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Nanoindentation study on the characteristic of shear transformation zone volume in metallic glassy films

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ABSTRACT

Based on the statistical data of first pop-in events during nanoindentation, the shear transformation zone (STZ) volumes of metallic glassy films with various compositions were estimated through the cooperative shearing model (CSM). The experimental results indicate that the STZ volume decreases with Poisson' ratio of the sample, which contradicts with previous observations using the rate-jump method. Moreover, the critical size for deformation mode transition (i.e. the localized shearing to homogeneous flow) at nanoscale could intrinsically be correlated with STZ volume.

Metallic glasses have great potential to be utilized as engineering materials for their excellent mechanical properties, such as large elastic limit, high strength and strong wear resistance [1–4]. However, the limited size and lack of ductility are currently the two critical bottlenecks hinder the development of metallic glasses for commercial applications. In order to overcome the catastrophic failure, and improve workability, tremendous research efforts have focused on revealing the deformation mechanism and establishing the structure-properties correlation in metallic glasses [5,6]. Owning to the original work of Argon [7], the deformation unit with a local rearrangement of atoms, also referred to shear transformation zone (STZ) has been widely applied to analysis the low-temperature deformation of metallic glasses. Being different from structure defect, STZ is defined by its transience, i.e., it can only be identified from the atomic structures before and after deformation. The details of the STZ evolution are mostly studied by computer simulations, on its shape, configuration and activation mechanism [8]. In recent years, following the cooperative shear model (CSM) by Johnson and Samwer [9], two distinct experimental methods have been developed to estimate the STZ volume [10,11]. Both measurements, namely rate-jump method and statistical method are conducted on the nanoindentation [12]. The measured STZ volume displayed a strong correlation with the ductility of bulk metallic glasses [10] and would be influenced by the structure state [13], temperature and strain rate [14]. However, the results determined by rate-jump method were in doubt. Though it was widely applied, rate-jump method has its limitations due to the fact that the STZ volume can only be calculated from this method if strain rate sensitivity (SRS) is greater than zero. It is well documented in literature that SRS can be negative for most of the BMGs at room temperature under quasi-static loading [14–17]. Bhattacharyya et al. revealed that pile-up in nanoindentation would significantly affect the value of SRS by rate-jump method [18], thus the questionable STZ volume.

For several years, size effect of metallic glasses have attracted numerous attentions that enhanced plasticity and strength were both validated in the nanoscale samples, accompanied with a deformation model transition from localized shearing to homogeneous flow [19–23]. Very recently, Volkert et al. [24] revealed that SRS kept constant among the samples with different sizes, indicating the same underlying mechanism in both localized and homogeneous deformation. This implies that the STZs play an important role on the apparent size effect of plastic deformation in metallic glasses. However, the exact response of STZs on the deformation mode transition, which critical size is material-dependent, is far from understanding. With this in mind, the seven kinds of thin metallic glassy films (~1.5 μm in thickness) were prepared. These are La–Co–Al, Cu–Zr, Cu–Zr–Al, Zr–Cu–Ni–Al, Ni–Nb, Co–Ta–B and W–Ru–B, respectively, providing clean and smooth surface for testing. Relying on spherical nanoindentation, STZ volume was carefully estimated by statistical analysis of first pop-in data. Here, we aim to study the general variation trend of STZ volume

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among various components, for further bridging the deformation mechanism and plastic characteristic in small-scaled metallic glasses.

The metallic glassy thin films were deposited on clean silicon wafer in a DC magnetron sputtering system at room temperature in pure argon gas. The power on the target was fixed at 200 W and working argon pressure was set about 1 mTorr. The sputtering time was sample-dependent that longer duration was applied on the target with higher melting point, enables the same thickness in each film. The thicknesses of the as-deposited films were measured by a surface profilometer (Dektak 150). By means of X-ray energy dispersive spectrometer (EDS) attached on the SEM, the chemical compositions of both films can be accurately detected. The as-deposited films were $La_{57}Co_{18}Al_{25}$, $Cu_{52}Zr_{48}$, $Cu_{44}Zr_{44}Al_{12}$, $Zr_{55}Cu_{15}Ni_{13}Al_{17}$, $Ni_{60}Nb_{40}$, $Co_{51}Ta_{9}B_{40}$ and $W_{40}Ru_{29}B_{31}$. The amorphous nature of the films was confirmed by X-ray diffraction with Cu K_α radiation (not shown here).

The nanoindentation experiments were conducted at constant temperature of 20 °C on Agilent Nano Indenter G200 with a spherical indenter, with an effective radius of 3.15 μm upon calibrating on standard fused silicon. The displacement and load resolutions of the machine are 0.01 nm and 50 nN, respectively. The as-deposited films could be directly applied on nanoindentation testing due to the ultralow roughness of metallic glassy film surface [25]. 120 tests with load-control mode were conducted on each sample to detect the relationship between the applied load and penetration depth (P vs. h). The loading rate was 0.5 mN/s. All the nanoindentation tests were carried out until thermal drift reduced to below 0.05 nm/s.

