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Ductile-to-brittle transition in a Ti-based bulk metallic glass

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The effects of changes in cooling rate on the density, elastic constants and fracture energy have been determined for Ti40Zr25-Cu12Ni3Be20 bulk metallic glass. It is shown that changes in cooling rate directly affect the measured density and elastic constants, while the fracture energy, ranging from 148.9 to only 0.2 kJ m^{-2}, is also shown to correlate with these changes in cooling rate/density/elastic constants. The critical Poisson's ratio for toughness in this alloy system is 0.35, somewhat higher than that obtained in other metallic glasses, although consistent with recent theoretical predictions. Further correlations with these changes in toughness on the fracture surface appearance are provided.
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The unique combination of properties of bulk metallic glasses (BMGs)—i.e. strength, elastic limit, hardness, corrosion and wear resistance—make them potentially useful for a variety of structural applications. However, exploitation of BMGs is hindered by their lack of plasticity/toughness. Recent work has been investigating various intrinsic and extrinsic ways to improve the plasticity/toughness of BMGs via alloy modification, use of a composite structure and change of processing conditions [1–11]. Recently, fracture energy correlations with the ratio μ/B (or alternatively, ν) for a range of BMG alloys has been demonstrated as well as for annealing-induced embrittlement [12]. Independent work has similarly shown a correlation between compressive plasticity and Poisson's ratio [2,13]. Recent experimental observations have also provided a correlation between the fracture toughness and the length scale of the "dimple"/"vein pattern" for various brittle and tough BMGs [14]. However, the generality of such observations to other BMG systems has not been widely demonstrated. Recent discoveries of ductile Ti-based glassy alloy ingots [15] have enabled quantification of the effects of cooling history on microstructure, elastic constants and their effects on deformation and fracture behavior. The present paper on a range of Ti–BMGs illustrates that tuning the elastic constants by cooling rate can control the deformation and fracture toughness behavior of Ti–BMGs.

Commercial-grade elements (with purities of 99.5% or higher) were used to produce the alloy ingots. Four alloy ingots with a nominal composition Ti40Zr25Cu12-Ni3Be20 were made in an arc furnace under a flowing argon atmosphere with an ingot weight of 10 g (labeled as AM1, AM2, AM3, AM4). Rods (3 mm diameter) and rectangular bars ($5 \times 5 \times 65$ mm^3) were prepared by copper-mold suction casting under an argon atmosphere (labeled as AC1 and AC2, respectively). The amorphous nature and the phase structure of as-made ingots and as-cast rods/bars were confirmed by X-ray diffraction (XRD) using Cu K_α radiation. The pulse–echo overlap technique with 10 MHz piezoelectric transducers was used to measure the shear and longitudinal wave speeds, enabling calculation of the elastic constants. Density measurements were carried out by Archimedes' principle and the accuracy was evaluated to be 0.004 g cm^{-3}. Samples with a nominal dimension of $4 \times 6 \times 24$ mm^3 for notch toughness testing were machined directly from these as-made ingots, while the as-cast rods and bars were directly used for testing. Three-point bend notch toughness experiments were conducted at room temperature using a MTS model 810 servohydraulic testing rig under displacement control with a rate of 0.1 mm min^{-1}. The cylindrical rods and bars were notched using a slow-speed diamond wire

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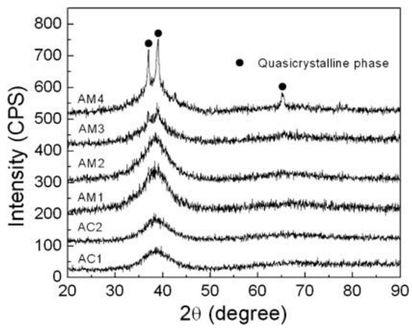

Figure 1. XRD patterns of four as-made 10 g ingots (AM1, AM2, AM3 and AM4) and as-cast (AC1: 3 mm diameter rod, AC2: rectangular bar $5 \times 5 \times 65$ mm^3) Ti40Zr25Cu12Ni3Be20 BMG subjected to different cooling rates.

saw with a root radius of 110 μm to a depth of approximately half the diameter/width, and notch toughness tests were performed. Fatigue precracked tests were not conducted due to limitations in sample dimensions, although this is known to affect the magnitude of toughness [9,16]. Scanning electron microscopy (SEM) was used to examine the fracture surfaces. A Buehler Micromet hardness tester with a diamond Vickers indenter was used to determine the microhardness using a load of 500 g and a loading time of 10 s. Each microhardness value was the average of 10 individual measurements.

