Effect of residual stresses on nanoindentation creep behavior of Zr-based bulk metallic glasses

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Abstract

The effect of residual stresses on the time-dependent deformation of a bulk metallic glass is investigated by the nanoindentation technique. In order to induce residual stresses, a beam sample was elastically bent and constrained in a steel ring. The upper side of the beam experiences the tensile residual stress, the lower side the compressive residual stress, and the central line nearly nil stress. Afterward, nanoindentation creep tests were performed on this stressed sample at room temperature. The creep rate is apparently higher on the tensile side, and remains lower and nearly fixed on the compressive side. The data were analyzed using the viscoelasticity iso-strain Kevin model. Individual strain contributions from anelasticity and creep were evaluated. The behavior can be explained by the joint influence of the residual stress and indentation loading.

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1. Introduction

Various bulk metallic glasses (BMGs) such as the Mg-based [1], Ti-based [2], and Zr-based amorphous alloys [3] have been well developed in past decades. Unlike crystalline materials, the non-crystalline structures of BMGs with short-range-ordered atomic configuration have shown unique mechanical and physical properties, such as excellent corrosion resistance [4], electromagnetic properties [5], large limit of elastic deformation [5], high strength [6], good fatigue properties [7], and so on. These properties suggest great application potentials as advanced structural or functional materials in various fields.

In most cases, to obtain micro- and nano-scaled mechanical properties of BMGs like hardness, yield strength and elastic modulus under localized deformation, the nanoindentation techniques have been applied [8,9]. Moreover, BMGs can be further investigated by nanoindentation using several testing modes such as bending, fatigue, and creep tests [10–15]. In recent years, nanoindentation testing has been employed for analyzing the time-dependent deformation mechanism in the Zr-based [16], Ti-based [17], and Fe-based [18] BMGs at ambient temperature. According to these results, the creep behavior appeared to be affected by the factors such as the applied load and loading rate. Under a higher peak load, a higher creep displacement could be induced. In addition, a higher creep displacement can also be achieved with increasing loading rate. These findings are in agreement with the results obtained from crystalline materials under similar test conditions [19–21]. On the other hand, the influence of residual stress on the mechanical response of BMGs, either originated from rapid cooling or subsequent loading in processing or in use, has not been fully researched.

Recently, Wang et al. [22] reported the variation of hardness in a BMG with tensile or compressive residual stress. They found that the onset of yielding largely decreased under tensile residual stress, but only slightly increased under compressive residual stress. In those studies, the Zr-based BMG beam was elastically bent and constrained in a steel ring. After detailed examinations, the hardness was found to strongly decrease over the tensile side and slightly increase in the compressive side under the elastic bending mode. The work-softening behavior in this Zr-based BMG was mainly attributed to the tensile residual stress. These experimental results were also consistent with the three-dimensional finite-element simulations [22].

In this study, we conducted creep tests on an elastically-stressed Zr52.5Al4.5Ti14.5Cu17.5Ni14.5 BMG sample and evaluated the time-dependent creep properties at various locations. The effect of residual stress on the creep behavior of this BMG is explored.
2. Experimental procedures

The Zr52.5Al10Ti5Cu17.9Ni14.6 BMG beam, with the dimensions of 3, 0.6, and 15.3 mm in width, thickness, and length, respectively, was cut from a 7 mm diameter rod (cast by suction casting) by electrical discharge machining. To enforce an applied stress in the specimen, this beam was elastically bent and constrained in a steel ring as our previous work [22]. This would impose a tensile residual stress on the upper side, compressive residual stress on the lower side, and nil residual stress along the central line. The lateral (cross-sectional) surface of the bent beam was upward mounted with epoxy and then mechanically polished to a mirror finish before subsequent testing.

The time-dependent creep tests were conducted on the specimen at ambient temperature by a MTS Nanoindentation XP system equipped with a Berkovich tip. Constant load indentation creep tests were carried out with a constant loading rate of 1 mN s\(^{-1}\) to different peak loads of 100, 200, 300, and 400 mN. To investigate the effect of loading rates, experiments at constant loading rates of 0.1, 1, and 10 mN s\(^{-1}\) were performed under the load control mode to the load of 400 mN. In the creep tests, the peak load was held for 30–500 s, and the displacement was measured at numerous locations across the beam vertical axis, \(y\), from the top to the bottom. The central neutral line is set as \(y = 0\), and the compressive and tensile sides are referred as \(y < 0\) and \(y > 0\), respectively.

