Intragranular Twinning, Detwinning, and Twinning-Like Lattice Reorientation in
Magnesium Alloys

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4 **Abstract:**
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8 Deformation twinning plays a critical role on improving metals or alloys ductility,
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10 especially for hexagonal close-packed materials with low symmetry crystal structure. A
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12 rolled Mg alloy was selected as a model system to investigate the extension twinning
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14 behaviors and characteristics of parent-twin interactions by nondestructive *in situ* 3D
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16 synchrotron X-ray microbeam diffraction. Besides twinning-detwinning process, the
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18 “twinning-like” lattice reorientation process was captured within an individual grain
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20 inside a bulk material during the strain reversal. The distributions of parent, twin, and
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22 reorientated grains and sub-micron level strain variation across the twin boundary are
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24 revealed. A theoretical calculation of the lattice strain confirms that the internal strain
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26 distribution in parent and twinned grains correlates with the experimental setup, grain
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28 orientation of parent, twin, and surrounding grains, as well as the strain path changes. The
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30 study suggests a novel deformation mechanism within the hexagonal close-packed
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32 structure that cannot be determined from surface-based characterization methods.
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47 **Keywords:** Magnesium alloy; Twinning; Synchrotron X-ray; Deformation mechanism
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1. Introduction

The application of magnesium alloys as lightweight structural materials is hindered by their low formability, the lack of a mechanistic understanding of their twinning, and the detwinning behavior in a polycrystalline state. Magnesium (Mg) alloys are of particular interests among all commercial structural materials, due to their unique properties of low density (2/3 that of aluminum and 1/4 that of iron), high strength-to-weight ratio, and high specific stiffness [1, 2]. Magnesium possesses two “easy” deformation modes: the $\langle a \rangle$ slip on basal planes and the $\{10\bar{2}\}\langle 10\bar{1} \rangle$ tensile twinning [3-6]. Tensile twinning, which leads to a tensile strain parallel to the c-axis, is the focus of recent studies due to the importance and easy activation in hexagonal close-packed (HCP)-structured metals and alloys [7-14]. When the tension direction is parallel to the c-axis at room temperature, the $\langle a \rangle$ dislocation slip cannot operate, which makes the tensile twinning the only active deformation mode to accommodate the strain along the c-axis. The tensile twinning results in a 86.3° reorientation of the basal poles, and, thus, detwinning may occur in the twinned materials during reloading in the opposite direction [7, 8, 11, 15-18]. The unfavorable grains for slip can be re-orientated into a favorable orientation, because of these twinning and detwinning operations. Recently, a twinning-like lattice reorientation in a submicron Mg single crystal was reported, which resembles the conventional $\{10\bar{2}\}\langle 10\bar{1} \rangle$ tensile twinning but without a crystallographic mirror plane through *in situ* TEM [12]. It leads to a 90° lattice reorientation that provides the plastic strain but it is not simple shear. Although providing the temporal information as opposed to *ex situ* metallographic studies, *in situ* investigations such as the scanning electron microscopy (SEM) and transmission electron microscopy (TEM), or electron

backscatter diffraction (EBSD) are limited to surface or thin-film information [12, 19-22]. Critical questions that cannot be answered by such methods include: Are the twins nucleated inside a bulk sample at the “hot spots” where the resolved shear stress of twin dislocation systems maximizes? Surface-based observations, such as *in situ* EBSD found that detwinning takes place by shrinkage of the twins. Would these findings be valid inside the bulk? Answers to these questions require a non-destructive *in situ* bulk measurement on the length scales commensurate to the twin activities.

In previous studies, the *ex situ* polychromatic X-ray micro Laue diffraction with differential aperture X-ray microdiffraction (DAXM) technique was employed to characterize the deformation twinning behavior in the HCP system. The geometrically necessary dislocations (GND) were examined in T1-type tensile twinning in the pure Ti, $\{10\bar{1}2\} < \bar{1}011 >$, and surrounding grains after four-point bending, based on the reconstructed peak streak in the commercial purity titanium with a moderately strong texture, which found that the existence of a large orientation gradient in front of the twin tip and a high GND population near twin boundaries due to the pile-ups of the dislocations [23]. Another study focused on the T2-type tensile twinning, $\{11\bar{2}1\} < \bar{1}\bar{1}26 >$, in a rolled pure Ti plate sample with a moderate texture after four-point bending, which identified different types of edge GNDs in both the matrix and twin grains [24]. These *ex situ* studies did not resolve the strain distribution in the matrix and twin grains, which is essential for the theoretical-modeling work. Recently, an *in situ* measurement of a rolled AZ31 Mg alloy reported the grain orientation evolution and lattice strain distribution in the matrix and twin grains beneath the sample surface during $\{10\bar{1}2\} < \bar{1}011 >$ tensile twinning deformation [25]. However, the detwinning and/or

