Experimental Techniques for Uncovering Deformation Mechanisms in Nanocrystalline Al Thin Films

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Abstract

Nanocrystalline materials have been shown to be harder and stronger than their coarse-grained counterparts, but what is not fully understood is how these materials deform. Attempts to characterize the mechanisms that govern the mechanical response are inhibited by the challenges associated with obtaining reasonable volumes of material to test. Here we report on the fabrication of nanocrystalline thin films with vapor deposition and the development of techniques for mechanical testing of submicron thin films. Samples, in which a Si frame is utilized for ease of handling of the specimens, were fabricated using MEMS-inspired techniques, and results for uniaxial tensile tests of ~380 nm thin film aluminum samples with an average grain size of 36 nm are presented.

Introduction

The mechanisms by which nanocrystalline (nc) materials (i.e. grain sizes that are < 100 nm) accommodate plastic deformation are different from those observed in their coarse-grained counterparts. Molecular dynamics simulations and direct TEM observations [1-3] point to an apparent change in the underlying mechanism that controls plastic deformation in materials with grain sizes that are less than ~50 nm. A transition occurs from normal dislocation slip to grain boundary-mediated events and partial dislocation activity.

Previous attempts at synthesizing nc materials using multiple step processing techniques have yielded samples that are not fully-dense [4]. As a result, a one-step processing method, namely sputtering, was employed to create high quality thin film microsamples of nc-Al. Materials with dimensions at these length scales are readily utilized in the emerging field of microelectromechanical systems (MEMS) and the properties must be thoroughly evaluated to ensure reliability of these devices. Paradoxically, the small sizes associated with these films impart technical challenges for mechanical testing. To elucidate the mechanical response and deformation mechanisms of this class of material, a unique Microsample tensile testing apparatus was developed and implemented to conduct mechanical tests of nc metals.

Experimental Methods

Material Synthesis and Specimen Fabrication

Fabrication techniques inspired by the microelectro-mechanical systems (MEMS) industry were employed to create microtensile freestanding thin film samples as illustrated in Fig. 1. These methods allow for the creation of high purity, high quality films, well-defined sample geometry, and batch processing. The processing was performed on 3" (001) Si wafers in order to create a "frame" for handling such fragile submicron films, as shown in [5]. First, a 4 μm thick thermal SiO_2 layer was grown on the Si wafer and used as a mask for backside window etching. Second, using KOH as an anisotropic etchant, a window was defined on the backside of the specimen that allows for the release of the gage section. The backside etch was stopped with ~ 50 μm of Si remaining. Third, a liftoff technique was employed to pattern the aluminum geometry on the frontside. Finally, a pulsed dry gas etchant of XeF_2, which selectively (1000:1)

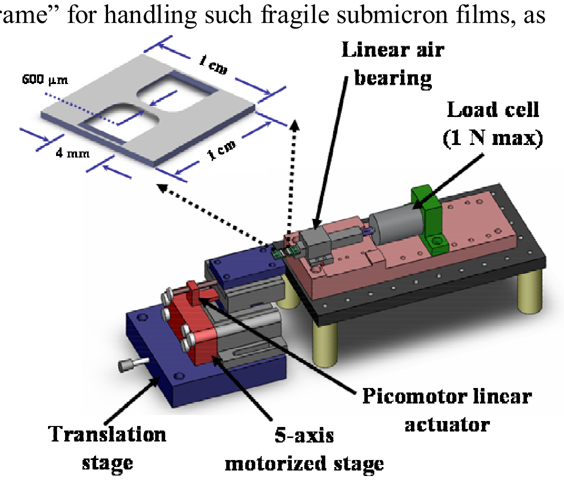

Fig. 1: Schematic of microtensile testing setup and sample geomtry.

reacts with Si over Al, was used to remove the supporting Si layer. Since dicing of a brittle wafer can be cumbersome, lines were patterned around the outside of the specimen with a thickness such that the KOH etchant closed a v-groove channel at a calculated depth; greatly simplifying separation of the dies. Physical vapor deposition methods are viable means of synthesizing high quality nanocrystalline films using one-step processing, but impart a technical challenge in terms of mechanical testing since the films must be kept submicron in order to keep the grain size in the regime of interest. DC-magnetron sputtering of an Al target (99.999% purity) was used with pulsed deposition to minimize the growth of columnar grains through the thickness of the film. Fig. 2 shows the resulting microstructure of the Al films and the cross-sectional morphology. Measurements from ~250 grains exhibit a lognormal distribution of grain size, with an average value of 36 ± 16 nm. X-ray pole figure analysis evidenced the lack of any strong texture, but the presence of weaker textures could not be ruled out since the thin films yield such low diffracted intensity.

