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Nekouie, Vahid, Anish Roy, and Vadim V. Silberschmidt. 2019. “Indentation Study of Mechanical Behaviour of Zr-cu-based Metallic Glass”. figshare. https://hdl.handle.net/2134/25911.

Indentation study of mechanical behaviour of Zr-Cu-based metallic glass

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Abstract

It has been well known that plastic deformation of bulk metallic glasses (BMGs) is localised in thin shear bands. So, initiation of shear bands and related deformation should be studied for comprehensive understanding of deformation mechanisms of BMGs. In this paper, indentation techniques are extensively used to characterise elastic deformation of Zr-Cu-based metallic glass, followed by a systematic analysis of initiation and evolution of shear bands in the indented materials. Our results, obtained with a suggested wedge-indentation technique, demonstrated initiation of shear bands in materials volume.

Key words: Metallic glass; shear bands; indentation.

1 Introduction

Modern high-tech industries rely on the manufacture and synthesis of advanced materials that are stronger than conventional ones. Bulk metallic glasses (BMGs) have received much scientific and technological attention due to their advanced mechanical properties such as a high ratio of elastic limit to the Youngs modulus and higher fracture toughness, when compared to their crystalline counterparts of the same composition. This is typically attributed to the absence of a long-range order in their atomic structure and a lack of defects such as dislocations, which control ductility in traditional metallic materials. BMGs are brittle and exhibit negligible plasticity in the macro-scale. Some recent experiments on sub-micron and nano-sized BMG specimens showed that the process of shear localisation become more stable and less catastrophic, when compared to a response exhibited by large-size samples [1]. These desirable and unique properties of metallic make them an ideal candidate for many applications such as MEMS (micro-electromechanical systems), miniaturised biomedical devices and implants as well as in micro-robotics. A number of mechanistic theories have been proposed to describe the plastic flow and deformation behaviour of BMGs. Some popular theories are concepts of free volume and shear transformation zones (STZs) proposed in Argon and Spaepens model, based on motion of atoms [2, 3]. The deformation mechanism of metallic glasses based on these concepts is realised homogenously or inhomogenously, depending on the levels of strain rate, temperature and applied stress [4]. A significant amount of experiments was carried out to understand deformation mechanisms. Prior studies showed

that shear bands with a characteristic thickness of the order of 10-20 nm were responsible for deformation of BMGs at low temperatures and/or high stresses. More recently, deformation-induced crystallisation was observed in a number of BMGs that led to substantial plastic deformation [5]. These changes in shear bands were not only induced by bending or compression of BMGs [6], but were the results of their nanoindentation or microhardness testing [7], ball milling or cold rolling process [8]. However, various research groups suggested contradicting conclusions on shear band crystalisation, based on similar experiments performed with various BMG systems [4, 9]. Thus, a question on propensity for crystallisation is still open. Traditional indentation techniques have been used extensively over years to determine mechanical properties and deformation mechanisms of metallic glasses [10]. These techniques helped researchers to perform mechanical characterisation at micro-scale and analyse the mechanism of plastic flow in BMGs. Although shear bands typically initiate beneath the indenter, in nano- and micro-indentation experiments, by their very nature, shear bands could be observed only after they evolved to the surface. So, a wedge indentation experiment was designed to overcome the limitation of nano- and micro-indentation to observe the initiation and propagation of shear bands under the indenter surface [1]. As the length of the wedge indenter was considerably larger than its width, the wedge indentation experiments also allow numerical modelling to be simplified to a 2D formulation. There are different, sometimes contradicting hypotheses about the deformation mechanisms of BMGs at microscale. Hence, further studies are required to understand initiation and propagation of shear bands in the volume and at the surface of metallic glasses. In this study, a Zr-Cu-based metallic glass is characterised using nano- and micro-indentation techniques. A thorough structural characterisation of shear bands around the indented region was carried out to understand the nature of shear banding in BMGs.

2 Experimental procedure

For our studies, a beam-shaped alloy with nominal composition of $Zr_{48}Cu_{36}Al_8Ag_8$ was prepared at IFW Dresden, Institute for Complex Materials, , Germany by arc-melting the pure elements (99.9% Zr, 99.99% Cu). BMG specimens were cut and polished to mirror-like finish with the roughness of some 5 nm. Indentation tests were conducted to characterise the shear bands with a nano indentation test system (Micro Materials Ltd.) using a spherical and Vickers indenters. A series of nano- and micro-indentations were conducted on the polished surface of the samples with a loading rate of 2 mN/s. A wedge indenter made of high-speed steel, with a nominal angle of 60 and an edge radius of 19.5 m was designed and manufactured in-house (Figure 1). The indentation tests reported here were conducted at ambient temperature. XRD analysis of as-cast samples was carried out to study formation of crystalline phases. In order to reconfirm the crystallography, the samples were thinned to electron transparency and observed using transmission electron microscopy (TEM). Scanning electron microscopy (SEM) was used to observe evolution of shear bands on the deformed surfaces.

