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Microstructure and ultrahigh strength of nanoscale Cu/Nb multilayers

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ABSTRACT

The microstructure and mechanical properties of Cu/Nb multilayers were investigated by X-ray diffraction, transmission electron microscopy, scanning electron microscopy and nanoindentation. Ultrahigh strength of 3.27 GPa is achieved at the smallest layer thickness of 2.5 nm, which agrees well with the theoretical prediction based on the deformation mechanism of crossing of dislocations across interfaces. After that, the strength decreases with the increasing layer thickness and the transition of the deformation mechanism to confined layer slip occurs at the layer thickness of 6.5 nm. Additionally, strength of the Cu/Nb multilayers increases with increasing loading strain rate because of enhanced strain hardening.

1. Introduction

Metallic multilayers have attracted extensive attention because of their promising mechanical properties and significant theoretical interest over the past couple of decades [1–3]. It is generally accepted that for layer thickness (h) above approximately 5 nm, the flow strength of multilayers is dependent on the individual layer thickness and several deformation mechanisms have been proposed to describe the plastic deformation in this size scale [4]. While for layer thickness below 5 nm, where the deformation mechanism is thought to be transmission of single glide dislocations across interfaces, the flow strength is independent on layer thickness and the maximum value is determined by interfacial properties [5]. Hoagland et al. have proved that for different interface types, such as coherent interfaces and incoherent interfaces, the factors that affect the resistance to dislocation slip transmission are quite different [6]. For coherent interfaces, such as interfaces in Cu/Ni system, the most important effect on strength derives from the coherency strains and the maximum attainable flow strength should be equal to the coherency stress [7], which has been confirmed by experimental data later [8]. On the other hand, for incoherent interfaces, such as interfaces in Cu/Nb system, the case is more complex and atomistic simulations are applied to understand the properties of the interfaces and estimate the maximum attainable flow strength. Recently, Wang et al. have investigated the interaction of glide dislocations with Cu/Nb interfaces and found that a single mixed dislocation, from either Cu or Nb, cannot cross the interface until the resolved shear stress (RSS) is increased to extremely high values in excess of 1.1 GPa [9]. This result indicates that the attainable strength maximum in Cu/Nb system should exceed 3.4 GPa. However, such maximum strength or hardness has not yet been observed experimentally, although a lot of researches have been made on the room temperature strength of the Cu/Nb system using nanoindentation or micropillar compression [4,10–13]. The discrepancy between the model prediction and experimental data may be due to the sensitivity of the strength to strain rate. As revealed by atomic modeling, dislocation climb is involved in the process of slip transmission even at room temperature, and reactions between interfacial dislocations assisted by climb could lead to annihilation of dislocation content, resulting in less strain hardening [14]. The room temperature strength values of Cu/Nb multilayers that have been reported were frequently tested at strain rate on the order of 10^{-4} s^{-1}, quite smaller than the strain rate that usually used in atomistic simulations. This lower strain rate may promote the dislocation climb process, release strain hardening, and then reduce the strength value. In fact, the dependence of yielding strength on the strain rate has been observed in several nanocrystalline materials [15,16]. Although there is no clear understanding of the deformation mechanism, it is postulated that the confined nanoscale grain structure plays an important role in this peculiar phenomenon.

Herein we investigate the room temperature strength of Cu/Nb system using nanoindentation with a constant loading strain rate (LSR) of 0.05 s^{-1} and discuss the length-scale deformation mechanism at such strain rate. Moreover, varying loading strain rate tests are carried out to further investigate the dependence of the strength on strain rate. The experiment results confirm that ultrahigh strength over 3 GPa can be achieved in Cu/Nb multilayers with small layer thickness at relative high strain rate and room temperature dislocation climb is involved in the plastic deformation process. The mechanical properties of Cu/Nb mulitlayers have been intensively studied because of their remarkably high strength, fatigue resistance

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[17] and thermal stability [18]. Due to higher He solubility than either Cu or Nb and ability to prevent the degradation of material properties by the growth of bubbles, Cu/Nb multilayers are markedly more resistant to radiation damage, and thus can be applied to extend the operating limits and lifetimes of nuclear reactors [19–21].

2. Experimental details

Cu/Nb multilayers consisting of alternating equivalent Cu and Nb individual layers with h ranging from 2.5 to 20 nm were prepared at room temperature by alternate electron beam evaporation deposition using an ultrahigh vacuum (UHV) chamber. The base vacuum of the chamber was 5×10^{-6} Pa. The deposition rates were 0.8 Å/s for both Cu and Nb. The nominal thickness of the individual Cu and Nb layers was monitored by an in situ quartz oscillator. All the multilayers were deposited onto Si (100) wafers with native oxide. The deposition began with Cu layer and ended with Nb layer. The total thickness of multilayers was approximately 400 nm. Monolithic Cu film and Nb film of 1 μm were also deposited onto Si (100) in the same condition for comparison.

