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Scripta Materialia 67 (2012) 736–739

Scripta MATERIALIA

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Comments and Reply

Comment on: Size effects on yield strength and strain hardening for ultra-thin Cu films with and without passivation: A study by synchrotron and bulge test techniques

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Received 19 January 2012; revised 8 April 2012; accepted 18 April 2012
Available online 17 May 2012

Copper sub-micron films are structured as columnar arrays of grains and are virtual two-dimensional polycrystalline solids. By modelling their plastic deformation by a two-dimensional flow of sliding deformable grains, a new perspective of the mechanical tests of Gruber et al. [Acta Mater. 56 (2008) 2318] is attained that reveals remarkable regularities which are missed by the more conventional analysis of the authors. The model shows that two-dimensional plasticity has significant qualitative differences from the three-dimensional counterpart.
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Keywords: Thin films; Plastic deformation; Grain boundary sliding; Modeling

In a recent paper, Gruber et al. [1] report the isothermal mechanical testing of a number of copper sub-micron films from 15 to 765 nm thickness. While straining the sample, they measured the two principal true stresses, in the loading and transverse directions, by diffracting in situ the X-rays provided by a synchrotron source. The diffraction pattern reveals the distortions of the crystal cells of the metallic film, which are proportional to the true stresses operating on the film. Our analysis of the data suggests that the films undergo a two-dimensional flow regime when plastically deformed. Regrettably, Gruber et al. failed to realize this because they interpreted their experimental results in a more conventional way.

Because of the mismatch of the Poisson ratios of the metallic film and the substrate, any strain in the loading direction is associated not only with a stress σ_L in that direction, but also with a comparable stress σ_T in the transverse one. Gruber et al. measured both stresses with high accuracy, but did not solve the biaxial character of the deformation univocally when interpreting the data. In Figure 9 of their paper they attempt to introduce an equivalent effective uniaxial stress resorting to the von Mises formula $\left(\sigma_L^2 - \sigma_L\sigma_T + \sigma_T^2\right)^{1/2}$ for the effective yield stress in two dimensions, which rests on the assumption that the yield point is an intrinsic property of the material [2]. However, the flow stresses in Figure 9 of Gruber et al. clearly correspond to strain states far from the yield point. In Figures 10 and 11 the stress σ_L in the loading direction is identified with the flow stress, disregarding the stress in the transverse direction, which defies the commonly accepted idea that multiaxial plastic deformation is driven by deviatoric stresses. We show in what follows that the deformation is actually driven by the difference $\sigma_L{-}\sigma_T$ between the principal stresses, which always has a plateau when plotted against the strain and is almost independent of the film thickness, no matter how structured σ_L and σ_T may be. The high flow stresses claimed by Gruber et al. are strongly influenced by the substrate through σ_T.

The problem has current technical importance because the structural dimensions of today microsystems are on the length scale of 100 nm or less, and tensile and compressive stresses in them can reach hundreds of megapascals, either in service or during the fabrication process. Reliable design demands a precise knowledge of the mechanical properties of sub-micron metallic

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http://dx.doi.org/10.1016/j.scriptamat.2012.04.018

films, together with a good understanding of the response of the film to strongly varying stresses.

Normal-view and cross-sectional micrography has shown that polycrystalline sub-micron films have grain sizes that are larger than, or of the same order as, the film thickness, with grains traversing the entire film [1,3]. Typical films are structured as columnar arrays of grains, which in the plane of the film exhibit random equiaxed shapes and crystallographic orientations. However, partial crystallographic ordering is detected in the direction normal to the film plane. The grains of sub-micron copper films, either self-standing or on a supporting substrate, show a strong tendency to orient the (1 1 1) crystal direction along the normal to the film surfaces [1,3]. A sub-micron metallic film of this kind configures a physical realization of a two-dimensional polycrystalline solid. The importance of this goes beyond the technical applications because it provides a means for the laboratory testing of theoretical models for plastic flow with reduced dimensionality. High-precision methods for elucidating the intrinsic thin film mechanical properties when testing them on substrates [1], and techniques for the fabrication and mechanical testing of self-standing beams of sub-micron thickness [4], have been developed only very recently.

If we assume that grain boundary sliding is the dominant mechanism for the plastic deformation of the film, we can reduce the general theoretical formalism of Ref. [5] to two dimensions to analyse the data. The basic hypothesis of the model is that adjacent grains can slide past each other over long distances by way of the shear stresses actuating in their shared boundaries, at the same time accommodating their shapes by internal mechanisms to prevent voids at the interfaces and to preserve matter continuity. The sliding-induced stress fields associated with grain shape accommodation are of little relevance because they are assumed to be much weaker than the shear stress causing grain sliding. The latter is linearly related to the relative speed $|\Delta\vec{v}|$ between adjacent grains, and has a threshold τ_c below which no elementary sliding process can occur [6].

