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Subsurface deformation during Vickers indentation of bulk metallic glasses

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Abstract

Bonded interface technique was employed to examine the nature of subsurface deformation during Vickers indentation in two kinds of bulk metallic glasses (BMGs), $Pd_{42.1}Ni_{39.77}P_{18.13}$, and $Zr_{56.69}Cu_{26.96}Al_{10.95}Ni_{5.4}$. Quantitative information such as the shear band spacing, pile-up length, subsurface deformation zone size etc. was recorded for indentation loads ranging from 50 to 5000 g. Experimental results show that both the BMGs have an average hardness value of ~550 VHN with slightly higher hardness at low loads. Observations of deformation zones indicate that they deform appreciably through the shear band mechanism. For both bulk indentation, as well as the interface indentation, the normalized size of the deformation zone was found to be independent of the applied load. Two types of shear bands, radial, and semi-circular in nature, have been observed. These results are compared with similar studies made on ductile metals and silicate glasses.

Keywords: Bulk metallic glasses; Vickers indentation; Bonded interface technique; Shear band; Subsurface deformation

1. Introduction

The plastic flow in metallic glasses at high stresses and low temperatures occurs inhomogeneously, localizing into slip bands along planes of maximum shear. Shear band formation is associated with localized atomic shear-rearrangements, correlated to regions of excess free volume [1]. In late 1980s and early 1990s, it was discovered that metallic glasses could be processed in bulk form in certain ternary, quaternary, and higher order compositions due to the sluggish crystallization kinetics in them [2]. The availability of metallic glasses in bulk form facilitates studying their mechanical behavior in detail, with possible structural applications in mind. Hence, research focused towards understanding the micro-mechanisms of deformation and in developing the constitutive stress–strain relationships in this class of material is actively being pursued. Indentation tests are excellent means for such studies, especially needing only a small volume of material. Deformation by indentation is inherently stable, at least macroscopically, because the contact area between the indenter and the material being deformed increases during the course of indentation to support whatever load is imposed.

This work was initiated with the objective of conducting systematic studies on the deformation behavior associated with indentation in bulk metallic glasses (BMGs), with particular focus on elucidating the subsurface deformation characteristics. For this purpose, a Pd-based ternary alloy and a Zr-based quaternary alloy were selected. Bulk and interface indentation experiments, the latter employing the bonded interface technique, were carried out and the preliminary results of these studies are reported in this paper.

2. Materials and experiments

The Pd-based alloy ingot, whose nominal composition is $Pd_{42.1}Ni_{39.77}P_{18.13}$, was first prepared by arc melting pure metals in a purified argon atmosphere. The BMG was then obtained by repeated melting of the molten alloys, fluxed with B_2O_3, in a quartz tube. Finally, the tube was water quenched to form ~4 mm diameter BMG rod. 2 mm thick plates of the Zr-based alloy, whose nominal composition is $Zr_{56.69}Cu_{26.96}Al_{10.95}Ni_{5.4}$, was formed by melting pure metals in argon atmosphere and then chill-casting it in plate shaped copper mould. The as-cast alloys were characterized

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using X-ray diffraction, differential scanning calorimetry, and transmission electron microscopy, to ascertain their amorphous nature.

Indentation specimens for the bonded interface technique were prepared by cutting the BMG specimens first into two halves and then polishing them to a 1 μm finish prior to bonding them using a high strength adhesive. In order to minimize the bond layer thickness and to obtain a high strength bond, specimens were put into PVC moulds and then filled up with cold mount resin. After this the top surface was polished carefully so that the indentation face was flat and horizontal. Vickers diamond indentations were carried out both on the bonded interface, as well as away from it. Indentations on the interface were carried in such way that one of the indentation diagonals coincided with the interface. Maximum indentation loads applied varied from 500 to 5000 g for the interface indentation whereas for bulk indentation (i.e., indentation away from interface) loads ranged from 50 to 2000 g. The bonded interface was subsequently opened by dissolving the adhesive in acetone. Deformation zones both under the indenter tip (subsurface) as well as around the bulk indentations were examined using a scanning electron microscope and parameters like shear band spacing, plastic zone size etc. were carefully quantified.

