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## Damage induced by helium ion irradiation in Fe-based metallic glass



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### HIGHLIGHTS

- Metallic glass Fe<sub>68</sub>Zr<sub>7</sub>B<sub>25</sub> could maintain amorphous state after the irradiation.
- A series of crystallization behaviors occurred in metallic glass Fe<sub>80</sub>Si<sub>7,43</sub>B<sub>12,57</sub>.
- The surface of tungsten appeared blisters at the fluence of 1.0  $\times$   $10^{18}$  ions/cm^2.
- Surfaces of Fe-based metallic glasses cracked at the fluence of  $1.6 \times 10^{18} \text{ions/cm}^2$ .

### A R T I C L E I N F O

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# 1. Introduction

One of the most urgent problems in the future fusion reactors is the choice of irradiation resistant materials. The tungsten is considered to show a good irradiation resistance and can be applied to the future fusion reactors due to its excellent properties such as high melting point, good thermal conductivity, low thermal expansion coefficient, low physical sputtering rate, low tritium retention, etc. [1] However, long-term irradiation of thermal neutrons, hydrogen isotope particles, helium ions, impurity particles, etc. will result in damage such as blistering, swelling, cracking, etc.

### G R A P H I C A L A B S T R A C T



### ABSTRACT

The changes in structure and surface morphology of metallic glasses  $Fe_{80}Si_{7,43}B_{12.57}$  and  $Fe_{68}Zr_7B_{25}$  before and after the irradiation of He ions with the energy of 300 keV were investigated, and were compared with that of the tungsten. The results show that after the  $He^{2+}$  irradiation, metallic glass  $Fe_{68}Zr_7B_{25}$  still maintained amorphous. While a small amount of metastable  $\beta$ -Mn type phase nanocrystals formed in metallic glass  $Fe_{80}Si_{7,43}B_{12.57}$  at the fluence of  $4.0 \times 10^{17}ions/cm^2$  (19dpa). The nanocrystals transformed into  $\alpha$ -Fe phase and tetragonal  $Fe_{2}B$  phase as the fluence increased to  $1.0 \times 10^{18}ions/cm^2$  (47dpa). Then the new orthogonal  $Fe_{3}B$  phase and  $\beta$ -Mn type phase nanocrystals appeared when the fluence increased further, and the quantities of nanocrystals increased. Blisters and cracks appeared on the surface of tungsten under the irradiation fluence of  $1.0 \times 10^{18}ions/cm^2$ , however only when the fluence was up to  $1.6 \times 10^{18}ions/cm^2$ , could cracks and spalling appear on the surfaces of metallic glasses.

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in tungsten [2]. And this damage will shorten the service life of the tungsten and cause pollution to the core plasma, thereby affecting the operation of the fusion reactors.

Structured by long-range disorder and short-range order, metallic glasses are thought to absorb the damage induced by irradiation due to their heavily disordered amorphous structure [3]. And because of their good physical and chemical properties such as high strength and excellent corrosion resistance, metallic glasses have attracted widespread attention from the researchers. Fe-based metallic glasses are supposed to be applied in the future fusion reactor irradiation environment due to their wide super-cooled liquid region, relatively high crystallization temperature, and a lack of neutron activation element. The researchers carried out some studies about the irradiation-induced damage in Fe-based

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metallic glasses: Umakoshi et al. used high energy electron to irradiate FeZrB metallic glass and discovered that  $\alpha$ -Fe phase nanocrvstalline formed after the irradiation [4]; Rodriguez et al. observed recrystallization phenomenon in amorphous Fe<sub>80</sub>B<sub>20</sub> ribbons during the high energy Au ion irradiation experiments [5]; Rizza et al. explained the crystallization in the Fe-based metallic glasses after the irradiation of Pb ion with an energy of 5 GeV by the relaxation of the high level of energy deposited in electronic excitations along the path of ions [6]; Kane et al. found that after the electron irradiation, the width of the first coordination shell of Fe atoms decreased and the amorphous matrix was transforming to more ordered phase in metallic glass FeSiBNb [7]; Sun et al. irradiated amorphous FeSiNbZrB ribbons with 5 MeV Xe ions and discovered that after irradiation, the number of nanocrystalline increased with the increase of irradiation fluences, but the size of nanocrystalline saturated as the ion fluences increased [8]. As the potential candidates of irradiation resistant materials in the fusion reactors, Fe-based metallic glasses need to sustain the bombardment of thermal neutrons, hydrogen isotope particles, helium ash, sputtering impurity atoms and high heat load. Therein, helium ion irradiation can induce the formation of helium bubbles, causing performance deterioration to the materials.

