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Published in:
Defect and Diffusion Forum

DOI:
10.4028/www.scientific.net/DDF.368.3

Defect and Diffusion Forum
ISSN: 1662-9507, Vol. 368, pp 3-6
doi:10.4028/www.scientific.net/DDF.368.3
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Submitted: 2015-11-11
Revised: 2016-02-10
Accepted: 2016-03-18
Online: 2016-07-05

Serrated plastic flow of various metallic glasses during nanoindentation

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Keywords: metallic glasses, nanoindentation, pop-ins

Abstract. Nanoindentation experiments were executed on amorphous metallic ribbons made of $Fe_{40}Ni_{40}B_{20}$, $Cu_{47}Ti_{35}Zr_{11}Ni_6Si_1$ and $Zr_{65}Cu_{17.5}Ni_{10}Al_{7.5}$ that differ in microhardness and glass forming ability. The individual serrated plastic flow events were analyzed in a wide range of the loading rates. In the individual pop-in events of the load-displacement (*P-h*) curve the contributions of plastic deformation (Δh_{pl}) were calculated depending on the loading rate and the alloy composition. It is concluded that the contribution of the serrated plastic deformation flow varies with the composition of the alloy. The highest plastic deformation for the individual pop-ins was observed for Zr-based metallic glasses.

Introduction

Metallic glasses exhibit unique properties and their combination, including the high strength and high hardness, makes them attractive for practical applications. The deformation mechanism of amorphous materials is different from the crystalline counterparts due to inhomogeneous plastic deformation that is highly localized into narrow shear bands [1, 2].

Discontinuities in the load-displacement (*P-h*) curves are produced during load rate-controlled nanoindentation and correlated with the discrete shear banding events [3]. Wright et al. [2] and Golovin et al. [4] found that the onset of the plasticity occurred at a discrete displacement burst (commonly termed “pop-in” or “serration”) during nanoindentation of Zr-based bulk metallic glasses. Serrated flow is characterized by repeating cycles of stress drop during displacement rate-controlled experiments followed by elastic reloading without an increase in the load [5]. It is generally accepted that the development of shear transformation zones leads to the deformation localized into shear bands and therefore to inhomogeneous flow in metallic glasses [1]. The inhomogeneous nature of the deformation in metallic glasses is reflected in the serrated plastic flow with the complex topology. Jang et al. [6] and Bei et al. [7] observed that the number of pop-ins correlates directly with the number of shear bands observed on the surface of the sample around the indent. The displacement discontinuities increase in the size with increasing load or displacement. The origin of serrated flow in metallic glasses is still unclear but, its role in the formation of shear bands is generally accepted.

In this work we focus on the serrated flow of plastic deformation and the differences in the discontinuities in the *P-h* curves during indentation for FeNiB, CuTiZrNiSi and ZrCuNiAl types of metallic glasses that have the different glass forming ability over a wide range of loading rates.

Experimental materials and methods

The samples of the amorphous metallic ribbons with the nominal compositions of $Fe_{40}Ni_{40}B_{20}$, $Cu_{47}Ti_{35}Zr_{11}Ni_6Si_1$ and $Zr_{65}Cu_{17.5}Ni_{10}Al_{7.5}$ (at. %) were used for the nanoindentation experiment. The ribbons with the cross-sections of 10 mm × 0.018 mm, 1.72 mm × 0.02 mm and 1.13 mm × 0.01 mm, respectively, were prepared by rapid melt quenching on a spinning metallic disc.

Before nanoindentation experiment the specimens were mechanically polished to mirror finish and tested using nanoindentation equipment MTS NanoIndenter® XP with a cube corner diamond tip. For the tip calibration procedure fused silica was used. The indentation measurements were performed at room temperature in the load rate-control mode up to the maximal load of 250 mN using five loading rates from 0.05, 0.1, 1, 10, and 100 $mN.s^{-1}$ followed by holding for 1s and unloading. The high data acquisition rate up to the 100 Hz was chosen for resolving rapid dynamic events. For each measurement up to twenty-five indents were made. After nanoindentation, the morphology of the indents and the shear bands of the ribbons was observed by XL30S SEM-FEG scanning electron microscope (SEM).

