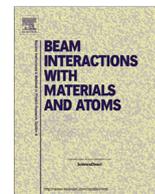




Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

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## Effect on structure and mechanical property of tungsten irradiated by high intensity pulsed ion beam

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### ARTICLE INFO

#### Article history:

Received 20 July 2016

Received in revised form 20 March 2017

Accepted 20 March 2017

Available online xxxxx

#### Keywords:

High intensity pulsed ion beam

Tungsten

Irradiation damage

### ABSTRACT

The anti-thermal radiation performance of tungsten was investigated by high intensity pulsed ion beam technology. The ion beam was mainly composed of  $C^{n+}$  (70%) and  $H^+$  (30%) at an acceleration voltage of 250 kV under different energy densities for different number of pulses. GIXRD analysis showed that no obvious phase structural changes occurred on the tungsten, and microstress generated. SEM analysis exhibited that there was no apparent irradiation damage on the surface of tungsten at the low irradiation frequency (3 times and 10 times) and at the low energy density ( $0.25 J/cm^2$  and  $0.7 J/cm^2$ ). Cracks appeared on the surface of tungsten after 100-time and 300-time irradiation. Shedding phenomenon even appeared on the surface of tungsten at the energy densities of  $1.4 J/cm^2$  and  $2.0 J/cm^2$ . The surface nano-hardness of tungsten decreased with the increase of the pulse times and the energy density. The tungsten has good anti-thermal radiation properties under certain heat load environment.

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### 1. Introduction

During the development of nuclear fusion research, one of the most urgent problems to solve is irradiation damage of plasma facing materials. The plasma facing materials in nuclear fusion reactor mainly suffer neutron irradiation, plasma irradiation and two kinds of strong thermal irradiation that were transient state and steady state. The effects of the transient or steady high heat load would shorten the lifespan of the plasma facing materials and cause security issues. So they need to show excellent irradiation-resistance performance, good thermal conductivity, low sputtering rate, high deuterium/tritium solubility and good adaptability with plasma for application. The nuclear fusion devices are still on the research stage. So studying irradiation effects on candidate materials can be conducted only by simulating the irradiation conditions approximately.

High intensity pulsed ion beam (HIPIB) is a new and multidisciplinary cross technology involved in plasma physics, nuclear physics and materials [1,2]. The technology is a combination of ion irradiation and strong heat radiation with features of instant, high heat and high energy deposition. The temperature gradient on the surface of materials caused by HIPIB can achieve over  $10^8 K/s$ , the surface can be heated rapidly and melted within one pulse [3–5], and non-equilibrium metastable phase such as nanocrystals form

on the surface of crystalline materials in the subsequent rapid cooling [6–8]. The materials may be etched by sheets [9] and generated serious irradiation damage due to the shock waves and extremely high thermal stress generate during the HIPIB irradiation. The thermal load on the surface of materials can be up to  $3.6 \times 10^4 MW/m^2$  induced by HIPIB when the pulse width and the energy density are 70 ns and  $0.25 J/cm^2$ , respectively, which is much higher than the thermal load threshold ( $30 MW/m^2$ ) of steady state in nuclear fusion reactor. Therefore HIPIB irradiation can be used for simulating the transient heat load conditions induced by unstable plasma in nuclear fusion reactor and the damage caused by it in materials are well worth studying.

Tungsten with the features of high melt point, good thermal conductivity, low expansion coefficient, low sputtering rate and tritium retention rate has already been selected as surface material for tokamak divertor [10]. Due to the good adaptability with plasma and other excellent properties, tungsten is considered as one of promising candidate materials for plasma facing components [11], and the irradiation resistance of tungsten attracts extensive attentions. The aggregation of helium atoms in tungsten metal were simulated by Wang et al. [12], James Gibson et al. investigated the changes of micro-mechanical properties of tungsten under the irradiation of  $W^{12+}$  and  $He^+$  in high temperature, and found that the hardness of tungsten increased after the irradiation [13]. Barabash et al. discovered that the structural phase of tungsten changed significantly and the toughness of tungsten decreased after neutron irradiation [14]. M. Battabyal et al. found

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that the hardness of tungsten increased after the  $\text{Fe}^{2+}$  and  $\text{He}^{2+}$  irradiation, and doped oxidic particles to tungsten could improve the strength of tungsten significantly [15]. HIPIB is an efficient method of irradiation with an intense thermal effect; it can be used to detect the capacity of anti-thermal stress of tungsten. However, the study of HIPIB irradiated tungsten is scarce at present.

