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Nanoindentation in Metallic Glasses with Different Plasticity

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Abstract. Nanoindentation and thermomechanical experiments on three types of metallic glasses with different glass forming ability were carried out. The nanoindentation behaviour at room temperature was associated with the creep at elevated temperatures. Different discontinuity populations and their shape observed on the nanoindentation loading curves were compared with morphology of plastically deformed indentation regions. The influence of different thermal stability of studied alloys on the nanoindentation was studied as well.

Introduction

The metallic glasses have been extensively studied for a number of years, but the nature of the plastic deformation mechanisms is still unclear. The instrumented nanoindentation helps in study of their deformation behaviour.

During nanoindentation in metallic glasses the discontinuities or bursts on the load-displacement curves, named as pop-ins, were observed. These pop-ins are connected with the discrete shear banding events [1]. The pop-in events are generally observed at nanoindentation during the loading at higher deformations. In comparison to the other shapes of tips, the cubic corner indentor has the lower tip angle. Therefore at the indentation it can penetrate deeper into the metallic glass and causes higher plastic deformation and the pop-ins are more pronounced. The presence of pop-ins was found to be sensitive to the loading rate. The lower loading rates promote more serrations or displacement bursts [2 - 4].

The combination of glassy nature and metallic bonds makes the deformation mechanisms in metallic glass remarkably different from crystalline metals. Inhomogeneous deformation occurs via the catastrophic event of shear band creation under adiabatic conditions. At micro level the ductile behaviour is saved and the plastic deformation at room temperature is sustained by cooperative shearing of atomic clusters termed shear transformation zones (STZs) [5]. At present a lot of metallic glasses are known with a wide range of specific properties. The non-crystalline structure and lack of longe range order is common for all these metallic materials.

The goal of our experimental work was experimentally observe plastic deformation at room temperature by nanoindentation and relate it with their thermal characteristics.

Experimental materials and methods

For the experiments the amorphous alloy ribbons with the nominal composition of $Zr_{65}Cu_{17.5}Ni_{10}Al_{7.5}$, $Cu_{47}Ti_{35}Zr_{11}Ni_5Si_1$ and $Fe_{40}Ni_{40}B_{20}$ (at. %) prepared by melt spinning method were used. Nanoindentation was performed on the polished samples using MTS NanoIndenter® XP with a cube corner tip. The indentation measurements were performed at room temperature in load-

control mode. After loading up to 250 mN with the rate of 1 mN.s^{-1} the short-time hold (10 s at maximum load of 250 mN) was applied and controlled unloading was followed. After nanoindentation, the morphology of indents and shear bands in their surrounding was observed by scanning electron microscope.

Thermal stability of metallic glasses was examined by DSC calorimetry using TA Q2000 calorimeter at the heating rate of 10 K/min in N2 atmosphere. Thermoplastic behaviour of all alloys was investigated using TA Q400EM thermomechanical analyser.

