Strain burst speeds in metallic glass micropillars

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\textbf{ABSTRACT}

Uniaxial microcompression and nanoscratch tests using the nanoindentation system on the Mg-, Au- and Zr-based metallic glass micropillars with diameters of 3.8 and 1 \textmu m were performed and compared. Strain burst phenomena were observed in all microcompression tests as indications of localized shearing. The strain burst speed of micropillars can be measured based on the raw displacement-time profile. The results indicate that strain burst speed of metallic glasses increases with increasing sample size, with decreasing wear resistance, and with decreasing ductility. The current study demonstrates that strain burst speed and wear characteristics can be regarded as promising indicators for the ductility of metallic glasses.

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1. Introduction

It is well known that bulk metallic glasses (BMGs) possess outstanding properties such as high yield strength, excellent wear and corrosion resistance, high fracture toughness and easy shaping/forming ability \cite{1–5}. Among all of unique properties, mechanical properties are the most important subject in applications as engineering materials. With the emerge of micro-electro-mechanical systems (MEMS) and other microscaled devices, the fundamental properties of micrometer-sized samples have become increasingly more important. Therefore, several studies have been conducted to study the sample-size effect of BMGs\cite{6–11}.

Such previous investigations focused mainly on the relationship between yield strength and sample size. In addition to sample-size effect on the BMG yield strength, several unusual features were observed in the deformation of such micropillars. For example, Schuster et al.\cite{9} suggested that theses small samples are unable to make the transition to a fully developed shear band since no veins or droplets are observed in the fracture surface. Cheng et al.\cite{10} demonstrated that global melting is observed in micropillars tested under high strain rate compression. Volkert et al.\cite{11} showed that homogeneous deformation dominates when shear-band spacing is greater than the dimension of test samples. Up to date, the detailed information about deformation mechanism of micrometer-sized BMGs still needs more efforts to explore.

In BMGs, the deformation mode is dominated by single shear along the principal shear plane, and the plastic deformation is accompanied by flow serrations\cite{12,13}. These flow serrations have been demonstrated to be caused by consecutive shear-band propagation on the principal shear plane\cite{14,15}, which eventually leads to the macroscopic localized shear. Compared to millimeter-scaled BMGs, localized shear is also revealed as the dominant deformation mode in micrometer-sized BMGs, and strain burst phenomena are observed in all microcompression tests. Based on the previous studies\cite{6–11}, the strain burst phenomenon in micropillars is usually indicative of a sudden propagation of a localized shear band, which is related to the flow serrations in BMG bulk samples. Thus, it is of interest to examine the relationship between the strain burst speed and many mechanical characteristics critical for MEMS applications.

2. Experimental

To assess the strain burst speed, we selected the brittle-natured Mg\textsubscript{65}Cu\textsubscript{25}Gd\textsubscript{10}, moderate brittle Au\textsubscript{49}Ag\textsubscript{5.5}Pd\textsubscript{2.3}Cu\textsubscript{26.9}Si\textsubscript{16.3}, and ductile phase-separated Zr\textsubscript{63.8}Ni\textsubscript{16.2}Cu\textsubscript{15}Al\textsubscript{5} amorphous alloys (all compositions in atomic percent). The BMGs were prepared by injection casting or suction casting into a water-cooled Cu mold with an internal cylindrical-shaped cavity of 3 mm in diameter. Detailed information about the preparation procedure of the Mg-, Au- and Zr-based BMGs can be found elsewhere\cite{16–18}. X-ray diffraction...
respectively. The corresponding strain rates vary from 1 to 25 nm/s for 1 μm deformed with different prescribed displacement rates ranging from 0.25 to 25 nm/s for 1 μm pillars and 0.95–95 nm/s for 3.8 μm pillars, respectively. The corresponding strain rates vary from 1 × 10⁻⁴ to 1 × 10⁻² s⁻¹. To measure strain burst speed as precisely as possible, the pillar samples compressed at a high strain rate were tested using a high data acquisition rate of 100 Hz or 400 Hz. Due to the limitation of storage capability in each microcompression test, the low strain-rate compression tests were forced to use a lower data acquisition rate ranging from 0.25 to 25 nm/s for 1 μm pillars and 0.95–95 nm/s for 3.8 μm pillars, respectively. The more ductile Zr BMGs show the slower strain burst speed. The difference can be up to 3–7 times. Moreover, the strain burst speeds of the 3.8 μm pillar samples are appreciably higher, being 9.1–10.6, 5.0–8.0 and 1.8–2.5 μm/s for the Mg-, Au- and Zr-based BMG micropillars, respectively. The alloy dependence is the same as the 1 μm micropillars, and the difference in speed can be over one order of magnitude.

