Indenter Geometry Affecting Indentation Behaviors of the Zr-Based Bulk Metallic Glass

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Indenter dependent indentation behaviors of the Zr-based bulk metallic glass, such as adhesion, serrated flows, shear bands, residual indent morphologies, were found and analyzed in this paper. An interesting phenomenon is presented that adhesion of the Zr-based bulk metallic glass appears during the indentation test using the cube-corner indenter but it’s not observed when using the Berkovich indenter. Corresponding to the adhesion behavior, “dirty” but new surfaces with the nano-scale dimple structure are formed by the cube-corner indenter while relatively smooth surfaces with few discrete shear bands are obtained by the Berkovich indenter. Indentation experiments indicate that the adhesion force between the cube-corner indenter and the Zr-based bulk metallic glass depends on the loading rate, the maximum penetration load and the cyclic loading number. These phenomena will enhance understanding of shear band formation, shear-induced softening, and adhesion of bulk metallic glasses.

Keywords: indenter geometry, bulk metallic glass, adhesion, serrated flows, shear bands, indentation parameters

1. Introduction

Since their discovery in 1960,1) bulk metallic glasses (BMGs) with amorphous microstructures have been given huge attentions because of their excellent properties,2–6) such as high hardness, fracture strength, elastic limit, strength, corrosion resistance, good thermoplastic processability and biocompatibility. However, their poor ductility and highly localized plastic deformation in shear bands leading to macroscopically catastrophic failure, limit their applications for real structural materials. Both their excellent properties and embarrassing shortcomings make bulk metallic glasses become topics of considerable research for potential engineering applications. Bulk metallic glasses with the random or chaotic structure, lack crystalline defects such as grain boundaries and dislocations compared with conventional metals. So, the conventional dislocation-mediated deformation mechanism used for crystalline materials is not applicable for BMGs. Via tensile tests7–9) compressive tests,10–12) three-point bending tests13–15) and indentation tests,16–18) deformation mechanisms of BMGs have been studied for many years but physical processes and mechanisms related to initiation, formation and propagation of shear bands are still not very clear. In this study, we focus on another feature—indenter dependent behaviors, especially the adhesion behavior between the indenter and the Zr-based bulk metallic glass, which may be also related to shear bands but is rarely investigated up to now.

2. Materials

The Zr-based BMG—Zr_{46.5}Cu_{45}Al_{7.5}Ti_{1.5} was provided by the Research Group of Prof. Jianzhong Jiang in Zhejiang University. It was produced by arc-melting the mixtures of Zr, Cu, Al, and Ti elements in a Ti-gettered high-purity argon atmosphere. More information about the specimen preparation, its amorphous nature and related properties can be found in Refs. 19 and 20.

3. Experiments and Results

Before experiments, the sample was polished by the extreme precise lapping/polishing machine (Bni62, BN Technology Corporation). Then, indentation experiments were carried out by a self-made nanoindentation device, which has been introduced and calibrated in the Ref. 21). Two kinds of diamond indenters, the Berkovich indenter and the cube-corner indenter, were used, which were bought from Syntong-mdp LTD, Switzerland. Both of the curvature radiuses are less than 150 nm. During indentation experiments, the closed loop displacement control mode was selected and both the loading and unloading time were 60 s. Indentation curves obtained by the Berkovich indenter and the cube-corner indenter are illustrated in Figs. 1(a) and 1(b) respectively.

Comparing Figs. 1(a) and 1(b), an interesting phenomenon is observed that the penetration load becomes to be reverse direction when the penetration load reduces to zero in Fig. 1(b) but it is not observed in Fig. 1(a), which indicates that adhesion appears between the pyramid surface of the cube-corner indenter and the sample surface. Meanwhile, for different maximum penetration loads and different loading rates, the adhesion behavior is also not observed in Fig. 1(a), indicating that the maximum penetration load and the loading rate don’t affect occurrence of the adhesion behavior of the Zr-based BMG when using the Berkovich indenter. On the contrary, for different maximum penetration loads and different loading rates, the adhesion behavior is observed for all curves obtained by the cube-corner indenter as shown in Fig. 1(b). In previous research, this difference is rarely reported and studied because the Berkovich indenter is the most commonly used indenter.

