Altering strength and plastic deformation behavior via alloying and laminated structure in nanocrystalline metals

C. Gu [a], F. Wang [b,*], P. Huang [a,*], T.J. Lu [b,c], K.W. Xu [a]

[a] State Key Laboratory for Mechanical Behavior of Material, Xi'an Jiaotong University, Xi'an 710049, China
[b] State Key Laboratory for Strength and Vibration of Mechanical Structures, Xi'an Jiaotong University, Xi'an 710049, China
[c] MOE Key Laboratory for Multifunctional Materials and Structures, Xi'an Jiaotong University, Xi'an 710049, China

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ABSTRACT

Nanoindentation and electron microscope techniques have been performed on sputtering deposited monolayered nanocrystalline CuNb and multilayered CuNb/Cu thin films. Microstructural features, hardness and surface morphologies of residual indentation have been evaluated to identify the effects of alloying and laminated structure on strength and plastic deformation behavior of nanocrystalline metals. By altering the content of Nb in CuNb alloy and adding crystalline Cu layers into CuNb alloy, the volume fraction of amorphous phase in CuNb alloy and interface structures changed dramatically, resulting in various trends that are related to hardness, indentation induced pileup and shear banding deformation. Based on the experimental results, the dominant deformation mechanisms of the CuNb and CuNb/Cu thin films with various Nb contents were proposed and extended to be discussed.

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1. Introduction

Nanocrystalline (NC) metals with novel mechanical properties have been the focus of research efforts over the past few decades. NC metals usually exhibit a higher strength compared with their coarse grain counterparts, but a lower ductility that greatly limits their engineering application. In general, the limited ductility in NC metals mainly attributes to lack of available strain accommodation processes and low work hardening capability [1]. Therefore, tremendous efforts have been applied to improve the ductility of NC metals by introducing nanotwins [2,3] or fabricating metals with binomial grain size distribution [4,5].

Other than the aforementioned methods, nanoscale multilayered structure has also been proved to be an effective way to achieve desirable strength and ductility of metallic materials. Specifically, the strength and ductility of metallic multilayers were attributed by modulation periods and the choices of the constituent layers [6]. Generally, the hardness of multilayer thin films with equal individual layer thickness (h) will increase with decreasing h. The dominated strengthening mechanism of these multilayers has been systematically studied. Hall–Petch (H–P) relation, the confined layer slip (CLS) model and the interface crossing model had been successfully applied on explaining the strength of crystalline/crystalline

multilayer thin films with h ranging from micrometer to a few nanometers [7]. The structure and strength of the interfaces between the heterogeneous phase layers have been proposed to play an important role in determining the deformation behavior of the multilayers. However, the ductility is still strictly limited in most of the multilayer systems with equal individual layer thickness. In recent years, by adding very thin crystalline layers into amorphous or crystalline phases, the ductility of these laminate structure materials has been effectively improved [8–10] by the existence of crystalline/amorphous interfaces (CAIs) or crystalline/crystalline interfaces.

Alloying is another way to effectively enhance the strength and thermal stability for pure NC metals [11]. By alloying, both grain size and stacking fault energy of NC metals should decrease, resulting in high strength and strong twinning formation ability. Moreover, the volume fraction of amorphous phase in NC alloys could be increased by increasing solute atoms content, leading to plastic deformation transformed from NC-like to amorphous-like behavior [12], i.e., from dislocation dominated to shear bands (SBs) dominated. This characteristic of nanocrystalline alloys provides an effective way to systematically study the deformation behavior of both crystalline and amorphous phases in an identical alloy system. Furthermore, the structure of alloy phase could affect the interface structure when adding heterogeneous phase layers into the monophase alloy. As mentioned above, while the interface structure could strongly affect the deformation mechanisms of the laminated structured thin films, the specific impact of the interface

with different structures could also be systematically studied in the alloy system.

In the present study, by considering the effects of both alloying and multilayered structure, the microstructure and mechanical properties of CuNb alloy thin films with and without crystalline Cu layers were examined mainly by nanoindentation test and electron microscope analysis, to explore the effects of interface structure formed between CuNb and Cu layers on the strength and plastic deformation behavior.

2. Experimental details

CuNb alloy thin films with various Nb contents f_{Nb} were deposited on single crystal Si substrate by magnetron sputtering under argon pressure of 0.3 Pa at room temperature. Direct current (DC) and radio frequency (RF) power co-sputtering pattern were used for CuNb depositing, in which RF power with a fixed value of 100 W for Cu deposition, while DC power of 20 W, 60 W and 100 W were used for Nb deposition. The layer thicknesses of all the CuNb thin films were maintained as 1500 nm. Energy dispersive spectrometer (EDS) result showed that Nb contents were 9.3 at%, 22 at% and 36 at% for the CuNb alloys deposited under DC powers of 20 W, 60 W and 100 W, respectively.

