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# Intragranular twinning, detwinning, and twinning-like lattice reorientation in magnesium alloys☆

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ABSTRACT

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



## 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 <a> slip on basal planes and the {10.2}⟨10.1⟩ 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 <a> 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 {10.2}⟨10.1⟩ 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

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(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, $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$, 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 $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twinning deformation [25]. However, the detwinning and/or lattice reorientation behavior during reverse loading were not explored and remained unclear.

An in-depth understanding of the deformation and failure mechanisms in Mg polycrystals requires a fundamental knowledge of the twinning activities. For example, how the deformation twinning-detwinning operate inside a parent grain? Does the twinning-like lattice reorientation exist in a bulk HCP-structured material? What is the lattice strain distribution around the parent-twin interface? In this study, the tensile twinning behavior within an individual grain inside a polycrystalline rolled AZ31B Mg alloy was investigated *in situ* using the state-of-the-art synchrotron X-ray microbeam. A special 3D DAXM technique to achieve the sub-micrometer scale is used to spatially resolve the lattice orientations and strains with the focused polychromatic and monochromatic X-ray, respectively. The twinning − detwinning and/or twinning-like lattice reorientation processes, as driven by a macroscopic compression and tension strain path, were identified by the grain orientation map during in-plane compression and reverse tension. The internal strain and diffraction peak intensity variation of a certain *hkl* inside an individual grain was also measured. A theoretical model was developed to simulate the strain field across the parent-twin interface. The twinning-like lattice reorientation was characterized for the first time using the diffraction method in a bulk Mg sample and the parent-twin boundary were found to differ from surface-based measurements.

## 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 h 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 Fig. 1(a). 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 to 30 keV) was applied with a 0.5 × 0.5 μm $^2$ 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 × 0.5 μm$^2$ 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 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:

$$\varepsilon^{hkl} = \frac{d^{hkl} - d_0^{hkl}}{d_0^{hkl}} \tag{1}$$

where $\varepsilon^{hkl}$ 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 diffratometer, 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-