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Postprint

This is the accepted version of a paper presented at *Materials Research Society (MRS)*, 2011.

Citation for the original published paper:

Masood, A., Ström, V., Oak, J., Belova, L., Ågren, J. et al. (2011)

The observation of surface softening in Fe-based metallic glass.

In: *Mater. Res. Soc. Symp. Proc. Vol. 1300* (pp. 5-). Materials Research Society

<http://dx.doi.org/10.1557/opl.2011/399>

N.B. When citing this work, cite the original published paper.

Permanent link to this version:

<http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-101828>

The observation of surface-softening in Fe-based metallic glass

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ABSTRACT

Depth sensing nano-indentation investigations have been performed to determine the radial dependence of the hardness through the cross section of a Fe-based bulk glassy rod. We have found the hardness of the material decreases along radius from the center to the outermost surface. This phenomenon is attributed to the ‘cooling rate induced surface softening’. Furthermore, a significant change (~15 %) in elastic modulus is noticed along the radius as well.

INTRODUCTION

The study of mechanical properties of bulk metallic glasses (BMGs) is an intriguing field of research from fundamental as well as application point of view [1-17]. One of the major areas of active pursuit being carried out is how to improve the ductility of these materials, an essential factor for structural applications [2-5, 7]. A recent breakthrough in this direction is the discovery of induced surface softening in BMGs. It is emphasized that the surface softening by residual stresses induced by shot-peening may improve the ductility in amorphous glassy materials [7]. Such surface softening can also be obtained by thermal tempering [7, 8]. However the occurrence of such a phenomenon is extremely rare in the case of BMG because of their high thermal conductivity. In the present letter, we report the manifestation of surface softening by Fe-based BMG.

Among the different classes of BMGs, the Fe-based materials are particularly interesting because they exhibit high strength and excellent soft magnetic properties along with very high saturation magnetization [1, 6-10]. The lack of ductility hinders them from different structural applications. Although there are many studies regarding the magnetic properties of these materials, the mechanical properties of these materials are not well investigated. In the present work, we focus on the mechanical properties of Fe₇₁B₂₃Nb₆ bulk metallic glass. Our study indicates that, the hardness and the elastic modulus of the material considerably decreases at the outermost surface in comparison to the central part indicating the occurrence of ‘surface-softening’. This kind of surface softening may originate due the different cooling rate at central and outer part of the rod during casting. Earlier experimental studies indicate that FeBNb system has some unique network like structure which enables it to have high mechanical strength along with appreciable compressive plasticity [15]. Therefore it is considered as an excellent material for structural application. The current work is to gain further insight into the mechanical properties of the FeBNb system.

EXPERIMENTAL DETAILS

The glassy amorphous rod of well known BMG composition of $\text{Fe}_{71}\text{B}_{23}\text{Nb}_6$ of diameter ~ 0.5 mm was fabricated from the ingot of the alloys by conventional Cu-mold technique. The ingot of the alloy was prepared by induction melting of the constituent elements with purity $\sim 99.99\%$. The induction melting was carried out in the presence of highly pure Ar gas to rule out the any possibility of oxidization. The prepared rod was characterized by x-ray diffraction (XRD) technique (using Cu-K α radiation) as well as the magneto-thermo-gravimetry analysis (MTGA). The XRD study indicated the amorphous nature of the sample. In addition, magnetic- thermo-gravimetric (MTGA) measurements are used to confirm the absence of any trace of crystalline phase in the material.

For studying the mechanical properties, nano-indentation experiment was carried out on the sample. We have used the nano-indenter (NANO INDENTER[®] II, nano instruments Inc., Oak Ridge, TN) with Berkovich diamond tip. For such study, the sample was mechanically polished to get the geometry required for the experiment as shown in Fig. 1. At the start of the experiment, the load frame compliance and diamond tip area calibration were performed with the help of fused silica sample provided by the instrument manufacturer. All indentation experiments were performed with force control mode at the loading rate of 25mN/sec and keeping peak load at 300 mN. Two hold segments of 20 sec and 2 sec were taken for each load-unload cycle at maximum load and at the 80% of unload respectively. The central axis along the length of the rod is considered as a reference line. The positions of indentations were also indicated in Fig. 1.

