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Fracture toughness of free-standing nanocrystalline copper–chromium composite thin films

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Abstract—In this paper, a hybrid method of experiments and numerical analyses for measuring the fracture toughness of electron-transparent thin films (∼50–100 nm thick) with a nanocrystalline grain size is presented. Electron-transparent, free-standing copper–chromium composite thin films were produced by electron beam deposition coupled with electron beam lithography and deformed in situ in a transmission electron microscope (TEM) in tension. Crack growth in these nanocrystalline thin films was observed and recorded in situ in the TEM. The recorded crack opening profiles are used to estimate the local as well as the global fracture toughness of the nanocomposite by employing inverse analyses. The yield strength, the plastic hardening modulus and the toughness of the copper matrix are determined by the inverse finite element method by matching numerical crack opening profiles with the experimental counterpart. Knowing the matrix toughness, crack kinking angles at the copper–chromium interfaces are used to estimate the interface toughness. The global composite toughness is then obtained by estimating the bridging forces of crack-face ligaments with a limit analysis. The inverse analyses give the yield stress as ∼800 MPa, the plastic hardening modulus as ∼1 GPa and the local toughness as ∼64 J m^{-2} for the nanocrystalline copper matrix. The toughness of the copper–chromium interface is determined to be ∼27 J m^{-2}; this weak interface provides crack-face bridging that increases the global toughness of the composite film by ∼38% to ∼89 J m^{-2}.
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1. Introduction

Nanocrystalline (nc) metals (materials with grain size <100 nm) in general display high yield strength, hardness and wear resistance often at the expense of ductility and toughness [1–3]. It is also generally accepted that as the grain size decreases within this regime, plastic deformation is no longer dominated by dislocation motion in the interior of grains; much of the deformation is accommodated at grain boundaries, and grain boundary sliding begins to play a significant role in the deformation process [1–3]. Thin films, <200 nm in thickness, and deposited from the vapor state, often exhibit a nc microstructure, the grain size being limited by film thickness and being comparable to the film thickness. Keller et al. [4] and Arzt [5] reported that the tensile strength increases with decreasing film thickness as a consequence of both film thickness and grain-size effects on dislocation motion and Arzt, in his extensive review [5], noted the need for further studies to elucidate underlying mechanisms contributing to strengthening in polycrystalline thin films. Such size effects on strength and also on low ductility [6], and various possible deformation mechanisms [7–9] responsible for the size effects, have also been reported. In summary, the yield strength and the toughness of such thin films depend not only on the grain size d but also on the thickness h of the film [10–14], i.e. $\sigma_y(d,h)$ and $G_c(d,h)$. However, it has been experimentally difficult to measure σ_y and G_c for free-standing thin films [15] and to delineate the two effects.

In considering the thickness effect on the yield strength of a free-standing metallic film, interaction between the free surfaces and grain boundary plasticity is believed to alter the yield strength of thin films once the thickness becomes comparable to the grain size. Depletion of grain-boundary dislocation sources near free surfaces is considered to elevate the yield strength for smaller thickness. Such grain-boundary source depletion is considered to be due to dislocation image forces and the consequences are similar to the high strength, low hardening behavior of single crystal nanostructures where dislocation starvation mechanisms have been identified [16]. Nevertheless, exact mechanisms responsible for the thickness dependence of the yield strength are yet to be determined. Regarding the thickness dependence, Espinosa et al. [11] showed that the yield strength of a copper film more than doubles from 160 to

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345 MPa when the thickness is decreased from 1000 to 200 nm. Hirakata et al. [13] showed that $\sigma_y = \sigma_a + \sigma_b (h_0/h)^{1/2}$ with $\sigma_a = 17.4$ MPa, $\sigma_b = 5095.5$ MPa and $h_0 = 1$ nm for a copper film with its thickness in the range of $200 < h < 1000$ nm. The interplay between d and h for the yield strength makes it more complicated to predict the thickness dependence of the toughness for ultrathin films.

In contrast to the grain-size dependence of yield strength, grain-size dependence of toughness is far less understood, since the toughness is determined by dislocation plasticity at crack tips, void nucleation strength and growth and linkage mechanisms of multiple dispersed voids [17–19]. Furthermore, fracture mechanisms are also thought to vary as the yield strength increases as a consequence of grain size reduction to an ultrafine scale and the dominant plasticity mechanisms change. Karimpoor et al. [20] and Haque and Saif [21] showed that the toughness of nc metal thin films is much less than those of conventional bulk metals. It is postulated that a small number of dislocation sources and a thickness-limited glide distance of dislocations in thin films result in decreased fracture toughness when the thickness is of the same order of magnitude as the grain size.