Results and Discussion

The DSC traces for all used alloys are shown in Fig. 1 (left). On all curves the exothermic peaks indicate the crystallization of the amorphous alloys in one or two stages. At temperatures below the crystallization, in the case of Zr and Cu based alloys, a continuous weak decrease on the DSC trace, connected with the heat capacity decrease due to the glass transition, is visible. The lowest crystallization temperature (onset at 402.5 °C) has the Fe based alloy. At higher temperature (onset at 427.2 °C) the Zr based amorphous alloy becomes to crystallize and the Cu based alloy with a determined onset of crystallization of 442.5 °C shows to be more stable. Whereas in the case of Fe based alloy no significant glass transition is visible, the Cu based and especially Zr based alloys show the remarkable glass transitions. The glass transition temperatures were estimated as inflexion points on the decrease part of the heat flow curve. For Zr based metallic glass the glass transition temperature was found to be as 394.5 °C and for Cu based alloy 442.5 °C.
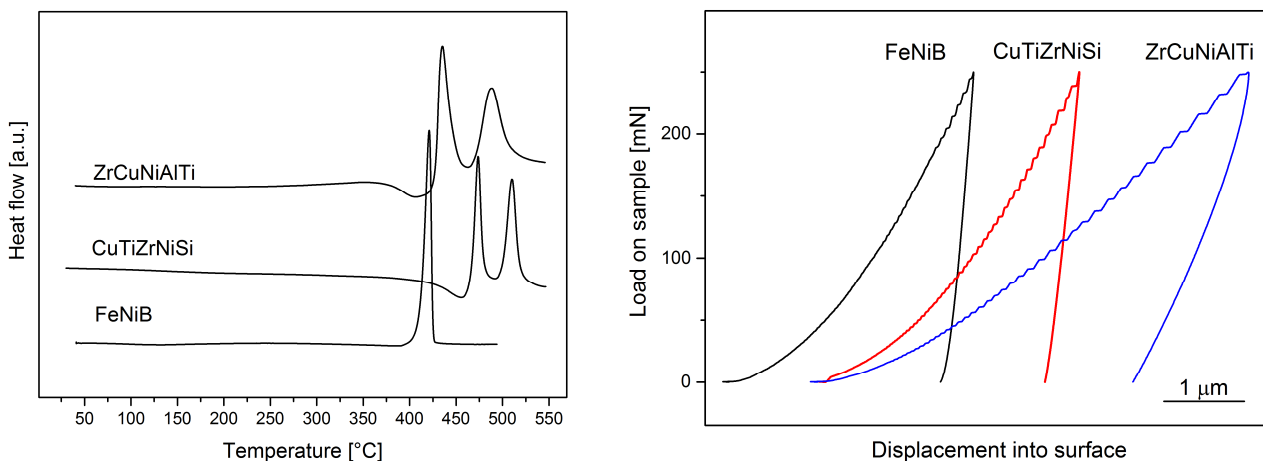

Fig. 1 DSC traces with crystallization peaks and glass transitions at temperatures below the crystallization (left), and the nanoindentation curves with pop-ins for indicated metallic glasses (right).
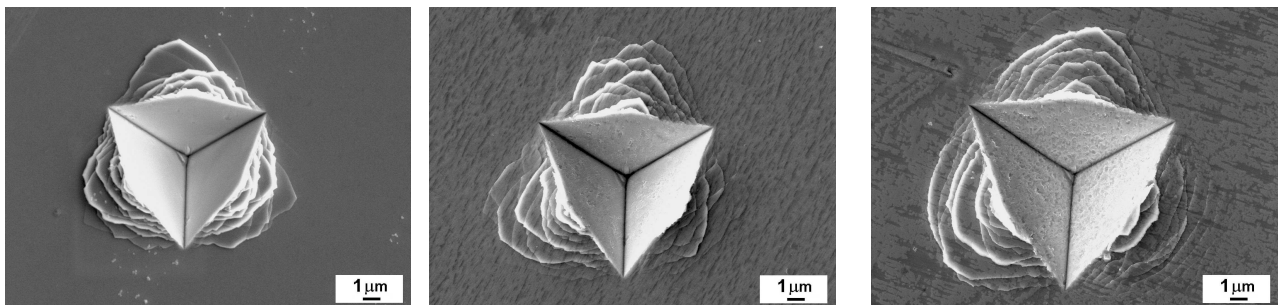

Fig. 2 Indent morphology for Fe (left), Cu (middle) and Zr based alloy (right) corresponding to the traces in Fig.1 (right).

For description and comparison of the indentation on the different alloys one typical indentation event and its SEM image were chosen. The nanoindentation curves in Fig. 1 (right) indicate the similar hardness for Fe and Cu based alloys, whereas the Zr based alloy exhibits larger plastic deformation and therefore lower hardness. For all alloys the serrations are present on the loading curves. The serrations develop with the loading increase. At low loadings, the serrations or individual pop-ins vanish in the noise. As the load raises the individual pop-ins are more and more developed and the period of their occurrence becomes larger. For Fe based alloy the pop-ins occur at higher loading, whereas in the case of Zr based amorphous alloy the serrations become visible at lower loading. In the case of Cu based alloy the intermediate behaviour can be seen.