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4 lattice reorientation behavior during reverse loading were not explored and remained
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6 unclear.
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10 An in-depth understanding of the deformation and failure mechanisms in Mg
11 polycrystals requires a fundamental knowledge of the twinning activities. For example,
12 how the deformation twinning-detwinning operate inside a parent grain? Does the
13 twinning-like lattice reorientation exist in a bulk HCP-structured material? What is the
14 lattice strain distribution around the parent-twin interface? In this study, the tensile
15 twinning behavior within an individual grain inside a polycrystalline rolled AZ31B Mg
16 alloy was investigated *in situ* using the state-of-the-art synchrotron X-ray microbeam. A
17 special 3D DAXM technique to achieve the sub-micrometer scale is used to spatially
18 resolve the lattice orientations and strains with the focused polychromatic and
19 monochromatic X-ray, respectively. The twinning – detwinning and/or twinning-like
20 lattice reorientation processes, as driven by a macroscopic compression and tension strain
21 path, were identified by the grain orientation map during in-plane compression and
22 reverse tension. The internal strain and diffraction peak intensity variation of a certain *hkl*
23 inside an individual grain was also measured. A theoretical model was developed to
24 simulate the strain field across the parent-twin interface. The twinning-like lattice
25 reorientation was characterized for the first time using the diffraction method in a bulk
26 Mg sample and the parent-twin boundary were found to differ from surface-based
27 measurements.
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2. Experimental and Modeling Methods

2.1 Experimental materials

A commercial rolled AZ31B magnesium alloy (a nominal composition of 3.0 % Al, 1.0 % Zn, and Mg as balance, in weight percentage) in H24 temper with a typical rolling texture was chosen. A plate dog-bone specimen was prepared with its axial direction along the rolling direction (RD) and surface-normal direction parallel to the normal direction (ND) of the rolling plate. The sample gauge section was 2 mm length x 1.5 mm width x 1.5 mm thickness. After sample machining, the specimens were annealed at 345 °C for 2 hours to remove the existing residual stresses. The average grain size of the annealed AZ31B magnesium alloy varied from 20 to 100 μm .

2.2 *In situ* 3D x-ray Laue diffraction measurement

A customized loadframe was specially designed for the *in situ* synchrotron X-ray microbeam diffraction measurements. In the current study, three strain levels were chosen for the *in situ* synchrotron X-ray microbeam diffraction based on our previous experience, e.g., undeformed state, – 2.4 % compressive strain (twinning dominant deformation), and – 7% compression – relative + 3% reverse tension strain during the reverse tension (the beginning of detwinning dominant deformation). The mechanical testing was under strain control using digital image correlation method for *in situ* synchrotron X-ray microbeam diffraction measurement.

The *in situ* measurements were carried out using 3D X-ray Laue diffraction microscope at 34ID-E, Advanced Photon Source (APS), Argonne National Laboratory (ANL), USA. The 3D X-ray Laue diffraction microscope uses the synchrotron X-ray

microbeam to probe the local crystal structure, orientation and strain tensors with sub-micrometer spatial resolution, which is achieved by the DAXM technique [23, 25-28]. The schematic of *in situ* 3D-XRD is illustrated in the supplementary Fig. 1. The sample was 45° to the incoming beam and GE detector. The loading direction was marked in the coordinate X direction. The movement of a platinum wire profiler was parallel to the sample surface. For the grain orientation mapping, the white beam (the energy range from 7 – 30 keV) was applied with a 0.5 x 0.5 μm² beam size. The step size was 2 μm along the loading direction (X-axis) and sample depth direction (Y-axis), respectively. The monobeam (the fixed beam energy for a specific *hkl*) was employed for the energy scan at certain locations (the fixed locations at X-axis) with a 0.5 x 0.5 μm² beam size. The step size along the sample depth direction (Y-axis) was 1 μm. The depth along the sample-surface-normal direction equals the spatially resolved depth divided by $\sqrt{2}$, due to the 45° inclination of the sample surface normal to the incoming beam direction. In the current paper, the spatially resolved depth from the measurement is adopted. In the current study all of the Laue patterns are indexed with respect to the sample surface normal direction [Q vector of (00.1) planes in the sample surface normal direction]. The lattice strain was calculated, using the well-known equation below:

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

where ε^{hkl} is the lattice strain of a certain *hkl*, d_0^{hkl} and d^{hkl} are the d-spacings of certain *hkl* before and after deformation, respectively. The lattice parameters of an annealed powder sample ($a = 0.32037(5)$ nm and $c = 0.52003(2)$ nm) were measured at the

VULCAN engineering diffractometer, Spallation Neutron Source (SNS), ORNL, which is used to calculate d_0^{hkl} in the current study. It is worth to mention that some pixels are not automatically indexed (the white color in Fig. 3) because of the indistinct Laue patterns at these positions, but these patterns can be manually indexed.

