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Nanoindentation Study of the Influence of the Loading Rate on the Deformation of Metallic Glasses

Mária Huráková[1,a,*], Kornel Csach[1,b], Alena Juríková[1,c], Jozef Miškuf[1,d], Štefan Demčák[2,e], Václav Ocelík[3,f], Jeff Th.M. De Hosson[3,g]

[1] Institute of Experimental Physics, Slovak Academy of Science, Watsonova 47, 040 01 Košice, Slovakia

[2] Department of Environmental Engineering, Faculty of Civil Engineering, Technical University of Košice, Vysokoškolská 4, 040 01 Košice, Slovakia

[3] Department of Applied Physics, Faculty of Mathematics and Natural Sciences, University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands

[a,*]hurakova@saske.sk, [b]csach@saske.sk, [c]akasard@saske.sk, [d]miskuf@saske.sk, [e]stefan.demcak@tuke.sk, [f]v.ocelik@rug.nl, [g]j.t.m.de.hosson@rug.nl

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Abstract. Nanoindentation experiments at the loading rates from 0.05 to 100 mN.s^{-1} on the amorphous FeNiB alloy were executed. We found that the serrations in the load-displacement (*P-h*) curve are more pronounced at lower loading rates and gradually disappear upon increasing loading rate. We have estimated the contribution of the inhomogeneous plastic deformation from pop-in events on the *P-h* curves. The pop-in population was compared with the morphology of indents.

Introduction

Plastic deformation of amorphous metallic materials is fundamentally different from that in crystalline solids, due to the lack of long-range order in the atomic structure. Dislocations and defects carry plastic strain in crystalline solids while in amorphous alloys inhomogeneous plastic deformation is highly localized into narrow shear bands. These alloys exhibit several unique deformation phenomena due to the dominance of shear banding at low temperature plasticity [1-4].

Recently nanoindentation experiments have provided insights into mechanisms of metallic glass deformation [5, 6]. The displacement bursts in the load-displacement (*P-h)* curves produced during load rate-controlled nanoindentation are correlated with the discrete shear banding events [7]. Wright et al. [2] and Golovin et al. [8] observed that the onset of the plasticity during nanoindentation of Zr-based bulk metallic glasses occurred at a discrete displacement burst ("pop-in"). Serrated flow is characterized by repeating cycles of a sudden stress drop during displacement rate-controlled experiments followed by the elastic reloading [9]. It is assumed that the serrations are associated with shear band nucleation and their propagation. The number of pop-ins correlates directly with the number of shear bands observed on the surface of the sample around the indent. Schuh and Nieh [5-7] identified a rate dependence on the nanoindentation response of metallic glasses. They showed that the slow indentations promote a serrated flow, while more rapid indentations can partially or even completely suppress the appearance of such bursts. The serrated flow strongly depends on the loading rate of nanoindentation, the lower loading rates promote more serrations or displacement bursts. In this work we concentrate on inhomogeneous plastic deformation process during nanoindentation of FeNiB type of metallic glass over a wide range of loading rates.

Experimental materials and methods

The samples of amorphous metallic ribbons with nominal composition of $Fe_{40}Ni_{40}B_{20}$ (at. %) were used for the nanoindentation experiments. The ribbons with the cross-sections of 10 mm × 0.018 mm were prepared by rapid melt quenching on a spinning metallic disc.

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The specimens were mechanically polished to mirror finish and tested using nanoindentation equipment MTS NanoIndenter® XP with a cube corner diamond tip. For tip calibration procedure the fused silica was used. The indentation measurements were performed at room temperature in load rate-control mode up to the maximal load of 250 mN using five loading rates from 0.05 to 100 mN.s^{-1}. 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 carried out. After nanoindentation, the morphology of indents and shear bands of the ribbon was observed by XL30 ESEM-FEG scanning electron microscope (SEM).

Results

Localized plastic flow of the deformation in the indent area was observed by scanning electron microscopy. Fig. 1 represents the plastic flow around cube corner indents on the surface of the glass for loading rates 0.1, 10 and 100 mN.s^{-1}. Due to the cube corner indenter tip form and the maximum used loading of 250 mN the indent and the deformed volume with shear bands are relatively large. The size of the indent and the form of plastic deformed area seem to be preserved for all loading rates applied. For the whole interval of the loading rates, no principal differences in the shear band density or shear band pattern were observed.

