Understanding Low-cycle Fatigue Life Improvement Mechanisms in a Pre-twinned Magnesium Alloy

Wei Wu¹ and Ke An^{1,*}

1. Chemical and Engineering Materials Division, Oak Ridge National Laboratory, Oak Ridge,
TN 37831, USA

***Corresponding authors**

Ke An, Ph.D.
Lead Scientist
Chemical and Engineering Materials Division
Oak Ridge National Laboratory
1 Bethel Valley Rd
Oak Ridge
TN 37831, USA
Office: +1-865-919-5226
Fax: +1-865-574-6080
E-mail: kean@ornl.gov

Abstract:

The mechanisms of fatigue life improvement by pre-twinning process in a commercial rolled magnesium (Mg) alloy have been investigated using real-time in situ neutron diffraction under a continuous-loading condition. It is found that by introducing the excess twinned grains through pre-compression along the rolling direction the fatigue life was enhanced approximately 50%, mainly resulting from the prolonged detwinning process and inhibited dislocation slip during reverse tension. Moreover, after pre-twinning, the reduction of the rapid strain hardening during reverse tension leads to a compressive mean stress value and more symmetric shape of stress-strain hysteresis loop. The pre-twinning has significant impacts on the twinning-detwinning characteristics and deformation modes during cyclic loading and greatly facilitates the twinning-detwinning activities in plastic deformation. The cyclic straining leads to the increase of contribution of tensile twinning deformation in overall plastic deformation in both the as-received and pre-twinned sample. The mechanisms of load partitioning in different groups of grains are closely related to the deformation modes in each deformation stage, while the fatigue cycling has little influence on the load sharing. The pre-twinning process provides an easy and cost-effective route to improve the low-cycle fatigue life through manufacturing and processing, which would advance the wide application of light-weight wrought Mg alloys as structural materials.

Keywords: Magnesium alloy; Mechanical properties; Anisotropy; Neutron diffraction

1. Introduction

The development of lightweight, high strength, and cost-effective materials is essential for the energy-efficient vehicles, which will reduce the weight of automobile, consumption of fossil fuel, and green-house gas emission, without compromising the performance and reliability [1-3]. The wrought magnesium (Mg) alloy is a perfect candidate to meet those criteria. As load bearing components, the materials suffer from cyclic loading, which could result in the catastrophic failure during the service. Therefore, the improvement of fatigue properties in currently commercially available Mg alloys is certainly worth the effort of researchers to promote the wide application of Mg alloys as structural materials [4-6]. There are many ways to enhance mechanical properties in Mg alloys, including the rare earth element addition [7-10], optimizing the microstructures [11-18], and controlling the initial texture [19-21], and so on. It can be achieved by metallurgy, heat-treatment, manufacturing and processing (rolling, extrusion, drawing, bending, and stretching), etc.. In previous studies [22-27], it has been reported that the alternate $\{10.2\}$ twinning and detwinning processes are often involved in highly textured wrought Mg alloys during cyclic straining, which can be influenced by chemical composition, microstructure, and initial texture, et al.. Among them, manipulating the initial texture through pre-twinning can be easily and cost-effectively accomplished by manufacturing and processing, which will efficaciously modify the low-cycle fatigue properties of wrought Mg alloys.

The hexagonal close-packed (HCP) structured Mg has two easy deformation modes, namely, basal $\langle a \rangle$ slip $\{00.2\}\langle 11.0 \rangle$ and tensile twinning $\{10.2\}\langle 10.1 \rangle$ [28, 29]. The $\{10.2\}$ tensile twinning can be activated by tension parallel to the c-axis or compression perpendicular to the c-axis, which leads to a sudden reorientation of lattice approximately 86.3° [22, 23]. The

detwinning process can be initiated during the stress- or strain-reversal in the re-orientated lattice in the twinned region. The comprehensive studies [20, 22-24, 26, 30-43] have been conducted in the past two decades to characterize the twinning and/or detwinning process in HCP-structured crystalline materials during plastic deformation, strain-path changes, and cyclic loading.

The asymmetric shape of the stress-strain hysteresis loop is usually observed in wrought Mg alloys during fully reversed low-cycle fatigue when the total strain amplitude is larger than ± 0.005 [39]. In our previous study [26], the low-cycle fatigue behavior of a rolled AZ31 Mg alloy has been investigated using real-time in situ neutron diffraction under continuously loading condition at total strain amplitude ± 0.02 , at room temperature. It was found that the asymmetry of the stress-strain hysteresis loop was attributed to the early exhaustion of the detwinning process, due to the insufficient twinned grains. It is expected that pre-twinning will provide a large amount of twinned grains that would change the twinning and detwinning characteristics during cyclic loading, and therefore affect the low-cycle fatigue life. Hitherto, the research regarding the effect of pre-twinning on low-cycle fatigue behavior of wrought Mg alloys is sparse. It has been demonstrated that pre-twinning by pre-tension in “ND” sample (loading direction parallel to ND of the rolling plate) derogated the low-cycle fatigue life, because of the development of the tensile mean stress and ductility loss caused by the pre-tension [44]. On the contrary, the pre-twinning by pre-compression in “RD” sample (loading direction parallel to RD) enhanced the fatigue life by reducing the mean stress [45, 46]. Thus, the pre-twinning process could have either positive or negative impact on the low-cycle fatigue properties, which highly depends on the initial texture, loading path, and sample orientation.

