Quasi-static and dynamic deformation behaviour of Zr-based bulk metallic glass

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Abstract. Nano- and micro-indentation studies were carried out to characterise a plasticity mechanism through the evolution of localised shear bands that drive material's deformation at sub-micron length scale. Initial deformation of Zr-based bulk metallic glass (BMG) was investigated with nanoindentation tests using a spherical indenter. The indentation cycle reflects an elastic deformation with the yielding load of approx. 3 mN. For designed cycling indentation, hardening and softening phenomena were observed in nano- and micro-indentations, respectively. High-precision dynamic mechanical relaxation measurements were performed using a Dynamic Mechanical Analyzer (DMA), on decreasing frequency from 160 Hz to 0.1 Hz. A mechanical response of the BMG surface to a concentrated impact load was also studied. The obtained results indicated that the studied Zr-based BMG behaved as an elastic-perfectly plastic material at macroscale with discrete plasticity events at smaller length scales.

1. Introduction

Metallic glasses are a relatively new class of materials used in many functional and structural applications ranging from golf club to nano devices [1]. They were initially manufactured with rapid quenching techniques by exposing the molten alloys to very high cooling rates, ~ 10^6 K/s [1]. In the late 1980s, Inoue and co-workers [2-4] found a large number of new bulk metallic glasses in a variety of multi-component alloy systems comprising the rare-earth-based systems that have cooling rates less than 100 K/s and thicknesses reaching several centimeters. The first commercial BMG, $Zr_{41.2}Cu_{12.5}Ni_{10}Ti_{13.8}Be_{22.5}$ alloy, was fabricated by Johnson and Peker in 1992 with a critical cooling rate of 1 K/s. Metallic glasses are well-known for their distinctive mechanical properties, which can be exploited for a wide range of technological applications [2]. Unlike crystalline materials, amorphous metals lack an orientation long-range order. However, there is evidence of a crystal-like short-range order, which leads to unusual structural properties and non-conventional deformation mechanisms [6]. As a result, metallic glasses show excellent engineering properties such as high values of the Young`s modulus and elasticity limit, higher fracture toughness when compared to inorganic glasses and ceramics, high yield stress and high specific strength. Typically, inorganic glasses are brittle at room temperature, exhibiting a smooth fracture surface as a results of mode-I brittle fracture. BMGs, to the contrary, deform by localized shear deformation at room temperature, and are ductile in bending and rolling deformation. Under uniaxial tension or compression, BMG specimens undergo shear fracture immediately after yielding without appreciable macroscopic plastic strain.

For over a century, indentation has been conducted to probe the mechanical behaviour of materials for a wide range of engineering applications. The main reason for its ubiquitous use is its intrinsic experimental simplicity since indentation needs minimal specimens preparation and can be performed several times on single specimens, and can probe various volumes of materials via appropriate choice of load and tip geometry [7]. Instrumented nano- and micro-indentation on flat plates of BMG is ideal for the study of plastic deformation in amorphous materials because of limited global ductility in uniaxial testing conditions [8, 9]. In current study, purely elastic deformation of BMGs is analysed by using nano-indentation with a spherical indenter. Spherical indenters are finding popularity, as this type of indenter provides a smooth transition from elastic to plastic contacts. Instrumented indentation experiments are capable of showing some mechanical properties directly reflecting the inhomogeneous BMGs without the adverse effect of macroscopic casting defects. The frequency-dependent modulus is measured using Dynamic Mechanical Analyzer (DMA) to evaluate storage and loss moduli (E_1) and (E_2) and loss tangent$\left(\tan\delta = \frac{E_1}{E_2}\right)$. Additionally, the dynamic response of materials under concentrated impact loading at elevated rates is also investigated.