In this study, HIPIB with high heat load was used to irradiate tungsten metal to simulating the transient condition induced by unstable plasma in tokamak nuclear fusion device. The changes of performance and structure of tungsten before and after irradiation were investigated to provide reference data to the probability of tungsten which served as plasma facing materials.

## 2. Experimental methods

The purity of the polycrystalline tungsten used in this study was 99.95 wt. %, and the size was  $10 \times 10 \times 2 \text{ mm}^3$ . Surfaces of tungsten were polished to a mirror finish and then cleaned ultrasonically with alcohol and acetone prior to the HIPIB irradiation. The irradiation experiment was performed on the TEMP-4M accelerator (high-power ion beam sources) in the Tomsk Polytechnic University [16,17]. Graphite and low hydrogen polymers were used as the anode, and the stainless steel was used as cathode of the diode. The ion beam was mainly consisted of  $\text{C}^{n+}$  (70%) and  $\text{H}^+$  (30%), the acceleration voltage was 250 kV, the pulse width was 70 ns and the pulse interval was 10 s. The times of total pulses were 3, 10, 100 and 300 respectively when the energy density was  $0.25 \text{ J}\cdot\text{cm}^{-2}$ , and the energy density was  $0.25 \text{ J}\cdot\text{cm}^{-2}$ ,  $0.7 \text{ J}\cdot\text{cm}^{-2}$ ,  $1.4 \text{ J}\cdot\text{cm}^{-2}$ ,  $2.0 \text{ J}\cdot\text{cm}^{-2}$  respectively when the total pulses were 3 in the irradiation experiment.

The structural changes of the tungsten before and after irradiation were analyzed by Grazing Incident X-ray diffractometer with Cu  $\text{K}\alpha$  radiation (GIXRD, D8 Discover). The surface topography of tungsten before and after irradiation was observed by scanning electron microscope (SEM, Zeiss Supra55 (VP)), and the changes of nanohardness of tungsten before and after irradiation were measured by micro-nano indenter (FISCHERSCOPE HM2000) with a Berkovich indenter, the load was 300 mN, the load time was 20 s, and the measurements were carried out on the surface of samples.

## 3. Results and discussion

### 3.1. Effect of irradiation on tungsten phase structure

Fig. 1 shows the GIXRD patterns of tungsten before and after HIPIB irradiation with energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times. It can be seen that the main structure of tungsten before the irradiation was body-centered cubic (bcc), and it remained without significant change after HIPIB irradiation with different times.

Fig. 2 shows the GIXRD patterns of tungsten before and after HIPIB irradiation with 3 times and different energy density. It can be seen that no apparent changes occurred on the phase structure of tungsten. Figs. 1 and 2 indicated that the phase structure of tungsten used in this study didn't change significantly after irradiation with the pulse numbers from 3 to 300 and the range of energy density  $0.25\text{--}2.0 \text{ J}\cdot\text{cm}^{-2}$ . HIPIB irradiation is a process with rapid heating and rapid solidification which can induce phase transformation in other crystal materials (such as high-speed steel, nickel alloy and so on [18,19]) easily. However, there was no obvious phase transformation occurred in the tungsten used in this study, it indicated that the phase structure of tungsten was stable. Thus tungsten with body-center structure, high melt point and good heat conductivity had good resistance against HIPIB irradiation and the phase transformation occurred in the tungsten rarely.

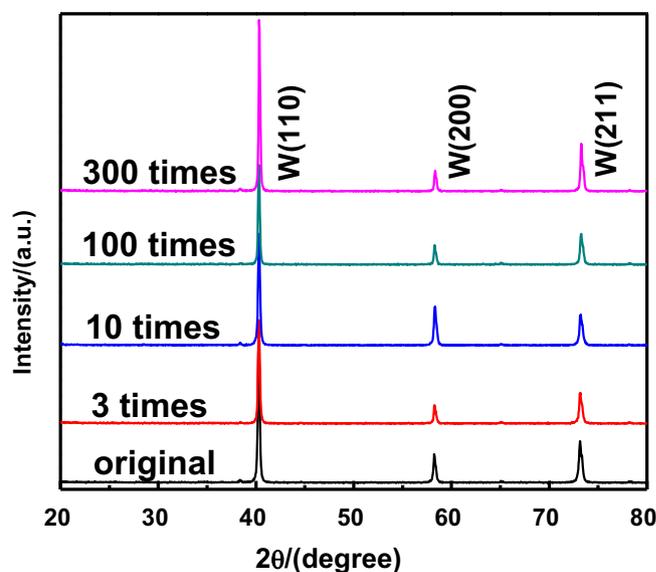


Fig. 1. The GIXRD patterns of HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times.

