7.1 EFFECTS OF ION IRRADIATION ON BAM-11 BULK METALLIC GLASS –

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OBJECTIVE

The goal of this study is to investigate the radiation behavior of the bulk metallic glass BAM-11 and to determine if it is a viable candidate for high-radiation structural applications in fusion systems.

SUMMARY

Bulk metallic glasses are intriguing candidates for structural applications in nuclear environments due to their good mechanical properties along with their inherent amorphous nature, but their radiation response is largely unknown due to the relatively recent nature of innovations in bulk metallic glass fabrication. Microstructural and mechanical property evaluations have been performed on as-cast and heat-treated $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ bulk metallic glass (BAM-11) irradiated with 3 MeV Ni^+ ions to 1 and 10 dpa. TEM showed no evidence of radiation damage or crystallization following ion irradiation, and changes in hardness and Young's modulus were 10-15%. In addition, irradiation-induced damage was seen to saturate at or below the 1 dpa dose level.

PROGRESS AND STATUS

Introduction

Amorphous metallic glasses were first synthesized in the 1960s [1] and have since received considerable scientific attention due to their appealing properties, including their good thermal conductivity, high strength, good ductility, and corrosion resistance [2-4]. Metallic glasses are an intriguing candidate for use in radiation environments due to their lack of crystalline structure, which prohibits the formation of conventional radiation defects such as vacancy-interstitial Frenkel pairs and dislocation loops that occur in crystalline solids. A more thorough review of radiation effects in bulk metallic glasses is available in the previous semi-annual report (for period ending December 31, 2013).

In the first portion of this study, we investigated ion irradiation effects in the bulk metallic glass BAM-11 (Composition: $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$). Specimens were irradiated with 3 MeV Ni^+ ions to 0.1 and 1.0 dpa at room temperature and 200°C. Nanoindentation hardness and Young's modulus both decreased by 6 to 20% in samples irradiated at room temperature, with the sample irradiated to 1.0 dpa experiencing the greatest change in mechanical properties. However, no significant changes in properties were observed in the samples irradiated at 200°C, and transmission electron microscopy showed no visible evidence of radiation damage or crystallization following ion irradiation at any of the tested conditions.

Here, we continue the work on this bulk metallic glass and investigate the effects of higher irradiation doses on the material and the effects of pre-irradiation heat treatments.

Experimental Procedures

A $Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ alloy (BAM-11) was fabricated by arc melting in an argon atmosphere using a mixture of base metals with the following purities: 99.5% Zr, 99.99% Cu, 99.99% Ni, 99.99% Al, and 99.99% Ti. The alloy was then remelted and drop cast in a Zr-gettered helium atmosphere. The rod was then cut into sections of 8 by 3 by 1 mm and mechanically polished to a mirror finish. Some samples were given a 48 hour heat treatment at 300°C, well below the material's glassy transition temperature of 393°C [5].

After fabrication and heat treatment, BAM-11 specimens were ion irradiated with 3 MeV Ni^+ ions at perpendicular incidence. Samples were irradiated to fluences of 4.2 x 10^{14} and 4.2 x 10^{15} ions/cm^2, or peak damage levels of 1 and 10 dpa at the University of Tennessee/ORNL Ion Beam Materials Laboratory (IBML). Ion range and damage event profiles were used to determine the fluence to dpa conversion and were generated by the SRIM software using an average displacement energy of 40 eV [6]. The most common ion range depth was calculated to be 1.36 µm, with implanted Ni concentrations reaching 5.5 x 10^{18} atoms/cm^3 at this depth. Damage levels at the sample surface were calculated to be about 40% of peak levels.

Post-irradiation microstructural characterizations of the irradiated bulk metallic glass specimens were performed via transmission electron microscopy (TEM). TEM foils were fabricated using an FEI Quanta Dual-beam focused ion beam (FIB)/SEM with a final thinning step of 2kV Ga^+ ions at a glancing angle of about 4° in order to minimize ion beam milling damage. Samples were then analyzed in a Phillips CM 200 TEM operating at 200 kV using the techniques of bright field (BF) imaging, selected area electron diffraction (SAED), high resolution TEM (HRTEM), and x-ray energy dispersive spectroscopy (EDS) performed in scanning TEM (STEM) mode.

