Mechanical twinning and detwinning in pure Ti during loading and unloading — an *in situ* high energy X-ray diffraction microscopy study

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Abstract

Far-field high energy X-ray diffraction microscopy (HEDM) was used to study $\{10\bar{1}2\} < \bar{1}011 >$ twinning in Ti. Twin nucleation **within** a bulk parent grain is observed at a resolved shear stress (RSS) of 225MPa. During unloading, the RSS on the twin plane reversed sign, providing a driving force for detwinning. Formation of the twin, however, prevented the parent grain from returning to its original stress state even after complete unloading. The twin morphology and surrounding environment were **examined using** near-field HEDM.

Text

Mechanical twinning and de-twinning are important plastic deformation mechanisms in hexagonal metals. The most commonly observed twinning mode is $\{10\bar{1}2\} < \bar{1}011 >$ [1-5]. Studies using electron backscatter diffraction (EBSD) [6-8] as well as high energy X-ray diffraction microscopy (HEDM) [9] show that twin nucleation occurs even in grains with medium to low Schmid factors computed from the external loading geometry. However, local stress states in a polycrystalline aggregate can be very different from the global stress [10]. To assess whether a critical resolved shear stress (CRSS) exists for twin nucleation, as assumed in many models [11-13], it is necessary to measure local stress states in individual grains just prior to twin nucleation. Furthermore, *in situ* microscopy and neutron diffraction studies have shown that twinned volumes can shrink or disappear when loading is removed or reversed [14-16]. The local stress conditions that lead to detwinning remain unclear. Direct measurements provide evidence needed to validate constitutive models.

The HEDM suite of techniques [17-20] coupled with *in situ* mechanical testing enables the required grain level investigations. Far-field HEDM (ff-HEDM) can spatially map grains from a bulk polycrystalline specimen. By analyzing diffraction patterns taken at different specimen rotation angles, the crystallographic orientations, centers of mass (COM), and average strain tensors for each grain can be determined [21-24]. With knowledge of single crystal elastic constants, stress tensors can then be computed. Aydiner et al. [25] first applied ff-HEDM to identify twin formation in Mg during compression. More recently, Bieler et al. [26] studied twin nucleation in Ti and found that twin nucleation may or may not occur on the twin variant with

the highest resolved shear stress. Strain transfer from neighboring grains could account for the latter behavior.

In the present study, a further HEDM investigation of twinning in Ti is described. For the first time, a spatially resolved twinning-detwinning event was measured *in situ* in the interior of a bulk specimen in a loading-unloading test. Stress evolution in the parent grain and the twin were monitored throughout the test. Issues related to the stress condition for twin nucleation, residual stresses due to twin formation, and the driving force for detwinning are examined.

The investigated material was grade 1 commercially pure Ti with the composition given in [26]. The grain size of this material was $\sim 100\text{ }\mu\text{m}$ as determined from initial EBSD analysis. A tensile sample with a cross sectional area of $1\times 1\text{ mm}^2$ and gauge length of 5mm was prepared. It has a strong c-axis texture with a population of about eight times that of a random distribution close to the tensile axis. The sample was mounted in a specially designed load frame [19] at the Advanced Photon Source (APS) beamline 1-ID and deformed incrementally in tension with 37 steps. A $100\text{ }\mu\text{m}$ tall by 1.5 mm wide monochromatic X-ray beam ($E=65.4\text{ keV}$) illuminated the entire cross section of the sample. To employ ff-HEDM (Figure 1), the load was held constant while the sample was rotated about the tensile axis (Z) over a 140° range and diffraction patterns are recorded as ω sweeps through 1° intervals at each deformation step. The X-ray area detector (2048×2048 pixels spanning a $0.4\text{m} \times 0.4\text{m}$ area) was placed approximately 1m downstream of the sample. The measurement was performed in 11 contiguous layers of the sample so the total investigated volume was $1.1\text{mm} \times 1\text{mm} \times 1\text{mm}$. A digital image correlation (DIC) camera was

used to ensure that approximately the same volume was illuminated at all 37 deformation steps, which is critical for tracking individual grains through the test. The stress-strain curve, derived from the load-displacement data assuming uniform macro-scale deformation, is shown in Figure 1; this agrees closely with analysis of the DIC data. The sample experienced elastic deformation during the first 7 steps (up to 0.2% strain), plastic deformation during the next 23 steps (0.2% to 3% strain), and unloading during the last 7 steps. The stepwise unloading allowed the tracking of detwinning.