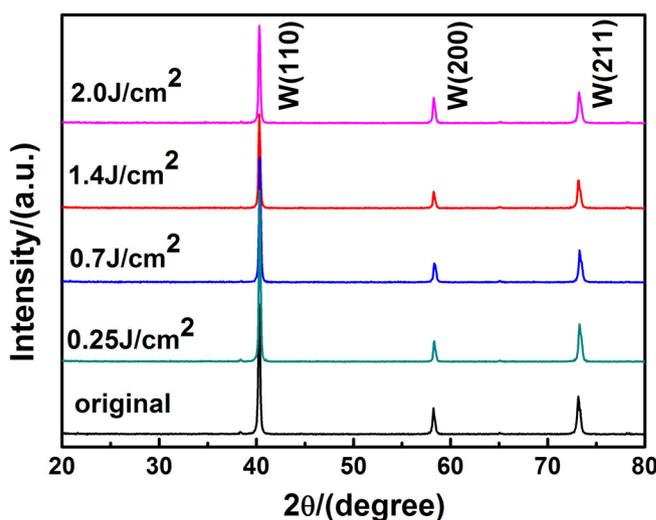
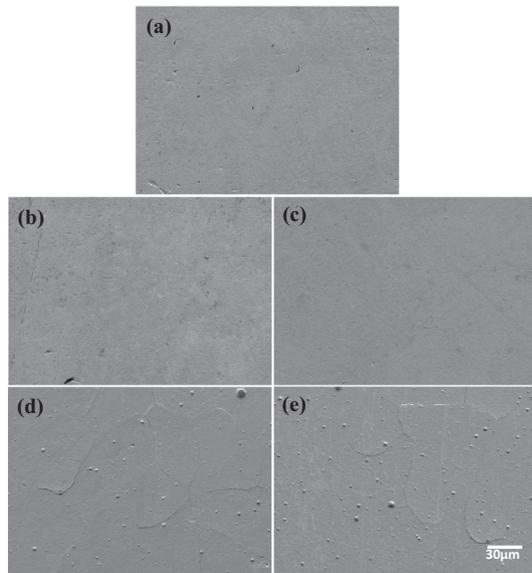


Fig. 2. The GIXRD patterns of HIPIB irradiated tungsten with 3 times and different energy density.

### 3.2. Effect of irradiation on surface topography, composition and micro-stress in tungsten

The surface morphology of tungsten before and after HIPIB irradiation with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times are shown in Fig. 3. It can be seen from Fig. 3(a) that the surface of original tungsten was smooth. Fig. 3(b) and (c) show the surface topography of the tungsten after 1 and 3 times irradiation, respectively. It is observed there were no significant changes appeared on the surface of tungsten compared with the original sample. In other researches, the craters form on the surface of some crystal materials such as high-speed steel [20], titanium alloy [21] and so on after HIPIB irradiation, while the mechanism of the craters formation is unclear until now, but it is to be sure that the craters formation is connected with the impurities existed in the material. However, no craters formed on the surface of tungsten due to the high melt point and high purity (99.5 wt.%) of the tungsten used in this study, the content of low-melting impurities was little. It



