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Influence of helium ion irradiation on the morphology and microstructure of CN-G01 beryllium

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Abstract

Beryllium is considered as a candidate of ITER first-wall (FW) armor and neutron multiplier in fusion reactors. To assess the irradiation resistance of CN-G01 beryllium, which has been accepted as an alternative ITER-grade beryllium in China, 180 keV helium ions with fluences of $1.0 \times 10^{17}$ ions cm$^{-2}$, $5.0 \times 10^{17}$ ions cm$^{-2}$ and $1.0 \times 10^{18}$ ions cm$^{-2}$ were implanted at room temperature. The theoretical simulation of energy loss, damage distribution and helium concentration were proceeded by SRIM. In this paper, we report the experimental exploration of ion fluence effect on surface morphology and microstructure of irradiated samples. Field Emission Scanning Electron Microscope (FESEM) analysis depicted the formation and growth of helium bubbles at different irradiation ion fluences. No obvious exfoliation or cavities was observed on the surface at all ion fluences, suggesting a reliable radiation resistance of CN-G01 beryllium. Atomic force microscopy (AFM) morphology showed that the maximum height of bubbles was 47.8 nm. Surface roughness values increased slightly due to the formation of defects and bubbles on the irradiated beryllium surface. Nevertheless, the structural analysis demonstrated by grazing incidence x-ray diffraction (GIXRD), indicated an obvious preferred orientation on (101) peak at various ion fluences. Annihilation of defects caused by a small rise of the localized temperature could explain the increasing intensity of diffraction peak (101).

1. Introduction

Nuclear fusion energy is expected to be an attractive way to solve energy issue in the future [1, 2]. International Thermonuclear Experimental Reactor (ITER), as a critical step on the road of the development of fusion energy from the experiment scale to a power plant scale, is now under construction in France [3–5] to demonstrate the feasibility of fusion energy through international collaboration. Beryllium is served as the armor material of the ITER-First Wall (FW) and the neutron multiplier of the tritium breeding blanket because of its low atomic number, neutron breeding potential and high thermal conductivity [6–8]. In the ITER Final Design Report 2001, S-65C beryllium from Brush Wellman (USA) and DShG-200 beryllium from Russian Federation (RF) have been identified as reference beryllium grades [9, 10], owing to their low impurity content, high flexural rigidity and excellent thermal fatigue. As a participant in ITER, China also developed its own fabrication technique and such beryllium named as CN-G01 produced by a vacuum hot press (VHP) approach [11, 12]. The basic qualification tests have been performed according to the specification for the ITER-grade beryllium. The results confirmed that the physical and thermo-mechanical properties of the CN-G01 have satisfied the ITER requirements [13]. In other words, the CN-G01 beryllium can also be used in the ITER-FW and the tritium breeding blanket.

During operation of fusion reactor, beryllium metal is exposed to neutrons and produce a large number of helium ions inside the material by undergoing neutron multiplication reaction: $2\text{Be} + n \rightarrow 2\text{He}^+ + 2n$ [14]. High helium concentration could cause significant effect on the beryllium materials. For examples, P P Liu et al

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Table 1. Chemical composition of CN-G01 beryllium (wt. %).

| Product grade | Be  | BeO | Al  | C   | Fe  | Mg  | Si  | U   | Others |
|---------------|-----|-----|-----|-----|-----|-----|-----|-----|--------|
| Values        | >99 | 0.75| 0.006| 0.060| 0.050| 0.003| 0.009| 0.0015| <0.04  |

[15] reported the microstructure evolution of beryllium irradiated by 30 keV helium ion with a dose of $1 \times 10^{17}$ He$^+$ cm$^{-2}$ at room temperature. The helium bubbles and different types of dislocation loops were observed after irradiation. Morishita et al [16] investigated the microstructural changes of beryllium samples (provided by NGK Insulators) irradiated by 8 keV helium ions at temperatures ranging from room temperature to 873 K. It is found that, in all irradiated beryllium samples, the tiny bubbles were formed by the gas driven process and the radiation-induced vacancy migration phenomenon, which is independent on temperature. Furthermore, other types of defects including blisters, exfoliation, flakes and pinholes were observed on the surface of the irradiated beryllium samples. In addition, Kupriyanov et al [17] explored the effect of neutron irradiation on the performance of seven beryllium grades (DshG-200, TR-30, TshG-56, TRR, TE-30, TIP-30 and S-65). The results indicated that the properties and radiation damage were different for different beryllium grades. However, studies on the helium irradiation behavior of the newly proposed beryllium grades CN-G01 are insufficient. Hence, to further assess the properties of CN-G01 beryllium, more experimental investigations should be conducted, especially on the effect of helium ions on the performance of the CN-G01 beryllium.

