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On the strain rate sensitivity of plastic flow in metallic glasses

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ABSTRACT

The nanoindentation technique was employed to examine the strain rate sensitivity, m, and its dependence on the structural state of a Zr-based bulk metallic glass (BMG). The free volume content in the BMG was varied by examining samples in the as-cast (AC), shot-peened (SP), and structurally relaxed (SR) states. Hardness values measured at different loading rates and over a temperature range of 300–423 K as well as the strain-rate jump tests conducted in the quasi-static regime at room temperature, show that m is always negative. All the load–displacement (P–h) curves in this temperature regime exhibit serrated load–displacement responses, indicating that the shear band mediated inhomogeneous plastic flow governs deformation. Such localization of flow and associated softening is the raison d'être for the negative m. Significant levels of pile-up around the indents were also noted. The order in the average values of hardness, pile-up heights, and the displacement bursts on the P–h curves was always such that SR > AC > SP, which is also the order of increasing free volume content. These observations were utilized to discuss the reasons for the negative strain rate sensitivity, and its dependence on the structural state of metallic glasses. It is suggested that the positive values of m reported in the literature for them are possibly experimental artefacts that arise due to large pile ups around the indents which lead to erroneous estimation in hardness values.

1. Introduction

It is now well accepted by the metallic glass research community that plastic flow units in these amorphous materials are the shear transformation zones (STZs), which are clusters of atoms undergoing collective shear deformations under the influence of the applied stress [1–4]. STZs occur preferably in those locations of the material where free volume is more, i.e., atomic packing efficiency is relatively less. Above the glass transition temperature, T_g, of the alloy, profuse activation of the STZs occurs and the stress field associated with them relaxes instantaneously. This leads to homogenous flow of the glass [5]. Below T_g, on the other hand, plastic deformation at higher stresses manifests in the form of shear bands, which are flow bands within which plastic deformation localizes into [6]. Such localization of flow, and concomitant flow softening during deformation, in combination with the fact that there are no intrinsic barriers such as grain boundaries to the shear bands, results in near-zero ductility of bulk metallic glasses (BMGs). Consequently, considerable recent research has gone into understanding the origins and kinetics of shear bands with a view to ascertain as to how they control ductility and toughness of BMGs [7–10].

One of the important aspects of plastic flow, which has received relatively less attention as far as metallic glasses are concerned, is the strain rate sensitivity (SRS) that provides critical information about the plastic flow characteristics of a material. For quantifying the SRS of a material, the flow stress, σ, and the applied strain rate, $\dot{\varepsilon}$, are related through the equation: $\sigma = A\dot{\varepsilon}^m$; the parameter m embodies the SRS. For most engineering metals and alloys, m is positive, ranging between 0 and 0.1 at room temperature and can increase further at higher temperatures [11]. A positive and high value of m in combination with significant strain hardening often implies resistance to localization of plastic deformation and hence is preferred—especially in situations where superplasticity is desired—as it would act against necking during tensile deformation [11]. It is also possible to estimate the activation volume, V, and energy, Q, of the fundamental deformation processes (such as dislocation glide or climb), and in turn, develop an understanding of the underlying physics of plastic deformation from the value of m [1,12,13]. It is important to emphasize here that such estimation is only possible for cases where m is positive.

In the context of metallic glasses, flow above T_g is Newtonian, hence $m=1$ [14]. Below T_g, a survey of the available literature—almost all of which report experiments that were conducted at

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Table 1
Summary of the literature on the strain rate sensitivity in metallic glasses.

Material	Test conditions	Strain rate sensitivity	Reference
$Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$ (Vitreloy 105) and a composite of it reinforced with 6% graphite	Rate jump tests in compression with $\dot{\varepsilon}$ between 3.3×10^{-3} and 3.7×10^{-4} s^{-1}.	Negative ($\approx -0.001 \pm -0.0005$)	Dalla Torre et al. [15]
Vitreloy 105	Compression at various T (77–350 K) and $\dot{\varepsilon}$ between 3.33×10^{-5} and 0.2 s^{-1}.	Positive between 77 to 200 K and negative above 200 K (≈ -0.002)	Dubach et al. [16]
Vitreloy 105	Compression with $\dot{\varepsilon}$ between 2.34×10^{-3} and 1.87×10^{-1} s^{-1}	Negative (≈ -0.0026)	Jiang et al. [17]
$Pd_{40}Ni_{40}P_{20}$	Compression with $\dot{\varepsilon}$ between 3.3×10^{-5} and 2×10^{3} s^{-1}	Negative for both flow and fracture stresses	Mukai et al. [18]
Pd-20% Si	Compression at different $\dot{\varepsilon}$ (10^{-4}–10^{-2} s^{-1})	Fracture strength reduces with $\dot{\varepsilon}$, yield strength remains nearly invariant	Maddin and Masumoto [19]
$Zr_{57}Ti_5Cu_{20}Ni_8Al_{10}$	Compression over a range of $\dot{\varepsilon}$ between (10^{-4}–3×10^{3} s^{-1})	Failure stress increases with $\dot{\varepsilon}$	Hufnagel et al. [20]
Zr/Hf based BMGs	Dynamic compression	Fracture strength reduces with increasing $\dot{\varepsilon}$	Li et al. [21]
$Zr_{38}Ti_{17}Cu_{10.5}Co_{12}Be_{22.5}$	Quasistatic and dynamic compression over $\dot{\varepsilon}$ (10^{-5}–10^{3} s^{-1})	Fracture stress reduces with increasing $\dot{\varepsilon}$	Xue et al. [22]
Injection and in situ suction cast $Zr_{65}Al_{7.5}Cu_{17.5}Ni_{10}$	Compression, $\dot{\varepsilon}$ between 1.6×10^{-5} and 1.6×10^{-1} s^{-1}	Injection cast BMG shows negative m. In situ suction cast BMG does not show any SRS.	Xue et al. [23]

room temperature—suggests that m is either zero or slightly negative as seen from the summary presented in Table 1 [15–23].