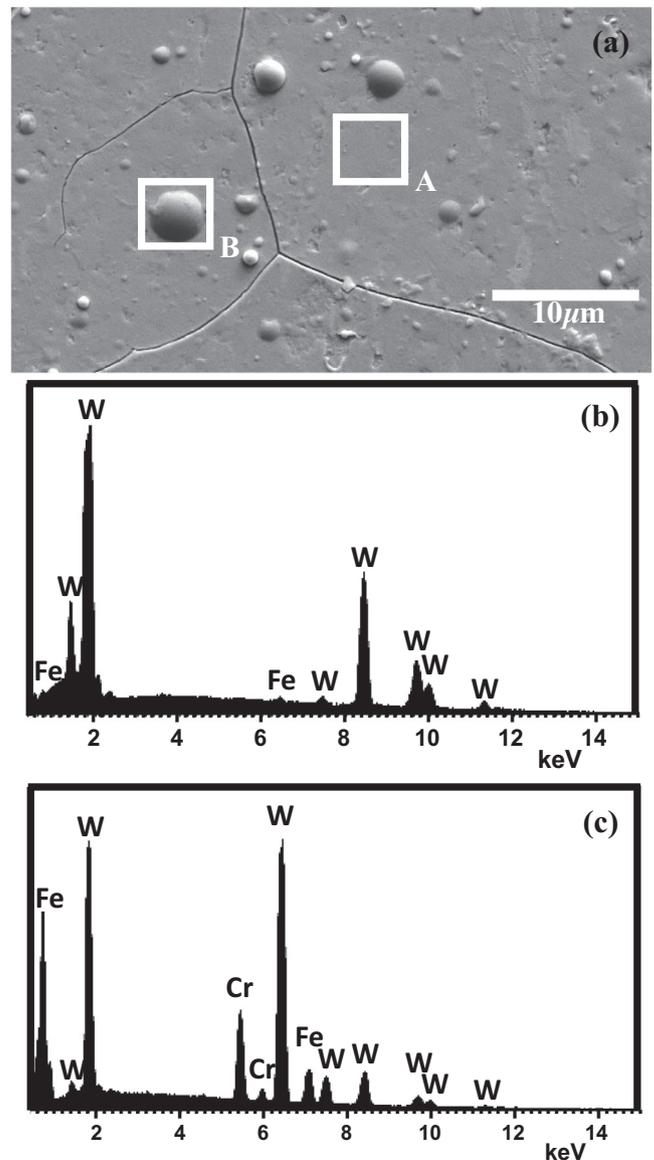
**Fig. 3.** The SEM surface morphology of HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different time as (a) original sample, (b) 3 times, (c) 10 times, (d) 100 times, (e) 300 times.

can be seen from Fig. 3(d) and (e) that salient points and cracks formed on the surface of the tungsten after 100 and 300 times irradiation and the number of salient points increased with the irradiation times.

Since salient points on the surface of tungsten may be induced by the irradiation of ion beam, so energy spectrum analysis of the tungsten samples had been done. Fig. 4 shows the energy spectrum of salient points and the smooth areas on the surface of tungsten after HIPIB irradiation with the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and 300 times.

It can be seen from Fig. 4(b) that the mainly component of smooth areas in the sample was tungsten, while Fig. 4(c) indicated that the components of the salient points were Fe, Cr elements besides W element after 300 times irradiation. The Fe, Cr elements were introduced by the erosion of cathode. When the pulse numbers were few, the salient points were not easy to form due to the content of Fe, Cr elements was little, so there were no salient points appeared in Fig. 3(b) and (c). Fe and Cr accumulated on the surface of the tungsten metal with the increasing of irradiation times, and the remelting occurred on the surface made W atoms and Fe, Cr atoms aggregate to form salient points.

In Fig. 5 the GIXRD patterns of (110) plane in tungsten before and after HIPIB irradiation is displayed for the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times. It can be seen that comparing with original sample, the diffraction peak of tungsten moved towards high angle after 3 times irradiation. Namely the interplanar spacing of tungsten became smaller after 3 times irradiation, it was caused by stresses generating in the surface layer [22]. The diffraction peak of tungsten continued shifting to the high angle after 10 times irradiation which indicated that the stresses increased with the times of irradiation. Stresses accumulated in the surface layer, and cracks formed on the surface of samples when the total stresses exceeded the ultimate tensile strength of weak grain boundaries after 100 and 300 times irradiation. The morphology of the cracks along grain boundaries was showed in Fig. 3(d) and (e). There are three main reasons for the formation of the stresses generated in the tungsten samples: (1) The high temperature generated during the HIPIB irradiation led to melt and then the tungsten cooled rapidly so that it cannot achieve the steady state, stresses generated in the subsurface of samples, and the recoil



**Fig. 4.** The energy spectrum of different superficial areas on HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and 300 times as (a) SEM surface morphology, (b) energy spectrum of A area (the smooth area) in a, (c) energy spectrum of B area (the salient point) in a.

effect caused by the etching of the incident ions would also produce stress in the sample, (2) Extremely high temperature gradient also cause the thermal stresses, (3) High energy ion beam implantation lead to lattice deformation. HIPIB impacted on the surface during each irradiation which resulted in that the strength of the material decrease, and stresses accumulated in the surface layer with the increasing of irradiation times, then cracks formed on the surface of tungsten when total stresses exceeded the ultimate tensile strength of weak grain boundaries.