While the micropillar strain burst speeds show both alloy systems and pillar size dependences, the loading strain-rate effect seems to be minor. In view of Table 1, the strain burst speeds at a high strain rate are only slightly higher than those at a lower strain rate. For the ductile Zr-based pillars, there is hardly distinct strain rate dependence. Namely, the strain burst speed is insensitive to the applied strain rate, which is consistent with the results of Chen et al. [15]. Note that the applied loading velocity for these micropillars is in the range of 0.25–95 nm/s, which are much lower than the burst speeds (0.2–10.6 μm/s).

The representative load-displacement and displacement-time curves are shown in Fig. 3(a) and (b), respectively. Independent of the metallic glass systems (Mg-, Au- or Zr-based), micropillar diameters (1 or 3.8 μm) and compression strain rates (1 × 10⁻⁴–1 × 10⁻² s⁻¹), sudden strain bursts are distinctly observed, as the example demonstrated in Fig. 3(b). Note that the more brittle-natured Mg- and Au-based BMG micropillars (with ∼0% plastic strain for the Mg-based bulk rod compression specimens [16] and ∼1.5% plastic strain for the Au-based bulk rod compression specimens, obtained in our parallel study) exhibit one or two strain bursts over the micro-compression process, while the ductile phase-separation Zr-based BMG micropillars (with ∼13% plastic strain for their bulk rod compression specimens [18]) show multiple strain bursts and these bursts proceed in a progressive fashion. The recorded displacement-time information and the data extracted from strain burst speed allow a detailed analysis of strain burst characteristics during compression. Table 1 summarizes all of the calculated strain burst speeds of the Mg-, Au-, and Zr-based 1 and 3.8 μm pillars at different strain rates.

By close examination of strain burst speed data in Table 1, there are clear trends showing the BMG alloy systems and the micropillar size dependences. For the 1 μm pillar samples, the strain burst speeds are scattered in the range of 0.7–1.6, 0.4–1.5 and 0.2–0.3 μm/s for the Mg-, Au- and Zr-based BMG micropillars, respectively. The more brittle Mg and Au BMGs exhibit faster strain burst speeds. The difference can be up to 3–7 times. Moreover, the strain burst speeds of the 3.8 μm pillar samples are appreciably higher, being 9.1–10.6, 5.0–8.0 and 1.8–2.5 μm/s for the Mg-, Au- and Zr-based BMG micropillars, respectively. The alloy dependence is the same as the 1 μm micropillars, and the difference in speed can be over one order of magnitude.

3. Results and discussion

For the microcompression tests, the typical appearances of the deformed pillars are shown in Fig. 1 for the 3.8 μm Mg-, Au- or Zr-based BMG micropillars. It is apparent that localized shear deformation is still the dominant deformation mode. To observe the microstructure of pillar sample, TEM samples are fabricated by FIB with a trenching and liftout technique. Fig. 2(a) and (c) show the SEM images of the 1 μm Zr-based BMG pillar sample before and after microcompression test. And Fig. 2(b) and (d) are the bright field TEM images of a lamella prepared from the undeformed and deformed micropillars. The corresponding selected area diffraction patterns are shown in the insets. According to TEM observations, the deformed Mg-, Au- and Zr-based BMG pillars are still all of the amorphous structures.

The microcompression samples were prepared using the dual focused ion beam (FIB) system of Seiko, SM13050 SE, following the method developed by Uchic and Dimiduk [19]. Detailed description of the preparation methods have been described elsewhere [6–8]. In the current study, the cylindrical micropillars with diameters of 3.8 and 1 μm were fabricated. Microcompression tests were performed in an MTS nanoindenter XP with a continuous stiffness measurement mode using a flat punch indenter with an equilateral triangle cross-section measuring 13.5 μm in side length. These samples were deformed with different prescribed displacement rates ranging from 0.25 to 25 nm/s for 1 μm pillars and 0.95–95 nm/s for 3.8 μm pillars, respectively. The cross-section pro-
on these three BMGs, following the studies of Hodge et al. [20] and Wang et al. [21]. The wear resistance of three BMGs can be estimated based on the results of scratch tests. In general, strain burst speed may be closely related to the friction coefficient of metallic glasses. Imagining the bicrystal sliding, slide resistance is directly linked to the friction between two crystals. It is the best way to measure the “intrinsic” friction coefficient of a BMG if we use a BMG tip to slide over the same BMG surface. Even though it is unavailable to achieve this measurement in the current situation, measuring the coefficient using a diamond tip can still provide useful relative information and basic trend.

