Global melting of $Zr_{57}Ti_5Ni_8Cu_{20}Al_{10}$ bulk metallic glass under microcompression

Sheng Cheng[a)]
Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee 37996, USA

Xun-Li Wang
Neutron Science Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 39831, USA

Hahn Choo
Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee 37996, USA and Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA

Peter K. Liaw
Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee 37996, USA

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Global melting was observed in micron-sized pillar samples tested under microcompression. In addition to a higher strength that was reproduced in samples with different sizes, a large plastic strain was also observed prior to the final fracture, as compared to bulk counterparts. The global melting was a result of the final fracture as evidenced by the acoustic emission at the end of the microcompression. However, the increased strain rate and large plastic strain might have contributed to the temperature increase prior to the fracture. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2813623]

Bulk metallic glasses (BMGs) possess several outstanding properties that make them desirable structural materials for microelctromechanical system (MEMS) devices.[1] First, these amorphous alloys contain no defects that cause yielding in crystalline materials, such as dislocations, and thus have high strength and hardness desirable for the high-stress and wear-prone conditions often encountered by the small MEMS parts. Second, the BMGs offer a large elastic strain before yielding, low mechanical damping, and high resilience, properties desirable for many MEMS applications.[2,3] Third, in the absence of grain boundaries and having uniform microstructure at compositions where multiphase mixtures would prevail in the crystalline state, the BMGs can be superior to conventional alloys in terms of corrosion resistance.[4] Combining all these traits, BMGs are obviously attractive candidates as MEMS materials. In terms of plasticity, bending tests of metallic glass wires and thin plates usually indicate a considerable bending ductility,[5] even though monolithic BMGs usually fail within ~1% of plastic strain in conventional compression test due to the localization of plastic deformation in ultrathin shear bands.[6] Micrometer-diameter wires can be tested in tension, but the BMGs exhibit little or no macroscopic tensile ductility such that it is difficult to determine yield stress and intrinsic plasticity.[7] A quantitative compression test on the micron scale would therefore be of scientific significance.

However, so far, there have been only limited quantitative measurements in this respect. A recent study on a Pd-BMG investigated strength and the shear banding behavior of micron-sized samples with diameter sizes from 2 to 20 μm under microcompression tests.[8] The study found little size effect on the strength but suggested that the strength was still higher than that in bulk samples. Moreover, multiple shear bands were found operating in the samples and the plastic strain was in the form of shear band burst, but no melting was observed in the samples. Similar observation was also made in Mg-BMG microcompression tests.[9] In common is that the microcompression tests in these studies were performed under a quasistatic prescribed strain rate, i.e., 10^{-4} s^{-1}. How the micron-sized samples behave under a higher strain rate has not been explored. This paper presents the microcompression tests of a Zr-based metallic glass, $Zr_{57}Ti_5Ni_8Cu_{20}Al_{10}$, under a prescribed strain rate of 10^{-3} s^{-1}. The behavior is discussed and compared to that of bulk samples.

Cylindrical micropillars were prepared using dual focused ion beams (FIB) machining with a method developed by Uchic *et al.*[10] Briefly, in the first step, rough cutting with a high ion beam current was conducted to dig the outer trench and outline a column. Fine machining to reach the final diameter was performed in a progressive manner with gradually reduced beam currents. The current density used for the final trim is ~0.1 nA to reduce the possible changes caused by the Ga ions to the surface. To minimize the taper formed in micropillars due to the beam convergence in the first step, a second step of 360° FIB trimming was applied to "lathe" off the taper, with the sample column tilted by 2°–5°. The aspect ratio (height/diameter) of the finished microposts is ~2, following the recommendation provided by finite element modeling.[11]

Microcompression tests were performed using MTS Nanoindenter XP systems in the continuous stiffness mode and a truncated cone-shaped diamond indenter that applies the compressive load. In order to check the alignment, a shallow indentation (up to 1 μm deep, depending on the

[a)]Author to whom correspondence should be addressed. Tel.: (865) 974-2683. FAX: (865) 974-4115. Electronic mail: scheng1@utk.edu

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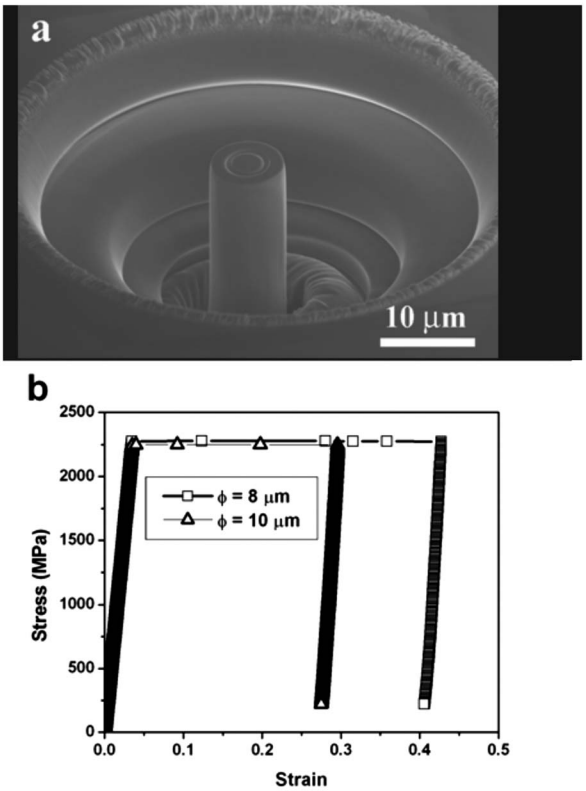