Stemming from the lack of direct in situ observation, the effect of pre-twinning on the deformation mechanisms during low-cycle fatigue remains unclear, such as 1) what is the reason

for fatigue life improvement after pre-twinning from a microscopic point of view, 2) how the twinning and detwinning activities vary after the pre-twinning, and 3) what controls the load partitioning in different groups of grains during deformation? The current paper concentrates on investigation of the above-mentioned three subjects by using real-time in situ neutron diffraction method under a continuous-loading condition to provide the first-hand evidence during cyclic straining.

2. Material and Methods

2.1 Experimental materials

A commercial AZ31B Mg alloy after rolling (a nominal composition of 3.0 % Al, 1.0 % Zn, and Mg as balance, in weight percentage) with typical rolling texture was selected in the current study. The initial texture of the rolling plate was published elsewhere [24]. The low-cycle fatigue samples were machined according to the American Society for Testing and Materials (ASTM) Standard E606-04 in a cylindrical dog-bone shape, with 8 mm diameter and 16 mm gage length. The axial direction of fatigue specimens was parallel to the rolling direction (RD) of the rolling plate. An arrow was marked on the end of each specimen to indicate the normal direction (ND) of the rolling plate in the radial direction. Before the mechanical loading, all specimens were annealed at 345 °C for 2 hours to release the residual stress generated from the manufacturing and sample machining. The microstructures of annealed AZ31B rolled Mg alloy in transverse direction (TD)-ND and TD-RD plans are demonstrated in Fig. S1(a) and (b), respectively. The average grain size of annealed sample was approximately 40 μm .

2.2 Mechanical testing

The fully reversed low-cycle fatigue experiments were conducted at a total strain of ± 0.02 , at room temperature with triangular loading waveforms and 1 Hz frequency. It is worth mentioning that the fatigue tests were started from compression, unlike the conventional low-cycle fatigue tests starting from tension, in order to initiate the twinning dominated deformation at the beginning. The constant strain rate, $3.33 \times 10^{-5} \text{ s}^{-1}$ was applied during neutron diffraction measurements on one sample, designed as “As-received sample”. The 1st, 2nd, 5th, 10th, 20th, 50th, and 80th cycles were chosen for the real-time in situ neutron diffraction measurements.

Another sample was pre-compressed to -0.04 strain under a constant strain rate, $2.22 \times 10^{-5} \text{ s}^{-1}$, and then unloaded to 0 N. Then the same experiment scheme as the as-received sample was applied, except the strain rate was changed to $1.11 \times 10^{-5} \text{ s}^{-1}$ and the neutron diffraction measurements were performed in 1st, 2nd, 5th, 10th, 20th, 30th, 40th, 50th, 60th, 70th, 80th, 90th, 100th, 110th, 120th, and 130th cycles.

2.3 Neutron diffraction measurements

The real-time in-situ neutron diffraction measurements were conducted using the recently-developed state-of-the-art VULCAN Engineering Diffractometer at the Spallation Neutron Source (SNS) of the Oak Ridge National Laboratory (ORNL) under a continuous loading condition [47-49]. The detailed experiment setup has been described in detail elsewhere [25, 26, 38, 50, 51]. Only a brief description is provided here. The VULCAN MTS loadframe was laid horizontally. The specimen was aligned with loading direction parallel to RD and the horizontal-radial direction along ND. The incident beam was 45° to the sample. The two detector banks were located at $\pm 90^\circ$ to the incoming neutron beam. One detector bank recorded the diffraction patterns in axial direction, and the other received diffraction patterns in horizontal-radial direction, during the mechanical loading. The neutron beam slits was defined as $5 \text{ mm} \times 5 \text{ mm}$, with 5 mm receiving collimators. The high intensity mode and a 20 Hz chopper speed were chosen for the as-received sample, while 30 Hz chopper speed was selected for the pre-twinned sample.