Fig. 1(a) shows the typical P–h curve of the Cu–Zr film at maximum load 20 mN. Here, the pop-in events with the scale of 1–2 nm can be observed. The initial loading curve can be completely fitted by the Hertzian elastic contact theory [26], given by:

$$P = \frac{4}{3} E_r \sqrt{R} h^{1.5} \tag{1}$$

where E_r is the reduced elastic modulus which accounts for that the elastic displacement occur in both the tip and sample, R is the tip radius. And the elastic constant of the film can be deduced by:

$$\frac{E_s}{1 - \nu_s^2} = \left(\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i} \right)^{-1} \tag{2}$$

where E and ν are the elastic modulus and Poisson's ratio, with the subscripts s and i represent the sample and the indenter, respectively. For commonly used diamond tip, $E_i = 1141$ GPa and $\nu_i = 0.07$. It should be mentioned that the Hertzian fitting line exactly deviates from the P–h curve at the position of first pop-in, as shown in the inset of Fig. 1(a), indicating the transition from elastic to elastic–plastic once the first pop-in emerges, which could be regarded as the onset of yielding in metallic glass [27]. The loads of first pop-in were plotted with the measurements, as shown in Fig. 1(b). It implies that the occurrence of yielding event of metallic glass was uniform and scattered during the nanoindentation, probably is due to the stochastic process of the shear banding nucleation. According to Bei's theory, the maximum shear stress τ_m underneath the indenter when the first pop-in event occurs represents the shear strength for the onset of plasticity [27]. For a spherical indenter, the τ_m happens at about half the elastic contact radius $a = \sqrt{Rh_e}$ right below the contact surface, given by:

$$\tau_m = 0.445 P_m \tag{3}$$

where P_m is the mean pressure given by:

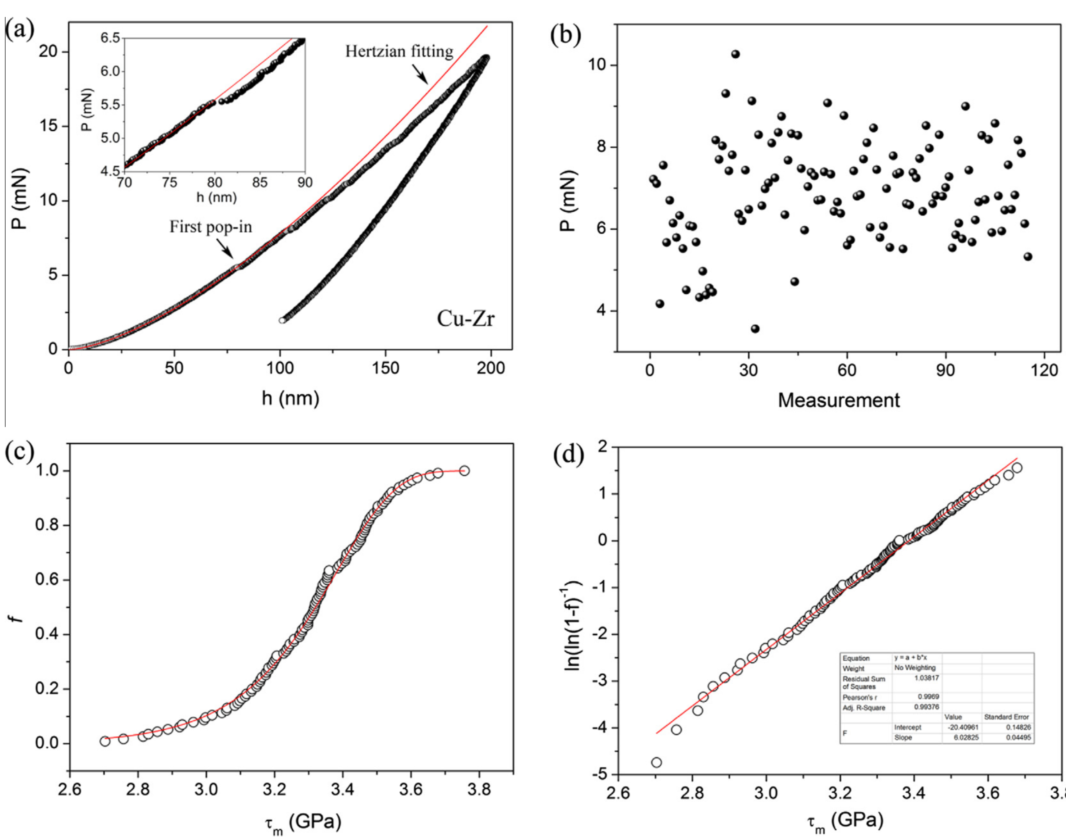

Fig. 1. (a) Representative load–displacement (P–h) curve for the Cu–Zr film at a peak load 20 mN and the first pop-in can be clear observed. The Hertzian fitting overlaps the elastic loading curve; (b) the load of first pop-in versus measurement; (c) cumulative probability distribution of the maximum shear stress τ_m at first pop-in; (d) The value of $\ln[\ln(1-f)^{-1}]$ versus maximum shear stress τ_m, linear fitting is employed to estimate the activation volume.

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