3. Results

3.1. Bulk indentation

Fig. 1 shows the variation of the measured Vickers hardness (VHN) as a function of the maximum applied load for the Pd- and Zr-based BMGs, respectively. The hardness values for the Pd-based BMG are within the range of 550–600 VHN when the applied load is between 50 and 200 g. However, when the load is increased to more than 200 g, the hardness values gradually decrease, reaching ∼530 VHN at 2000 g load. The trend in the hardness versus load was similar in the case of the Zr-based alloy, a high hardness of ∼550 VHN was obtained when the applied loads were small and a constant hardness of ∼530 VHN for loads of 500 g and above. The observation of slightly higher hardness values at low loads can be attributed to the higher resistance to shear band nucleation at these loads [3].

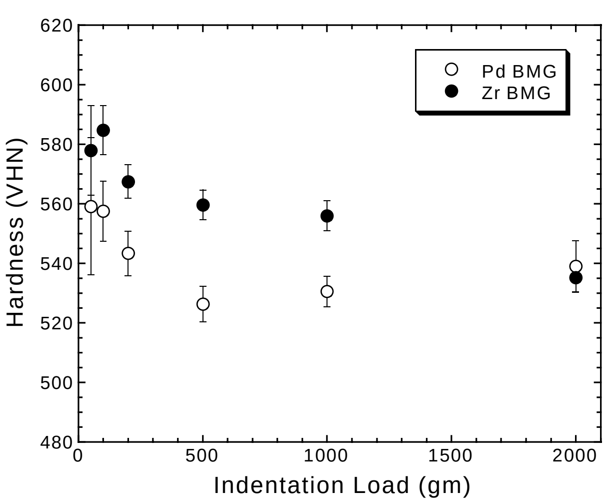

Fig. 1. Vickers hardness (VHN) as a function of the maximum applied load for the two BMGs examined.

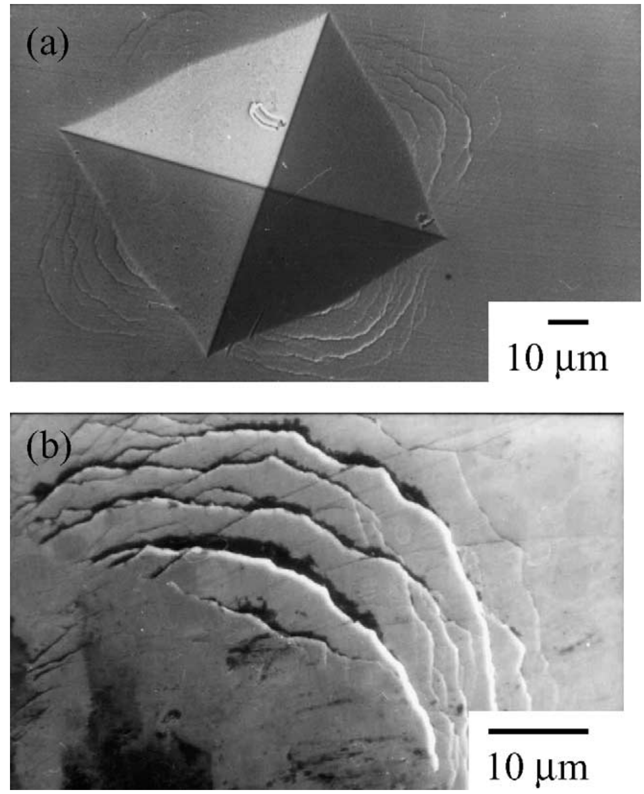

Fig. 2. (a) Representative optical micrograph showing the top-view of an indentation impression obtained with a load of 2000 g in the Pd-based BMG and (b) tilted image showing the step-like nature of the shear bands.

