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Consolidation of mechanically alloyed $Mg_{49}Y_{15}Cu_{36}$ powders by vacuum hot pressing

P.Y. Lee [a,*], C. Lo [a], J.S.C. Jang [b]

[a] *Institute of Materials Engineering, National Taiwan Ocean University, Keelung, Taiwan*
[b] *Department of Materials Science and Engineering, I-Shou University, Kaohsiung, Taiwan*

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Abstract

The preparation of $Mg_{49}Y_{15}Cu_{36}$ metallic glass powders was accomplished by mechanical alloying of pure Mg, Y, and Cu after 10 h of milling. The thermal stability of these $Mg_{49}Y_{15}Cu_{36}$ amorphous powders was investigated by differential scanning calorimeter (DSC). The T_g, T_x and ΔT_x are 450, 492, and 42 K, respectively. The as-milled $Mg_{49}Y_{15}Cu_{36}$ powders were then consolidated by vacuum hot pressing into disc compacts with a diameter and thickness of 10 and 1 mm, respectively. After vacuum hot pressing, bulk $Mg_{49}Y_{15}Cu_{36}$ metallic glass with nanocrystalline precipitates homogeneously embedded in a highly dense glassy matrix was obtained. The applied pressure during consolidation can enhance the thermal stability and prolong the existence of amorphous phase inside $Mg_{49}Y_{15}Cu_{36}$ powders.

Keywords: Mechanical alloying; Metallic glass; High-pressure; Thermal analysis

1. Introduction

Recently, new bulk metallic glass (BMG) with a wide supercooled liquid region exceeding 20 K have been prepared in a number of Mg-based alloy systems [1–3]. These new alloys are expected to expand the application fields of bulk metallic glasses due to their high specific strength to weight ratio, good ductility and relatively low cost. Mg-based BMG are generally prepared by high-pressure die-casting or mold casting method. Mg materials must be melted before these processes. As melted Mg reacts with oxygen violently, a complicated device is required to control the atmosphere. In contrast, solid Mg does not react as easily with oxygen as its liquid counterparts. Therefore, an alternative way to prepare amorphous alloy is *via* solid-state amorphization reaction (SSAR). The techniques to synthesize amorphous alloys *via* SSAR include hydrogenation, multilayer interdiffusion, and mechanical alloying (MA) [4]. As previous investigations demonstrated, mechanical alloying has been used successfully to synthesize amorphous alloy powders [5,6]. In this paper, we report on the glass formability and thermal stability of mechanically alloyed $Mg_{49}Y_{15}Cu_{36}$ powders prepared by high-energy ball milling. The consolidation of $Mg_{49}Y_{15}Cu_{36}$ metallic glass powders into bulk form by a vacuum hot-pressing method was also investigated in details.

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 composition $Mg_{49}Y_{15}Cu_{40}$ and then canned into a 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 [5]. The as-milled powders were consolidated in a vacuum hot pressing machine to prepare bulk samples with a diameter of 10 and 1 mm in thickness. The hot pressing was performed at 473 K for 30 min under a pressure of 0.72, 0.96 and 1.20 GPa, respectively. The structure of the as-milled and bulk samples was analyzed by X-ray diffractometer (XRD, Siemens D-5000 diffractometer) and scanning electron microscopy (SEM, Hitachi S-4100 SEM). Thermal analysis was investigated using a Dupont 2000 differential scanning calorimeter (DSC), where the sample was heated from room temperature to 650 K under a purified argon atmosphere at constant heating rates of 40 K/min.

3. Results and discussion

Fig. 1 shows the X-ray diffraction patterns of the starting and as-milled $Mg_{49}Y_{15}Cu_{36}$ powders for different of milling time. At the early stage of milling, crystalline Mg, Y, and Cu peaks were observed. The peak intensities decreased with increasing milling time. With further ball milling up to 7.5 h,

* Corresponding author. Tel.: +886 2 24622192; fax: +886 2 24625324.
E-mail address: pylee@mail.ntou.edu.tw (P.Y. Lee).