However, Pan et al. [13], who employed the nanoindentation technique to measure the hardness, H, at different loading rates, report m to be positive for a wide variety of BMGs. We began the experimental work reported in the current paper with the purpose of examining as to how m varies with the structural state of a BMG, by employing the nanoindentation technique. Our objective, to start with, was to use the methodology suggested by Pan et al. [13] for analysis of the results and possibly relate parameters such as Q and V to the free volume content in the BMG. The structural state of a glass can be varied by either annealing the glass below its T_g or by subjecting it to intense plastic deformation [24,25]. These processes effectively change the free volume content in it; prior studies have shown that such a change can alter the mechanical properties such as pressure sensitivity and toughness of the BMGs markedly [25,26]. However, how SRS of BMGs is influenced by the free volume content is not known yet. Contrary to the initial expectations, our experimental results always yielded negative m values unlike that of Pan et al. [13]. In trying to find what could be the reasons for this discrepancy, we examine the effect of pile-up of material around the indenter during plastic flow and shear band mediated plasticity, on SRS of BMGs. In this process, we also show that the positive values of m reported by Pan et al. [13] are most likely experimental artefacts.

2. Material and experiments

Nanoindentation studies were performed on a fully amorphous Zr-based BMG, Zr41.2Ti13.8Cu12.5Ni10Be22.5 (Vitreloy 1, T_g=625 K), in three different structural states: as-cast (AC), structurally relaxed (SR) and shot-peened (SP). SR samples were obtained by annealing the AC samples in vacuum at 563 K (which is well below T_g) for 12 h whereas the SP samples were obtained by shot-peening the polished AC specimens with cast steel balls (280 μm mean diameter) as per the AMS-S-1316 standard. The thickness of the deformed layer in the SP samples is ~40 μm [27]. Further structural characterization details of the specimens in all the three states can be found in reference [25]. Prior calorimetric studies have shown that while structural relaxation leads to a reduction of free volume vis-á-vis the AC state, shot peening leads to an increase in the free volume [25].

Both load- and displacement-controlled nanoindentation experiments were conducted on mirror polished surfaces of BMG specimens in a Hysitron Triboindenter equipped with a Berkovich tip of 50 nm radius. In load-control mode, the peak loads, P_{max}, were maintained at either 9 or 250 mN, and the loading rates, dP/dt, were 0.4, 0.8, 4 and 8 mN/s for P_{max}=9 mN and 4, 8, 16, 30, 60 and 120 mN/s for P_{max}=250 mN. For each combination, 15 indentations were performed. Rate jump tests were performed in displacement-control mode to monitor how load, P, changes with increasing displacement rate, dh/dt. In these tests, the material is first loaded to h=50 nm at dh/dt=50 nm/s. Then, dh/dt is increased either to 150 or 500 or 1000 nm/s in three different sets of tests and loaded to h_{max}=150 nm before unloading. All the aforementioned tests were performed at room temperature (~300 K).

In addition, load-controlled tests at temperatures, T=373 and 423 K were also performed at the abovementioned dP/dt values with P_{max}=9 mN so as to investigate the effect of temperature on SRS of the BMG. The same nanoindenter equipped with high temperature heating facility was used for this purpose. The indenter and the sample were held for 1800 s at the desired temperature before the start of indentation. In all cases, the indents were scanned over an area of 20 μm × 20 μm, by using the indenter tip, after complete unloading so as to obtain images of the indentation impressions.

3. Results

Representative P–h responses obtained on the BMG in the three structural states with P_{max}=9 mN are displayed in Fig. 1(a). (The P–h curves obtained with P_{max}=250 mN are similar, and hence not shown here but are given in Fig. S1 of the Supplementary material (SI).) The curvature of the P–h curve during the loading segment is the highest for SR state, followed by AC and then SP states. The loading segments of the P–h curves (P_{max}=9 mN) for all three structural states show discrete displacement bursts, commonly referred to as pop-ins, as evident from the inset of Fig. 1(a). Images of the indents, particularly those made with P_{max}=250 mN showed considerable pile-up due to plastic flow around the indents. One such image is displayed as Fig. 1(b). This pile-up changes the effective contact area and hence the routinely-used Oliver–Pharr method for extracting H from the P–h curves would lead to errors [28]. A way to circumvent this problem and estimate H accurately is to use the Meyer's hardness [29], defined as $H=P_{max}/A_t$, where A_t is the imprint area of the indent, which is the true contact area, and is estimated from the scanned indentation impression images. Here afterwards, we only use Meyer's hardness in this paper.