



Mechanical characteristics of Mg–Cu–Zr thin film metallic glasses

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ARTICLE INFO

Available online 27 March 2012

Keywords:

Crystalline/amorphous laminates
Interface energy
Micro-compression
Shear fracture

ABSTRACT

For improving the ductility of metallic glasses, nanocrystals within the amorphous matrix are frequently intentionally added. In this study, the MgCuZr thin film metallic glasses (TFMGs) with a positive mixing heat between Mg and Zr are fabricated via co-sputtering in an attempt to separate the pure Mg nano-particles. The microstructure and mechanical properties of the sputtered MgCuZr thin films and their FIB-machined micropillars are examined as a function of Mg content from 39 to 100 at.%. From the nanoindentation and micropillar load–displacement curves, the Mg-rich metallic glass composites exhibit smoother nature and more ductile behavior. Meanwhile, due to strong (0002) basal texture, the Mg-rich thin films and micropillars possess strong modulus (~80 GPa), hardness (~3.5 GPa), and yield stress (~1.5 GPa), coupled with more ductile behavior. All of these would allow promising applications in micro-electro-mechanical systems.

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1. Introduction

Bulk metallic glasses (BMGs) have been widely studied due to their excellent mechanical properties such as high strength and elastic limit [1,2]. Moreover, many researchers note the potential application on the biodegradable [3] and micro-electro-mechanical systems (MEMS) [4] applications due to the lack of periodic atomic packing, indicating that no defects, such as grain boundaries, dislocations, and plane defects exist in amorphous alloys. However, the shear bands, as well as the inhomogeneous plastic deformation characteristic, are still the critical key for the potential application. To solve this problem, two major concepts are announced. The first one is the suppression of shear band propagation [5,6]. By using the interaction between shear bands and crystalline reinforcement phases, the shear bands can be absorbed and stopped [5,7]. Another one is the formation of multiple shear bands by using the phase separation technique [6,8].

In addition to the BMGs, thin film metallic glasses (TFMGs) become popular lately. Both monolithic and multilayered TFMGs are currently under study [4,9–11]. Due to the limitation of film or layer thickness, the shear transition zones (STZs) cannot aggregate and develop into principal shear bands [12]. Such monolithic and multilayered TFMGs can reach the strength of 1–2 GPa with much more improved compressive or tensile plasticity.

In this study, we developed strong amorphous alloys and nanocomposites by using the simple one-step co-sputtering. Here, we combine the two well-known Zr–Cu and Mg–Cu alloy systems to fabricate the MgCuZr alloy thin films. Utilizing the high diffusion mobility of Mg, we can easily fabricate nanocomposites composed of amorphous matrix and nanocrystalline reinforcement phases in various volume ratios.

The mechanical characteristics of a series of MgCuZr alloy thin films are analyzed through nanoindentation and microcompression.

2. Experimental details

A series of MgCuZr alloy thin films was deposited on Si substrates by co-sputtering with pure Mg and Cu₅₀Zr₅₀ (in at.%) alloy targets, which were set on the radio frequency (RF) and direct current (DC) cathodes, respectively. The purity of Mg and Cu₅₀Zr₅₀ targets is 99.9% and 99.9%, respectively. Before deposition, the base pressure of the chamber maintained less than 5×10^{-7} Torr. Thus, the composition of MgCuZr alloy thin films is controlled by altering the power values of Mg and Cu₅₀Zr₅₀ cathodes. The film thickness of all MgCuZr alloy thin films was kept at ~2.5 μm. The resulting MgCuZr alloy thin films have the compositions of Mg₃₉Cu₃₁Zr₃₀, Mg₄₃Cu₃₂Zr₂₅, Mg₄₈Cu₂₈Zr₂₄, Mg₅₅Cu₂₆Zr₁₉, Mg₇₃Cu₁₄Zr₁₃, Mg₇₈Cu₁₂Zr₁₀, Mg₈₄Cu₈Zr₈, and pure Mg, as determined by energy-dispersive spectroscopy (EDS) equipped in JEOL 6330 field emission scanning electron microscopy (FESEM). These samples are named as Mg39, Mg43, Mg48, Mg55, Mg73, Mg78, Mg84, and Mg, respectively.

