Cyclic Deformation and Fatigue Damage in Magnesium Single Crystal under Fully Reversed Strain-Controlled Tension-Compression in $[10\bar{1}1]$ Direction

Qin Yu^{1,2}, Yanyao Jiang^{1,*}, Jian Wang^{2,*}

¹Department of Mechanical Engineering, University of Nevada, Reno, Reno, NV 89557, USA

²Material Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Abstract

Cyclic deformation and the resulted fatigue are experimentally investigated in magnesium single crystal subjected to fully reversed strain-controlled tension-compression in $[10\bar{1}1]$ direction at room temperature. Plastic instability is found near the end of the first detwinning reversal. Twin-twin boundaries and secondary twins are induced by interaction of primary $\{10\bar{1}2\}$ twins. Microcracks are developed at primary twin boundaries and twin-twin interaction sites. Final fracture is **most likely** caused by cleavage-cracking on $\{10\bar{1}1\}$ - $\{10\bar{1}2\}$ double twin and shearing fracture on the basal plane.

Keywords: Magnesium single crystal, Cyclic deformation, Twinning-Detwinning, Fatigue.

* Corresponding authors: Yanyao Jiang: Phone: 1-775-784-4510, Fax: 1-775-784-1701, Email: yjiang@unr.edu; Jian Wang: Phone: 1-505-667-1238, Fax: 1-505-667-8021, Email: wangj6@lanl.gov

Hexagonal close packed (HCP) magnesium (Mg) is considered as potential weight-reduction material in aerospace and automobile industries [1-3]. Great efforts have been made to study the cyclic deformation of cast and wrought Mg alloys [4-7]. Inconsistent results of cyclic deformation behavior are present in Mg alloys owing to various microstructural influences, such as alloying composition [8], inclusion [9], and heterogeneity of grain and phase structures [10]. Inherently, due to the presence of grain boundaries and the indeterminate internal stresses, the mechanical response caused by a single deformation mode is very difficult to be characterized. Henceforth, it is valuable to study the cyclic deformation in Mg single crystal.

Limited experimental studies have been conducted to explore the cyclic deformation in HCP single crystals [11-16]. For Mg single crystal, work on cyclic plasticity was limited to crystals oriented for basal slips [13, 14]. By carrying out fully reversed strain-controlled tension-compression experiments in single crystal Mg oriented for basal slips, Stevenson and Sande [13] found that duplex basal slips are operative at high plastic strain amplitude. Kwadjo and Brown [14] characterized the dislocation configuration associated with different stages of the cyclic stress-cumulative plastic strain (CSPSC) curve for single basal slips. Work dedicated to studying the twinning-dominated cyclic deformation in Mg single crystals is very limited [15,16]. Recently, Yu et al. [15] carried out fully reversed strain-controlled tension-compression experiments to study the cyclic deformation behavior in [0001]-oriented Mg single crystal utilizing *in situ* optical microscopy (OM). Six $\{10\bar{1}2\}$ twin variants subjected to the same resolved shear stress can be activated simultaneously. The current effort aims to extend the investigation of twinning-dominated cyclic deformation in Mg single crystal loaded in the $[10\bar{1}0]$ direction. Different from the [0001]-oriented cyclic loading, two of the six $\{10\bar{1}2\}$ twin variants are subjected to a maximum resolved shear stress with a Schmid factor (SF) of 0.499, and the remaining four twin variants have a lower SF of 0.125. Apart from characterizing the cyclic stress-strain response, development of local twin structures and the resulted fatigue damage are studied by electron backscatter diffraction (EBSD) and scanning electron microscopy (SEM).

Mg single crystals were fabricated into small dog-bone shaped plate specimens having a gage length of 5 mm and a cross section of 3 mm $\times$ 3 mm. The loading axis is aligned with the $[10\bar{1}0]$ direction. The flat specimen surfaces are parallel to either the $(\bar{1}2\bar{1}0)$ plane or the $(\bar{1}2\bar{1}3)$ plane. Detailed description of specimen preparation and the experimental setup can be referred to Ref. [15]. No twins were found in the un-deformed specimen. Fully reversed strain-controlled

tension-compression experiments were carried out in ambient air at three strain amplitudes: 0.5%, 0.75%, and 1%. The strain rate was 4.0×10^{-4} /s when *in situ* OM was used to observe the specimen surface. A strain rate at 4.0×10^{-3} /s was used for the rest of the loading cycles. All the experiments started with a compressive loading reversal. In addition to *in situ* OM observation, EBSD characterization of twin structures was carried out in the specimens unloaded from the first and third compressive peaks at the strain amplitude of 0.75% and in the specimen unloaded from the 30th compressive peak at the strain amplitude of 1%. After fatigue fracture, microcracks and the macroscopic fracture surfaces were examined using SEM and EBSD.

