MECHANICAL BEHAVIOR OF NANO GRAINED METALS

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Summary: The paper presents (1) a novel instrumentation for studying mechanical behavior of nano scale free standing thin films in-situ in Scanning or Transmission electron microscope, as well as under optical microscope, (2) results on mechanical behavior of nano grained Al and gold films (3) mechanistic interpretation of the experimental data.

Thin metal films are extensively used in electronics and micro/nano mechanical systems where components are often less than 100nm. These metal structures are typically poly crystalline in nature with abundant grain boundaries. The fundamental role of such boundaries in determining the thermomechanical properties is not well understood, a study that is often challenged by the limitations of the instrumentation that allows testing nano structures.

Experimental procedure: We have developed a micro mechanical experimental method to study the stress-strain response of free standing thin metal films with thickness 20 nm or higher. It allows in-situ observation of the microstructue of the sample in transmission electron microscope (TEM) when the sample's stress-strain behavior is measured. Hence, the fundamental micro structural mechanisms that give rise to the mechanical behavior can be investigated. Figure 1 shows the SEM micrograph of the micro apparatus. It is a tensile test stage made of Single Crystal Silicon (SCS), 10mm long, 3mm wide and 100μm thick. The freestanding sample is made of sputtered Aluminum [1]. One end of the sample is attached to a force sensor beam, A_1A_2, while the other end to the support beams B_1B_2. A piezo motor is employed to stretch the stage along its longitudinal direction which strains the sample. Two markers gaps, A and B, measure the force and stretch of the sample: marker A gives the displacement of the beam A_1A_2, and hence the force on the sample (spring constant of A_1A_2 is calibrated using a nano indenter), and the difference between the gaps A and B gives the stretch of the sample.

Results: We have successfully carried out in-situ uniaxial tensile experiment in TEM on 100nm thick Al film while its stress-strain response is measured. Figure 2 shows the quantitative stress-strain response and the in-situ transmission electron micrograph at regions C and D of the stress-strain curve. To the best our knowledge, this is the first time that such quantitative measurement and qualitative observation of material's microstructure have been performed. Note that the grains are mostly less than 100nm, there are very few dislocations and they do not generate during straining, but grains rotate with increasing strain as is evident from the change of contrast of the same grain under increased loading. Furthermore there is no evidence of voids or intergranular cracks in the film.

We have employed the instrument to test Aluminum films with grain sizes 10-80nm with the corresponding thicknesses of 30-200nm. The results are shown in Table 1. We note that as grain size decreases, (1) elastic modulus decreases, (2) yield stress increases and reaches a maximum value when the grain size is 50nm. With further decrease of the grain size, yield stress decreases. We also find that metal shows non-linear elastic behavior with small plastic deformation when grain size is small. Experiments on gold films show similar behavior.

Interpretations: As grain size decreases, grain boundary to volume ratio increases. Atomic arrangements near the boundary region is distorted compared to the granular counterpart to accommodate the misfit between the two adjacent grains. The distorsion is induced by various planar defects such as dislocations, steps and lattice dilations which result in a region with lower elastic modulus. Thus, as the metal is strained, due to higher elastic compliance, the boundary region takes more strain that the granular counterpart. With increasing strain, the boundary region becomes more distorted, and its compliance increases. Thus, the overall metal response becomes increasingly softer. Upon unloading, the metal returns to the unloaded configuration with small permanent deformation. Figure 3 shows a mechanistic model that considers the metal as composed of grains with size d and grain boundaries with thickness δ. If the modulus of the boundary is half that of the grains, and δ=1.1nm, then the effective modulus of the metal as a function of grain size agrees well with experiments (Table 1).

With decreasing grain size, there is thus a change of deformation mechanism. The dislocation slip ceases simply due to lack of dislocations, and grain boundary mechanisms begin to play a major role in taking deformation. Yield stress increases. If the grain size is such that there are not many dislocations for plasticity, but the size is not small enough that the abundance of dislocation is yet sufficient for the boundary mechanisms to facilitate the deformation, then the metal may show highest strength. For Al, 50nm appears to be this transition size.

[1] A. Haque and M. T. A. Saif, *Scripta Materialia*, 47, pp 863-867, 2002.