2. Experimental procedure

In this study, rectangular beam-shaped samples with a rectangular cross-section of a Zr-based metallic glass with nominal composition of $Zr_{48}Cu_{36}Al_8Ag_8$ (at %) with length of 40 mm, width of 10 mm and thickness of 2 mm were prepared at IFW Dresden, Institute for Complex Materials, Germany. The indentation tests were conducted with a Nano Test (Micro Materials Ltd.) system equipped with a spherical indenter with a radius of 5 µm and 50 µm. The samples with length of 10 mm, width of 10 mm and 2 mm thickness were adapted for these measurements. The samples were polished to mirror-like appearance with average roughness of around 5 nm. For the polishing process, the samples were initially mounted in epoxy resin (Epofix, Struers Ltd.) and then polished using a semi-automatic polishing machine (TegraPol-25, Struers Ltd.) with a rotating sample holder (TegraForce-5, Struers Ltd.) and an automatic feeding system for the polishing media (TegraDoser-5, Struers Ltd.). As a typical polishing cycle, mounted samples were initially ground using a polishing disc with grit size of 400 (35 µm), followed by 600 (25 µm), 1200 (15 µm), and finally 5000 (3 µm). Subsequently, polishing was continued by using a diamond paste consisting of particles of 1 µm in size. After finishing each polishing step and before starting the next one, the mounted samples were removed from the specimen holder, cleaned with deionised water and dried with a heat gun. When the final stage was completed, in order to get a clean surface without any contamination all the samples were subjected to ultrasound in deionised water for two minutes, sprayed with ethanol and dried using a heat gun. All the tests were conducted in a displacement control mode, and single loading as well as multiple unloading-reloading experiments were performed. The loading and unloading rates were 0.1 mN/s and 2 mN/s for single loading and the incremental loading-unloading experiments respectively, followed by the holding time of 1 s for all experiments. The maximum applied depth ranged between 5000 nm and 20000 nm for micro-indentation, and 200 nm and 2000 nm for nano-indentation. Three-point bending (3PB) was also used to determine the Young`s Modulus $(E)$ and Poisson`s ratio (ν) which are 86 GPa and 0.35, respectively. The load was applied by the mechanical test system operating with the displacement rate of 2 mm/min. Additionally, high-precision dynamical mechanical relaxation measurements were performed with DMA system using SD TA861.The experimental frequency range was from 0.2 Hz and 160 Hz at constant temperature. The elastic modulus can be derived from the measurements. A specimen in the shape of rectangular beam with length of 40 mm, width of 10 mm and thickness of 2 mm was tested in a 3PB configuration. The accuracy of frequency-dependent modulus measuring for DMA was better than 2%. For impact measurements, a pendulum-based impact was performed using the Nano Test system with a diamond spherical probe of radius $R = 5$ µm. The operating principles of the experimental set are outlined in [10].

3. Result and discussion

3.1. Elastic deformation in indentation

To investigate the initial elastic deformation, a maximum load of 6 mN was applied to the samples in the nano-indentation tests in this study, which is around the estimated yielding load of approx. 3 mN for the typical Zr-based BMGs corresponding to 5 μm spherical tip based on the work of Packard *et al*. [11]. Figure 1(a) and (b) show the response to nano-indentation loading, holding and unloading cycle

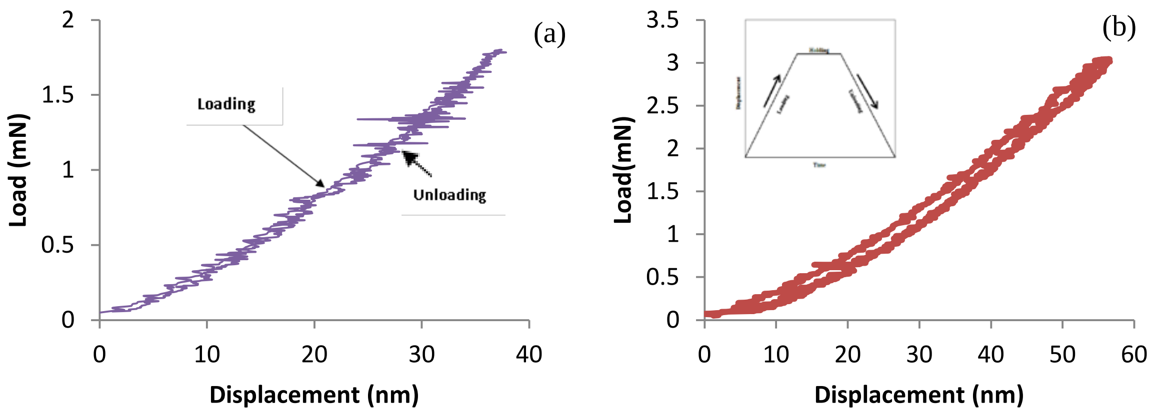

Figure 1. Typical load-displacement plot for Zr-based BMGs at loading rate of 0.1 mN/s: (a) purely elastic deformation; (b) initial plastic deformation

with maximum loading of 2 mN and 3.5 mN, respectively. It can be seen from Figure 1(a) that the loading and unloading responses are similar, with no obvious disparity between the two. This indicated an overall elastic response of the material under nano-indentation.

