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# Mechanical behavior of bulk metallic glass prepared by copper mold casting with reversed pressure



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#### 1. Introduction

Bulk metallic glasses (BMGs) are generally prepared by fast solidification technologies to freeze the melt and avoid crystallization. Metallic glasses (MGs) have no typical crystalline microstructures and crystallographic defects such as dislocations and interfaces, which makes them exhibit outstanding properties. In general, the fracture strength of BMG is higher than the corresponding crystalline alloy but the plasticity is rather poor. From standpoint of fracture, Xu et al. (2010) claimed that cracks in BMGs were often hard to initial but relatively easy to propagate, so Schuh et al. (2007) deemed that the mechanical behavior of BMGs generally had "quasi-brittle" manner. As well known, brittle materials are inclined to fracture at a stress much lower than yield strength, when they contain typical cast defects such as inclusions, cast pores and surface flaws. During uploading, cast defects might produce local stress concentration in BMG samples, which make the initiation of cracks turn easier and cause the embrittlement of BMG. For example, Gao et al. (2011) found that the fracture strength of a Zr-based BMG was very sensitive to the size of cast pores. Therefore, cast defects should be regarded as a key role in BMGs to study further.

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#### ABSTRACT

The effect of applied reversed pressure on mechanical properties of bulk metallic glass (BMG) prepared by copper mold casting was investigated by quasi-static compression experiment and Weibull statistics. The BMG samples prepared by negative pressure casting exhibit higher strength and larger plasticity compared with those prepared by positive pressure casting. The applied reversed pressure plays a key role not only in the formation of cast defect at macro-scale but also for the distribution of free volume at micro-scale, which is responsible for the variable values of strength and plasticity, respectively. In copper mold casting process, negative pressure is also beneficial for reducing porosity content and homogeneous distribution of free volume, which in turn leads to the improvement of the reliability of BMGs.

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Cast defects were usually affected by the employed casting technologies or casting parameters. In traditional castings, the formation of cast defects has been proven to be associated with casting temperature (Campbell, 2006), casting atmosphere (Chen et al., 2008a) and electromagnetic treatment (Dong et al., 2015). As a fast solidification casting, copper mold casting (CMC) is often used for preparing small metallic castings. In the processes of CMC, cooling rate caused by copper mold is very fast so that the filling of melt is relatively difficult. Therefore, variable applied pressures are often employed to improve the mold-filling ability of CMC (so-called low-pressure casting). However, up to date, rarely literature has concerned the effect of applied pressure on the microstructure, cast flaw formation and mechanical properties of BMGs. In the present work, based on a typical Ti-based BMG with nominal composition of  $Ti_{41}Zr_{25}Be_{28}Fe_6$  (Gong et al., 2012), the mechanical properties of 2-mm-diameter rod samples prepared by reversed pressure will be comparatively studied.

#### 2. Experimental

High-purity (>99.9%) metal elements Ti, Zr, Be and Fe were used as starting materials and melted by arc-melting to prepare one master alloy ingot (about 20 g in weight) with nominal composition of  $Ti_{41}Zr_{25}Be_{28}Fe_6$ . This ingot was inverted and re-melted for at least five times to make homogenization of chemical composition. The obtained ingot was divided evenly into four smaller parts (each for

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Fig. 1. Schematic diagrams of cast methods. (a) Negative pressure casting and (b) positive pressure casting.



**Fig. 2.** XRD spectra of typical NPC sample and PPC sample. The inset shows an optical image of an as-cast rod with a diameter of 2 mm.

5 g). Two of them were re-melted by arc-melting and the melt drops were subsequently sucked into a copper mold by negative pressure (called negative pressure casting and denoted as NPC) as shown in Fig. 1(a). The other two small ingot parts were re-melted by induction melting and the melt drops were injected into the identical copper mold by argon gas with a positive pressure of ~0.2 MPa. This casting method was termed as positive pressure casting (denoted as PPC) as shown in Fig. 1(b). The temperature of the melt was under real-time monitoring by a double color laser infrared thermometer to control the casting temperature in both cast processes.

