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Effects of size on the strength and deformation mechanism in Zr-based metallic glasses

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ABSTRACT

We report results of uniaxial compression tests on $Zr_{35}Ti_{30}Co_6Be_{29}$ metallic glass nano-pillars with diameters ranging from ~1.6 μm to ~100 nm. The tested pillars have nearly vertical sidewalls, with the tapering angle lower than ~1° (diameter >200 nm) or ~2° (diameter ~100 nm), and with a flat pillar top to minimize the artifacts due to imperfect geometry. We report that highly-localized-to-homogeneous deformation mode change occurs at 100 nm diameter, without any change in the yield strength. We also find that yield strength depends on size only down to 800 nm, below which it remains at its maximum value of 2.6 GPa. Quantitative Weibull analysis suggests that the increase in strength cannot be solely attributed to the lower probability of having weak flaws in small samples – most likely there is an additional influence of the sample size on the plastic deformation mechanism.

1. Introduction

Emergence of size effects in mechanical properties is an intriguing phenomenon: reduction of material size to the order of its microstructure has been found to give rise to unique properties due to the engagement of fundamentally different physical processes at nano-scale (Arzt, 1998). For example, when the extrinsic sample dimensions are reduced below the minimum distance for dislocation multiplication in face centered cubic (fcc) metals, the conventional dislocation multiplication mechanism ceases and the materials are left in a dislocation-starved state, leading to fundamentally different from bulk mechanical response such as size-dependent strength and discrete plastic flow (Greer et al., 2005; Uchic et al., 2004). In the absence of well-defined plasticity carriers like dislocations in crystalline metals, metallic glasses show significantly different mechanical behavior from their crystalline counterparts such as high ceramic-like strength, increased elastic limit, and catastrophic failure under uniaxial loads at room temperature (Schuh et al., 2007). The plastic deformation of metallic glasses is generally known to occur through collective atomic rearrangements called shear transformation zones (STZ) (Argon, 1979; Falk and Langer, 1998), and their temporal and spatial correlation determines the deformation mode (Schuh et al., 2007). At elevated temperatures, the STZs are uniformly distributed under an applied stress, and the plastic deformation is homogeneous. At low temperatures, however, the STZs are densely populated within a narrow region, and the deformation quickly localizes into what is usually called a shear band (Schuh et al., 2007). Even though the specific mechanism of shear banding process remains controversial, the general consensus is that an embryonic shear band forms when the secondary STZs are activated near the primary STZs by the assistance of the free-volume, and catastrophic failure occurs when this embryonic shear band instantaneously propagates across the sample at the yield stress.

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While several research groups have tried to understand the effects of length scale on the deformation of metallic glasses and on shear band propagation, both theoretical and experimental reports are inconsistent and controversial (Ashby and Greer, 2006; Chen et al., 2009, 2010; Cheng et al., 2007; Dubach et al., 2009; Hofmann et al., 2008; Lai et al., 2008; Lee et al., 2007; Li and Li, 2006; Schuh et al., 2004; Schuster et al., 2007, 2008; Shan et al., 2008; Shimizu et al., 2006; Volkert et al., 2008; Wu et al., 2009; Zheng et al., 2007). For example, there exist at least three different theoretical estimations of the critical length scale, each based on a different phenomenology of the shear band mechanism. One such report compares the characteristic strain rate of shear banding with the magnitude of Debye frequency, allowing Schuh et al. (2004) to predict the characteristic shear band scale to be on the order of 50–500 nm, where the shear band becomes unstable and propagates instantly. In another study, by calculating the internal temperature increase due to frictional heating, Shimizu et al. (2006) predicted 100 nm as the maximum size of the embryonic shear band, below which shear localization would not occur. Finally, Ashby and Greer (2006), as well as Hofmann et al. (2008) analyzed the plastic zone size near the tip of an opening crack in a bending or tensile experiment and concluded that fast fracture does not occur in samples whose dimensions are below this plastic zone size, on the order of 1 μm–1 mm for most metallic glasses.

