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Scratch induced deformation behavior of hafnium based bulk metallic glass at multiple load scales

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ABSTRACT

The scratch induced deformation behavior of Hafnium based bulk metallic glass (BMG) at *micro*- and *macro*-load scales is reported in this study. The micro-scratch deals with a normal load range of 1000–8000 μN, whereas the macro-scale scratches are made with a normal load reaching up to 5 N. Increase in the load beyond 2000 μN changes the deformation mechanism from plowing to fracture and chipping. The plastic strain is mostly accommodated by shear band formation with a significant amount of free volume generation. A change in the strain rate helps in the nucleation of shear bands and prevents fracture and chipping in the case of micro-scratches made at ramp loading as compared to constant load. Higher strain rate and heat generation in the track causes increased strain hardening in macro-scratch, which could be attributed to increased probability of nano-crystallization. Complex shear band structure is evolved during scratching, which is attributed to the mixed type of loading, consisting of compressive normal load and lateral shear load across scratch-track.

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1. Introduction

Bulk metallic glasses (BMGs) are known for their high fracture strength, low elastic limit, and low plasticity. But, the shear band which forms during deformation introduces failure surfaces along oblique planes relative to the loading axis. This 'self-sharpening' behavior in BMGs occurs during dynamic impact as well. This phenomenon is the key for efficient performance as kinetic energy penetrators [1,2]. Hf based bulk metallic glasses (BMG) have been proposed as potential candidates for kinetic energy penetrators due to the high density of Hf, which makes the penetrators more effective [1,2]. A few studies have also been carried out on Hf based alloys to correlate their glass forming ability with deformation and mechanical behavior [1–8].

Bulk metallic glasses have also shown their potential as coatings in dry bearings for use in the outer space [9–11], hard facing alloys, wear resistant coatings for sensors, medical implants, and magnetic heads [9,12]. These applications introduce sliding contact on BMG surfaces, which demands an understanding of their tribological behavior. The machining of BMGs into the final components also uses a single-point tool where deformation conditions can be closely mimicked by scratch experiments [13]. Literature available on micro- and macro-scale scratch studies on Cu, Ni, Zr and Mg based BMGs reveals several aspects of deformation and failure [12–20]. Coefficient of friction (COF) increases with the applied normal load both at micro- and macro-scales [12,14–16]. But, scratch velocity does not have any effect on the COF [12]. The scratch (wear) resistance of BMGs increases with the hardness [17,19], but does not follow a linear relationship, as in Archard's equation [19]. A change in the wear mode is observed from rubbing and plowing to cutting with an increasing normal load for soft Mg-based BMGs [18]. In situ micro-scratching of Fe-based BMG revealed a transition from ductile plowing to shear band formation and serrated-chipping with an increasing load [20]. Thus, it is very difficult to predict tribological behavior for a certain BMG composition by deriving the similar principle from another BMG. Nevertheless, few commonalities are found in scratch behavior of BMGs. The deformation during scratching is mostly accommodated by shear bands [13,16,17,20]. Higher load causes formation of wider shear bands to accommodate larger deformation [13,20]. A higher scratch velocity leads to finer shear bands which are caused by the activation of multiple shear bands simultaneously to accommodate the applied strain quickly [17]. A study by Maddala et al. [9] on $Cu_{50}Hf_{41.5}Al_{8.5}$ (*closest BMG composition to the alloy used in this study*) reported tribological behavior in as-cast and annealed samples under sliding wear condition in ball-on-disk wear mode. Wear resistance is found to linearly increase with devitrification, due to formation of nanocrystallites [9]. The wide variation in tribological behavior of bulk metallic glasses is due to their unique structure, which is further complicated by different loading conditions.

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Tribological damage generally starts at lower (nano/micro) scale on the surface and aggravates to a macro-scale leading to failure. Thus, it is also important to analyze the tribological behavior at different length scales to fully understand the surface failure mechanism.

Based on the abovementioned scenario, the aim of the present study is to understand the scratch induced deformation behavior of Hf-based BMG, at different modes (*constant* vs. *ramp*) and magnitudes (*micro-Newton* vs. *Newton*) of loading. Microstructural alterations are correlated with the scratch based deformation behavior of Hf-BMG. The formation of shear bands as a result of loading type is analyzed to understand the specific arrangement of the shear bands at the bank/edge of the scratch groove. It is emphasized that scratch induced deformation behavior of Hf-based BMG has never been reported in the literature.

2. Experimental procedures

The Hf-based BMG alloy was prepared by mixing the 99.99% pure elements (Hf, Cu, Nb, Ni and Al) and arc melting them in a Ti-gettered argon atmosphere (~5×10^4 Pa). The ingot obtained was melted several times in order to enhance its homogeneity. Further, the arc melted ingot was re-melted and quenched into rod form (Ø = 3 mm) and was prepared by suction casting it into a copper mold. The alloy used for this study was of the following composition (atomic %): Hf-46.3; Cu-28.2; Ni-13.9; Al-6.2; and Nb-5.4 (referred as Hf-BMG hereafter).

X-ray diffraction (XRD) was carried out using Cu Kα (λ = 1.542 Å) radiation in a Siemens D-5000 X-ray diffractometer operating at 40 kV and 40 mA. A scan rate of 0.2°/min was used. The transmission electron microscope (TEM) sample was prepared by focused ion beam (FIB) milling technique. A Philips/FEI Tecnai F30 high resolution transmission electron microscope (HRTEM), operating at an accelerating voltage of 300 kV, was used to study the BMG sub-structure and capture the selected area diffraction pattern. Glass transition temperature T_g, and melting temperature, T_m, were measured using SDTQ600 (TA Instruments, New Castle, DE). The differential scanning calorimetry (DSC) curve was analyzed from 50 °C to 1200 °C at a scan rate of 20 °C/min in an argon environment.

