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Experimental studies of shear bands in Zr-Cu metallic glass

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ABSTRACT

Shear bands are the key feature that control deformation in bulk metallic glasses (BMGs). This study provides a comprehensive analysis of plastic deformation of a Zr-Cu-based BMG at room temperature. Experiments were conducted to observe the evolution of shear bands in the material. It was shown that shear bands formed discretely in the material which allows for material deformation to occur across it. Additionally, individual shear bands were characterised to obtain a better understanding of shear-band-induced plasticity. Assessment of mechanical properties, such as hardness and elastic modulus, indicate the deformed regions of the material were weaker than undeformed regions. No compositional or structural changes were found in shear-band of the studied BMG suggesting generation of local free volume in the deformed region.

1. Introduction

Bulk metallic glasses (BMGs) have attracted considerable attraction as structural materials due to their high strength and superior elasticity [1–6]. At room temperature, inelastic deformation behaviour in BMGs are strongly governed by the formation of shear bands (SBs), making an investigation of their origin, evolution and properties a pre-condition for interpretation of mechanisms of plastic deformation of BMGs [7,8]. Several studies were recently carried out, for example, on propagation dynamics of an individual shear band (SB) and interactions between them [9,10], effects of extrinsic factors including sample size, machine stiffness, testing temperature and strain rate on shear-banding behaviour [11–14] and *in-situ* or quasi-*in situ* observations of SB evolution [15–17].

Localisation of shear deformation induces intense strain softening in BMGs; mechanism for which are debated [18]. Deformation-induced nanocrystallisation was observed in some BMGs under large plastic deformation [7,11,19], under compression [20], nanoindentation [21] and ball-milling processes [14]. It was suggested that *in-situ* nanocrystallisation within SBs resulted in bifurcation of their propagation, indicating branching and healing mechanisms contributing to energy-dissipation processes that were responsible for plastic deformation at the microscale. An origin of nanocrystallisation was explained in terms of heating in shear bands, free-volume generation, shear strains or ultrahigh strain rates. Schuh and co-workers proved that SBs in a Zr-based metallic glass demonstrated no obvious long-range order [5,14]. This hypothesis was confirmed in a study by Wilde and Rösner [22], who found no crystallisation in SBs of $Al_{88}Y_7Fe_5$ glass at room temperature. Thus, the question of propensity for crystallisation in SBs of BMGs is still open. With different, sometimes contradicting hypotheses about deformation mechanisms of BMGs at the microscale, further studies are required to understand initiation and propagation of SBs in the volume and on the surface of metallic glasses. Structural characterisation of SBs around a deformed region will help us understand the nature of their formation and evolution [23]. This could help link SB propagation with inelastic deformation of BMGs.

As localised shear-band formation is responsible for inelastic deformations in BMGs, this study focus on characterisation of shear bands as well as their formation and evolution. It provides a new insight related to shear bands in Zr-Cu-based metallic glass and its deformation under homogeneous (uniaxial compression) and inhomogeneous (wedge indentation) loading conditions. To achieve this, individual SBs were characterised with TEM. Nano- and wedge indentations were carried out to analyse the evolution of SBs through the volume of the BMG employing a surface-decoration technique.

2. Material and experimental procedure

A BMG with a nominal composition of $Zr_{48}Cu_{36}Al_8Ag_8$ (at.%) was selected for this study. The alloy was produced at IFW Dresden, Institute for Complex Materials, Germany. The supplied samples were cut using wire electric discharge machining (EDM) into rectangular specimens of approximately 40 mm × 2 mm × 2 mm for a three-point bending test and 40 mm × 5 mm × 2 mm for wedge indentation

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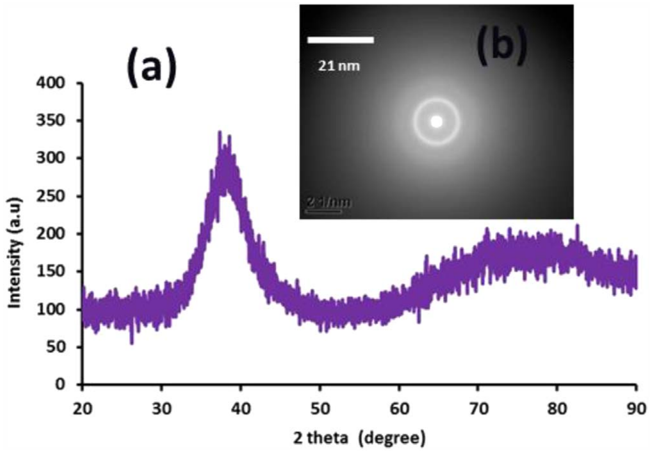

Fig. 1. XRD pattern (a), with corresponding SAED patterns shown in inset (b), of as-cast $Zr_{48}Cu_{36}Al_8Ag_8$.

experiments. All samples were polished to mirror finish before experimentation. Structure of the studied alloy was inspected with X-ray diffraction (XRD) using a Rigaku Ultima-III diffractometer with Cu-Kα radiation. The scans were carried out over several locations on the samples that were characterised with TEM (JEM-2000FX, JEOL Ltd., Tokyo, Japan) before and after deformation. For TEM analysis, the samples were thinned to electron transparency using a focussed ion beam system (FIB, FEI Company, Hillsboro, USA). As shown in Fig. 1, only broad amorphous peaks were presented without any indication of crystalline Bragg peaks. The dark field TEM image indicates that no crystalline phases were found, further confirming the glassy nature of the samples. The halo SAED pattern of the as-cast sample (Fig. 1b) shows a set of diffuse rings, which is typical of an amorphous structure. The composition of the locations, where the SAED was obtained from, was tested *in-situ* with EDS. The measurement showed that the elemental concentrations were close to the nominal values of the BMG material which is 47.9 at.% Zr, 36.5 at.% Cu, 7.9 at.% Al and 7.7 at.% Ag.