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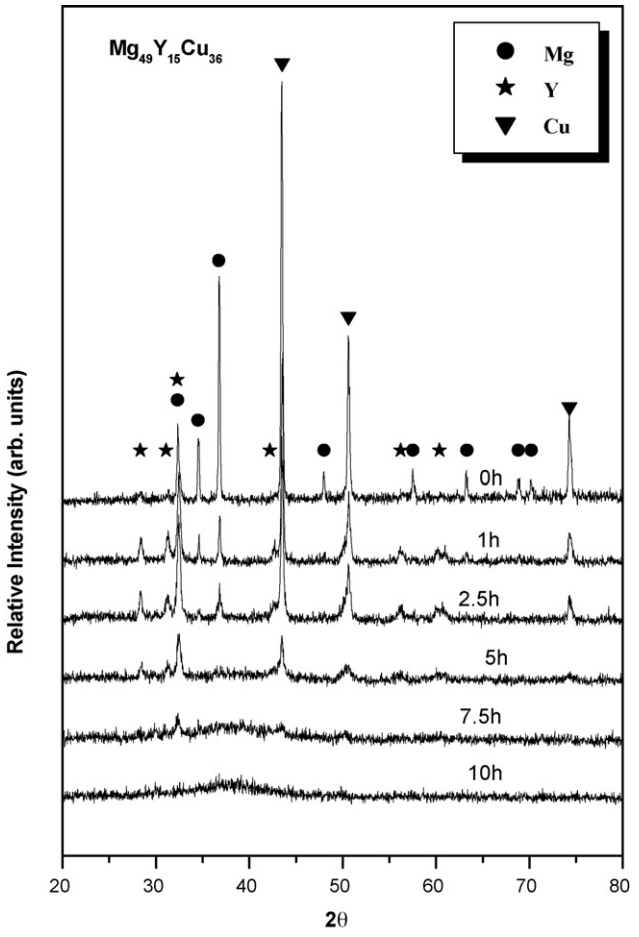

Fig. 1. X-ray diffraction patterns of mechanically alloyed $Mg_{49}Y_{15}Cu_{36}$ powders for various milling time.

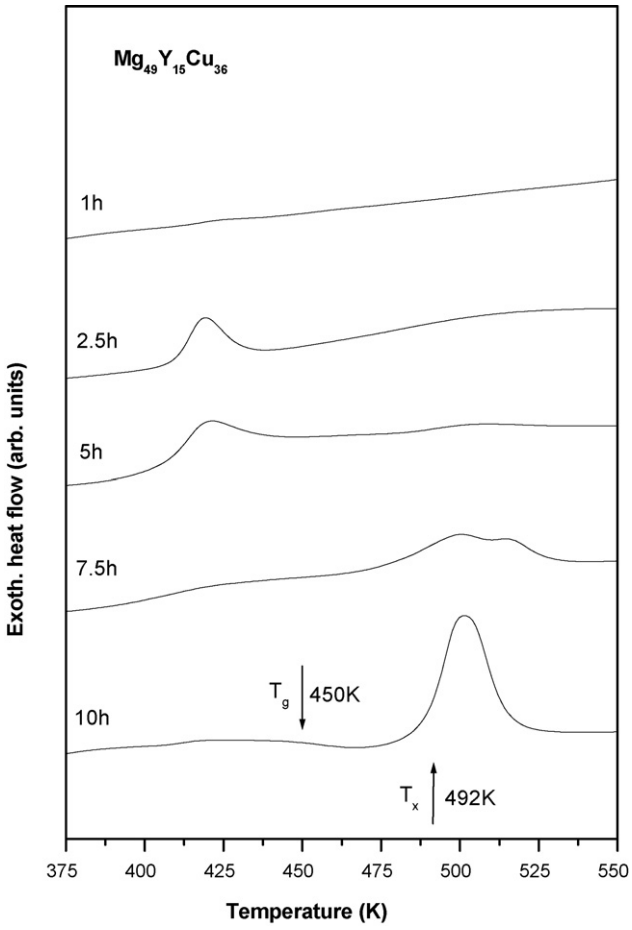

Fig. 2. DSC traces of mechanically alloyed $Mg_{49}Y_{15}Cu_{36}$ powders as a function of milling time.

a broad diffraction peak at $2\theta = 35$–40° is the main feature of this patterns, indicating that the as-milled powders are predominantly amorphous. Fully amorphous powders were successfully prepared after 10 h of milling.

