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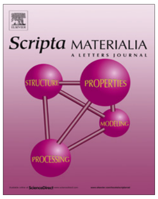

Strain rate sensitivity of Cu/Ta multilayered films: Comparison between grain boundary and heterophase interface

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ABSTRACT

Grain boundaries and heterophase interfaces both play important roles in enhancing the strain rate sensitivity (SRS) of engineering materials as they often serve as obstacles for dislocation motion. In this work, however, we carried out nanoindentation tests on Cu/Ta multilayers prepared with a wide range of modulation periods (Λ) and modulation ratios (η) and found negative contribution of incoherent interfaces to SRS. Activation event extension aided by incoherent interface shear was suggested as the dominating mechanism for rate related deformation process in Cu/Ta multilayers. The results provided valuable insights into the fundamental roles of grain boundaries and heterophase interfaces in plastic deformation.

Nanostructured multilayered films (NMFs) have large volume fraction of interfaces, which is the main reason that metallic NMFs exhibit unique mechanical properties such as high hardness and strength attractive for a range of practical applications. While this has provoked a significant theoretical interest in exploring the underlying mechanisms [1–5], the strain rate sensitivity (SRS) of metallic NMFs is a key parameters that could shed light on their rate controlling deformation mechanism [5]. The large SRS value (m) achieved can help suppress localization at large deformation rates.

Abundant experimental findings have led to the interesting discovery that SRS is highly sensitive to the average grain size (d) and refinement of grains to nanoscale in metals such as Cu, Ni, W, Mo and Ti resulted in enhanced SRS [6–17] due to the primary obstacle of grain boundaries (GBs) that hinder dislocation motion. Nevertheless, the variation trend of m in NMMs with individual layer thickness (h) is complex as two different constituent phases coexist. For instance, recently, Lu reported that Mg/Ti multilayers did not show significant change in m when h was changed, which is radically different from their crystalline components [18]. Then it is interesting to explore how m is influenced by the layer thickness and dissimilar interfaces.

As the SRS of NMFs is yet fully understood, it becomes therefore crucial to compare the roles of heterophase interfaces and GBs played in rate dependent plastic flow. To this end, extensive experimental examinations were conducted on incoherent Cu/Ta multilayer films with varying period ratio Λ and thickness ratio η (=h_{Cu}/h_{Ta}) in this work, in order to have in-depth research into the effects of length-scale-effect as well as interface on SRS.

With reference to Table 1, Cu/Ta multilayers with different combinations of Λ (=Cuh_1 + Tah_2, h_1 and h_2 representing separately the thickness of Cu and Ta layers) and η (=Cuh_1/Tah_2) were deposited to the total thickness of 1 μm on Si (100) at room temperature by magnetron sputtering. The base pressure prior to sputtering was 6.3×10^{-5} Pa and the Ar pressure during sputtering was 6.11×10^{-1} Pa. The microstructures of the as-deposited films were characterized by X-ray diffraction (XRD) using an improved RigakuD/max-RB X-ray diffractometer with Cu Ka radiation and a graphite monochromator. The modulation of each multilayer was investigated using JEOL 2100F high resolution transmission electron microscopy (HRTEM) operated at 200 kV. Upon manual polishing, cross-sectional TEM samples were precisely prepared via Gatan Precision Ion Polishing System 691 by using Ar ion. The mechanical properties of the Cu/Ta films were evaluated by nanoindentation testing via a dynamic contact module device equipped with the Nanoindenter XP system (MTS, Inc.), under continuous stiffness measurement (CSM) mode with a frequency of 45 Hz at room temperature. To accurately evaluate the strain rate sensitivity of the films, nanoindentation was performed at loading

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Table 1
Experimental setting of Cu/Ta multilayer films with $\eta = 1$, and $\Lambda = 60$ nm, 120 nm.

$\eta = 1$	Cu2/Ta2 Cu100/Ta100	Cu10/Ta10 Cu125/Ta125	Cu30/Ta30 Cu200/Ta200	Cu60/Ta60
$\Lambda = 60$ nm	Cu10/Ta50	Cu20/Ta40	Cu30/Ta30	Cu50/Ta10
$\Lambda = 120$ nm	Cu20/Ta100	Cu40/Ta80	Cu60/Ta60	Cu100/Ta20

strain rates (LSR) of 0.2 s^{-1}, 0.1 s^{-1}, 0.05 s^{-1}, 0.01 s^{-1} and 0.005 s^{-1}, respectively, with the maximum penetration depth of 200 nm. Each strain rate was tested for at least 16 points to minimize measurement error.

The XRD patterns of the present Cu/Ta multilayers (not shown for brevity) exhibited strong texture of Cu (1 1 1)//Ta (1 1 0). The corresponding TEM images of Fig. 1a showed clear modulation structures, which were in line with our set values. Careful microstructure examinations revealed that the grain size in both Cu and Ta scaled with the layer thickness. Same as the XRD results, the electron diffraction images presented in Fig. 1b also showed the Kurdjumov–Sachs (KS) crystallographic orientation relation. HRTEM observations in Fig. 1c show that the interface is atomically flat and sharp without any second phase over a large area. The inverse fast Fourier transform (FFT) in Fig. 1d show that one-dimensional mismatch present along the interface is expected to result in an array of misfit dislocations.