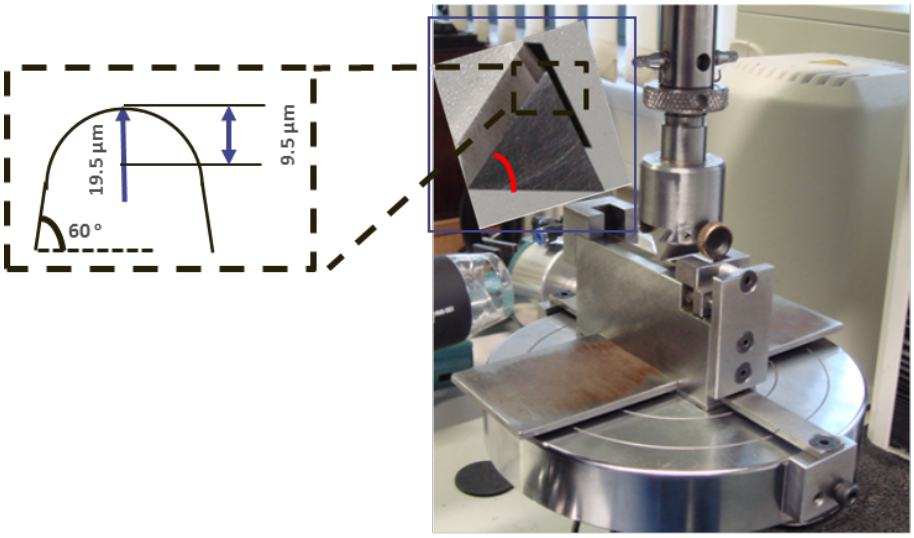


Figure 1: Experimental set-up for wedge indentation and its dimension

3 Results and Discussion

The amorphous nature of the supplied samples of BMG was initially investigated using X-ray diffraction (Figure 2); their microstructure was further characterised with TEM. The TEM results confirmed the amorphous nature of the alloy, as the first halo ring of a Selected Area Electron Diffraction (SAED) pattern did not show any presence of nanocrystals.

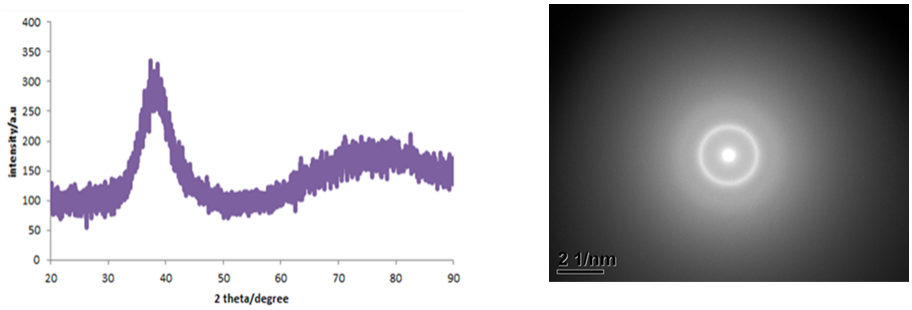


Figure 2: (a) XRD pattern of as cast $Zr_{48}Cu_{36}Al_8Ag_8$, (b) corresponding SAED patterns.

3.1. Microindentation

Multiple unloading-reloading experiments were conducted using a spherical indenter with diameter of 50 m at loading rate of 2 mN/s to investigate the variation of elastic modulus with depth in BMG specimens. The maximum indentation depth ranged from 6 m to 18 m, and three partial unloads down to 20% of the peak load at each step were applied in these steps. Shear bands formed at loads in excess of 10 N. As shown in Fig. 3, shear bands moving from various initiation points crossed each other; however, shear bands nucleated later were arrested by already nucleated ones.

