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Size dependence of creep behavior in nanoscale Cu/Co multilayer thin films

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ABSTRACT

Cu/Co multilayers with periodicity of 4–40 nm were prepared by electron beam evaporation deposition. Microstructure and room temperature creep behavior were investigated by X-ray diffraction, transmission electron microscopy and nanoindentation test. The results show that superlattice structure forms with decreasing periodicity and coherent interfaces come into being at low periodicity of 4 nm. Size dependence of the creep behavior is observed and power-law creep parameters including stress exponent and size sensitivity index are calculated by dimension analysis. A dislocation model for predicting the steady-state deformation of multilayers with semi-coherent interfaces is presented. Nanoscale effects are explained by dislocation generation and annihilation mechanisms involving single dislocations slip in confined layers and dislocations climb at the interfaces, respectively. Model predictions agree well with experimental observation.

1. Introduction

The influence of scale on the mechanical properties of multilayers has long been of interest since large hardness enhancement compared with monolithic coatings of the constituent materials has been found [1,2]. Through studying the evolution of film strength as a function of periodicity, the length-scale-dependent strengthening mechanisms have been well documented [3]. However, scale dependence of rate-controlling deformation mechanisms in multilayers has less been investigated, even though some researchers have already focused their eyes on room temperature creep phenomena in thin films due to the necessary to practical applications [4–6]. Recently, size dependence of power-law creep observed in nanocrystalline has received much attention and several models have been proposed [7–9]. It is believed that, different from coarse-gained materials, dislocations in nanocrystalline are mainly located at high-angle boundaries, and thus both the rates of dislocations generation and dislocations recovery are enhanced. As a result, explicit size dependence of steady-state deformation appears. Plastic deformation in nanoscale multilayers is expected far more complex than that in nanocrystalline because interfaces which play a key role in the plastic deformation have very different structures in different types of systems. In fact, discrepant size-dependent creep behavior has already been reported in several multilayer systems [10–12]. It is obvious that the rate-controlling deformation mechanisms in multilayers is still unclear and is open for investigation.

With the development of depth-sensing indentation technique, it is accepted that nanoindentation test provides a simplest and most direct way to probe the mechanical properties, including creep, of thin films [13,14]. As one of the widely used method, constant-load indentation (CLI) experiment records the hardness value with decreasing indentation strain rate during the load-holding segment, so the tabulation of several hardness-indentation strain rate pairs can be applied to analyze the creep process [15–18]. Although nanoindentation creep behavior is considerably more complex than that measured by conventional uniaxial tensile tests, creep parameters such as stress exponent are in good agreement with those values obtained by conventional methods [19–22].

Cu/Co is one of the most studied multilayer systems for it is an ideal candidate for magnetic sensors and electronic switching elements due to its giant magnetoresistance (GMR) [23]. In the present work, we investigate room temperature indentation creep in Cu/Co multilayers using the CLI method. Creep parameters including stress exponent and size sensitivity index are calculated by dimensional analysis. A dislocation model based on dislocations generation and annihilation at interfaces is developed to account for the nanoscale effects.

2. Experimental details

Cu/Co multilayers were deposited onto Si(1 0 0) wafers at room temperature by alternate electron beam evaporation deposition of Cu and Co using an ultra high vacuum (UHV) chamber. The base vacuum of the chamber was 5×10^{-7} Pa. Prior to the deposition of Cu/Co multilayers, a 10-nm-thick Ti layer was deposited to improve the cohesion between the film and the substrate. The deposition rates were 0.5 Å/s for both Cu and Co. The nominal thickness of individual Cu and Co layers was

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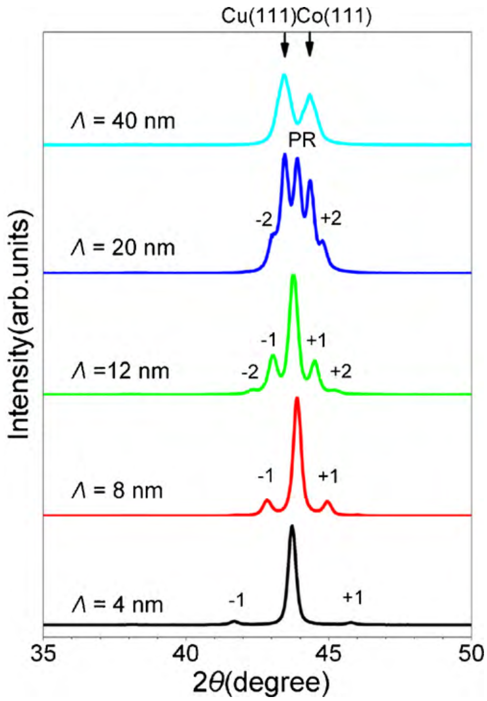

Fig. 1. The XRD patterns for all the Cu/Co multilayers.

monitored by an in situ quartz oscillator and all the multilayers were controlled to have equivalent Cu and Co layers but different periodicity ranging from 4 nm to 40 nm. The total thickness of films was approximately 600 nm.