The morphological observations of the indent shape for all alloys show the large deformation due to the cubic tip indenter and the maximal used load. Around the indent the deformed region with shear bands is present. The total deformed volume size for all examined alloys corresponds to the plastic deformation during nanoindentation according to the traces in Fig. 1 (right). The massive shear bands are parallel and divide the deformed region into platelet parts. The individual plate edges have the irregular form. The complicated edge form is caused by acting of secondary shears in alternative shearing variants. The deformation in amorphous structure is carried out via the creation and propagation of shear bands. The observation of deformation and failure of amorphous metallic glasses has shown that the shear band generates and propagates under quasi-adiabatic conditions [6, 7, 8]. High accumulated energy released by a localize shear is associated with the local heating of thin shear band zone [9]. The shear is then occured by shearing of rigid parts along the local heating softened bands. Comparison of dominant shear band topology (Fig. 2) with the form of loading curves (Fig. 1 right) does not show a simple correspondence between the pop-ins population and the principal shear band morphology in the plastic deformed region for individual alloy types.
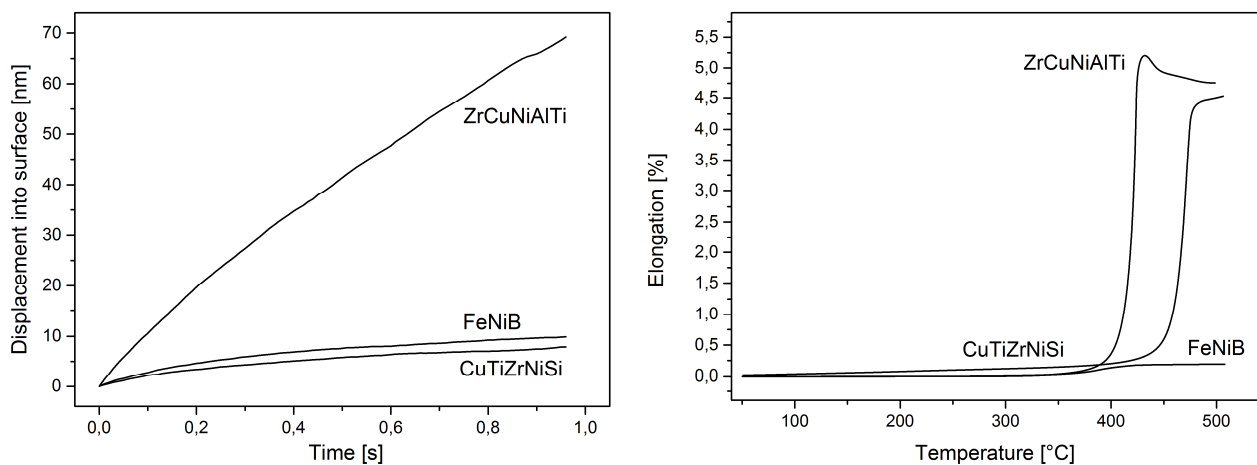

Fig. 3 Short-time nanoindentation creep for examined alloys (left) and the temperature dependence of the homogenous deformation for used metallic glasses (right).

Due to the metastable nature of the metallic glasses structure, their deformation properties are often time and temperature sensitive. Fig. 3 (left) demonstrates the difference in a short time stress relaxation during nanoindentation at room temperature. Whereas the Fe and Cu alloys exhibit similar rigidity, the Zr based alloy shows the significant time dependent stress relaxation. The ability of amorphous alloys to undergo homogeneous thermally activated deformation was studied by measuring their elongation under applied mechanical tension during linear heating. By the subtraction of measured elongation curve at low applied stress and that one performed at high stress it is possible to separate simultaneously a contribution of thermal expansion, contraction due to the structural relaxation and all apparatus effects [10]. The elongation difference during heating run at high and low applied stress for studied alloy types can be seen in Fig. 3 (right). For Zr type of amorphous alloy the elongation at temperatures above 380 °C dramatically raises. Similar

behaviour for Cu type alloy is observed at the temperature of about 50 °C higher. The dramatic elongation increase is connected with the viscosity decrease at glass transition. This softening is substantial lower for Fe alloy type. Although the hardness behaviour is generally connected with the yield stress, we assume that for the interpretation of indentation behaviour, such as the pop-in populations and their shape for different metallic glasses, their ability for homogeneous deformation should be taken into account. The reason for this argument is in local heating in shear bands during inhomogeneous plastic deformation of metallic glasses.

Summary

Nanoindentation of different amorphous metallic glasses shown, a different tendency to form serrations on the loading curves during the penetration of the cubic indenter. At selected loading rate of $1mNs^{-1}$ the lowest serration (pop-in) population was observed for FeNiB alloy, whereas the mostly developed pop-ins were present during nanoindentation on Zr based alloy with the high glass forming ability.

Acknowledgment

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