GIXRD analysis with different incident angles was applied for samples which were after HIPIB irradiation with the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and 300 times, the GIXRD patterns were showed in Fig. 6. Choosing the diffraction peak of (211) plane which corresponds to high angle in order to compare shifts of the diffraction peak, the incident angles were  $1^\circ$ ,  $2^\circ$  and  $3^\circ$ . It can be seen from Fig. 6 that compared with the original sample, after irradiation, the shift of the diffraction peak corresponding to (211) plane in tungsten was maximum ( $0.2^\circ$ ) when the incident angle was  $1^\circ$ ;

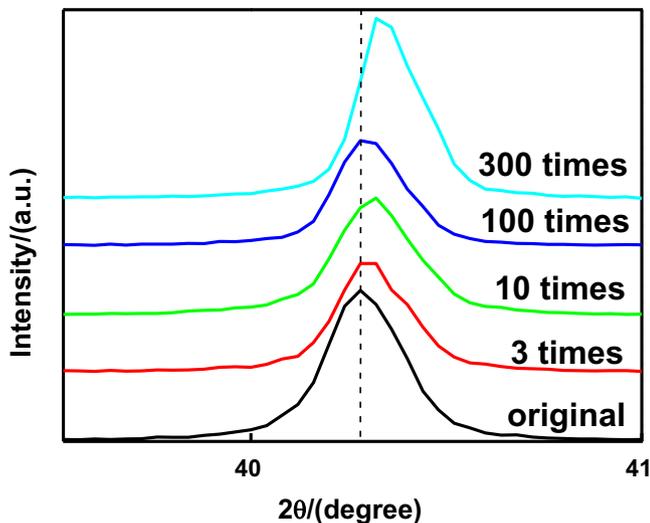


Fig. 5. The GIXRD patterns of body-centered cubic phase (110) plane in HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times.

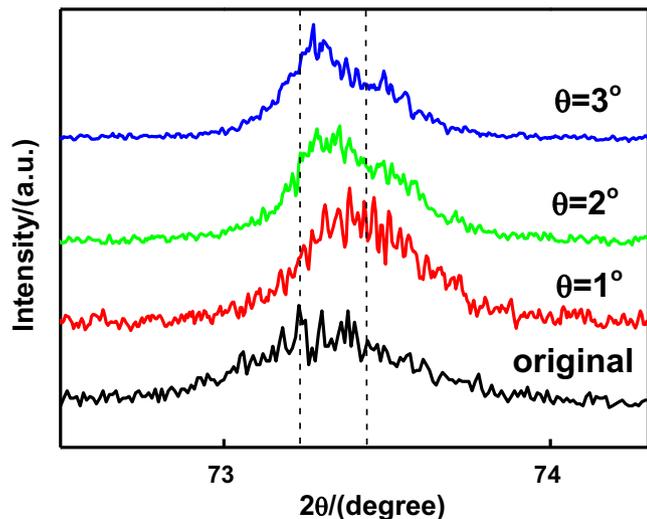


Fig. 6. The GIXRD patterns of the (211) plane in original tungsten and HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and 300 times, the incident angle of X-ray are  $1^\circ$ ,  $2^\circ$ ,  $3^\circ$ .

the shift decreased when the incident angle was  $2^\circ$  and the shift decreased further when the incident angle increased up to  $3^\circ$ . Because different incident angle of GIXRD means different detected depth of materials. In the different depth of the material, the amount of the diffraction peak was different, it indicated that there was a stress gradient generated in tungsten after irradiation, thus the quantitative analysis for the stresses existed in the surface of tungsten couldn't be completed.

Fig. 7 shows the SEM surface morphology of tungsten before and after the HIPIB irradiation with 3 times and different energy density. It is observed that the surface of original tungsten was smooth. Comparing with original tungsten, there were no significant changes appeared on the surface of tungsten after HIPIB irradiation with the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  which was showed in Fig. 7(b), while the scratch on the surface generated by the polish vanished and the surface of tungsten was erosion, remelting might occurred as the same time under HIPIB irradiation with the energy density of  $0.7 \text{ J}\cdot\text{cm}^{-2}$  which was showed in Fig. 7(c). It can be seen that from Fig. 7(d) and (e), irradiation damage such

