Serrated Plastic Flow in a Zr-based Bulk Metallic Glass During Nanoindentation *

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We investigate plastic deformation of $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ bulk metallic glass using depth sensing nanoindentation. Numerous serrations in the load–displacement curves during indentation, shear bands and pile-ups around the indent were observed. The results revealed that the serrated plastic flow behaviour in this alloy depends strongly on the indentation strain rate.

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Recently, bulk metallic glasses (BMGs) have attracted great interest due to their unique physical, mechanical, and chemical properties.[1–8] However, BMGs loaded under unconstrained conditions usually fail catastrophically with little global plasticity. This deformation behaviour has limited the applications of BMGs as engineering materials. Therefore, considerable efforts have been made to understand the plastic deformation behaviour during the past decades. The plastic deformation of bulk metallic glasses (BMGs) is fundamentally different from that in crystalline solids, because of the lack of long-range order in the atomic structure. At low temperature (well below the glass transition), plastic deformation of BMGs is highly localized into thin shear bands.[9] Mechanistically, these shear bands are self-organized assemblies of smaller units of plasticity, e.g., volume elements of material containing 30–50 atoms that individually undergo local shear transformation. The operation of shear bands in the atomic scale or nanoscale gives rise to several peculiar mechanical properties at the macroscopic scale. Typically, in indentation or compression, plastic flow of BMGs is usually serrated with many small displacement bursts or load drops.[10] This serrated flow phenomenon is not unique to BMGs. In crystalline materials, it can be observed as the Portevin–Le Chatelier (PLC) effect. The physical basis for appearance of the PLC effect is a negative strain rate sensitivity, which originates from dynamic strain aging (DSA) caused by the interaction of moving dislocations with mobile impure atoms.[11] In contrast, the origin of serrated flow in BMGs has not yet been clarified.

When studying the process of shear bands and serrated flow instability as well as their physical nature at atomic scale or nanoscale, specific methods and devices with high spatial and time resolution are required. In this connection, the depth sensing nanoindentation technique has been proven to be a powerful tool and is increasingly being used to probe the mechanical properties of highly localized regions in materials.[12–17] During indentation, the depth of a rigid indenter tip is monitored as it is pressed into a tested material. The resulting load–displacement response reflects elastic and/or plastic deformation. Recent investigations have demonstrated that depth sensing nanoindentation can be used to probe the serrated plastic flow behaviour in BMGs.[18–22] However, the precise nature of serrated plastic flow and its correlation with the formation and development of shear bands in BMGs still remains unclear. In this Letter, we extend the use of the depth sensing nanoindentation technique to study the effect of the indentation strain rate on the serrated flow behaviour of a Be-bearing Zr-based BMG, and a mechanistic explanation for the unique experimental observations will be presented as well.

$Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ bulk metallic glass was produced by arc melting the pure elements together under a purified Ar atmosphere in ingots of the desired composition. Each ingot was then re-melted twice to ensure a homogeneous composition. Then 8-mm diameter rods of metallic glass were made by suction casting the molten alloy into a copper mould. The cylinders thus obtained were confirmed to be non-crystalline by conventional x-ray diffraction. Prior to indentation, the specimen was polished electrolytically using a methanol solution containing 33% volume fraction HNO_3. Indentation tests were performed using an MTS Nanoindenter XP system (Oak Ridge, TN)

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operated in the continuous stiffness mode. The system has load and displacement resolutions of 50 nN and 0.1 nm, respectively. The indentations were made in a constant strain rate mode (the indentation strain rate is defined as $\dot{\varepsilon} = (1/h)dh/dt$, where h is the indentation displacement). To investigate the effect of the strain rate on the serrated plastic flow, we carried out four strain rate levels of indentations, namely 0.02, 0.05, 0.1, and $0.2\,\mathrm{s}^{-1}$. For all indentations, a diamond Berkovich indenter was used and care was taken to ensure that the thermal drift of the instrument was maintained below 0.05 nm/s. Microscopic observations on the indents were conducted using an optical microscope (Neophot21).