For both the alloys and at all the loads, pile-up of the material was observed around all the bulk indentations, occurring in the form of semi-circular shear bands that appear to emanate from the edge of the indentation. Representative optical micrograph showing top-view of the indentation impression is shown in Fig. 2a. Fig. 2b shows a SEM image obtained by tilting the sample, illustrating the step-like and wavy nature of the shear bands. The size of the pile-up (i.e., distance from the edge of the indenter imprint to the outer most shear band), δ was found to increase with load as shown in Fig. 3. However, δ in the Zr-alloys is comparatively smaller (∼15 μm at the load of 2000 g) to that observed in the Pd-based alloy (∼20 μm at the load of 2000 g). This indicates to a higher resistance to deformation in the Zr-based BMG, possibly a result of the partial crystallization present in the as-cast material itself [4].

In order to examine for the possible indentation impression size (which in turn depends on the applied load)

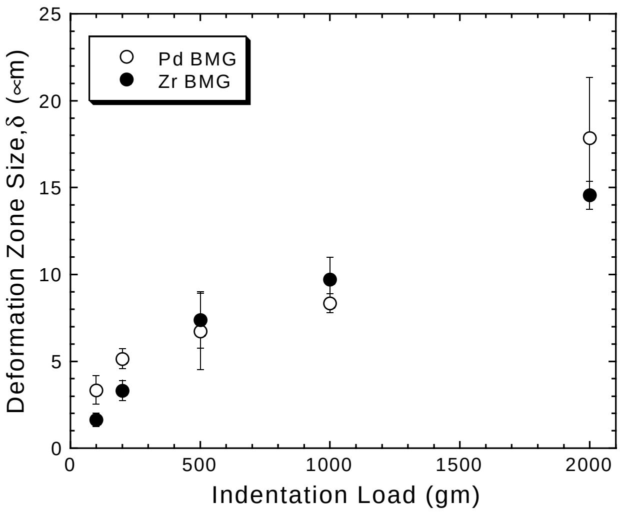

Fig. 3. Variation of the size of the deformed region, δ (distance from the edge of the indenter imprint to the outer most shear band), as a function of applied load.

dependency on the pile-up length, normalized pile-up lengths, i.e., ratio of δ and the total deformation size (distance from the center of the indent to the outer most shear band in the pile-up), Δ, at various loads were computed and compared. It was found that δ/Δ is independent of the applied load, with values of ∼0.35 and ∼0.29 for the Pd-based and the Zr-based BMGs, respectively. These values are substantially smaller than the deformation zones that are observed in ductile alloys [5–7].

3.2. Subsurface deformation

Fig. 4a shows deformation region underneath a Vickers indenter for the Pd-based BMG for an applied indentation load of 5000 g, obtained by employing the bonded interface technique. The deformation zone is semi-circular in nature containing a high density of shear bands. Atomic force microscopy and stereo-pair imaging using SEM confirmed that most of these were indeed shear bands and not cracks [4]. Two types of shear bands can be identified within this deformed zone, those that are semi-circular (out-of-plane displacements) while the other is radial (in-plane shear displacements). The density of the semi-circular shear bands is considerably larger vis-à-vis the radial shear bands in the case of Pd-based BMG. Tilting of the specimen confirms that the deformed zone is hemispherical in nature, with semi-circular shear bands forming steps on it.

