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APPLICATION NOTE

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In-Situ Micropillar Compression

Nanomechanical Testing in the SEM with Simultaneous EBSD

Examining the mechanical behavior of microscale structures requires instrumentation and techniques capable of revealing phenomena that occur at the sub-micron- and nano- scale. The scanning electron microscope (SEM), especially when coupled with electron backscatter diffraction (EBSD), can provide the spatial resolution required to observe the mechanical response and to characterize physical deformation of such microscale structures. When coupled with a nanomechanical test instrument designed for use in the SEM, a complete characterization of the behavior involved is possible.

The **PI 85 SEM PicoIndenter®** is a fully quantitative nanomechanical test instrument designed to be used in the SEM. With a compact, vacuum-compatible design it can easily be interfaced with commercial SEM systems. Plus, with a sub-nanometer displacement noise floor and a sub-microNewton force noise floor, it offers sensitivity ideal for nanoscale *in-situ* testing. Utilizing a creative sample geometry allows for simultaneous SEM imaging, nanomechanical testing, and EBSD mapping.

In this work, the **PI 85** was used to test an individual

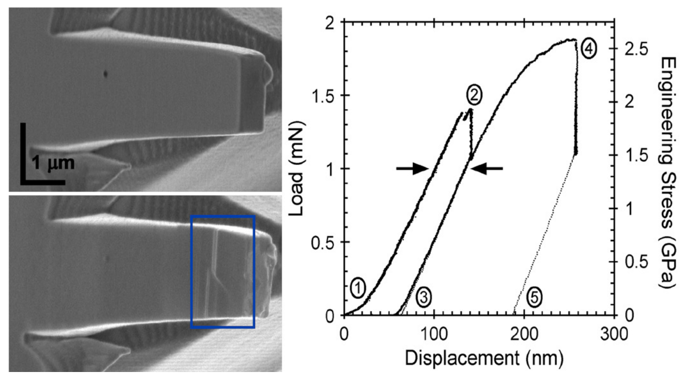

Figure 1: GaAs pillar before (top) and after (bottom) compression. The blue rectangle indicates an area of plastic deformation, which is shown in more detail in Figure 3. The load–displacement curve shows the mechanical response of the pillar. The engineering stress shown was calculated by dividing the load by the cross-sectional area.

pillar of GaAs. The pillar was fabricated from single-crystal GaAs using focused ion beam (FIB) milling. In the microscope the pillar was oriented such that the compression axis (the [001] direction of the pillar) was perpendicular to the electron beam and there was an angle of 20° between the EBSD detector screen and the (110) face of the pillar. A conductive-diamond flat-punch tip was used for the compression.

The pillar was compressed using displacement-control mode with a displacement rate of 2 nm/s. Before and after images of the pillar and the load–displacement

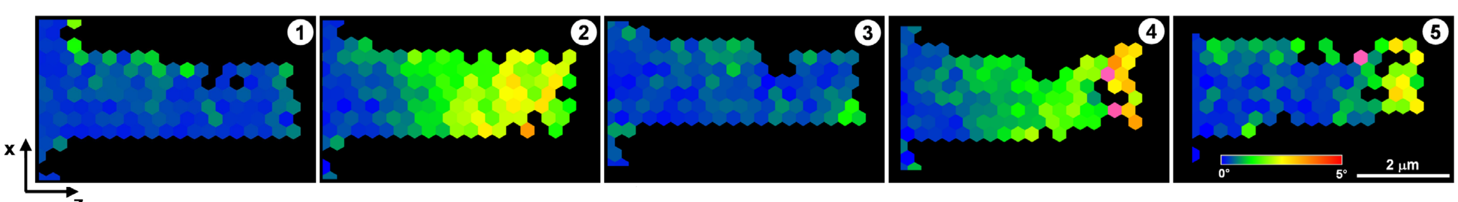

Figure 2: EBSD orientation maps acquired before, during, and after loading. The numbers indicated correspond to those in the load–displacement curve shown in Figure 1.

data acquired during the test are shown in Figure 1. The EBSD patterns in Figure 2 show the crystallographic orientation of the pillar before nanomechanical testing, at peak load (via a 220 second hold segment at maximum displacement) and after completing the nanomechanical test. In the first loading cycle, 1→2→3, the pillar was loaded to a peak load of nearly 1.5 mN and the resulting deformation was predominantly elastic. The change in orientation observed in the EBSD map acquired at peak load was caused by elastic bending of the pillar by as much as 3°.

The pillar was then loaded a second time, 3→4→5, this time to a higher peak load of nearly 2 mN. After the second loading cycle, substantially more permanent deformation was observed in the pillar both in the SEM image (Figure 1) and as evidenced by the orientation change indicated by the EBSD map (Figure 2). An initial inspection of the post-compression SEM image (Figure 1) shows surface steps near the free end of the pillar, which could have been mistakenly attributed to dislocation slip bands. However, a subsequent high-resolution inverse pole figure (IPF) map of the top of the pillar was then acquired, shown in Figure 3. This region corresponds to the area outlined by the blue rectangle in Figure 1. The IPF map and pole figure in Figure 3 reveal the presence of two deformation twins which formed during compression.

The relatively large amount of plasticity observed in this pillar is unlike the behavior observed in bulk GaAs, which typically shows very little room-temperature ductility. This plasticity can be explained by the geometry and small volume of the pillar. Dislocations are known to move on {111}-type planes in GaAs, with full dislocations dissociating into two partials. In small volumes the leading partial dislocation, which has a higher Schmid factor than the trailing partial, can cut through the entire crystal and annihilate at the pillar surface before the trailing partial moves, creating an intrinsic stacking fault. Twin boundaries can form by partial dislocations passing on neighboring (111) planes. This mechanism is responsible for the large plasticity observed at this small length scale.

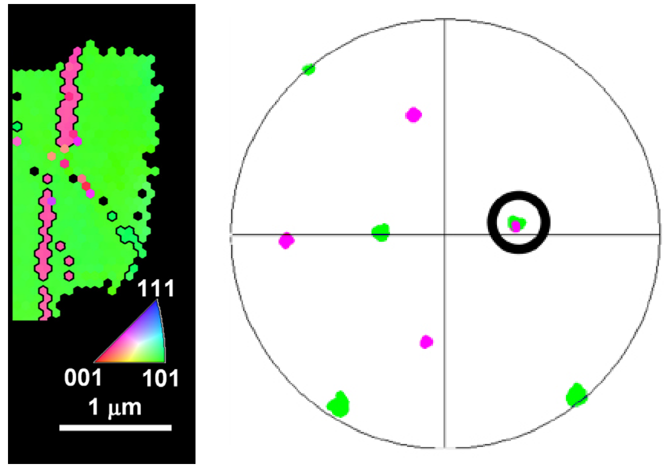

Figure 3: Inverse pole figure map (left) and <111> pole figure (right) illustrate the formation of two twinned areas created in the end of the pillar.

Conclusion

This work (1) demonstrates the first *in-situ* EBSD measurements acquired during compression of a micropillar in the SEM. Furthermore, by analyzing the EBSD data using advanced cross-correlation methods described in Ref. 2, it is possible to determine the full stress and strain tensors at each point. The combination of these three high-resolution techniques provides a complete picture of the behavior of the pillar before, after, and during testing.

References:

1 Niederberger, Mook, Maeder, and Michler, Mater Sci Eng A, 2010.

2 Maeder, Mook, Niederberger, and Michler, Philos Mag, 2010.