To study SB evolution, interrupted wedge indentation was performed on the BMG component. The wedge indenter was made of high-speed steel (HS 6-5-4), with a wedge angle of 60° and an edge radius of 19.5 μm, designed and manufactured in-house (see Fig. 2). The tests were conducted with an Instron 3345 testing machine, with a displacement rate of 0.5 mm/min. The unloaded samples were analysed with SEM (LEO 1530VP, Carl Zeiss SMT, Germany) on their top and front surfaces (see Fig. 2). First, an initial indentation with a load of 500 N was made on a beam-shaped specimen with dimensions 40 mm × 5 mm × 2 mm. This was sufficient to initiate shear bands near the point of load application. Next, ten 6 × 6 sets of grid lines were milled using FIB with 30 kV acceleration voltage and 10 pA beam current on the front surface of the beam. The milled lines had a length of 20 μm, width of 0.2 μm and spacing of 1 μm (Fig. 5). Following this,

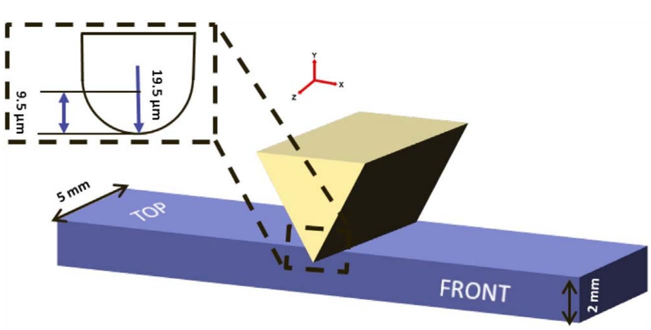

Fig. 2. Schematic of wedge indentation setup for SB observation with wedge dimensions.

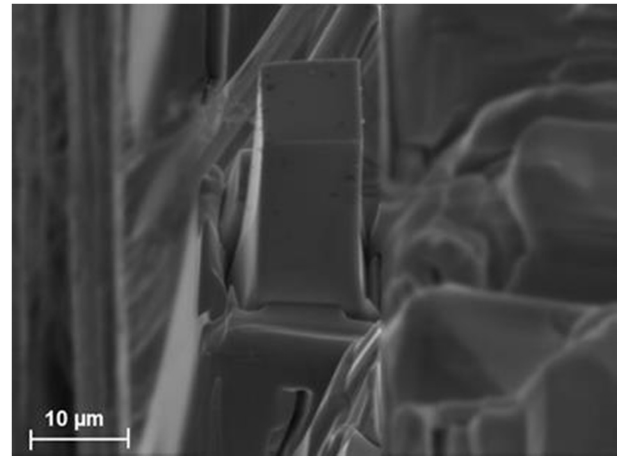

Fig. 3. Micropillars of as-fabricated $Zr_{48}Cu_{36}Al_8Ag_8$ with square cross-section of 10 μm × 10 μm.

wedge indentation was performed by loading-unloading at 1 kN and 2 kN. The gridlines were observed with SEM after each unloading step (Fig. 5a–d).

Subsequently, to characterise the SBs, a series of nano-indentation (Nanotest 600, Micro Materials Wrexham UK) experiments were conducted at and near the SBs (Fig. 9). A fracture surface obtained from conducting a three-point bend test to failure was characterised using a Vickers indenter under peak loads of 500 mN and a loading rate of 2 mN/s with a 30 s hold time at peak load to assess variation in hardness.

Specimens for microcompression studies were prepared from beam-shaped samples of as-cast material and from the fracture surface area (see details in Section 3.3.2). To reduce the effect of a taper, micropillars (see Fig. 3) with a nominal square cross-sectional area of 10 μm × 10 μm and an effective height of 25 μm were prepared with a top-down milling method with FIB by successively reducing the cross-section of the pillar during the milling process. The pillar obtained from the fractured surface was milled to ensure a flat top surface. Ion beam with a 30 kV accelerating voltage was used where an initial current of 20 nA was gradually reduced to 1 nA as the pillar cross section decreased. The tests were performed with a nanoindentation system (MTS nanoindenterXP) using a flat punch tip with an equilateral triangle cross-section with an inside length of 50 μm. All tests were performed at a constant nominal displacement rate of 8 nm/s with a total indenter displacement of 2 μm. The resulting evolution and morphology of microstructure were examined with SEM.

3. Shear-band characterisation

3.1. Shear-band evolution

The surface-decoration technique was used to track formation and evolution of multiple SBs [24]. Shear bands were examined at ten different locations as shown in Fig. 4.

It is known that surface imperfections serve as preferred sites for initiation of SBs [25]. We performed several studies to ensure the surface decoration itself do not serve as SB initiation sites. Our studies indicate no obvious differences in SB evolution in specimens with polished and decorated surfaces.

As illustrated in Fig. 5, SBs formed a localised deformation zone with slip occurring across these bands as marked by arrows in Fig. 5. This indicates that once an SB was formed, the material volume slipped across it discretely (Fig. 5b). Our experimental studies show that SBs do not propagate gradually across specimens (no diffusion) associated with SB displacement that occurred in a localised area as proposed by Liu and co-workers [26].