The typical scratch depth profiles of three different BMGs are shown in Fig. 4(a). The scratch depths of the Mg-, Au- and Zr-based BMGs are ~367, ~316 and ~250 nm, respectively. The cross-section profiles for these three BMGs are measured by a surface scan under a slight load (100 μN) at a halfway point of scratch distance, as indicated in Fig. 4(b). Fig. 5 shows the SEM micrographs showing the worn scratches in three different BMG alloys. It is readily seen that material pile-up occurred in the scratch tracks and shear bands formed at the sides of such scratches. The total volume of materials removed during scratch test can be qualitatively measured by scratch depth and cross-section profile due to the fixed Berkovich tip.
indenter geometry. Subsequently, the wear coefficient, $K$, and wear resistance $R_W$ of three different BMGs can be calculated based on the Archard wear equation \[22\] and the modified Archard wear equation \[20,21\]. Here, the wear resistance is a reflection of the difficulty of bond breaking in the worn material. The Archard wear equation can be expressed as:

$$V_W = KSN,$$  \hspace{1cm} (1)

where $V_W$ is the total volume of material removed by the wear, $K$ is the wear coefficient, $S$ is the total sliding distance, $N$ is the normal load, and $H$ is the material hardness. The wear coefficient is also one of the wear properties. For the ease of engineering applications, the Archard wear equation can be modified as:

$$R_W = \frac{SN}{V_W},$$  \hspace{1cm} (2)

where $R_W$ is the wear resistance with a unit of Pa. Taking proper values for each variable into Eqs. (1) and (2), the average wear resistances of three BMGs are calculated to be $0.97 \times 10^{11}, 2.17 \times 10^{11}$ and $4.17 \times 10^{11}$ Pa, respectively, as tabulated in Table 1. The lower wear resistance in the Mg-based samples indicates the lower resistance of bond breaking. During shear-band propagation, the Mg samples tend to impose a lower resistance for shear-band propagation, resulting in a higher burst speed.

In Table 1, the wear characteristics exhibit a direct relationship with the strain burst speed for three BMGs, namely, based upon bicrystal sliding model, the higher wear resistance will show the lower strain burst speed. This trend is very consistent with that obtained from microcompression tests. It suggests that the argument of strain burst speed in microcompression tests can be rationalized by the nanoscratch tests.

It is also intended to apply another perspective to explain the different strain burst speeds in three different BMGs. From the plastic shear resistance point of view, Wang et al. \[23\], who performed nanoindentation tests on the Mg-, Au- and Zr-based BMGs, and found that the plastic shear resistance is dependent on the shear modulus. In our current study, the shear modulus of the Mg- and Au-based BMGs is $18.6$ \[24\] and $26.45$ \[17\], respectively. For the Zr-based BMG, the shear modulus of $Zr_{63.8}Ni_{16.2}Cu_{15}Al_{5}$ cannot be found in the literature, but for the alloys with similar compositions, $Zr_{64.1}Cu_{15.75}Ni_{10.12}Al_{10}$ and $Zr_{65.5}Cu_{15}Ni_{10}Al_{10}$, the shear modulus are $28.46$ and $30.27$ GPa \[25\], which are both higher than the shear modulus of the Mg- or Au-based BMGs. For this reason, the higher modulus in the Zr-based alloys will be inherent with a higher bond strength, which will lead to higher plastic shear resistance and lower strain burst speeds during the microcompression tests.

As reported by Song et al. \[14\] and Chen et al. \[15\], shear-band propagation in BMGs consists of three steps: acceleration, steady state, and deceleration, indicating the continuous dissipation of kinetic energy carried by a travelling shear band. In BMGs, when shear bands form and start to propagate, these shear bands have a sufficient kinetic energy to propagate through the entire test sample. However, in metallic glassy micropillars, Schuster et al. \[9\] reported that a fully developed shear band was difficult to be formed in micropillar samples due to the lack of sufficient space. This suggest that these micropillar samples appears unlikely to make the transition to final or mature shear band, which is also reported in the literatures \[26,27\]. Thereby, the propagation speed of ill-developed shear band in metallic glassy micropillars should be different from that in BMG bulk samples, and much slower shear-band propagations would be recorded in metallic glassy micropillars. Here, we arbitrarily take strain burst speed as shear-band propagation speed in BMG micropillars. Note that the burst speeds recorded in bulk specimens are typically in the range of $100–500$ \(\mu\text{m/s}\) \[15\], and the burst speeds in the current micropillars are only $0.2–10.6$ \(\mu\text{m/s}\). It is reasonable to assume that such ill-developed shear-band propagation is limited within the initial acceleration step.

