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Fe-based bulk metallic glass composites without any metalloid elements

J.E. Gao, Z.P. Chen, Q. Du, H.X. Li, Y. Wu, H. Wang, X.J. Liu, Z.P. Lu*

State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083, China

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Abstract

Glass-formation of Fe-based bulk metallic glasses (BMGs) and their composites is strongly dependent on their metalloid content. A good Fe-based glass former usually needs a metalloid content of ~ 20 at.% or above; however, the high content of the metalloids usually has side-effects on the performance of the alloys. In this paper, we developed a series of metalloid-free Fe-based BMG matrix composites with a diameter of more than 10 mm in the Fe-Co-La-Ce-Al-Cu system. During cooling, phase separation took place, i.e. Fe-rich and Fe-depleted liquids formed. Upon subsequent cooling, the body-centered cubic (bcc)-Fe(Co, Al) solid solution and Ce(Fe, Co)₂ intermetallic phase precipitated out of the Fe-rich liquid and the remaining Co-La-Ce-Al-Cu liquid was eventually vitrified to form the amorphous matrix. Due to elemental partitioning, it was found that, for a given Fe content, there exists an optimum compositional range for Co and Ce so that a large fraction of the glassy matrix can form. In addition, the final microstructure of the current composites was strongly dependent on the cooling rates applied: excessively fast cooling rates restricted the diffusion and precipitation of the Fe atoms, which reduced formation of the glassy matrix.

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1. Introduction

In the past decades, Fe-based bulk metallic glasses (BMGs) and their composites have attracted much attention not only because of their superior properties, such as high fracture strength, high hardness, good corrosion resistance and excellent magnetic properties, but also because of their low cost of manufacture, thanks to from the plentiful natural resources of Fe [1–7]. Unfortunately, the shortcomings of the Fe-based BMGs, such as limited glass-forming ability (GFA) and room-temperature brittleness, severely restrict their widespread use. It is widely recognized that the metalloid elements (i.e. B, C, Si and P) are key glass-forming constituents in Fe-based alloy systems [8,9], and a good Fe-based glass-former often contains a metalloid content of \sim 20 at.% or above [3,10–13]. Nevertheless, it was found that the high content of the metalloid elements usually brings in side-effects on the soft magnetic properties and plastic deformation at room temperature in these Fe-based BMGs [14,15]. It is also important to develop Fe-based BMGs and their composites that contain no metalloid elements.

In our study, we made an attempt to design Fe-based BMG matrix composites with no metalloids by taking advantage of phase separation. We demonstrate this new strategy in the Fe-based Fe-Co-La-Ce-Al-Cu system, which possesses extraordinarily large formability with a maximum attainable size over 10 mm. The formation mechanism and phase competition upon solidification will be explored in detail.

2. Alloy design strategy

In an alloy system, if the enthalpy of mixing between the two main components is positive and there also exists a miscibility gap between them, phase separation tends to take place. Such a concept has been used to fabricate

^{*} Corresponding author. Tel.: +86 10 82375387; fax: +86 10 62333447. *E-mail address:* luzp@ustb.edu.cn (Z.P. Lu).

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MG composites containing either two kinds of amorphous phases or glassy-crystalline structures at various microstructural length scales [16–20]. To date, a variety of BMGs with good GFA have been synthesized in Pd- [21,22], Zr-[23,24], Mg- [25], Cu- [26], Fe- [11], Co- [27], Ni- [28], Ti-[29], La- [30], Ce- [31] and Y-based [32] alloy systems. Among these alloy systems, the largest critical size for glass formation is up to 80 mm [22]. Based on the concept of phase separation, we can select a known BMG with a good GFA. element A-based $A_m B_n C_y D_y E_z$ e.g. (m+n+x+y+z=100 at.%) as our starting composition, and then add an element M having a positive enthalpy of mixing and a miscibility gap with the main component A. As a result, two liquids, i.e. the M-rich region and Mdepleted region, will form at high temperatures. Due to the phase separation, the M-depleted region would have a composition close to the starting A-based alloy, which has a good GFA. During solidification, it can be thus anticipated that crystalline phases will be precipitated out of the M-rich liquid, while the M-depleted liquid will vitrify. As such, an M-based BMG composite consisting of the M-rich crystalline phases embedded in the A-based BMG matrix can be fabricated. The total volume fraction of the BMG matrix can be adjusted by the amount of the M addition. In realistic cases, the interaction between M and the other constituents will make the metallurgical process much more complex than as stated above.

