Sample size effect and microcompression of Mg_{65}Cu_{25}Gd_{10} metallic glass

C. J. Lee and J. C. Huang

Institute of Materials Science and Engineering, Center for Nanoscience and Nanotechnology, National Sun Yat-Sen University, Kaohsiung, Taiwan 804, Republic of China

T. G. Nieh

Department of Materials Science and Engineering, the University of Tennessee, Knoxville, Tennessee 37996-2200, USA

(Received 18 July 2007; accepted 27 September 2007; published online 19 October 2007)

Micropillars with diameters of 1 and 3.8 μm were fabricated from Mg-based metallic glasses using focus ion beam, and then tested in compression at strain rates ranging from 6×10^{-5} to 6×10^{-1} s^{-1}. The apparent yield strength of the micropillars is 1342–1580 MPa, or 60%–100% increment over the bulk specimens. This strength increase can be rationalized using the Weibull statistics for brittle materials, and the Weibull modulus of the Mg-based metallic glasses is estimated to be about 35. Preliminary results indicated that the number of shear bands increased with the sample size and strain rates.


The strengths of face-centered-cubic single crystals are known to be strongly dependent on the sample dimensions, especially when the dimension is close to micron or submicron scales.1–5 For example, the strengths of Ni or Au micro-sized single crystal specimens are several times higher than that of the bulk specimens, and the strength can be even further increased by more than one order of magnitude in submicron pillar specimens. This dramatic effect occurs when the size of samples is smaller than the characteristic length for multiplication, which results in the dislocation starvation.

In contrast, bulk metallic glasses (BMGs) do not have a long range ordered structure. The plastic deformation of BMGs at room temperature proceeds in the form of highly localized shear bands.6,7 Yielding is induced by shear band nucleation and propagation. BMGs are not expected to work harden because localized shear bands do not exert long range interactive force like dislocations do. Thus, once yielding starts shear band propagates very rapidly and leads to catastrophic failure.

It has been recently shown that the strength of Mg-based BMGs is sensitive to the specimen size.8 Specifically, the strength is higher in a smaller size sample. There is yet another sample size effect in BMGs. Schuh et al.9 recently employed nanoindentation to study the shear band dynamics and identified a region (homogeneous region II), which was characterized by extensive emission of multiple shear bands. Gao et al.10 also developed a free-volume-based, thermoviscoplastic constitutive law to describe the inhomogeneous deformation in amorphous alloys and concluded that homogeneous region II actually corresponded to a region with fine shear bands, and the shear band spacing decreases with increasing strain rate. The question then arises as to what would happen when the specimen size is reduced to a range where the specimen can no longer support even one single shear band. A preliminary result10 indicated that the size scale is probably around 1 μm or less. In the current paper, we fabricated microscale pillar specimens and subsequently tested them in compression to study their mechanical response as a function of specimen size and strain rate.

We selected a Mg_{65}Cu_{25}Gd_{10} (in at. %) amorphous alloy for this study.

Compression samples were prepared using the dual focus ion beam system (FIB) of Seiko, SMI3050 SE, following the method developed previously.5 Firstly, the 30 keV and 7–12 nA currents Ga beam in FIB was faced to the surface of the BMG disk to mill a crater with a bigger size island being located in the center. Secondly, the same voltage and smaller currents of 3 down to 0.2 nA were used to refine the preserved island in the center to a desired diameter and height of pillars. Two kinds of micropillars with diameters of 3.8 and 1 μm, and the respective height-to-diameter ratio of 2–2.5 and 2.5–3 were fabricated. These pillars, as shown in Fig. 1, are tapered because the divergence of ion beam, and the convergence angle is about 2.5°.

Microcompression tests were performed with an MTS nanoindenter XP under the continuous stiffness measurement mode using a flat punch, which was machined out of a standard Berkovich indenter also by FIB. The projected area of the tip of the punch is an equilateral triangle of 13.5 μm. A prescribed displacement rate of 0.25–500 nm/s, which corresponds to the initial strain rates of 6×10^{-5}–6×10^{-2} s^{-1}, was used to deform the pillars. The load-displacement data are presented in Fig. 2. Traditionally, the curves are readily converted into the engineering stress and strain curves, with the assumption that the sample is uniformly deformed. However, the assumption is obviously violated since a bulk metallic glass is deformed by the emission of highly localized shear bands, as will be shown later. Thus, we choose to present the load-displacement data for the convenience of discussion.
Since the pillars are uniformly tapered, the total displacement $\Delta h$ is

$$\Delta h = \frac{1}{h_0} \int_0^{h_0} \Delta u(x) dx,$$

where $h_0$ is the height of the pillar $\Delta u(x)$ is the local displacement. In the elastic range, with $\sigma = E\epsilon$ and $\sigma(x) = P/A(x)$, where $P$ and $A$ are instantaneous load and sample cross section area, respectively, Eq. (1) is deduced to

$$\Delta h = \frac{P}{h_0} \int_0^{h_0} \left( 1 + \frac{2 h_0}{d_0} \sin \theta \right) dx,$$

where $d_0$ is the radius of the pillar and $\theta = 2.5^\circ$ is the taper angle. As $\sin \theta \approx \theta$, we obtain

$$E = \left( \frac{P}{\Delta h} \right) \left[ \frac{\ln \left( 1 + \frac{2 h_0}{d_0} \right)}{\pi d_0 \theta} \right].$$

