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Stress induced atomic-scale damage and relaxation in bulk metallic glasses

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ABSTRACT

The nanoscale Electric Contact Resistance (nanoECR) system with a high resolution is used to study the possible structural change in bulk metallic glasses (BMGs) after stimulation of cyclic stress. It is found that the electrical current traveling through the fatigue-tested $Zr_{50}Cu_{40}Al_{10}$ BMG specimen is much lower than that of as-cast one at the same electric voltage. Excluding the effect of sample geometry, it is revealed that the increase in the electric resistivity of the fatigue-tested BMG is due to a more disordered structure. Furthermore, our results show pronounced atomic-scale damage and relaxation in the fatigue-tested BMG, shedding quantitative insights into the process of structural evolution in BMGs under cyclic loadings.

1. Introduction

As a new class of metallic materials, bulk metallic glasses (BMGs) have many attractive mechanical properties, such as extremely high strength and hardness, large elastic limit and high wear resistance, due to their complex disordered atomic structures [1–3]. Such non-crystalline structures are thermodynamically metastable in nature, and they have a strong tendency for structural relaxation upon temperature changes or stress perturbations [4,5]. For thermal relaxation, encouraging progresses have been made. Generally, the thermal relaxation includes physical aging and crystallization, when the temperature increases, the BMGs tend to shift to corresponding equilibrium states [6,7]. For the stress induced relaxation, most studies were focused on the dynamic mechanical relaxation, it was found that the β relaxation is the precursor of α relaxation, and the activation energy of β relaxation is related to the potential energy barriers of the shear transformation zones [8–10]. However, atomic-scale details for the stress induced relaxation are still unclear. In spite of the numerous works about the micro- and macro-scale damages (shear band and crack) in BMGs [11,12], the atomic scale damage mechanism of BMGs is still regarded as a long-standing problem, which is crucial for the in-depth understanding of the deformation mechanism of BMGs.

The reason for the above situation is twofold: first the underlying structural unit for the atomic scale evolution process is unclear. Although free volume and shear transformation zone (STZ) models were widely used for understanding the deformation in BMGs, microscopic scale damage involves the interaction between the individual structural units, which are not accounted for by those mean-field models. Secondly, effective detection of the tiny local atom rearrangement is difficult. Recent findings of nanoscale mechanical heterogeneity of BMGs imply a heterogeneous atomic structure in BMGs [13,14], which has been directly confirmed by the geometric frustration of the local atomic order [15]. Based on the structural heterogeneity, the newly proposed core–shell or flow unit model provides us a new insight into the deformation mechanism of BMGs [16,17], and it views the amorphous structure as an atomic-scale composite, with basic block composed of liquid-like cores (LLCs) embedded in the solid-like shells (SLSs). The LLCs are the soft regions with loosely packed atoms, under applied stress

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they are prone to responding non-affinely; while the SLSs are the relative hard regions with densely packed atoms, they build up the elastic matrix and undertake the affine deformation of BMGs. Based on the core–shell model, anelasticity and yielding of BMGs have been successfully interpreted [18]. Nevertheless, how the structural heterogeneity evolves under stress is seldom studied. Besides, it is well established that the relaxation of metallic glasses leads to the changes of the electrical resistance [19,20]. Therefore, we will make efforts to probe the internal structural rearrangement of BMGs based on electrical response of the BMGs. The purpose of the present work is to study the stress-induced structural evolution details of BMGs based on the core–shell model. To this end, fatigue-tested BMG specimens, with greater number and larger magnitude of atomic rearrangement regions after thousands of cyclic stress stimulations, were selected to compare the electrical response with that of as-cast BMGs. Qualitative results have been unveiled by analyzing the results of the electric response based on the in-situ nano Electric Contact Resistance (nanoECR) measurement in the nanoindentation system.

