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APPLICATION NOTE

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Orientation Mapping of Thin Films Under Loading*

In-Situ Tensile Testing Using Push-to-Pull (PTP) with PI 95 TEM PicoIndenter®

Nanocrystalline Thin Films

Nanocrystalline materials exhibit superior mechanical properties such as hardness, strength and fatigue, compared to their coarse-grained counterparts. However, due to strain localization and low strain hardening, nanostructured metals and alloys often show poor ductility. To correlate deformation mechanisms to grain orientation and size changes, a new approach of imaging while deforming a thin film in tension using a **Push-to-Pull (PTP)** device was developed, Fig. 1. Automated crystal orientation mapping (ACOM) in microprobe STEM mode was applied to quantify microstructural changes at high resolution. The **PTP** device, an in situ tensile apparatus, was designed to work seamlessly with Hysitron's commercially available **PicoIndenter** instruments for TEM and SEM.

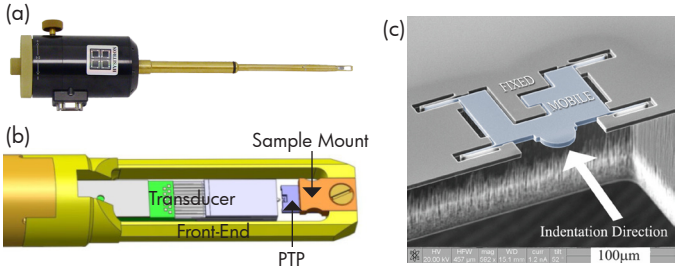

Figure 1: (a) TEM **PI 95**, instrumented nanomechanical test holder, (b) Front-end, transducer and sample mount, and (c) **PTP**.

Sample Preparation - Thin Film on PTP

Gold thin films were deposited on carbon coated mica by RF magnetron sputtering using a target of Au with 99.99% purity. The film showed {111} texture in the growth direction. To transfer the film from mica onto the **PTP**, the sample was immersed in a deionized water-ethanol mixture to release the thin film from the substrate, and the film floating on the surface was picked up by a **PTP** device. The spring system of the **PTP** was recovered by cutting the film along the trenches using FIB, Fig. 2a. FIB was also used to cut a dog-bone shape sample of approximately 2 µm in width and 2.4 µm in length, Fig. 2b.

PI 95 with Automated Crystal Orientation Mapping

The **PI 95 TEM PicoIndenter**® was used to conduct *in-situ* strain-rate controlled tensile experiments. After thin film sample preparation, the **PTP** device was mounted to the **PI 95** sample mount, and the sample mount was attached to the front-end of the instrument, Fig. 1. An FEI Tecnai F20 ST operated at 200 kV in µpSTEM mode and equipped with an ASTAR system (NanoMegas) was used for ACOM-TEM data acquisition. Using spot size 8, gun lens 6, extraction voltage of 4.5 kV and 50 µm C2 aperture, a probe with ~1 nm diameter and a convergence semi-angle of 1.4 mrad was generated.

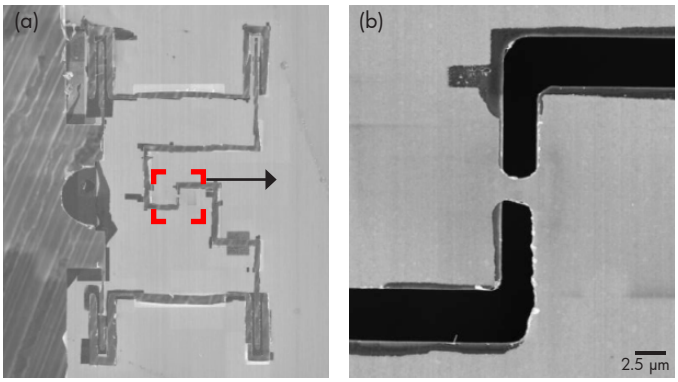

Figure 2: (a) Low and (b) high magnification images showing Au thin film on **PTP** *[1].

A maximum final strain of 9.7%, which was directly measured in STEM, was applied to the thin film with a strain rate of $6x10^{-4}$ 1/s. At the end of each displacement ramp, 45 minute hold segments were allowed for image acquisition. Two distinct regions were observed during loading, Fig. 3: initial straining up to 4.5% did not show any significant load increase indicating that the film was unbending (unfolding or straightening the bent film), and then the load increases rapidly indicating that the film is under tensile stress.

*[1] **Combination of in-situ straining and ACOM TEM: a novel method for analysis of plastic deformation of nanocrystalline metals**, A. Kobler, A. Kashiwar, H. Hahn, and C. Kübel, *Ultramicroscopy*, **128**, 68-81 (2013); DOI: 10.1016/j.ultramic.2012.12.019.

Mechanical Properties and Grain Size Distribution

Load-displacement data was recorded after the film fractured to determine the stiffness of the **PTP** device, which was used to calculate the actual force applied to the film and to obtain the stress–strain curve. The maximum load on the film at the point of rupture was 229 µN corresponding to an ultimate tensile strength of 1.6-2.0 GPa. This range of UTS is in reasonable agreement with other published results.

