



Synthesis of impurity-insensitive Zr-based bulk metallic glass

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ABSTRACT

The glass forming ability (GFA) and mechanical properties of Zr-based bulk metallic glasses (BMGs) are extremely sensitive to oxygen and other metalloids in raw materials, which severely limits the wide commercial application of this material for the drastic increase of manufacturing cost. In this paper, an impurity-insensitive $Zr_{50}Ti_4Y_1Al_{10}Cu_{25}Ni_7Co_2Fe_1$ bulk metallic glass with large component number was synthesized. This alloy shows critical diameter (d_c) of 16 mm and similar thermal stability and mechanical properties using both high-purity and low-purity raw materials. The effect of large component number on impurity-insensitivity is discussed in detail. Successful formation of impurity-insensitive Zr-based BMG indicates that increasing component number would be an effective strategy for reducing the influence of harmful impurities on BMGs, which provides a useful guideline for fabrication of low-cost industrial productions.

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1. Introduction

In recent years, bulk metallic glasses (BMGs) have attracted extensive attention due to their special structure and properties [1,2]. Among them, Zr-based BMGs show unique mechanical properties (i.e., high elasticity, low young's modulus, high Poisson's ratio, high strength and good plasticity [3–5]), outstanding superplastic forming ability [6], significant corrosion resistance [7] and superior biocompatibility [8], and are promising in the future as advanced materials in various applications.

Nevertheless, in order to realize the widespread commercial application of Zr-based BMGs, merely remarkable material performance is inadequate. In fact, low manufacturing cost is an important factor for industrial production of materials. However, it has been reported that Zr-based BMGs are sensitive to oxygen and other metalloids in raw materials, which would lead to significant decrease in their glass forming ability (GFA) and mechanical properties [9–13]. As a consequence, high-purity raw materials are required in fabrication of Zr-based BMGs for good performance. Obviously, this would drastically increase the manufacturing cost, which to some extent hampers the commercial application of Zr-based BMGs. Therefore, the key factor for industrial production of low-cost BMGs is to reduce the harmful effect of impurities on GFA and mechanical properties.

To diminish the negative effect of impurities on Zr-based BMGs, minor addition of elements (e.g., rare-earth elements) which have strong interaction with oxygen and other metalloids was commonly used [9,12–14]. It should be noted that, the aim of this

strategy is to absorb impurities to lower their influence on glassy matrix. However, it could not entirely eliminate these impurities in the glassy matrix, for both Zr and Ti show high capability for absorbing these elements as well. In addition, as various impurities are contained in the raw materials, minor addition of single element may not be sufficient for the purification. Since removing all the impurities out of glassy matrix is extraordinary difficult, it is necessary to enhance the impurity-insensitivity of Zr-based BMGs, which could maintain the GFA and mechanical properties with the existence of a certain amount of impurities.

Considering the adverse effects of harmful impurities on glassy matrix, designing glass forming compositions with large component number would be a valid solution for synthesizing Zr-based BMGs with high impurity-insensitivity. It was found that large component number would lead to complex interaction between constituent elements and high mixing entropy in the alloys [15], which would result in sluggish diffusion of elements [16]. Based on these effects, the impurities could be possibly dispersed in the glassy matrix steadily, and even the nucleation of intermetallic phases caused by these impurities occurs, further growing process would be impeded. Moreover, it was reported that the composition with large component number would have complex crystallization behavior [15], and the complex competition between these crystalline phases might inhibit the precipitation of impurity-induced crystalline phases. Therefore, it is worth expecting that, large component number would be beneficial to enhance the impurity-insensitivity in BMGs.

In this work, an octonary $Zr_{50}Ti_4Y_1Al_{10}Cu_{25}Ni_7Co_2Fe_1$ BMG with high impurity-insensitivity was designed and the specimens containing different levels of impurities were fabricated. Our findings indicate that this composition with large component number is impurity-insensitive in GFA, thermal stability and mechanical properties, which is significant to the development of BMGs for commercial productions.

