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ScienceDirect
Acta Materialia 59 (2011) 5146–5158
Acta MATERIALIA
www.elsevier.com/locate/actamat

# Softening and dilatation in a single shear band

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Received 26 January 2011; received in revised form 15 April 2011; accepted 21 April 2011
Available online 2 June 2011

**Abstract**

Shear band is the key feature that controls the plastic deformation process of metallic glasses (MGs). However, the investigation directly on the shear band and its properties is rarely conducted as the band is perceived as extremely narrow. We have conducted a controlled experiment to form a single shear band in the specimen, which enabled us to probe shear-induced softening and dilatation directly on the shear band itself. Extreme dilatation and free volume increase as high as 1.14% and 1.40% respectively, have been observed, resulting from a drastic structure change due to severe plastic flow in the band. Nanoindentation on the individual shear band reveals significant softening of 36% and an unexpected wide width up to 160 μm, three magnitudes higher than what has been reported. These prove beyond doubt the dilatation as a mechanism for softening. The correlation between the free volume content and softening is discussed. Our findings provide a new insight for understanding the deformation behavior of metallic glass.


*Keywords:* Shear band; Softening; Free volume; Dilatation; Shear band thickness

## 1. Introduction

Metallic glasses, especially the recently discovered bulk metallic glasses (BMGs), have attracted broad attention due to the fact that BMGs possess remarkable physical, chemical and mechanical properties, as well as great potential for applications in many areas [1–5]. However, plastic deformation of BMGs is highly localized into shear bands, resulting in a limited plastic strain and catastrophic failure at room temperature. Unlike ductile crystalline materials where the plastic deformation is uniform due to the occurrence of work-hardening, plastic deformation of metallic glasses is concentrated within narrow bands, i.e. shear bands [6]. Shear band is the unit of the plastic deformation in metallic glasses, and investigation of the properties of shear bands will aid us to better understand the mechanism of plastic deformation of metallic glasses. Much work had been done to investigate the formation mechanism and properties of shear band in the past decades [7–14]. However, most of the studies performed on the shear band of BMGs were either by simulation [7,8] or by experiments, but on a composite, i.e. shear bands embedded in an undeformed matrix of BMGs due to the formation of multiple shear bands [9–14]. Tang et al. [9] found that the specimen (the composite) was softened after deformation, but they did not provide the information on the shear band itself. Bei et al. [10] reported that the hardness was reduced by 10%, even with the homogeneous compressive deformation of 80%. They used the model of composite and roughly deconvoluted the hardness of the shear band to be 1.4 GPa, which is about 80% reduction compared to the undeformed region. Statistical analysis of the nanohardness data obtained from an extensively deformed region by Yoo et al. [12] showed that the deformed region is always softer than the undeformed region. They further calculated the hardness of the shear band according to Johnson's expanding-cavity model, and showed that the averaged shear band hardness is about 3.25 GPa.

On the other hand, dilatation is considered as the mechanism of deformation-induced localization, but the

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doi:10.1016/j.actamat.2011.04.047

dilatation in metallic glass is rarely investigated directly. Dilatation due to shear in metallic glass is only supported indirectly by density measurements by Cahn et al. [15]. However, the evidence is not convincing and the conclusions are somewhat inaccurate as the test was not performed directly on the deformed region due to the formation of multiple shear bands during rolling.

Many have tried to investigate the shear band through serve deformation. However, the difficulty is that increasing apparent plasticity will only increase the number of shear bands and decrease the shear band spacing [9–14], not increase the degree of deformation within the band. This has actually limited the amount of deformation in each band. In general, there are three difficulties to investigating one shear band directly in a composite setting (shear band and an undeformed region). Firstly, it is hard to determine the location of the shear band. The surface steps associated with shear bands have to be erased by polishing to create a flat surface. Once that is done, the traces of shear bands disappear, making the precise location of the shear band impossible. Secondly, shear bands typically comprise only a small volume fraction of a deformed specimen, but bulk characterization techniques average over both the shear bands and the (presumably undeformed) remainder of the material. Thirdly, even if a high degree of nominal deformations had been carried out, the plastic strain in a single shear band is still small. One character is that the total hardness decrease is only 10% or less.