The event-based data reduction software, VULCAN Data Reduction and Interactive Visualization software (VDRIVE) was used for data reduction after neutron diffraction measurements [52]. In the present paper, the binning time of 120 and 90 s were selected for as-

received and pre-twinned sample, respectively, which are the shortest time bins without compromising the data quality for single peak fitting. In the meantime, the averaged loadframe information over the binning time, such as displacement, force, stress, and strain, were synchronized with the “chopped” diffraction patterns. To eliminate the neutron beam fluctuation during the measurement, the diffraction patterns were normalized to the incoming beam energy. Moreover, the instrument background was subtracted from the diffraction patterns for the normalization of diffraction peak intensity. The lattice strain of certain hkl , ε^{hkl} , was calculated using the well-known equation as follows,

$$\varepsilon^{hkl} = \frac{d^{hkl} - d_0^{hkl}}{d_0^{hkl}} \quad (1)$$

where d_0^{hkl} is the reference d-spacing before deformation and d^{hkl} is the d-spacing after deformation. The reference d-spacing, d_0^{hkl} , was measured for 500 and 1500 s, which are much longer than the binning time, for the as-received and pre-twinned sample, respectively, to diminish the propagated d_0^{hkl} statistical error. Since the rolled Mg samples are highly textured, some of hkl s cannot be detected or have very low peak intensities for the reference measurements, leading to a bad fitting of d_0^{hkl} from the single peak fitting. After deformation, the missing peaks will experience the appearing-disappearing sequence due to the twinning-detwinning process. By using the inappropriate d_0^{hkl} , the fake lattice strain information will be attained. To solve this problem, the lattice parameters, a and c , were obtained from the whole pattern fitting using Rietveld method in axial and radial direction separately in both the as-received and pre-twinned sample. Then, the d_0^{hkl} in the missing hkl s was calculated and used for the lattice strain calculation.

3. Results

3.1 Low-cycle fatigue behavior

The stress-strain hysteresis loops of the as-received sample in the selected fatigue cycles are presented in Fig. 1(a). The asymmetric shape of the stress-strain hysteresis loops was observed throughout fatigue life in the as-received sample. The stress-strain hysteresis loop of the 1st cycle with a sigmoidal shape (or S-shape) was obviously different from the other cycles with a concave-up shape, during reverse tension. Since the 5th cycle, the stress-strain curves in the tension were almost overlapping. A slight strain hardening was observed in each fatigue cycle during compression, which increases after the 50th cycle.

The mechanical stress-strain responses of the pre-twinned sample in the chosen fatigue cycles are displayed in Fig. 1(b). It is noticed that the stress-strain hysteresis loops of the pre-twinned sample were relatively symmetric in shape during cyclic loading. The shapes of the hysteresis loops were quite similar during fatigue testing, except that the area of the hysteresis loops increased with the fatigue cycles, indicating the strain hardening behavior. Specifically, the area of hysteresis loops changed apparently in the first 5 cycles, and then became relatively stable. A relative rapid strain hardening behavior was observed by the end of tension and compression since the 50th cycle. The fatigue life of the pre-twinned sample was extended to 131 cycles, comparing with 87 cycles in the as-received sample.

The variation of tensile and compressive peak stress as well as the mean stress as a function of number of cycles for both the as-received and pre-twinned samples are presented in Fig. 1(c) and (d). The tensile peak stress of the as-received sample marginally increased in the first 5 cycles, and then saturated in the following fatigue cycles, while the compressive peak

stress kept increasing throughout the fatigue life. The compressive stress in the first 10 cycles increased faster than after the 10th cycle. The tensile and compressive peak stresses of pre-twinned sample increased constantly in the entire fatigue life. It is noticed that in the first 10 cycles and after the 40 cycles, the tensile and compressive stress increased relatively faster than cycles between the 10th and 40th cycle. After pre-twinning, the tensile peak stress was apparently decreased, while the compressive peak stress was slightly increased. It resulted in the compressive mean stress in the pre-twinned sample, which is thought to be the main reason for the prolonged fatigue life, comparing with the tensile mean stress in the as-received sample, as demonstrated in Fig. 1 (d). It was also found that the mean stress of the as-received sample stayed unchanged in the first 4 cycles, and then reduced rapidly from the 5th to the 10th cycle, followed by a gradual decrease until the sample failure. In the pre-twinned sample, the mean stress increased slightly in the first 10 cycles, and then remained unchanged.

3.2 Microscopic responses

In our previous studies [25, 26, 38], it has been demonstrated that the real-time in situ neutron diffraction was powerful to investigate the deformation dynamics and mechanisms during strain-path changes and cyclic loading in a rolled AZ31B Mg alloy. In the present research, the same methodology was adopted to characterize the effect of pre-twinning on deformation mechanisms during low-cycle fatigue.

The diffraction peak intensity evolutions of certain *hkl*s in the loading direction in the selected cycles for the as-received and pre-twinned samples are illustrated in Fig. 2(a) and (b), respectively. The twinning and detwinning processes were perceived during cyclic loading in each selected fatigue cycle in both as-received and pre-twinned samples, such as the diffraction