At each deformation step within each layer, individual grains were indexed using the 140 diffraction patterns recorded during ω rotation. Indexing procedures are described in [26]. Overall, three analysis programs — *PeakSearch*, *Transformation*, *GrainSpotter* — were applied successively using the FABLE software package [27]. These steps identify peaks in the diffraction patterns, assign to each a reciprocal lattice vector, $\mathbf{g}_{hkl}$, and determine grain orientations from the pool of $\mathbf{g}_{hkl}$'s. A grain is considered indexed when a minimum of 30 crystallographically consistent reciprocal lattice vectors are associated with a given lattice orientation. The minimum completeness (fraction of expected $\mathbf{g}_{hkl}$'s assigned to the grain) is 70%. An independent Matlab code, following [26, 21], was used to obtain the center of mass (COM), lattice strain, and stress tensor for each indexed grain.

Figure 2 illustrates the identification of a twin in layer 6 (the middle layer). In Figure 2(a), $\{0001\}$ pole figures from layer 6 at three different strains are shown. The c-axis texture is evident as most grains have their $\{0001\}$ pole close to the Z direction. At a global strain of 1.6%, a twin had nucleated. A parent grain (referred to as Grain 1) was identified for the twin

according to two criteria: (1) the misorientation between Grain 1 and the twin (86.3° around $\langle 1\bar{2}10 \rangle$) agrees with the theoretical orientation relationship for $\{10\bar{1}2\} \langle \bar{1}011 \rangle$ twinning; (2) Grain 1 is physically near the twin. Figure 2(b) shows the grain COM map at the three macroscopic strains. The square shape of these maps is consistent with the $1 \times 1 \text{ mm}^2$ cross section of the sample. Grain 1 and its neighboring grains (Grains 2-10) are highlighted. At 1.6% strain, the COM of the twin (T) is about $115 \mu\text{m}$ away from the COM of Grain 1, indicating that the twin formed near the edge of a large grain. Except for Grains 3 and 6, all other neighboring grains of Grain 1 had „hard“ orientations with $\{0001\}$ close to the tensile axis (Figure 2(a)). Note that the COMs of some grains at 1.6% strain are shifted compared with 0.9% strain. This is likely because of vertical offset in the measured volume between different load steps (despite the usage of DIC), which will affect the COM position for grains that are partially located in a layer. This vertical offset may also explain why some neighboring grains are missing at 1.6% strain — should these grains lie outside the measured layer 6 at this load step.

The stress tensor for each grain was determined from the measured lattice strain of different (hkl) planes [21]. Figure 3(a) shows the values of σ_{ZZ} in all grains prior to macroscopic loading. Grains near the upper and lower edges of the sample show negative and positive σ_{ZZ} values respectively, indicating that there was an initial macroscopic internal bending stress in the sample that either preexisted or resulted from mounting of the sample. Figure 3(b) shows the evolution of σ_{ZZ} in selected grains. In all grains, σ_{ZZ} increased rapidly in the elastic stage (up to 0.2% strain) and changed modestly in the plastic stage (0.2% to 3% strain). The twin was first observed at 1.5% strain; σ_{ZZ} in the twin is below the value in the parent and most of the

neighboring grains, indicating the stress relief expected due to load partitioning between the hard-oriented parent grain and the soft-oriented twin [25]. Upon unloading, σ_{ZZ} in both Grain 1 and the twin quickly dropped and became negative when the load was completely removed. After unloading, σ_{ZZ} in Grain 1, though negative, was 116MPa higher than the initial unloaded state. The formation of the twin evidently prevented Grain 1 from returning to its original stress state. A similar observation is found for Grain 5 that is close to the twin. In contrast, σ_{ZZ} in Grain 2 is almost the same before loading and after unloading. σ_{ZZ} in Grain 6 gradually increased after yielding, indicating strain hardening in this soft-oriented grain (see Figure 2), but after unloading, σ_{ZZ} returned to its before-loading value. The last two σ_{ZZ} values for Grain 6 come from the analysis of layer 5, where Grain 6 is also present, as Grain 6 was not indexed in layer 6 in the last two unloading steps.