Fig. 1 Stress-strain hysteresis loops of magnesium single crystals subjected to fully reversed tension-compression in [10-10] direction at strain amplitudes of 0.5%, 0.75%, and 1%.

Fig. 1 presents the stress-strain hysteresis loops for the Mg single crystals subjected to fully reversed tension-compression in the $[10\bar{1}0]$ direction at three strain amplitudes of 0.5%, 0.75%, and 1%, respectively. The stress-strain hysteresis loops at the strain amplitudes under investigation are associated with three featured deformation modes: twinning-dominated, detwinning-dominated, and non-basal slips-dominated accompanied by detwinning. Twinning occurs under the compressive reversal when the resolved shear stress exceeds the critical resolved shear stress (CRSS) for tension twinning. The CRSS to activate tension twin is identified from the initial compression phase. The directly measured value is 2.2~3.0 MPa, which is very close to that (~2.4 MPa) obtained in the [0001] direction [15]. Detwinning accompanies the entire sigmoidal shaped tensile loading reversal. At the inflection point of the sigmoidal curve, the strain hardening rate converts from a gradually decreasing low value to a rapidly increasing high value. The significant increase in the strain hardening rate is ascribed to the operation of non-basal slips [17-19]. By testing a notched plate under monotonic tension in the [10-10] direction, Reed-Hill and Robertson [19] observed the non-basal slip band traces on the specimen surface of Mg single crystals. It was suggested that the non-basal slip occurring under tension in [10-10] direction was of duplex prismatic type [19].

A distinctive phenomenon of plastic instability, which is not observed in the [0001]-oriented Mg single crystal, is discovered near the end of the first tensile reversal in the $[10\bar{1}0]$ -oriented Mg single crystal. To describe the phenomenon in detail, the specimen tested at $\Delta\epsilon/2 = 0.75\%$ is taken as an example (red lines in Fig. 1). Beyond the inflection point of the tensile loading

reversal, the steady plastic flow proceeds smoothly but terminates at point A (see Fig. 1, $\sigma = \sim 115$ MPa and $\varepsilon = \sim 0.5\%$) where a sudden stress drop ($|\Delta\sigma_1| = 4$ MPa) followed by a short flow plateau ($\Delta\varepsilon_1 = 0.06\%$) occurs. At the end of the plastic flow plateau, a second rapid stress drop with a significant decrease in stress ($|\Delta\sigma_2| = 30$ MPa) is observed. The second stress drop is followed by an immediate stress increment by 10 MPa and a subsequent plastic flow plateau ($\Delta\varepsilon_2 = 0.09\%$), which continues until reaching the tensile peak strain of 0.75%. The currently observed unstable plastic flow is similar to the inhomogeneous plastic deformation associated with Lüders band propagation in carbon steels [20,21] and the serration of the stress-strain curve (Portevin-Le Chatelier (PLC) effect) observed in aluminum alloys [22-24]. Unlike the high-frequency serration with small stress drop and increment (around 5 MPa in average) in the PLC effect, the magnitude of stress drop in the [10-10] Mg single crystals is rather random, varying from ~ 4 MPa to as high as ~ 30 MPa. Moreover, both the stress increment and the plastic flow plateau can be followed after a stress drop. The underlying mechanisms of plastic instability might be related to dislocation-solute interaction (or dynamic solute aging) and dislocation-dislocation interaction [20-24]. Without the presence of solutes in the pure Mg single crystals, the dislocation-solute interaction can be ruled out as a possible cause of the observed phenomenon and therefore, the dislocation-dislocation interaction can be a probable cause. Noting that such a plastic instability is not observed in the [0001]-oriented Mg single crystal near the end of the first compressive reversal [15], it is conjectured that the involved non-basal dislocations could originate from the duplex prismatic slips rather than the second-order pyramidal slips. This speculation is enlightened by the experimental findings in Mg single crystals that pyramidal slips operate under *c*-axis compression [25,26] whereas duplex prismatic slips operate in [10-10]-tension [19].

It is noticed that, different from wrought Mg alloys [6], polycrystalline pure Mg [27], and single crystal Mg oriented in [0001] direction [15, 16], significant cyclic softening is observed in the [10-10] Mg crystal during tensile loading reversals. In addition, the maximum stress upon which plastic instability occurs in the first tensile reversal is not a monotonic increasing function of the applied strain amplitude. These observed deformation features are likely associated with the dislocation-dislocation interactions that are influenced by the initial amount of tension twinning created by the preceding compressive strain. Nevertheless, the detailed mechanisms of

plastic instability occurring near the end of the initial detwinning phase in $[10\bar{1}0]$ -Mg single crystal are not clear and require further investigations.