peak intensity of (00.2) grains experienced an increasing-decreasing sequence accompanying with decreasing-increasing of the (10.0) diffraction peak intensity in the same detector bank (for the detailed description of twinning-detwinning characteristics during cyclic loading based on neutron diffraction results, refer to [26]). It was found that the maximum value of (00.2) peak intensity at -0.02 strain kept unchanged until the 10th cycle. After that, the maximum value of diffraction peak intensity of (00.2) grains increased in each cycle, suggesting the twinning activities keep enhancing with the cyclic loading. Moreover, the minimum value of (00.2) diffraction peak intensity always went back to 0 in the first 10 cycles, implying the full detwinning occurred. However, in the 50th and 80th cycles, the diffraction peak intensity of (00.2) grains at $+0.02$ strain was higher than 0, indicating the formation of residual twins. While the neutron diffraction measurements were being performed under a continuous-loading condition and for the pre-twinned sample, there was a neutron beam loss from the 2nd to 10th cycles, resulting in the data missing in that period of time, as shown in Fig. 2(b). However, this does not prevent us from making a reasonable understanding that the general trend of diffraction peak intensity in certain *hkl*s. It was similar to the as-received sample, such as, during compression the twin volume fraction increased with the fatigue cycles. Nevertheless, the diffraction peak intensity of (00.2) grains at $+0.02$ strain remained almost the same value, much higher than 0, in the entire fatigue life. It suggests that the twinned grains, owing to the pre-twinning process, were abundant enough for the detwinning process during reverse tension.

The diffraction peak intensity evolution of (10.0), (00.2), and (11.0) grains at the maximum and minimum strains for both the as-received and pre-twinned samples are presented in Fig. 2(c) and (d). The diffraction peak intensity of (00.2) grains at minimum strain began to increase after the 5th cycle for the as-received sample, as shown in Fig. 2(c). In the meantime, the (10.0) peak

intensity reduced slightly after the 5th cycle, and the (11.0) peak intensity decreased continuously. It suggests that the twinning activities were enhanced with the increase of fatigue cycles. On the other hand, the diffraction peak intensity of (00.2) grains at maximum strain was higher than 0 since the 50th cycle, as a result of the appearance of the residual twins. It was noticed that the (11.0) diffraction peak intensities at the maximum strain reduced after the 10th cycle, stemming from that the combination of the incomplete detwinning process, involvement of the dislocation slips, and the grain rotation during deformation. The general trend of diffraction peak intensities of certain *hkl*s at the maximum and minimum strains for the pre-twinned sample was comparable for the as-received sample. The differences lay in that the diffraction peak intensity of (00.2) grain at the maximum strain was much higher than 0 and kept almost unchanged. It is interesting to note that the diffraction peak intensity of (10.0) grains at the maximum strain kept increasing since the 60th cycle, while the (11.0) peak intensity decreased, which is thought of resulting from the grain rotation. In our previous studies [25,38], it was found that because the macroscopic stress dropped rapidly in the elastic region during reverse loading, it could introduce the complex internal stress state in some localized regions and favor the grain rotation. The similar phenomenon was also reported in NiTi shape memory alloy [53].

The diffraction peak intensities of the (00.2), (10.0), and (11.0) grains in the loading direction as a function of engineering stress during tension and compression in each selected fatigue cycle for both as-received and pre-twinned samples are demonstrated in Fig. 3(a)-(l). The capitalized letters, “D”, “DT”, “T”, and “TR”, represents dislocation, detwinning, twinning, and transition from twinning during compression (or detwinning during tension) to dislocation, respectively, which are used throughout the current paper. In tension, the plastic deformation experienced

detwinning – transition from detwinning to dislocation – dislocation dominated deformation in the as-received sample in the 1st cycle, as indicated in Fig. 3(a), (c), and (e), resulting in a sigmoidal shape of the stress-strain curve. Since the 2nd cycle, the detwinning process and transition region were prolonged and the dislocation dominated deformation disappeared. It is worth to mention that the diffraction peak intensities of different *hkl*s almost stopped changing by the end of tension deformation. It suggests that the detwinning process was exhausted and dislocation slips ruled over. The activation of dislocation slips is responsible for a rapid strain hardening in the stress-strain curve. On the contrary, for the pre-twinned sample, the detwinning dominated deformation modes governed the entire plastic deformation region in the 1st cycle, as shown in Fig. 3(b), (d), and (f). Although the transition from detwinning to dislocation dominated deformation mode was witnessed since the 20th cycle, it is obvious that the detwinning process was still dominating accompanied by the limited dislocation slip activities. It is intriguing that although there were sufficient twinned grains for the detwinning process in the pre-twinned sample as displayed in Fig. 2(b), the dislocation slip was activated anyway during the late stage of compression, which is the main reason for the relative rapid hardening behavior. In compression, the plastic deformation was governed solely by twinning dominated deformation in the 1st cycle for the as-received sample, and the transition from twinning to dislocation dominated deformation region was not identified until the 50th cycle, as displayed in Fig. 3 (g), (i), and (k). For the pre-twinned sample, the twinning – transition from twinning to dislocation dominated deformation were detected in the entire fatigue life in Fig. 3(h), (j), and (l). The slope of the diffraction peak intensity vs. engineering stress curves in the 50th and 80th cycles in transition region was still very steep in the as-received sample, suggesting the combination of twinning dominated deformation and the limited dislocation slips, simply because there were

enough parent grains suitable for tensile twinning process under compressive loading condition. However, because the pre-twinning process consumed a lot of parent grains, when the further compression (-0.04 strain) was applied to the sample, the tensile twinning process tended to be exhausted and the dislocation slips became more and more active, as a result of which the diffraction peak intensity vs. engineering stress curves in transition region in the pre-twinned sample became more and more flat with the increase of the fatigue cycles. The developed dislocation slip activities in the transition region accounted for the more intense strain hardening behavior during compression in the pre-twinned sample than the as-received sample.

