Acta Materialia 154 (2018) 172–181

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Effects of notches on the deformation behavior of submicron sized metallic glasses: Insights from *in situ* experiments

R. Lakshmi Narayan [a, b], Lin Tian [b], Danli Zhang [b], Ming Dao [c, ***], Zhi-Wei Shan [b, **], K. Jimmy Hsia [d, *]

[a] Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA 15213, USA
[b] Center for Advancing Materials Performance from the Nanoscale (CAMP-Nano), Hysitron Applied Research Center in China (HARCC), State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an, Shaanxi 710049, PR China
[c] Department of Materials Science and Engineering, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139, USA
[d] Department of Mechanical Engineering and Department of Biomedical Engineering, Carnegie Mellon University, Pittsburgh, PA 15213, USA

ARTICLE INFO

Article history:
Received 7 March 2018
Received in revised form
15 May 2018
Accepted 17 May 2018
Available online 19 May 2018

Keywords:
Metallic glass
Notch sensitivity
Strength
Ductility
Nano mechanical test
In situ TEM

ABSTRACT

Reducing the size of metallic glasses (MG) to submicron or nanoscale levels improves their strength and ductility. However, there is no clear consensus in the literature regarding their mechanical behavior in the presence of a flaw or notch. In this work, quantitative tensile tests on notched submicron sized CuZr MG specimens were conducted inside a transmission electron microscope to study their deformation characteristics. Strength was found to be notch insensitive for shallow notched thick specimens, although reducing specimen dimensions and increasing notch sharpness enhances it by 14%. It was reasoned that the severity with which shear bands are geometrically constrained determines the strength and fracture morphology of notched specimens. Softening, accompanied with a transition to necking failure, occurs when the width of ligament that connects the notches is smaller than 80 nm. The competition between shear band propagation and plastic zone growth-mediated homogeneous activation of shear transformation zones was found to be responsible for this brittle to ductile transition. Current results provide unique insights into the various design aspects to be considered for reliable engineering of small scale components.

1. Introduction

Metallic glasses (MGs), in addition to possessing an impressive suite of mechanical properties like high strength, resilience and wear resistance [1], are easily processable [2–6], which makes them ideal materials for designing miniaturized components with intricate shapes in nano electromechanical (NEMS) devices [3,6]. A concomitant feature of designing intricate shapes is that the fabricated components, accidentally or intentionally, contain notches, incisions and sharp corners. These artifacts locally elevate or concentrate stresses when the component is mechanically loaded. Therefore, the applicability of MGs in NEMS devices will critically depend on their mechanical behavior, more specifically their strength and plasticity, in the presence of notches.

It has been observed that the *notch sensitivity* of strength, for most engineering materials, is a function of length scale. For instance, the tensile strength of bulk sized crystalline components is highly notch sensitive, wherein they become weaker in the presence of notches [7,8]. However, when the component dimension is reduced to the nanoscale, the strength and toughness become insensitive to notches or flaws [9–14]. This contrasting behavior has been attributed to the differences in the way plasticity, mediated by dislocations or grain re-alignments, interacts with the internal microstructures (e.g., grain boundaries and twins) and external geometry (sample dimensions), respectively, at the two extreme length scales [13,15,16]. In the case of MGs, a clear consensus on the notch sensitivity of tensile strength and plasticity has not yet been established at these extreme length scales. While experiments and simulations on bulk alloys by Kimura and Masumoto [17], Wang et al. [18] and Pan et al. [19] encourage the notion of increased nominal strength with increasing notch sharpness,

* Corresponding author.
** Corresponding author.
*** Corresponding author.
E-mail addresses: mingdao@mit.edu (M. Dao), zwshan@xjtu.edu.cn (Z.-W. Shan), kjhsia@cmu.edu (K.J. Hsia).

https://doi.org/10.1016/j.actamat.2018.05.041
1359-6454/

similar experiments by Flores and Dauskardt [20] indicate an inverse correlation between the two. Furthermore, Qu et al. [21,22] assert that MGs are notch insensitive materials. At the submicron and nano scales, molecular dynamics (MD) [23] and continuum simulations [24,25] predict higher strength and ductility of notched tensile MG specimens compared to their unnotched counterparts. Conversely, experiments conducted by Gu et al. [26] suggest that the strength and ductility of MG specimens decrease in the presence of notches.

One reason for this lack of consensus regarding notch sensitivity of MGs, compared to the well-established trends in crystalline materials, is the obscurity associated with the structure and deformation mechanisms in the former. In crystalline materials, owing to atomic periodicity, defects like dislocations, twins and interfaces can be easily identified as the principal flow units of deformation with well-defined characteristic length scales. However, in MGs, isolating flow units is a non-trivial process as its atoms are arranged non-periodically and the microstructure has no distinguishable features. Therefore, although theories and models involving shearing of loosely packed atomic clusters, also known as shear transformation zones (STZs) [27], have been developed, it is difficult to isolate or characterize them directly. Furthermore, the strains in MGs localize rapidly during deformation and the STZs develop into shear bands (SBs). To understand the notch sensitivity of deformation from the perspective of STZs, free volume and SB dynamics poses a unique challenge. This challenge is amplified by the lack of systematic experimental investigations at the nano scale, which is nevertheless difficult to perform in the first place. In this work, we conduct notched tensile experiments, on a Cu-Zr MG, inside a transmission electron microscope (TEM) using a quantitative mechanical testing system. The specimen dimensions and notch aspect ratios are systematically varied to examine subtle changes in strength and plasticity. By incorporating key features of the existing deformation models, the results are then analyzed and compared with predictions in literature.

