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Effect of Silicon on microstructures of iron after high-temperature deuterium implantation

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Abstract: To study the effect of silicon on microstructures in CLAM (China Low Activation Martensitic) steel after irradiation, the microstructures of Fe and FeSi model alloys were investigated after implanted deuterium ions at 773K. TEM (Transmission Electron Microscope) observation and EDX (Energy Dispersive X-ray Spectrum) analysis have been carried out. The results showed that tiny voids were observed in Fe after implantation, while no void observed in FeSi model alloys. Instead, there were some carbides which indicated silicon played an important role in the improved irradiation resistance. Fast diffusing mechanism was adopted to analyze the effect of silicon during irradiation.

Introduction

CLAM steel is considered as the primary Chinese test blanket module of International Thermonuclear Experimental Reactor (ITER) and in the designs of FDS series PbLi blankets for the future fusion reactors[1,2]. The mechanical properties of CLAM steel were paid much attention by researchers[3]. As is well known, the microstructure can greatly affect the mechanical properties of steels[4]. In the irradiation condition, the most important microstructure evolution are the formation of defects and precipitates. However, it is difficult to obtain the exact results because there are many alloy elements in CLAM steel. So it is necessary to analyze the evolution of defects and possible precipitates separately.

Silicon is a strong element to form ferrite which can improve mechanical properties of steels [5,6]. Early studies found that the addition of silicon reduced the binding force, increased the irradiation embrittlement, thereby, unfavorable to the irradiation resistance. However, silicon can resist the irradiation swelling rate in austenite steel. Besides, silicon is not the main reason to cause irradiation embrittlement. Silicon affects relative parameters of thermodynamics and kinetics of phosphorus segregation in grain boundary. Therefore, the segregation of phosphorus could be induced and lead to embrittlement. If there is little phosphorus in steel, for example FeSi model alloy in this study, silicon maybe resist irradiation embrittlement.

In this paper, the microstructures of Fe and FeSi model alloys were observed. Irradiation swelling theory was used to simulate the evolution of bubbles in small displacement damage. Fast diffusing mechanism was used to analyze the effect of silicon. As modeling research, the result aimed to the effect of silicon on microstructures of CLAM steel.

Experimental

Fe and FeSi model alloy were prepared to disks with 100μm thick and 3mm diameter. Heat treatment was done at 1023K and held for half hours then air cooling. The final TEM specimens were polished by twin-jet electro-polisher using 5% $HCLO_4$-95%C_2H_5OH polishing solution.

Deuterium ions implantation was performed at 773K up to a fluence of 5×10^{17} D^+/cm^2 by an ion accelerator at Institute of Semiconductors of the Chinese Academy of Sciences. The accelerator voltage was 58keV for Fe calculated by SRIM (the Stopping and Range of Ions in Matter). After implantation, TEM observation was done using Tecnai F20 FETEM (Field Emission Transmission Electron Microscope).

Result and discussion

As a contrast, microstructures of post-implanted Fe were also observed, shown in Fig.1. TEM examinations of the post-implanted specimens revealed a high density and small size voids[7]. The size of which was approximately 20 nm and the number density was $1\times10^{11}/cm^2$.

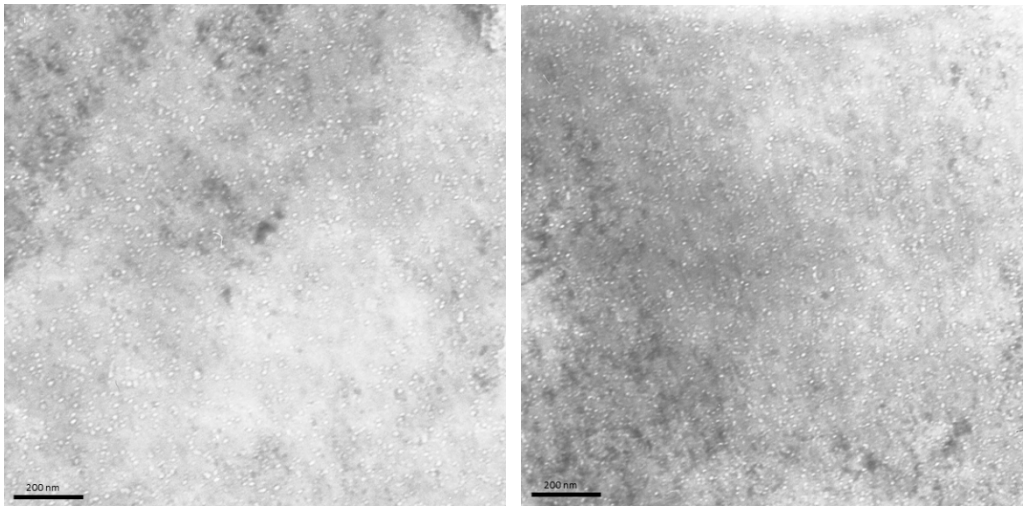

Fig. 1 microstructures of Fe after deuterium implatation at 773K

These voids seemed to be polyhedron[8]. To calculate the swelling rate conveniently, According to the swelling rate formula, these voids were supposed as regular spherical. The equation is as follows:

$$S = \frac{\Delta V}{V} = \frac{\pi}{6xyt}\sum d^3 \tag{1}$$

Where, x, y means the area of the field of view, nm; t means the thickness of the field of view calculated by extinction coefficient ξ_{eff} g(3g), nm; d is the mean diameter of voids, nm. According to Fig. 1, the swelling rate was 0.025%.