Next, we investigate the variation of hardness and elastic modulus with depth in the sample. The 'load/partial-unload' technique allows for hardness and modulus measurements at different indentation depths in the sample at the same location**.** A large plastic zone was formed under the indenter tip during indentation, commensurate with prior experiments [12-13]. The plastic zone contained a high density of shear bands, which is ideal for the investigation of deformation-induced hardening and softening effects [13]. The results show the dependence of hardness on the penetration depth. As shown in Figure 2(a) and (b), particular hardening and softening behaviors were observed when the multiple unloading-reloading tests were conducted during the indentation experiment with the same loading rate. In Figure 2(b), the onset of yielding upon each reloading is noted to take place always at a higher load than the load immediately before the previous unloading, apparently suggesting hardening effect. In addition, the hardness increased from 3.7 GPa to 5.7 GPa with a sharp enhancement in first 10 cycles and a slight rise in the following 10 cycles.

This also indicates that there is a work hardening behavior in the metallic glass at nanoscale due to increasing the hardness. However, the micro-indentation results (Figure 2(a)) indicate that there is large-scale softening associated with each load-unloading step, and hardness decreased from approximately 4.6 GPa to some 3 GPa. Such strain softening occurs in the development of shear transformation zones, which bring about an increase of free volume. At room temperature, the deformation-induced creation of free volume cannot be compensated by thermally-assisted structural relaxation and its accumulation results in formation of shear bands. Hence, this phenomena proves that plastic deformation at microscale in metallic glass is accompanied by dilatation, i.e. creation of free volume [13].

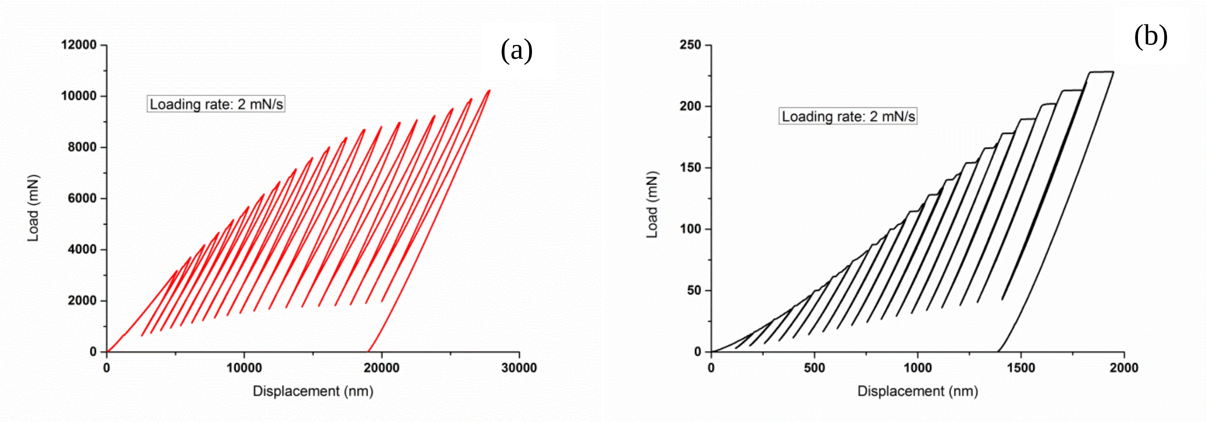

Figure 2. Indentation load-displacement curves of Zr-based BMG for incremental loading-unloading at loading rate of 2 mN/s (a) micro-indentation; (b) nano-indentation

3.2. Dynamic Mechanical Analysis (DMA)

Dynamic Mechanical Analysis (DMA) is one of the useful techniques to measure mechanical and viscoelastic properties as a functional of temperature, frequency and time when they are subjected to periodic stress cycles. The types of materials that can be analysed with this technique include thermoplastic, thermoset, metals, composites and ceramics. DMA provides quantitative and qualitative information such as the Young`s and shear moduli, damping characteristics, polymer structure and morphology, flow and relaxation behaviour. Figure 3 shows the frequency dependence of storage modulus (E_1), loss modulus (E_2) and damping ($tan\delta$) of the studied Zr-based metallic glass determined with DMA for a frequency range from 0.2 Hz to 160 Hz. It can be seen that the dynamic characteristics of the metallic glass are effectively independent of the frequency. The dynamic properties of the metallic glass are directly related to the microstructure and it can be concluded that the BMG behaves like a purely elastic material. The maximum value of storage modulus, loss modulus and the loss factor are 129 GPa, 3.57 GPa and 0.025, respectively.