Hardness and elastic modulus were measured using an MTS XP nanoindenter, with the indentations performed normal to the mechanically polished control and irradiated surfaces [7]. All tests were performed using a Berkovich diamond indenter (3 sided pyramidal tip) in continuous stiffness measurement mode at a constant indentation rate [(dP/dt)/P] of 0.05/s with a maximum applied load of 15 mN, resulting in a maximum indentation depth of ~350 nm. For statistical purposes, each sample was indented a total of ~16 times in different locations and the averages of those results are reported here. Hardness and elastic modulus were calculated using the Oliver and Pharr method [8,9]. The area function of the tip and machine stiffness of the nanoindenter were calibrated by indenting on a standard fused silica sample [10].

Mechanical Properties

Hardness and elastic modulus data generated through nanoindentation tests are shown as a function of indenter depth in Figure 1. Average data for an indenter depth of 200 nm, which represents a compromise in depth to minimize surface effects while also minimizing contributions to the data from unirradiated regions, is presented in Table 1.

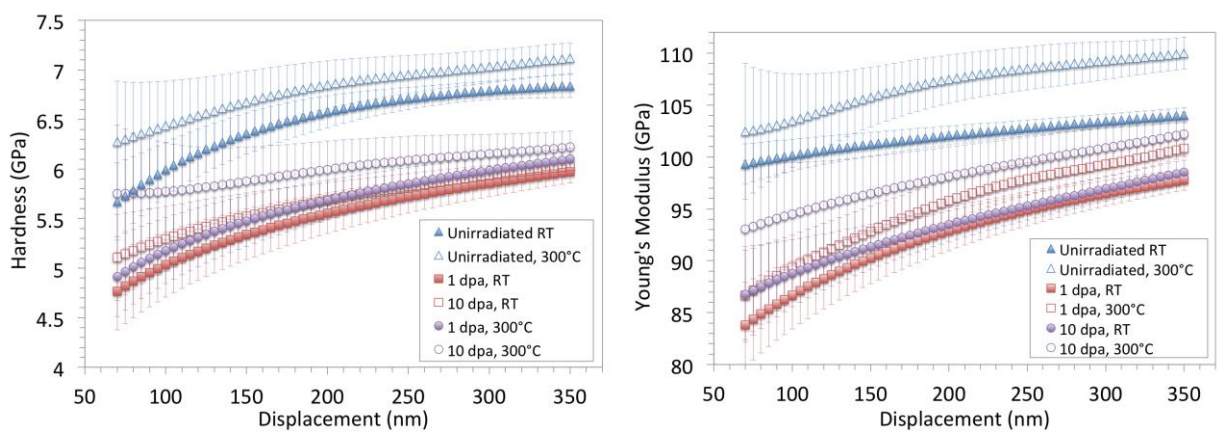

Figure 1. Hardness and elastic modulus as a function of indenter depth in the unirradiated and irradiated BAM-11 specimens.

Table 1. Summary of nanoindentation results on virgin and irradiated BAM-11 at a depth of 200 nm.

	Unirr.	Unirr., HT	1.0 dpa, RT	10 dpa, RT	1.0 dpa, HT	10 dpa, HT
Average Hardness* (GPa)	6.6	6.9	5.6	5.7	5.7	6.0
Std. Dev. Of Hardness (GPa)	0.2	0.2	0.2	0.2	0.2	0.3
Average Young's Modulus* (GPa)	102.1	107.5	92.9	93.5	95.8	98.1
Std. Dev. Of Young's Modulus (GPa)	0.9	2.3	1.9	1.7	2.0	2.8

For both heat-treated and as-cast samples, hardness and modulus dropped for the irradiated specimens as compared to the sample in the unirradiated condition. There was also a slight increase in hardness and modulus in the heat-treated condition as compared to the virgin condition for all samples. The drop in hardness following irradiation for both the as-cast and heat-treated samples was on the order of about 15%, while the drop in modulus was on the order of about 10%.

While hardness and modulus values were observed to drop, the irradiation-induced change does not markedly differ between the samples tested at 1 and 10 dpa, indicating a saturation of irradiation damage in the bulk metallic glass. This indicates that BAM-11 may be resistant to much higher radiation doses than those tested here.