The lattice strain evolutions of certain interested *hkl*s in the 1st cycle in the as-received and pre-twinned samples are illustrated in Fig. 4(a)-(f). It is noticed that the lattice strains of *hkl*s are not originated from 0 in Fig. 4(b), because the residual strain was built up from the pre-twinning process. During the twinning dominated deformation and in the axial direction in the 1st compression, the (11.0) grains tended to carry more loads than other grains and the (00.2) twin grains had a relative small lattice strain in the as-received sample [Fig. 4(a)], while the (00.2) twin grains shared more loads in the pre-twinned sample [Fig. 4(b)]. During the transition from twinning to dislocation dominated deformation in pre-twinned sample [Fig. 4(b)], the (00.2) twin grains had a tendency to convey more loads, while the (11.0) grains began to yield. It should be pointed out although the lattice strain in (11.0) parent grains was higher than the (00.2) twinned grains in the axial direction in the pre-twinned sample, the population of (00.2) grains is much higher than other orientation grains, due to the pre-twinning process, as displayed in Fig. 2(b). During the reverse tension, the plastic deformation was dominated by detwinning, transition from detwinning to dislocation, and dislocation slip, sequentially, in the as-received sample. The lattice strain responses in different groups of grains in each deformation stage are dissimilar in

the axial direction in Fig. 4(c), specifically: i) during detwinning dominated deformation [the (10.0) and (11.0) peak intensity increased and (00.2) peak intensity decreased], the (10.0) and (11.0) grains shared more load than other orientation grains, especially the (11.0) grains, and (00.2) grains yielded immediately; ii) during transition region [the (10.0) and (11.0) peak intensity recovered and (00.2) peak gradually disappeared], the lattice strain of (10.0) and (11.0) grains increased rapidly; and iii) during dislocation dominated deformation [diffraction peak intensities cease changing], the (11.0) grains started to yield and the (10.0) and (10.1) grains received more load. Since the detwinning deformation mode governed the plastic deformation during reverse tension in the pre-twinned sample, the lattice strain evolution in pre-twinned sample in Fig. 4 (d) was very much alike the lattice strain variation in as-received sample during detwinning dominated deformation region in Fig.4(c). During compression, the general trend of lattice strain evolution in the as-received and pre-twinned samples in Figs. 4(e) and (f) were identical to the 1st compression in Figs. 4(a) and (b), respectively.

The lattice strain variations of different groups of grains in both the axial and radial direction are demonstrated in Fig. 4(g)-(j) for both the as-received and pre-twinned samples in the 50th cycle. During the detwinning dominated deformation in the tension, the lattice strain in the (11.0) grains increased rapidly and the (00.2) grains stopped changing in the axial direction in the as-received sample, as displayed in Fig. 4(g). In the transition from detwinning to dislocation dominated deformation region, it is obvious that the loads were transferred from (11.0) to (10.0) grains. The overall trend of the pre-twinned sample in Fig. 4(h) is similar to the as-received sample, except that the (00.2) lattice strain increased linearly during tension. During compression and in the twinning dominated deformation region, the (11.0) lattice strain increased significantly and the (00.2) lattice strain increased slowly, as presented in Fig. 4(i). When the sample was

further compressed, the loads were transferred from (10.0) and (11.0) grains to the (00.2) grains. It is apparent in Fig. 4(j) that in the pre-twinned sample the (00.2) twin grains yielded immediately upon the twinning dominated deformation and began to share more loads in the transition region.

4. Discussion

4.1 Deformation mechanisms

The evolutions of the peak stress, 0.2 proof stress, stress at the dividing point between twinning/detwinning and the transition region, and deformation modes during cyclic loading in the as-received and pre-twinned samples are elaborated in Fig. 5(a) and (b), respectively. The R1 (region 1), R2, R3, and R4 represent twinning, transition from twinning to dislocation, detwinning, and transition from detwinning to dislocation dominated deformation modes, and are filled with meshed patterns in color blue, purple, red, and green, respectively. During compression, the plastic deformation of the as-received sample was controlled by the twinning process, as shown in Fig. 5(a). Moreover, with the increase of fatigue cycle, the area of R1 became wider and wider, implying that the twin volume fraction increased over the fatigue cycles. The transition from twinning to dislocation dominated deformation region, R2, was distinguished since the 20th cycle and had a tendency to expand over fatigue cycles in Fig. 5(a), but the tensile twinning still ruled over as exhibited in Fig. 3(g), (i), and (k). After pre-twinning, the range of R1 remained almost unchanged in Fig. 5(b), signifying a stable tensile twinning process in the pre-twinned sample in the entire fatigue life. Moreover, the transition region, R2, was identified in the 1st cycle, mainly because the parent grains were fully twinned during cyclic loading after the pre-compression. In the transition region of the pre-twinned sample, the tensile twinning process was almost exhausted and the dislocation slip became the dominant deformation mode, as shown in Fig. 3(h), (j), and (l). The range of the transition region stayed nearly constant until the 20th cycle, after that, transition region grew wider and wider.

