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Regular Article

Texture dependent strain rate sensitivity of ultrafine-grained aluminum films

Ehsan Izadi, Jagannathan Rajagopalan *

Department of Mechanical and Aerospace Engineering, School for Engineering of Matter, Transport and Energy, Arizona State University, Tempe 85287, USA

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ABSTRACT

We investigated the strain rate sensitivity (SRS) of freestanding ultrafine-grained aluminum films with similar thickness (~240 nm) and mean grain size (~275 nm) but highly dissimilar texture. For (110) textured bicrystalline films, flow stress increased by 14% ($m = 0.017$) as the strain rate was varied from ~7×10^{-6}/s to 5×10^{-3}/s. In contrast, for non-textured films the flow stress increased by more than 90% ($m = 0.103$) over a similar strain rate range. The drastic difference in SRS can be explained by texture-induced changes in deformation mechanisms of the films, as revealed by *in situ* TEM straining experiments.

Ultrafine-grained (UFG) and nanocrystalline (NC) metals exhibit a number of appealing mechanical properties such as high strength and increased wear resistance [1]. Metallic thin films with UFG/NC microstructures are employed in numerous applications including structural coatings, interconnects in semiconductor devices, in thermomechanical applications and as structural and electrode components in micro-electro-mechanical systems (MEMS) [2–4]. In many of these applications, and particularly in MEMS, the metallic films may be subjected to a wide range of loading rates. For instance, in MEMS based high frequency oscillators or thermomechanical switches the actuation times can vary from microseconds to milliseconds.

It is known that the mechanical response of UFG/NC metal films depends on the rate at which they are deformed. Therefore, in order to gain reliable operation of MEMS, the mechanical behavior of metal films over various loading rates needs to be understood [4,5]. Strain rate sensitivity (SRS) studies of thin metal films have typically focused on mechanical behavior at rates between 10^{-6}/s and 10^{-3}/s [6–9], but more recently a wider range of strain rates [4,10–12] have been explored. SRS studies of UFG/NC metals have been mainly directed towards elucidating the effect of mean grain size on their rate dependent mechanical response. These studies have shown that across a wide range of metals SRS increases and activation volume decreases [6,7,10,13] as the grain size becomes finer.

But in addition to the mean grain size, texture is also known to significantly influence the deformation behavior of UFG/NC metal films. Torre et al. have shown that differences in texture lead to variation in the yield stress and ultimate tensile stress of NC nickel foils [14]. Similarly, experiments on nanoscale aluminum (Al) films with similar mean grain size and thickness but different textures have revealed significant differences in flow stress and Bauschinger effect [15,16]. However, the effect of texture on the SRS of metallic films has not been systematically investigated so far.

In this study, we investigated the SRS of two sets of nanoscale Al films with nearly identical thickness (~240 nm) and mean grain size (275–285 nm), but substantially different textures. Our results unambiguously show that SRS of UFG Al films is strongly dependent on film texture. Films with a strong (110) out-of-plane texture and composed only of two in-plane grain variants (bicrystalline films) show significantly smaller SRS compared to films with a random orientation of grains (non-textured films). The flow stress of the bicrystalline films increased by 14% when the strain rate was increased from ~7×10^{-6}/s to 5×10^{-3}/s. In contrast, the flow stress of non-textured films increased by over 90% over a similar strain rate range.

The bicrystalline Al film (labeled as textured film) and the non-textured Al film were synthesized by carefully controlling the deposition conditions. To obtain the textured film, the native silicon dioxide layer on a (001) oriented silicon (Si) wafer was removed through hydrofluoric acid etching and the wafer was immediately transferred to the sputtering chamber to avoid regrowth of the oxide layer. Al was then deposited on the bare Si wafer using DC magnetron sputtering,

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* Corresponding author.
E-mail address: jrajago1@asu.edu (J. Rajagopalan).

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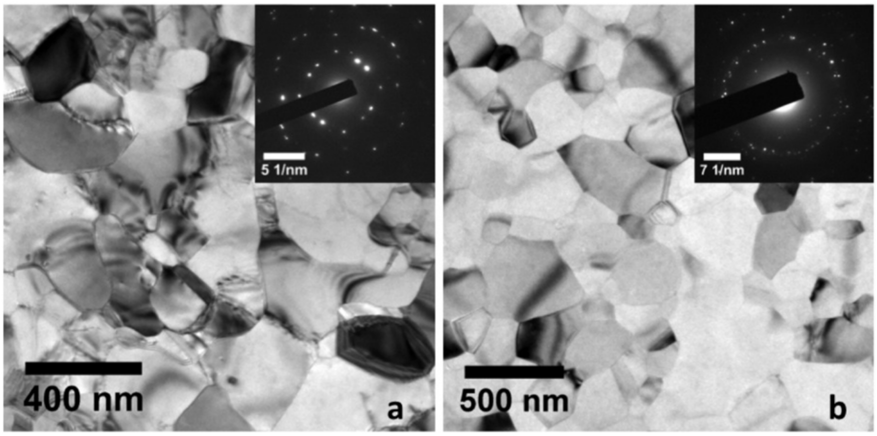