Micro-scratch studies were carried out using the 2D scratching module of a Hysitron Triboindenter TI-900. A pyramidal diamond Berkovich probe with a 100 nm tip radius was used for making the scratches. Scratches were made with both *constant* and *ramp* normal loads. The constant loads used were 2000, 4000, or 8000 μN, whereas the ramped scratches were made at an increasing normal loading rate of 80 $\mu N \cdot s^{-1}$ in the range of 0–8000 μN. All scratches were 10 μm long and made with a lateral displacement rate of 1 $\mu m \cdot s^{-1}$. The surface profile of the scratch grooves was captured immediately after each scratch, using the scanning probe microscope (SPM) integrated in the triboindenter. A CSM Instrument NanoScratch Tester with a high load cantilever and a 50 μm cono-spherical diamond indenter was used to perform the macro-scratches in the ramp loading mode with a normal load range of 0–5 N. The scratches were 1.5 mm long and performed at a lateral displacement rate of 8.33 $\mu m \cdot s^{-1}$ and at an increasing normal loading rate of ~30 $mN \cdot s^{-1}$. Surface profile of the bank of the scratch was obtained using the SPM. Three or more scratches were made in each condition. All the error values reported in the plots and tables are based on the variations shown amongst these three scratches in each condition.

A JEOL JSM-633OF field emission scanning electron microscope, operating at 15 kV, was used to observe the macro- and micro-scratches. ImageJ software [21] was used for a quantitative analysis of the microstructural features.

3. Results and discussion

3.1. Structural characterization of the Hf-based BMG

Fig. 1a shows the XRD pattern of the Hf-based BMG alloy. The pattern shows a broad peak at a 2θ range of ~35–45°, which is the signature

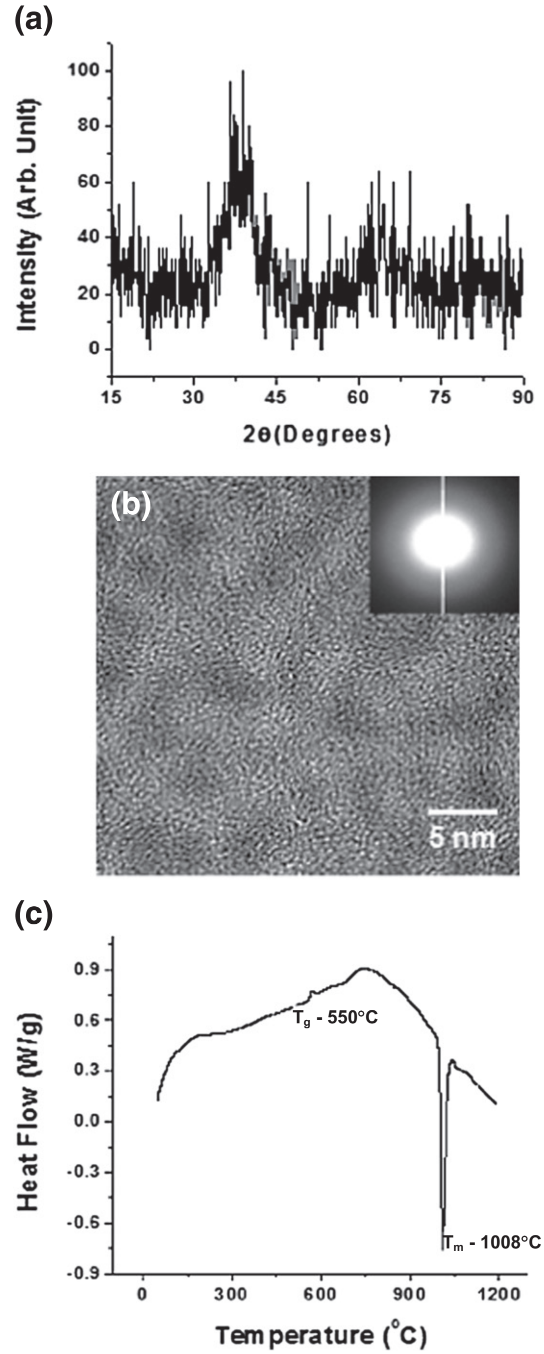

Fig. 1. (a) X-ray diffraction pattern of Hf based BMG; (b) HRTEM image with selected area diffraction pattern as inset and (c) DSC plot for Hf-BMG.

of amorphous structure at bulk scale. Fig. 1b provides high resolution TEM image of Hf-BMG at nano-scale. The absence of lattice fringes and crystalline structure in Fig. 1b indicates the amorphous structure. Selected area diffraction pattern, presented as an inset in Fig. 1b also shows the signature of amorphous structure without any diffraction spots. Fig. 1c presents the DSC curve with an exothermic signal followed by the glass transition. T_g is assessed to be ~550 °C. The devitrification temperature (T_X) is found to be 570 °C at the onset of the crystallization peak.

3.2. Micro-scratch behavior: constant load

Fig. 2 shows the results from the micro-scratch tests performed at constant normal loads of 2000, 4000, and 8000 μN. This is a composite

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