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2. Experimental

Alloy ingots with nominal composition of $Zr_{50}Ti_4Y_1Al_{10}Cu_{25}Ni_7Co_2Fe_1$ (in at.%) were prepared by arc melting the mixtures of the raw materials under a high-purity argon atmosphere. Two ingots using pure Zr and Ti (purity above 99.9 wt.%), and sponge Zr and Ti (the compositions are listed in Table 1), respectively, were fabricated (denoted as Z1, Z2 hereinafter, for convenience). Other elements utilized in this paper were pure metals with purities of above 99.9 wt.%. Moreover, for further evaluation of the impurity-insensitivity of alloys, two levels of impurities were added in this composition, one is the addition of 0.02 at.% Cr, 0.02 at.% Mn, 0.02 at.% C and 0.02 at.% Si in Z2 (namely, (Z2) $_{0.9992}Cr_{0.02}Mn_{0.02}C_{0.02}Si_{0.02}$, denoted as Z3), and the other is the addition of oxygen by melting for 1 min and casting the ingots of Z3 under argon atmosphere with oxygen partial pressure of 1 Pa (denoted as Z4). All the ingots were melted four times to ensure compositional homogeneity. Rod-shape specimens with 2 mm in diameter were prepared using copper mold injection casting method, and those with 16 mm and 18 mm in diameter were produced via copper mold tilt-pour casting. Except Z4, all the fabrication processes were under high purified Ar atmosphere. To analysis the microstructure of the specimens, a Bruker AXS D8 X-ray diffractometer with Cu K_{α} radiation was utilized. A JXA8100 electron probe microanalyzer (EPMA) was used for the investigation of the microstructures of the specimens. A NETZSCH DSC 404C differential scanning calorimeter (DSC) was carried out to characterize the thermal behaviors of the glassy specimens at a heating rate of $0.33\text{ K}\cdot\text{s}^{-1}$. A SANS 5504 testing machine was employed and the specimens of 2 mm in diameter and 4 mm in height were adopted in the compressive tests to investigate the mechanical properties of Z1–Z4 at a strain rate of $4.17 \times 10^{-4}\text{ s}^{-1}$ at ambient temperature. At least five specimens of Z1–Z4 were examined in the test. A JEOL 6010 scanning electron microscope (SEM) was carried out for the observation of fractured surfaces of Z1–Z4 specimens. A JEOL 2100F transmission electron microscope (TEM) was utilized for further observation of the specimens. These specimens were cut from the rods for the test of mechanical properties by electron-discharging machine and ground to less than $50\text{ }\mu\text{m}$ in thickness, and then etched through ion milling.

3. Results

XRD patterns of Z1–Z4 specimens with 16 mm and 18 mm in diameter are shown in Fig. 1. The main hump without obvious Bragg's peaks corresponding to crystalline phases could be observed in the XRD patterns of the Z1–Z3 specimens with 16 mm in diameter, indicating the formation of fully glassy structure within the resolution limit of XRD. In contrast, crystalline peaks could be identified in the patterns of rod specimens with 18 mm in diameter, demonstrating that Z1–Z3 have d_c of 16 mm. The crystalline peaks emerge in the curve of Z4 specimen with 16 mm in diameter, indicating that further addition of oxygen in Z3 would lead to slight decrease of GFA. However, these crystalline peaks are much smaller than broad scattering peak in the XRD pattern of Z4 specimen with 16 mm in diameter, indicating high volume fraction of glassy phase. This result demonstrates that this alloy could effectively reduce the harmful influence of a certain amount of impurities on GFA.