In this work, the irradiation effect of helium ions (180 keV) on the microstructure and morphology of the CN-G01 beryllium under different ion fluences ranging from $1.0 \times 10^{17}$ ions cm$^{-2}$ to $1.0 \times 10^{18}$ ions cm$^{-2}$ were investigated. Displacements per atom (dpa) and helium ions concentration in the beryllium were evaluated by the Monte Carlo code SRIM-2013. The crystallographic characteristics of beryllium were determined by grazing incidence x-ray diffraction (GIXRD). The surface morphology of reference and irradiated CN-G01 beryllium were characterized by field emission scanning electron microscope (FESEM) and atomic force microscopy (AFM).

2. Experimentation

The beryllium CN-G01 used in this work was fabricated by Ningxia Orient Ground Co. Ltd, China. The sample was manufactured by powder metallurgy vacuum hot pressing techniques. While the bulk density is 99.9% of the theoretical density. The major chemical composition of CN-G01 beryllium sample is presented in table 1. The purity of CN-G01 beryllium is higher than 99.0 wt. % and the content of BeO impurity is less than 1.0 wt. %. Specifically, due to ITER application safety considerations, the maximum content of uranium is specified as 0.0030 wt. %. For the grade CN-G01 beryllium, the content of uranium measured in the qualification process is below 0.0020 wt. %, where the ITER application requirement has been achieved.

CN-G01 beryllium samples were cut into flat specimens with dimensions of $10 \times 10 \times 1$ mm. After being ground with SiC papers and diamond pastes, beryllium samples were polished in an electrolytic cell by using electrolyte consisting phosphoric acid, glycerol, gelatin, anhydrous ethanol and sulphuric acid in a certain ratio. Before helium ions irradiation experiment, the samples were cleaned ultrasonically in alcohol bath to remove impurities attaching on surface.

The polished samples were implanted with helium ions at the 320 kV Multi-Discipline Research Platform for Highly Charged Ions in Institute of Modern Physics, Chinese Academy of sciences (IMP, CAS) [18]. Beryllium were mounted on a sample holder in a high-vacuum chamber at a pressure of $5 \times 10^{-4}$ Pa. The ion beams were swept in two perpendicular directions to a uniform distribution, where the samples were irradiated by 180 keV helium ions in vacuum at room temperature. The ion fluences were $1.0 \times 10^{17}$ ions cm$^{-2}$, $5.0 \times 10^{17}$ ions cm$^{-2}$ and $1.0 \times 10^{18}$ ions cm$^{-2}$, respectively. Before ion irradiation test, the prediction of damage distribution and ion range of CN-G01 beryllium were estimated by means of widely used Monte Carlo code SRIM (Stopping and Range of Ions in Matter) 2013 software [19, 20].

After implantation, both references and helium ion irradiated beryllium samples were characterized. Microstructure evolution in beryllium during irradiation was conducted by grazing incidence x-ray diffraction (GIXRD) pattern using Cu Kα radiation (1.54 Å). Field Emission Scanning Electron Microscope (FESEM) was used to observe the evolution in surface morphology. The helium bubble size was measured by Nano Measurer. While atomic force microscopy (Dimension Icon AFM from Bruker Nano) was used to determine the surface morphologies and roughness. Thus the morphological and structural results of the samples irradiated at different ion fluences were compared to examine the changes induced by the helium ions in CN-G01 beryllium samples.
3. Results and discussion

3.1. SRIM calculations

To investigate the radiation damage in CN-G01 beryllium caused by helium ions, a theoretical simulation of energy loss, damage distribution and helium concentration were proceeded in the quick calculation mode of SRIM. The percentage of ion energy loss has been listed in Table 2, where helium ions irradiation-induced ionization energy loss was 96.56% (approximately 173.81 keV), indicating that the ionization process prevailed in irradiated beryllium. Figure 1 presents the distribution of the ionization energy loss in irradiated beryllium. There are two distinct plots in the figure, red plot represents energy loss from the incident ions, and blue plot represents energy loss from recoils. One should have noticed that the ions lose more energy on the surface. Thus, beryllium surface is prone to defects and irradiation damages after irradiation.

