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Size-dependent hardness and tensile plasticity of Ta-$Zr_{61}Cu_{17.5}Ni_{10}Al_{17.5}Si_4$ nanolaminates

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

[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

Ta/$Zr_{61}Cu_{17.5}Ni_{10}Al_{17.5}Si_4$ crystalline/amorphous (C/A) nanolaminates with different Ta and ZrCuNiAlSi layer thicknesses were deposited by magnetron sputtering on both Si and polyimide substrates. Nanoindentation and uniaxial tensile tests were separately performed to study their size-dependent hardness and plasticity. Tensile plasticity was evaluated by considering both the tensile cracks and nanoindentation morphologies observed under scanning electron microscope (SEM). C/A nanolaminates with crystalline layer thickness of 40 nm and amorphous layer thickness of 5 nm exhibited the best tensile plasticity, which was much better than pure Ta and $Zr_{61}Cu_{17.5}Ni_{10}Al_{17.5}Si_4$ monolayers. For nanolaminates having identical Ta layer thickness, the hardness always increased with decreasing amorphous layer thickness. Combined with cross-sectional high resolution transmission electron microscopy (HRTEM) observation, the size-dependent deformation mechanisms of C/A nanolaminates were discussed.

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1. Introduction

Although nanocrystalline and amorphous materials attracted tremendous attention due to their unique mechanical properties [1–4], the engineering application of these materials had been greatly limited by their poor plasticity. The poor plasticity mainly attributed to the increasingly difficulty for dislocation operation in nanocrystalline materials and the catastrophic failure through mature shear band (SB) formation and propagation in amorphous materials [5,6]. To solve this problem, combining nanocrystalline and amorphous nanoscale thin layers to form crystalline/amorphous (C/A) nanolaminates was proposed to effectively improve plasticity relative to either nanocrystalline or amorphous materials [7–9].

For a C/A nanolaminates, individual-layer-thickness dependent dislocation and shear transformation zone (STZ) motions inside the crystalline and amorphous thin layers, respectively, played crucial roles in determining its mechanical properties [7]. Other than size effects related to individual layer thickness, selection of lattice structure of the crystalline layers was also a crucial issue, which was not addressed until very recently. Previously, for nearly all the nanolaminates, face-center cubic (fcc) latticed Cu was chosen as the crystalline constituent layer [7,8,10–14], and only a few studies considered other lattice structures, *e.g.*, body-centered cubic (bcc) [9] and hexagonal close-packed (hcp) [15]. As crystalline layers having different lattice structures could exhibit quite different deformation behaviors because of their own unique slip systems, it is yet clear whether improved plasticity could be achieved by adding amorphous materials into bcc latticed metals and how the plastic deformation mechanism of nanolaminates is affected by amorphous layer thickness.

In the present study, bcc Ta and $Zr_{61}Cu_{17.5}Ni_{10}Al_{7.5}Si_4$ amorphous alloy were selected as the constituent materials to prepare Ta/$Zr_{61}Cu_{17.5}Ni_{10}Al_{7.5}Si_4$ C/A nanolaminates. By testing the mechanical properties of samples having different Ta and/or $Zr_{61}Cu_{17.5}Ni_{10}Al_{7.5}Si_4$ layer thicknesses, the optimized combination of individual layer thickness was investigated. The underlying deformation mechanisms were explored by examining the indentation morphologies, tensile cracks and atomic scale microstructures.

* Corresponding author.
** Corresponding author.
E-mail addresses: wangfei@mail.xjtu.edu.cn (F. Wang), huangping@mail.xjtu.edu.cn (P. Huang).

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2. Experimental details

2.1. Material preparation

Ta/ZrCuNiAlSi nanolaminates with various individual layer thicknesses were deposited on both Si(100) and polyimide substrates via magnetron sputtering. For reference, monolayer Ta and ZrCuNiAlSi thin films were also prepared. Firstly, a circular polyimide substrate layer, diameter 50 mm, was placed on a pallet and then covered with a baffle with five rectangular holes (3 mm in width and 40 mm in length). Secondly, two fan-shaped silicon chips were fixed on the edge of a baffle. Finally, the deposition device was placed inside a vacuum chamber. Thin films of both C/A nanolaminates and monolayers were deposited on Si and polyimide substrates simultaneously in order to ensure the consistency of deposition parameters. Thin films deposited on Si substrates were used for nanoindentation testing, while those deposited on polyimide substrates were for tensile testing.

Before depositing, the direct-current (DC) power was connect with a 99.99% pure Ta target and the radio-frequency (RF) power was connect with a ZrCuNiAlSi alloy target. During depositing, the DC power and RF power were fixed at 100 W and 150 W, respectively. Ta and ZrCuNiAlSi monolayers were prepared by single operation of DC power and RF power, respectively, while the Ta/ZrCuNiAlSi nanolaminates were prepared by working alternatively with DC power and RF power. Two series of Ta/ZrCuNiAlSi nanolaminates were prepared. For seriesI, the thickness of Ta layer was fixed at 20 nm while ZrCuNiAlSi layer thickness was varied from 2.5 nm to 20 nm. For seriesII, the thickness of Ta layer was fixed at 40 nm and the ZrCuNiAlSi layer thickness was varied in the same manner as series I. For both series I and II thin films, the total film thickness was fixed as 1500 nm by carefully controlling the deposition time.