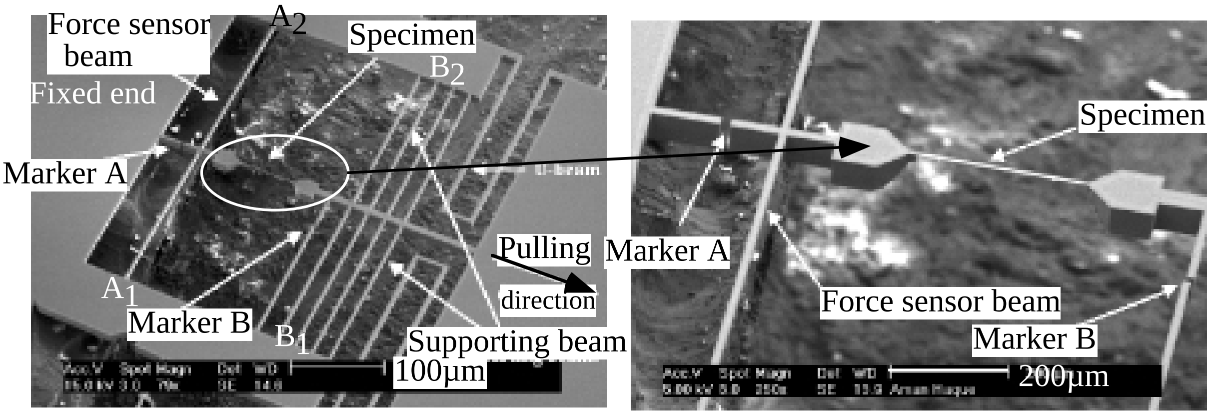

Figure 1. Scanning electron micrograph of the micro stage and the smaple. The stage is 100µm deep, 10mm long and 3mm wide.

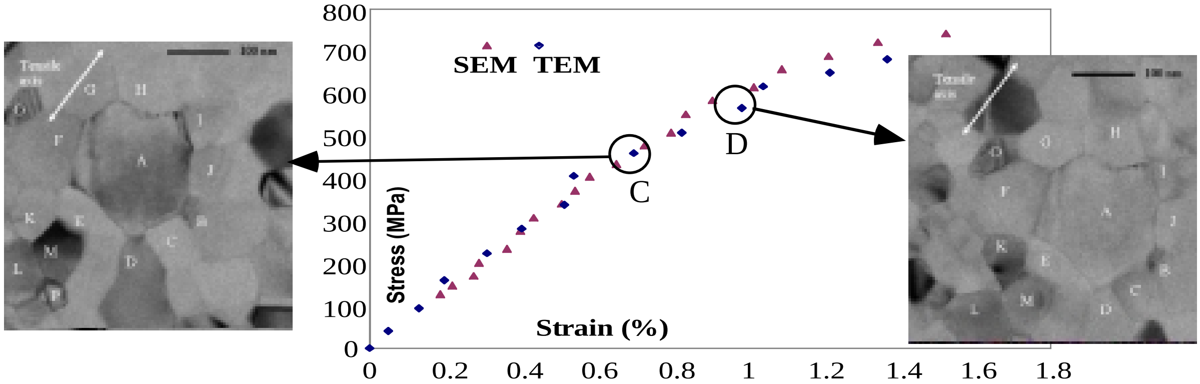

Figure 2. Stress-strain behavior of 100nm thick aluminum film studied in Scanning and Transmission electron microscopes.

Film thickness t (nm)	Grain size d (nm)	E (GPa)	Yield stress (GPa)	Ultimate stress (MPa)	Ultimate strain ε_u (%)
200	80	70	327	531	1.995
100	50	68	710	726	1.525
50	20	63	328	497	1.099
30	10	60	249	318	0.638

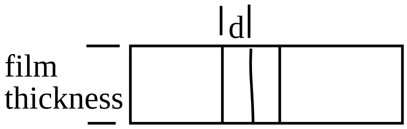

Table 1. Mechanical properties of Al films with varying thickness and grain sizes

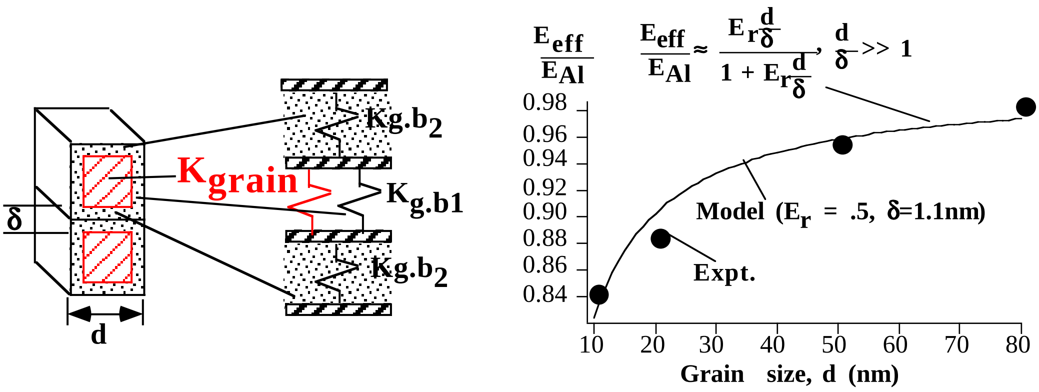

Figure 3. An elastic model of a polycrystalline metal with grains and grain boundaries. If $E_r = E_{gb}/E_g = .5$, and the grain boundary thickness is 1.1nm, then the model prediction matches well with the experimental data.