Substitute $d_0 = 0.5 \mu m$ and $h_0 = 2.5 \mu m$, we finally have

$$E = 2.64 \times 10^{12} \left( \frac{P}{\Delta h} \right),$$

where $P$ and $\Delta h$ are in the units of Newton and meter, respectively. Thus, the elastic modulus is the slope of load-displacement curve multiplied by a constant of $2.64 \times 10^{12}$. The current result of improved strength caused by a decrease in sample size is in accord with a previous study on millimeter-scaled BMG. Also noted in Table I is the fact that the average strength of the 1 $\mu m$ pillar ($\sim 1500$ MPa) is higher than that of the 3.8 $\mu m$ pillar ($\sim 1390$ MPa). This demonstrates that the sample size effect can extend from the millimeter to micron range. The strength improvement from millimeter-scaled sample to micron-scaled pillar is noted to be about 60%–100%, which is quite remarkable.

After yielding, the load remains essentially constant at different strain rates, as evident in Fig. 2. The corresponding engineering stress-strain curves, not shown here, have a similar shape. This particular shape of stress-strain curve usually, according to the conventional wisdom, leads to a conclusion that BMG behaves like a perfectly plastic material. However, the raw displacement-time data, as given in Fig. 3, seem to reveal a different message. Specifically, data in Fig. 3 indicate the displacement (or strain) takes place almost instantly. In other words, strain does not occur in a gradual fashion but in the form of burst, even though the tests were conducted under strain rate control conditions. Every strain burst event, regardless of the strain rate, proceeds within about 1 s, suggesting that the strain rate during these bursts was at least $10^{-3}$ s$^{-1}$. There is also a notable trend that, for both micropillars, a faster compression rate results in a larger strain burst. The instant strain burst is peculiar but is usually indicative of a sudden propagation of a localized shear band.

The morphology of compressed pillar samples is shown in Fig. 4. It is well known that bulk Mg-BMGs generally fracture into fragments at the onset of yielding since they have a low Poisson’s ratio of $\sim 0.32$. However, the present micropillar samples do not exhibit such a catastrophic fracture mode even at a “macroscopic” strain of 10%–15%. In fact, all of them deformed by macroscopic shear, presumably resulted from localized shear band propagation. In principle, shear band is anticipated to initiate from the corner of contact between specimen and compression punch. This is the location where the sample experiences the maximum stress, not only because it has the minimum cross section as a result of sample taper, but also due to the large constraint caused by the friction between the test specimen and punch. Indeed, it is clearly shown in Fig. 4, in which all pillars are observed to deform by this shear mode after yielding. Furthermore, immediately following the yielding, the upper part of the specimen begins to slide along a major shear plane. However, the effective load-bearing cross-sectional area always remains constant, as the punch impresses onto the bottom part of the
An increase in strength with decreasing sample size can be rationalized in the following. For brittle materials, the variability of their strengths is expected based on their flaw sensitivity and may be analyzed using the Weibull statistics. The Weibull equation describes the fracture probability $P_f$ as a function of a given uniaxial stress $\sigma$ in the form of

$$P_f = 1 - \exp \left[ - \left( \frac{\sigma - \sigma_0}{\sigma_m} \right)^m \right],$$

where $\sigma_0$ is a scaling parameter, $m$ is the Weibull modulus, and $V$ is the volume of the tested sample. The parameter $\sigma_m$ denotes the stress at which there is a zero failure probability, and is usually taken to be zero. Assume the characteristic flaw causing fracture in both millimeter and micron samples are the same, then, at a fixed fracture probability, i.e., $P_f=$ constant, the above equation reduces to

$$V \left( \frac{\sigma}{\sigma_0} \right)^m = \text{const.}$$

Since $V \propto d^3$, where $d$ is the diameter of the compression sample, we obtain

$$d^3 \sigma^m = \text{const.}$$

As listed in Table I, the apparent strengths of the 3 mm and 1 $\mu$m Mg-BMG samples are 800 and 1580 MPa, respectively. Insert these values into Eq. (7), the Weibull modulus is calculated to be about 35, which is within the range of the $m$ values recently reported for Zr$_{48}$Cu$_{45}$Al$_7$ ($m=$73.4) and (Zr$_{48}$Cu$_{45}$Al$_7$)$_{98}$Y$_2$ ($m=$25.5). The above analysis supports the observation that the compression stress increases with decreasing specimen size.

The authors gratefully acknowledge the sponsorship from the National Science Council of Taiwan, Republic of China, under the Project No. NSC 96-2218-E-110-001. This work was also partially supported by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under Contract No. DE-FG02-06ER46338 with the University of Tennessee.

---