In addition, from Figs. 1(a) and 1(b), another difference can also be observed that the loading portions of the indentation curves obtained by the Berkovich indenter are relatively smooth but there are obvious fluctuations in the loading portions of the indentation curves obtained by the cube-corner indenter. Fluctuations in the loading portions actually indicate occurrence of serrated flows, which have be
widely observed in previous research.\textsuperscript{22}) In order to highlight this difference, the loading portions of indentation curves obtained by the Berkovich indenter and the cube-corner indenter with the same maximum penetration load of 148 mN are given in Fig. 2(a). Meanwhile, Figs. 2(b) and 2(c) illustrate local enlarged views of Fig. 2(a) in the range of 120–148 mN, illustrating the indenter dependent serrated flow behavior of the Zr-based bulk metallic glass in detail.

From the indentation curves shown in Fig. 1(b), we can roughly see that the adhesion force \( f \) increases with the increasing of the maximum penetration load. Considering that the loading rate is also changed, this affecting law may be questioned. In order to better study influences of indentation parameters such as the loading rate, the maximum penetration load and the cyclic loading number, on the adhesion force \( f \) between the indenter and the BMG, the experiment method with a single variable was used. Considering that the maximum penetration load of the nanoindentation device in the Ref. \textsuperscript{21}) is only 500 mN, the load sensor was changed to be a new one with a larger measuring range of 2.5 N for studying effects of indentation parameters in wider ranges. Then, indentation experiments with different loading rates, maximum penetration loads and cyclic loading numbers were carried out, and experimental results are summarized in Fig. 3. From Fig. 3, we can see that the adhesion force \( f \) is dependent on the indentation parameters. In Figs. 3(a) and 3(b), the adhesion force has the decreasing trend with the increasing of the loading rate but it increases with the increasing of the maximum penetration load. In Fig. 3(c), the adhesion force for the first indentation is relatively large and it obviously decreases with the increasing of the cyclic loading number.

In order to understand what has happened when the Berkovich indenter and the cube-corner indenter contact with the sample surface and reveal reasons leading to the difference between Figs. 1(a) and 1(b) at the points that indenters are departing from the sample surface, residual morphologies of the Zr-based BMG after indentation tests were measured and results are shown in Fig. 4. In Fig. 4(a), only a few discrete shear bands appear around the indent, and pyramidal surfaces formed by the Berkovich indenter are relatively smooth and not distinctly different from the initial surface. Being very different from the Berkovich indent, many concentric shear bands appear around the cube-corner indent, and relatively “dirty” but new surfaces are formed by the cube-corner indenter. What’s more, new phenomena as shown in Fig. 5 are obtained when high magnification was used to observe local regions of residual indents. Interestingly, some discrete shear bands appear on the pyramidal surfaces formed by the Berkovich indenter in Fig. 4(a) but they are not obvious like that around the indent. In Fig. 5(b), the nano-scale dimple structure is obviously observed on the pyramidal surfaces formed by the cube-corner indenter. The
reason for absence of the nano-scale dimple structure in some regions in Fig. 5(b) is that the cube-corner indenter takes them away when it is departing from the sample surface. The dimple structure and the river or vein pattern have been observed on fracture surfaces of BMGs during tensile tests,\textsuperscript{26)} compressive tests,\textsuperscript{10,27,28)} and three-point bending tests,\textsuperscript{13–15)} but they are rarely reported during indentation tests because the Berkovich indenter is the most commonly used indenter for indentation tests which usually leads to relatively smooth residual surfaces as shown in Fig. 4(a) even under a relatively large maximum penetration load of about 1 N. On the contrary, the obvious dimple structure is formed as shown in Fig. 6 using the cube-corner indenter even under a very low maximum penetration load of 50 mN.

4. Discussion

Even with very different testing principles, the dimple structure formed during the tensile tests, compressive tests, three-point bending tests and indentation tests may have the
similar formation mechanism related to shear-induced softening and local temperature rise, but initiation, formation and propagation of shear bands may be different during indentation tests which are confined in very localized regions.

The only difference between the Berkovich indenter and the cube-corner indenter is that they have different face angles, 65.3° for the Berkovich indenter and 35.3° for the cube-corner indenter. According to this difference, possible reasons leading to the difference between Figs. 4(a) and 4(b) are given as follows.

