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Mg–Y–Cu bulk metallic glass prepared by mechanical alloying and vacuum hot-pressing

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Abstract

We have studied the amorphization behavior of $Mg_{85-x}Y_{15}Cu_x$ (x = 20–40) alloy powders synthesized by mechanical alloying technique. The as-milled powders were mainly amorphous after 10 h of milling. The thermal stability of these $Mg_{85-x}Y_{15}Cu_x$ glassy powders was investigated by differential scanning calorimeter (DSC). The ranges of Tg, T_x and ΔT_x are around 430–459, 467–497, and 30–46 K, respectively. The $Mg_{49}Y_{15}Cu_{36}$ glassy powders exhibit the largest supercooled region of 46 K. The amorphization behavior of $Mg_{61}Y_{15}Cu_{24}$ was examined in details. Amorphous phases gradually became dominant after 7.5 h of milling and fully amorphous powders formed at the end of milling. The thermal stability of $Mg_{61}Y_{15}Cu_{24}$ glassy powders was similar to that of melt-spun $Mg_{60}Y_{15}Cu_{25}$ amorphous alloys. $Mg_{61}Y_{15}Cu_{24}$ bulk metallic glass with homogeneously embedded nanocrystalline precipitates was successfully prepared by vacuum hot pressing. It was found that the applied pressure during consolidation could enhance the thermal stability and prolong the existence of amorphous phase inside $Mg_{61}Y_{15}Cu_{24}$ powders.
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1. Introduction

Recently, new metallic glasses with a wide supercooled liquid region exceeding 20 K have been prepared in a number of Mg-based alloy systems, such as Mg–Ni–Ln (Ln = Ce, La), Mg–Ca–Al, Mg–Cu–Y, and Mg–Cu–Y–TM (TM = Ag, Zn, Al, Li) [1–3]. A wide supercooled region, though not necessarily implying improved glass forming ability, is an important indicator for bluk metallic glass (BMG). The supercooled liquid region is defined by the temperature range, $\Delta T = T_x - T_g$, between the glass transition temperature (T_g) and crystallization temperature (T_x). The increase of ΔT means that the stability of the supercooled liquid state against crystallization increases and, therefore, enables the formation of bulk metallic glass (BMG) by conventional casting techniques at low cooling rates ranged from 1.5 to 100 K/s. Indeed, $Mg_{65}Y_{10}Cu_{25}$ BMG with diameter up to 7mm has been produced by Inoue et al. [4]. These new alloys are expected to expand the application fields of bulk metallic glass due to their high specific strength to weight ratio and relatively low cost.

It is well-known that both high reduced glass transition temperature T_g/T_m and large supercooled liquid region ΔT are essential for the formation of BMG by rapid solidification [5]. However, this also restrict BMG formation to near-eutectic compositions where supercooling can be realized without nucleation of crystalline phases. Mg-based BMG are generally prepared by high-pressure die-casting [5] or mold casting method [6]. Mg materials must be melted before these processes. As melted Mg reacts with oxygen violently, a complicated device is required to control the atmosphere. BMG have also been prepared successfully by extruding atomized amorphous $Mg_{85}Y_{10}Cu_5$ [7], $Mg_{87.5}Y_{7.5}Cu_5$, and $Mg_{70}Ca_{10}Al_{20}$ powders [8]. A clean atomization process to control oxygen and moisture within 1 ppm, however, is required to prepare corresponding amorphous powders. In contrast, solid Mg does not react as easily with oxygen as their liquid counterparts. Therefore, an alternative way to prepare amorphous alloy is via solid-state amorphization reaction (SSAR processes) [9]. The techniques to synthesize amorphous alloys via SSAR include hydrogenation [10], multilayer interdiffusion [11], and mechanical alloying (MA). As previous investigations demonstrated, amorphization by mechanical alloying has been observed for a variety of binary and ternary alloy systems [12–14]. The product material of mechanical alloying is in powdered form and is suitable for compaction

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and densification into various shapes. In this paper, we report the glass formability and thermal stability of mechanically alloyed $Mg_{85\text{-}x}Y_{15}Cu_x$ ($x=20$–40) powders prepared by high-energy ball milling. The amorphization behavior of $Mg_{61}Y_{15}Cu_{24}$ and its BMG compaction was also investigated.

2. Experimental procedure

Elemental powders of Mg (99.9%, <325 mesh), Y (99.8%, <325 mesh), and Cu (99.5%, <100 mesh) were weighed to yield the desired compositions: $Mg_{85\text{-}x}Y_{15}Cu_x$ ($x=20$–40), and then canned into an SKH 9 high speed steel vial together with Cr steel balls under an argon-filled glove box, where a SPEX 8000D shaker ball mill was employed for MA. General details of mechanical alloying process are given elsewhere [15]. The bulk samples were prepared by consolidating the as-milled powders in a vacuum hot pressing machine at 453 K under a pressure of 1.20 GPa. The structure of the as-milled and bulk samples was analyzed by X-ray diffractometer (XRD, Siemens D-5000 diffractometer) and synchrotron extended X-ray absorption fine structure (EXAFS) technique. Thermal analysis was investigated using a Dupont 2000 differential scanning calorimeter (DSC) at a heating rate of 40 K/min. EXAFS measurements were performed at the Wiggler-C beamline of the National Synchrotron Radiation Research Center (NSRRC) in Hsinchu, Taiwan. Detailed data analysis procedures are available elsewhere [16].