X-ray diffraction (XRD) experiment was adopted to investigate the microstructure using Rigaku D/max-RB X-ray diffractometer with Cu Kα radiation. Transmission electron microscopy (TEM) with selected-area diffraction (SAD) was performed using a JEM2010 high-resolution electron microscopy (HRTEM) with 200 kV accelerating voltages. The mechanical properties of the multilayers, as well as the Cu and Co monolithic layers, were investigated by a Nano Indenter XP (MTS Systems Corp.) with a displacement resolution of less than 0.01 nm and 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 tests. The hardness of the multilayers was measured by a continuous-stiffness measurement (CSM) technique with a load strain rate of 0.05 s^{-1}. Indentation creep process was tested at room temperature by a constant-load indentation (CLI) method. A series of indents was made on each sample at an inter-indent spacing of 50 μm, and the mean values of the creep data were then calculated in order to minimize the deviation of results after the extreme values were rejected.

3. Results and discussion

3.1. Microstructure

X-ray spectra recorded with standard θ–2θ reflection geometry of Cu/Co multilayers is shown in Fig. 1. For the multilayer with Λ = 40 nm, only two main Bragg reflections located at 43.4° and 44.3° are observed, indicating strong ⟨1 1 1⟩ texture along the film normal. When the periodicity decreases to 20 nm, another principal reflection located at 43.9° appears, which is not the characteristic of Cu or Co plane spacing but intermediate between the spacing characteristics of Cu (1 1 1) and Co (1 1 1). We presume that this principle peak is the reflection of the epitaxial Co/Cu (1 1 1) planes. With the decreasing periodicity, the reflections of Cu (1 1 1) and Co (1 1 1) disappear and only the principal reflection can be observed. Moreover, the principal reflection is flanked symmetrically by some additional satellite peaks and their position agrees well with the periodicity predicted by the deposition rate calibrations. The XRD spectra indicates that superlattice structure forms with the decreasing periodicity as 2.0% lattice mismatch within close-packed planes between Cu (1 1 1) and Co (1 1 1) is accommodated and manifested by the single Bragg reflection at ~43.9°.On the other hand, it is well known that full coherent interfaces can form only below a critical layer thickness h_c [24]. When layer thickness is above this critical value, a square grid of misfit dislocations between semi-coherent interfaces will form to relax coherency stresses. Experimental observations in Cu/Co superlattices have revealed that coherent interfaces remain for layers up to 4 nm [25,26], thus, we assume that full coherent interfaces will come into being at Λ = 4 nm in Cu/Co multilayers.

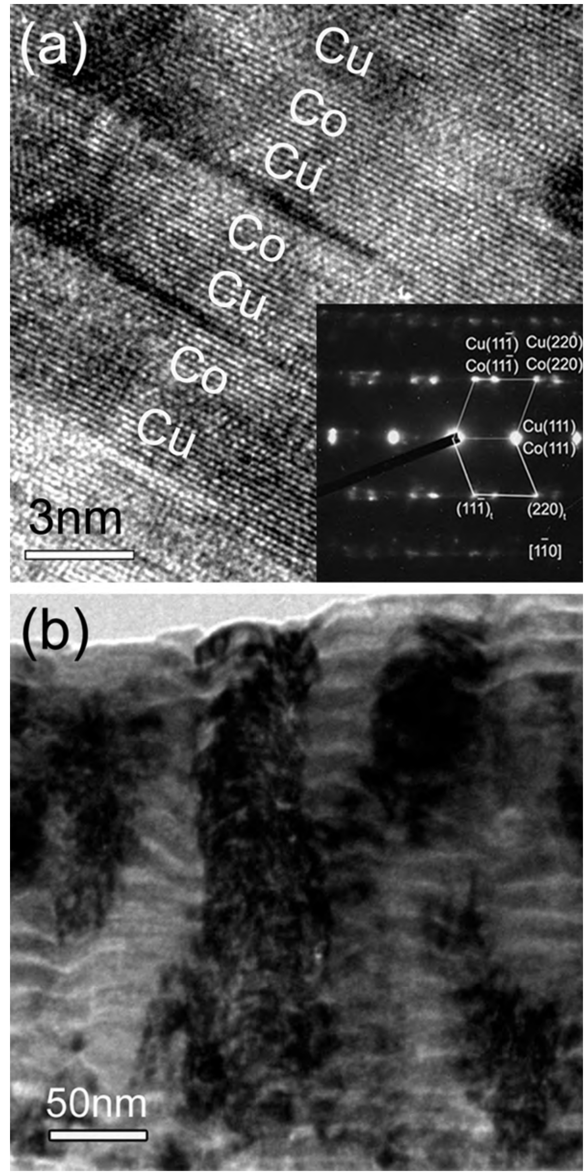

Fig. 2. (a) The cross-section HRTEM image and the corresponding SAD pattern confirm coherent interfaces in the multilayer with Λ = 4 nm. (b) The transmission electron micrograph taken in bright field of the multilayer with Λ = 20 nm shows the layered structure with columnar grains.

Cross-section transmission electron micrographs provide a more intuitive view of the interfacial structure at small periodicity. Fig. 2(a) shows the high-resolution electron micrograph of the multilayer with Λ = 4 nm. Layered structure is still maintained at Λ = 4 nm and coherent interfaces between Cu and Co layers have been observed. Moreover, the corresponding SAD pattern with [1 $\bar{1}$ 0] zone axis, as inserted in Fig. 2(a), also confirms the coherence with the orientation relationship of $(1\,1\,1)_{Cu}//(1\,1\,1)_{Co}$. Apart from this, a twining structure with (1 1 1) twin planes parallel to the layer interfaces is implied. Fig. 2(b) shows the bright field image