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SciVerse ScienceDirect

Scripta Materialia 69 (2013) 207–210

Scripta MATERIALIA

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In situ study of the evolution of atomic strain of bulk metallic glass and its effects on shear band formation

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Received 18 February 2013; revised 13 March 2013; accepted 17 March 2013
Available online 22 March 2013

The tensile behavior of TiZrNiCuBe metallic glass was studied using synchrotron X-ray diffraction and scanning electron microscopy. Shear band formation was carefully controlled by the introduction of a stress gradient due to a semi-circular notch. Micro- and atomic-level strains were calculated by the *Q*-space method and pair distribution function analysis, respectively. The atomic-pair strains for the area near the notch increase exponentially at the final deformation stage. This can be directly linked to the shear-induced dilatation resulting from the shear band formation.

Keywords: Metallic glasses; Synchrotron X-ray diffraction; Pair distribution function; Strain; Shear bands

Bulk metallic glasses (BMGs) often have an excellent combination of exceptional physical, mechanical and chemical properties, that are superior to their crystalline counterparts of identical chemical composition [1]. They are promising materials for applications which require very high strength, wear and/or corrosion resistance. However, BMGs usually have very low plasticity (<2%) in compression and nearly zero plasticity in tension [2], seriously limiting their structural applications on any industrial scale. Hence, the study of their intrinsic deformation mechanisms is of scientific and technological importance. Since the emergence of BMGs, numerous studies have concentrated on understanding how to increase their global plasticity, especially how to control the nucleation and propagation of shear bands. Low plasticity makes it very difficult to measure the local stress/strain at the location where shear bands nucleate and to track the evolution of the stress/strain along the path of shear band propagation. In 2005, Poulsen et al. [3] demonstrated that synchrotron X-ray diffraction (SXRD) is a suitable technique for characterizing the stress/strain fields in amorphous materials. Using an in situ loading rig, X-ray diffraction patterns were acquired on the BMG samples at different locations at different load steps [4]. The strains calculated from the scattering intensity spectra agreed well with the macroscopic measurement using a load–displacement curve [5]. Meanwhile, in situ transmission electron microscopy (TEM) [6–9] and high-resolution scanning electron microscopy (SEM) [10] have been also used to study the nucleation and propagation of shear bands. For example, Chen et al. [8] used in situ TEM compression tests to study the intrinsic size effects on the deformation mode of metallic glass pillars, and Matthews et al. [6] used in situ straining TEM to study shear band propagation, while Bouzakher et al. [10] used in situ SEM to investigate the redirection of shear bands in a stress field. However, combining electron microscopy with SXRD in situ studies to investigate the correlation between local atomic rearrangement and shear band formation has not been reported so far. In this paper, we describe the in situ deformation behaviors of unnotched and notched Ti-based BMG samples using the complementary techniques of SXRD and SEM. The aim was to investigate the underlying relationship between local atomic rearrangement and shear band formation. Micro- and atomic-level strains were calculated from SXRD patterns, while separate in situ SEM tests were performed under identical conditions so that shear band formation and propagation could be observed.

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1359-6462/$ - see front matter
http://dx.doi.org/10.1016/j.scriptamat.2013.03.016

The strain evolution of different atomic pairs under different loads was quantified and correlated with the nucleation and propagation of shear bands.

Alloy ingots with a nominal composition of $Ti_{40}Zr_{25}Ni_3Cu_{12}Be_{20}$ (at.%) [11] were prepared by arc melting, then casting in a copper mold to form plates of $3 \times 30 \times 60\ mm^3$. Dog-bone-shaped tensile samples having a gauge length of 6.75 mm, a thickness of 0.3 mm and a width of 1.5 mm (Fig. 1) were electrical discharge machined from the cast plates. For some samples, a semi-circular notch with a diameter of 250 μm was machined on one edge in the middle of the gauge length. The side surfaces of all the tensile samples were ground using SiC abrasive papers and finally polished with 1 μm diamond suspension before testing. In situ tensile tests were carried out at room temperature using a Deben Microtest 2000 module. The X-ray diffraction measurements were performed at the Joint Environmental, Engineering and Processing beamline (I12) of Diamond Light Source (Oxfordshire, UK). Figure 2a shows the layout of the experimental setup. The loads were applied using load-step control until sample failure. A monochromatic X-ray beam of a spot size of $0.25 \times 0.25\ mm^2$ and a Pixium RF4343 two-dimensional (2-D) detector were used to acquire diffraction patterns at the locations marked by P1, P2 and P3 on the sample, as shown in the Figure 1 inset. Each acquisition took 4 s. A high X-ray energy (98.856 keV) and a short sample-to-detector distance of 472.85 mm were used to acquire diffraction patterns up to a scattering vector Q of 20 $Å^{-1}$ ($Q = 4\pi \sin\theta/\lambda$). Complementary in situ SEM observations of shear band nucleation and propagation under identical load conditions were carried out inside Zeiss Evo60 SEM operated at 20 keV.