Other than the CuNb alloy, multilayered $CuNb_x$/Cu (x=9.3 at%, 22 at% and 36 at%) thin films, in which the contents of Nb in CuNb layers were identical to that of the monophase CuNb alloys, were also prepared by alternatively depositing a 140 nm CuNb layer and a 10 nm Cu layer of 10 cycles. Thus the total layer thicknesses were also 1500 nm for all the $CuNb_x$/Cu multilayers. In addition, the interface structures of the three kinds of multilayers varied with the Nb content, the detailed information of the monolayer $CuNb_x$ and multilayer $CuNb_x$/Cu samples is shown in Fig. 1. Structural characterization of Cu, Nb and CuNb alloy thin films was performed by X-ray diffraction (XRD, using a D3290 PANalyticalX'pert PRO with Cu-Kα radiation) analysis, and the microstructural features of CuNb and CuNb/Cu samples were examined by using high-resolution transmission electron microscopy HRTEM (JEOL JEM-2100F operating at 200 kV). The mechanical properties were investigated by using a MTS Nanoindenter XP system (MTS, Inc.) under Continuous Stiffness Measurement (CSM) mode. The radius

of the Berkovich tip was calibrated to be around 50 nm, and the applied strain rate was set as a constant of 0.05 s^{-1}. The residual indentation morphologies of each sample were characterized by using scanning electron microscope (SEM).

3. Results

3.1. Microstructural features

3.1.1. Monophase CuNb alloy

Fig. 2 shows the XRD patterns of pure Cu, Nb and CuNb alloys with various Nb contents. Specifically, Fig. 2(a) indicates that CuNb9.3 at% alloy exhibited nearly identical diffraction peaks as pure Cu with face-centered cubic lattice structure, indicating that Nb atoms may nearly completely dissolve in Cu lattice as substitutional solid solution and no precipitated phase was formed. As f_{Nb} increased, the diffraction peaks of CuNb22 at% and CuNb36 at% moved to lower angle range that is close to the angles corresponding to Nb diffraction peaks. Detailed and magnified diffraction patterns of CuNb9.3 at%, CuNb22 at% and CuNb36 at% are shown in Fig. 2(b). Although the sharp peak of Cu(111) at 43.5° appeared in CuNb9.3 at% alloy, broad hump appeared roughly around angle of 40.5° in CuNb22 at% and Cu(200) and Cu(311) peaks disappeared except for a relatively stronger Cu(220) peak at 73.0°. For CuNb36 at% alloy, similar broad hump centered at 41° appeared and Cu(220) peak was weakened. Therefore, the diffraction patterns shown in Fig. 2 indicate that the CuNb alloys transformed from crystal-like to amorphous-like lattice structures as f_{Nb} increased.

3.1.2. $CuNb_x$/Cu multilayers

Cross-sectional TEM images of $CuNb_x$/Cu multilayers are shown in Fig. 3. For CuNb9.3 at%/Cu, the bright field and dark field images shown in Fig. 3(a) and (b), respectively, indicate that adding Cu layers could be clearly observed as pointed by white arrows. The diffraction pattern in the upper left of Fig. 3(a) shows that the lattice parameter of CuNb9.3 at%/Cu is the same as pure Cu, and is consistent with XRD results shown in Fig. 2. In Fig. 3(b), a single grain in CuNb9.3 at%/Cu is marked by the yellow circle curve, which indicates that the in-plane grain size of CuNb9.3 at%/Cu is about 50 nm. Detailed HRTEM image of the yellow dashed box in Fig. 3(b), which
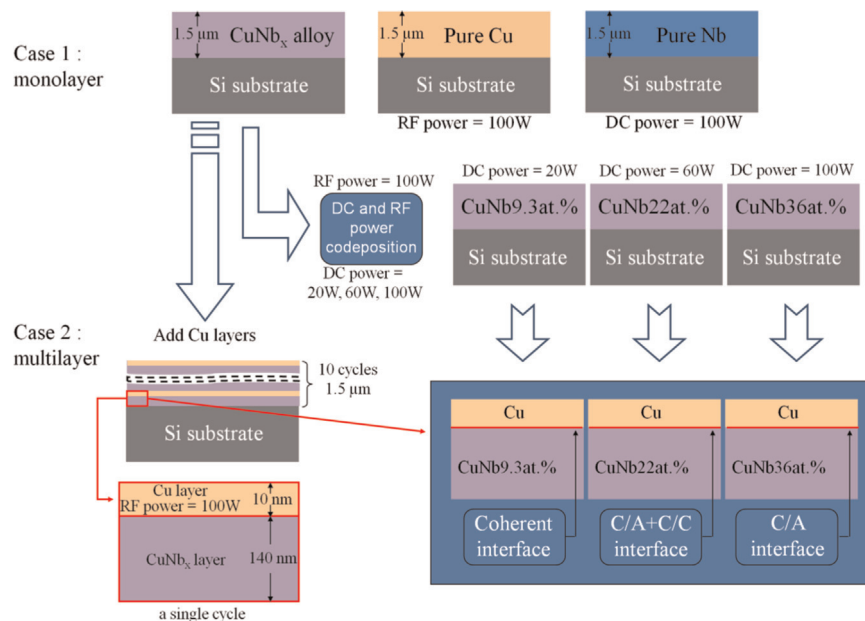

Fig. 1. Schematic diagram of the fabrication of the monolayer $CuNb_x$ films and multilayer $CuNb_x$/Cu films with different interface structures.