On the other hand, by utilizing the micro-compression approach now commonly used to study size-dependence in single crystals, several groups reported a correlation between the reduced size and several mechanical properties: yield strength (Chen et al., 2009, 2010; Cheng et al., 2007; Dubach et al., 2009; Lai et al., 2008; Lee et al., 2007; Schuster et al., 2007, 2008; Volkert et al., 2008), plasticity (Cheng et al., 2007; Shan et al., 2008) and transition to homogeneous deformation in compression (Volkert et al., 2008) and tension (Guo et al., 2007). However, these experimental results are also widely divergent. For instance, the strength of the metallic glasses has been reported to increase (Cheng et al., 2007; Lai et al., 2008; Lee et al., 2007), decrease (Volkert et al., 2008), or be independent of size (Chen et al., 2009, 2010; Dubach et al., 2009; Schuster et al., 2008). The characteristic size at which the transition from localized to homogeneous deformation occurs has also been found to vary substantially: from 400 nm (Volkert et al., 2008) to 200 nm (Chen et al., 2010) and 100 nm (Guo et al., 2007), or not observed at all down to 250 nm (Schuster et al., 2008) and 150 nm (Wu et al., 2009). These discrepancies can be understood, in part, by recognizing that experimental artifacts due to the imperfect pillar geometry and loading misalignment have a significant impact on the results of uniaxial compression experiments, especially in metallic glasses. Not accounting for the pillar tapering angle, for example, results in a non-trivial over/underestimation of the measured strength (Schuster et al., 2008; Zhang et al., 2006), and the less-than-flat pillar top topology has been found to suppress shear band formation and propagation (Wu et al., 2009). A detailed discussion on these and other geometric artifacts is given in Section 3.

In this study, we report the results of uniaxial nano-pillar compression experiments on the $Zr_{35}Ti_{30}Co_6Be_{29}$ metallic glass with the diameters ranging from ~1.6 μm to ~100 nm. In the course of this work we discovered the emergence of two unique and useful properties: (1) a yield strength increase from 1.7 GPa to 2.6 GPa as the pillar diameter is reduced below ~800 nm, remaining at that high value with subsequent diameter reduction and (2) once the lateral dimension is decreased to 100 nm, the formation of shear bands ceases, and the material exhibits homogeneous plasticity while maintaining its high strength, findings similar to the recently reported results of uniaxial tension experiments on nano-pillars made from the same bulk metallic glass (Jang and Greer, 2010).

2. Experimental procedures

A ~3 mm in diameter and 1 mm in thickness disk was cut from a $Zr_{35}Ti_{30}Co_6Be_{29}$ metallic glass rod, mechanically polished down to ~50 nm by Alumina powder paste and subsequently annealed at 325 °C for 1 h. The compression samples were fabricated on the polished surface of the disk using the FEI Nova 200 Focused Ion Beam (FIB). The cylindrical compression samples were fabricated through top-down milling by sequentially reducing the inner and outer diameters of the annulus patterns at the final ion beam condition of 30 kV and 10 pA. The aspect ratio (height/diameter) of the compression pillars was maintained at 4–5. We took special measures to ensure that the tapering angle, often associated with the use of the top-down FIB milling, is less than 1° for pillars larger than 200 nm in diameter, and less than ~2° for 100 nm and 200 nm pillars, as can be seen in Figs. 1 and 5. The pillars larger than 200 nm also have a nearly-perfectly flat top surface as can be seen in Fig. 1.

Uniaxial compression experiments were performed with a custom-made flat punch indenter tip in the Nanoindenter G200 (Agilent Technologies) and in the SEMentor, a custom-made *in situ* mechanical deformation instrument, where the deformation process can be observed throughout the experiment in a Scanning Electron Microscope (SEM). To eliminate the additional complexity of strain rate effects, all tests were performed at a constant nominal displacement rate (0.5–8 nm/s) controlled by a feedback algorithm, resulting in the global strain rate of 1.0×10^{-3} s^{-1}. Pre-calibrated load frame and support spring compliances were used to separate the specimen-only response from the measured raw load and displacement data. The contribution to displacement arising from the pillar-substrate contact was also removed using the Sneddon-based method utilized by Greer et al. (2005). By this methodology, the true pillar height on a rigid substrate can be determined by equating the measured contact stiffness and its theoretical value. Post-testing morphology and the evolved microstructure due to deformation were examined in SEM and Transmission Electron Microscope (TEM) (FEI Tecnai F30 and F20). TEM foils were prepared by lifting out a micron-sized lamella using the nano-manipulator (AutoProbe 200, Omniprobe, Inc.), and transferring it onto a Cu grid. Unlike pillars with larger diameters, the 100 nm pillar was fabricated directly on the lamella, compressed in the SEMentor while attached to the TEM grid, and subsequently analyzed.