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# 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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Far-field high-energy X-ray diffraction microscopy (HEDM) was used to study $\{10\bar{1}2\}\langle\bar{1}011\rangle$ twinning in Ti. Twin nucleation within a bulk parent grain is observed at a resolved shear stress (RSS) of 225 MPa. 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.


*Keywords:* High-energy X-ray diffraction microscopy; Titanium; In situ mechanical testing; Twinning; Detwinning

Mechanical twinning and detwinning are important plastic deformation mechanisms in hexagonal metals. The most commonly observed twinning mode is $\{10\bar{1}2\}\langle\bar{1}011\rangle$ [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 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.

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The investigated material was grade 1 commercially pure Ti with the composition given in Ref. [26]. The grain size of this material was ∼100 μm as determined from initial EBSD analysis. A tensile sample with a cross-sectional area of 1 × 1 mm$^2$ and gauge length of 5 mm 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 μm tall by 1.5 mm wide monochromatic X-ray beam ($E = 65.4$ keV) illuminated the entire cross-section of the sample. To employ ff-HEDM (Fig. 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 m × 0.4 m area) was placed approximately 1 m downstream of the sample. The measurement was performed in 11 contiguous layers of the sample, so the total investigated volume was 1.1 mm × 1 mm × 1 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 macroscale deformation, is shown in Figure 1; this agrees closely with analysis of the DIC data. The sample experienced elastic deformation during the first seven steps (up to 0.2% strain), plastic deformation during the next 23 steps (0.2% to 3% strain) and unloading during the last seven 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 Ref. [26]. Overall, three analysis programs – *PeakSearch*, *Transformation* and *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 Refs. [26,21], was used to obtain the 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: (i) 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; and (ii) 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 × 1 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 μm 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 (Fig. 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 $\sigma_{ZZ}$ in all grains prior to macroscopic loading. Grains near the upper and lower edges of the sample show negative and positive $\sigma_{ZZ}$ 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 $\sigma_{ZZ}$ in selected grains. In all grains, $\sigma_{ZZ}$ increased rapidly in the elastic stage (up to 0.2% strain)

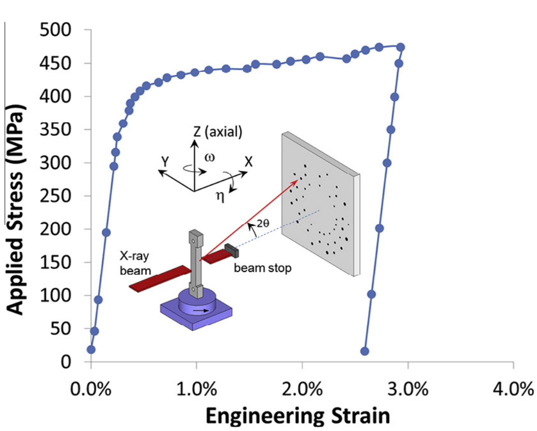


**Figure 1.** Far-field HEDM setup at APS 1-ID along with the coordinate system used for data analysis. The sample was deformed by tension with 37 deformation steps, as shown in the stress–strain curve.

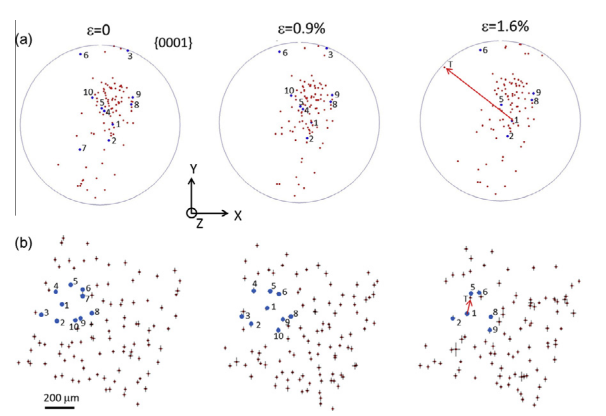


**Figure 2.** Identification of a twinning event in layer 6. (a) {0001} pole figures at three different global strains. The normal to the figure is the tensile axis. The orientation marked "T" appeared after 1.6% strain; this spot has the twin orientation relation to Grain 1 (hence, the parent grain). (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).