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A Damage-tolerant Bulk Metallic Glass at Liquid-nitrogen

Temperature

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Tensile tests and notch toughness tests were conducted on $Zr_{61}Ti_2Cu_{25}Al_{12}$ glass (ZT) at room temperature and liquid-nitrogen temperature. The tensile strength of ZT was improved from 1.63 GPa at room temperature to 1.72 GPa at liquid-nitrogen temperature. Micro-notches with a root radius of 1–3 µm were introduced to test the notch toughness of ZT at room temperature and liquid-nitrogen temperature. The test results revealed that the notch toughness of ZT at liquid-nitrogen temperature is comparable to that of ZT at room temperature. The combination of high yield strength and notch toughness of ZT at liquid-nitrogen temperature is comparable to that of the best cryogenic engineering materials.

KEY WORDS: Metallic glasses; Yield strength; Fracture toughness; Liquid-nitrogen temperature

1. Introduction

Lightweight and high-performance cryogenic engineering structures require materials with a combination of high strength and high fracture toughness^[1]. The reduced thermal activation at cryogenic temperatures typically produces increased strength^[2]. However, a ductile-to-brittle transition on cooling often occurs in widely used structural materials such as body-centered cubic carbon steels and hexagonal close packed Ti alloys^[1,3]. This reveals an inherent difficulty in combining both high strength and high toughness for cryogenic engineering applications. Although a variety of materials exist with strength and toughness that are weakly temperature-dependent (e.g. Ni steels with fine grain size, face-centered cubic stainless steels, Ni-based superalloys, Al alloys and Cu alloys) and are used in cryogenic engineering structures^[1], a long-standing goal remains the combination of high strength and high fracture toughness at cryogenic temperatures. Since both the yield strength and ductility of nanostructured metals and alloys can be significantly improved at cryogenic temperatures compared with room temperature^[4,5], an inverse temperature-dependent toughness results^[3]. However, some BMGs such as Ti₄₀Zr₂₅Cu₁₂Ni₃Be₂₀ $glass^{[6]}$, $Pd_{79}Ag_{3,5}P_6Si_{9,5}Ge_2$ $glass^{[7]}$, and $Zr_{61}Ti_2Cu_{25}Al_{12}$

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glass^[8] (ZT) are much more damage-tolerant than nanostructured metals and alloys at room temperature and their combination of high yield strength and fracture toughness extend well beyond the traditional limit ranges established by the strongest and the toughest materials. Furthermore, both strengthening and ductilization of bulk metallic glasses (BMGs) at cryogenic temperatures have also been reported^[2,9–14] due to retarded shear banding. Other work has shown reductions in shear-band viscosity, shear-slip velocity, and the magnitude of stress drop in serrated flow, with decreasing temperature^[12,13]. These observations imply that some BMGs might combine sufficiently high yield strength and high fracture toughness at cryogenic temperatures.

Previous work has shown that $ZT^{[8,15]}$ exhibits a combination of high yield strength and high fracture toughness at room temperature, comparable to the best structural materials known^[8]. The present work further shows that the strength of ZT increases slightly at liquid-nitrogen temperature (77 K) with notch toughness that is comparable to that obtained at room temperature. These results reveal an excellent combination of toughness and strength of ZT at 77 K.

2. Experimental

 $Zr_{61}Ti_2Cu_{25}Al_{12}$ ^[15] master alloy ingots were prepared by arc melting pure elemental pieces under Ti-gettered argon atmosphere in a water-cooled copper hearth. The ingots were flipped and re-melted 4 times to ensure chemical homogeneity. $Zr_{61}Ti_2Cu_{25}Al_{12}$ glassy rods with a diameter of 5 mm and plates with dimensions of 3.2 mm \times 8 mm \times 62 mm were fabricated