Through conducting a controlled deformation of metallic glass, we demonstrate a method to form one single yet stable shear band in the sample. Under this condition, the shear band or sheared region can be distinguished from the matrix easily. Forming one dominant shear band enables us not only to increase the degree of the deformation in the materials, but also to perform a clean study on the deformed region, such as measuring its hardness directly and unambiguously, and estimating the corresponding free volume generated in the shear band. Since all the plastic strain is concentrated, the shear band region is severely plastically deformed, leading to a drastic change in structure and dilatation. This has resulted in a record jump in free volume and significant increase in volume, which consequently causes a significant softening in the band. Our findings prove that dilatation certainly is a mechanism of softening. The present results provide strong evidence that the thickness of a well-developed shear band is well above what was previously reported.

## 2. Experiments

Alloy ingots with nominal composition of $Zr_{69.5}Cu_{12}Ni_{11}Al_{7.5}$ were prepared from elemental metals (purity > 99.5%) by arc-melting under a Ti-gettered Ar atmosphere. From the master alloy, a sample rod with a diameter of 2 mm and length of 70 mm was then produced by copper mold casting. The amorphous structure of the as-cast alloy prepared was examined by X-ray diffraction (XRD, Philips $\chi'$ Pert Pro) with Cu K$\alpha$ radiation and transmission electron microscopy (TEM, Joel-2010). The TEM foils were prepared following a series of thinning processes, namely mechanopolishing, dimpling and finally ion-milling. To reduce the possible effect of ion-milling on the structure of the samples, a voltage of 3 kV was used.

Samples with different plastic strain (2%, 4%, and 6%) were prepared by uniaxial test with a Zwick/Roell 020 testing machine at strain rate of $2 \times 10^{-4}$ s$^{-1}$. The compression specimens with a diameter of 2 mm and a length of 4 mm were cut from the cast rod, and the ends were polished carefully to ensure parallelism. The morphology of the deformed samples was examined with a scanning electron microscope (SEM, FEI Quanta 200). The thermal response of as-cast and deformed samples was investigated with differential scanning calorimetry (DSC, Perkin-Elmer DSC-7) at a heating rate of 20 K min$^{-1}$ in a flow of argon. A second run under the identical condition was used to determine the baseline after each measurement.

Nanoindentation was conducted using a triboindenter (Hysitron Inc., Minneapolis, MN), with a Berkovich diamond tip mounted to a low thermal conductivity shaft mounted on an atomic force microscope (AFM). A maximum load of 5 mN with a loading rate of 0.5 mN s$^{-1}$ (with a 10 s hold time at the peak load) was applied. The hardness in different samples or regions was measured from the load–displacement curve of nanoindentations by the method of Olive and Pharr [16]. In order to validate the hardness of the shear band, microindentation was also carried out on the 6% sample using a Vickers diamond with a load of 200 g and a dwell time of 10 s. The spacing between each indent is 50 μm.

The dimension changes of the as-cast and deformed sample were investigated by thermomechanical analysis (TMA, Q400EM) at a heating rate of 20 K min$^{-1}$ from 293 K to 593 K in a flow of argon with a load of 20 mN. To remove the free volume, the samples were kept warm for 1 h at 593 K. The accuracy of displacement could be 15 nm, which is enough for the dimension change during heating.

## 3. Results

### 3.1. The structure and hardness of the as-cast alloy

Fig. 1a shows the XRD pattern of the as-cast $Zr_{69.5}Cu_{12}Ni_{11}Al_{7.5}$ alloy. The alloy is basically amorphous, as indicated by a broad typical diffraction hump with the absence of any detectable crystalline peaks. The detailed microstructure was further examined by TEM. Fig. 1b shows typical characteristics of amorphous structure, and the corresponding selected area electron diffraction (SEAD) pattern shows the faint and diffuse ring. No trace of crystalline phases could be detected in the sample within the solution of the TEM apparatus used.

The hardness of the as-cast sample was measured by nanoindentation at an interval of 100 μm, which showed