Figure 1. The sketch of the segment of the rod, which was used for nano-indentation experiment. The positions of the indentations are indicated with X- mark.

RESULTS AND DISCUSSIONS

The XRD and MTGA results confirm the amorphous nature of the sample. The results obtained from the nano-indentation experiments were analyzed on the basis of Oliver-Pharr's approach to determine the contact depth at maximum load (h_c), contact stiffness (S), the contact area (A_{o-p}) [16]. The elastic modulus (E) and hardness (H) were calculated by taking the average of the values in each row by using those parameters. The typical load-displacement curves for the indents at the reference line and outermost position of the rod have been presented in Fig. 2.

Figure 2. The typical load-displacement curve for the indents at reference line and at the outer most position of the rod.

We have used atomic force microscopy (AFM) technique to visualize the imaging profile of all the indents and also subtracted the ‘pile-up’ contribution to the obtained contact area from the indentation experiment following the method proposed by Kese et al. [17]. The corrected values of hardness obtained from the nano-indentation experiment are in good agreement with the values measured by micro-indenter for the same sample. A typical AFM image of an indent along with the possible pile-up contribution has been shown in Fig. 3. The variation of E and H (before and after subtracting ‘pile-up’ contribution) with the positions of indents from the reference line is shown in Fig. 4. From Fig. 4 it is evident that, in the indentation experiment, the pile-up material can introduce significant errors in the calculation of H and E . Therefore that correction is absolutely necessary in order to obtain the actual values of those parameters.

Figure 3. AFM image of the indents which shows the pile ups around the indents.

Both the quantities (E and H) are found to be decrease along the radius from the reference line (Fig. 4). In this study the value of H is the lowest at the surface, which can be considered as an indication of surface-softening of the material as reported for other systems [8]. The different values of E at different positions points out that the local atomic arrangements at different locations are probably not similar although overall structure of the sample is amorphous. In addition the elastic modulus is found to decrease by about 15 % from the reference line to the outer surface of the sample (Fig. 4).

Figure 4. Variation of elastic modulus (E) and hardness (H) along the radius of the rod from the reference line. (a) Shows value of E before and after correcting contribution of pile-up material. (b) Shows value of H before and after correcting the contribution of pile-up material.

Cooling rate during casting plays a vital role in the formation of BMG [8]. During casting, even a small difference in cooling rate inside the material can lead to variation in local atomic arrangement resulting in change of mechanical properties [8]. Recently, Liu et al. [8] have shown that the elasticity and hardness decrease along radius in the case of Zr-based BMG rod in the same manner as our observation in the present study. They have argued that because of the maximum cooling rate at the surface, the hardness as well as the elastic modulus becomes considerably less in comparison with the central part (cooling rate induced surface softening) [8]. The surface softening in the present case could also originate as the effect of the higher cooling rate at the surface compared to central part. Another recent study by Zhang et al. [7] points out that the induced surface softening can be beneficial in dramatic enhancement of ductility. They have produced surface softening in Zr-based BMG Vitreloy 1 by shot-peening, which improves the ductility significantly. In contrast to that case, surface softening in the present case occurs spontaneously as the consequence of higher cooling rate at the surface compared to central part. However the percentage of decrease in hardness at surface is almost of the same order of magnitude as observed in the case of Zr-alloy.

CONCLUSION

We have studied the radial distribution of the mechanical properties of a BMG material of nominal $\text{Fe}_{71}\text{B}_{23}\text{Nb}_6$ composition. The cooling rate induced surface softening is observed for this material. In addition, we see a significant change in elastic modulus along the radius. This phenomenon indicates that the local atomic arrangement is highly influenced by the cooling rate during casting process.

ACKNOWLEDGEMENTS

Authors are thankful to Dr. K. O. Kese and Dr. T. Tamaki and Dr. A. Biswas for intense discussion. The work is financially supported by Hero-M Excellence Centre at KTH, Sweden. AM is thankful to HEC, Pakistan for providing doctoral fellowship.

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