EBSD observations of twin structures are presented in Fig. 2. All the twin structures represent a material state unloaded from the compressive peak stress where the material has been twinned. The z-direction in the figure denotes the loading axis oriented in the $[10\bar{1}0]$ direction. To facilitate discussions, T_i ($i = 1$ to 6) are used to denote the $(10\bar{1}2)$, $(01\bar{1}2)$, $(\bar{1}102)$, $(\bar{1}012)$, $(0\bar{1}12)$, and $(1\bar{1}02)$ primary twin variants, respectively. T_{ij} ($i, j = 1$ to 6) represent secondary twin T_j activated within the primary twin T_i .

Figs. 2a and 2b show the twin structures in the material unloaded from the first and third compressive peaks, respectively, at $\Delta\epsilon/2 = 0.75\%$. Two twin variants, T_1 and T_4 having a maximum SF of 0.499 and identical zone axis, are developed. In Fig. 2a, lenticular-shaped twins are observed. The boundaries of two neighboring twins having the same variant meet and coalesce (arrow A in Fig. 2a). When two twin variants meet, no twin transmission or penetration occurs but twin-twin boundaries (TTB) form (arrow B in Fig. 2a) [28, 29]. In the specimen unloaded from the third compressive peak (Fig. 2b), the number of twin lamellae and the number of TTBs are significantly increased. It is noticed that the absolute compressive peak stress increases from 9.36 MPa at the first loading cycle to 15.12 MPa at the third loading cycle (Fig. 1). The positive correlation of the increased compressive peak stress and the multiplied TTBs suggests that TTB plays an effective role in the material hardening during cyclic deformation. To be specific, the incoming twinning dislocations (TDs) can be piled up at the barrier TTB, which exerts a repulsion force on the successive impinging TDs. To further grow the twin, a large stress that overcomes the backstress at TTBs is required. Such a hardening effect is amplified as more TTBs are developed.

Fig. 2 EBSD observation of twin structures: (a) unloaded from the first compressive peak at 0.75% strain amplitude; (b) unloaded from the third compressive peak at 0.75% strain amplitude; (c) unloaded from the 30th compressive peak at 1% strain amplitude; (d) magnified area in (c) showing secondary tension twin.

Fig. 2c illustrates the twin structures developed in the material unloaded from the 30th compressive peak at $\Delta\epsilon/2 = 1\%$. All six twin variants are observed. The activation stress for T_2 , T_3 , T_5 , and T_6 is larger than that for T_1 and T_4 since the SF of T_2 , T_3 , T_5 , and T_6 is 0.125 which is

lower than that (0.499) for T_1 and T_4 . As a result, T_2 , T_3 , T_5 , and T_6 can be only activated when the material is cyclically hardened during the later stage of cyclic deformation at a larger strain amplitude, such as the loading condition ($N = 30$ cycles, $\Delta\epsilon/2 = 1\%$) in Fig. 2c. The development of all the six twin variants can lead to TTBs formation between either two co-zone twin variants (Type I) or two twin variants that do not share the same zone axis (Type II) [29]. The Type II twin-twin interaction can nucleate secondary tension twin at the TTB. A detailed analysis of the feasibility of secondary twin transmission can be found in Ref. [29]. In the current study, the observation of secondary twins can be visualized in Figs. 2c and 2d. An EBSD analysis shows that a secondary twin T_{21} is developed at the TTB formed between two primary twins T_1 and T_2 . It is established that the formation process of secondary tension twins is energetically unfavorable, which contributes to material hardening as well as the formation of TTBs [29]. To summarize, an experimental observation by EBSD in Fig. 2 shows that the twinning-associated cyclic hardening in $[10\bar{1}0]$ -oriented Mg single crystal is due to the incessant formation of TTBs and secondary twins with the increasing number of loading cycles.

Fig. 3 Microcracks and fracture surface in magnesium single crystal subjected to fully reversed tension-compression in $[10\bar{1}0]$ direction: (a) and (b) specimen surface; (c) fracture surface; (d) microcracks; (e) cleavage cracking and shearing fracture.

An examination of fatigue damage and fracture surface in $[10\bar{1}0]$ -oriented Mg single crystal is illustrated in Fig. 3. Since the specimen loaded at $\Delta\epsilon/2 = 0.75\%$ and 1% were not separated into two parts, the specimen tested at $\Delta\epsilon/2 = 0.5\%$ which was fractured into two parts is used to examine the fatigue damage. Fig. 3 shows the SEM observations on the specimen surface (Figs. 3a, b, d, and e) and the fracture surface (Fig. 3c). On the specimen surface away from the macroscopic fracture surface (Fig. 3d), microcracks are distributed mainly in two specific regions: the serrated primary twin boundaries (arrows A in Fig. 3d) and the severely fragmented twin-twin interaction sites (arrow B in Fig. 3d). The length of microcrack is very short (less than $10\text{ }\mu\text{m}$), implying that the microcrack propagation along twin boundaries and TTBs is limited. The current observation of microcrack is similar to that obtained in the $[0001]$ -oriented Mg single crystal [15].