Figure 1 shows the XRD patterns of the four as-made 10 g ingots, as-cast 3 mm diameter rod and as-cast rectangular bar. The as-cast samples (AC1 and AC2) and as-made ingots (AM1 and AM2) show broad diffraction peaks consistent with the lack of long-range order that characterizes amorphous systems. However, for the as-made AM3 and AM4 ingots, although the amorphous diffuse diffraction background is still dominant, distinct crystallization peaks appear, indicating the formation of a quasicrystalline phase [15]. As shown in Table 1, the density substantially increases from 5.384 to 5.475 g cm^{-3} for the samples in the order AC1 to AM4 as shown in Figure 1. The density depends on the rate at which they were cooled from the melt. The higher the cooling rate, the lower the density. Therefore, the cooling rate at which the samples were cooled from the melt decreases in the order AC1, AC2, AM1, AM2, AM3, AM4. A density increase with decreasing cooling rate was also reported in Pd40Ni10Cu30P20 BMG [17]. The microhardness, shown in Table 1, also increases from 504 to 592 HV with decreasing cooling rate. The increases in microhardness and density are consistent with the denser atomic configuration and evolution of quasicrystalline phase that occur with lower cooling rates due to the increased time for the supercooled liquid to relax during solidification.

The elastic properties were measured ultrasonically. The elastic moduli are listed in Table 1. It is found that the shear modulus μ increases from 34.3 for AC1 to 40.1 GPa for AM4, consistent with the structural relaxation and precipitation of quasicrystalline phase. The changes in bulk modulus B are significantly smaller than the changes in shear modulus, providing a decrease in Poisson's ratio. The Young's modulus E changes dramatically from 93.3 for AC1 to 107.4 GPa for AM4.

The notched toughness (K_c) values were calculated using the maximum load at fracture, summarized in Table 1. The load–displacement traces for samples AM1, AM2, AM3 and AM4 were linear to fracture, while for samples AC1 and AC2, nonlinear traces to fracture were observed. Included in Table 1 is the fracture energy, G_C, calculated as $G_C = K_c^2(1 - \nu^2)/E$. The plane strain plastic zone radius r_p at failure is also included in Table 1; this was estimated using $r_p \approx K_c^2/6\pi\sigma_0^2$, where σ_0 can be calculated using $\sigma_0 = HV/3$. r_p for most of the samples studied is very small compared to the specimen diameter or thickness, indicating that nominally plane strain conditions can be assumed. However, the high toughness samples (e.g. AC1 and AC2) produce a mixture of plane stress and plane strain due to the high toughness. Figure 2 provides the correlation between fracture energy and the ratio of shear modulus to bulk modulus (μ/B) as well as Poisson's ratio (ν). A strong correlation between fracture energy and ν (or μ/B) is observed. The fracture energy increases quite rapidly with increasing ν (or decreasing μ/B) with a distinct transition from tough to brittle behavior. The 3 mm diameter rod exhibits high fracture energy, reaching 148.9 kJ m^{-2}, which is among the largest reported to date for BMGs, although this sample is not in plane strain condition. The Ti–BMG composite with quasicrystalline precipitation exhibits very low toughness, approaching the ideally brittle behavior associated with oxide glasses. The variation of elastic constants in Ti40Zr25Cu12Ni3Be20 BMG enabled the identification of a well-defined transition from plasticity to brittleness, confirming the existence of a critical Poisson's ratio of 0.35, above which plastic deformation occurs, or a critical μ/B ratio of 0.33, below which plastic deformation occurs. The critical Poisson's ratio for plasticity in Ti40Zr25Cu12Ni3Be20 BMG is somewhat higher than the critical Poisson's ratio 0.32 in Fe-based BMGs and Mg-based MGs and the 0.31–0.32 inferred from a range of BMG compositions as well as annealing-induced embrittlement [12,13,18]. Combined with previous reports of the lack of plasticity in some Zr- [19,20] and

Table 1. The measured density (ρ), microhardness, elastic moduli, notch fracture toughness (K_c) and calculated notch fracture energy (G_C) and calculated plane strain plastic zone radius (r_p) for Ti40Zr25Cu12Ni3Be20 BMG.

Alloy	ρ (g cm^{-3})	HV	μ (GPa)	B (GPa)	E (GPa)	ν	K_c (MPa $m^{1/2}$)	G_C (kJ m^{-2})	r_p (μm)
AC1	5.384	504	34.3	109.9	93.3	0.358	126.3	148.9	312
AC2	5.391	505	34.7	108.4	94.1	0.355	102.2	96.9	203
AM1	5.421	513	36.0	109.6	97.3	0.352	39.7	14.2	30
AM2	5.446	522	36.9	110.0	99.6	0.349	13.5	1.6	3.3
AM3	5.467	534	37.4	109.9	100.7	0.347	8.2	0.6	1.1
AM4	5.475	592	40.1	111.1	107.4	0.339	5.4	0.2	0.3

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