Results

Load-displacement (*P-h*) curves during indentation with loading rates *dP/dt* ranging from 0.05 to 100 $mN.s^{-1}$ in all studied alloys are presented in Fig. 1. It is evident that Fe- and Cu-based metallic

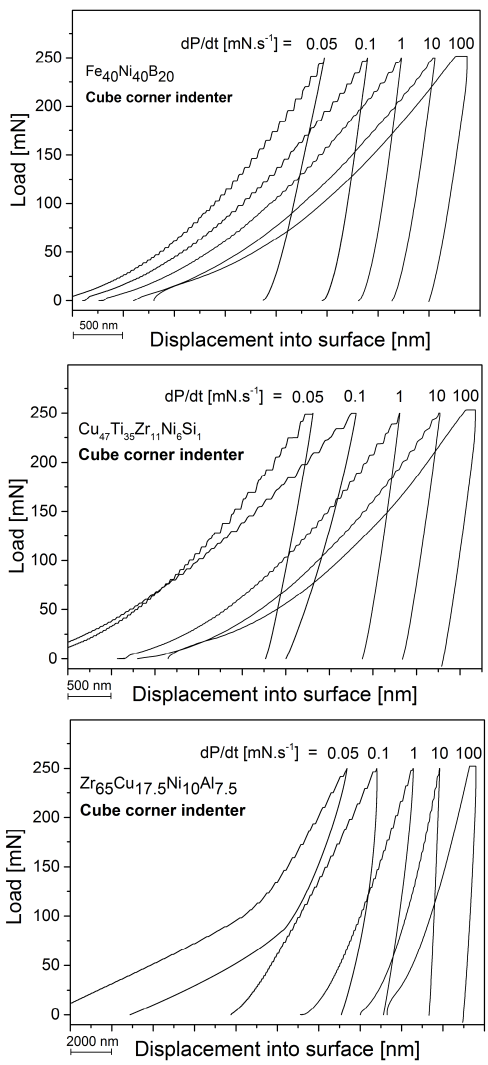

Fig. 1 *P-h* curves during indentation for all observed alloys.

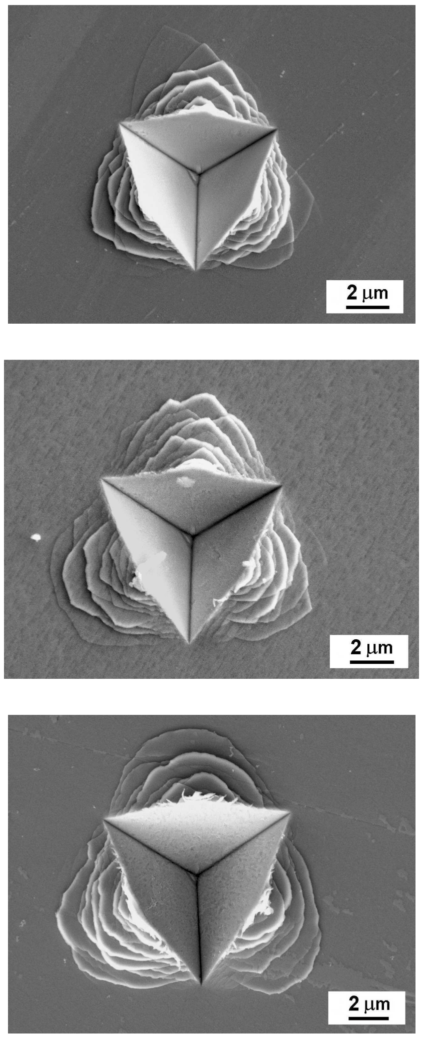

Fig. 2 Indent morphologies at the rate of 0.1 $mN.s^{-1}$ for all three alloys.

glass ribbons have similar shapes of indentation curves for all loading rates. In Zr-based metallic glass ribbon the shape of indentation curves changes with increasing loading rate. For all alloy compositions the smooth line of indentation curves becomes zig-zag one. It is clear visible that at lower loading rates (from 0.05 to 1 mN.s^{-1}) the pop-in events are more pronounced and gradually disappear with increasing loading rate. These discontinuities in the loading curves were analyzed in detail. The concept of discrete plasticity ratio [6-11] was used for the separation of the elastic, homogeneous and inhomogeneous plastic deformation contribution from the total instantaneous deformation for the individual pop-ins.