Instability of shear bands was observed in the form of nucleation of several secondary shear bands from the primary ones in the course of deformation. A large plastic zone was formed under the indenter tip during indentation [12, 13]. It contained a high density of shear bands; this is ideal for investigation of deformation-induced hardening and softening effects [14]. The obtained results showed a dependence of elastic modulus on penetration depth, indicating a work-softening phenomenon in the studied metallic glass, especially at microscale. The values of reduced (Er) and elastic (E) moduli obtained from the curve using the Oliver-Pharr method are given in Table 1. The reduced and elastic moduli decreased from 48 GPa to 38 GPa and 41 GPa to 33 GPa, respectively. This phenomenon is often referred as indentation size effect (ISE) [12, 15], manifested by a decrease in the elastic modulus with an increase in the indentation depth. A large number of shear bands were activated by indentation; this reduced the reaction force on the indenter, leading to a reduction in material stiffness.

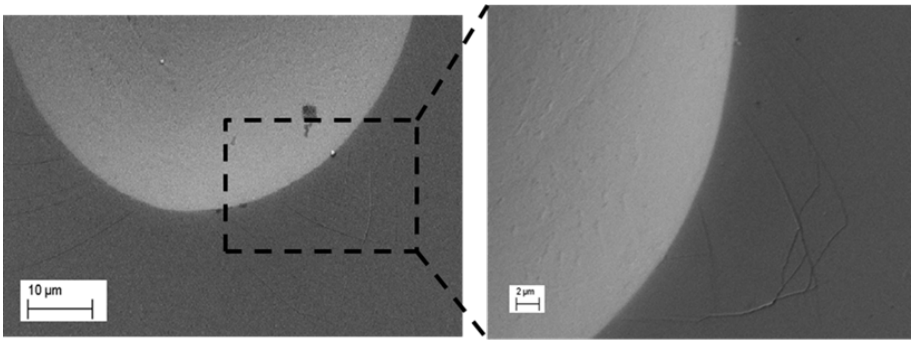


Figure 3: SEM images of micro-indentation in $Zr_{48}Cu_{36}Al_8Ag_8$.

Table 1: Reduced modulus (Er) and elastic modulus (E) of $Zr_{48}Cu_{36}Al_8Ag_8$ at various depth.

Cycle	Indentation depth (µm)	Reduced modulus (GPa)	Youngs modulus (GPa)
1	6.2	48	41.7
2	12.1	40	34.8
3	18.2	38	33.06

3.2 Shear-band initiation

The aim of this experiment was to characterise initiation of the first plastic event in order to calculate the required stress for shear bands initiation of the metallic glass investigated in this study. Based on the nano-indentation experiments [1], the first pop-in event occurred at applied force of 4 mN resulted in indentation depth of 0.060 m. The corresponding total area of contact and stress required for the first pop-in were 1.88 m2 and 2.12109 N/m2, respectively. It is necessary to determine the contact area A_c in wedge-indentation experiments in order to predict the approximate force necessary for initiation of shear bands. This force can be obtained using the following equation:

$$F = \sigma A_c \qquad \text{Eq.1}$$

It was found that the required load would be around 500 N; hence, testing was carried out using the beam-shaped samples in a compression mode with a constant displacement rate of 0.05 mm/min using the wedge indentation technique and the load levels between 200 N and 500 N. To avoid contact problem at low loads, surfaces of the wedge indenter were prepared with the use of a surface grinder in an attempt to make the surface uniform while maintaining the 60 angle.