From a free volume point of view, a propagating shear band initially accelerates, as a result from free volume creation, and, then, reaches a steady state as a result of the balance between the creation and annihilation of free volume. After the dissipation of kinetic energy, the propagating shear band decelerates and is arrested. The last step represents the overwhelming role of free volume annihilation over creation. Shear-band propagation can be regarded as a process of the competition between the creation and annihilation of free volume. Therefore, for BMG micropillar samples, after the creation of free volume upon the shear band starting propagation, shear-band propagation would be easy to be temporarily stopped by the rapid annihilation of excess free volume due to insufficient kinetic energy for the free volume use-up.

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**Table 1**

Summary of data measured using microcompression tests and nanoscratch tests.

<table>
<thead>
<tr>
<th>BMG</th>
<th>Strain rate (s(^{-1}))</th>
<th>Strain burst speed (µm/s)</th>
<th>Wear resistance (10(^{11}) Pa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg-based BMG</td>
<td>$1 \times 10^{-3}$</td>
<td>$0.7 \pm 0.2$</td>
<td>$9.1 \pm 1.2$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-4}$</td>
<td>$0.8 \pm 0.1$</td>
<td>$10.0 \pm 3.1$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-5}$</td>
<td>$1.5 \pm 0.4$</td>
<td>$10.6 \pm 2.9$</td>
</tr>
<tr>
<td>Au-based BMG</td>
<td>$1 \times 10^{-4}$</td>
<td>$0.4 \pm 0.1$</td>
<td>$5.4 \pm 1.4$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-5}$</td>
<td>$0.5 \pm 0.1$</td>
<td>$5.0 \pm 0.7$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-6}$</td>
<td>$1.5 \pm 0.3$</td>
<td>$8.0 \pm 1.5$</td>
</tr>
<tr>
<td>Zr-based BMG</td>
<td>$1 \times 10^{-4}$</td>
<td>$0.2 \pm 0.1$</td>
<td>$2.1 \pm 1.0$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-5}$</td>
<td>$0.3 \pm 0.2$</td>
<td>$1.8 \pm 0.8$</td>
</tr>
<tr>
<td></td>
<td>$1 \times 10^{-6}$</td>
<td>$0.3 \pm 0.1$</td>
<td>$2.5 \pm 1.1$</td>
</tr>
</tbody>
</table>

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**Fig. 4.** (a) Scratch depth profile versus scratch length and (b) cross-section profile and for the Mg-, Au- and Zr-based BMGs.
In our previous study on the millimeter-scaled BMG compression specimens [15], the more ductile BMG will show the slower shear-band propagation. The current results on the metallic glassy micropillars also demonstrate the same trend. The concept of slower shear-band propagation would lead to the more ductile behavior appears to be able to extend from the millimeter-scaled to micrometer-scaled range for metallic glasses. It seems that the strain burst speed and wear characteristics might be regarded as a promising indicators for the ductility property of metallic glasses.

Nevertheless, the distinct difference in shear-band propagation between BMG bulk and micropillars samples may be attributed to another possible reason: the instrumental or methodological artifact for nanoindentation systems. Presumably, the flat punch tip might have lost contact with micropillar sample under a certain load when shear band propagates. As a result, strain burst speed (or shear-band propagation speed) is only a lower limit value. To characterize the strain burst speed in metallic glasses, a more accurate testing nanoindentation machine equipped with a high data acquisition rate is necessary. The qualitative information in shear-band propagation might be useful for getting a rough picture, but the more accurate quantitative measurement of strain burst speed needs to be further investigated in future.

4. Conclusions

Through the microcompression tests on micrometer-sized Mg-, Au- and Zr-based metallic glassy micropillars, the strain burst speeds, or the shear-band propagation speeds, are characterized and analyzed. Coupled with our previous results [15], the following conclusions can be drawn.

(1) The burst speeds seem to exhibit the sample size dependence from the slowest in the 1 μm pillars, to the faster in the 3.8 μm pillars, and to the much faster in the millimeter-sized bulk samples.

(2) The burst speeds also exhibit alloy system dependence. The burst speeds are the slowest in the ductile Zr-based pillars, followed by the faster in the moderate brittle Au-based pillars and then the fastest in the brittle Mg-based pillars. The trend is consistent with our previous finding from the BMG bulk samples.

(3) From the compression and sliding experiments, it appears that the more ductile metallic glasses will exhibit the slower burst or shear band propagation speeds, as well as the higher sliding resistance. The burst speed and wear characteristics might be promising indicators for the ductility property of metallic glasses.

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