Load-displacement (*P-h*) curves during nanoindentation with loading rates *dP/dt* ranging from 0.05 to 100 mN.s^{-1} are presented in Fig. 2 (left). The details of the final parts of loading sequence on the *P-h* nanoindentation curves for all loading rates are shown in Fig. 2 (right). It is evident that at lower loading rates (0.05, 0.1 and 1) the pop-in events are more pronounced and gradually disappear with the increasing loading rate.

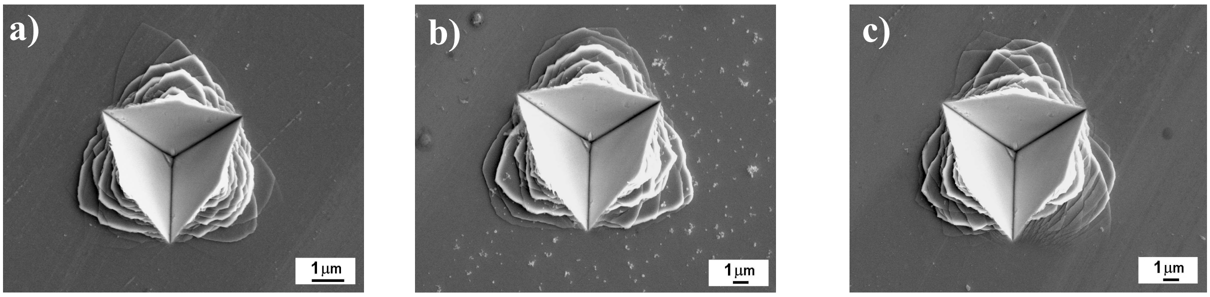

Fig. 1 Morphology of the indents after the nanoindentation with the loading rate of 0.1 mN.s^{-1} (a), 10 mN.s^{-1} (b) and 100 mN.s^{-1} (c).

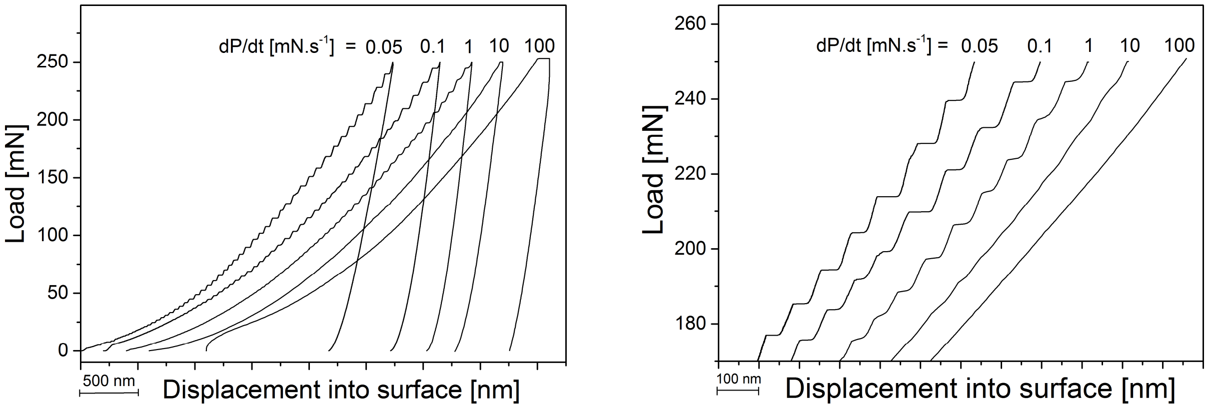

Fig. 2 Load-displacement (*P-h*) nanoindentation curves for various loading rates (left) and detail of these curves (right). Individual *P-h* curves are horizontally shifted for the clarification.