The nature of the as-deposited MgCuZr alloy thin films was characterized by Siemens D5000 X-ray diffractometer (XRD) from 20° to 60° using Cu–Kα radiation. Thin foils for plane-view transmission electron microscopy (TEM) observations were mechanically ground only from the silicon substrate side. Then, the thin foils were ion-thinned from the silicon side via Gatan precision ion polishing system model 691 with cold stage. The TEM foils were observed via Tecnai G20 field emission transmission electron microscopy (FETEM).

The mechanical properties of MgCuZr alloy thin films were measured using firstly the MTS nanoindenter XP (Agilent Technologies) equipped with a standard Berkovich tip, under the loading-rate-control mode. The peak load and loading rate in all tests were set to be 5 mN and 0.1 mNs^{−1}, respectively. The elastic modulus and hardness were

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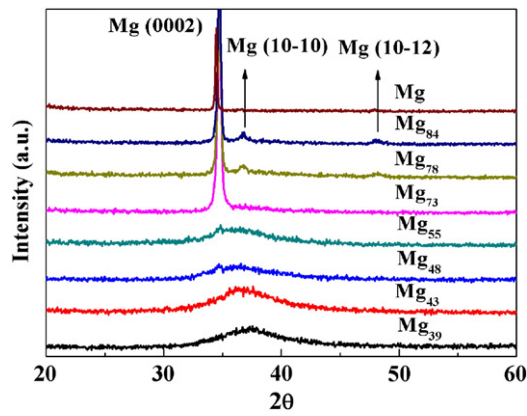


Fig. 1. XRD diffraction patterns of the MgCuZr alloy thin films. The films with high Mg contents have a strong (0002) basal plane texture, making other peaks difficult to be seen.

determined by the average data of all tests. Then, micropillars (measuring $\sim 1 \mu\text{m}$ in diameter and $\sim 2 \mu\text{m}$ in height) were machined by FIB and subjected to microcompression testing using the same MTS nanoindenter XP equipped with a flat-end tip [13,14], under the loading-rate-control mode at 0.02 mN s^{-1} . The peak load was set at 1.5 mN for Mg39 and Mg43, 1.3 mN for Mg48, 1.2 mN for Mg55, Mg73, Mg78 and Mg84; and 0.8 mN for pure Mg, due to the fact that these pillars are intrinsically weaker with increasing Mg content.

3. Results and discussion

The XRD results of the MgCuZr alloy thin films are shown in Fig. 1, indicating a transition from fully amorphous to crystalline phases with increasing Mg content. For the thin films with a lower Mg, such as Mg39 and Mg43, they are basically fully amorphous. With increasing Mg content from 48 to 55 at.%, a very minor peak at 34.7° is found, corresponding to the (0002) diffraction plane of the hexagonal close-packed (HCP) Mg. For Mg78, Mg84 and pure Mg, minor peaks at 36.8° and 48° corresponding to (10 $\bar{1}$ 1) and (10 $\bar{1}$ 2) planes are also seen. The strong (0002) basal plane texture is clearly seen. Here, we can find that the transition stage from fully amorphous to fully crystalline phase in the MgCuZr alloy system resides from 43 to 73 at.% Mg. This transition composition range is somewhat wider than other thin film systems, such as ZrCuTiTa [15], Cu–W [16] and Ag–Ni [17] (the latter three are all narrow within ~ 10 at.%).

The mutual heat of mixing for Mg–Cu, Zr–Cu and Mg–Zr, equals to -3 , -23 , and $+6 \text{ kJ/mol}$, respectively [18], meaning Mg would be pushed out by Zr. As Mg content is greater than 73 at.%, the dominant

Mg would supersaturate Zr and Cu, forming the Mg-based crystalline phase. Note that we might consider that Zr could also precipitate out in the Mg matrix when Mg content is high. However, this did not happen. This is because that, with the highly negative heat of mixing between Zr and Cu, these two atoms would attract each other strongly, forming tight Zr–Cu bonds and difficult to be separated. The strong attractive force between Zr and Cu suppresses the Zr precipitation in the Mg matrix when the Mg content is high.

