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Alloying effects on ductility of nanostructured Cu-X (X = Zr and W) thin films

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ABSTRACT

Alloying effect on tensile ductility of nanostructured Cu-X (X = Zr and W) thin films was studied in comparison. Both Zr and W atoms segregated at grain boundaries (GBs) and increased the GB cohesion energy, leading to similar increase of ductility in as-deposited Cu-X films. After annealing treatment, however, changes in ductility showed different alloying effect: the Cu-Zr one increased while the Cu-W one decreased when compared with their as-deposited counterparts. This discrepancy was rationalized by different microstructural evolution that intergranular CuZr amorphous layer was produced in the Cu-Zr film while intergranular W grains were formed in the Cu-W one.

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The promise of greatly enhanced strength (hardness) for structural materials has been the driving force behind the increasing research effort on the mechanical properties of nanostructured materials [1,2]. However, the low tensile ductility at room temperature limits their practical usage, in particular for the nanocrystalline (NC) metals with columnar grains (when the loading direction is perpendicular to the boundary) [3,4]. This is because dislocations are emitted from one GB and absorbed by the opposite GB, without dislocation accumulation inside grains thus showing weak strain hardening ability [5]. Atomic shuffling within the boundary is required to accommodate the incoming plastic strain brought by each dislocation. When this process is inefficient, cracking occurs. Once a crack has nucleated, the high density of GBs can act as an easy path for the crack propagation [6].

In fact, previous findings have clearly uncovered that alloying can significantly tune the microstructure at GBs and grain interiors, hence influencing mechanical properties of nanostructured thin films. For example, Hecman et al. [7] studied the tensile ductility of columnar nanotwinned Cu-Al alloys and found it varied from ~2% to ~5%, depending on the Al contents. Khalajhedyati and Rupert [8] studied the uniaxial compressive deformation of electrodeposited NC Ni–W, uncovering the importance of GB states for the failure of materials, with added structural disorder isolated as a positive feature for improving failure resistance. Actually, disorder at a GB can also be negative. For example, amorphization due to S at GBs in Ni is a well-known case of segregation-induced embrittlement [9]. Most recently, Gibson and Schuh [10] developed a model for describing how cohesive energy of a boundary is altered by equilibrium segregation. This model was shown to capture well-known literature data, with embrittling agents in Fe and Au generally being found to be non-metals or metalloids. However, it is unclear whether it is applicable in binary Cu alloyed thin films. In addition, effects of microstructure evolution on ductility of columnar NC metals need more study.

It is well known that the binary Cu-Zr system with very negative enthalpy of mixing exhibits great glass formation ability associated with a wide glass-forming composition range [11,12], while the Cu-W system with high positive enthalpy of mixing and there does not exist any Cu-W compound in its equilibrium phase diagram [13]. In this work, we elaborately prepare the NC Cu-X (X = Zr and W) alloyed thin films with columnar grains. It uncovers that the segregation of Zr and W at GBs increases the ductility both. However, the annealing treatment, which is employed to trigger a more pronounced segregation, shows opposite influence on the ductility of Cu-Zr and Cu-W alloyed thin films.

The Cu-X (X = Zr and W) alloyed thin films with a thickness of 1.5 μm were co-sputtered from elemental targets on 125 μm-polyimide substrates (DuPont Kapton®) by DC magnetron sputtering. With a constant power for Cu target, the sputter power for the X target was adjusted to realize different compositions (from 0 at.% to 15 at.%) of the binary films. The samples were annealed in a high vacuum annealing furnace at 200 °C for 2 h. TEM samples were examined by using JEOL-2100F high resolution (HR) electron microscope with an accelerating voltage of 200 kV to reveal the microstructural features. The scanning transmission electron microscopy (STEM) and energy-dispersive spectroscopy (EDS) capabilities of the JEOL-2100F TEM were used for