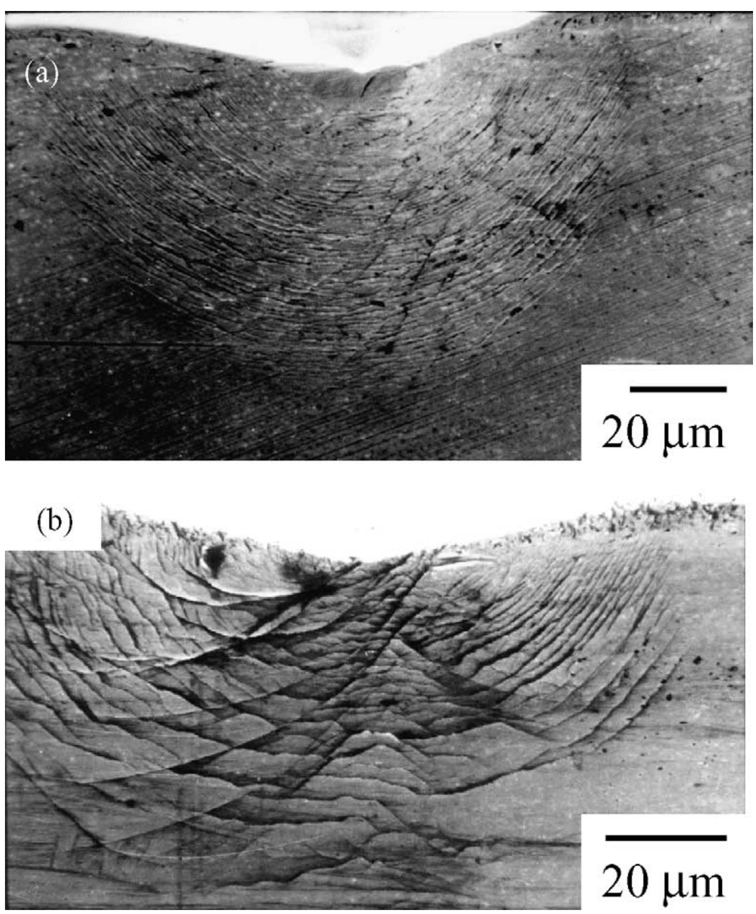

Fig. 4. Micrographs showing the deformed region underneath a Vickers indentation (load = 5000 g) obtained using the bonded interface technique. (a) Pd-based BMG and (b) Zr-based BMG.

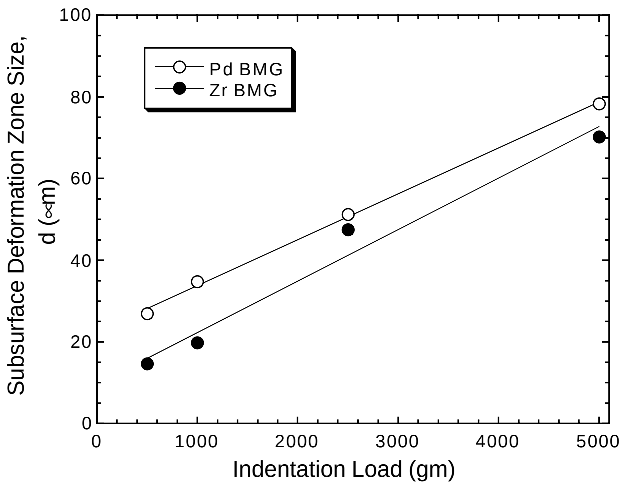

Fig. 5. Variation of the subsurface deformation zone size, *d*, as a function of the indentation load.

Fig. 4b shows the nature of subsurface deformation in the Zr-based BMG for an applied load of 5000 g. Again, a hemispherical deformation zone is seen. However, the radial bands are dominant in this case. The intersection angle of the radial bands is ∼90°, making them appear like slip lines.

Distance of the outermost shear band from the tip of indenter imprint, *d* (considered as the size of the subsurface deformation zone), was plotted as a function of the load in Fig. 5. For both the alloys investigated, *d* increases with increase in load, similar to that seen in bulk indentation. However, the *d*/*D* ratio (where *D* is the distance from the indented surface to the outermost semi-circular shear band) remains constant at ∼0.85 for both the alloys examined. Furthermore, the size of the subsurface deformation zone is much larger than the pile-up length during bulk indentation.

For the Pd-based alloy, the spacing between the semi-circular shear bands was found to be independent of the distance from indenter tip, with an inter-band spacing of 0.75 μm for the 2500 g load and ∼1 μm for the 5000 g load. In the case of the Zr-based glass however, the inter-band spacing was found to increase from about 1–2 μm at close to the indenter tip to a spacing of 8–10 μm for the outermost bands.