During reverse-tension, the region of detwinning dominated deformation, R3, in the as-received sample increased significantly from the 1st to the 2nd cycle and enhanced faintly over

the fatigue cycles in Fig. 5(a). The dislocation dominated deformation only appeared in the 1st cycle. In the transition from detwinning to dislocation dominated deformation region, R4, the detwinning process was almost completed and dislocation slip turned out to be more and more active in the as-received sample in Fig. 3(a), (c), and (e). The range of transition region changed little over the cyclic straining in Fig. 5(a). It is noted that approximately 45% tensile peak stress came from the transition region. In the pre-twinned sample, the plastic deformation during tension was mainly determined by detwinning dominated deformation mode in Fig. 5(b). The transition region, R4, did not appear until the 10th cycle and continuously enlarged over the fatigue cycling. In the transition region, between detwinning and dislocation slip, the former was still in predominant position, as displayed in Fig. 3(b), (d), and (f).

Therefore, deformation mechanisms have been greatly influenced by pre-twinning process, including: 1) stabilizing the tensile twinning dominated deformation, 2) promoting the dislocation slips in the transition region during compression, 3) facilitating the detwinning process during reverse tension, and 4) restraining the dislocation slips during tension. All of these can be considered to be the contributors to the improvement of fatigue life in the pre-twinned sample.

The shape of the stress-strain hysteresis loop was modified after the pre-twinning process, as displayed in Fig. 1(a) and (b). In our previous study [26], it is claimed that the asymmetry of the stress-strain hysteresis loop results from the early exhaustion of the detwinning process. In the current study, the same characteristic was observed in the as-received sample in Fig. 1(a) with the tensile peak stress much higher than the compressive peak stress in Fig. 1(c). However, after pre-twinning, the shape of the stress-strain hysteresis loop in the pre-twinned sample became relatively symmetric. As mentioned in 4.1, the detwinning process governed the plastic

deformation and dislocation slip in transition region was significantly inhibited during reverse tension in the entire fatigue in the pre-twinned sample. It results in the elimination of the rapid strain hardening, which is thought of the main reason for the relative symmetric shape of the hysteresis loop. It is reasonable to believe that the symmetry of the stress-strain hysteresis loop can be further adjusted by manipulating the pre-twinning process, i.e., pre-compression to a smaller strain, less than -0.04 in the current paper. The more systematic and sophisticated researches on the effect of pre-twinning on low-cycle fatigue behavior of wrought Mg alloys will be carried out.

To better understand the effect of twinning and detwinning process on low-cycle fatigue behavior in a rolled Mg alloy with and without pre-twinning, it is vital to distinguish contribution of tensile twinning on plastic deformation over the fatigue cycles. In previous study [35], the percentage of the tensile twinning deformation in overall plastic deformation was estimated using Eq. 2, as follows,

$$\frac{\Delta\epsilon_{twin}}{\Delta\epsilon_{plastic}} = \bar{m}S \frac{\Delta\nu}{\Delta\epsilon_{plastic}} \quad (2)$$

where $\frac{\Delta\epsilon_{twin}}{\Delta\epsilon_{plastic}}$ is the percentage of the tensile twinning deformation in overall plastic

deformation, $\bar{m}$ is averaged orientation factor, S is the characteristic shear of the twin system,

and $\frac{\Delta\nu}{\Delta\epsilon_{plastic}}$ is the slope of the normalized intensity vs. plastic strain curve. In the current paper,

the $\bar{m}$ is 0.3 by assuming that averaged orientation factor does not change during deformation

and S is 0.131 for the tensile twinning in Mg. The normalized diffraction peak intensities of the

(00.2) grains in the axial direction as a function of plastic strain during tension and compression

in the as-received and pre-twinned samples are presented in supplementary Fig. S2. It should be clarified that the slope of the normalized intensity vs. plastic strain curve was calculated only from the portion of twinning/detwinning dominated deformation in each cycle and no transition region was included for the data fitting. The estimated $\frac{\Delta\epsilon_{twin}}{\Delta\epsilon_{plastic}}$ in the twinning and detwinning dominated deformation regions as a function of number of cycles for as-received and pre-twinned samples is displayed in Fig. 6. For both the as-received and pre-twinned samples, the percentage of the tensile twinning deformation in overall plastic deformation increased with the increase of fatigue cycles. After the pre-twinning, the percentage of the tensile twinning deformation in overall plastic deformation increased about 40~50%, stemming from that more twinning and detwinning activities during deformation in the pre-twinned sample than the as-received sample. It is interesting to note the general trends of the percentage increase over fatigue cycles in the as-received and pre-twinned sample were in the same fashion, suggesting that the rate of percentage increase with fatigue cycles mainly came from materials.