An X-ray diffractometer (XRD, Rigaku D/max2500) was used to confirm the amorphous structure of the as-cast rods. The glass nature of the rods were measured by differential scanning calorimeter (DSC, Netzsch STA 409) at a heating rate of 20 K/min. Compression samples, with a diameter of 2 mm and a length of about 4 mm, were cut from BMG rods by a slow diamond saw. The uniaxial compression tests were carried out on a WDW-100 testing machine at a strain rate of  $4 \times 10^{-4} \text{ s}^{-1}$  at room temperature. The fracture surface and side view of the deformed samples were examined by a scanning electron microscopy (SEM, LEO 1530).

#### 3. Results

#### 3.1. Identification of amorphous structure

Fig. 2 shows typical XRD spectra of  $Ti_{41}Zr_{25}Be_{28}Fe_6$  BMG rods prepared with reversed pressure. Each sample for XRD test was cut from the middle part of as-cast rods with a length of  $\sim$ 70 mm (A typ-

ical rod is shown in the inset of Fig. 2). It has been found that both samples exhibit typical amorphous structure in which the spectrum possesses no obvious sharp fraction peak and only contained a diffuse scattering peak in the 20 range of  $30^{\circ}-45^{\circ}$ . It has been reported in an earlier work (Gong et al., 2015) that  $Ti_{41}Zr_{25}Be_{28}Fe_6$  BMG has a critical cast thickness with full amorphous structure of over 10 mm, thus the 2-mm-diameter glassy rod can be easily made neither by NPC or PPC. In addition, Fig. 2 also shows that the amorphous structural feature of the BMG sample cast with reversed pressure has no obvious difference based on XRD analysis.

Fig. 3 displays the DSC curves of the samples that were cut from the upper, middle and bottom part (see the inset in Fig. 2) of a NPC rod and a PPC rod, respectively. It is very clearly that these DSC curves are very similar ones which possess obvious glass transition feature and crystallization feature. The thermal physical parameters including the onset glass transition temperature  $T_g^{\text{onset}}$ , the onset crystallization temperature  $T_x$ , the crystallization peak temperature  $T_{p1}$  and  $T_{p2}$ , the supercooled liquid region  $\Delta T$  and the structural relaxation enthalpy  $\Delta H_0$  are all listed in Table 1. Except  $\Delta H_0$ , the other thermal parameters are highly consistent, indicating the similar glassy nature of the samples.

Free volume was deduced to be some point defect in BMGs (Spaepen, 1977) and strongly affected the mechanical behavior of BMGs (Chen, 2008). Structural relaxation enthalpy  $\Delta H_0$  was often regarded as the indicator of free volume, which has been demonstrated by Slipenyuk and Eckert (2004). Fig. 3 also shows the structural relaxation enthalpy by the slashed area on each curve and the calculated  $\Delta H_0$  values are listed in Table 1. The PPC samples exhibit relatively scatter  $\Delta H_0$  data than that of NPC samples resulting in a mean value of  $7.2 \pm 1.4 \text{ Jg}^{-1}$ . It should be noted that the standard deviation value  $1.4 \text{ Jg}^{-1}$  is about ~20% of the total  $\Delta H_0$  value, which implies that the distribution of free volume in PPC samples is heterogeneous. The mean  $\Delta H_0$  value of NPC samples is  $8.2 \pm 0.3 \text{ Jg}^{-1}$  and the deviation is only ~4% of the total value. Therefore, we may concluded that the free volume distribution in NPC rod is relatively more homogenous than that of PPC samples.

#### 3.2. Mechanical properties

Fig. 4 displays the engineering stress-strain curves of BMG samples prepared with reversed pressure, respectively. To better understand the repeatability and reproducibility of data, at least 20 samples for each group were tested. It is found that the plastic strain  $\varepsilon_p$  of NPC samples is in a range of 0.35–9.3% with a mean value of  $3.5 \pm 2.8\%$  and the  $\varepsilon_p$  value of PPC samples is 0.3-4.5%, about  $2.2 \pm 0.7\%$ . Thus it apparently shows that the plasticity of NPC sample is better than that of PPC sample. It is worthy noticing that, for NPC samples, the error is comparable to the mean value, which reflects a discrete distribution of data. It is implied that the large

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