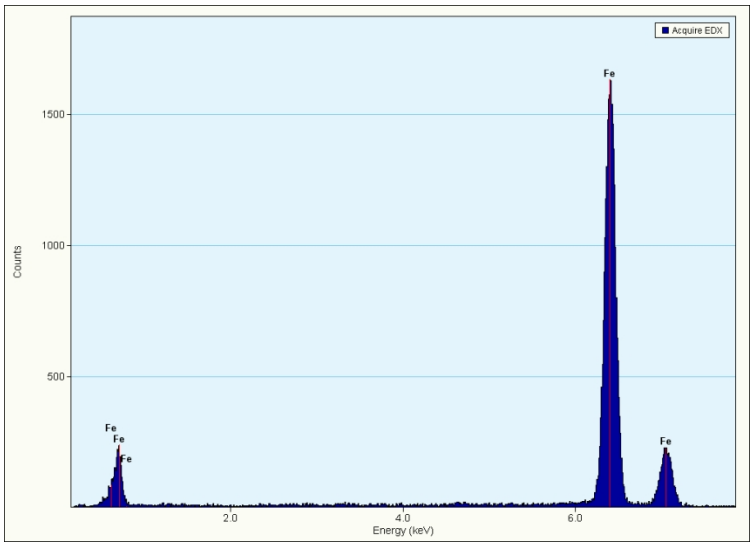

Fig. 2 the composition of Fe after deuterium implatation at 773K

From Fig 2, no precipitate or impurity was observed. That means, there were only high density and small size voids in Fe after implantation. Fig. 3 illustrates micorostructures of FeSi model alloy after deuterium implantation at 773K. Different from microstructures in Fe, there seemed to be some precipitates in FeSi model alloy whose size was 500 nm.

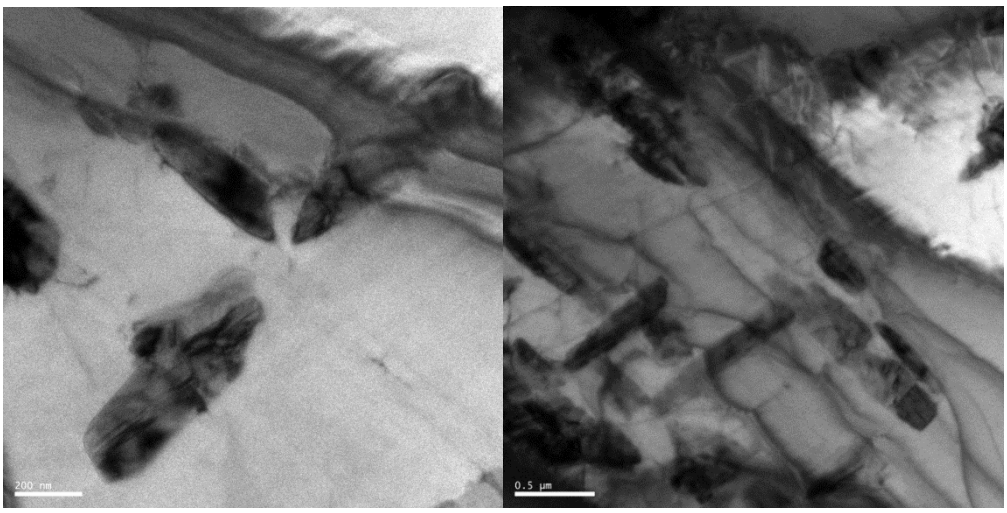

Fig. 3 microstructures of FeSi after deuterium implatation at 773K

And there was no obvious void which meaned the addition of silicon effectively suppressed the formation of void. The irradiation swelling resistance of silicon may be due to the fast diffusing mechanism[9,10]. When materials are suffered from irradiation, the swelling could be divided into three stages, that is, incubation, steady-state and saturation regime. The effect of silicon on irradiation swelling mainly lies in the extension of incubation. Silicon is a fast diffusing element with large diffusion coefficient at high temperature. As an alloy element in steel, the diffusion of silicon is through vacancy diffusion. As a result, the fast diffusion of silicon could obviously improve the effective mobile ability of vacancy, decrease the supersaturation of vacancy, thus reduce the nucleation rate of void and suppress the formation of void.