3. Results and discussion

3.1. Amorphization behavior of $Mg_{61}Y_{15}Cu_{24}$

Fig. 1 shows the X-ray diffraction patterns of the starting and as-milled $Mg_{61}Y_{15}Cu_{24}$ powders as a function of milling time. At the early stage of milling, crystalline Mg, Y, and Cu peaks were observed. The peak intensities decreased gradually with increasing milling time. An amorphous indicating broad diffraction peak at $2\theta=33$–38° can be noticed after 5 h of milling treatment and became dominant after 7.5h of milling. Fully amorphous powders formed at the end of milling. The gradual decrease of the elemental crystallinity at the early milling stages and successive formation of amorphous phases is similar to what is observed during the amorphization by mechanical alloying in many binary [12] or multicomponent [13] systems.

While X-ray diffraction technique served as a long-range probe, synchrotron EXAFS technique can be used to reveal the local atomic variations during MA. Fig. 2a shows the EXAFS spectra of Cu K edge (i.e. 8979 eV) of the as-milled powders. The amplitude of oscillations is observed to decrease as the milling time increases. This indicates that the crystallinity of Cu elements decreased as the milling process proceeded. After determining the experimental threshold (i.e. E_0), conversion into k space, weighting, and the Fourier transformation of the $k^2\chi(k)$ into real space, the radial distribution function (RDF) of the local atomic environment of detected atom (i.e. Cu) can be determined [16]. Magnitudes of the RDF indicate the coordination numbers of detected atom at various distances. Fig. 2b shows the corresponding RDFs of the as-milled powders where the magnitudes of the nearest neighbor contributors is observed to decrease significantly after 5 h of milling. This indicates that Cu element starts to amorphize with other elements. The magnitudes decreased further with increasing milling time. This implies that amorphous phases become dominant gradually. Similar results have also been found in Fe–Ta, Zr–Ti–Al–Ni–Cu and Ni–Zr–Ti–Si systems [13,16,18].

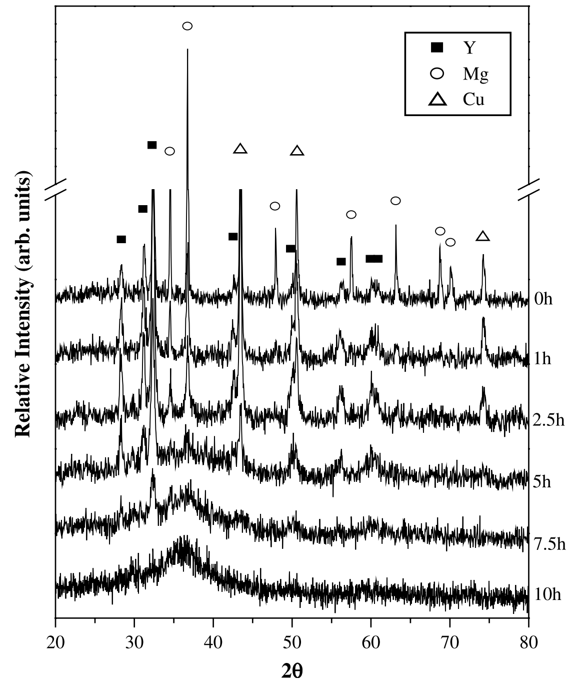

Fig. 1. X-ray diffraction patterns of mechanically alloyed $Mg_{61}Y_{15}Cu_{24}$ powders as a function of milling time.

The thermal stability of $Mg_{61}Y_{15}Cu_{24}$ amorphous powders were investigated by differential scanning calorimetry and the corresponding DSC scans were shown in Fig. 3. It can be seen that the amorphous powders exhibits an endothermic heat event due to the glass transition followed by a sharp exothermic peak indicating the successive stepwise transformations from a supercooled liquid state to crystalline phases. The glass transition and crystallization temperatures were defined as the onset temperatures of the endothermic and exothermic DSC events, respectively. The glass transition temperature (T_g) and the crystallization temperature (T_x) are 432 and 471 K, respectively. The supercooled liquid region ΔT_x is 39 K. For melt-spun $Mg_{60}Y_{15}Cu_{25}$ amorphous alloys, Kim et al. [19] have reported that T_g,T_x and ΔT_x are 439, 477 and 38 K, respectively. MA is a high-energy ball milling process involving the collision of fine powders with milling balls and vial. The oxygen and iron contents in mechanically alloyed powders are always more than 1 at.% due to the large surface area of the fine particles and iron contamination from the milling tools. Seidel et al. [20] have investigated the effect of oxygen and iron on the thermal properties of mechanically alloyed and rapidly quenched Zr–Al–Cu–Ni and Zr–Ti–Cu–Ni

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