Microstructure

Similar to the previous thrust in which samples were irradiated to a maximum of 1.0 dpa, no significant microstructural changes were observed in the BAM-11 BMG samples after irradiation to 1 or 10 dpa in either the as-cast or heat treated conditions. TEM revealed the samples to be feature-free, and diffraction showed the samples to be amorphous. HRTEM confirmed that no crystallites were present in the sample. Additionally, no dislocations or point defect-like structures were visible upon tilting on two different axes in the TEM. EDS performed on the samples did not reveal any surprises in terms of the elements in the specimens. Microstructures of the as-cast and heat-treated samples are shown in Figure 2.

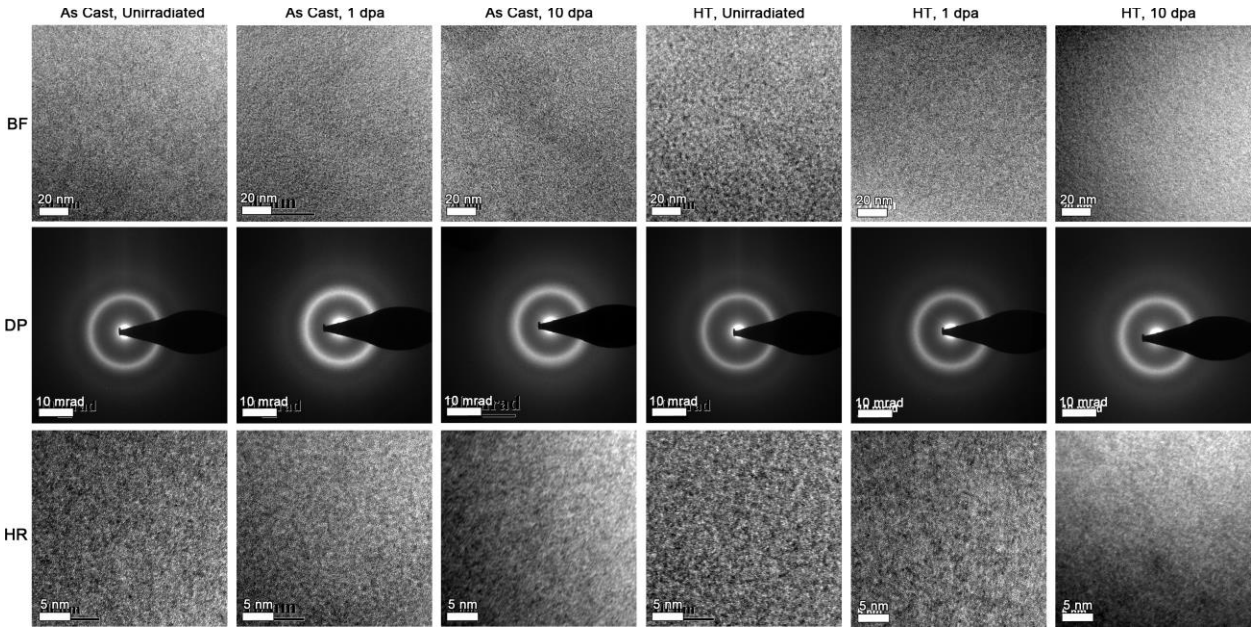

Figure 2. TEM micrographs consisting of BF images at 135kx, diffraction patterns, and high resolution images at 580kx of as-cast and heat-treated ion-irradiated BAM-11 irradiated to 1 and 10 dpa. No changes in crystallinity or any defect structures were observed.

CONCLUSIONS

After irradiation with 3 MeV Ni^+ ions to dose levels of 1 and 10 dpa at room temperature, both as-cast and heat-treated amorphous BAM-11 bulk metallic glass specimens were found to exhibit no observable changes in microstructure and only minor (10-15%) drops in hardness and elastic modulus. In addition, damage in the material seems to saturate at or below the 1 dpa dose level based on mechanical property measurements.

Overall, the favorable constitutive response of BAM-11 following irradiation to low fluence levels indicates that the alloy may have applications as a structural material in nuclear applications, albeit only in low temperature scenarios where the alloy stays below its glassy transition temperature. Further testing of the bulk metallic glass at dose levels up to 100 dpa may be necessary to ensure that irradiation damage does indeed saturate as expected.

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