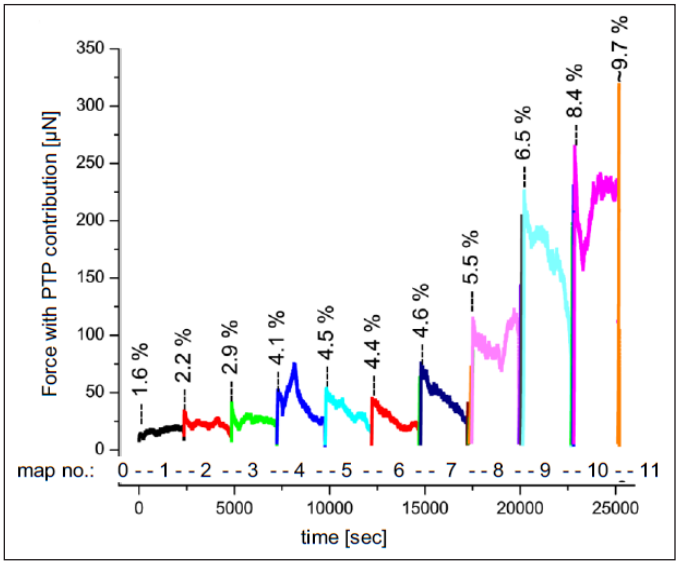

Figure 3: Load versus time plot with strain% and ACOM captured map numbers *[1].

The orientation maps before deformation showed a typical bimodal structure of the nanocrystalline films. Quantitative analysis revealed an average crystallite size of ~37 nm including a small number of anomalous growth grains with ~150 nm diameter. The global grain size evolution shows no significant change in grain size during initial loading (straightening the film), and an increase of ~2.5% during actual sample loading. The grain growth was also accompanied by a change in overall grain shape from an initially slight elliptical shape with the minor axis parallel to the straining direction to equiaxed after straining.

Grain Orientation and Texture

Even though the global grain size analysis initially showed no significant increase in grain size, local analysis of the orientation maps revealed that individual grains started deforming plastically already during the early stages of loading. For example, the orientation maps showed that big grains grow by "eating" small grains already during "unbending".

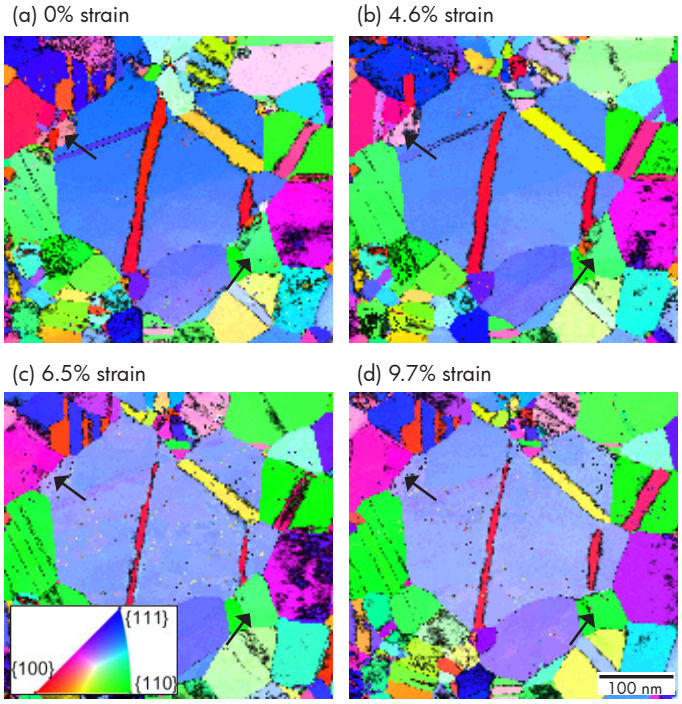

Figure 4: Microstructural evolution with increasing tensile strain indicates smaller grains (indicated by black arrows) are merging with the large blue grain. Inset shows color code of crystal orientation *[1].

The size of grain 1 in Figure 4 decreased by approximately 6% during the "unbending" stage, which was followed by another 20% shrinkage during the "loading" stage. This can be described as micro-plasticity, which accommodates the early stages of deformation, followed by grain growth as part of macro-plastic deformation. Furthermore, it was observed that twinning and detwinning occurred simultaneously in different grains throughout the deformation experiment, and grain rotation of individual grains was observed during later stages of straining to accommodate the deformation.

Conclusion

In this study, a new method of imaging and quantifying crystallographic changes in nanocrystalline thin films during tensile deformation has been presented. The combination of **PI 95** with **PTP** and ACOM-TEM provides a reliable local and global method of orientation mapping with significant advantages over classical BF/DF TEM and HRTEM imaging. This approach can potentially be applied to a wide range of nanocrystalline materials with a variety of dimensions and shapes.

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