3. Results and discussion

To confirm the existence of residual stresses in the bent BMG beam sample, we first carried out nanohardness measurements across the specimen thickness. Fig. 1 presents the variations of the hardness, modulus, and indentation depth from the compressive to tensile side of the BMG beam. The reference hardness (denoted as \(H_0\)) and elastic modulus (\(E_0\)) are the “unstressed” hardness and modulus obtained at \(y = 0\), which are 6.1 GPa and 101 GPa for the current sample. The hardness remains nearly constant from the compressive side to the central line (\(y = 0\)), but the hardness is seen to decrease on the tensile side up to \(\sim 20\%\) in Fig. 1(a); this result is consistent with that reported previously [22]. By contrast, the modulus values are basically scattered around 101 GPa; the dependence of modulus with the locations from the tensile to compressive sides is not pronounced, as seen in Fig. 1(b). The constancy of modulus is expected since modulus is a material intrinsic property. The indentation depth exhibits the opposite trend in Fig. 1(b) with respect to the hardness, being lower and relatively constant on the compressive side and gradually increasing on the tensile side.

Prior to each creep testing, the load was gradually applied using a constant loading rate. Fig. 2(a) shows such load-displacement nanoindentation curves under the load control mode with different peak loads from 100 to 400 mN at a given loading rate of 1 mN s\(^{-1}\). With increasing peak load, the displacement obviously increases. Serrated plastic flows (i.e., pop-ins) can be seen from the curves, associated with the shear-banding operations [23]. It is noted that the serration magnitude becomes higher at larger loads because the constant loading rate (for example 1 mN s\(^{-1}\)) would actually lead to a decreasing indentation strain rate for a higher load level [23]. And the lower strain rate would in-turns induce more apparent serrations. Here, we will not further discuss in details the serration phenomenon since it is not the focus of this paper.

The creep displacement curves as a function of holding time for different peak loads from 100 to 400 mN at a given loading rate of 1 mN s\(^{-1}\) are shown in Fig. 2(b). The creep displacement is seen to rapidly increase in the beginning and then to slow down during the holding period. Similar observations have also been reported in other BMGs, such as the Ce-based [24], Pd-based [13], Fe-based [18], Mg-based [14], and Ti-based [17] metallic glasses. Note that the origin of the time axis for the hold time (\(t = 0\ s\)) marks the onset of creep straining, excluding the loading time from 0 mN to the peak load. It is also noted that the total creep displacement increases with increasing peak load, as evident in Fig. 2(b).

Fig. 2(c) shows the indentation displacement curves at 400 mN with different loading rates of 0.1, 1 and 10 mN s\(^{-1}\). It is clear that the indentation displacement increases with increasing loading rate. The results are reasonable, since excess free volumes that can be induced during deformation. According to Spaepan’s free volume theory [25,26] the production rate of free volume during the deformation is proportional to the strain rate. A material indented at a higher loading rate will surely experience a higher strain rate. Therefore, the higher loading rate would create more excess free volume during the deformation. And, a BMG possessing a higher amount of free volume can be less resistant to the creep behavior. These results are consistent with the previous studies [16]. Similar data and curves were also collected from different locations in the test sample. For brevity, we do not display them here.

The viscoelastic (anelastic and viscoplastic) deformation can be empirically described by a combination of a linear spring and dashpot in an iso-strain condition, i.e. the Kelvin model, which is commonly used for describing the creep of polymeric materials, but can also be applied to metallic glasses, i.e.,

\[
h = J\left(1 - \exp\left(-\frac{t}{\tau}\right)\right) + kt, \tag{1}\n\]

where the first term is referred to the anelasticity and the second term to the creep, and \(J\) is the amplitude of the anelasticity.
contribution, $\tau$ is the characteristic time constant for anelasticity, and $k$ is the coefficient for creep contribution. In what follows, we are fitting the equation to extract the underlying time-dependent material properties under the influence of tensile or compressive residual stresses. From systematic fitting for all the data on various sample locations, loaded at various loads and loading rates, the datum bank can be used to analyze the influence of residual stress and indentation condition on the time-dependent creep behavior of the Zr-based BMG.

