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Bulk and microscale compressive behavior of a Zr-based metallic glass

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Micropillars with diameters of 3.8, 1 and 0.7 μm were fabricated from a two-phase Zr-based metallic glass using focus ion beam (FIB), and then tested in compression at strain rates from 1×10^{-4} to 1×10^{-2} s^{-1}. The apparent yield strength of the micropillars ranges from 1992 to 2972 MPa, or 25–86% increase over that of the bulk specimens. This strength increase can be rationalized by the Weibull statistics for brittle materials.

Keywords: Metallic glasses; Micropillars; Compression test; Shear bands

Over the past decades, bulk metallic glasses (BMGs) have attracted extensive interest because of their unique properties such as good corrosion resistance, a large elastic limit, as well as high strength and hardness. However, the high strength of BMGs is often accompanied by a virtually zero plastic strain in tension and limited plasticity in compression. The brittleness problem severely impedes further exploitation of this material. To overcome this problem, many researchers made attempts to develop an extrinsic composite microstructure within the glassy matrix [1–4], or an intrinsic structure, such as dual-phase BMGs or in situ precipitated nanocrystals [5–8]. The basic idea is effectively to block or hinder the propagation of shear bands during deformation. For example, we have developed a phase-separated ductile Zr–Ni–Cu–Al BMG, exhibiting high compression ductility more than 30% [8].

Recently, metallic glasses are seen as the potential material for imprinting, molding and microelectromechanical systems (MEMs) because of their high strength, hardness, and processing flexibility in the supercooled liquid region. Thus, intense efforts have been made to study the properties of small-sized samples. For example, it has been found that the strength of the face-centered cubic (fcc) single crystals such as Ni and Au [9,10] are a strong function of the specimen size in the micrometer range. This dramatic effect was proposed to be a result of the reduced specimen size which is smaller than the characteristic length for dislocation multiplication, resulting in dislocation starvation. In contrast, the BMG pillars do not deform by dislocation-mitigated processes [11,12]; instead, the plastic deformation in BMGs at room temperature is highly localized within shear bands or shear transformation zones (STZs) [13–15]. Our previous study on the brittle Mg–Cu–Gd-based glasses (no plastic compression strain in the bulk specimens) in the form of micropillars measuring 3.8 and 1 μm in showed a sudden strain burst, manifested as a constant flow stress, and no work-hardening [16]. Every strain burst event, regardless of the strain rate, proceeds within about one second, suggesting the strain rate during these bursts was at least 10^{-1} s^{-1}. There were very few shear bands, especially in the 1 μm pillar sample at a low strain rate: only one single shear band was present. It is thus of interest to examine the shear banding behavior of a much more ductile phase-separated Zr-based glass micropillars.

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A ductile two-phase $Zr_{63.8}Ni_{16.2}Cu_{15}Al_5$ (in at.%) BMG was used in the current investigation due to its remarkable mechanical properties in the bulk form [8]. The cylindrical micropillars with a diameter of 3.8, 1.0, and 0.7 μm are fabricated using the FIB milling. The Zr-based BMGs were prepared by injection–casting into a water-cooled Cu mold with an internal cylindrical-shaped cavity of 2 mm in diameter. Detailed description of the casting methods have been described elsewhere [8]. The 2 mm BMG rods were sliced into disks of 1.5 mm in height by diamond cut, and then were ground with SiC paper from #1200 to #1400. Finally, the disk surface was polished to mirror finish with a diamond polishing paste, from 1 μm grit to 0.25 μm, prior to the FIB machining.

The microcompression samples were prepared using the dual focus ion beam system (FIB) of Seiko, SMI3050 SE, following the method developed by Uchic and Dimiduk [17]. A Ga beam operated at 30 keV and 7–12 nA was initially directed perpendicular to the surface of the BMG disk to mill a crater with a much bigger size (around five times of the corresponding pillar) island located in the center. Then, the same voltage and smaller currents of 0.7–0.09 nA were used to refine the preserved island in the center to a desired diameter and height of the pillar. A series of concentric-circle patterns were utilized to machine the pillars. The diameter, *d*, of a pillar, e.g., the 3.8, 1 and 0.7 μm in this paper, is referred to the diameter at the half-height position. However, due to slight tapered shape and the first initiation of shear banding at the top surface [16], all engineering flow stresses were calculated using the diameter of the top surface.

Microcompression tests were performed in an MTS nanoindenter XP with the continuous stiffness measurement mode using a flat punch indenter with an equilateral triangle cross-section measuring 13.5 μm in side length, which was also machined by FIB. These specimens were deformed in a prescribed displacement. The corresponding strain rates vary from 1×10^{-4} to $1 \times 10^{-2}\ s^{-1}$. The data reported below are all engineering stresses and strains.

