Thin Film Bulge Test of Nanocrystalline Metals

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Abstract
Free-standing nanocrystalline copper (Cu) films with grain size around 39 nm are fabricated by thermal evaporation and characterized by the plane-strain bulge test. Young’s modulus and yield stress at a 0.2% offset are about 110 ~ 130 GPa and 400 MPa, respectively. Results show that no grain growth is observed and the predominant plastic deformation mechanism is grain boundary sliding accompanied by dislocation mechanisms within the grains to accommodate grain boundary sliding.

Summary of Research
In the present study, nanocrystalline copper (n-Cu) thin films are synthesized by thermal evaporation and the mechanical properties are explored using the “plane-strain bulge test” introduced by Vlassak and Nix [1]. The results are compared to previously published studies on the mechanical properties of similar free standing thin films with a micrometer grain size [2] and with nanometer grain size [3].

The n-Cu films are prepared by thermal evaporation (Edwards Auto 306 Vacuum Evaporator). The base pressure in the chamber is 1 x 10^-6 Torr and high purity (99.995%) Cu is used. A current of 2.4 A is applied through the tungsten boat to melt the Cu source which yields a deposition rate of 0.1~0.2 nm/s. During the process, the substrate temperature is no higher than 70°C.

As shown in Figure 1, the fabrication procedure is modified from that of Xiang et al. [4]. A (100) Si wafer with a 500 nm thick Si₃N₄ layer deposited on both sides by low pressure chemical vapor deposition (LPCVD) is used. Positive photo-resist AZ5214 (CLARIANT), ultraviolet photolithography (wavelength of 365 nm) and reactive ion etching (RIE, 250 mTorr, 250 W in CF₄/O₂ atmosphere, TECHNICS Series 800) is employed to open a rectangular window through the Si₃N₄ on one side of the wafer. Next, the Si wafer is anisotropically etched in 30 wt.% potassium hydroxide (KOH) solution at 80°C until a free-standing rectangular Si₃N₄ membrane remains on the opposite side of the wafer. The n-Cu is then deposited on the Si₃N₄ membrane by thermal evaporation and another RIE etching with lower pressure and power (150 mTorr and 150 W) is used to remove the Si₃N₄ so that a freestanding n-Cu film is obtained. A schematic of the specimen is shown in Figure 2.
Materials

Scanning electron microscope (SEM) micrograph and x-ray diffraction (XRD) studies show the average grain size of the specimens are in the range of 36 nm ~ 41 nm.

Figure 3 shows the plane-strain stress-strain curves for the six n-Cu films examined and three curves of the coarse-grained Cu films from [2] are also included for comparison. The Young’s moduli of the n-Cu films, compiled in Table 1, are in the range of 111 ~ 132 GPa, in good agreement with published values [5].

![Figure 3: Plane-strain stress-strain curves of six n-Cu films.](image)

There does not seem to be a pronounced thickness dependence of the yield stress for the n-Cu films as there is for the coarse-grained films at the strain rate of $5 \times 10^{-6}$ s$^{-1}$. Most of the specimens show a consistent range of 410 ± 10 MPa. As shown in Figure 3, the n-Cu films can achieve a ultimate stress of about 450 MPa, which is 50% higher than that of the coarse-grained Cu, 300 MPa, reported in [2]. Moreover, the results show that the n-Cu has less macroscopic ductility than the coarse-grained thin films and there is no obvious yield point in the data of Figure 3.

Figure 4 shows the progression of a crack-like feature near the edge of the free standing film. There is an uplift of material near the tip consistent with a Mode III ripping behavior. This is evidence of grain boundary sliding, however close inspection reveals a series of highly planar strain localizations which pass through several grains parallel to the Mode III crack tip, which suggests the possibility of dislocation activity. The deformation mechanisms associated with both types of failure are consistent with studies [6, 7] which state the deformation mechanism in n-Cu with grain sizes of those in the present experiments are thought to be grain boundary sliding accompanied by dislocation mediated plasticity within the nanoscale grains to accommodate grain boundary sliding.

Gianola et al. [3] reported two classes of stress-strain response for n-Al. The specimens in the present study are quite similar to one class which had been deposited using sputter deposition at a similar vacuum to present study ($10^{-6}$ Torr). The specimens exhibited a high yield stress, and limited ductility due to shear localization which occurred at a very low level of plastic strain. The authors [3] show that the differences in response can be attributed to an effective absence of grain growth in specimens while stress-assisted grain growth occurred in another class of specimens which had been deposited at higher vacuum. That, combined with an absence of perceptible grain growth in our specimens, suggests that impurities in the grain boundaries tend to pin grain boundaries to limit grain growth just as in [3, 8].

**References**