4.2 Load partitioning

It is noticed that the lattice strain evolution in the pre-twinned sample is very similar with the as-received sample in the 50th cycle. Overall, in both the as-received and pre-twinned samples during the twinning/detwinning dominated deformation, the (00.2) twinned grains in the axial direction tended to yield, while the lattice strain in (11.0) parent grains was built up rapidly. During transition from twinning to dislocation dominated deformation region, the loads were transferred from the (10.0) and (11.0) parent grains to (00.2) twinned grains. The population of (10.0) and (11.0) parent grains is very low due to the tensile twinning process, while the (00.2) twinned grains turned out to be the majority in the axial direction. During the transition from the

detwinning to the dislocation-dominated deformation, the (11.0) detwinned grains yielded accompanying with the lattice strain increased in the (10.0) detwinned grains. In the dislocation dominated deformation region, the loads were transferred from (11.0) grains to the (10.0) and (10.1) grains. Subsequently, it is obvious that the load partitioning in different groups of grains are closely correlated with the deformation mode changes.

The lattice strain evolutions of certain interested *hkl*s in the fatigue cycle right close to the sample failure are exhibited in supplementary Fig. S3(a)-(d) for both the as-received and pre-twinned samples. It is worth mentioning that the general trend of the lattice strain evolution in different groups of grains by the end of fatigue life is analogous to in the 50th cycle, suggesting that the fatigue cycling has little impact on the loading sharing mechanisms in different *hkl*s during deformation.

4.3 The improvement of low-cycle fatigue life by pre-twinning

It has been demonstrated that the shape of stress-strain hysteresis loops became more symmetric in pre-twinned sample in Fig. 1(b) than the as-received sample in Fig. 1(a). Moreover, the tensile peak stresses were significantly reduced and the compressive peak stresses were slightly increased after the pre-twinning in Fig. 1(c), which leads to the development of the compressive mean stress in the pre-twinned sample in Fig. 1(d). In the previous macroscopic mechanical behavior studies [45, 46], the prolonged fatigue life after pre-twinning was attributed to the reduction of the mean stress in the current experimental configuration (loading along RD), which agrees very well with experimental observation in the present research. Moreover, it was concluded that the pre-twinning promoted the detwinning process, which essentially altered the twinning-detwinning characteristics during cyclic loading [46]. Later, it was verified by optical

microscopy observation after fatigue failure that the residual twins in the pre-twinned samples were less than in the as-received sample, indicating the detwinning process was stimulated [45]. One step further, in the present study, the deformation mechanisms were characterized in the selected fatigue cycles during cyclic straining through the direct in situ observation by neutron diffraction method, which provides the in-depth understanding of the fatigue life improvement by pre-twinning.

Besides the macroscopic mechanical behavior, the in situ microscopic response acquired by the neutron diffraction provides the insight of the fatigue life improvement mechanisms. After the pre-twinning process by compression along RD in the current study, a large amount of twinned grains were formed as demonstrated in Fig. 2(b), which essentially altered the initial texture of the pre-twinned sample comparing with the as-received sample before the low-cycle fatigue testing. The diffraction peak intensity of (00.2) grains was always higher than 0 after reverse tension to the maximum strain, + 0.02, in the entire fatigue life in Fig. 2(b), indicating that there are enough twinned grains generated from the pre-twinning to aid the detwinning process. It is obvious that the detwinning dominated deformation controlled the plastic deformation of the pre-twinned sample during reverse tension in Fig. 5(b). Although the transition from the detwinning to dislocation dominated region was perceived since the 10th cycle in the pre-twinned sample, the detwinning was still predominant in the transition region as illustrated in Fig. 3(b), (d), and (f). It resulted in only a limited strain hardening in the stress-strain curve in Fig. 1(b), signifying that the dislocation slip was activated but not comparable with detwinning. It is thought that the rapid strain hardening during reverse tension in the as-received sample is harmful for the fatigue life, since it would result in the stress concentration at weak locations of the specimen and facilitating the formation of fatigue crack. It is believed that

the removal of strain hardening region in tension by prolonging the detwinning process is one of the main reasons for the prolonged fatigue life in the pre-twinned sample. It also can be used to explain the fatigue life reduction in ND samples (the loading direction parallel to the ND) after pre-twinning process under the fully-reversed low-cycle fatigue, as shown in previous research [45]. The low-cycle fatigue behavior of the ND sample has a different twinning-detwinning sequence compared with the RD sample, i.e. the twinning-detwinning process occurs during tension-compression in ND sample and compression-tension in RD sample, respectively [20]. It results in the rapid strain hardening occurs during compression in ND sample, owing to the early exhaustion of the detwinning process and the activation of dislocation slips [30]. Based on the experimental observation in Fig. 5, it can be deduced that the rapid strain hardening during compression in ND sample would be eliminated after the pre-twinning process because the detwinning process was sustained by the sufficient twinned grains, while the work hardening would appear in tension that is thought to be the main reason for the fatigue life reduction. In addition, the higher compressive peak stress was perceived in the pre-twinned sample than in the as-received sample after the pre-twinning in the current study, which usually is beneficial for the fatigue life by facilitating the crack closure.