2. Experimental details

It is clear that resolving this atomic evolution issue requires an experimental technique possessing a sufficiently high sensitivity and spatial resolution to directly capture the collective atomic-scale processes leading to a local activation of the deformation units. In this study, we will show that nanoECR, with micro-volt, pico-amp and milli-second resolutions, offers us the unique opportunity to achieve this goal. A schematic drawing of the nanoECR test is shown in Fig. 1(a). Through the conducting boron doped diamond indenter (with an electric resistivity $3.3 \times 10^{-2}\ \Omega$ m) impressed on the BMG micropillar (mounted on conductive stage), an electric current, which carries the dynamic evolutional information of the internal atom structure during the deformation process, can be in-situ monitored, and the relative change in the resistance and resistivity can be calculated. The relative resistivity were analyzed to uncover the evolution of the atom structure during the loading process, and comparative analysis were carried out to reveal the structure difference between the as-cast and fatigue-tested BMGs. It should be pointed out that absolute resistivity value of the metallic glass cannot be directly measured by the nanoECR test, as the relative resistance extracted here includes the resistance of conductive diamond tip, metallic glass sample and the conductive stage connected in series during the loading process. Because the deformation only occurs in the micropillar, the atomic evolution process in BMGs can be accurately detected. Cylindrical amorphous rods of $Zr_{50}Cu_{40}Al_{10}$ with a diameter of 8 mm were prepared by the tilt-casting method in an arc-melting furnace and subsequently machined into the button-head fatigue specimens [21,22]. Prior to tension–tension fatigue tests, the samples were polished to 1200 grit to eliminate surface's influence. Then, employing a computer-controlled Material Test System (MTS), the fatigue tests were carried out at a fixed stress range of 2015-201.5 MPa with a R ratio ($R = \sigma_{min.}/\sigma_{\max.}$, where $\sigma_{min.}$ and $\sigma_{\max.}$ are the applied minimum and maximum stresses, respectively) of 0.1 under a load-control mode, using a sinusoidal waveform with frequency of 10 Hz. At this stress range, the fatigue life of the BMG is around 1650 cycles, the detailed information of the fatigue test can be found in Ref. [21]. Afterward, utilizing FEI Focused Ion Beam instrument and following the traditional step-by-step concentric milling method [23,24], three micropillars with a top diameter of ~1 μm and height of ~2 μm were milled out from the polished surfaces of specimens sectioned from one as-cast BMGs and about 3 mm away from the fracture surface (in order to avoid the effect of fracture surface) of one fatigue-tested BMGs, respectively. Then, in-situ nanoECR measurements were performed on the micropillars with an applied constant voltage of 0.1 V and the load increasing to a medium level 1.2 GPa with loading, holding and unloading times all set to 10 s (as shown in Fig. 1(b)) utilizing the TI 950 Hysitron nanoindentation instrument equipped with a nanoECR module. Note that the yield strength of the BMG is ~2 GPa, and the medium stress level used here can ensure a fully Ohmic junction between the flat indenter and the micropillar.

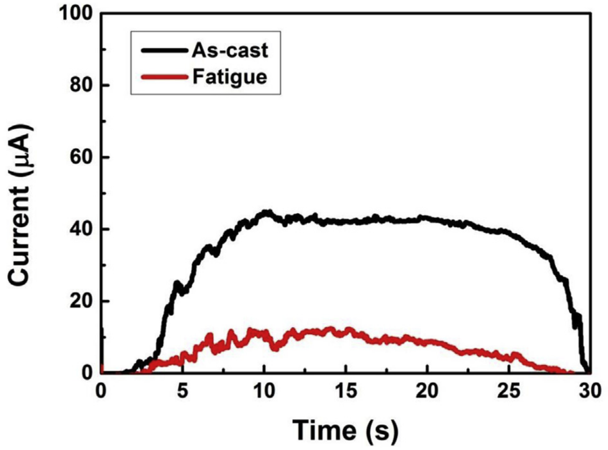

Fig. 2. The electric currents detected using nanoECR for the as-cast and fatigue-tested BMGs.

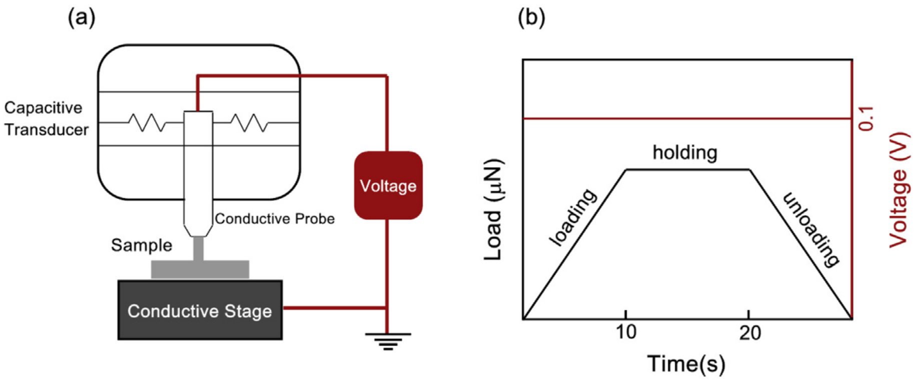

Fig. 1. (a) Sketch of the apparatus for nanoECR experiments. (b) The experimental load and voltage schemes for the nanoECR experiments.