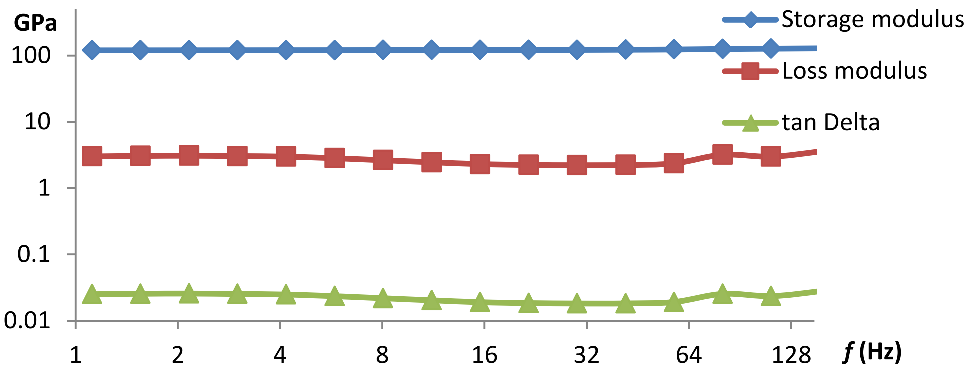

Figure 3. Frequency-dependence of storage and loss moduli of metallic glass measured with DMA at room temperature

3.3. Probe impact mode for low cycle fatigue

The probe impact mode can be used for investigating low cycling fatigue, work hardening and dynamic hardness. In this experiment, the pendulum is moved away from the specimen by a known distance and then released to produce a single impact. Successive impacts can be made at a single point until failure occurs. In most cases, an initial period of fatigue damage generation occurs, in which cracks develop and expand, but in which no appreciable increase in penetration depth in observed. Figure 2(a) and (b) illustrate that there is an evidence of strain hardening (as shown in nano-indentation results, Figure 2(b)) as there is a significant increase in displacement (penetration depth) in first 30 seconds, where the cracks coalesce, and a slight enhancement afterwards. Additionally, these results indicate that the Zr-based BMG was an elastic-perfectly plastic material since the material is linear-elastic and as the load increases to the yield point its stress remained constant and strain increased.

(a) (b)

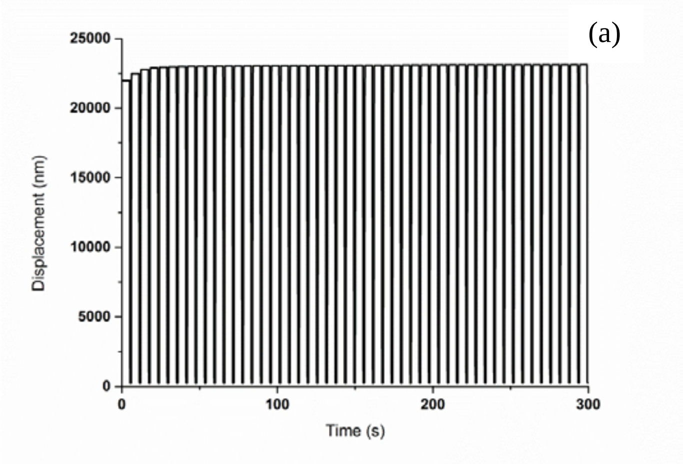

Figure 4. Typical displacement-time plot of impact results for Zr-based BMGs (a) for 300 s; (b) for 60 s. One impact every five second and failure of the metallic glass occurred after 30 s.

4. Conclusion

The initial stage of elastic deformation of BMG was investigated via nano-indentation test with a spherical indenter. It was found that the atomic structure has an effect on increasing initial elastic deformation. Strain-hardening and -softening were also observed for the Zr-based BMG during plastic deformation using controlled instrumented nano- and micro-indentation techniques. The dynamic elastic response of the BMG samples was studied at various frequencies at constant temperature showing that the dynamic characteristics of the metallic glass are effectively independent of the frequency. The indentation impact characterisation findings indicated that the BMG shows pure elastic behaviour.

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