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diffraction- and atomic-contrast imaging, and also for elemental mapping of the thin films. Uniaxial tensile testing was performed on a micro-force test system (MTS® Tytron 250) at a constant strain rate of $1.0 \times 10^{-4} s^{-1}$ at room temperature. All samples with a gauge section of 30 mm long and 4 mm wide were strained to about 10%. The critical macroscopic strain ε_{cri} [14], characterizing the microcrack formation on the microscopic level, rather than a rupture strain or elongation, was used to represent the ductility of the films. The residual stresses were measured using $\sin^2\psi$ method by X-ray diffraction (PANalytical X'pert PRO MRD), which were relatively small and of the same order of magnitude for all samples. The morphologies of the microcracks were examined by a scanning electron microscope (JSM-7000F SEM).

Fig. 1(a) and (e) show the typical cross-sectional microstructures of the as-deposited Cu-3.0 at.% Zr and Cu-14.5 at.% W films, respectively, both of which exhibit the nano-sized columnar grains along the growth direction. The GBs are nearly perpendicular to the film free surface, while the twinning planes are nearly parallel to the film surface. The average twin lamellae thickness seems to be ≤5 nm, which may be related to the alloy elements [15–17]. Such small nanotwins were also observed in the as-deposited Cu-Al [18] and Cu-W [19] alloyed thin films. Representative cross-sectional TEM images of annealed Cu-3.0 at.% Zr and Cu-14.5 at.% W films are shown in Fig. 1(b) and (f), respectively. Compared with their as-deposited counterparts, it can be seen that nanotwins and columnar grains are maintained without obvious change in their sizes after annealing. Representative planar TEM images of the as-deposited and annealed Cu-7.0 at.% Zr films are shown in Fig. 1(c) and (d), respectively. It can be seen that the as-deposited film exhibits the nano-sized grains with blurred GBs. After annealing treatment, nano-sized grains were still maintained. Fig. 1(g) and (h) are planar TEM images of the as-deposited and annealed Cu-2.7 at.% W films, respectively. The nano-sized grains are quite stable with clear GBs. The selected area electron diffraction patterns in Fig. 1 show that the films are random polycrystalline samples.

Since Zr and W show negligible solubility in the Cu lattice according to the phase diagram at the equilibrium state, the as-deposited Cu-X alloyed thin films are often supersaturated solid solutions with slight segregation of solute X at GBs [15,19]. Elemental mapping in the TEM by EDS was performed for the annealed Cu-Zr and Cu–W samples. Fig. 2(a) and (b) show the element distribution of annealed Cu-7.0 at.% Zr and Cu-2.7 at.% W films, respectively. The EDX mapping analyses of the rectangular regions reveal a nanoscale heterogeneous structure that Zr and W atoms mainly distribute at the GBs. Only a little of Zr and W can be detected within the NC grain interior after annealing at 200 °C. High-resolution transmission electron microscopy (HR-TEM) observations were carried out to determine the GB structure of annealed samples. Fig.2(c) and (d) demonstrate the existence of continuous and thicker CuZr amorphous interfaces at GBs of the annealed Cu-3.0 at.% Zr and Cu-7.0 at.% Zr films, respectively, compared with the as-deposited films. The annealing treatment induces Zr segregation at GBs and promotes amorphous intergranular films (AIFs) formation in the present Cu-Zr films. This is consistent with Shi and Luo's [20] interfacial thermodynamic models and GB diagrams, showing that higher temperatures usually promote formation of thicker AIFs. By contrast, HR-TEM images of the annealed Cu-2.7 at.% W and Cu-8.8 at.% W films (Fig.2 (e) and (f), respectively) show that small NC W grains are nucleated at GBs. Vüllers and Spolenak [21] also showed that thermal treatment could trigger segregation of spherical W agglomerates with sizes of ~9–15 nm at the GBs.