Fig. 1. (a) Bright-field TEM image of the textured Al film with a mean grain size of 275 nm. Selected area diffraction (SAD) of the film showing an (110) out-of-plane texture with two in-plane variants rotated 90° with respect to each other (inset). (b) Bright-field TEM image of the non-textured Al film with a mean grain size of 285 nm. Selected area diffraction (SAD) of the film showing the lack of texture (inset).

which resulted in heteroepitaxial growth with the following relationship: Al(110)//Si(001), Al[001]//Si[$1\bar{1}0$] and Al(110)//Si(001), Al[001]//Si[110] [17]. Thus, this film consists of just two grain families with (110) out-of-plane texture which are rotated 90° in plane with respect to each other. The non-textured film was obtained using a similar process except that it was sputter deposited on a Si(001) wafer with the native silicon dioxide layer intact. The oxide layer disrupts the epitaxial growth of Al, leading to a film with random orientation of grains. Both the textured film and non-textured film were deposited to a thickness of ~240 nm at 5.5 nm/min. The chamber base pressure during deposition of the textured and non-textured film was 8×10^{-8} Torr and 3×10^{-7} Torr, respectively.

The microstructure of the films was examined through transmission electron microscopy (TEM) and X-ray diffraction (XRD). Based on plan-view TEM images, the textured Al film had a mean grain size of 275 nm and the non-textured film had a mean grain size of 285 nm (Fig. 1). The images also indicated a columnar grain structure with one grain traversing the thickness of the film. To perform the experiments, dog-bone shaped freestanding samples were co-fabricated with MEMS based tensile testing devices (Fig. 2a) using microfabrication techniques outlined in [18]. The MEMS devices have built-in gauges to track the sample deformation. A piezoelectric actuator (Physik Instrumente) was used to load the devices and a CMOS camera (Thor Labs) was used to acquire images of the gauges during the experiments. A custom MATLAB™ program, which tracks prescribed features across a series of images using cross-correlation techniques, was used to measure the displacement of the gauges and thus the sample strain.

A miniature s-beam load cell (Futek), arranged in series with the MEMS device, measured the total force (F_{tot}) on the device (Fig. 2b). As evident from the equivalent mechanical model of the device, $F_{tot} = F_S + F_U + F_A$, where F_S is the force on the sample and F_U and F_A are the force on the U-beams and the Alignment beams, respectively. The combined stiffness (K) of the U-beams and Alignment beams was measured in a separate experiment after the sample had fractured. K multiplied by the displacement (x) gives $F_U + F_A$, from which F_S and thus the stress on the sample was obtained.

In all the experiments, loading was along the [001] direction of the Al[001]//Si[110] grain family for the textured film and along an arbitrary direction for the non-textured film. To calculate the uncertainty in stress

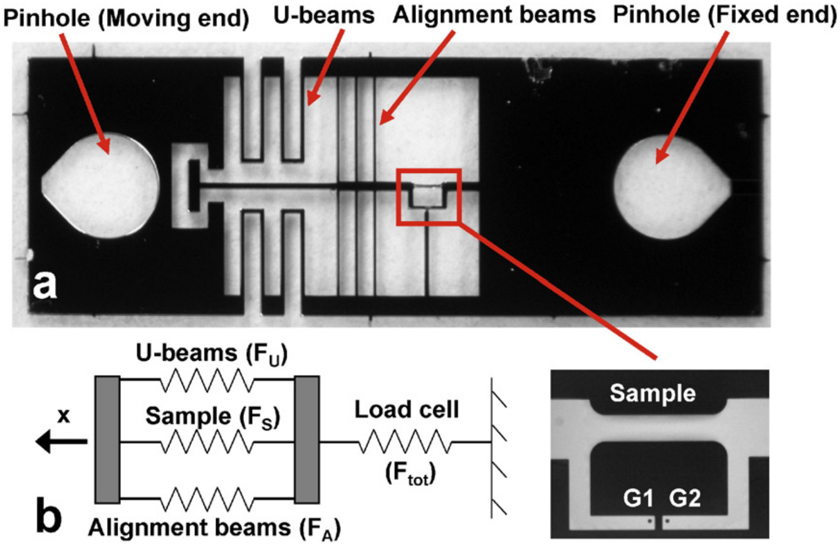

Fig. 2. a) MEMS device for performing constant strain rate experiments on freestanding metal film samples. The nominal strain on the sample is obtained by tracking the displacement of gauges G1 and G2. b) Equivalent mechanical model of the MEMS device. The U-beams and Alignment beams are in parallel with the sample and hence their displacement (x) is the same. The load cell is arranged in series with the MEMS device.