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Intrinsic size effect of CuTa/Cu nanolaminates with unequal modulation ratios

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ABSTRACT

In order to study the intrinsic size effect of nanolaminates with unequal modulation ratios, CuTa/Cu nanolaminates with identical Cu layer volume fraction but different Cu layer thicknesses were prepared by varying the number of Cu layers. The hardness and indentation morphology of each CuTa/Cu nanolaminate was characterized by means of nanoindentation and scanning electron microscope. Furthermore, the microstructures of CuTa/Cu nanolaminates with different interfacial configurations were examined under high resolution transmission electron microscopy. The deformation of CuTa/Cu nanolaminates was found to be dominated by the thickness ratio between CuTa and Cu layers as well as the interfacial microstructure.

Amorphous materials attracted increased attention due to their fundamental scientific importance and engineering application potential [1,2]. However, the application of amorphous thin films is seriously limited by nearly zero plasticity due to catastrophic failure caused by shear bands (SBs) formation and propagation [2]. An effective way to enhance the mechanical properties of amorphous materials was to introduce crystalline layers into monolayer amorphous thin film, forming the amorphous/crystalline (A/C) nanolaminate structure [3]. The deformation mechanism of A/C nanolaminate was found to be strongly size dependent, because the formation or propagation of SBs in amorphous layer and dislocation motion in crystalline layer were both size dependent [4–6]. Existing research on A/C nanolaminates mainly focused on structures with unequal modulation ratios [7–9], as A/C nanolaminates with equal modulation ratios exhibited the worst plasticity behavior [10]. Traditionally, to investigate the size effect of A/C nanolaminates with unequal modulation ratios, the thickness of amorphous or crystalline layer was fixed while the thickness of the other layer was altered [7,8], or the modulation wavelength was fixed while the modulation ratio was varied [10]. For these structures, the volume fraction of both C layer and A layer changed when individual layer thickness in A/C nanolaminate was altered. Under such conditions, although the mechanical properties of the nanolaminate changes with varying layer thickness, it also changes with varying volume fraction of the two constituent layers. That is, the hardness would be affected by both the size-dependent strengthening mechanism and the volume fraction factor. This additional volume fraction factor would also affect the plasticity of the system as the plasticity might be improved by increasing the volume fraction of ductile constituent layers. As a result, it was hard to clarify whether the change of mechanical properties was mainly caused by the size factor or by volume fraction factor. And the question arises as what is the real size effect without the influence of volume fraction factor, i.e., the intrinsic size effect, in nanolaminate systems with unequal modulation ratios. In order to investigate the intrinsic size effect of A/C nanolaminates, the volume fraction of both the crystalline layer and the amorphous layer should be kept constant.

In the present study, series of CuTa/Cu A/C nanolaminates with fixed Cu layer volume fraction were prepared. While discussing the size-dependent deformation mechanism, the thickness of Cu layer and CuTa layer in each series of A/C nanolaminates was varied by varying the number of Cu layers. Furthermore, by varying the volume fraction of Cu layer in two different series of samples, the size effect of CuTa/Cu nanolaminates having identical Cu layer thickness but different Cu layer volume fractions were also discussed. In addition, by controlling the Ta content in CuTa layer, CuTa/Cu nanolaminates having identical structural configuration but different interfacial structures were prepared. The effect of interfacial structure on the deformation mechanisms of these samples was analyzed.

CuTa monolayers and CuTa/Cu nanolaminates were deposited on Si(100) wafers by magnetron sputtering. CuTa monolayers were deposited using the pure Cu and Ta targets co-sputtering mode, which were

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connected with radio frequency (RF) power and direct current (DC) power, respectively. During deposition, the RF power was fixed at 100 W while the DC power was fixed at 60 W and 80 W, respectively, so as to obtain two CuTa monolayers with different Ta contents. Energy disperse spectroscopy (EDS) results showed that the Ta content of the CuTa monolayer deposited with a DC power of 60 W and 80 W was separately 34 at.% and 42 at.% (denoted below as CuTa34 and CuTa42). Subsequently, by depositing alternatively the CuTa layer and the Cu layer, CuTa/Cu nanolaminates were prepared. Specifically, samples of CuTa34/Cu and CuTa42/Cu with Cu layer thickness of 50 nm, 20 nm, 10 nm and 5 nm were prepared by fixing the Cu layers volume fraction at 1/15 (i.e., the total thickness of Cu layers was 100 nm) and varying the number of Cu layers as 2, 5, 10 and 20, respectively. Further, the volume fraction of Cu layers was set as 2/15 for CuTa34/Cu, i.e., the total thickness of Cu layers was 200 nm. Again, with the number of Cu layers varied as 2, 5, 10 and 20, the Cu layer thickness became 100 nm, 40 nm, 20 nm and 10 nm. For both CuTa monolayers and CuTa/Cu nanolaminates, the total thickness was fixed at 1.5 μm.

The microstructural features of CuTa monolayers and CuTa/Cu nanolaminates were investigated by examining their cross-sectional images under high-resolution transmission electron microscopy (HRTEM, JEOL JEM-2100F operating at 200 KV). Nanoindentation tests were carried out using a MTS Nanoindenter XP system (MTS, Inc.) under Continuous Stiffness Measurement (CSM) mode. Berkovich indenter was chosen to measure the hardness of all samples. The indentation depth was 200 nm and 1500 nm (i.e., equal to layer thickness) for hardness test and residual indentation morphology examination, respectively. In each test, a total of 12 indents were performed, with the applied strain rate fixed at 0.05 s^{-1}.

