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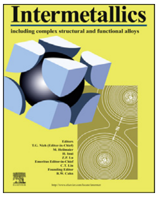

On the subsurface deformation of two different Fe-based bulk metallic glasses indented by Vickers micro hardness

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Bonded interface technique was employed to examine the nature of subsurface deformation during Vickers micro indentation in two iron-base bulk metallic glasses, $(Fe_{0.9}Ni_{0.1})_{77}Mo_5P_9C_{7.5}B_{1.5}$ (BMG-1) and $Fe_{40}Co_8Cr_{15}Mo_{13}Y_2C_{16}B_6$ (BMG-2). Quantitative information such as the subsurface deformation zone size was recorded for indentation loads ranging from 200 to 5000 gr. The results showed that the BMG-2 specimens had an average hardness value higher than those observed in the BMG-1 specimens. The trends of the hardness vs. indentation load in the BMG-1 specimens were found to be related to the pressure sensitive index, while in the BMG-2 specimens, the cracking events and deformation-induced creation of free volume were responsible for the hardness tendency change. Observations of the deformation zones indicated that they deformed noticeably through two types of semi-circular and radial shear bands and the density of the radial shear bands was much more in the annealed specimens compared to the as-cast specimens. The relaxed and partially crystallized BMG-2 specimens exhibited cracking, ripple-like pattern as well as cracking and fragmentation, respectively. A simplified $R = CP^{0.5}$ model was used to analyze the shear band zone size in the subsurface and specimens brittleness.

1. Introduction

Fe-based bulk metallic glasses (BMGs) have recently been considered as potential engineering materials due to their excellent properties [1]. The plastic deformation of BMGs is mostly brought about by the formation of the shear bands [1–3]. As a result, understanding the formation and propagation mechanisms of shear bands, deformation morphological characteristics, and the shear band zone size (SBZS) are the active areas of research on BMGs. The indentation test, extensively used in recent years, is considered to be an attractive method to investigate the mentioned properties of BMGs [4–12]. Bonded interface technique, with no significant effect on the contours of equal strain [4–12], is a very useful means to study the shear band characteristics in brittle materials such as ceramics, glasses, and metallic glasses.

Indentation test is an excellent means of conducting studies on the plastic deformation of BMGs. Being independent of the specimen size, easy to perform, and localized plastic deformation around the indenter are the main reasons that indentation is considered for the investigation of the deformation behavior of BMGs [4,5]. By employing the bonded interface technique, which offers the potential to directly observe the deformation morphology, the initiation and evolution morphologies of shear bands beneath the different indenters were systematically studied in certain BMGs [4–12]. Jana et al. [4,5], Tao et al. [9], Zhang et al. [11], etc. have conducted extensive studies on BMGs to study the shear bands features. The results indicated that the plastic zone beneath the indentation was mainly characterized by the semi-circular shear bands. In addition, several sets of radial shear bands were noticed under high loads and/or in the annealed BMGs specimen.

This work was initiated to examine the deformation morphology associated with the Vickers micro indentation of as-cast, annealed $(Fe_{0.9}Ni_{0.1})_{77}Mo_5P_9C_{7.5}B_{1.5}$ (BMG-1), and $Fe_{40}Co_8Cr_{15}Mo_{13}Y_2C_{16}B_6$ (BMG-2) bulk metallic glasses to identify the microscopic features resulting in the ductile [1] and brittle [13] behavior of the BMG-1 and BMG-2 specimens, respectively. Both interface via bonded interface technique and bulk Vickers micro indentation experiments were conducted. Additionally, the influence of the annealing treatments, inducing relaxation and partial

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crystallization, on the hardness and shear band morphology were investigated. The motivation for this study originates from the results of recent studies [1,13] demonstrating the ductile and brittle behavior of the BMG-1 and BMG-2 specimens. On the other hand, the result of the studies demonstrating that annealing the as-cast BMGs can markedly alter their mechanical behavior [14,15], is another motivational reason.

2. Materials and methods

The Fe-based multi-component master alloys with a nominal composition of $(Fe_{0.9}Ni_{0.1})_{77}Mo_5P_9C_{7.5}B_{1.5}$ (BMG-1) and $Fe_{40}Co_8Cr_{15}Mo_{13}Y_2C_{16}B_6$ (BMG-2) were prepared by the Vacuum Arc Remelting (VAR) in a water-cooled Cu-mold. The master alloy ingots were obtained using Fe–B (containing 15 Wt% B), Fe–P (containing 12 Wt% P), Fe–C (containing 5 Wt% C) pre-alloys, and commercially pure elements like Fe (99.9% purity), Ni (99.99% purity), Co (99.9% purity), Cr (99.9% purity), Y (99% purity) as well as Mo (99% purity). The master alloy ingots were re-melted four times for homogenization. The cylindrical rods of Fe-based bulk metallic glasses with a size of 2.5*60 mm were produced by water-cooled Cu-mold suction casting technique. A Philips X'Pert PRO x-ray diffractometer (XRD) using Cu-Kα ($\lambda = 0.1541$ nm) and a PerkinElmer differential scanning calorimeter (DSC) at a heating rate of 20 K/min were adopted to verify the structure of the specimens and the thermal behavior related to glass transition, crystallization, and melting events. To establish relaxation and partial crystallization, in a high vacuum tube furnace, the as-cast specimens were given isothermal annealing treatment at 653 and 690 K (BMG-1 specimens) as well as 783 and 843 K (BMG-2 specimens) for 60 and 240 min, respectively. Then the BMGs specimens were cut into two halves and each specimen was polished to a mirror finish prior to bonding the polished surfaces using high strength glue. The bonded specimens were mounted and subsequently their top surfaces were ground and polished to a mirror finish. Vickers micro indentation tests were performed along the bonded interfaces as well as away from it, in the bulk on a Wolpert D-6700 tester. The loads were selected within the range of 200–5000 gr with a holding time of 30 s. After the tests, the specimens were un-mounted and the bonded interfaces were opened by dissolving the glue in an acetone bath; then, the specimens were cleaned with ethanol in a supersonic cleaner. A Philips Scan MV 2300 scanning electron microscope (SEM) and a ZEISS optical microscope (OM) were utilized to observe the microstructural features and the deformation morphology beneath the indentation imprint. The density measurements were carried out at room temperature according to the Archimedean technique with the electronic balance of ±0.001 gr precision.