2.3 *Ex situ* EBSD measurement

The *ex situ* EBSD measurements were conducted using the JEOL-6500 field emission scanning electron microscope at 20 kV with a 1 μm step size at the ShaRe facility, Oak Ridge National Laboratory (ORNL). The EBSD specimens were deformed at the same strain levels following the same deformation history as the *in situ* 3D x-ray Laue diffraction measurement.

2.4 Theoretical modeling

As shown in Figs. 1(b), the incident x-ray beam travels adjacent to a protruded twin grain, which can be modeled by a twin dislocation loop. The method to calculate the lattice strain, $\varepsilon_{\mathbf{k}_2-\mathbf{k}_1}$, is given below. We first present the solution of the elastic boundary value problem of a circular dislocation loop of a radius, R , lying on the (x_1, x_2) plane, $x_3 = 0$, with the center at the coordinate origin. The Burgers vector can be arbitrary, thus including both prismatic and shear components.

We follow the formulation in Mura [29] and the procedure in Gao and Larson [30] for the anisotropic elasticity, giving the displacement field as

$$u_m(\mathbf{x}') = -\int_A b_i c_{ijkl} \frac{\partial G_{mk}(\mathbf{x}, \mathbf{x}')}{\partial x_l} dA_j, \quad (2)$$

where c_{ijkl} are elastic constants, $dA_j = n_j dA$, n_j is the normal of the dislocated plane, and the integration and differentiation are conducted for $\mathbf{X}$. The Green tensor, $G_{ij}(\mathbf{x}, \mathbf{x}')$, denotes the i -th displacement component at a location, $\mathbf{X}$, caused by the j -th point force applied at $\mathbf{x}'$. The summation convention is assumed for repeated subscripts. To calculate the strain tensor, we need the displacement gradients:

$$\frac{\partial u_m(\mathbf{x}')}{\partial x'_n} = - \int_A b_i c_{ijkl} \frac{\partial}{\partial x'_n} \left[\frac{\partial G_{mk}(\mathbf{x}, \mathbf{x}')}{\partial x_l} \right] dA_j = \int_A b_i c_{ijkl} \frac{\partial^2 G_{mk}(\mathbf{x}, \mathbf{x}')}{\partial x_l \partial x_n} dA_j. \quad (3)$$

The Green tensor is the elastic displacement field in an infinite anisotropic space subjected to a point force at the coordinate origin, which can be solved by the combination of the Stroh formalism and two-dimensional Fourier transform [30, 31]. The Fourier transform converts real coordinates (x_1, x_2) to imaginary coordinates (ξ_1, ξ_2) . The Stroh formalism is used to derive the general solution in the two-dimensional plane spanned by an arbitrary wavevector, $\xi = (\xi_1, \xi_2)$, and x_3 . Boundary conditions and applied loads are used to determine the unique solution. The Green tensor in real space is [30]

$$G_{ij}(x_1, x_2, x_3) = \begin{cases} \frac{1}{4\pi^2} \iint \frac{1}{\xi} \bar{\mathbf{A}} \langle \exp(-i\xi \bar{p}_* x_3) \rangle \bar{\mathbf{A}}^{-1} (\mathbf{B}^{-1} + \bar{\mathbf{B}}^{-1})^{-1} \exp(-i\xi_1 x_1 - i\xi_2 x_2) d\xi_1 d\xi_2, & x_3 > 0 \\ \frac{1}{4\pi^2} \iint \frac{1}{\xi} \mathbf{A} \langle \exp(-i\xi p_* x_3) \rangle \mathbf{A}^{-1} (\mathbf{B}^{-1} + \bar{\mathbf{B}}^{-1})^{-1} \exp(-i\xi_1 x_1 - i\xi_2 x_2) d\xi_1 d\xi_2, & x_3 < 0 \end{cases} \quad (4)$$

Note that complex scalar, p_a , and matrices, $\mathbf{A}$ and $\mathbf{B}$, depend on elastic constants and ξ_α/ξ , which is the direction of the wavevector. A bar on top of a complex variable denotes the complex conjugate. The representation of $\langle \dots \rangle$ is

$$\langle \exp(-i\xi p_* x_3) \rangle = \begin{bmatrix} \exp(-i\xi p_1 x_3) & & \\ & \exp(-i\xi p_2 x_3) & \\ & & \exp(-i\xi p_3 x_3) \end{bmatrix}. \quad (5)$$