The inset of Fig. 1(a) illustrated schematically the present CuTa/Cu nanolaminates: the thickness of Cu layers varied with the number of Cu layers by fixing the volume fraction of Cu layers in the nanolaminates. Fig. 1(a) and (c) presented nanoindentation hardness results for all the nanolaminates. Two series of CuTa/Cu nanolaminates were compared: series I had identical Cu layer and CuTa layer microstructures but different Cu layer volume fractions while series II had the same Cu layer thickness and Cu layer volume fraction but different interfacial structures.

Fig. 1(a) plotted the hardness of CuTa34/Cu as a function of Cu layer thickness for series I, where the total Cu layer thickness for CuTa/Cu-100 and CuTa/Cu-200 was 100 nm and 200 nm, respectively. For CuTa34/Cu-100, the hardness increased with increasing Cu layer thickness. In contrast, For CuTa34/Cu-200, the hardness decreased with increasing Cu layer thickness. This quite different variation trend of hardness between these two groups of samples indicated that the two sample groups possessed different deformation mechanisms even when they had identical Cu layer thickness.

The hardness of A/C nanolaminate system was considered to be affected synthetically by the crystalline layer, the amorphous layer and the interface. A simple relationship of rule of mixture always used to reveal the underlying deformation mechanism of the nanolaminate structure [3,4,7]. The hardness of present CuTa/Cu samples calculated by the rule of mixture, H_{ROM}, was given by:

$$H_{ROM} = H_{CuTa} \cdot f_{CuTa} + H_{Cu} \cdot f_{Cu} \quad (1)$$

where H_{CuTa} and H_{Cu} were the hardness of CuTa layer and Cu layer, and f_{CuTa} and f_{Cu} were the volume fraction of CuTa layer and Cu layer, respectively. In previous studies, amorphous materials were found to be size-independent over a wide range of samples sizes [11–14]. As a result, H_{CuTa} should be constant in Eq. (1). However, the crystalline layer exhibited distinct size effect [15–17]. To confirm the size-dependent strengthening mechanism of Cu layer, the TEM images of CuTa34/Cu were examined as shown in Fig. 2(a)–(c). From the selected area electron diffraction (SAED) pattern of CuTa34/Cu-100(5) shown in Fig. 2(a), both amorphous halo and crystalline diffraction spots could be

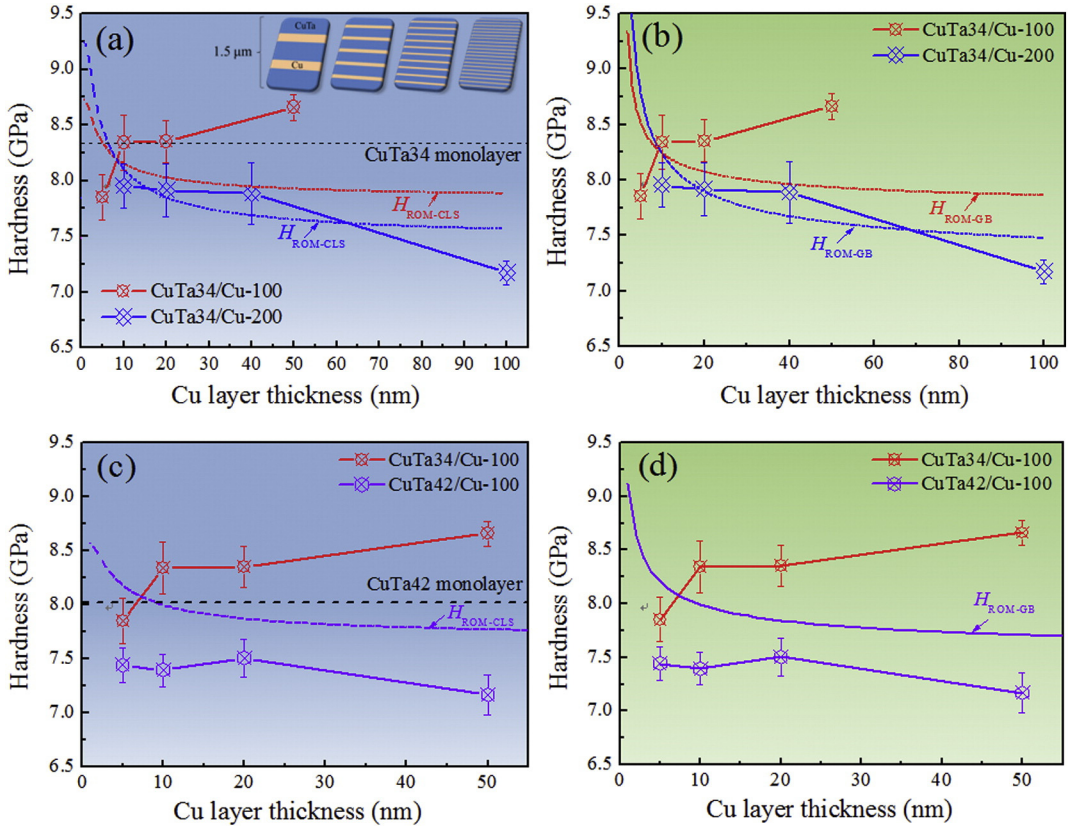

Fig. 1. (a) and (b) nanoindentation hardness of CuTa34/Cu-100 compared with that of CuTa34/Cu-200, the inset of (a) presented the schematic illustration of CuTa/Cu nanolaminates, (c) and (d) nanoindentation hardness of CuTa34/Cu compared with that of CuTa42/Cu.