2.2. Mechanical behaviors

The hardness of all the samples was evaluated using a Nanoindenter XP system (MTS, Inc.) under Continuous Stiffness Measurement (CSM) mode, with a fixed strain rate of 0.05 s^{-1}. The indentation depth was fixed as 200 nm for hardness testing and the tip was Berkovich indenter with radius ~50 nm. For each measurement, 16 indents were carried out and at least 10 effective data were involved in the eventual analysis. In addition, deep indentations with penetration depth of 1500 nm were performed for each thin film sample to further characterize the deformation behavior. For each sample, tensile tests were also carried out via a Universal Testing Machine at a constant strain rate of 10^{-5} s^{-1}, and the gauge length and elongation were fixed as 30 mm and 10%, respectively.

2.3. Microstructural investigation

The microstructures of Ta/ZrCuNiAlSi nanolaminates, especially the Ta-ZrCuNiAlSi interface structures, were examined under HRTEM (JEOL JEM-2100 F operating at 200 KV). TEM sample was prepared by cutting a small piece from specimen deposited on Si substrate. Indentation morphologies and tensile cracks were observed under SEM.

3. Results and discussion

3.1. Deformation behaviors

The morphologies of tensile cracks were used to characterize the plasticity of all samples. Fig. 1 showed the SEM images of the tested samples, with low magnification (1 K) and high magnification (20 K) images separated by dashed lines in Fig. 1(a)–(j). For pure Ta and ZrCuNiAlSi thin films, cracks were clearly observed in both low and high magnification images as shown in Fig. 1(a) and (b). Similarly, cracks also appeared in all Ta(20)/Am(*x*) (*x* means a variable parameter with value of 2.5 nm, 5 nm, 10 nm or 20 nm) nanolaminates as shown in Fig. 1(c)–(f) (sample with Ta layer thickness of *m* nm and ZrCuNiAlSi layer thickness of *n* nm was referred as Ta(m)/Am(n) hereafter). In contrast to tensile testing of free standing films or bulk materials, where only a mature fracture formed, the formation of multiple cracks in the present study was attributed to the adhesion force between the deposited film and the polyimide substrate. Upon tensile deformation, the film was homogeneously elongated with polyimide. When the strain exceeded the breaking elongation of the film, cracks formed on its top surface and evenly distributed along the length direction. The cracks in the SEM image were caused by different elastic recovery percentages between the film and the polyimide. As the thickness of polyimide (200 μm) was much larger than the film (1.5 μm), the elastic recovery percentage of polyimide was negligibly affected by the film and should be, approximately, the same in all the samples. For a thin film deposited on polyimide, better plasticity means larger elongation with the polyimide, resulting in a narrow crack separation distance after elastic recovery. In extreme cases, if the plasticity of the film was better than the experimentally setting strain value, no crack would be observed.

The crack separation distance shown in Fig. 1 was an average of ten cracks which were distributed uniformly across the samples. As the values in Fig. 1(c)–(f) were larger than those in Fig. 1(a) and (b), the plasticity of Ta(20)/Am(x) nanolaminates was even worse than both monolayer Cu and ZrCuNiAlSi. In particular, upon increasing the Am layer thickness to 20 nm, the value shown in Fig. 1(f) was much larger than the other Ta(20)/Am(x) nanolaminates, corresponding to the worst plasticity among all the samples. By further increasing the thickness of Ta layer to 40 nm, the crack morphologies of Ta(40)/Am(x) became quite different: contrary to Ta(20)/Am(x) where the largest crack separation distance was observed in the sample having the thickest Am layer, the largest crack separation distance appeared in Ta(40)/Am(x) having the thinnest Am layer as shown in Fig. 1(g). Furthermore, Fig. 1(j) indicated that the crack separation distance of Ta(40)/Am(x) with the largest Am layer thickness, *i.e.*, Ta(40)/Am(20), was smaller than all other Ta(20)/Am(x), indicating an enhanced plasticity. For Ta(40)/Am(5) and Ta(40)/Am(10) shown in Fig. 1(h) and (i), respectively, cracks in both low and high magnification images were nearly invisible. Especially, note that the magnification of Ta(40)/Am(5) and Ta(40)/Am(10) in high magnification images was 25 K, larger than the others with 20 K magnification. These results indicated that plasticity had been effectively improved when the Ta layer thickness was set as 40 nm and Am layer thickness as 5 nm or 10 nm. As cracks in Ta(40)/Am(5) and Ta(40)/Am(10) were nearly the same, it was hard to clarify which sample had the better plasticity. Consequently, residue nanoindentation morphologies of these samples were investigated as shown in Fig. 2.

Fig. 2(a) and (f) presented the indentation morphologies of monolayer Ta and ZrCuNiAlSi, respectively. Cracks and shear bands (SBs) in Fig. 2(a) and (f) represented typical indentation morphologies of brittleness crystalline and amorphous phase materials. However, the indentation morphologies became more complex when Am layers were added into crystalline Ta. For Ta(20)/Am(x), a switch from cracks to SBs was observed when Am layer thickness was increased from 2.5 nm to 20 nm as shown in Fig. 2(b)–(e). Interestingly, for Ta(20)/Am(5), cracks and SBs were both observed. In contrast, for Ta(40)/Am(x), no SBs was observed as Ta layer thickness was increased. For Ta(40)/Am(10), in particular, both SBs

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