4. Discussion

A clear advantage of the bonded-interface indentation technique is that the deformation pattern can be imaged, which provides insights into the governing deformation mechanism [5]. However, its primary disadvantage is that the adhesive layer may relieve the elastic constraint for plastic flow that would have been present otherwise in the bulk indentation, introducing changes to the size and shape of the deformed zone as well as possibly altering the indentation mechanism itself. Mulhearn [5] has conducted extensive studies on various types of metals to examine this possibility and concluded that the relaxation of constraint may affect the extent of the deformed zone and the slope of the strain gradient, but has no significant effect on the contours of equal strain. On this basis, the experimental observations made in the current study can be considered to represent the true behavior of the material underneath the indenter.

Mulhearn [5], Samuels and Mulhearn [6], and Dugdale [7] have examined the subsurface deformation regime due to indentation of ductile metals, by employing various types of techniques, and have mapped the shape of the elastic–plastic and other low strain boundaries of the deformed zone. Their results suggest that the indentation by the Vickers indenter occurs by a compression of a set of concentric hemispherical shells rather than a cutting type mechanism, implying that plastic flow of ductile metals is dominated by the compression mechanism underneath the indenter. This type of behavior is best described by the expanding cavity model wherein a hemispherical radial mode of deformation is assumed [8,9].

The observation of concentric semi-circular shear bands for the Pd-based BMG, which are surface steps produced by the shear bands propagating to the relatively unconstrained free surface that is available at the bonded interface, substantiates that the mechanism governing the plastic flow in this alloy is similar to that seen for ductile metals. This is not surprising since the Pd-based alloy's continuum response is similar to that of a ductile metal, with an elastic–perfectly plastic uniaxial compressive response. Whereas, the plastic flow in ductile metals is macroscopically homogeneous (an averaged response of multitude of discrete dislocation motion), shear band propagation in metallic glasses is inhomogeneous in nature and hence results in several distinct bands within the deformed zone. Furthermore, the contours of the semi-circular shear bands are similar to the Mises equivalent strain contours underneath pyramidal indenters predicted by Giannakopoulos et al. [10], who have conducted detailed three dimensional finite element models for pressure sensitive solids. Donovan [11], who has examined the geometry of plastic flow under both spherical and Vickers in indentation in a $Pd_{40}Ni_{40}P_{20}$ amorphous alloy with the aid of sectioning and etching, has also arrived at a similar conclusion.

The radial bands dominant in the case of the Zr-based BMG appear to be a result of the cutting mechanism proposed by Tabor [12]. It is interesting to note that the subsurface deformation pattern observed in this glass is very similar to that seen in silicate glasses. However, subtle differences between the deformation pattern of the Zr-based BMG and the silicate glasses do exist. For example, Hagan [13] reported that the angle at which the radial flow lines meet in a soda–lime glass is 110° whereas it is ∼90° in the case of the Zr-based BMG.

With respect to the substantial difference in the flow patterns observed in the as-cast Pd-based BMG and the as-cast Zr-based BMG, the presence of dendrite-like crystallites in the latter case is thought to be the reason for the significantly lower deformation as well as different morphology of subsurface deformation [4]. Indeed, the deformation morphology in the Pd-glass, which is subjected to annealing heat treatments in order to systematically crystallize it, shows a subsurface shear band pattern that is similar to that seen in the Zr alloy [4].

5. Conclusion

In summary, the observations of the bulk and subsurface deformation zone made by indentation reported in this work show that the as-cast glass can deform appreciably through the shear band mechanism. Furthermore, the normalized deformed zone sizes, both for bulk indentation as well as the interface indentation, are independent of the applied load. Finally, inter-band spacing is independent of the location whereas it appears to depend on the load applied, increasing with increasing load. Research is underway to further analyze and understand the observed deformation behavior and to elucidate the governing constitutive response with the aid of modeling to that best describes the observed phenomenon.

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