Typical surface morphology of indented area after nanoindentation was observed by scanning electron microscopy. Figure 2 shows the localized plastic flow around indents on the surface of all alloys for the loading rate of 0.1 mN.s^{-1}. The indents and the deformed volumes with shear bands are relatively large due to the used indenter tip form and the maximum used load of 250 mN. Only a small influence of the sample compositions on the final shape of indent area was observed. Similar pictures were obtained for all applied loading rates. No principal differences in the shear band density or shear pattern were found in the whole interval of the loading rates. We conclude that with increasing loading rate the disappearance of pop-ins is not directly related to the changes in shear band morphology in the plastic zone around the indent.

The origin of serrated flow of inhomogeneous deformation in metallic glasses is still unclear but, it is certainly connected with the formation of shear bands. The serrated flow on *P-h* curves can be also considered for the transition from perfectly elastic behavior to plastic deformation which was also observed by Bei et al. [7]. We calculated the plastic deformation contributions (Δh_{pl}) for individual pop-in events depending on the indentation depth for the loading rates of 0.05 and 0.1 mN.s^{-1}. In Fig. 3 there are these dependences for all examined sample compositions.

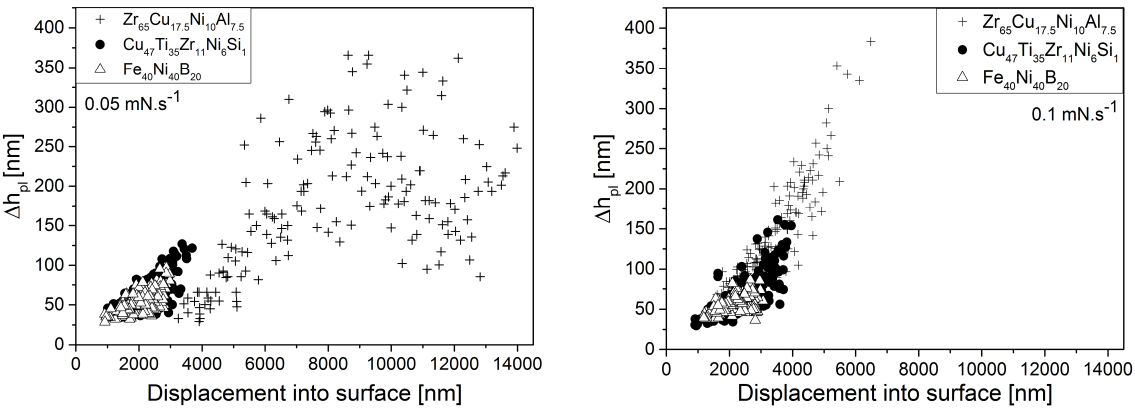

Fig. 3 The plastic deformation contribution at elemental pop-in event for indicated alloys at loading rate 0.05 mN.s^{-1} (left) and 0.1 mN.s^{-1} (right).

The plastic deformation contribution observed for Zr-based amorphous alloy was higher than for Fe- and Cu-based amorphous alloys. As the loading rate increases, the instantaneous plastic deformation occurs at lower indentation depths. This influence is more significant for Zr-type glass in the comparison to Fe and Cu types. The differences in the deformation behaviour of examined metallic glasses can be attributed to specific distribution of activation energy in the alloys with different chemical composition [12].

Summary

Nanoindentation experiments over a wide range of deformation rates reveal the presence of the deformation discontinuities on all three types of amorphous alloys ($Fe_{40}Ni_{40}B_{20}$, $Cu_{47}Ti_{35}Zr_{11}Ni_6Si_1$ and $Zr_{65}Cu_{17.5}Ni_{10}Al_{7.5}$) at loading rates up to 10 mN.s^{-1}. These discontinuities on the *P-h* curves start at the load up to 50 mN. As the indent penetrates deeper, the discontinuities are more pronounced. These discontinuities vanish for the loading rates of above 10 $mN.s^{-1}$. More developed discontinuities are created for the materials with lower microhardness and so lower strength (Cu- and Fe-based). The deformation discontinuities were analyzed using discrete plasticity ratio concept. This ratio shows comparable values for all examined alloy compositions. The observation of indent region morphology does not show a simple correlation between the presence of pop-ins and the shear band morphology.

Acknowledgment

This work was supported by Slovak Academy of Sciences- grant VEGA 2/0045/14.

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