The loading segments of the load–displacement (P–h) curves for typical indentations for four different strain rates are shown in Fig. 1. Serrated plastic flow was observed at all strain rate levels involved in the present study, but became less prominent as the strain rate was increased. This observation is similar to that for Be-free Zr-based bulk metallic glasses[21] and Al-based glass.[22]

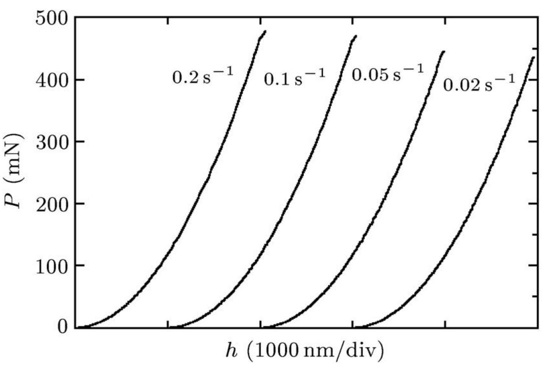

Fig. 1. Load versus displacement at various strain rates.

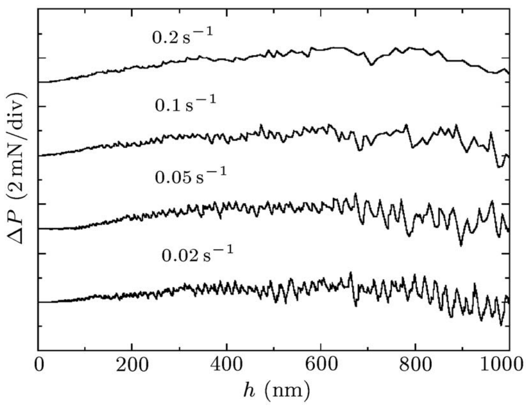

Fig. 2. Effect of indentation strain rate on the load deviation.

To demonstrate further characteristics of this serrated flow, these measured P–h curves, as shown in Fig. 1, were fitted with smooth power law functions

$$P_{\mathrm{fit}} = Ch^m, \tag{1}$$

where C and m are the loading rate-dependent fitting constants. For a given penetration depth h, the corresponding loads p_{fit} can be determined using Eq. (1), whereas the corresponding experimental values of loads, p_{exp}, can be obtained from the curves illustrated in Fig. 1. Obviously, the amount $\Delta P = P_{\mathrm{exp}} - P_{\mathrm{fit}}$ can characterize the deviation of the load from that necessary for keeping the global behaviour. The $\Delta P - h$ curves at different strain rates are shown in Fig. 2. It can be seen that the oscillation frequency of the load deviation ΔP is higher at lower strain rate than that at higher strain rates, whereas the oscillation magnitude is insensitive to the indentation strain rate.

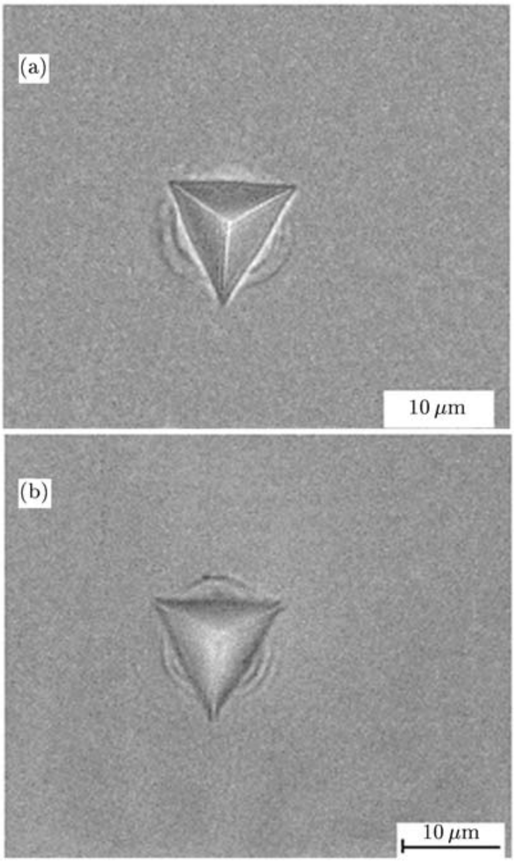

Fig. 3. Optical micrographs of indentation patterns formed at strain rates of (a) $0.02\,\mathrm{s}^{-1}$ and (b) $0.2\,\mathrm{s}^{-1}$.