For the Mg single crystal, the elastic constants, $C_{11} = 59.7$, $C_{12} = 26.2$, $C_{13} = 21.7$, $C_{33} = 61.7$, and $C_{44} = 16.4$ (unit: GPa), are given in the crystallographic coordinates. A tensor transformation is first performed to obtain c_{ijkl} in the local coordinates given in Fig. 1(b). The Green tensor in Eq. (4) and the displacement gradients in Eq. (3) are obtained by linear algebraic calculations that determine p_a , $\mathbf{A}$, and $\mathbf{B}$, and then by the numerical quadrature (using an adaptive Simpson method with a quadrature tolerance of 10^{-4}). From the strain components, the lattice strain in the $\mathbf{k}_2 - \mathbf{k}_1$ direction can be easily calculated by a tensor transformation.

The incoming beam, $\mathbf{k}_1$, is in the Y-direction, and the diffracted beam, $\mathbf{k}_2$, is in the Z-direction. According to the coordinates in Fig. 1, $\varepsilon_{\mathbf{k}_2 - \mathbf{k}_1}$ is the projected strain in the sample surface normal direction. From the inset in Fig. 3, the c-axis in the parent grain is roughly in the YZ plane, and makes an angle of about $\beta \approx 29^\circ$ with respect to the Y axis, while the c-axis in the twin grain is roughly in the X axis. Therefore, the twin dislocation

plane and the YZ diffraction plane make an angle of $\tan^{-1}\left(\frac{c}{\sqrt{3}a}\right)$ in Fig. 1(b). A geometric consideration gives

$$\mathbf{k}_2 - \mathbf{k}_1 = \begin{Bmatrix} -\sin\left(\frac{\pi}{4} - \beta\right) \cos\left(\tan^{-1}\left(\frac{c}{\sqrt{3}a}\right)\right) \\ -\cos\left(\frac{\pi}{4} - \beta\right) \\ -\sin\left(\frac{\pi}{4} - \beta\right) \sin\left(\tan^{-1}\left(\frac{c}{\sqrt{3}a}\right)\right) \end{Bmatrix}, \quad \mathbf{k}_1 = \begin{Bmatrix} \cos\beta \cos\left(\tan^{-1}\left(\frac{c}{\sqrt{3}a}\right)\right) \\ \sin\beta \\ \cos\beta \sin\left(\tan^{-1}\left(\frac{c}{\sqrt{3}a}\right)\right) \end{Bmatrix}. \quad (6)$$

It should also be noted that although it appears that the incident beam is adjacent to the twin dislocation plane in Fig. 3(b), it may not be located at $x_2^0 = 0$. Thus, we choose three representative locations, given by

$$\mathbf{P}_0 = (1.1, 0, 0)R, \quad \mathbf{P}_{\pm} = (1.1 \cos 20^\circ, \pm 1.1 \sin 20^\circ, 0)R, \quad (7)$$

which are in the unit of the dislocation loop radius R .

3. Results

The stress-strain response of a rolled AZ31B Mg alloy [11] is shown in Fig. 2 (a). During compression, a low yield strength, followed by a slow strain hardening up to 7% strain and then a relatively rapid strain hardening behavior, was observed. During the reverse tension, a relatively higher yield strength was detected, while a shortened slow work hardening region spanning from the strain of -5.7 to -2% strain was observed. When the sample was further stretched, a rapid strain hardening, followed by a steady stress-strain flow, was detected. The microstructural evolutions at selected strain levels are illustrated in Fig. 2(b)-(d). Before deformation, the material is basically twin-free, as shown in Fig. 2(b). At strain levels (ii) and (iii), the type of twin is exclusively the $\{10.2\}\langle 10.1\rangle$ tensile twinning. A large amount needle-shaped twin grains can be observed with various width from 1 to 16 μm , as shown by the electron backscatter diffraction (EBSD) images in Fig. 2 (c) and (d).

The grain orientation maps are constructed to characterize the twinning and detwinning behavior of a rolled AZ31B Mg alloy at three strain levels using *in situ* white beam synchrotron X-ray microbeam diffraction measurements, as shown in Fig. 3. The coordinates and geometric setup in the uniaxial test and diffraction measurements can be found in Fig. 1. Several grains can be identified from the sample surface to a depth of $\sim 140\ \mu\text{m}$, as displayed in Fig. 3. The grain orientation of 8 grains is displayed in the supplementary Fig. S1. The (00.1) poles in the most of grains (except grain ⑥) are close to ND, which is known as the typical rolling texture. In Fig. 3, the loading direction is horizontal, and the Y-axis represents the depth direction of the x-ray beam. The pixels with various colors represent different grain orientation. It should be mentioned that the