2. Experimental methods

A melt spun Cu_{49} Zr_{51} MG was used in this study. Free-standing, double notched tensile plate specimens were fabricated using focused ion beam (FIB, FEI Nanolab 600). The final FIB trim and notch cut were performed using the ultra-mild milling conditions of 16 kV and ~1.5 pA. Since milling with the Ga ion beam can induce structural changes, which in turn can also potentially influence the mechanical behavior of the material, the composition of the specimens at different points was determined using energy dispersive X-ray spectroscopy (EDX) in the TEM. The Ga content in each specimen did not exceed 1%, suggesting that the milling conditions would have negligible influence on the results of this study. To further eliminate the effects of FIB induced damage, a nano mill (M1040, Fischione) was employed as a final step to slightly thin the specimen. Thickness and cross section area of the gauge section for each specimen was measured after fracture using a scanning electron microscope (SEM). For more details on preparing free standing tensile specimens, refer to Tian et al. [28,29]. The uniaxial tensile tests were carried out using the quantitative capability of a Hysitron PI95 TEM PicoIndenter. The tests were performed under displacement control mode at a nominal strain rate of 1×10^{-3}/s. The test specimen was gripped and aligned inside a JEOL 2100 F TEM operating at 200 kV. Owing to the non-trivial nature of designing and performing these experiments, only up to 3 tests could be repeated for each specimen condition.

Since the electron beam can generate heat and potentially influence the results of the mechanical tests, imaging, inside the TEM, was performed at an e-beam current density of 2×10^{-2} A cm^{-2}. Previous tests on the same material, with similar dimensions as used in this paper, have confirmed that the deformation characteristics are unaffected by the e-beam, at or below this current density [28,29]. In addition, since a large heat-conducting tungsten grip is in intimate contact with the small specimen, heating effects would be negligible. All the tests were recorded by a charge-coupled device camera (Gatan 833 CCD), placed inside the TEM, operating at a capture rate of 10 frames/s.

After conducting the tensile tests the fractured cross-section of each specimen was viewed inside the high resolution SEM. This was primarily done to measure the dimensions (thickness and width) of the unnotched ligament in each specimen. Note that since the length of all the ligaments are of the order of 20–150 nm, variations in their thicknesses are assumed to be negligible.

3. Results and discussions

3.1. Effect of specimen thickness

A representative image of a free standing double notched tensile specimen is displayed in Fig. 1(a). The total width, w_t, and gauge length, L, of each specimen is 300 nm and 1700 nm, respectively. In these tests, the notch height, r, and the notch depth, d, as illustrated in Fig. 1(b), are both 75 nm, and the un-notched ligament length, w_u, is 150 nm for every specimen. Only the specimen thickness t was varied from 200 nm to 400 nm. The schematic illustration and the *in situ* TEM image of the specimen-grip assembly is provided in Tian et al. [28,29]. The nominal stress, σ_n, is measured as the ratio of applied load and the initial cross section of the ligament that connects the notches, whereas the nominal strain, ε_n, is measured as the change in gauge length, ΔL, divided by L. Fig. 2(a) displays the representative stress-strain curves from tensile tests on the notched specimens (individual curves have been shifted relative to each other). Irrespective of the specimen thickness, the stress-strain curves are characterized by an approximately linear elastic portion, up to ε_n ~4.5–5%, followed by fracture.

Un-notched MG specimens with similar dimensions also exhibit a similar stress-strain response under uniaxial tension [28,29]. A higher strain to failure, ε_f ~4–5% in submicron sized specimens, compared to ε_f ~2% in bulk MGs, is a consequence of the former's smaller volume. Smaller volumes delay the onset of catastrophic failure because an embryonic SB (formed from activated STZs) is geometrically confined, thus preventing it from maturing into a macroscopic SB [28,30]. This allows locally stronger regions in the structurally heterogeneous MG to deform elastically even after the strain exceeds 2%.

It was also observed that the peak nominal stress, σ_s, of 200 and 300 nm thick specimens are comparable, and are as high as ~3.92 ± 0.14 GPa (See Fig. 2(a)). However, when t is increased to 350 nm and 400 nm, σ_s reduces to ~3.72 ± 0.17 GPa. Previous studies show that the strength of unnotched specimens with similar dimensions is also in the range of 3.5–3.9 GPa [28]. Therefore, the relative invariance in ε_f and σ_s, regardless of whether the specimen contains notches, *tentatively* suggests that the mechanical properties of submicron scale MGs are notch insensitive.

Next, we examined the fractured specimens to see if the mode of failure is also insensitive to the presence of notches. In unnotched tensile specimens, fracture occurs when a dominant SB nucleates at some surface undulation and propagates along a plane oriented at an angle, θ ~55-60°, to the loading axis [26,29]. While notched specimens with $t \geq 350$ nm appear to fail in the same manner as unnotched specimens, i.e. fracture occurs along a plane inclined at some angle to the loading axis (See Fig. 2(b) and Video S1a), the fractured side profiles of thinner specimens ($t \leq 300$ nm) are perpendicular to the loading axis (See Fig. 2(c) and Video S1b).