The micro-compression pillar samples have an approximate height-to-diameter ratio of 1:2.5, and the taper angle from the top to the bottom is about 2–3°. The morphologies of representative micropillars after compression are shown in Figures 1–3. The deformation mode of these micropillars is invariably the localized shear banding, independent of the specimen size. The first shear band is initiated from the corner of the contact surface between the specimen and compression indenter punch, where the sample has the least cross-sectional area and thus experiences the maximum stress.

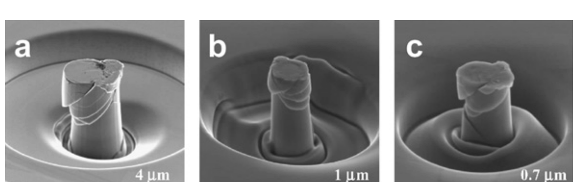

Figure 1. SEM micrographs showing the appearance of deformed pillars at a strain rate of $1 \times 10^{-4}\ s^{-1}$: (a) 3.8 μm, (b) 1 μm, and (c) 700 nm.

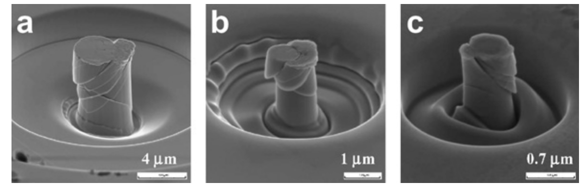

Figure 2. SEM micrographs showing the appearance of deformed pillars at a strain rate of $1 \times 10^{-3}\ s^{-1}$: (a) 3.8 μm, (b) 1 μm, and (c) 700 nm.

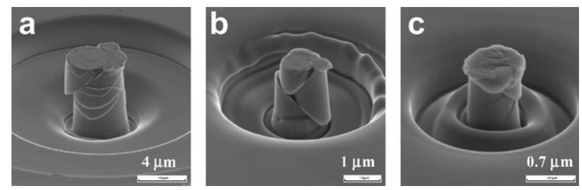

Figure 3. SEM micrographs showing the appearance of deformed pillars at a strain rate of $1 \times 10^{-2}\ s^{-1}$: (a) 3.8 μm, (b) 1 μm, and (c) 700 nm.

With increasing straining, additional shear bands are triggered continually. From Figures 1–3 we can observe that sample shear-off does not occur immediately. This phenomenon is also reflected in the engineering stress–strain curves presented in Figures 4a–c.

It is pointed out that an engineering stress–strain curve is normally converted from the load–displacement data under the assumption that the specimen is uniformly deformed. In this study, the deformation of Zr-based BMG micropillars is dominated by the emission of shear bands in a manner of "strain burst" to release the energy, similar to that in crystalline solids [9]. However, in contrast to that in the case of Mg-based BMG micropillar which exhibits only one single strain burst (Fig. 4d), strain bursts in the present ductile Zr-based glassy micropillars are multiple and they appear to proceed in a progressive fashion. The increment of the load or the transformed engineering stress in each step varies from 100 to

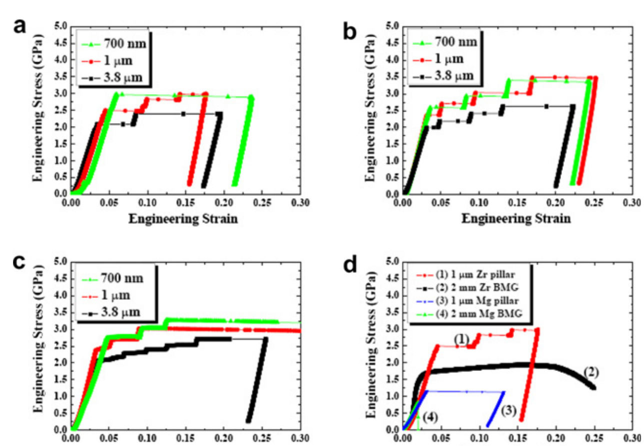

Figure 4. The micro-compression engineering stress–strain curves of the 3.8 μm, 1 μm and 700 nm micropillars at different strain rates of (a) $1 \times 10^{-4}\ s^{-1}$, (b) $1 \times 10^{-3}\ s^{-1}$, and (c) $1 \times 10^{-2}\ s^{-1}$. The comparison of the curves for the Zr and Mg 2 mm bulk compression specimens, and the 1 μm Zr and Mg micropillars, compressed at $10^{-4}\ s^{-1}$ is presented in (d).