The simulation results clearly indicates that, when the energy of incident ions was consumed, He\(^+\) ions would remain inside the material. The helium concentration and the displacement per atom (dpa) in function of depth are shown in Figure 2, respectively. While helium ions distributed from 0 nm to 1095 nm in depth and peaked at 935 nm. The displacement damages of the irradiated beryllium have been roughly estimated by using the formula\:[21, 22]:

\[
dpa = \frac{\text{displacement per } \AA \text{ ion } \times \text{fluence}}{\text{atomic density of the target}}
\]  

The calculation result of displacements per atoms 2.4 dpa, 12.0 dpa and 23.9 dpa and the peak helium concentration is 5.94 at.% 29.7 at.% and 59.4 at.%, corresponding to the ion fluences of 1.0 \times 10^{17} \text{ ions cm}^{-2}, 5.0 \times 10^{17} \text{ ions cm}^{-2} and 1.0 \times 10^{18} \text{ ions cm}^{-2}.

3.2. Surface morphology

The FESEM images demonstrate the surface morphologies of reference sample and irradiated CN-G01 beryllium sample at different ion fluences as shown in figures 3(a)–(d). In the case of reference sample, the surface appears to be smooth and homogeneous, without any impurity or cracks on the top (figure 3(a)). While

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**Table 2. Energy loss of helium ions in the beryllium calculated by SRIM-2013.**

| Energy loss (%) | Ionization | Vacancies | Phonons |
|----------------|------------|-----------|---------|
| Ions           | 96.56      | 0.05      | 0.49    |
| Recoils        | 0.76       | 0.09      | 2.06    |

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**Figure 1.** The distribution of ionization energy loss in beryllium irradiated by 180 keV He\(^+\) ions.
after helium irradiation, certain irradiation-induced bubbles appear on the surface and the density of bubbles increases together with the ion fluence. At low ion fluence of $1.0 \times 10^{17}$ ions cm$^{-2}$, a small amount of tiny bubbles appear and sparsely distribute on the surface, as shown in figure 3(b). When the fluence is increased to $5.0 \times 10^{17}$ ions cm$^{-2}$, the average size of bubbles is significantly increased and formed a regular circle shape. By continuously increasing the ion fluence to $1.0 \times 10^{18}$ ions cm$^{-2}$, the final agglomeration and coalescence of bubbles have been shown in figure 3(d) and the maximum diameter of bubble was found to be 535 nm. The distribution of bubbles size is shown in figure 4. The average size is measured to be 76.29 nm, 140.65 nm, 257.15 nm at $1.0 \times 10^{17}$ ions cm$^{-2}$, $5.0 \times 10^{17}$ ions cm$^{-2}$ and $1.0 \times 10^{18}$ ions cm$^{-2}$, respectively. Similar bubbles also appeared on the surface of other beryllium grades after irradiation. Dutta et al [23] observed bubbles with tri-modal distributions consisting of very small ($\sim 20–50$ nm), medium ($\sim 50–100$ nm) and very large ($>200$ nm) on beryllium samples after irradiated by 60 keV helium ions. In this work, the size of irradiation-induced bubbles in CN-G01 beryllium is medium at low ion fluence and very large at high ion fluence.

