the stress reaches -134 MPa during the first loading reversal, tension twins are activated and the strain hardening rate keeps a constant low value until the minimum compressive strain C1.1. Fresh tension twins are observed mostly in the large grains (Fig. 1(C1.1)) and the TVF is approximately 29% at C1.1 (Fig. 2). During unloading after the first compressive peak strain, a significant Baushinger effect is exhibited, indicating activation of detwinning [8,9]. This can be attributed to the internal tensile stresses developed in the twinned grains [9]. The tensile reversal exhibits a pronounced sigmoidal shape. Fig. 1(C1.2) presents the EBSD microstructure at zero strain after loading from the first compressive peak strain. Tension twins can be clearly found both inside the grains and near the grain boundaries. The TVF at C1.2 is 4%, indicating that the detwinning process has not been fully completed at the zero strain. After C1.2, a rapid increase of strain hardening rate is exhibited, which is due to the activation of non-basal slips during the later stage of detwinning [8-17]. When the strain reaches 4%, as shown in Fig. 1(C1.3), detwinning is completed with very few residual tension twins. Interestingly, some {10-11}-{10-12} double twins can be detected, as shown by the black arrows in Fig. 1(C1.3). These double twin boundaries are confirmed to have a 38° misorientation angle about the $\langle 2-1-10 \rangle$ rotation axis. It is well known that during monotonic tension, a strain of 4% cannot induce double twins [5]. Double twins were reported to occur at an approximately 22% tensile strain [5] in an extruded Mg alloy, which immediately led to the final fracture of the material [5,6]. However, in the current study of cyclic deformation, double twins are observed in the material at 4% strain after prior compression to -4%. The observation of double twins at a tensile strain of 4% rather than at a much larger strain near the fracture point [5] has never been reported. The activation of double twin can be ascribed to the increased localized stress and the multiplied local defects induced by the prior twinning and detwinning process under reversed loading. It should be emphasized that double twins found at C1.3 do not lead to the abrupt and immediate fracture of the material.

When the specimen is reloaded for a second compressive loading reversal, tension twins are activated again. Unlike the smooth twin boundaries developed during the initial compression phase (Fig. 1(C1.1)), the twin boundaries formed during the second compressive loading reversal are serrated, as shown in Fig. 1(C1.4). In addition, the lenticular shaped twin lamellae are found to be fragmented (see arrows in Fig. 1(C1.4)). The TVF at C1.4 (zero strain) is approximately 20% (Fig.2). When the strain reaches -4% in the second compressive loading reversal, as shown in Fig. 1(C2.1), most of the matrix is twinned. The TVF increased from 29% at C1.1 to 70% at C2.1. The number of tension twin boundaries is significantly decreased in Fig. 1(C2.1) as compared to that in Fig. 1(C1.1). This is caused by the coalescence of twin boundaries occurring between two neighboring twins of the same variant.

After five loading cycles, the stress-strain hysteresis loop exhibits an almost fully

symmetrical shape. Both the tensile reversal and the compressive reversal show a sigmoidal shape. During the compressive reversal, tension twins persist to be activated. C1.4 and C6.4 are the same positions of zero strain in the compressive reversals of the first and sixth loading cycles, respectively. The influence of cyclic loading on twinning behavior can be investigated by comparing the microstructures at C1.4 and those at C6.4. First, the number of twin lamellae is increased from about 35 in Fig. 1(C1.4) to more than 100 in Fig. 1(C6.4) within the observation area shown in Fig.1. Moreover, the twin lamellae in Fig. 1(C6.4) are narrower and more fragmented as compared to those in Fig. 1(C1.4). The widths of twin lamellae in Fig. 1(C6.4) are less than $2\mu\text{m}$ whereas most in Fig. 1(C1.4) are between $3\mu\text{m}$ and $6\mu\text{m}$. Despite the smaller width of the twin lamellae at C6.4, the TVF (40%) at C6.4 is much higher than that (20%) at C1.4. This is due to the significantly increased number of twin lamellae developed during cyclic deformation.

Cyclic loading results in repeated twinning-detwinning, which enhances the twin nucleation sites and inhibits twin growth. Such effects are inherently caused by the accumulation of residual dislocations during cyclic deformation. The residual dislocations consist of two major types. First, the gliding twinning dislocations (TDs) can be either retained inside the grains or pinned at the grain boundaries as a result of repeated twinning-detwinning [22]. Second, basal slips are operative during twinning-detwinning due to their very low critical resolved shear stress (CRSS) [3]. Non-basal slips can be also activated after twinning exhaustion and after completion of detwinning. The glide dislocations due to slips can multiply and interact with twins, resulting in certain dislocation substructures within the grains and at the vicinities near the twin/grain boundaries [23]. As directly evidenced by TEM observation in Ref. [23], cyclic loading increases dislocation density in the matrix and generates dislocation substructures due to intense secondary slips and secondary twins inside the primary twin. Certain accumulated dislocation substructures accompanied by the increased local stress can act as effective twin nucleation sites. As the dislocation density is increased in the cyclically hardened material, an increasing number of twin nucleation sites is present. This is confirmed by the observation that the number of nucleated twin lamellae is increased after cyclic loading (C6.4) as compared to that at the first loading cycle (C1.4). With respect to twin growth, residual dislocation substructures accumulated inside the grain can block the glide of TDs during the twin growth, leading to inhibition of twin growth and henceforth resulting in a smaller twin width (compare Fig. 1(C1.4) and Fig. 1(C6.4)). Such a barrier effect of residual defects to the gliding TDs can also cause a nonuniform growth of the twin boundaries, resulting in serrated boundaries of the fragmented twins as observed in Fig. 1(C6.4).

