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In Situ TEM Investigation of the Deformation Mechanisms and Microstructural Changes in Ultrafine-grained Non-textured Aluminum Film Using Automated Crystal Orientation Mapping

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Automated crystal orientation mapping in TEM (ACOM-TEM), with a precessing nanoprobe electron beam scanning over the specimen to collect spot diffraction patterns, is highly suitable to study the deformation mechanisms and monitor the microstructural evolution of UFG/NC metals during deformation. This technique enables direct acquisition of orientation/phase map over micron-sized areas while enhancing the ability to identify grains, microtexture and twin boundaries using the orientation maps of the sample that are extracted from the indexed diffraction patterns.

In this study, we use ACOM-TEM in combination with quantitative in situ TEM load-unload straining experiments (Fig. 1a) using a custom MEMS device to track orientation changes in hundreds of grains in a freestanding non-textured, UFG aluminum film (thickness 200 nm, mean grain size 180 nm) and correlate those changes with the macroscopic stress-strain response of the film [2-3].

Our results show extensive grain orientation changes during loading, with both the fraction of grains that undergo rotations and their magnitude increasing with strain. The rotations are reversible in a significant fraction of the grains during unloading, leading to notable inelastic strain recovery [2-4]. More surprisingly, a small fraction of grains rotate in the same direction during both loading and unloading, even though the applied stress is substantially different. At 1.9% strain during loading, more than 70% of the grains experienced rotations ranging from 0.2° to 6°. Similarly, more than 60% of the grains underwent rotations during unloading as the sample strain was decreased from 1.9% to 1.6%. Among the grains that showed in plane rotations during unloading, about 49% of the grains underwent reverse rotations. The rest of the grains exhibited uncorrelated rotations (31%) or forward rotations (20%), which was consistent with redistribution of stresses triggered by reverse yielding of plastically soft grains.

The ACOM-TEM measurements also provide evidence for reversible as well as irreversible grain/twin boundary migration in the film. Several grains showed an increase/decrease in size during loading, which is consistent with previous studies on deformation induced grain growth in UFG and NC metals. However, we observed changes in grain size even during unloading, when the applied stress had been considerably reduced. Figs. 1 provide two different examples of this phenomenon. Fig. 1c shows a grain that reduced in size while loading, continued to shrink during unloading, and was completely annihilated. Fig. 1d, in contrast, shows a grain that exhibited reversible growth. The size of this grain increased during loading by ~ 33% (from ~9300 nm2 to ~12,400 nm2) but shrunk (to 6700 nm2) when the sample was unloaded. We also observed that the length fraction of Σ3 boundaries increased from 10% (at 0.7% strain) to ~20% (at 1.9% strain) during loading and it decreased to ~15% after unloading to 1.6% strain. However, continuous detwinning during both loading and unloading was also observed in some grains (Fig. 1e).

Overall, the microstructural observations point to a spatially inhomogeneous stress distribution in the film that constantly evolves during both loading and unloading. The presence of these reverse and forward grain rotations during unloading is also consistent with the qualitative observations of the evolving microstructure. As shown in Fig. 1, there is evidence of both reversible and irreversible migration of grain/twin boundaries. It has been shown that these grain boundary migrations are primarily driven by the local stress state. Therefore, a reverse migration of a grain boundary during unloading likely reflects a reversal in the local stress state. In contrast, the continued migration of a grain/twin boundary would suggest that the local stress state remains similar to that during loading [5].

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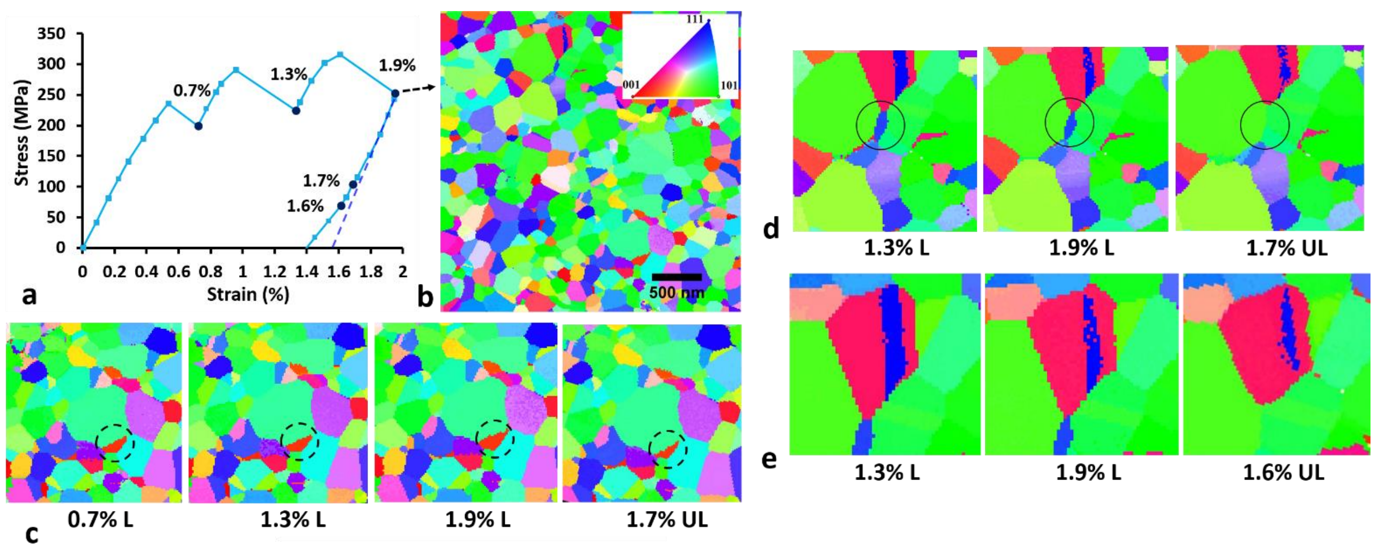

Figure 1. a) Stress-strain response of non-textured UFG Al film during in situ ACOM-TEM experiment. b) ACOM- map showing the color-coded out of plane orientation of the grains in the scanning area. c) Reversible change in size of a grain (marked by the black dashed circle) during loading and unloading. The size of the grain increased during loading but reduced as the sample was unloaded. d) Progressive reduction in size and complete annihilation of a grain and reversible change in size of another grain (marked by the black circle) during loading and unloading, respectively. e) Detwinning in a grain during loading and unloading. ‚L' denotes loading and 'UL' denotes unloading in the figure.

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