Fig. 3(a) and (d) show the dependence of the critical macroscopic strain (ε_{cri}) of Cu-X alloyed thin films on X (X = Zr and W) contents before and after annealing treatment. For the as-deposited films, one can see that ε_{cri} increases with the increasing additions in both Cu-Zr and Cu-W films. However, after annealing, ε_{cri} increases in Cu-Zr films and decreases in Cu-W films, compared with their as-deposited counterparts. Moreover, the ε_{cri} of Cu-15 at.% W films is even smaller than that of Cu-W films with W content ≤8.8 at.%. Recent studies demonstrated that nanotwins could improve the ductility [22]. However, nanotwins in the present alloyed films are stable and nearly sustain the same size before and after annealing. These changes in ductility should be attributed to the change in GB structures rather than the nanotwins.

To verify this claim, representative morphologies of microcracks were examined by SEM, as shown in Fig. 3(b, c) and (e, f) for Cu-Zr and Cu-W thin films, respectively. All the tensile directions are vertical to the microcracks, as indicated by the white arrows. One can see that all these alloyed films failed by means of intergranular cracking. The

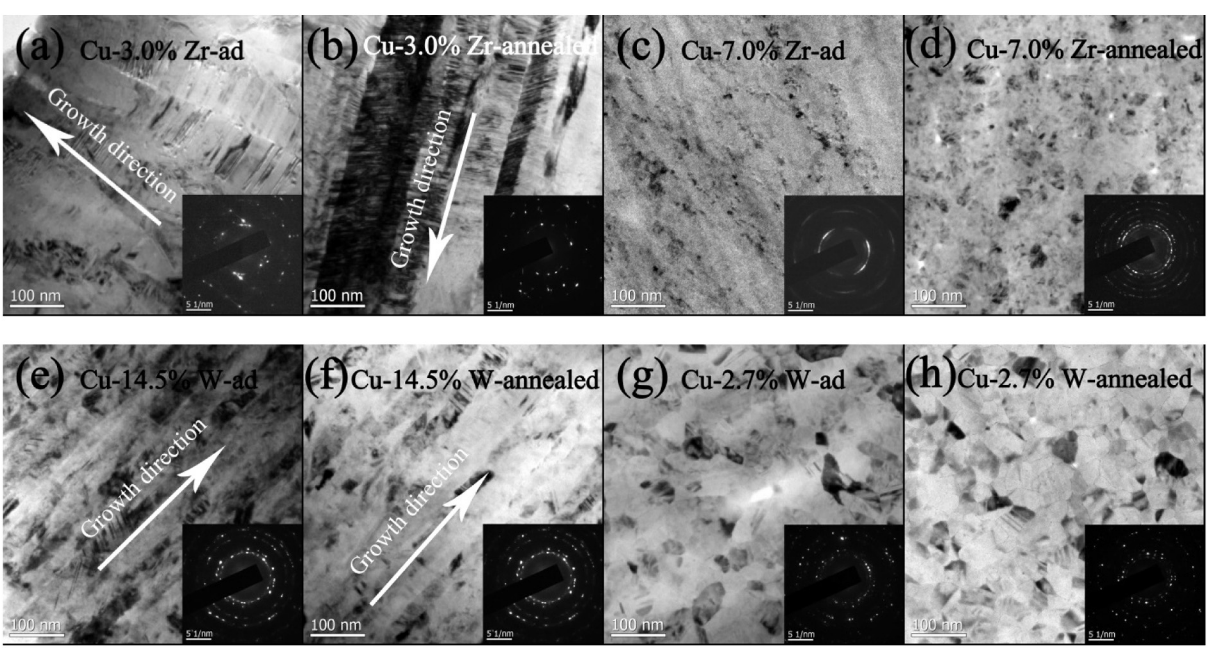

Fig. 1. (a) and (b) are cross-sectional TEM images of the as-deposited (ad) and annealed Cu-3.0 at.% Zr films, respectively; (c) and (d) are planar TEM images of the as-deposited and annealed Cu-7.0 at.% Zr films, respectively; (e) and (f) are cross-sectional TEM images of the as-deposited and annealed Cu-14.5 at.% W films, respectively; (g) and (h) are planar TEM images of the as-deposited and annealed Cu-2.7 at.% W films, respectively. Inserts in the bottom-right are corresponding SAD patterns.

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