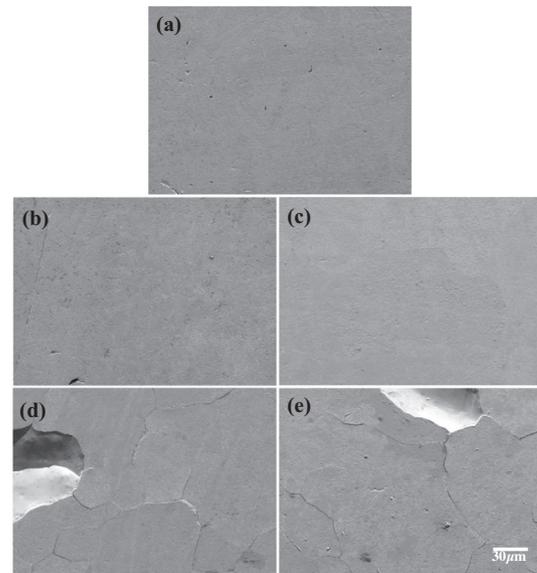


Fig. 7. The SEM surface morphology of HIPIB irradiated tungsten with 3 times and different energy density as (a) original sample, (b)  $0.25 \text{ J}\cdot\text{cm}^{-2}$ , (c)  $0.7 \text{ J}\cdot\text{cm}^{-2}$ , (d)  $1.4 \text{ J}\cdot\text{cm}^{-2}$ , (e)  $2.0 \text{ J}\cdot\text{cm}^{-2}$ .

as cracks and exfoliation occurred on the surface of tungsten when the energy density of irradiation up to  $1.4 \text{ J}\cdot\text{cm}^{-2}$  and  $2.0 \text{ J}\cdot\text{cm}^{-2}$ . Combining with Fig. 3, it can be drawn that the increase of ion energy density has more significant effects on irradiation damage of tungsten than the increase of pulse numbers.

Fig. 8 shows the GIXRD patterns of (110) plane in tungsten before and after HIPIB irradiation with 3 times and different energy density. It can be observed that comparing with original sample, the diffraction peak of tungsten shifts toward high angle after irradiation with the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  which indicates that the interplanar spacing of tungsten became smaller and stresses generated in the surface layer of tungsten due to the residual stress induced by the irradiation. The diffraction peak of tungsten shifts toward high angle further when the irradiation energy density up to  $0.7 \text{ J}\cdot\text{cm}^{-2}$  which declared the stresses increased with the irradiation energy density. There was no crack appeared on the

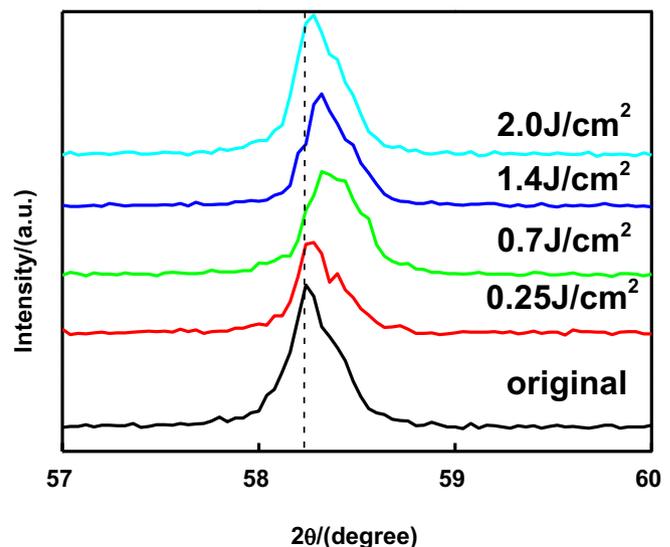


Fig. 8. The GIXRD patterns of body-centered cubic phase (200) plane in HIPIB irradiated tungsten with 3 times and different energy density.

surface of tungsten under this condition as it shows in Fig. 7(c), it indicated that the stresses accumulated in the surface layer didn't exceed the ultimate tensile strength of grain boundaries of tungsten. Fig. 8 exhibited that the shift of diffraction peak of the irradiated tungsten decreased when the energy density of irradiation up to  $1.4 \text{ J}\cdot\text{cm}^{-2}$ , and it decreased further when the energy density of increased to  $2.0 \text{ J}\cdot\text{cm}^{-2}$ . This was due to cracks and exfoliation appeared on the surface of tungsten (shows in Fig. 7(d) and (e)) which resulted in the stresses accumulated on the surface of tungsten released and the interplanar spacing restored partly.