It is known that the toughness of a plate approaches the plane-strain toughness when the thickness is larger than the crack tip plastic zone size, while the toughness increases towards the plane-stress toughness as the thickness is reduced to less than the plastic zone size. This phenomenon is caused by the constraining effect of three-dimensional homogenized-continuum plasticity. However, once the thickness is further reduced to less than a critical thickness, h_{cr}, free-surface effects on micromechanisms of plasticity make the fracture toughness, $K_{IC}(d,h) = \sqrt{EG_c(d,h)}$, of nominally ductile metals decrease with the thickness [12,19,22,23], where E is Young's modulus. For example, Begley et al. [24] noted that the fracture toughness increased from 6.9 to 8.5 $\text{MPa}\sqrt{\text{m}}$ for copper thin films when the thickness is increased from 200 to 600 nm, which is in agreement with experimental results by Hirakata et al. [13], who measured $K_{IC} = 2.34$, 6.63 and 7.81 $\text{MPa}\sqrt{\text{m}}$ for copper films of $h = 100$, 500 and 800 nm, and $d = 170$, 280 and 370 nm correspondingly in that order.

Approaches adopted previously for the evaluation of the fracture toughness of thin films include notched tensile tests [12,13,19,22,25], channel cracking tests [24,26], bulge tests [27,28], scratch adhesive tests [29–31] and indentation tests [14,32,33]. The fracture toughness of such films has been determined from the simple relation $K_I = Y\sigma\sqrt{\pi a}$, where Y is a factor related to specimen geometry and a is the crack size, utilizing linear elastic fracture mechanics (LEFM) as it applies to cracked specimens under tensile loading [13,19,22,26,27]. However, significant plasticity in the immediate vicinity of a propagating crack can be present [18,19] and causes errors in this relation for the estimation of fracture toughness. Preparation of a notched free-standing specimen is also difficult and inconvenient, and is usually made by a line-incisor or a sharp razor for micron-thickness films [12] and a focused ion beam system for sub-micron thickness films [13,22,27]. Moreover, it is difficult to preserve propagating cracks normal to the loading direction in tensile tests as this requires elaborate alignment fixtures to be utilized when testing such thin specimens to ensure valid results. The fracture toughness of a thin film on a substrate has been quantitatively estimated using channel cracking tests coupled with a micromechanical model relating applied strain to average crack spacing [24,26]. This approach has uncertainties associated with the process to obtain crack spacings, mechanical properties of the substrate, inelastic deformation in thin films and delamination at the interface. Scratch and indentation tests have inherent problems such as dependence of indentation tip sharpness, uncertainty in measuring crack lengths and applied forces, interface delamination and semi-empirical relations based on analytic or numerical solutions of half-space radial cracking problems. In addition, it is also difficult to precisely measure applied forces and indentation-induced cracks in ultrathin films where shallow indentation depths are required to make a sharp crack in a thin film on a substrate. While in situ transmission electron microscopy (TEM) straining techniques [19,22,23] allow the detailed examination of the evolution of microstructural events ahead and in the vicinity of an advancing crack tip, the lack of precise knowledge of the stress and strain states at the advancing crack tip makes the technique less amenable (if not impossible) for direct quantification of fracture energy. The methodology described in this paper is an attempt to overcome this deficiency.

In this paper, we introduce three new inverse methods to extract elasto-plastic properties of individual parts of a Cu/Cr microcomposite, local toughnesses of the nc Cu matrix and the Cr/Cu interface, and the global toughness of the composite, from TEM images of crack growth in the free-standing film. The first method is the matched-asymptotic inverse finite element method (MAI-FEM) [34–39] to measure the mechanical properties of an ~100 nm thick Cu film of ~50 nm grain size, directly from the crack opening geometry observed in in situ straining tests. The Cu film is reinforced with a geometric array of through-thickness cylindrical Cr pillars/disks of ~500 nm diameter. Here, we use MAI-FEM to evaluate the yield strength, the plastic linear hardening modulus and the toughness of the film. The second is an interface-crack kinking analysis to extract the Cr/Cu interface toughness using crack kinking angles that are made by matrix–pillar interface decohesion and naturally observed in the in situ experiments. The third is an interaction J-integral method [40] coupled with limit analysis [41] to evaluate the global toughness of the composite film, taking into account the crack bridging forces of Cu ligaments made by microcracks of debonding Cu/Cr interfaces that trails the advancing crack tip. It is emphasized that atomic scale mechanisms are not explicitly examined in this study although their manifestations are collectively reflected in the crack geometry/shape and sequence of events that ensue in the crack growth process. In this approach, in contrast to tensile and indentation techniques, it is not necessary to directly measure applied forces. Previously, Pallares et al. [42] reported a method similar to the MAI-FEM presented here for evaluating the stress intensity factor of a sharp crack under elastic deformation, based on William's series expansion to match measured crack opening profiles. However, the present MAI-FEM for cracks blunted by concentrated plastic deformation embedded in a background plastic-deformation far-field employs an inverse finite element (FE) analysis to evaluate the yield strength and the hardening parameter as well as the toughness. The estimation of the interface toughness by using a kinked crack analysis, introduced here, is distinctly an innovative approach since analytical and/or experimental

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