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by suction casting method using water-cooled copper mold. The fully amorphous nature of the ZT samples was confirmed by the diffuse X-ray diffraction pattern (Scintag X-1 X-ray diffractometer) and DSC traces (Perkin-Elmer DSC-7) as shown in Fig. 1. Tensile testing was conducted on samples with gage dimensions of Φ 1.5 mm imes 10 mm that were machined from ascast rods and polished with SiC papers. Bend bars for toughness testing with dimensions of 3 mm \times 6 mm \times 26 mm were cut from the bottom part of as-cast plates with a low-speed diamond blade and polished with SiC papers. Single edge notches with a length of 2.8 mm were introduced using a diamond wire saw with a radius of 100 µm. Micro-notches, with a root radius of $1-3 \mu m$ were obtained by repeatedly sliding a diamond-coated razor blade over the saw-cut notch with lubricant. The final depth of the notch is 3 mm. Toughness tests and tensile tests were conducted at room temperature and liquidnitrogen temperature on an Instron 1361 electromechanical machine at a displacement rate of 0.3 mm min^{-1} and an initial strain rate of 5 \times 10⁻⁴ s⁻¹ respectively. The cryogenic temperature tests were conducted by immersing the samples into liquid nitrogen.

3. Results and Discussion

Fig. 2(a) shows the tensile engineering stress-crosshead displacement curves for ZT at 298 K and 77 K, respectively. It is proposed^[16] that more elastic energy is needed to activate shear-transformation zones at cryogenic temperatures than at room temperature. In the present work, the yield strength increased from 1.63 GPa at 298 K to 1.72 GPa at 77 K (as listed in Table 1). Compared to the reported 18% strength improvement for $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{59}Cu_{31}Al_{10}$ BMGs^[10], the 5.5% increase for ZT is low. However, the improvement of strength of ZT is larger than that of Cu₅₇Zr₄₃ BMG which exhibited a strength increase of only 1.5%. In contrast to the compressive ductility improvements reported for other BMGs at cryogenic temperatures^[9,12,13]</sup>, there is no obvious tensile ductility of ZT at either room temperature or 77 K, similar to that found for other BMGs in the literature [10,17,18]. In the uniaxial tensile stress state, there is no frictional force that is available in compression. The tensile stress state enhances strain softening and the instability of shear bands, producing a shear band that slips without limit. Even though it has been reported that both the viscosity and velocity of shear banding are reduced at cryogenic



Fig. 1 Fully amorphous nature of the present samples. (a) DSC trace showing glass transition and crystallization of ZT. (b) Diffuse Xray diffraction pattern of ZT without sharp crystalline Bragg peaks.



Fig. 2 Tensile tests and toughness tests of ZT at 298 K and 77 K. (a) Tensile stress-crosshead displacement curves of ZT at 298 K and 77 K. (b) Evolution of stress intensity factor with crosshead displacement for micro-notched samples. Catastrophic fracture occurred at the maximum K.

temperatures^[12,14], the tensile ductility of BMGs at cryogenic temperatures has not yet been be remarkably improved.

As shown in Fig. 3(a), a micro-notch with root radius of 1-3 µm was introduced by using a diamond-coated razor blade to investigate the notch toughness of ZT at 77 K. The loading traces of the toughness tests for the micro-notched samples tested at 298 K and 77 K are shown in Fig. 2(b). As listed in Table 1, the toughness of the micro-notched sample is 107.7 MPa m^{1/2} at 298 K. As reported elsewhere, the room temperature toughness of fatigue pre-cracked samples^[15] is 112 MPa m^{1/2}. We have obtained a room temperature fatigue pre-cracked toughness of 114.8 MPa $m^{1/2}$ for this material. As demonstrated presently, the micro-notch toughness of ZT at room temperature is very similar to the toughness values obtained at room temperature on fatigue pre-cracked samples. This type of behavior has been reported previously on very tough metallic glass samples^[6,15] where the proliferation of shear bands at the fatigue pre-crack essentially blunts the fatigue pre-crack. This has also been demonstrated in other work on $ZT^{[15]}$ and is consistent with that found in the present work. However, BMGs with lower toughness have shown quite different behavior regarding the effects of changes

Table 1 Measured yield strength (σ_y), fracture toughness of micronotched sample (K_c) and calculated plane stress plastic zone size (r_p) of ZT at room temperature (298 K) and liquidnitrogen temperature (77 K)

Temperature (K)	$\sigma_{\rm y}~({\rm GPa})$	$K_{\rm c} ({\rm MPa}{ m m}^{1/2})$	<i>r</i> _p (μm)
77	1.72	105.8	602
298	1.63	107.7	695

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