The thermal stability of $Mg_{49}Y_{15}Cu_{36}$ amorphous powders were investigated by differential scanning calorimetry and the corresponding DSC scans are shown in Fig. 2. 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 temperature (T_g) and the crystallization temperature (T_x) are 450 and 492 K, respectively. The supercooled liquid region ΔT_x is 42 K. Based on the DSC results, the 10 h as-milled $Mg_{49}Y_{15}Cu_{36}$ powders were consolidated into disk shape by vacuum hot pressing. The powders were hot pressed at 473 K under pressures of 0.72, 0.96, and 1.20 GPa for 30 min, respectively. Fig. 3 shows a typical consolidated sample of bulk metallic glass that exhibited smooth outer surface and metallic luster. Its polished cross-sectional view as examined by SEM is shown in Fig. 4. Because neither pores or voids are seen, this indicates bulk $Mg_{49}Y_{15}Cu_{36}$ with highly dense structure have been prepared successfully. Fig. 5 displays the XRD traces and DSC scans of the as-milled and bulk $Mg_{49}Y_{15}Cu_{36}$ specimens. A broad halo with overlapped sharp crystalline reflections is identifiable in the patterns for bulk sample. The crystalline peaks could be indexed to be Mg_2Cu and $Mg_{24}Y_5$ phase. Though not shown here, TEM observation confirms that fine precipitates with a grain size less than 10 nm are homogeneously dispersed in the glassy matrix. For

Fig. 3. The outer morphology of the $Mg_{49}Y_{15}Cu_{36}$ BMG after vacuum hot pressing at 473 K with a pressure of 1.2 GPa.

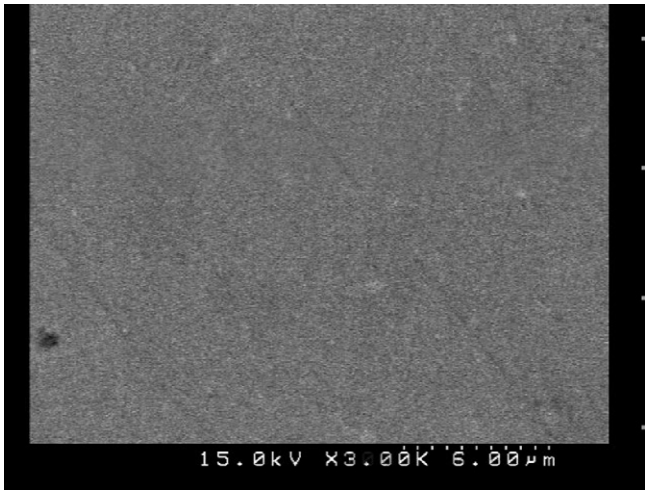

Fig. 4. Polished cross-sectional view of the $Mg_{49}Y_{15}Cu_{36}$ disk specimen after vacuum hot pressing at 473 K with a pressure of 1.2 GPa.