Experimentally, the SRS index m has been defined as the slope of the double logarithmic plot of hardness H and imposed nominal strain rate $\dot{\varepsilon}$, as [19–21]:

$$M = \frac{\partial \ln H}{\partial \ln \dot{\varepsilon}} \tag{1}$$

For the present Cu/Ta multilayers, Fig. 2a plotted the variation trend of m with h for selected values of Λ, with η fixed at 1. However, regarding the definitive effect of h, not much could be inferred from the experimental results, for each Λ, all the data points fell onto a straight line. Neither could a definitive relation between m and h be established in a way similar to the relationship between SRS and grain size d for Cu and Ta metals, e.g., significantly increasing m with decreasing grain size in nanocrystalline [12]. Similar results have been reported in other works but without explanation [18,22].

Unlike the remarkable influence of grain size on SRS in pure monolithic Cu and Ta, the insensitive SRS of Cu/Ta multilayers should be attributed to the presence of incoherent interfaces. In order to explore the interface effect on SRS, we further checked the Cu/Ta films with Λ fixed at 60 nm and 120 nm. As shown in Fig. 2b, a reduced m was observed when Λ was reduced from 120 nm (red dots) to 60 nm (black dots), which is contrary to the trend found in nanocrystalline Cu and Ta. With the maximum penetration depth varied from 100 nm to 400 nm, we also conducted comparison tests in Cu10/Ta10 and Cu30/Ta30. As shown in Fig. 2c, as the penetration depth was increased (i.e., more interfaces were crossed), a sharply reduced m was observed.

The results presented above demonstrated that increasing the number of incoherent interfaces has a negative effect on the strain rate sensitivity m of Cu/Ta multilayers, which is contradictory to continuum scale assumptions. In these assumptions, the role of interfaces in a multilayer was often treated in a phenomenological way as grain boundaries (GBs) in nanocrystalline, resulting in scaling laws that relate strength to spacing between interface barriers. However, acting as planar defects, the interfaces and GBs play quite opposite roles in the rate-related parameter m.

For clarification, we should first identify the general features between GBs and incoherent interfaces. During magnetron sputtering, GBs formed perpendicular to interfaces were believed to possess disordered atomic structure with 1–2 nm thickness, so that no dislocations could travel along GBs. Instead, deformation was characterized by dislocations absorbed and nucleated at GBs, then slip along defect-free grain interiors. To elucidate the rate-limiting

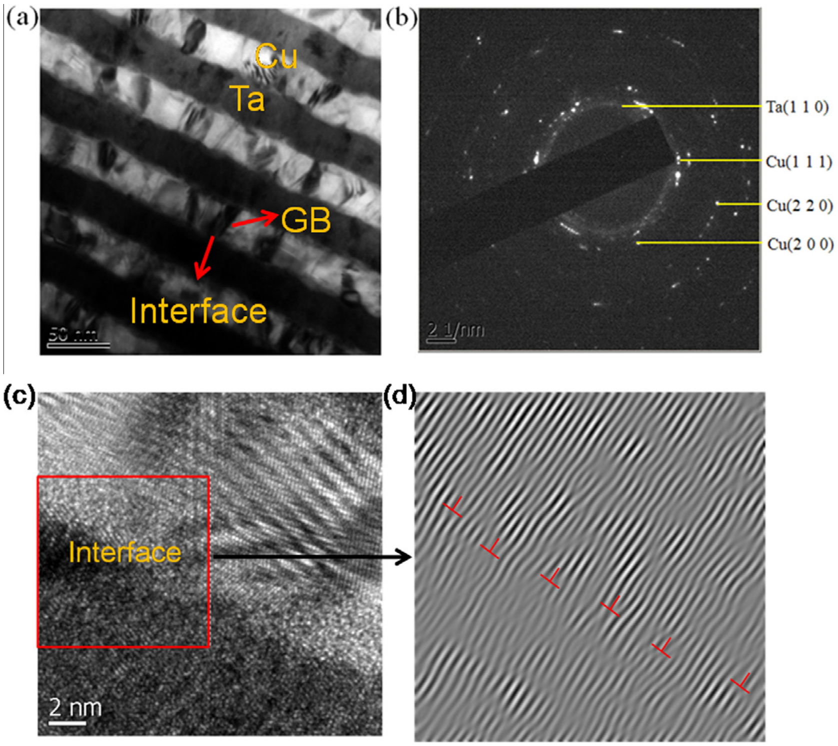

Fig. 1. TEM image of Cu30/Ta30 multilayered film: (a) cross-section; (b) electron diffraction. (c) Is the HRTEM image of Cu10/Ta10 showing the incoherent interface and (d) is the inverse fast Fourier transform (IFFT) HRTEM image of red squared box regions of (c). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)