The X-ray diffraction (XRD) experiment was carried out using Rigaku D/max-RB X-ray diffractometer with Cu Kα radiation. Microstructure observation was performed by a JEM2011 transmission electron microscopy (TEM) with 200 kV accelerating voltages. The mechanical properties of the multilayers were investigated by a Nano Indenter XP (MTS Systems Corp.) with a displacement resolution of 0.01 nm and a loading resolution of 50 nN. A Berkovich indenter, a three-sided pyramid with the same area-to-depth function as that of a Vickers indenter, was used in all the experiments. The hardness and modulus of the multilayers were measured by a continuous-stiffness measurement (CSM) technique with a constant LSR of 0.05 s^{-1}. Varying LSR nanoindentation tests are carried out on the multilayer with $h=10$ nm with LSR ranging from 0.01 to 0.5 s^{-1}. A frequency of 45 Hz was used to avoid the sensitivity to thermal drift. The maximum indentation depth was 400 nm. More details about this technique and analysis method can be found in references [22–24]. Ten indents were performed on each specimen and an average value is reported with error bars indicating the range. For reference, the mechanical properties of the Cu film and Nb film were also examined under the same experimental condition. After indentation, the indent images of hardness measurement at LRS $=0.05$ s^{-1} were characterized by field-emission scanning electron microscopy (SEM).

3. Results and discussion

3.1. Microstructure

Cross-section transmission electron microscopy was performed to confirm the layered structure of the Cu/Nb multilayers. Fig. 1 shows the bright field image of the multilayer with $h=20$ nm. Discrete layered structure with sharp planar interfaces between the two phases is clearly visible. Meanwhile, we can see that the multilayer show columnar structure with the columns mostly extending through the whole thickness of the film. The average width of the column grains is around 130 nm, much larger than the layer thickness.

Fig. 2 shows the high-angle symmetrical X-ray spectra for Cu/Nb multilayers in the 2θ range of 30–65°. One can see that only the peaks of Nb (110) and Cu (111) are rather strong, and the other diffraction peaks are quite weak. The results indicate that all the multilayers are polycrystalline with strong Nb (110) and Cu (111) out of plane texture. As the layer thickness decreases to 4 nm, some additional satellite peaks are observed to flank the principal reflections and their position agrees well with the periodicity predicted by the deposition rate calibrations. Meanwhile, the peaks of the Cu (111) and Nb (110) begin to shift to a low-angle. The appearance of satellite peaks is a sign of the formation of superlattice structure and it has been proved that

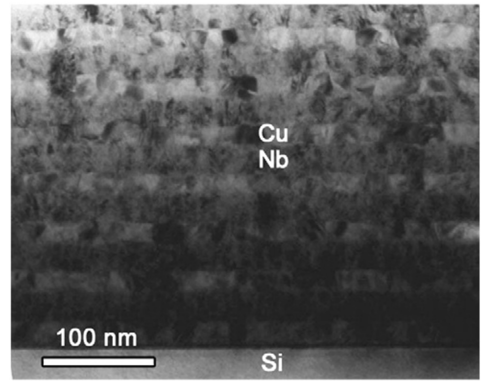

Fig. 1. The cross-section transmission electron micrographs taken in bright field of the multilayer with $h=20$ nm.

superlattice structure can be produced in ultrathin layers of two dissimilar multilayers [25]. In fact, X-ray pole figure measurements have confirmed that epitaxial relationship of Kurdjumov–Sachs orientation Cu{111}//Nb{110} and Cu<110>//Nb<111>exits in the Cu/Nb interfaces in ultra thin layer thickness [26]. Due to the large lattice mismatch of ~11%, misfit dislocations will be generated in the interfaces to accommodate the elastic strain, resulting in a compressive contribution to the interface stress [27,28]. Owing to the Poisson effect, the spacing perpendicular to the interfaces will expand, resulting in the shift of the diffraction peaks of Cu (111) and Nb (110) to a low-angle.

3.2. Mechanical properties

The nanoindentation data were measured continuously during the loading of the indenter by the CSM method, and then they were analyzed by the Oliver and Pharr method [22]. Fig. 3(a) and (b) show the variations in hardness and modulus with indentation depth of the Cu/Nb multilayer with $h=2.5$ nm for ten different continuous stiffness indentation tests, respectively. Both the curves approach

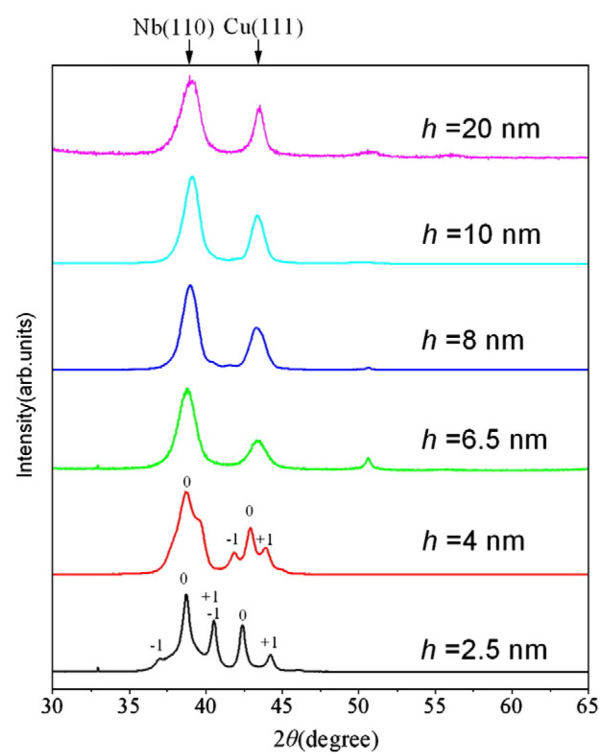

Fig. 2. The XRD patterns for all the Cu/Nb multilayers.