Based on Sneddon’s solutions,29) the contact pressure distribution \( P_r \) within a semi-infinite half-space loaded by a rigid conical indenter during elastic contact is given as

\[
P_r = \frac{E}{2(1 - \nu^2)} \frac{\cosh^{-1}(a/r)}{\tan \alpha}, \quad 0 \leq r \leq a
\]  

(1)

where \( E \) is Young’s modulus, \( \nu \) is Poisson’s ratio, \( a \) is the contact radius, \( r \) is the radius distance measured from the axis of the conical indenter and \( \alpha \) is the equivalent cone semi-angle, 70.3° for the Berkovich indenter and 42.3° for the cube-corner indenter. Equation (1) indicates that during elastic contact, the contact pressure distribution \( P_r \) is dependent on indenter geometry. For the same contact radius and radius distance, higher pressure is produced under the cube-corner indenter and it’s about three times the pressure under the Berkovich indenter. For the same pressure, the radius distance \( r \) can extend farther from the axis for the cube-corner indenter.

On the other hand, according to the slip-line theory,30) the maximum value of shear stress \( \tau_{max} \) which plays an important role in plastic deformation and yielding can be given as

\[
\tau_{max} = \frac{P_m}{2(1 + \nu)}
\]  

(2)

where \( P_m \) is the mean contact pressure. This equation suggests that for the same mean contact pressure, the smaller cone semi-angle leads to the higher maximum value of shear stress.

By combination of Eqs. (1) and (2), higher contact pressure, larger distribution regions and higher shear stress can be obtained by the cube-corner indenter because of its smaller cone semi-angle. So, for a given critical stress required for shear bands formation and propagation, the possibility that the shear bands can escape from under the contact to the free surface is greater for the cube-corner indenter. However, because of relatively low stress induced by the Berkovich indenter, many shear bands can not escape from under the contact to the free surface, and shear band propagation is inhibited by the surrounding low pressure regions. These may be reasons leading to the difference of shear band distribution between Figs. 4(a) and 4(b).

With regard to the nano-scale dimple structure illustrated in Fig. 5(b), it may be formed by melting and self-assembling of the material in the contact region because of the high pressure mentioned above and high temperature under the cube-corner indenter.16,31) Local heating during shear band operation31) and strong interfacial friction between the contact surfaces may be main reasons leading to local temperature rise. Shaper indenters with small face angles such as the cube-corner indenter induce high pressure and shear stress under the indenters. When the maximum value of shear stress \( \tau_{max} \) reaches the critical value, plastic deformation occurs. Shear bands form, extend and propagate to accommodate the high localized strain. According to the theory deduced by Lewandowski and Greer,31) remarkable
temperature excursions occur during shear band operation and they are more violent at high strain rates. When the elevated temperature exceeds the melting temperature of the BMG, materials in the contact regions will melt. During processes that the cube-corner indenter penetrates into and withdraws from the specimen surface, strong interfacial friction between the contact surfaces may also contribute to the temperature rise especially after melting of materials.32]

5. Conclusions

In summary, we study effects of indenter geometry on indentation behaviors of the Zr-based bulk metallic glass. An interesting phenomenon that the adhesion behavior is obviously observed during the indentation test using the cube-corner indenter but it’s not observed when using the Berkovich indenter. Corresponding to the adhesion behavior, “dirty” but new surfaces with the dimple structure are formed by the cube-corner indenter while relatively smooth surfaces with few discrete shear bands are obtained by the Berkovich indenter. Via further experiments, it is obtained that indentation parameters, such as the loading rate, the maximum penetration load and the cyclic loading number, affect the adhesion force. With the increasing of the loading rate and the cyclic loading number, the adhesion force decreases. On the contrary, the adhesion force increases with the increasing of the maximum penetration load.

In addition, by comparing the indentation curves obtained by the Berkovich indenter and the cube-corner indenter, the conclusion can be obtained that the cube-corner indenter with the small face angle promotes more obvious serrate flows than the Berkovich indenter.

The possible reasons leading to the difference mentioned above were discussed. The cube-corner indenter with the relatively small cone semi-angle leads to relatively high localized pressure and strain, promoting formation of shear bands which may further lead to remarkable temperature excursions because of shear band operation and strong interfacial friction.

Acknowledgments

This research is funded by the National Natural Science Foundation of China (Grant No. 51275198), Special Projects for Development of National Major Scientific Instruments and Equipments (Grant No. 2012YQ030075), National Hi-tech Research and Development Program of China (863 Program) (Grant No. 2012AA041206), and Program for New Century Excellent Talents in University of Ministry of Education of China (Grant No. NCET-12-0238). We also give our thanks to the Research Group of Prof. Jianzhong Jiang in Zhejiang University for providing the specimen.

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