comparison purposes, the corresponding data for as-milled powder isothermally annealed at 473 K for 30 min are also shown. Although the annealing temperature and time is the same as that of the hot-pressed sample, the XRD pattern is only consisting of crystalline peaks belong to Mg, Mg_2Cu and $Mg_{24}Y_5$ phase. The broad halo pattern was not observed which indicated the amorphous phase was vanished during annealing. This can be further confirmed by the absence of exothermic peak around 500 K on the DSC scan. The major difference between heat-treated and hot-pressed specimens is the employment of high pressure. The high pressure involved during consolidation can thus prolong the existence of amorphous phase inside $Mg_{49}Y_{15}Cu_{36}$ powders. It also noticed that partial crystallization was occurred for the samples hot-pressed at low pressure (0.72 GPa). However, with increasing pressure, the amount of residual amorphous phase in the hot-pressed samples increases, meaning that the crystalline fraction decreases when the powder is hot-pressed at 473 K under higher pressures. This result indicates that the consolidation pressure suppressed the crystallization in the $Mg_{49}Y_{15}Cu_{36}$ BMG. The crystallization behaviors of BMG under high pressure have been studied extensively [7–9]. In general, applied pressure on BMG might have three effects [10]. The first effect is densification, which could favor the appearance of the closest structure and favor the crystallization process. The second effect is suppression of atomic mobility at high pressures, which reduces the atomic diffusion in metallic glasses. The third effect is due to changes in relative Gibbs free energies of the glassy phase and crystalline phases by pressure. The effects can alter the sequence or the relative volume fraction of crystalline phases in the hot-pressed samples. For a certain amorphous phase, its crystallization behavior under pressure may depend on which is the dominating factor among the three effects. In the present study, due to the first effect mentioned above, pressure can promote the crystallization process at the lowest pressure of 0.72 GPa. However, with increasing pressure to ≥0.96 GPa, the long-range atomic diffusion in the BMGs becomes more difficult because the diffusion barrier was elevated by the reduction of volume and annihilation of excess free volume caused by high pressure. This means the second effect of suppression of atomic mobility at higher pressures becomes dominating factor, thus the amount of amorphous phase in the hot-pressed samples increases with increasing pressure. Similar results have also been found in $Al_{84}Ni_{10}Ce_6$, $Al_{82.5}Ni_5Y_8Co_2Zr_{2.5}$ [11] and $Zr_{55}Al_{10}Ni_5Cu_{30}$ [12] BMGs.

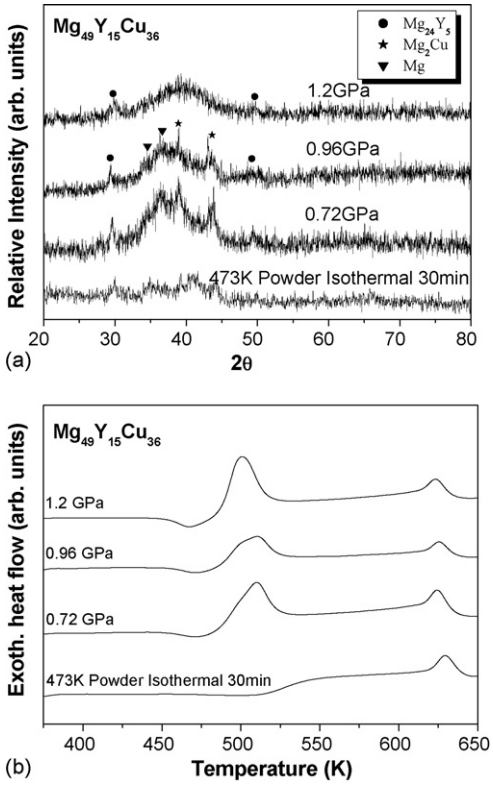

Fig. 5. (a) XRD patterns and (b) DSC traces of annealed powders and bulk $Mg_{49}Y_{15}Cu_{36}$ specimens.

4. Conclusion

We have studied the amorphization behavior of $Mg_{49}Y_{15}Cu_{36}$ alloy powders synthesized by mechanical alloying technique. The as-milled powders were mainly amorphous after 10 h of milling. The thermal stability of $Mg_{49}Y_{15}Cu_{36}$ amorphous powders was investigated by differential scanning calorimeter. The T_g, T_x and ΔT_x are around 450, 492, and 42 K, respectively. $Mg_{49}Y_{15}Cu_{36}$ bulk metallic glass with nanocrystalline precipitates homogeneously embedded in a highly dense glassy matrix 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 $Mg_{49}Y_{15}Cu_{36}$ powders.

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