5. Conclusions

The current paper aims at revealing the low-cycle fatigue life improvement mechanisms after the pre-twinning process using real-time in situ neutron diffraction under continuous loading condition. The major conclusions can be drawn as follows:

- (1) By promoting detwinning process and inhibiting the dislocation slip during reverse-tension through pre-twinning process, the fatigue life has been significantly improved, which is also responsible for the development of compressive mean stress and the symmetric shape stress-strain hysteresis loop in pre-twinned sample relatively.
- (2) The deformation mechanisms in each deformation stage during cyclic loading were significantly influenced by pre-twinning.
- (3) The pre-twinning process significantly improved the percentage of tensile twinning deformation in overall plastic deformation. The contribution of tensile twinning deformation in overall plastic deformation increased with fatigue cycles constantly for both the as-received and pre-twinned sample.
- (4) The load partitioning in different groups of grains was closely correlated with the deformation modes. The fatigue cycling has little influence on the load sharing mechanisms during deformation.
- (5) The pre-twinning process is straightforward and cost-effective for manufacturing the high fatigue resistance wrought Mg alloys. The systematic and sophisticated researches on the effect of pre-twinning on low-cycle fatigue behavior of wrought Mg alloys should be conducted to advance the applications as structural materials.

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Figure Captions

Figure 1. The hysteresis Loops of selected fatigue cycles for (a) the as-received sample and (b) pre-twinned sample. (c) and (d) The peak stress and mean stress as a function of fatigue cycles for both the as-received and pre-twinned samples, respectively.

Figure 2. The diffraction peak intensity evolution of certain *hkl*s in the loading direction in selected fatigue cycles for (a) the as-received sample and (b) pre-twinned sample. The diffraction peak intensity variation at the maximum and minimum strain as a function of fatigue cycles for (c) the as-received sample and (d) pre-twinned sample.

Figure 3. The diffraction peak intensity variation of certain *hkl*s as a function of engineering stress in selected fatigue cycles for as-received and pre-twinned samples, (a)-(f) during tension and (g)-(l) during compression.

Figure 4. Lattice strain evolution of certain interested *hkl*s in selected fatigue cycles during tension and compression for as-received and pre-twinned samples, (a) and (b) the 1st compression; (c)-(f) the 1st cycle; (g)-(j) the 50th cycle.

Figure 5. The peak stress, 0.2 proof stress, stresses at the dividing point between twinning/detwinning and transition region, and deformation modes as a function of number of cycles in (a) the as-received sample and (b) the pre-twinned sample.

Figure 6. The estimated $\Delta\epsilon_{\text{twin}} / \Delta\epsilon_{\text{plastic}}$ in the twinning and detwinning dominated deformation regions as a function of number of cycles for as-received and pre-twinned samples.

As-received Sample

Pre-twinned Sample

Figure 1 The hysteresis Loops of selected fatigue cycles for (a) the as-received sample and (b) pre-twinned sample. (c) and (d) The peak stress and mean stress as a function of fatigue cycles for both the as-received and pre-twinned samples, respectively.

As-received Sample, Loading direction, — (10.0) — (00.2) — (11.0)

Pre-twinned Sample, Loading Direction, — (10.0) — (00.2) — (11.0)

As-received Sample

Pre-twinned Sample

Figure 2 The diffraction peak intensity evolution of certain *hkl*s in the loading direction in selected fatigue cycles for (a) the as-received sample and (b) pre-twinned sample. The diffraction peak intensity variation at the maximum and minimum strain as a function of fatigue cycles for (c) the as-received sample and (d) pre-twinned sample.

Figure 3 The diffraction peak intensity variation of certain hkl s as a function of engineering stress in selected fatigue cycles for as-received and pre-twinned samples, (a)-(f) during tension and (g)-(l) during compression.

As-received Sample during Tension

Pre-twinned Sample during Tension

As-received Sample during Compression

Pre-twinned Sample during Compression

Figure 4 Lattice strain evolution of certain interested *hkl*s in selected fatigue cycles during tension and compression for as-received and pre-twinned samples, (a) and (b) the 1st compression; (c)-(f) the 1st cycle; (g)-(j) the 50th cycle.

As-received Sample

Pre-twinned Sample

Figure 5 The peak stress, 0.2 proof stress, stresses at the dividing point between twinning/detwinning and transition region, and deformation modes as a function of number of cycles in (a) the as-received sample and (b) the pre-twinned sample.

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