Fig. 1(C6.1) presents the microstructure at -4% of the sixth loading cycle. Comparing Fig. 1(C6.1) with Fig. 1(C1.1) and Fig. 1(C2.1), it can be seen that with increasing number of loading

cycles, TVF at -4% strain increases. The TVFs are 29%, 70%, and 90%, respectively, at C1.1, C2.1 and C6.1 (Fig.2). At C6.1, twinning has been exhausted and non-basal slips occur. With unloading from the compressive peak strain, detwinning occurs. Fig. 1(C6.2) shows the microstructure at a zero strain in the tensile reversal of the sixth loading cycle. It can be seen that detwinning has not been completed and the TVF in Fig. 1(C6.2) is approximately 43%. Loading from C6.1 to C6.2, approximately 52% of the twins developed during the compressive loading reversal are detwinned. At C6.2, some of the twins are fragmented and have serrated boundaries. This indicates that the obstacle effect of residual dislocations on the gliding TDs is operative during detwinning. The inhibition of detwinning caused by the residual dislocation substructures retards the twin shrinkage and results in the serrated twin boundaries.

Fig. 1(C6.3) presents the microstructure at 4% of the sixth loading cycle. At this point, detwinning is completed, and a small amount of residual twins is measured, as shown by arrow A in Fig. 1(C6.3). Moreover, as indicated by arrow B in Fig. 1(C6.3), compression twins are detected. The compression twin is confirmed to have a 56° misorientation angle about the $\langle 2\text{-}1\text{-}10 \rangle$ rotation axis. Compression and double twins activated from monotonic tension of a Mg alloy are known to cause a rapid material failure due to the severe slip localization within these twins [5,6]. In the current study, double twins are detected in the first loading cycle and compression twins are found after six loading cycles. Rather than fractured upon the occurrence of double/compression twin, the specimen fails under compression after experiencing 31 loading cycles [24]. The result indicates that double twins and compression twins can be induced under cyclic deformation but the formation of these twins do not lead to immediate fracture of the material.

For an extruded Mg alloy, compressive load results in tension twins and the subsequent tensile loading reversal leads to detwinning. The residual twins refer to the twins left in the material subjected to cyclic loading after the completion of the tensile loading reversal in a loading cycle. In order to understand the evolution of residual twins, Fig. 1(C21.3) presents the microstructure at the maximum strain of the 21st loading cycle. By comparing Fig. 1 (C1.3), Fig. 1(C6.3), and Fig. 1(C21.3), it can be seen that with increasing loading cycles, more residual twins can be found, and the size of the residual twin lamellae becomes larger. The TVFs are 0%, 0.6%, and 3%, respectively, at C1.3, C6.3, and C21.3 (see Fig. 2). The total volume fraction of the residual twins, however, is small even after 21 loading cycles under a large strain amplitude of 4%. Moreover, these residual twins are mainly distributed near the grain boundaries, as shown in Fig. 1(C21.3). This observation confirms that the increased dislocation density in the vicinities near the grain boundaries plays an important role in detwinning inhibition.

4. Conclusions

The twinning-detwinning evolution under cyclic deformation at a strain amplitude of 4% was investigated by using EBSD. Cyclic hardening caused by twinning/detwinning and dislocation slips increases twin nucleation sites but inhibits twin growth and twin shrinkage. The enhanced twin nucleation sites result in an increase in the number of twin lamellae and an increase in the twin volume fraction with increasing number of loading cycles. The inhibition of twin growth and shrinkage leads to serrated boundaries of fragmented twins. Double twins and compression twins can be introduced by cyclic loading and the formation of these twins does not lead to immediate fracture of the material. With increasing number of loading cycles, more and larger sized residual tension twin lamellae can be detected by EBSD, but the total volume fraction of the residual twins is trivial.

Acknowledgements

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Figures

Figure 1 Twinning-detwinning evolution of ZK60 Mg alloy during 4% strain amplitude cyclic deformation measured by EBSD.

Figure 2 Tension twin volume fraction (TVF) developed with loading cycles

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