FIG. 1. (a) A Zr-BMG micropillar with a diameter $\phi=8$ μm, trimmed using the two-step FIB machining. (b) The compressive stress-strain curves of the Zr-BMG micropillars under microcompressions.

sample hardness) was produced on the smooth surface away from the pillar sample. When a homogeneous indentation ring was observed, the system was considered well aligned. The load as a function of displacement was recorded at a prescribed displacement rate of 20 nm/s, corresponding to a strain rate of $\sim 10^{-3}$ s^{-1}. The data are then converted to engineering stress and engineering strain.

Figure 1(a) shows a representative as-cut pillar sample with a uniform diameter of $\phi=8$ μm. The aspect ratio is of 2.25 (diameter is 8 μm and the height is ~ 18 μm). The ring on the top surface [Fig. 1(a)] was made to serve as a reference for the second step lathe cutting. At this sample size level, the possible mechanical response change due to FIB damage was found insignificant according to a recent study.[8] Figure 1(b) shows the compressive stress-strain curves of micropillars with two diameters, i.e., $\phi=8$ μm and $\phi=10$ μm, respectively. The yield stress is very close for both samples, ~ 2.25 GPa, as compared to ~ 1.9 GPa of macroscopic samples made by the same materials.[6] This reproducible result suggests that the micropillar might indeed be stronger than macroscopic samples. The increased yield strength was also reported in the recent study of microcompression tests in Pd-based glass and Mg-based glass.[8,9] For the flat portion after yielding in Fig. 1(b), the load did not increase after the shear banding commenced, such that the compression punch moved at a high rate, but the data acquisition was at fixed rate. It resulted in relatively few data points. Apparently, the BMG did not exhibit intrinsic work hardening capability, as expected for a monolithic metallic glass. In fact, Fig. 1(b) suggests softening in the shear band,

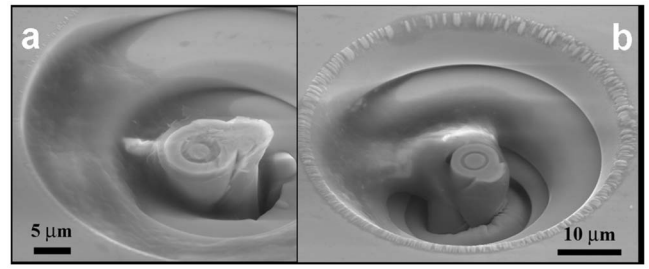

FIG. 2. SEM micrographs of the pillar samples with diameters of (a) $\phi=10$ μm and (b) $\phi=8$ μm after microcompression.

considering the effective cross-sectional area may be increasing during compression.[12]

The as-compressed samples were displayed in Fig. 2. One can see that the top surface and reference rings were well preserved, and the micropillars were pushed straightly down, indicating a good alignment. The apparent compressive plastic strain for sample a ($\phi=10$ μm) is $\sim 30\%$, while it is $\sim 40\%$ for the sample b ($\phi=8$ μm). The increased plastic strain is believed to be a result of the reduced sample size, whose effect is twofold: (1) In a conventional compression test, the shear offset of a shear band is on the order of microns prior to failure, which would represent a large plastic strain for a microsized sample, e.g., a 2.5 μm shear offset would be $\sim 16\%$ plastic strain for a 16 μm long sample (assuming a 45° shearing). In contrast, the same shear would only mean a 0.06% plastic strain for a 4 mm long bulk sample. (2) For Zr-based metallic glasses, the process zone size, d, determined by the squared ratio of the fracture toughness to the yield strength, is in the range of 0.1–1 mm.[1] This is the size of the plastic zone present at the tip of a sharp crack. In our case, d is much larger than the sample size, so brittle failure due to catastrophic crack propagation is expected to be discouraged, and thus the shear to continue to a large plastic strain. The large plastic strains observed in our tests testified the above arguments. The different plastic strains in the two samples might be related to the shear banding operation. Sample b in Fig. 2 apparently experienced a second primary shear, which would yield a higher plastic strain than sample a, which shows only one primary shear.