Figs. 3(a), (b), and (c) have the same length scale and the same location in each is marked with black rectangle. The grain orientation map of the un-deformed sample is illustrated in Fig. 3(a). The crystalline plane of grain ① in the sample surface normal direction is $(\bar{1}1.6)$, which is about 17.4° from the (00.1) plane. In the current study, the grain ①, which is approximately $35\ \mu\text{m}$ beneath the sample surface and $100\ \mu\text{m}$ in grain size, is chosen for the *in situ* measurement, because the twinning and detwinning activities in this bulk grain are quite representative during loading path in Fig. 2(a). The grain orientation of grain ① at a compression strain of -2.4% [point (ii) in Fig. 2(b)] is exhibited in Fig. 3(b). The red dashed curve denotes the grain boundary of grain ① before the deformation. In Fig. 3(b), a large portion of the parent grain has twinned, and only one twin variant is selected, while a small portion of the parent grain still remains untwinned at this deformation stage. The boundary of the untwinned portion is marked with solid red curves. The indexed Laue patterns of untwinned and the twinned portions are illustrated in the supplementary Fig. S2(a) and (b). It is demonstrated that the tensile twins locate at the blank area near the parent grain with approximately 85.5° (close to the theoretical value 86.3°) misorientation from the parent grain ①. Parent-twin boundary does not resemble the surface EBSD measurements (e.g., Fig. 2) or the post-mortem TEM images, which show sharp boundaries and needle-like or lenticular twins. Instead, the parent-twin boundary inside this bulk grain is irregular and has many sharp “finger-tips” in Fig. 3(b). Admittedly, the orientation map in Fig. 3 is just one slice of the entire grain, but one can

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4 imagine the three-dimensional reconstruction of the twin should possess irregular
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10 Figure 3(c) is the grain orientation map after – 7% compression – relative 3% reverse
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12 tension [point (iii) in Fig. 2(b)]. After this reverse tension, two surprising observations are
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14 found. First, the parent portion grows due to the shrinkage of the twinned portion. Very
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16 especially, the shrinkage occurs by “smoothening” some of the sharp finger-tips at the
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18 parent-twin boundary. Second, the other process that is equally probable is the nucleation
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20 of new twin variants in the previously twinned portion, which only occurs near the
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22 previous “finger tips”. In Fig. 3(d), two new needle-shaped twin areas are observed with
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24 their boundaries marked in color blue. It is interesting to notice that a very small
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26 reoriented grain marked in color black exists between the parent and twin portions, which
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28 has a different orientation from the parent grain (the boundary is marked in color black).
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30 Three distinct Laue patterns and the lattice orientations of the parent, twin, and reoriented
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32 areas are illustrated in Fig. S2(c)-(e) and Fig. 3, respectively, and the detailed lattice
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34 orientations of them are given in the supplementary Table 1.
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42 The diffraction peak intensity and lattice strain evolution of the $\{\bar{1}2.8\}$ peak as a
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44 function of resolved depth at a specific sample position (marked as a purple solid line on
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46 Fig. 3(c) and (d)) during the compression and reverse tension using the monobeam
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48 energy scan are displayed in Fig. 4. The diffraction peak intensity distribution across the
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50 parent-twin-parent grains during the compression is illustrated in Fig. 4(a). The
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52 diffraction peak intensity of $(\bar{1}2.8)$ decreases to zero around a depth of 76 μm , which
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54 means that other orientations (i.e., twins) appear. Comparing with Fig. 3(c), the tensile
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twins emerged at the same depth in the remaining parent portion. Hence, it can be concluded that the diffraction peak intensity decrease at approximately 76 μm in depth resulted from a tensile twin. This trend demonstrates that the monobeam energy scan results are consistent with the white beam grain orientation mapping. The grain boundaries and twin boundaries are highlighted as dashed red and purple lines, respectively, based on the diffraction peak intensity variation. The lattice strain of the $(\bar{1}2.8)$ peak in a parent grain as a function of resolved depth is presented in Fig. 4(c). In general, the lattice strain of the $(\bar{1}2.8)$ peak decreases with the increase of the resolved depth. The tensile lattice strain can be observed near the sample surface, while the compressive lattice strain appears inside the material. The lattice strain at the grain boundary near the sample surface is relatively low, which increases rapidly and reaches the peak value at a depth of 57 μm . Then, the lattice strain decreases promptly until a depth of 65 μm and changes slightly before crossing the twinned area. It is interesting to note that a dramatic lattice strain decrease can be discovered at a depth of 76 μm , where a twin appears in the remained parent grain. It indicates a large lattice strain gradient across the twinned area. After passing through the twinned area and before reaching to a depth of 83 μm , the lattice strain keeps on decreasing. A small lattice strain increase can be observed when approaching the grain boundary on the other side. A relatively high compressive lattice strain can be seen on the parent grain boundary deep into the material. Except for the sharp decrease of the lattice strain across the twinned area, the overall change of the lattice strain results from the heterogeneous strain field on the grain level (denoted as Type-II strain in diffraction studies), which deviates from the applied