The surface morphologic difference at different ion fluences can be explained by the interaction between helium ions with beryllium metal. When helium ions are implanted to samples, where helium ions will loss
energy on the surface (as shown in figure 1) and gradually accumulate inside the materials, inducing significant defects [24]. In precious study, it has been reported that up to ten helium atoms may be bond to one single vacancy [25]. Due to the strong binding of helium atom to vacancy, helium atoms can be easily trapped by vacancy-type defects to form helium–vacancy (He–V) clusters. By absorbing the surrounding helium atoms and vacancy defects, He–V clusters grow up to nanometer level and helium bubbles are gradually formed [22, 26, 27]. By means of increasing ion fluence, the bubble density is increased and some small bubbles will to contact with adjacent bubbles by chance, or attract more helium atoms to form a bigger one. After the coalescence process, an excess of internal pressure will be built up in the big helium bubble [28]. Once the internal pressure reaches to a high enough level, some bubbles will rupture and strip off. On the other hand, no obvious exfoliation or cavities was observed in this study, which hints excellent radiation resistance of CN-G01 beryllium.
Three-dimensional surface morphologies of irradiated area in CN-G01 beryllium samples were analyzed by AFM measurement. The reference beryllium exhibited a relatively smooth surface with the averaged roughness of only 0.88 nm. The irradiated sample surface has been changed in structure. When the ion fluence was $1.0 \times 10^{17}$ ions cm$^{-2}$, bulges and pits on the surface of irradiated beryllium were observed (figure 5(b)). At $5.0 \times 10^{17}$ ions cm$^{-2}$, the size of bulges and pits increased (figure 5(c)). It is observed that the irregular shape bulges were transformed into the semi-spherical bubbles. N J Dutta reported the heterogeneously distributed spike-like structures on the surface of irradiated beryllium [23]. In comparison, most of the bulges on the surface of CN-G01 beryllium have a spherical or ellipsoidal shape. Figure 6 demonstrated a simplified diagram of irradiated beryllium. Involved with FESEM analysis, it is clear to see that the bulges are irradiated-induced bubbles. At $1.0 \times 10^{18}$ ions cm$^{-2}$, the size of bubbles and pits further increased. The vertical scale of bubbles is up to 47.8 nm, which represents the height of bubbles. The averaged surface roughness of samples at different ion fluences were 2.62 nm, 6.34 nm and 7.63 nm, respectively, revealing that the surface roughness increased depending on ion fluence. Since ion irradiation could produce defects on the surface and induce bubbles inside, with ion fluence increasing, there exists a large number of defects and bubbles formed on the irradiated material surface. As a consequence, the roughness increased with the ion fluence.

3.3. XRD patterns analysis
Figure 7 shows the GIXRD diffraction patterns of CN-G01 beryllium samples before and after helium ions irradiation with different ion fluences. In the pattern of reference samples, diffraction peaks can be clearly
observed at 45.729°, 50.947°, 52.769°, 70.919° and 84.757°, corresponding to (100), (002), (101), (102) and (110) planes of hexagonal closed pack alpha phase beryllium (JCPDS No 71-0257). While after helium irradiation, no extra peaks have been observed in the diffraction of samples with different ion fluences. It has been reported in the previous study that the peaks of BeO (beryllium oxide) were speculated and the most intense peak recorded a shift towards the higher or lower Bragg’s angle region in other beryllium grades [23]. By contrast, no new phase appeared and the diffraction peaks position in irradiated CN-G01 beryllium samples remained unchanged, suggesting that the structure of CN-G01 beryllium is relatively stable. Figure 8 is magnified image of the most intense peak (101) of beryllium. It shows that the orientation of (101) plane is increased in all irradiated samples, indicating that irradiation leads the crystal to grow along the (101) plane. The microstructure changes of beryllium are related to the lattice disorder induced by helium ions irradiation. Defects generated inside the irradiated beryllium and the localized temperature will have a small rise. There may be annihilation of defects since the localized heat generated during the irradiation facilitates the recovery process in the materials. As a result, the diffraction peaks intensity of (101) increased.

4. Conclusions

Surface morphology and microstructure analysis of CN-G01 beryllium irradiated by 180 keV helium ions with different ion fluence have been comprehensively investigated in this study. Due to helium ions bombardments, tiny helium bubbles were formed at low ion fluence while agglomeration and coalescence of bubbles occurred at high ion fluence. No obvious exfoliation or cavities appeared on the surface, indicating an excellent radiation resistance of CN-G01 beryllium. AFM demonstrated that the maximum height of helium bubbles reaches to 47.8 nm on the sample surface and the roughness value increased slightly because of defects and bubble agglomeration that induced by the irradiation. It is worthy that the irradiated samples had a same preferred crystal growth orientation on (101) plane and no new phase was observed under all ion fluence from GIXRD patterns. The microstructure of CN-G01 beryllium is relatively stable, being similar to the present reported beryllium. This study demonstrates that irradiation of helium ions could affect the morphology and microstructure of CN-G01 beryllium, which provides a preliminary insight on the components changes of CN-G01 beryllium under ions bombardments. Studies on irradiation under higher influences will be detailed in the future.

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