3. Results

The amorphous state of the as-cast BMG-1 and BMG-2 specimens was characterized by XRD and DSC techniques. Fig. 1a shows the XRD patterns for the as-cast and the annealed specimens. All the specimens exhibited broaden diffuse peak without any detectable crystalline peaks on the XRD patterns, demonstrating the amorphous state before and after the annealing of the BMG-1 and BMG-2 specimens. Annealing the as-cast specimens as relaxation and partial crystallization processes do not change the XRD patterns significantly, the broad peak appears to get sharper with no crystalline peaks observed.

Fig. 1b shows the DSC curves of the as-cast BMG-1 and BMG-2 specimens. It can be seen that the two BMGs exhibit the distinct glass transition temperatures (T_g). Four exothermic peaks can be found on the DSC curve of the as-cast BMG-1 specimen, while three-step crystallization occurred in the as-cast BMG-2 specimen. The BMG-1 and BMG-2 specimens show small endothermic and exothermic events at 922 K and 1150 K, respectively; it may be attributed to the transformation from metastable crystalline phase(s) to stable crystalline phase(s) at the above temperatures.

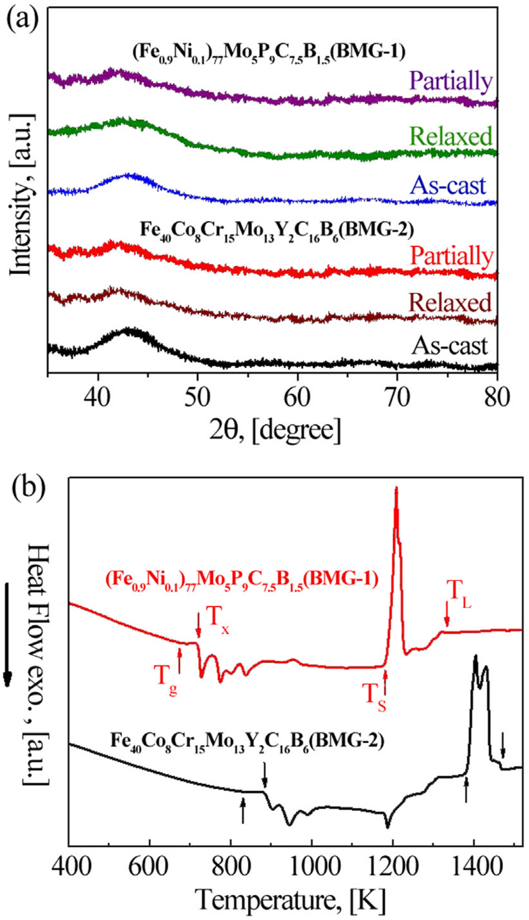

Fig. 1. Characteristics of the BMG-1 ($(Fe_{0.9}Ni_{0.1})_{77}Mo_5P_9C_{7.5}B_{1.5}$) and BMG-2 ($Fe_{40}Co_8Cr_{15}Mo_{13}Y_2C_{16}B_6$): a) XRD patterns of the as-cast and annealed specimens and b) DSC curves of the as-cast specimens. The Relaxation treatment for the as-cast BMG-1 and BMG-2 specimens was done at 653 and 783 K for 60 min, respectively. The as-cast BMG-1 and BMG-2 specimens were given iso-thermal partial crystallization treatment at 690 and 843 K for 240 min, respectively. T_g, T_x, T_S and T_L are glass transition, onset of crystallization, solidus and liquidus temperatures, respectively.

Fig. 2 shows the hardness measured on the top surface away from the interface at different loads and annealing conditions for both the BMG-1 and the BMG-2 specimens. The as-cast BMG-1 specimen (Fig. 2a) exhibits an increase of hardness with an increase in load. On the other hand, the hardness strongly depends on the load. For the annealed BMG-1 specimens, the tendency of hardness change is similar to the as-cast specimen. Hardness values also increase as loads increase below 500 gr and change smoothly for loads above 500 gr, but the values for the annealed specimens are higher than those for the as-cast specimen. This tendency to hardness change was also reported by Keryvin [16], Yu et al. [17], and Tang et al. [18]. For the BMG-1 specimens at all the load levels, the shear bands were observed around all the bulk indentations occurring in the form of semi-circular shear bands that appeared to emanate from the edge of the indentation (inset image in Fig. 2a).

For the as-cast and annealed BMG-2 specimens, a sharp decrease in the hardness with increasing load is observed (Fig. 2b). Typical indentation images on the top surface away from the interface (inset images in Fig. 2b) show cracks onset in the as-cast