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4 macroscopic uniform strain field. The lattice strain jump across the twinned area, denoted
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6 as Type-III strain, will be addressed in the Discussion section.
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10 The diffraction peak intensity variation of the $(\bar{2}1.\bar{8})$ peak in a parent grain as a function
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12 of resolved depth during reverse tension is demonstrated in Fig. 4(b). Similarly, the
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14 discrete diffraction peak intensity can be found in the range from a depth of 73 to 79 μm .
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16 As shown in Fig. 3(d), a twinned area locates in this range. The size of the twinned area
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18 in Fig. 4(b) is somewhat larger than in Fig. 4(a), due to the previous severe deformation
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20 (-7% compression). The lattice strain change of the $(\bar{2}1.\bar{8})$ peak in a parent grain as a
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22 function of resolved depth during reverse tension is shown in Fig. 4(d). Comparing Figs.
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24 4(c) with (d), it is obvious that the lattice strain moved toward the compression side,
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26 since the reverse tension is applied in the axial direction. This overall trend is exactly
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28 described by the grain-level heterogeneous Type-II strain in diffraction studies, which is
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30 caused by the grain-grain interactions. Overall, the lattice strain during the reverse
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32 tension decreases with the increasing resolved depth, which is similar to that during
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34 compression. As shown in Fig. 4(d), a lattice strain increase can also be perceived near
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36 the parent area on the sample surface side. Interestingly, the lattice strain remains almost
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38 unchanged when crossing the twinned area during the reverse tension. This intragranular
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40 Type-III strain near the twin zone will be discussed next. The lattice strain decreases
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42 when approaching the twin boundary on the left side and moving away on the right side
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44 of the twinned area. When getting to the parent area boundary from a depth of 87 to 92
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46 μm , the lattice strain remains constant.
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4. Discussion

It has been proposed that the detwinning process is mainly controlled by the migration of twin boundaries into the twins via the glide of the twinning dislocations, leading to a propagation of the parent grains into twin grains, which has been frequently observed and proved by *in situ* EBSD and TEM studies [22, 32]. However, post-mortem TEM studies sometimes find the nucleation of parent grains inside the twinned grain in the samples prepared by focus-ion milling and cutting from a bulk sample. Recently, another deformation mechanism has been reported by Liu and co-authors [12] that a twinning-like lattice reorientation resembles conventional $\{10.2\}\langle 10.1 \rangle$ tensile twinning but without a crystallographic mirror plane by *in situ* TEM. It provides the plastic strain by a 90° lattice reorientation, indicating that it is not a simple shear. However, this mechanism has not been experimentally verified in bulk Mg alloys during *in situ* deformation. The present study provides the direct observation of the plastic deformation process in an individual grain inside the polycrystalline Mg alloy during compression-tension experiments, which could be beneficial for the development of a new theoretical model to accurately capture the plastic deformation processes of the HCP-structured Mg. It was found that the original parent area is recovered, as present in Fig. 3(c), which is fulfilled by the motion of twinning dislocation from an untwinned area into a twinned area, i.e., the conventional detwinning mechanism. Moreover, the reoriented grain has a different lattice orientation with respect to the original parent grain, although the c-axes of the parent and detwinned area point at almost the same direction (4.6° apart in the current study). Based on the exact lattice orientations listed in Table 1 and grain orientation changes during deformation in Fig. 5, the schematic of the lattice orientation evolution is

presented in Fig. 3. Therefore, the experimental observation in the current study supports the co-existence of the two possible plastic deformation mechanisms, tensile twinning and twinning-like lattice reorientation, in the polycrystalline bulk material. In our previous neutron diffraction studies, it was suspected that the grain rotation occurred during the reverse loading after the twinning process [11, 16, 17, 33]. The current study provides the direct evidence to support the previous observation. Furthermore, all these processes occur near the irregular twin-grain boundary in Figs. 3(c) and (d), leading to a smoothed boundary during tensile loading stage.

It has been proven that the tensile twinning can be easily activated during the in-plane compression or through-thickness tension in rolled Mg alloys through *in situ* EBSD and neutron diffraction measurements [7-9, 11, 24, 34, 35]. In the current study, it is evident that the tensile twinning occurred during compression along the RD, as shown in Fig. 3(c). The majority of a grain has been twinned, and only a small portion of the parent grain remains at a compression strain of -2.4% . Besides that, in the current study, it is found that due to discontinuous twinning across the original parent area, the remainder of the parent grain is divided into several smaller regimes. Moreover, it is very interesting to notice that the sharp “finger-tip” features were developed at the parent-twin boundary during the twinning dominated deformation, while they became smoothed during detwinning deformation in Figs. 3(b) and (c).