Fig. 2(a)–(d) show the SEM images taken near the middle of Z1–Z4 specimens with 18 mm in diameter. Near-spherical particles with dark contrast corresponding to the crystalline phase could be observed in the glassy matrix. The main component of the crystalline phases in Z1–Z4

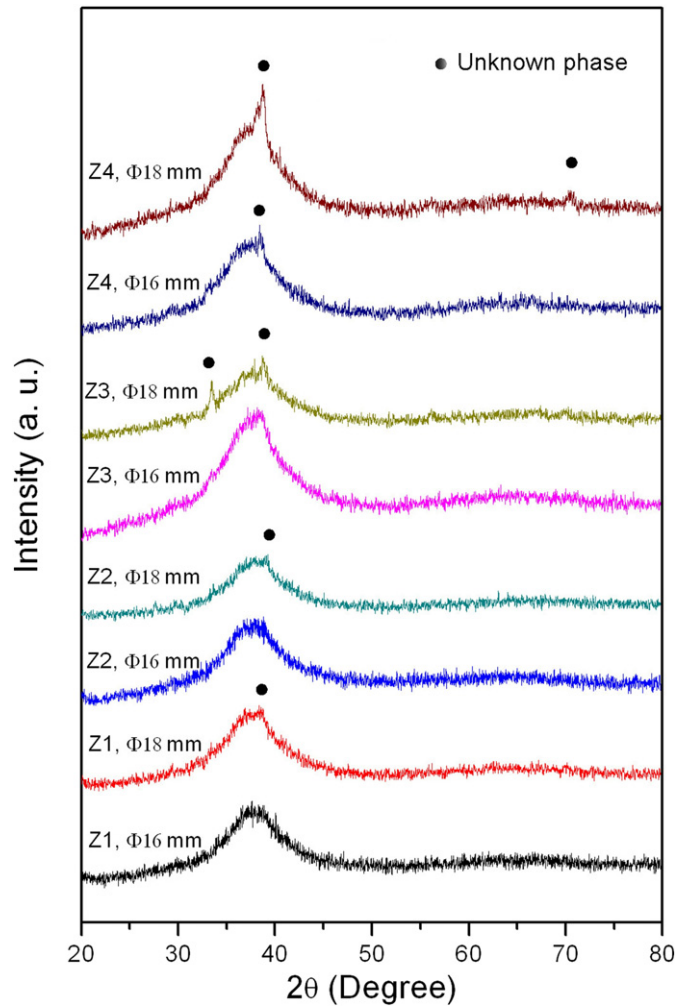


Fig. 1. XRD patterns of Z1–Z4 rod specimens with 16 mm and 18 mm in diameters.

was tested by energy dispersive X-ray spectrum (EDS) and listed in Table 2. From the results, it could be found that, the crystalline phases in these specimens have almost identical main composition, indicating that they are possibly the same phase. However, the size, volume fraction and distribution of the crystalline phases in Z1–Z4 are very different. It is shown in Fig. 2(a) that, a mass of tiny precipitates with several micrometers in diameter is well dispersed in the glassy matrix, and merely a few particles with larger diameters could be observed in Z1. Compared to Z1, Z2 possess more spherical particles with large sizes, but tiny precipitates are rarely observed (shown in Fig. 2(b)), while Z3 and Z4 show significant enhancement in the volume fraction of crystalline phases (shown in Fig. 2(c) and (d)), which is in accordance with the XRD results. Moreover, limited amount of crystalline particles in Z1 and Z2 is decomposed, and the morphologies of the decomposed microstructures are different, while this phenomenon is rarely observed in Z3 and Z4. The result indicates that, although main compositions of the phases in Z1–Z4 are extremely similar, these specimens display distinct differences in the phase formation and stability, which might result from the influence of impurities. In addition, from the EDS results, the

Table 1
The compositions of sponge Zr and Ti (in wt.%).

Raw materials	Zr + Hf	Ti	O	Mg	Cr	Mn	Si	C	Cl	Bal.
Sponge Zr	≥99.4	0.005	0.10	0.06	0.02	0.01	0.01	0.05	0.13	≤0.215
Sponge Ti	—	≥99.6	0.08	0.07	—	0.01	0.03	0.03	0.08	≤0.1

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