When acquiring diffraction patterns under tensile loads, the initial concentric diffraction rings become elliptical, expanding in the tensile direction and contracting in the transverse direction. To quantify these changes, 1-D scattering intensity spectra $I(Q)$ were extracted from the 2-D X-ray diffraction rings by using Fit2D [12] after subtracting the background using software ImageJ [13]. The mathematical procedure used is: (1) the diffraction rings acquired at different loads were segmented anticlockwise into 36 parts in the φ range of 0 to π using the Fit2D program; then (2) the scattering intensity within each segment was integrated [14].

The strain caused by the applied stress can be calculated by the relative shift in the position of the first peak of $I(Q)$ at that stress, analogous to the simple macroscopic definition of engineering strain, as follows [14]:

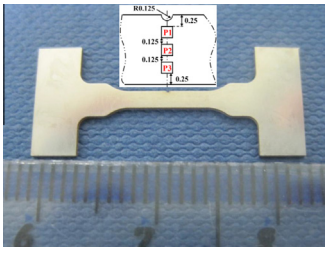

Figure 1. A dog-bone-shaped Ti-based BMG tensile sample. The inset shows an enlarged map of the middle of the notched sample with the three diffraction measurement locations, P1, P2 and P3 (units: mm).

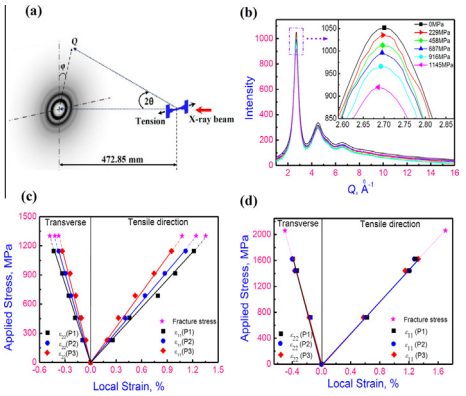

Figure 2. (Color online) The intensity curves and the stress–strain correlation. (a) The setup for the in situ tensile experiments; (b) typical scattering intensity spectra $I(Q)$ along the tensile direction, with an inset showing the shift of the first peak positions with applied stress; and strains at P1, P2 and P3, of the samples (c) with and (d) without a notch as a function of applied stress.

$$\varepsilon_i(\varphi_i, \sigma) = [Q(\varphi_i, 0) - Q(\varphi_i, \sigma)]/Q(\varphi_i, 0) \quad (1)$$

where $Q(\varphi_i, 0)$ and $Q(\varphi_i, \sigma)$ represent the positions of the first peak of $I(Q)$ curves at zero stress and σ, respectively, and φ_i is the azimuthal angle in the 2-D diffraction rings.

Figure 2b shows typical $I(Q)$ curves measured for a notched sample along the loading direction, with the inset showing the shift of the first peak positions with tensile stresses. As stress increases, the first peak position shifts towards smaller Q in the tensile direction ($\varphi_i = 0°$; see Fig. 2b inset) and towards higher Q in the transverse direction ($\varphi_i = 90°$; not shown here). Figure 2c and d shows the relationship between the local strains calculated using Eq. (1) at P1, P2 and P3, and the global tensile stresses applied on the unnotched and notched samples, respectively. For both samples, the strain increases linearly with increasing stress in both the tensile and transverse directions, as expected for a linear elastic material. For the unnotched sample, identical strains were obtained at P1, P2 and P3 (Fig. 2d), and the ratio of the strains between the tensile and the transverse directions gives Poisson's ratio of 0.330 ± 0.002. For the notched sample, different tensile and compressive strains were obtained, with $\varepsilon_{P1} > \varepsilon_{P2} > \varepsilon_{P3}$ (Fig. 2c), showing a clear strain gradient near the notch.

To study the strain evolution at atomic level, we calculated the total structural factor $S(Q)$ from $I(Q)$ using the Egami–Billinge procedures [15] and PDFgetX2 [16], and the reduced atomic pair distribution function (PDF), $G(r)$, by Fourier transformation of $S(Q)$ [14].

Figure 3a shows the PDFs $g(r)$ for the notched sample, with the inset showing the shifts of the first peak (Peak A: the 1st atomic coordination shell) under different stresses. Peak A shifts towards larger r with increasing applied stress, indicating the elongation of the 1st atomic coordination shell along the loading direction. Similar phenomena occur in the 2nd and 3rd atomic coordination shells (Peaks B and C, shown in Fig. 3a). These shifts can be converted into the strains for the respective atomic shells in both tensile and transverse