In addition to the crystallographic orientation map in Fig. 3, the use of the monochromatic x-ray beam reveals the lattice strain along the depth direction. In general, it is found that the lattice strain of the parent grain on the surface side is generally much higher than that inside the material for both twinning and detwinning processes, as shown

in Figs. 4(c) and (d). The lattice strain generally decreases gradually in the parent grain with the increase of the sample depth. The apparent lattice strain increase at the grain boundary of the parent grain on the surface side is observed during the twinning and detwinning dominated deformation.

Now we aim to interpret the sharp lattice strain jump across the twin grain in Fig. 4(c). From the geometric setup (Fig. 1) and the lattice orientations of the parent and twin grains (inset in Fig. 3), the twin plane and YZ diffraction plane are illustrated in Fig. 1(b). The finger-shaped twins area can be modeled by a stack of twin dislocation loops, or a super-dislocation loop with a Burgers vector, nb_s . The x-ray beam is in the Y-axis and runs across the dislocation plane at x_i^0 . Experimental results in Fig. 4(c) suggest a sudden drop of the lattice strain, $\varepsilon_{\mathbf{k}_2-\mathbf{k}_1}$ when the incident beam irradiated near the twin area. Referring back to Fig. 1(b), the incident beam may not go through $x_2^0=0$ because only one plane of XY is obtained in Fig. 3. Thus we tested points, $\mathbf{P}_0$ and $\mathbf{P}_\pm$. The lattice strain has no dimension, but our formulation (see the supplementary material) presents the strain in the form of $\varepsilon_{ij}R/nb_s$, where n is the number of dislocations in the protruded twins. Predicted results in Fig. 6 shows that when x_i^0 is located on the $x_2 \leq 0$ side of the twin dislocation loop, a noticeable drop, $\Delta\varepsilon_{\mathbf{k}_2-\mathbf{k}_1}$, can be found with a magnitude of $\Delta\varepsilon_{\mathbf{k}_2-\mathbf{k}_1} R/nb_s \approx 0.1$.

Experimental results in Figs. 4(c) and (d), in fact, have a superposition of the Type-II strain that is caused by the grain-grain interaction and the Type-III strain caused by the twin grain. In Fig. 4(c), the Type-II strain is monotonically decreasing, so that we are

only concerned with a sudden drop of the Type-III strain across the twin grain. This trend gives a lattice strain drop, $\Delta\epsilon_{\mathbf{k}_2-\mathbf{k}_1}$, of about 10^{-3} . Geometric features in Fig. 3(b) suggest that $R/nb_s \approx 100$, giving rise to a measured value of $\Delta\epsilon_{\mathbf{k}_2-\mathbf{k}_1} R/nb_s$ on the same order of magnitude as the prediction in Fig. 6. This agreement confirms the orientation mapping and the twin-parent interaction. The lattice strain drop vanishes in Fig. 4(d) during the detwinning stage. The orientation map in Fig. 3(d) identifies a twinning-like reoriented zone. The interface between this zone and the prior twinned zone is incoherent and consists of a high density of non-gliding dislocations. Additionally, this re-oriented zone is at quite a distance from the line scan, so that it has little effect in the lattice strain distribution in Fig. 4(d). Considering the observation of the reduction of the lattice jump from Figs. 4(c) to (d), and also noticing the planar scan in Fig. 3 is restricted to a two-dimensional slice of a rather three-dimensional solid, it is anticipated that in Fig. 4(d) there are new twin variants nucleated in the pre-existing twin grains. Equivalently speaking, a twin dislocation of a similar size but with a Burgers vector rotated by $\pm 2\pi/3$ is superposed onto Fig. 1(b), leading to a cancellation of the elastic strain fields. Therefore, the tensile strain produced inside the twin grain during the tensile twinning process could facilitate the motion of twinning dislocations toward the parent grain. Also, the unchanged lattice strain inside a twin grain with the apparently decreased lattice strain outside a twin grain could be beneficial for the detwinning process by moving the twinning dislocations into the twin grain.