At present, the crystallization behavior induced by the helium ion irradiation in Fe-based metallic glasses is not clear. In this study, we used  $\text{He}^{2+}$  ions with the energy of 300 keV to irradiate metallic glasses  $\text{Fe}_{80}\text{Si}_{7,43}\text{B}_{12.57}$  and  $\text{Fe}_{68}\text{Zr}_7\text{B}_{25}$  as well as tungsten to investigate the crystallization behavior and surface damage caused by the irradiation in the two metallic glasses, and employed tungsten as a comparison to provide the reference of possibility which the metallic glasses applied in the future fusion reactor irradiation environment.

#### 2. Experimental methods

The metallic glass ribbons Fe<sub>80</sub>Si<sub>7,43</sub>B<sub>12.57</sub> and Fe<sub>68</sub>Zr<sub>7</sub>B<sub>25</sub> used in this study were prepared by melt-quench method. The thickness of metallic glass Fe<sub>80</sub>Si<sub>7,43</sub>B<sub>12.57</sub> and Fe<sub>68</sub>Zr<sub>7</sub>B<sub>25</sub> is 35 µm and 40 µm, respectively. The tungsten used in this study had a purity of 99.95% with the size of 5 mm × 5 mm × 2 mm. Surfaces of tungstens were mechanically polished to a mirror finish and then all the samples cleaned ultrasonically with acetone and alcohol prior to the irradiation experiment. The ion beam irradiation experiment was completed on the 320 kV highly charged ion research platform at Institute of modern physics, Chinese academy of sciences. The samples were irradiated with He<sup>2+</sup> ions of 300 keV energy with fluences of 1.0 × 10<sup>17</sup>ions/cm<sup>2</sup>, 2.0 × 10<sup>17</sup>ions/cm<sup>2</sup>, 4.0 × 10<sup>17</sup>ions/cm<sup>2</sup>, 1.0 × 10<sup>18</sup>ions/cm<sup>2</sup> and 1.6 × 10<sup>18</sup>ions/cm<sup>2</sup>. To avoid the effect of irradiation ions flux on the material behavior [9,10], the flux for all the samples was 1.2 × 10<sup>13</sup>ions/cm<sup>2</sup>s.

The phase structure of the unirradiated and irradiated samples was studied by X-ray diffraction (XRD) with Cu K $\alpha$  radiation, and the changes on microstructure of samples were analysed by transmission electron microscope (TEM) with the operating voltage of 200 kV. The samples for TEM were prepared by focused ion beam (FIB) method. The surface morphology was characterized by scanning electron microscope (SEM). In addition, the ion range, concentration distribution of He atoms and DPA value in the metallic glasses and metal W were calculated by SRIM programme.

### 3. Results and discussion

3.1. Evolution of  $Fe_{80}Si_{7,43}B_{12.57}$  metallic glass microstructure under irradiation

Fig. 1 shows the GID-XRD patterns of metallic glasses



Fig. 1. XRD patterns of metallic glasses  $Fe_{80}Si_{7,43}B_{12,57}$  irradiated by He ions of 300 keV with different fluences.

 $Fe_{80}Si_{7,43}B_{12.57}$  before and after the He ion irradiation. It can be seen that the pattern of sample before irradiation presented broad peak, which demonstrated amorphous state of the original sample. After irradiation, no sharp diffraction peak appeared in the patterns of metallic glasses, which indicated that  $Fe_{80}Si_{7,43}B_{12.57}$  metallic glasses remained amorphous state as the main phase structure.

The cross-section TEM image and the corresponding SAED pattern of unirradiated metallic glass  $Fe_{80}Si_{7,43}B_{12.57}$  are shown in Fig. 2. From Fig. 2(a), it can be seen that no obvious damage was induced by Ga ions irradiation. And there was only one halo in the Fig. 2(b) indicating that the sample prepared by FIB technology was amorphous. That is, for metallic glass  $Fe_{80}Si_{7,43}B_{12.57}$ , preparation of TEM sample by FIB method would not cause crystallization.

The cross-section TEM image of metallic glass  $Fe_{80}Si_{7,43}B_{12.57}$ after the irradiation at a fluence of  $4.0 \times 10^{17}$ ions/cm<sup>2</sup> (19dpa) is given in Fig. 3(a). A white belt with a width of about 250 nm formed about 700 nm away form the surface, namely a helium bubble layer. Similar helium bubbles had also been observed by Iwakiri et al. [11] and Zhan et al. [12]. Fig. 3(b) and (c) show the selected-area electron diffraction images of regions A and B in Fig. 3(a), respectively. Only one halo can be observed in Fig. 3(b). It is illustrated that the metallic glass is still maintained amorphous in region A, the area which ions passed through. In Fig. 3(c), another two halos appeared, which shows that the nanocrystalline formed in region B, the area with helium bubble layer, and the two halos corresponded to (211) and (411) crystal planes of  $\beta$ -Mn type phase. Fig. 3(d) gives



Fig. 2. (a) The cross-section TEM image (b) the corresponding SAED pattern of pristine metallic glass  $Fe_{80}Si_{7,43}B_{12.57}$ 

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