



Effect of cooling rates on the structure, density and micro-indentation behavior of the Fe, Co-based bulk metallic glass



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ABSTRACT

The experiments demonstrate that ductility of the samples of bulk metallic glass (BMG) with the same chemical composition increased with decreasing sample size. It is shown that microhardness and density increases with decreasing the cooling rate. The fracture morphology of rods after compressive fracture were different on the cross section. Two characteristic features of the compressive fracture morphologies of metallic glasses (MGs) were observed in samples: smooth region and the vein pattern. Many parallel shear bands were observed on the deformed specimen with $\phi = 2$ mm in diameter. The results provide more understanding on the relationship among the cooling rate, structure and micro-indentation behavior of the Fe-Co-based BMGs.

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1. Introduction

Metallic glasses (MGs) also known as amorphous metals with metastable glassy states, are obtained by the rapid cooling of liquid alloys [1]. Because of their metastable structure metallic glass properties change both during ageing at room temperature and annealing at temperatures considerably below the crystallization temperature (T_x), which is associated with the phenomenon of the amorphous structure relaxation [2,3]. During the low-temperature annealing many changes of physical properties occur including density, specific heat, viscosity, stress relaxation, electrical resistance, internal friction, and above all fall of the plasticity [3,4]. It was also found that the physical properties of metallic glass and glass relaxation processes can be different depending on the casting conditions and thickness of the cast ribbon what is undoubtedly connected with the cooling rates and advancement of the relaxation processes at the fabrication stage of metallic glass [3].

Due to the attained geometrical shape, metallic glass is divided into conventional and bulk metallic glass (BMG). In the process of casting conventional metallic glass, the cooling rate is 10^4 – 10^6 K/s and they were confined to very thin sheets, ribbons or wires with the thickness in the range from 0.02 to 0.06 mm [1]. According to the effects of structural relaxation on the property changes of conventional metallic glasses induced by annealing, different cooling rates would result in the variation in glass transition temperature, density, structure sensitive properties such as hardness, and other properties. Thus the range of

application to the conventional metallic glass in the form of ribbons is limited because of their dimensions [5–7].

Bulk metallic glasses (BMGs) with different geometrical forms (ribbons/plates, rods, tubes) and various dimensions (thickness, diameter, full and inside diameter) are usually fabricated by rapid-quenching techniques in which the cooling rate is a key processing factor. When lower cooling rates are applied, from 10^6 to 1 K/s, the thickness of the ribbons is in the range 0.1 to 100 mm [5,8]. All BMGs are multi-compositional, and hence their elements tend to form characteristic local atomic clusters [9]. Following the principle of the efficient filling of space, Miracle [10] proposed face-centred cubic (fcc) packing of solute-centred clusters or short-range order (SRO) as the building scheme for metallic glass structures. Such a packing mode within a medium-range order (MRO) has been confirmed by Ma and co-workers [11] using atomic simulations and directly observed by Hirata et al. [12]. Recent studies [13] suggest that over the MRO, the clusters are connected via a fractal network with the dimension of 2.31, although there are some puzzles regarding this [14]. Regions between clusters are loosely packed clusters with large free volumes [15].

As known to many scientists, cooling rates play an important role in the fabrication of BMGs, although the dependence on it is not as great as in earlier times. Experiments indicate that structure and many physical properties are very sensitive to the cooling rate during fabrication [3,5–8,16–33]. Obviously, the cooling rate restricts the atomic rearrangement during the glass formation process and affects the properties of BMGs further [20]. For the sake of different cooling rates, it is probably that free volume will be different in samples produced by different methods. Since it is obvious that a free volume determines the density of metallic

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glasses it is possible that density of samples in different form (ribbon, rod, plate, ring) will be different [4].

Due to the lack of crystalline structure (lack of crystalline defects such as grain boundaries and dislocations) bulk metallic glasses (BMGs) may achieve unique mechanical, physical and chemical properties, which are superior to conventional metals and alloys. Chen fabricated an amorphous Pd-Cu-Si alloy with a diameter of up to 1 mm that could be considered to be a bulk metallic glass (BMG) [8]. Within several new bulk metallic glasses (BMGs) developed over the last decade, (Fe-Co)-Si-B-Nb glassy alloys play an important role because they combine a high glass-forming ability (GFA) with good soft-magnetic properties and very high compressive strength. As discussed in literature, the glass formation ability (GFA) of BMGs is best described by the temperature interval $\Delta T_x (= T_x - T_g)$ between the glass transition temperature (T_g) and the onset crystallization temperature (T_x). This parameter is also named as the super-cooled liquid region [34].

It was shown that the composition with the best combination of properties in this alloy family is $[(\text{Fe}_{0.5}\text{Co}_{0.5})_{0.75}\text{B}_{0.2}\text{Si}_{0.05}]_{96}\text{Nb}_4$, which has a good glass forming ability, soft magnetic properties, as well as an extremely high fracture strength of 3000–4000 MPa and ductile strain of 0.002 [35,36].

Based on their attractive properties, such as good soft magnetism [37,38] as well as mechanical behavior (high strength, high hardness) or excellent corrosion resistance and high wear resistance, many BMGs have potential applications in industry, medicine, energy systems, microelectronics, aeronautics and many other fields [39,40].