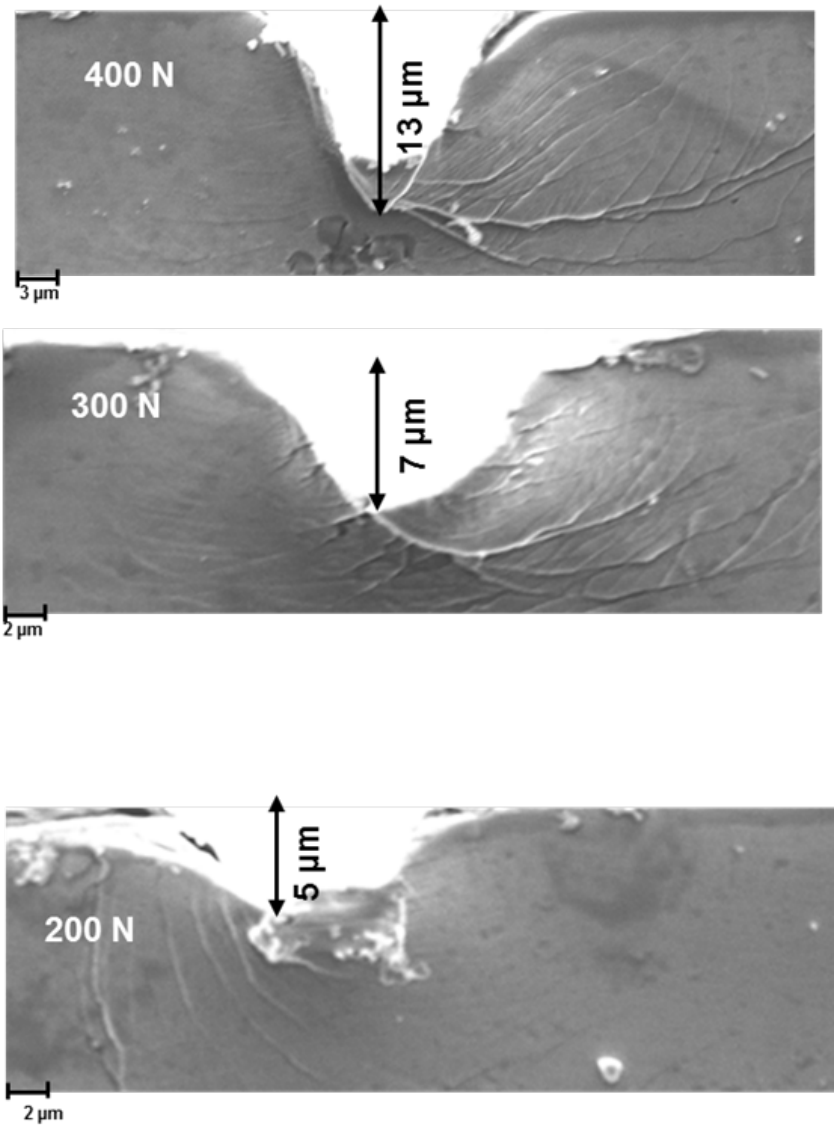


Figure 4: Evolution of shear bands pattern with load under wedge indentation: (a) 400 N, (b) 300 N, (c) 200 N.

The wedge indenter has an edge radius of 8 m and height of 5 m. Evolution of deformation pattern on the front surface of the specimen is presented in Fig. 4. The plastic depth increased from 5 m to 13 m by increasing the load from 200 N to 500 N, and serrated semi-circular slip-steps formed by shear bands were observed. The results show that the nucleation and initial propagation of shear bands occurred at loads below 200 N; there were no shear bands at 100 N load. For 200 N, the

indentation depth was 22.17 m before unloading obtained from F-D curve. The calculated stress was :

$$\sigma = \frac{F}{A_c} = 0.886 GPa$$

At 100 N, the shear band initiation stress was approximately 0.7 GPa, therefore, it can be estimated that, in wedge indentation the stress level required to initiate shear-band formation is between 0.7-0.9 GPa. Due to different shear bands morphology in wedge indentation, this value is not similar to the calculated stress required for shear bands initiation in nanoindentation with spherical indenter.

3.3 Comparison between glass and metallic glass

The purpose of this study was to compare fracture surfaces of soda-lime-silica glass and the studied Zr-Cu-based metallic glass using the wedge indentation technique at room temperature. The relationship established between mechanical behaviour and fracture feature can assist in elucidating the fracture mechanism. Wedge indentation was applied to both glass and metallic-glass specimens with dimensions of 40 mm x 4 mm x 2mm using loads of 500 N, 1 kN and 1.3 kN. Fractography studies showed that fracture surfaces of materials that fail in a brittle manner from surface cracks are characterized by a sequence of three distinct fracture features including mirror, mist and hackle regions, depending on the loading mode. For instance, there was no mist region observed on fracture surfaces formed in the mixed-mode failure [17]. A side view of wedge indentation for a glass specimen is presented in Fig. 5; here, classical concentric cone cracks were observed. The contact radius at maximum pressure was just within the outermost surface ring in Fig. 5, confirming that the cone fractures formed in the region of weak tension outside the subsurface compression zone. In addition, there was no detectable deformation observed beneath the contact circle; essentially, the material behaved as an ideally homogeneous solid. As shown in micrographs of subsurface damage at higher magnification in Fig. 5(c), hackle marking on the fracture surfaces of soda glass appeared as lances. Observation of fracture surfaces indicated that the propagating crack did not experience any energy-dissipation process such as plasticity or crack bridging, which could result in retarding the crack growth in a substantial manner.

As shown in Fig. 6, significant differences were found in the appearance of the fracture surfaces of specimens of the traditional and metallic glasses at microscale. In contrast to the former, shear bands in BMGs were not brittle and provided the ability to deform plastically, with many semi-circular shear bands created beneath the indenter. Shear bands bifurcate with increasing distance from the indenter tip, indicating branching and healing mechanisms contributing to energy-dissipation processes, which led to plastic deformation at microscale.