In the present study, the La–Ce–Al–Co–Cu alloy system in which the largest attainable diameter for full glass formation reaches up to 32 mm [33] was initially selected as our starting composition. It is known that the heat of mixing of the Fe–La, Fe–Ce and Fe–Cu atomic pairs is +5, +3and +13 kJ mol⁻¹ [34], respectively, and there is a miscibility gap between Fe and La/Cu [35]. Therefore, sufficient additions of Fe into the bulk glass-forming system could induce occurrence of the phase separation at the liquid La–Ce–Al–Co–Cu state. Upon subsequent cooling, it is possible to obtain Fe-based BMG matrix composite containing no metalloid elements.

3. Experimental

Multicomponent alloy ingots were prepared by arcmelting a mixture of pure (>99 at.%) Fe, Co, La, Ce, Al and Cu in a Ti-gettered argon atmosphere. Cylindrical rods with a length of 50 mm and different diameters were fabricated by a conventional suction-casting method using copper molds under helium atmosphere. Phase identification in the as-cast samples was examined by X-ray diffraction (XRD) with Cu K α radiation. Thermal properties associated with glass transition and crystallization events were investigated by differential scanning calorimetry (DSC) in a Thermal Analysis Q100 apparatus at a heating rate of 20 K min⁻¹. The melting and solidification behaviors of these alloys were characterized using differential thermal analysis (DTA) at a rate of 20 K min⁻¹. Microstructures of the samples were characterized by a Zeiss SUPRA55 field-emission scanning electron microscope (SEM) equipped with energy-dispersive spectrometry (EDS) and a transmission electron microscope (TEM, JEM-2100). For the TEM observation, specimens were mechanically pre-thinned to a thickness of ~40 µm, and then doublejet electropolishing was used at ~243 K with a solution of 5 vol.% perchloric acid and 95 vol.% methanol. Compression specimens with a diameter of 2 mm and a height of 4 mm were prepared by an electrical discharge machine. Uni-axial compression tests were performed at room temperature at an approximately constant loading rate of $2 \times 10^{-4} \text{ s}^{-1}$. Nanoindentation tests were performed using a MTS Nano Indentater XP instrument with a Berkovich indenter at a loading rate of 0.05 s^{-1} .

4. Results

4.1. Composition design

The maximum attainable size of newly developed BMG matrix composites as a function of chemical composition in the Fe-(Co-La-Ce-Al-Cu) alloy system is shown in Fig. 1. For the alloys containing more than 40-55 at.% Fe, the maximum attainable diameter of the Fe-based BMG matrix composites could reach up to 10 mm, while for the alloys containing less than 40 at.% Fe, the critical diameter is much larger. For the starting composition La_{325} $Ce_{32} Co_{15}Al_{10}Cu_{10}$ (hereafter refer as to Fe0), fully glassy rods of 32 mm in diameter can be produced [33]. As the content of Fe is increased, the maximum attainable size of the BMG matrix composites is decreased. To illustrate glass formation behavior and understand phase competition mechanism in the current Fe-(Co-La-Ce-Al-Cu) system, three alloys, i.e. $Fe_{30}Co_{21.4}La_{14.4}Ce_{23.4}Al_{4.8}Cu_{6.0}$, Fe₄₀Co_{18.4}La_{8.8}Ce_{27.2}Al_{2.4}Cu_{3.2} and Fe₅₀Co_{17.2}La_{7.2}Ce_{21.4}-Al_{1.8}Cu_{2.4} (hereafter referred to as to Fe30Co21, Fe40Co18 Fe50Co17, respectively), and were selected for demonstration.



Fig. 1. The critical thickness for the BMG matrix composites as a function of alloy compositions.

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