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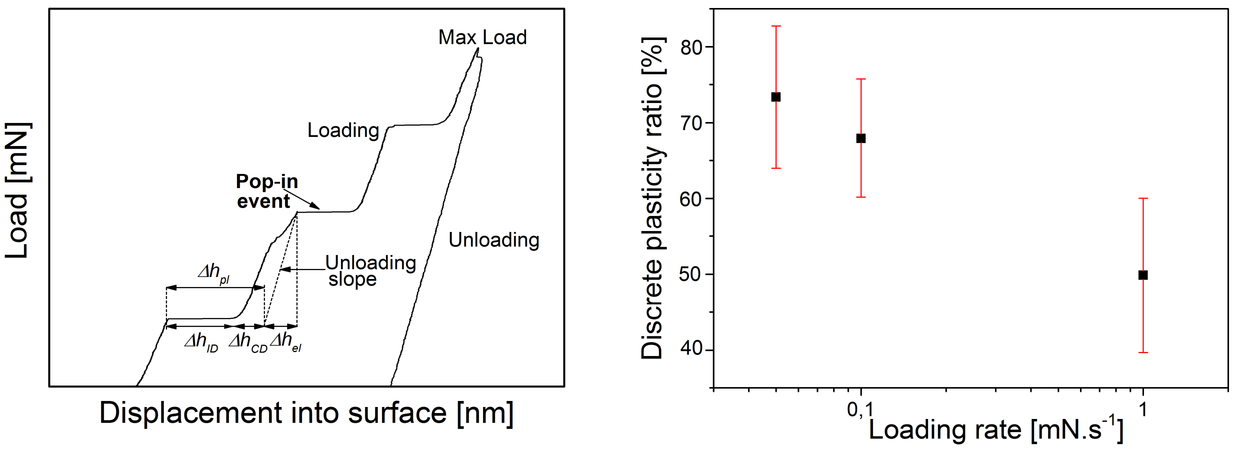

Fig. 3 The scheme of the estimation of the plastic and elastic contribution to the deformation at the individual pop-in event (left) and the discrete plasticity ratio as a function of the nanoindentation loading rate (right).

At lower deformation rates, as the displacement increases, the pop-ins are more developed. At low displacements, the frequency of pop-in occurrence is higher and with increasing the loading the period of individual pop-ins increases. Whereas the loading rate changes more than one order of magnitude (from 0.05 to the 1 mN.s^{-1}), the period of individual pop-ins decreases only slightly.

Estimating the contribution of the pop-ins to the total plastic deformation of the metallic glass is very useful for the study the rate-dependent transition phenomena. For quantitative description of serrated plastic deformation during nanoindentation the parameter called the discrete plasticity ratio is often used [5, 6, 10, 11]. The concept of this parameter is illustrated schematically in Fig. 3 (left). The total displacement recorded during nanoindentation is a superposition of three components: an inhomogeneous part of the plastic deformation $(\Delta \boldsymbol{h_{ID}})$, a continuous part of the plastic deformation $(\Delta \boldsymbol{h_{CD}})$ and an elastic part of the deformation $(\Delta \boldsymbol{h_{el}})$. It is expected that the elastic deformation will be fully recovered during unloading. Then the plastic portion of deformation $(\Delta \boldsymbol{h_{pl}})$ is simply given as a sum of $\Delta h_{ID} + \Delta h_{CD}$. Thus, the contribution of discrete plasticity to the total plasticity (called "discrete plasticity ratio") given as $\Delta h_{ID}/\Delta h_{pl}$ indicates the fraction of plastic deformation that can be attributed to the discrete pop-in events for a given indentation. This parameter is the index of the serration flow which is equal to zero for perfectly homogeneous flow and to the value of one for perfectly discretized deformation [6]. In Fig. 3 (right) the discrete plasticity ratio $\Delta h_{ID}/\Delta h_{pl}$ is plotted as a function of the indentation rate at loading rates of 0.05, 0.1 and 1 mN.s^{-1}. The average value and standard deviation were calculated from 150-180 pop-ins for each loading rate. For loading rates 10 and 100 mN.s^{-1} the pop-ins were not developed to the form suitable for regular quantification of individual pop-ins. For the cube corner indenter tip the discrete plasticity ratio decreases with the increasing the loading rate. The discrete plasticity ratio value of 0.75 at the loading rate of 0.05 mN.s^{-1} decreases by 30 % to the value of about 0.5 at the loading rate of 1 mN.s^{-1}.

It is generally accepted that the development of shear transformation zones leads to localized deformation, shear band 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. The origin of serrated flow in metallic glasses is still unclear but, it is certainly related to the formation of shear bands. We observed that the disappearance of pop-ins with increasing loading rate is not directly connected with the changes in shear band morphology in the plastic zone around the indent.

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Summary

The shape of the indent and its plastic zone does not depend on the loading rate over a wide range of loading rates of the nanoindentation of the metallic glass $Fe_{40}Ni_{40}B_{20}$. Serrated flow is found to be sensitive to the indentation depth and the deformation rate. At lower loading rates (0.05-1 mN.s^{-1}) the discrete deformation is more pronounced and the pop-in events are clearly visible. The increase of the loading rate in one order tends to the decrease the discrete plasticity ratio by the multiplying factor of about 0.7. The visible pop-ins disappear beyond loading rate higher than 1 mN.s^{-1}.

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