4 Conclusions

The microhardness study perfomed on $Zr_{48}Cu_{36}Al_8Ag_8$ clearly indicated the dependence of its elastic modulus on penetration depth at microscale due to activation of

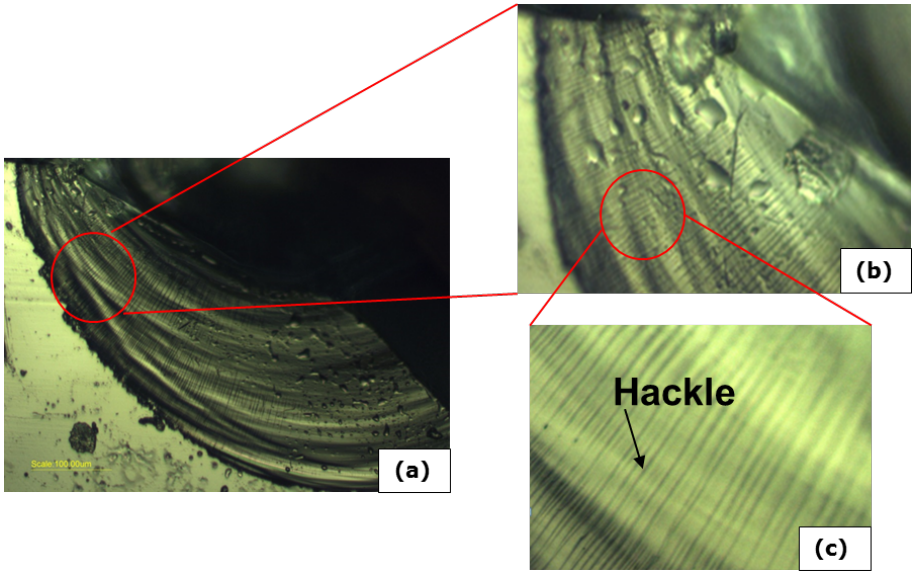


Figure 5: Optical micrographs of indented soda-lime-silica glass after applying 1.3 kN showing cone fracture form (a)and hackle fracture (b and c).

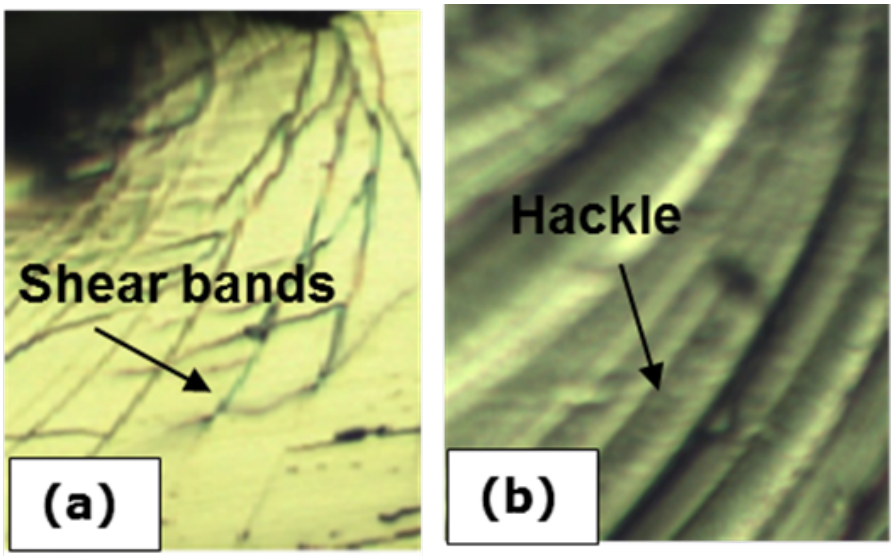


Figure 6: Optical micrograph of 1 kN wedge indentation on as-cast $Zr_{48}Cu_{36}Al_8Ag_8$ metallic glass (a) and soda-lime-silica glass (b).

a large number of shear bands. A relatively new technique wedge indentation was employed to calculate the required stress for shear bands initiation of the metallic glass. It was estimated that the level of stress required to initiate shear bands was between 0.7-0.9 GPa in wedge indentation. The wedge indentation technique was also applied to compare fracture surfaces of the soda-lime-silica glass and the studied Zr-Cu-based metallic glass at microscale. Observation of fracture surfaces indicated that the propagating cracks did not experience any energy dissipation in the traditional glass; on the contrary, the shear-band evolution in the metallic glass showed branching and healing mechanisms contributing to the plastic deformation

at microscale.

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