Extremely high temperature gradient formed on the surface when HIPIB acted on the materials, which resulted in two kinds of stress generated in opposite direction in the interior of the material. The two kinds of stress would cause compression - stretching effect in the material [23]. The effect caused by HIPIB irradiation resulted in stress accumulated around the defects in the material. Irradiation damage such as cracks and exfoliation appeared on the surface of the material when the total accumulated stresses exceeded the tensile strength of the material. Defects in tungsten concentrated on the grain boundaries, thus the compression - stretching effect caused by HIPIB irradiation made the stresses accumulate at the grain boundaries of tungsten. The stresses accumulated in the surface layer didn't exceed the tensile strength of the grain boundaries in tungsten, so there were no cracks appeared on the surface when the energy density of irradiation was low ( $0.25 \text{ J}\cdot\text{cm}^{-2}$ ) and the irradiation times was few. While the irradiation times increased to 100 and 300, the accumulated stresses exceeded the tensile strength at the grain boundaries of tungsten, thus cracks generated along the grain boundaries on the surface of tungsten as shows in Fig. 3(d) and (e). The stresses accumulated at the grain boundaries of the tungsten within one pulse increased with the energy densities of HIPIB, when the energy densities were  $1.4 \text{ J}\cdot\text{cm}^{-2}$  and  $2.0 \text{ J}\cdot\text{cm}^{-2}$ , a few times of irradiation resulted in the accumulated stresses exceeded the tensile strength of the grain boundaries, which made cracks and exfoliation generate as shown in Fig. 7(d) and (e), thus the stresses released. This indicated that the grain boundaries of tungsten weaken the resistance against the strong thermal stresses induced by HIPIB irradiation. And for tungsten, the impact of the increase of energy density for the surface irradiation damage is more significant than the impact caused by the increase of irradiation times.

### 3.3. Effect of irradiation on the nanohardness of tungsten

Fig. 9 shows the relationship between nanohardness and depth in tungsten before and after HIPIB irradiation with the energy density of  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times. It can be seen that the original tungsten had the highest nanohardness. Compared with the original sample, the nanohardness of the surface of tungsten decreased slightly after 3 times irradiation. The hardness decreased with the increase of irradiation times, it decreased more significant when the irradiation times increased to 100 and 300. The changes of nanohardness in tungsten before and after irradiation mainly depended on the synergistic effect of micro-stresses on the surface and annealing effect in the surface of tungsten induced by the high temperature generated during the HIPIB irradiation. The former enlarged the nanohardness while the latter diminished the nanohardness. During the HIPIB irradiation, the annealing effect played a leading role, the nanohardness of tungsten decreased after the irradiation, it decreased more significant with the increase of irradiation times due to the increase of annealing numbers.

The relationship between the nanohardness and depth in tungsten before and after HIPIB irradiation with 3 times and different energy densities is showed in Fig. 10. It shows that the original tungsten had the highest nanohardness. After 3 times irradiation, the nanohardness of surface of tungsten decreased slightly when

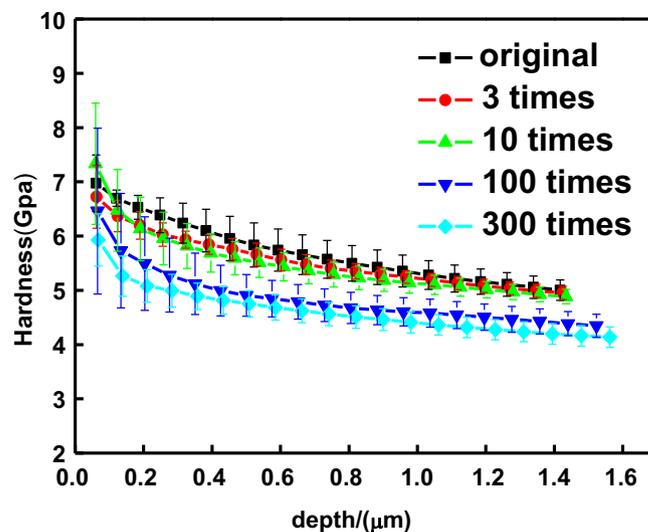


Fig. 9. The relationship between nanohardness and depth in HIPIB irradiated tungsten with energy density  $0.25 \text{ J}\cdot\text{cm}^{-2}$  and different times.