Fig. 2(a) shows the nanoindentation load–displacement curves, which can be divided into two groups. The first group for Mg39 to Mg55 possesses the pop-in events with increasing applied load, typical for metallic glasses or metallic glass composites under nanoindentation; and the second one for Mg73 to pure Mg shows smooth curves, typical for ductile crystalline metals. Pop-in events are the discontinuous strain caused by the shear band propagations within the metallic glasses. As the Mg particles are precipitated, the shear band propagations would be interacted and suppressed by the Mg particles in the first group. For the other group, crystalline Mg becomes the matrix and the deformation mechanism now is changed to dislocation activity, not shear banding. As a result, the load–displacement curve becomes smooth. The elastic modulus (E) and hardness (H) of the MgCuZr alloy thin films, measured by nanoindentation, are shown in Fig. 2(b). It is obvious that the modulus decreases from ~ 90 to $\sim 70 \text{ GPa}$, and hardness from ~ 5.5 to $\sim 1.5 \text{ GPa}$, with increasing Mg content. We can find that the trend of elastic modulus is slightly irregular, presumably resulting from the complicated influence of bonding length between Mg and Zr/Cu [15,19]. For Mg73 to Mg84, the hardness remains around 3.2 GPa. Basu et al. [20] also found that the modulus would rise with a high volume fraction of crystalline phase in partially crystalline La-based bulk metallic glass system. It appears that the modulus has been affected via the interaction and formation of rigid networks of crystalline phases. This is a tremendous improvement, compared with the hardness of about 0.4–0.8 GPa for commercial Mg alloys possessing grain sizes $\sim 100 \mu\text{m}$ [21], or with the hardness of 2 GPa for nanocrystalline Mg alloys [22].

Microcompression testing was utilized for extracting the uniaxial compression mechanical characteristics. As shown in Fig. 3(a), the metallic-glass or metallic-glass composite MgCuZr thin films, i.e., Mg39 to Mg73, exhibit the characteristics of strain bursts. The first apparent strain burst is usually defined as the yield stress. In comparison, some gradual transformation from elastic to plastic deformation can be found in Mg78, Mg84 and pure Mg. Fig. 3(b) shows the variation trend of the yield stress with increasing Mg content. Note that even for Mg78 and Mg84, with predominant Mg crystalline phase retained, the yield stress can still maintain around 1.5 GPa, significantly greater than the yield stress for pure Mg or commercial Mg alloys (around 0.1 to 0.4 GPa).

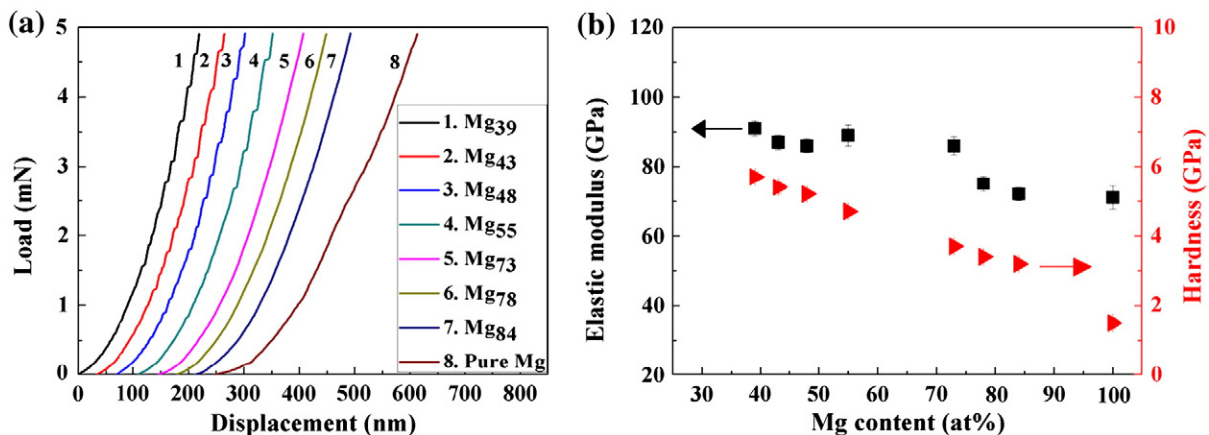


Fig. 2. (a) Load–displacement curves of the MgCuZr alloy thin films. For easy presentation, each curve in (a) is shifted about 40 nm to the right to separate these eight curves. (b) Variation of the elastic modulus and hardness extracted from nanoindentation of the MgCuZr micropillars as a function of Mg content. The datum scattering was small, with standard deviations around 2.0–3.5 GPa for modulus and 0.07–0.18 GPa for hardness.

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