In addition to the large plastic strain, surprisingly, apparent melting was observed in the samples after the microcompression. It appears as if the melt was squeezed out at a high

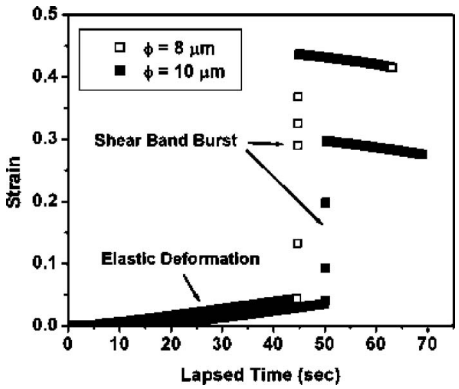

FIG. 3. The strain development as a function of loading time in microcompressions. The prescribed strain rates in the elastic deformation portion were approximately equivalent to 1×10^{-3} s^{-1}, but the average shear band bursting rates were ~ 1.3 s^{-1} for both samples.

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TABLE I. The parameters used in Eq. (2).

τ (MPa)	ΔL (μm)	ρ (kg/m^3)	C (J/kg K)	c_t (m/s)	κ (m^2/s)
1100	0.45	6.81×10^3	420	2470	3×10^{-6}

rate and was sprayed to the sidewall. In fact, the sample fracture followed the shear banding operation at the end of the microcompression. Even in these micron-sized samples, acoustic emission was clearly heard upon the sample fracture. Melt droplets were often observed in the fracture surface of BMG samples,[13–15] but massive melting as shown here was rarely observed in conventional compression tests of bulk samples. The massive melting in current microcompression suggests that the temperature rise in the sample volume was very significant. Wright *et al.* established a model based on the adiabatic heating in a single shear band due to the work done on the sample during plastic deformation,[15]

$$\Delta T = \frac{\tau \dot{u}_{\text{plastic}}}{K}\sqrt{\frac{\kappa \Delta t}{\pi}}, \tag{1}$$

where τ is the shear stress, $\dot{u}_{\text{plastic}}$ is the plastic displacement rate in the plane of the shear band, K is the thermal conductivity, κ is the thermal diffusivity, and Δt is the time elapsed during the unloading segment of a single serration.

They predicted that the temperature rise during a single serration is only on the order of a few degree Kelvin. However, if the same model was applied to the final failure event, where $\dot{u}_{\text{plastic}}$ is much larger, the temperature increase predicted for in a Pd-BMG sample could be sufficient to reach the melting temperature.[15] In our samples, the shear banding operation and the final fracture were essentially consequent events, and the shear banding burst rate was the plastic strain rate in the shear band. Figure 3 shows the strain development as a function of the lapsed time. In the elastic part, the loading was at the prescribed rate, i.e., $\sim10^{-3}$ s^{-1}, for both samples. However, after the samples began yielding (or the initiation of shear banding), despite the prescribed strain rate, the average shear banding burst was at a rate of 1.3 s^{-1}, which is three orders of magnitude higher than the prescribed rate. To give an estimate on the temperature rise, we can further derive Eq. (1) by replacing $\dot{u}_{\text{plastic}}$ with $\dot{u}_{\text{plastic}} = \Delta L/\Delta t$, where ΔL is the shear displacement along the shear band plane, and (with $K=\kappa\rho C$, where ρ is the sample density and C is the specific heat. Then Eq. (1) becomes

$$\Delta T = \frac{1}{\sqrt{\pi}}\frac{\tau \Delta L}{\rho C}\sqrt{\frac{1}{\kappa \Delta t}}. \tag{2}$$

If we take $\Delta t=0.2$ ns in the final failure event, $\Delta L = 0.45$ μm, as $\Delta L = 0.9 c_t \Delta t$, where c_t is the speed of a transverse sound wave ($c_t \approx 2.47\times10^3$ m/s). The temperature rise is estimated as 3940 K under an adiabatic condition (see Table I).[16]

In addition to plastic displacement rate $\dot{u}_{\text{plastic}}$ that could affect the temperature rise, the plastic strain was also reported to influence the temperature rise. In the recent investigations on a Zr-based BMG, by using high-speed thermographic-infrared camera, Jiang *et al.* investigated the impact of strain rate and strain level on the shear band temperature rise.[17] Their analysis suggested that both strain rate and shear strain could contribute to the temperature rise. The large plastic strain in our case might have generated heat prior to the final fracture, which could have contributed to the temperature rise. Considering the small volume of our samples, one should not be surprised that the generated heat at the point of fracture could drive a temperature rise above the melting point. By contrast, for the slow prescribed strain rate used in previous studies,[8,9] the actual strain rate during the shear band burst was not as high as in the current study, their plastic strain as result of a burst was also relatively small, and most importantly the tests were interrupted prior to the failure event. All these might help understand why no apparent melting was observed in their samples.

In summary, we have performed microcompression tests on a Zr-based BMG at a higher rate than previous investigations. The yield strength observed is higher than that found in conventional compression test of the same glass, consistent with the recently reported microcompression results. Global sample melting indicates that the temperature rise upon fracture was significant under a high strain rate and a large plastic strain.

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