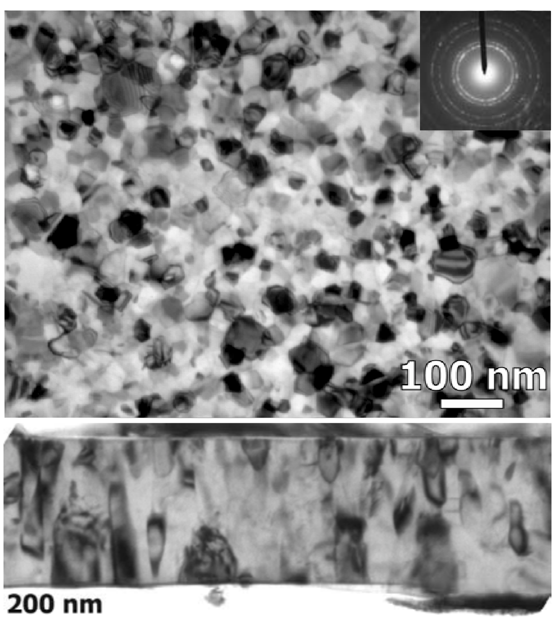

Fig. 2: TEM images of nc-Al films: (a) plan view and (b) cross-sectional view, showing slightly columnar morphology.

Mechanical Testing

The forces incurred in a tensile test of films of this size are very low (~0.06 N), and the precise alignment of the tensile axis with the loading apparatus is of paramount importance. To ensure alignment, a unique mechanical testing setup was designed and constructed, as shown schematically in Fig. 1. A fixed grip is attached to a high-resolution load cell (max. load = 1 N) and a second grip is attached to a piezoelectrically-actuated screw-driven linear motor. This grip was mounted on a 5-axis picomotor stage, which allows for precise alignment (with the aid of multiple microscopes). A UV-curable adhesive was used to fix the sample to the grips, and a rotary diamond blade was used to cut the Si support strips of the frame prior to testing. A linear air bearing provided nearly frictionless loading, and displacement was measured with a capacitance gage. All experiments were conducted at a rate of $\dot{\varepsilon} = 1 \times 10^{-4}\ s^{-1}$.

Results and Discussion

Representative stress-strain curves are presented in Fig. 3 for two Al samples of slightly different thickness of the same processing batch. As shown, the material exhibits high strength and significant ductility. A sharp instability is marked by the decrease in stress at higher strains, and manifests itself in the form of localized necking in the center of gage section. A deviation from the linear elastic regime is observed prior to a plateau in the stress. The extended plasticity in these samples implies that dislocation sources may still be active in this grain size regime. It should be noted that the columnar structure (although many areas show several grains through the thickness of the film) of the grains could induce free surface effects, which may not be representative of bulk nc-materials.

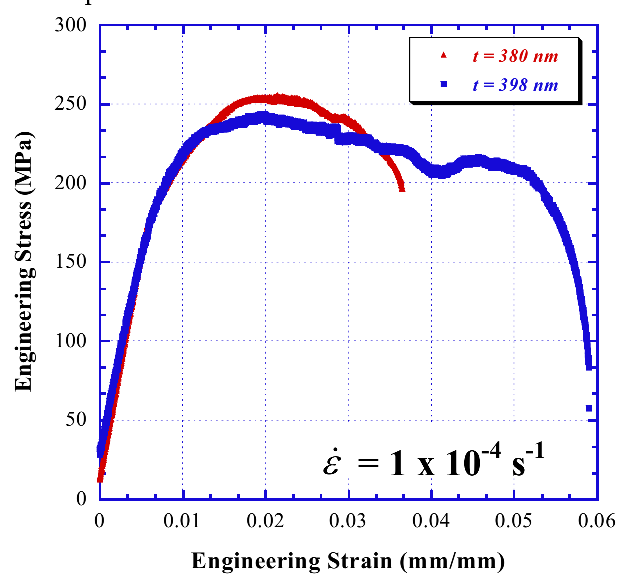

Fig. 3: Stress-strain curves of Al thin films for two thicknesses.

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