Metallic glasses (MGs) are quasi-brittle materials, because they do not possess sufficient intrinsic micromechanisms to mitigate high stress concentrations at crack tips. Contributing to this is an absence of strain hardening and a lack of intrinsic crack propagation barriers such as grain boundaries. In spite of these limitations, some MGs do exhibit toughness values comparable to crystalline structural alloys; on the other hand, others have very negligible ductility and macroscopic brittleness, with characteristics similar to oxide or silicate glasses [21].

High strength has been a long-standing objective pursued in metals and alloys. While the strength of a crystalline material is closely related to Pierce stress—the intrinsic frictional stress for dislocation motion—the fracture strength of BMGs is believed to be directly associated with the atomic bond due to the lack of defects [22,41]. Thus, the strength of a BMG is expected to be closely related to the physical parameters determined by atomic cohesive energy, such as glass transition temperatures (T_g), elastic modulus (E), and thermal expansion coefficients [22]. The relationship between strength and T_g is of particular interest since, T_g is also a key parameter governing the glass forming ability [41].

BMGs have strengths approaching the theoretical limit [42] but their plasticity at room temperature is typically very low. The high strength of BMGs is sometimes accompanied by plastic deformation and their deformation and fracture mechanisms are quite different from crystalline materials. It is well known that the strength of a crystalline material is associated with dislocation motion. Analogously, the strength of BMGs is likely to be associated with the shear band evolutions [41]. At temperatures below or around the glass transition (T_g) and rather high strain rates metallic glasses deform by the formation of localized shear bands [23] whereas homogeneous flow of the supercooled liquid is observed at elevated temperatures and low strain rates [23].

For the former case, it was previously considered that the compressive fracture usually proceeds along a shear plane inclined by 45° to the loading axis [23] i.e., the maximum shear stress plane. However, several recent systematic investigations on glasses in different alloy systems indicate that the shear fracture always deviates from the maximum shear stress plane either under compression or under tension [23].

Different from crystalline alloys, the shear plane deviates from the maximum resolved shear stress plane (45°) with the shear angle from 45 to 90° under tension and from 0 to 45° under compression [43]. This indicates that the failure of BMGs is not only controlled by the

deviatoric stress, but also by pressure or normal stress. For example, the difference in tensile and compressive yield strengths is due to the pressure sensitivity of deformation in BMGs [44].

The normal stress acting on a plane is of importance in propagating cleavage cracks. It is emphasized that such deviation from 45° is not symmetrical, as the tensile stress results in a more remarkable deviation (>5°) than that in compression case (<5°), implying the normal stress effect in tension is much more pronounced than that in compression.

In uniaxial tension, the plastic strain is almost zero [45]. For most of the known BMGs, plastic strain at room temperature is limited, <2%, even under compression, resulting from pronounced shear localization and work softening. Plastic deformation of metallic glasses at room temperature occurs through the formation and evolution of shear bands and is localized in thin shear bands [46]. Therefore thin shear bands are the inhomogeneous flow mode for BMGs.

The investigations on the fracture mechanism and fracture morphologies are very rare, which leads to a very weak understanding on some fundamental details about the fracture mechanisms of MGs. It should be noted here that attention in most work focuses mainly on the MGs with the shear fracture mechanism, which is the dominated fracture mechanism of most Zr-, Cu-, Ti-, Pd-, Pt-, Ni-based [47–49] and some other MGs, [50,51] not the MGs with a fragmentation mechanism including some Mg- [52,53], Co- [54] and Fe-based [55,56] MGs. The fracture behavior of Fe-based is significantly different in comparison with the well-studied Zr, Cu or Ti-based BMGs. The cooling rate is a critical factor to control the microstructure and mechanical properties of BMGs, which has a significance for the optimizing design of the materials.

Hence, a good understanding of fracture morphology and mechanical properties is important for designing performance of Fe, Co-based BMGs. The purpose of the paper was to investigate the effects of cooling rates on the structure, density, fracture morphology and micro-indentation behavior of the Fe, Co-based bulk metallic glass. In our previous work, the influence of cooling rate on the compression and fracture morphology was discussed [57].

2. Material and Methods

2.1. Material

The master alloy ingots with compositions of $\text{Fe}_{36}\text{Co}_{36}\text{B}_{19}\text{Si}_5\text{Nb}_4$ were prepared from pure Fe, Co, Nb metals and non-metallic elements: Si, B, in an argon atmosphere. The alloy composition represents nominal atomic percentages.

Rod-shaped samples were obtained by the pressure die casting the molten alloys into copper molds with diameters of $\phi = 2, 3$ and 4 mm in an argon atmosphere. Appearance of the rod samples with diameters of $\phi = 2, 3$ and 4 mm is shown in Fig. 1(a, b, c).

Lin and Johnson proposed a simple relationship between the sample dimension (R) and the cooling rate (dT/dt) as follows: $(dT/dt) = 10/R^2$ (cm) [58]. Thus, the achieved cooling rate in the rod-shaped samples with $\phi = 2, 3$ and 4 mm in diameter could be estimated to be 1000, ~444 and 250 K/s. Obviously the smaller the as-cast diameter, the larger the cooling rate achieved.

2.2. Experimental Procedure

The following experimental techniques were used: X-ray diffraction (XRD) phase analysis and TEM method to test the structure, differential scanning calorimetry (DSC) to test the thermal properties, microhardness measurements to test the mechanical properties. Fracture surfaces after decohesion process in compression and indentation morphologies after microhardness tests were observed by means of scanning electron microscopy (SEM). Density measurements were carried out by Archimedian principle for the samples weighing > 1.0 g and the accuracy was evaluated to be 0.005 g/cm³.

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