Supporting Online Material for

Plastic Deformation Recovery in Freestanding Nanocrystalline Aluminum and Gold Thin Films

Jagannathan Rajagopalan, Jong H. Han, M. Taher A. Saif*

*To whom correspondence should be addressed. E-mail: saif@uiuc.edu

DOI: 10.1126/science.1137580

This PDF file includes:

Materials and Methods
Figs. S1 to S3
References
Supporting Online Material

Materials and Methods

200 nm thick aluminum/gold films are sputter-deposited on a p-type Si wafer with a [100] out of plane orientation. Before deposition, the wafer is cleaned of organic residue as well as it native oxide and is baked to dehydrate. Then, the wafer with aluminum/gold film on top is optically patterned and subjected to anisotropic deep Si etching to produce the micro-electro-mechanical systems (MEMS) based tensile testing chip along with the freestanding aluminium/gold specimens. The features of the tensile testing chip and its detailed fabrication procedure are described in (S1). During deposition, the base pressure in the sputtering chamber is $5 \times 10^{-7}$ Torr and the purity of the aluminium and gold target used in sputtering is 99.999 % and 99.99 % respectively.

The schematic of the experiment is shown in Fig. S1. The freestanding specimens are held at one end by a Si force sensor and at the other end by a piezo actuator. Both the aluminum and gold specimens are initially buckled due to compressive residual stresses in as-fabricated condition. The compressive stresses in the specimens after buckling are negligible ($< 0.1$ MPa). Gauges G0, G1 and G2 are used to measure the force and strain in the sample. The change in gap between G0 and G1 gives a measure of the force, while the change in gap between G1 and G2 provides the elongation (strain) of the specimen.

The experiment starts with noting the initial distance, $b$, between the gauges, G1 and G2, when the specimen is buckled (state 1). The piezo is then actuated by increasing the voltage until the specimen straightens and the force sensing gauge, G1, just begins to move with respect to G0.
(state 2, G1-G2 gap = s), i.e., the specimen starts getting stressed. The voltage is then increased to deform the specimen to a predetermined strain level (state 3, G1-G2 gap = m). The specimen is then unloaded by lowering the voltage until the specimen starts to buckle and the force sensor indicates zero external stress (state 4, G1-G2 gap = p). Thus p-s is the plastic deformation and m-s is the total deformation. The chip is then completely unloaded so that the specimen buckles out. Then, the specimen is annealed by placing the chip on a hot plate while the chip temperature is monitored using a thermocouple. Strain recovery due to annealing is detected if p decreases after annealing; plastic strain is fully recovered if p-s goes to zero. The gauges are observed at high magnification during the experiments, leading to a strain and stress resolution of 0.005 % and 10 MPa for the aluminum specimens and 0.01 % and 1.5 MPa for the gold specimens.

During the experiments on the aluminum samples, the freestanding specimens were loaded at constant strain rate of ~ 1×10^{-4}s^{-1} by appropriately increasing the voltage on the piezo. The loading was halted at various points during deformation by holding the voltage constant for five minute periods to check for stress relaxation or creep. No stress relaxation or creep was seen during any experiment. The unloading rate for all experiments was ~ 1×10^{-3}s^{-1}. The same procedure was used for the gold specimens as well, except that the loading/unloading was halted for 2 minute periods instead of 5 minute periods at each point. No stress relaxation or creep was seen in this case as well.
Figure S1

Figure S1 displays the schematic (side view) of the experimental procedure. The specimen is shown along its length. It is initially buckled due to compressive residual stress. Upon actuation, the piezo first straightens the sample, and then strains it. The three gauges, G0, G1 and G2 provide a measure of stress and strain in the sample. State 1: The specimen has negligible compressive stress ($\sigma = 0$). State 2: specimen is straightened, but not strained (the gap between G0 and G1 has not changed compared to state 1), $\sigma = 0$. State 3: Specimen is deformed by $m$-$s$. G1 moves to the right giving the force on the sample ($\sigma = \sigma_m$). State 4: Specimen is unloaded, but kept straightened. G1 returns to its load free state ($\sigma = 0$). The plastic deformation is given by $p$-$s$. A reduction of $p$-$s$, after annealing, gives strain recovery in the specimen. Note that $p$ is measured by first letting the specimen buckle (by completely unloading the piezo) and then straightening it.

![Figure S1 schematic diagram](image-url)
Figure S2

Figure S2 displays the bright field transmission electron micrograph of the aluminum sample in (A) the as-fabricated condition and (B) after deformation and annealing at 240 °C, indicating no change in grain size on deformation or annealing.

![Figure S2](image)

Figure S3

Figure S3 displays the atomic composition, obtained through Auger electron spectroscopy, of the aluminum films in (A) the as-fabricated state and (B) after annealing at 240 °C, showing no major change in the impurity content on annealing. The composition of the oxide layer on the surface has been omitted.