Using the stress tensor for Grain 1, the resolved shear stress (RSS) for each twin variant was calculated and plotted in Figure 3(c). In early deformation, the RSS on the six variants were close to each other. This is expected because the c-axis of Grain 1 was nearly aligned with the tensile axis, so each twin variant was nearly symmetrically stressed. Upon yielding, the RSS values on the six twin variants developed a larger spread. The activated twin variant, VT 3, became the most stressed one after 1% strain. Twin nucleation occurred when the RSS on VT3 was about 225MPa. This value is consistent with the result in [26], where the three twin nucleation events occurred when the RSS in the respective parent grains are 200MPa, 240MPa, and 189MPa. It should be emphasized that a high RSS alone does not guarantee twin nucleation, as grain boundary defects are also important for the twin nucleus to form [7, 8]. The absence of twin formation on variants VT5 and VT6, even when the RSS exceeded 225MPa, can be

attributed to three possible reasons: (1) no appropriate grain boundary defects to assist nucleation were present; (2) nucleation and growth of VT3 made it difficult for other variants to be activated in the same parent grain; (3) VT5 or VT6 did nucleate, but were too small to be indexed. In the twin, the RSS on the twin plane along the twin shear direction is also shown in Figure 3(c). The negative value implies that the lattice in the twin is sheared in the opposite direction compared to the lattice in the parent grain across the twin interface — exactly what is expected during twinning. Upon unloading, the RSS in the twin moves toward positive values and eventually changes sign when the external load is reduced to 200MPa (third from the last data point). When the load was completely removed (last data point), the RSS in the twin became 91MPa, which is even greater than the value in the parent grain (~39MPa on VT3). This might be sufficient to initiate detwinning, since detwinning requires a relatively small reverse RSS [15]. In fact, it is found that the integrated intensity of the twin (0002) peak after complete unloading is about 20% lower than that at the beginning of unloading; this suggests that the volume of the twin shrank by approximately 20% during unloading. Stress close to the twin interface may be different from the grain-averaged stress of the twin and Grain 1, but it is not possible to study such in-grain stress variation using ff-HEDM.

After the mechanical test, the sample was taken out of the load frame, and a near-field HEDM [28-30] scan was performed near the central part of the far-field measurement volume to observe the geometry of some twins (this can now be done simultaneously with far-field measurements [31]). By placing the near-field HEDM detector only millimeters away from the sample and using a forward modeling strategy, the 3D grain structure can be reconstructed with high spatial resolution (~ 2 μ m). The X-ray beam for the near-field scan was ~ 4 μ m tall by 1.5mm wide. The

measurement scanned 100 slices along the sample gauge length, which covers a material volume of $400\mu\text{m}\times 1\text{mm}\times 1\text{mm}$. After reconstruction using a voxel size of $2\mu\text{m}$, a twin/parent grain pair (#182/#124) was found in the top slice whose misorientation angle/axis is consistent with $\{10\bar{1}2\} < \bar{1}011 >$ twinning (Figure 4). Based on the twin plane trace analysis, the location of #182 and #124 in the cross section, and the large size of #124, we conclude that these grains correspond to the twin and Grain 1 in layer 6 of the far-field measurement. The near-field map offers a better visualization of the twin morphology, showing that it was located adjacent to a grain boundary.

In summary, this work examines a twinning event in a bulk Ti sample during a loading-unloading test using *in situ* far-field and *ex situ* near-field HEDM. In this observed instance, the twin nucleated at 1.6% global strain on the most stressed twin variant in the parent grain. After the twin formed, the parent grain carried more stress than the twin. The RSS in the twin changes sign during unloading, providing a driving force for detwinning. Twin formation prevents the parent grain from returning to its original stress state when the load is fully removed. The morphology of this twin is revealed by an *ex situ* near-field HEDM scan. This work is among the first efforts to combine far-field and near-field HEDM techniques, a powerful method to study internal microstructure evolution in crystalline materials.

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Figure Captions

Fig. 1. Far-field HEDM setup at APS 1-ID along with the coordinate system used for data analysis. The sample was deformed by tension with 37 deformation steps, as shown in the stress-strain curve.

Fig. 2. Identification of a twinning event in layer 6. (a) $\{0001\}$ pole figures at three different global strains. The normal to the figure is the tensile axis. The orientation marked “T” appeared after 1.6% strain; this spot has the twin orientation relation to Grain 1 (hence, the parent grain). (b) Grain COM maps in the X-Y plane at these three strains. Positional standard errors in the X and Y directions are represented by a cross overlaid on each COM. At 1.6% strain, the twin was found in the vicinity of Grain 1 (close to Grain 5).

Fig. 3 (a) Distribution of σ_{ZZ} in all indexed grains in layer 6 at 0 strain. The σ_{ZZ} values in Grains 1-10 are indicated on the plot (in MPa). (b) Evolution of σ_{ZZ} in Grains 1, 2, 5, 6 and the twin through the test. Estimated uncertainty of σ_{ZZ} is about 33MPa. (c) RSS on six twin variants (VT) in Grain 1. VT 3 was the activated variant. RSS in the twin was also shown.

Fig. 4. The twin identified in layer 6 appears in the top slice of the near-field HEDM scan.

Figure 1
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