5. Conclusions

In summary, in the current study, the twinning and detwinning behavior in an individual grain in a rolled AZ31B Mg alloy has been investigated using *in situ* synchrotron X-ray microbeam diffraction on the sub-micrometer level. The twinning, detwinning, and twinning-like lattice re-orientation process have been observed in an individual grain inside a polycrystalline HCP-structured Mg alloy during *in situ* loading. The major conclusions can be drawn as follows:

1. It was found that during the early stage of deformation, a large portion of the parent grain was twinned, and only one twin variant was selected, while a small portion of the parent grain still remained untwined.
2. An irregular shape and many sharp “finger-tips” were perceived at the parent-twin boundary.
3. After the strain reversal, besides the detwinning, the lattice reorientation behavior was characterized through the experimental observation in the bulk Mg alloy for the first time.
4. The distinct lattice strain decrease in a parent grain was observed on two sides of the twin during the twinning dominated deformation, but no lattice strain variation was detected during the detwinning dominated deformation.
5. The micromechanical analysis of the lattice strain near the twin-dislocation loop confirms the above measurements and provides the additional evidence of the detwinning/lattice reorientation mechanisms.

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Figure Captions

Figure 1 (a) Schematic of synchrotron X-ray microbeam diffraction. (b) Geometric setup of the twin dislocation loop in the sample and diffraction coordinates.

Figure 2 (a) The stress-strain curve for a rolled AZ31B Mg alloy [11]. (b) – (d) The inverse pole figure maps in RD corresponding to three strain levels (i), (ii), and (iii), respectively, marked on Fig. 1(a).

Figure 3 The grain orientation maps: (a) – (c) before deformation, during compression, and reverse tension, respectively. (d) The zoom-in view of the area marked in (c) by green square dashed line. The letters, P, T, and N designate parent grain, twin grain, and lattice reorientation grain, respectively.

Figure 4 The diffraction peak intensity variations of the $\{\bar{1}2.8\}$ peak in a parent grain as a function of depth during compression and reverse tension are presented in (a) and (b), respectively. The lattice strain evolutions of the $\{\bar{1}2.8\}$ peak vs. depth at different locations during compression and reverse tension are illustrated in (c) and (d), respectively.

Figure 5 The (00.1) pole figure of parent grain ① in Fig. 2(a) during twinning-detwinning/lattice reorientation process.

Figure 6 Lattice strain in direction of $k_2 - k_1$ versus the distance along the k_1 direction. Black and red curves suggest a large drop of $\epsilon_{k_2 - k_1}$ near the twin dislocation loop.

Figure S1 The (00.1) pole figure for 8 distinct grains before deformation in Fig. 3(a).

Figure S2 The Laue patterns: (a) and (b) for parent and twin grains, respectively, in Fig. 3(b), (c) – (e) for parent, twin, and the reoriented grains, respectively, in Fig. 3(c).

Table 1 The lattice orientations of parent, twin, and reoriented grains.

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Figure(s)

Figure 1 (a) Schematic of synchrotron X-ray microbeam diffraction. (b) Geometric setup of the twin dislocation loop in the sample and diffraction coordinates.

100 μm

Figure 2 The stress-strain curve for a rolled AZ31B Mg alloy [11]. (b) – (d) The inverse pole figure maps in RD corresponding to three strain levels (i), (ii), and (iii), respectively, marked on Fig. 1(a).

Before Deformation, (i)

Figure 3 The grain orientation maps: (a) – (c) before deformation, during compression, and reverse tension, respectively. (d) The zoom-in view of the area marked in (c) by green square dashed line. The letters, P, T, and N designate parent grain, twin grain, and lattice reorientation grain, respectively.

Figure 4 The diffraction peak intensity variations of $\{\bar{1}2.8\}$ the peak in a parent grain as a function of depth during compression and reverse tension are presented in (a) and (b), respectively. The lattice strain evolutions of the $\{\bar{1}2.8\}$ peak vs. depth at different locations during compression and reverse tension are illustrated in (c) and (d), respectively.

- Parent/detwinned grain
- Twin grain
- ▲ Reorientation grain
- Twinning process
- Detwinning process
- -> Reorientation process

Figure 5 The (00.1) pole figure of parent grain ① in Fig. 2(a) during twinning-detwinning/lattice reorientation process.

Figure 6 Lattice strain in direction of $\mathbf{k}_2 - \mathbf{k}_1$ versus the distance along the $\mathbf{k}_1$ direction.
Black and red curves suggest a large drop of $\epsilon_{\mathbf{k}_2 - \mathbf{k}_1}$ near the twin dislocation loop.

Table 1 The lattice orientations of parent, twin, and reorientation grains.

	a (nm)	b (nm)	c (nm)
Parent grain	{-0.276907, -0.041018, -0.157145}	{0.273402, -0.114451, -0.123439}	{-0.076683, -0.451142, 0.249352}
Twin grain	{0.024366, 0.172616, -0.268362}	{-0.024421, -0.325446, -0.010265}	{-0.518645, 0.035244, -0.009997}
Reorientation grain	{0.199521, 0.115581, 0.22632}	{-0.31569, 0.030122, 0.037019}	{-0.034764, -0.454188, 0.249416}