Figure 1 represents a pair of adjacent two-dimensional grains. The x' and y' axes of the local frame of reference $(x'y')$, associated with the unitary vectors $\hat{\imath}'$ and $\hat{\jmath}'$, are normal and parallel, respectively, to the grain boundary shared by the grains. The main frame of reference (xy), with unitary vectors $\hat{\imath}$ and $\hat{\jmath}$, has its axes in the principal directions of the stress tensor. Denoting $\sigma_{i'j'}$, $i',j' = x',y'$, the components of the stress tensor in the $(x'y')$ frame of reference, the relative velocity $\Delta\vec{v}$ of the two grains is

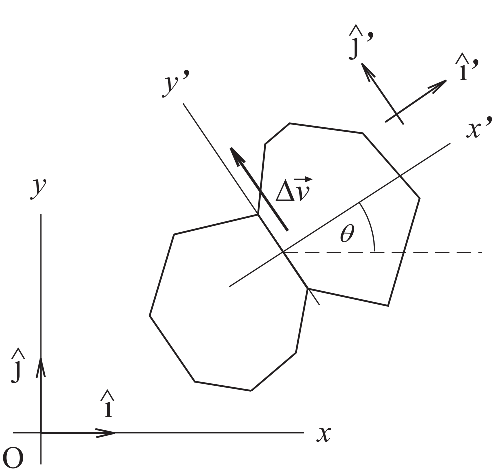

Figure 1. Schematic representation of two adjacent grains showing the local $(x'y')$ and main (xy) frames of reference, the corresponding unitary vectors and the relative velocity $\Delta\vec{v}$.

$$\Delta\vec{v} = \begin{cases} \mathcal{Q}(\sigma_{x'y'} - s'\tau_c)\hat{\jmath}' & \text{if} \quad \sigma_{x'y'} > \tau_c \\ 0, & \text{otherwise} \end{cases} \tag{1}$$

where $\mathcal{Q}$ is a proportionality coefficient and $s' = \sigma_{x'y'}/|\sigma_{x'y'}|$ is the sign of the shear stress $\sigma_{x'y'}$. The term $s'\tau_c$ ensures that $\Delta\vec{v} = 0$ when $|\sigma_{x'y'}| = \tau_c$. This expression for $\Delta\vec{v}$ has proven to hold with great accuracy for several aluminium, titanium and magnesium alloys [7]. The coefficient $\mathcal{Q}$ must not depend on either the shear stresses or the orientation of the grain boundary; therefore, its dependence on the normal stresses is only via the hydrostatic pressure invariant $p = -(\sigma_{x'x'} + \sigma_{y'y'})/2$ [8,9].

Replacing the well-known Mohr's formula $\sigma_{x'y'} = -(\sigma_x - \sigma_y)\sin\theta\cos\theta$, where σ_x and σ_y are the principal stresses and θ is the rotation angle defined in Figure 1, and $\hat{\jmath}' = -\sin\theta\hat{\imath} + \cos\theta\hat{\jmath}$, it gives

$$\Delta\vec{v} = \mathcal{Q}[-(\sigma_x - \sigma_y)\sin\theta\cos\theta + s'\tau_c](-\sin\theta\hat{\imath} + \cos\theta\hat{\jmath}) \tag{2}$$

which presumes that

$$\sin(2\theta) \geqslant \frac{2\tau_c}{|\sigma_x - \sigma_y|} \equiv \sin(2\theta_c) \tag{3}$$

and otherwise $\Delta\vec{v} = 0$. A grain boundary whose normal $\hat{\imath}'$ subtends with the principal direction x or y an angle smaller than θ_c is not able to slide because the in-plane shear stress is below τ_c, no matter how strong the external forces may be.

To link these equations with the velocity field $\vec{v}(x,y)$ of the plastically flowing film, consider two points, at (x,y) and $(x+\delta x,y)$. The macroscopically small segment δx intersects a large number n of grain boundaries, and then $\delta\, x = nd$, where d is the mean grain size. The relative velocity between the starting and final points of the segment δx is the sum of the n relative velocities between the consecutive grains it passes through. Thus

$$\frac{\vec{v}(x+\delta x,y) - \vec{v}(x,y)}{\delta x} = \frac{1}{nd}\sum_{k=1}^{n}\Delta\vec{v}(k) \tag{4}$$

where k numbers the successive grain boundaries intersecting δx. For the factor d in the denominator, however, the right-hand side of this equation defines an average. Consequently, in the proper limit,

$$\frac{\partial\vec{v}}{\partial x_i} = \frac{1}{d}\langle\Delta\vec{v}\rangle_i, \quad i = x,y \quad \text{or} \quad x_i = x,y \tag{5}$$

where the phrase $\langle\ldots\rangle_i$ means the average over all boundary orientations θ compatible with $x_i \geqslant 0$. With the latter restriction and recalling condition (3), this gives

$$\frac{\partial\vec{v}}{\partial x_i} = \frac{1}{\pi d}\int_{D_i} d\theta\Delta\vec{v}, \quad i = x,y \quad \text{or} \quad x_i = x,y \tag{6}$$

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