The variations of creep displacement as a function of indentation position at various loads (100–400 mN) and loading rates (0.1–10 mN s$^{-1}$) for different holding time periods (30–500 s) were systematically obtained. One of the examples is given in Fig. 3 in which data obtained across the sample under a peak load of 400 mN, loading rate of 1 mN s$^{-1}$, and a holding time of 500 s are summarized. On the compressive side, the residual stress only slightly affects the time-dependent displacement, varying around 8 nm. In comparison, a tensile residual stress can increase the displacement up to 12 nm, nearly 50% increment. For other load and loading rate combinations, this basic trend is universally observed. In the following, we will evaluate and directly compare the contributions from both anelasticity and creep.

From datum fitting and iteration, the variations of the $J$ coefficient and characteristic time constant $s$ at various sample locations, under a peak load of 400 mN, loading rate of 1 mN s$^{-1}$ for a holding time of 30 s are shown in Fig. 4(a). It is noted that the fitting is sensitive to datum scattering. Thus, measurements from longer holding period (>100 s) would result in a higher uncertainty for the extracted $J$, $s$, or $k$ values in Equation (1). Nonetheless, as indicated in Fig. 4(a), the anelasticity amplitude $J$ remains nearly constant at about 4.5 nm over the compressive side, but increases up to 8 nm over the tensile side, or about 80% increment. It suggests that the tensile residual stress can greatly accelerate the anelastic straining.

The variation of the characteristic time constant for anelasticity, $s$, is also plotted in Fig. 4(a). The time constant, again, remains basically fixed at $\sim$3.5 s over the compressive side and central line, but decreases down to $\sim$2 s over the tensile side. Since the time constant represents the time it takes to undergo anelastic straining for a portion of $1 - 1/e$ ($\sim$63%), thus the approximate time period for anelasticity contribution would be roughly $\sim$5.5 s for the compressive side and $\sim$3.2 s for the tensile side, suggesting that a tensile residual stress would shorten the time period to complete the anelasticity contribution. It is consistent with the notion that
there is more open space or free volume in the region with a residual tensile stress [22], so as to accelerate the anelastic displacement.

The creep contribution comes from the $k$ value in Equation (1). The variation of $k$ is shown in Fig. 4(b); it basically follows the trend of $J$, being nearly constant at $\sim 0.06$ nm/s over the compressive side and the central line, but increasing up to $\sim 0.12$ nm/s over the tensile side, or about 100% increment. This suggests that the anelasticity and creep contributions vary concurrently, both remaining nearly constant under a compressive residual stress, but increasing notably under a tensile residual stress. This is further confirmed by the plot in Fig. 4(c), where it can be seen that the anelasticity (A) and creep (C) components as a function of the holding time at $y/h = 0.3, 0, \text{ and } -0.3$ are basically varied in the same trend.

According to the work by Ye et al. and Dmowski et al. [27,28], the soft free volume zones would become active, inducing the anelastic deformation, even at a stress far less than the yield strength, i.e., before yielding. In our study, under the higher peak loads, the creep displacement signals would combine all the time-dependent deformation in both the elastic and plastic regimes. It is not easy to distinguish these two parts.

It is known that $\tau$ is correlated with temperature through an Arrhenius equation,

$$\tau = \tau_a \exp \left( \frac{\Delta F_a}{RT} \right),$$

(2)

where $\tau_a$ is the coefficient, $\Delta F_a$ is the activation energy for anelasticity, and $RT$ the usual meaning. Since we only perform creep tests at room temperature, it is impossible to calculate the activation energy. However, from the fact that $\tau$ in the compressive region is higher than that in the tensile region, the activation energy for the compressive side is anticipated to be higher than that for the tensile side. High temperature nanoindentation tests are currently underway to confirm this conjecture.

4. Conclusions

In summary, the time-dependent deformation (or creep) behavior at room temperature of an elastically bent Zr$_{52.5}$Al$_{10}$Ti$_5$. Cu$_{17.9}$Ni$_{14.6}$ is characterized by nanoindentation technique to study the effect of residual stresses. The creep displacement is observed to be only slightly affected by the compressive residual stress, but to increase appreciably up to almost 100% when the residual stress is tensile. The time-dependent nanoindentation displacement consists of two major parts, the anelasticity and creep. Through the datum fitting and analysis, it is found that the two parts contribute to the final displacement in a similar fashion when the sample is under residual stresses, namely, both remaining nearly constant under a compressive residual stress, but increasing notably under a tensile residual stress. However, the anelasticity part dominates the deformation initially but the creep part gradually takes over and eventually overwhelms the anelasticity part.

Acknowledgments

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