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Effect of the nanoindentation rate on the shear band formation in an Au-based bulk metallic glass

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Abstract

This study investigated the nanoindentation behavior of $Au_{49}Ag_{5.5}Pd_{2.3}Cu_{26.9}Si_{16.3}$ bulk metallic glass samples at loading rates ranging from 0.03 to 300 mN s^{-1}. Notable shear band pop-in events were observed. The pop-in size was observed to increase linearly with the load and decreased exponentially with the strain rate. A free-volume mechanism was proposed for interpreting these observations quantitatively. The results and analyses also shed light on the shear band nucleation and evolution processes in bulk metallic glasses.

Keywords: Metallic glasses; Plastic deformation; Hardness; Deformation inhomogeneities

1. Introduction

Bulk metallic glasses have aroused extensive interest in recent years because of their superior structural properties, such as ultra-high strength, a large elastic limit and high wear resistance [1–6]. The lack of line and planar defects in their amorphous structure, such as dislocations and grain boundaries, results in bulk metallic glasses with superior strength over their crystalline counterparts. However, their high strength is also accompanied by brittleness caused by highly localized shear band deformation, which greatly limits the applications of bulk metallic glasses.

Several recent efforts have been made at studying their shear band evolution patterns after monotonic loading experiments [7,8]. It was observed that the shear band size and density varied with the applied strain rate. However, in a deformed bulk metallic glass, the region of deformed shear bands is expected to be softer than that of non-deformed shear bands. As a result, it is possible for shear bands to initiate repeatedly from already weakened locations during these experiments. This phenomenon complicates postmortem micro-structural analysis, because a shear band observed under scanning electron microscopy/transmission electron microscopy may be a result of multiple shear band initiations at the same location. Thus, the size and density of the observed shear bands could not be directly correlated with shear band nucleation and evolution patterns.

Some state-of-the-art in situ characterization methods, e.g. nanoindentation and thermographic detection, have been used for probing the time and spatial arrangements of shear bands during the deformation of bulk metallic glasses [9–13]. The excellent time and displacement resolution of a nanoindenter, for instance, has been demonstrated in the analysis of shear band evolution [11–13]. Schuh et al. [11,12] recently studied the shear band pop-in phenomenon under different load, strain rate and temperature conditions using a Triboindenter®. They also successfully applied the free-volume theory for explaining the observed shear band evolution patterns. However, it is noted that the fruitful amount of pop-in data obtained from nanoindentation tests has not been fully analyzed. Thus, the goal of this paper was

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to extend the previous analyses of the pop-in size and distribution under various loads and strain rates. The analyses were then used for disclosing the nature of free-volume evolution, which controls the nucleation and propagation of localized shear bands.

2. Experimental methods

The bulk metallic glass material used in the present study was $Au_{49}Ag_{5.5}Pd_{2.3}Cu_{26.9}Si_{16.3}$, which was recently developed by Schroers et al. [14]. Alloy ingots of 3 mm in diameter were prepared by arc melting a mixture of elements with purities of >99.8%. X-ray diffraction and differential scanning calorimetry measurements were subsequently performed in order to confirm the amorphous nature of these samples and a glass transition temperature (T_g) of 400 K. The ingots were sliced into coupons with a thickness of 1 mm for nanoindentation experiments. The surfaces of these coupons were polished to a final grit size of 0.03 μm using diamond powders prior to the nanoindentation experiments.

Instrumented nanoindentation experiments were conducted using a Triboindenter® (Hysitron, Inc., Minneapolis, MN). The indenter has a high data acquisition rate of up to 10^4 s^{-1}. The experiments were performed under load control with a maximum load of 5 mN. Loading rates of 0.03, 0.1, 0.3, 1, 3, 10, 30, 100 and 300 mN s^{-1} were applied.