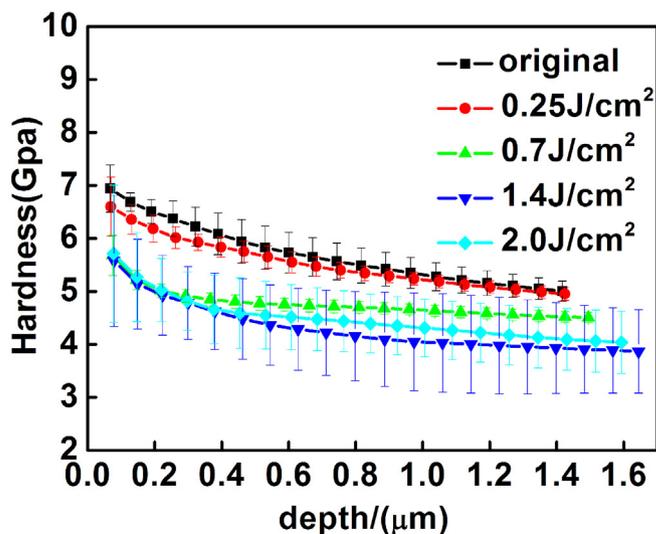


Fig. 10. The relationship between nanohardness and depth in HIPIB irradiated tungsten with 3 times and different energy density.

the energy density was  $0.25 \text{ J}\cdot\text{cm}^{-2}$ . While when the energy density of the irradiation up to  $0.7 \text{ J}\cdot\text{cm}^{-2}$ , the decrease became significant. It indicated that the energy densities of ion beams had greater influence on the nanohardness of the surface of tungsten. A higher energy density produced a higher temperature on the surface of tungsten during the irradiation and induced stronger annealing effect. Combined with the results of Fig. 9, it can be known that the impact of the increase of energy density on the nanohardness of the surface is more significant than the impact caused by the increase of irradiation times for tungsten.

HIPIB with the features of transient and high heat would generate extremely high thermal stresses in tungsten during the irradiation. The propagation of thermal stresses in the interior of tungsten result in high energy accumulation occurred at the grain boundaries of tungsten. The accumulated stresses might even exceed the ultimate tensile strength of grain boundaries in tungsten, and made serious irradiation damage such as cracks and exfoliation occurred on the surface of tungsten. And the high temperature induced by the HIPIB irradiation would bring out

annealing effect on the surface of tungsten, and then the nanohardness of tungsten decreased. It indicated that HIPIB is an efficient irradiation method so that it can be used to simulate the transient strong heat environment in the tokamak device and applied to the research on irradiation damage of materials. HIPIB also has the irradiation characteristics of ion beams and can implant ions into the surface of materials to simulate the effect of plasma irradiation on plasma facing materials.

There were no irradiation damage appear on the surface of tungsten when the energy densities of HIPIB were low ( $0.25\text{--}0.7\text{ J}\cdot\text{cm}^{-2}$ ) and the irradiation times were few (3–10), it indicated that tungsten has good anti-irradiation performance under high heat load so it could be used in the irradiation environment. Irradiation damage such as cracks and exfoliation appeared on the surface of tungsten when the energy densities of HIPIB were high and the irradiation times were major, so tungsten should be modified to improve its properties in order to apply to the environment with high heat load.

#### 4. Conclusion

In summary, the phase structure of tungsten was stable under HIPIB irradiation which indicated that tungsten has good resistance against the irradiation in the heat load environment. HIPIB is an efficient irradiation method and can be used to simulate the transient high heat environment in the tokamak device. Stresses accumulated at the grain boundaries in tungsten under HIPIB irradiation. Cracks and even exfoliation appeared on the surface of tungsten when the accumulated stresses exceeded the ultimate tensile strength of grain boundaries. During the HIPIB irradiation, impurity element accumulated on the surface of tungsten with the increasing of irradiation times. The salient points formed on the surface due to the remelting effect when the irradiation times were major. The nanohardness of the surface of tungsten decreased with the increase of irradiation times and energy densities, this was mainly because the high temperature generated by the irradiation induced an annealing-type effect. The impact of the increase of energy densities of HIPIB on the structure and properties is more significant than the impact caused by the increase of irradiation times for tungsten. The irradiation damage appeared on the surface of tungsten under high heat load environment limit the application of tungsten.

#### Acknowledgements

This work is financially supported by the National Science Foundation of China (No. 11375037 and No. 11675035). And the authors would like to thank Prof. Remnev G E at High-Voltage Research Institute at Tomsk Polytechnic University for his kind help on ion irradiation experiment.

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