A number of incomplete circular patterns of shear bands were observed in the piled up area around the indents. The typical morphologies of the indentation patterns produced at the highest and lowest strain rates are shown in Fig. 3. It can be seen from this figure that the number of shear bands is smaller and surface step size is larger outside the indent formed at low strain rate, while the number of shear bands is larger and the step size is smaller at high strain rate. This result is consistent with the observations made by Jiang and Atzmon for an Al-based metallic glass.[22]

Serrated plastic flow behaviour is usually observed in crystalline materials. There it is ascribed to non-continuum events, such as defect nucleation. Since shear bands are the only observable dominant feature during the inhomogeneous plastic deformation of BMGs, serrated flow in BMGs is surely related to the formation of shear bands. Physically, shear banding is a rate dependent process. The indentation strain rate affects the number of shear bands formed around the indent, as observed in Fig. 3, and consequently affects the serrated flow behaviour of BMGs. It can be seen from Figs. 1–3 that high strain rate results in a larger number of shear bands, whereas serrated flow is suppressed at high strain rates. Such an observation is also consistent with the previously study reported for Pd-based bulk metallic glass by Mukai *et al.*,[23] who observed that tensile failure at low strain rates occurs along a single shear band, whereas many intersecting shear bands were involved in fracture at high strain rates. The indentation studies on other BMGs,[21,22] not the Be-bearing Zr-based bulk metallic glass studied here, presented similar observations as described above. They demonstrated that at low strain rates single, isolated shear bands operate, whereas at high strain rates there are many shear bands operating within the specimen at the same instant. If the number of shear bands is sufficiently high, the contribution of an individual shear band to the total strain is indiscernible and, consequently, fewer serrations in P–h or $\Delta P - h$ curves can be observed. Obviously, our current study on the Be-bearing Zr-based bulk metallic glass supports this explanation.

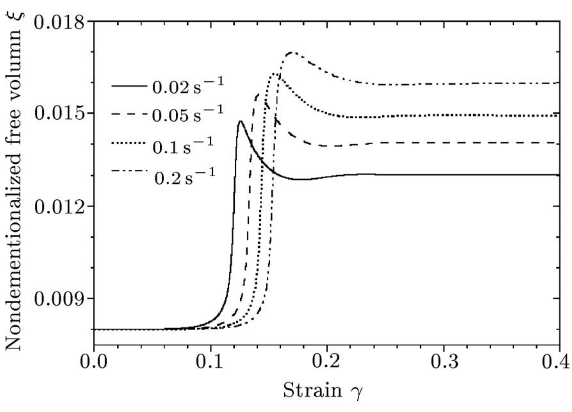

Fig. 4. Non-dimensional free volume concentration versus strain under different strain rates.

The important question now is about the reason why the number of shear bands formed in BMGs at high strain rate is higher than that at low strain rate. Up to now, this question still remains unclear. According to the free volume theory,[24,25] the formation of shear bands in metallic glasses is mainly due to the creation and coalescence of free volume in some local regions, whereas the creation and diffusion of free volume in metallic glasses are affected greatly by strain rate. According to Spaepen's microscopic plastic flow equation of metallic glasses,[24] the effect of strain rate on the nondimensional free volume concentration was investigated. The corresponding calculated results are shown in Fig. 4. It can be seen from this figure that the free volume concentration is higher at higher strain rates than that at lower strain rates. Hence, high free volume concentration at higher strain rates will induce a relatively larger number of shear bands in BMGs in comparison with the lower strain rate cases.

In summary, the depth sensing nanoindentation technique was used to probe the details of serrated plastic flow behaviour of $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ bulk metallic glass. The pattern of shear bands around the indent and its connection with the serrated flow behaviour have been examined by optical microscopy. The results demonstrate that the indentation strain rate has a significant influence on the serrated plastic flow behaviour and the formation of shear bands around the indent.

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