Based on the analysis above, silicon extended the incubation of swelling. FeSi model alloy still stayed in this stage under the irradiation dose of 5×10^{17} D^{+}/cm^{2}. So no void was observed. It indicates that silicon could suppress the formation of void in Fe. FeSi has the lower swelling rate than Fe.

Furthermore, the compositions of precipitates were analyzed by EDX. The results were shown in Fig. 4 and Table 1.

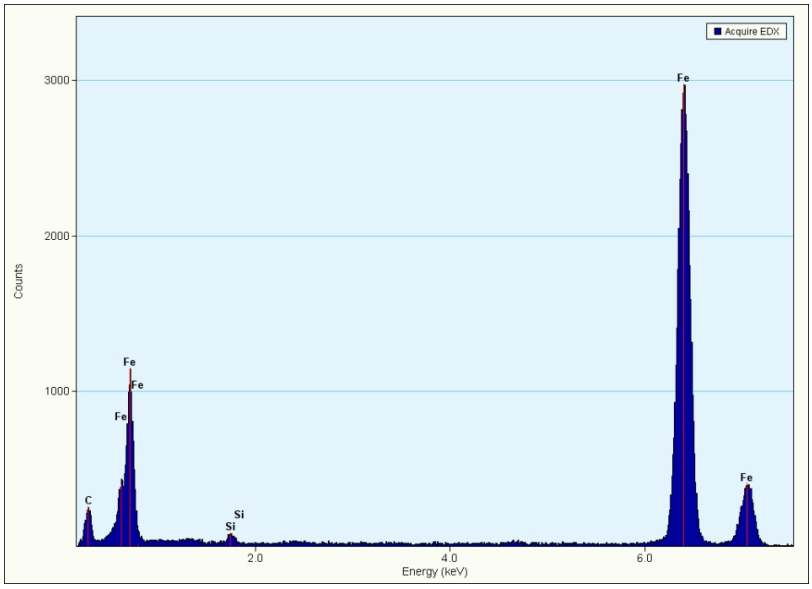

Fig. 4 the compositions of FeSi after deuterium implatation at 773K

Table 1 the compositions of precipitates of FeSi model alloy [wt.%]

element	C	Si	Fe
content	8.24	0.75	90.99

After implantation, the content of carbon in precipitates was obviously increased. Thus it can be seen that the formation of some carbides[11]. The research in strengthen toughening theory of alloy found that silicon could dissolve in ε-carbides, improved the stability of carbides, put off the transformation process from ε-carbides to θ-carbides[12]. And then it made the ε-carbides keep coherence with matrix, meanwhile, distributed evenly. As thus, alloy obtained well obdurability. Besides, silicon could strengthen martensite steel through solid solution and suppress the formation of Fe_3C, as a result, much carbon retained in martensite, which ensured the enough strength of martensite after tempering. Silicon could also affect the size and distribution of MC and M_2C and improve the effect of secondary hardening. Finally, it improves the strength of material further.

In addition, there were some dislocation loops in post-implanted FeSi model alloy with the mean size of 200 nm (shown in Fig.5). This is another reason which leads to hardening.

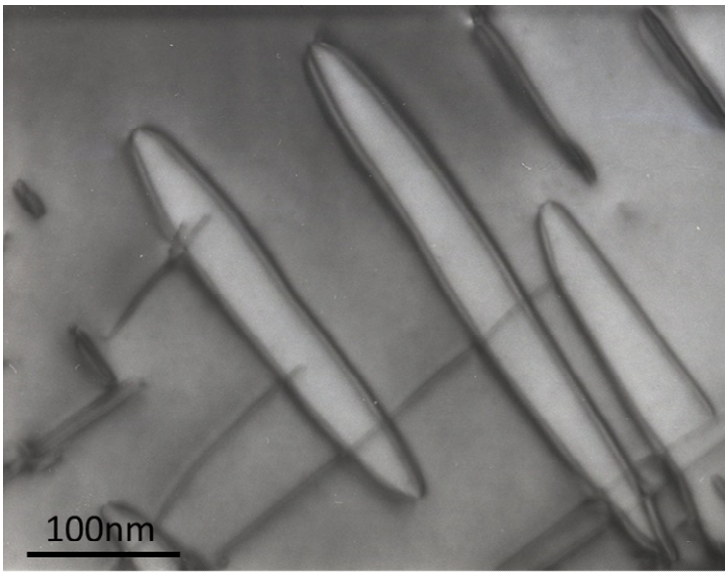

Fig. 5 the dislocation loops observed in FeSi after deuterium implantation at 773K

Conclusions

After deuterium implantation, voids were observed in Fe with the mean size of 20 nm and the number density of $1\times10^{11}/cm^2$. The irradiation swelling is about 0.025%. Carbides and dislocation loops were observed in FeSi, of which carbides act with silicon suppress the formation of void in Fe. FeSi has the lower swelling rate than Fe.

Acknowledgments

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