A three dimensional profile of the macroscopic fracture surface is schematically reconstructed (middle, Fig. 3). The macroscopic fracture surface consists of multiple cleavage-

like flat facets. On the $(\bar{1}2\bar{1}0)$ specimen surface, straight markings projected from the cleavage facets form three inclination angles with respect to the $[0001]$ direction (Figs. 3a and b): -35° , 35° , and 90° . The projected straight markings coincide with the traces of the crystal planes $(30\bar{3}4)$, $(\bar{3}0\bar{3}4)$, and (0001) on the $(\bar{1}2\bar{1}0)$ plane, as confirmed by the EBSD trace analysis. The crack facets $(30\bar{3}4)$ and $(\bar{3}0\bar{3}4)$ are associated with cleavage cracking along the boundary of $\{10\bar{1}1\}$ - $\{10\bar{1}2\}$ double twin, and the crack facet (0001) is related to Mode-II shearing fracture of the basal plane. The cleavage cracking on $\{20\bar{2}4\}$ plane was first observed and studied by Reed-Hill and Robertson [30-32] in Mg single crystal subjected to monotonic tension along the $[10\bar{1}0]$ direction. The $\{30\bar{3}4\}$ cleavage cracking marking was revealed to align with the boundary of $\{10\bar{1}0\}$ - $\{10\bar{1}2\}$ double twin, where $\{10\bar{1}1\}$ primary twin experiences a secondary $\{10\bar{1}2\}$ twinning. It was further found that the formation of double twin can lead to rapid cleavage cracking at the double twin boundary. As visualized in Fig. 3e, an inclined macrocrack of $\{10\bar{1}0\}$ - $\{10\bar{1}2\}$ twin type is connected to a microcrack on the $(\bar{1}012)$ twin boundary (arrow A in Fig. 3e). A trace analysis shows that the microcrack orientation coincides with the trace of $\{\bar{1}012\}$ crystal plane. The inclined microcracks are observed to predominantly align with the tension twin boundaries (Fig.3d). Therefore, the microcrack connected to the $\{10\bar{1}0\}$ - $\{10\bar{1}2\}$ macrocrack was most likely originated from tension twin cracking. Knowing that a large CRSS is required to activate $\{10\bar{1}0\}$ - $\{10\bar{1}2\}$ double twin [33, 34], it can be inferred from Fig. 3e that the double twin where the macrocrack is located is induced by the intensified stress field at the tip of the microcrack developed at the tension twin. The cleavage cracking at double twin can easily activate basal slips in the slightly-rotated crystals surrounding the crack. The locally aggravated basal slips can lead to Mode-II shearing fracture on the basal plane, as indicated by arrows B in Fig. 3e.

The followings are the major findings obtained from the current experimental investigation of cyclic deformation and fatigue in Mg single crystal subjected to cyclic tension-compression in $[10\bar{1}0]$ direction:

1. A plastic instability phenomenon is found in the first tensile loading reversal where the initial detwinning process accompanied by non-basal slips approaches exhaustion.
2. The two twin variants having the highest Schmid factor (SF) with an identical zone axis are activated in the early stage of cyclic deformation. The other four twin variants with a much lower SF can be activated as the material is cyclically hardened during the later stage of cyclic

deformation. The development of twin-twin boundaries and secondary tension twins effectively act as a twinning-induced hardening mechanism.

3. Repeated twinning and detwinning can initiate microcracks with limited propagation at primary twin boundaries and twin-twin interaction sites.

4. Based on the trace analysis, final fracture of the material is most likely dominated by the sudden cleavage cracking on $\{10\bar{1}0\}$ - $\{10\bar{1}2\}$ double twin boundary and the shearing fracture on the basal plane.

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Figures Captions:

Fig. 1 Stress-strain hysteresis loops of magnesium single crystals subjected to fully reversed tension-compression in $[10-10]$ direction at strain amplitudes of 0.5%, 0.75%, and 1%.

Fig. 2 EBSD observation of twin structures: (a) unloaded from the first compressive peak at 0.75% strain amplitude; (b) unloaded from the third compressive peak at 0.75% strain amplitude; (c) unloaded from the 30th compressive peak at 1% strain amplitude; (d) magnified area in (c) showing secondary tension twin.

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Figure 01
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Figure 02
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Figure 03

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