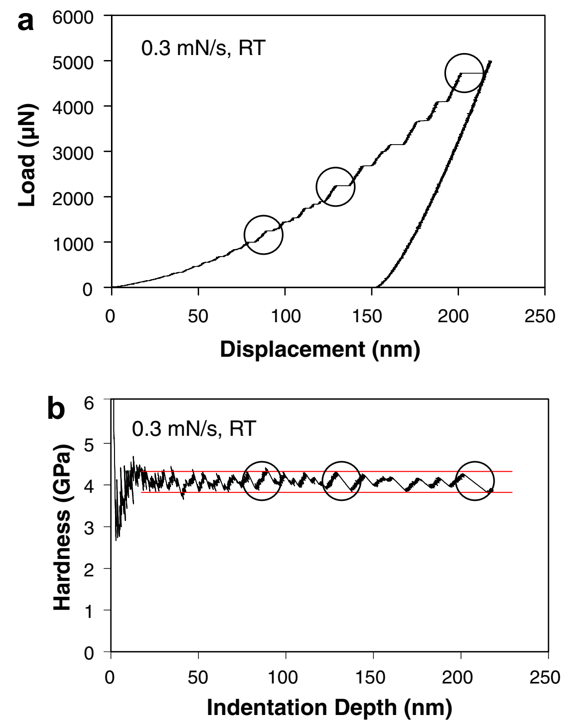

Fig. 1. Observed pop-in events during nanoindentation of an Au-based bulk metallic glass at a constant loading rate of 0.3 mN s^{-1} at room temperature. (a) *P–h* curve and (b) projected curve of the hardness versus indentation depth.

3. Results and discussion

3.1. Pop-in size versus the indentation depth

The *P–h* curve of Au-bulk metallic glass tested under nanoindentation at a loading rate of 0.3 mN s^{-1} at room temperature is plotted in Fig. 1a. A notable pop-in phenomenon was clearly observed. These pop-in steps represented instantaneous shear band bursts and have been previously observed in many other bulk metallic glass systems under nanoindentation [11–13,15–17]. This pop-in phenomenon has been characterized by its size (displacement increment), initiation depth, strain rate and temperature [11–13,17]. One of the distinct observations was that the pop-in size increased with the indentation depth, while the number of pop-in events decreased [11–13]. This was also observed in the present Au-bulk metallic glass, as shown in Fig. 1a. Intuitively, this observation appears to be "a natural consequence of the increasing length scale of the indentation geometry as the depth increases" [12]. However, a physical meaning of this scaling phenomenon has not been offered.

Greer et al. [13] pointed out that the elastic strain increment ($\delta h_e/h$) between two consecutive pop-in events was approximately a constant during nanoindentation of a bulk metallic glass. Here, δh_e is the elastic displacement increment between two pop-in events and h is the indentation depth. Since the elastic strain is proportional to the elastic stress, the elastic stress increment between each two consecutive pop-in events should also be a constant. Furthermore, since a bulk metallic glass behaves like a perfectly plastic material, the yielding strength (or hardness) does not change with the depth and, thus, this stress increment must be balanced out by the same amount of stress drop during a pop-in event. This indicates that all pop-in events were terminated by a similar amount of stress reduction during nanoindentation. This stress reduction is apparently caused by a free-volume increase inside an active shear band. When a shear band is activated, a part of the applied mechanical energy is consumed for creating defects and the free volume inside the shear band. According to the free-volume theory [18–20], a lower threshold stress than the activation stress would be needed for propagating the shear band further and this results in softening of the shear band [7,21]. The amount of stress reduction should follow the statistical distribution of the free volume in shear bands, which is determined by the applied stress, not the load.

In order to verify this implication, the data in Fig. 1a were re-plotted as the hardness versus indentation depth in Fig. 1b. Note that the hardness is proportional to the stress. Here the hardness H equals P/A, where P is the applied load and A is the indentation area calculated by the area function of the indenter tip [22]. The pop-in steps in Fig. 1a are projected into hardness serrations in Fig. 1b. The hardness reduction (Fig. 1b) at each serration corresponds to a pop-in event (Fig. 1a) and the hardness increase (Fig. 1b) at each serration corresponds to the elastic portion between two pop-in events (Fig. 1a). It is