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# Effect of He and H synergy on mechanical property of ion-irradiated Fe-10Cr alloy

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ABSTRACT

The effect of helium and hydrogen synergy on mechanical property (hardness and elastic modulus) and microstructural has been investigated in Fe-10Cr alloy as a reduced-activation ferritic/martensitic steel model following single-$He^+$, $H^+$ and sequential-($He^+$ + $H^+$) ion irradiation at 773 K, to provide basic understanding concerning the development of fusion reactor components. Nano-indentation results showed that pronounced irradiation hardening was induced by single and sequential ion irradiation. However, hardening due to sequential (He + H) ion irradiation (48%) was smaller than that of single ion irradiations. The elastic modulus of Fe-10Cr alloy encountered a decrease by sequential-(He + H) ion irradiation. No remarkable modulus changes were measured in samples with single He and H ion irradiation. The Orowan mechanism was adopted to correlate damage microstructure and hardening, indicating that the hardening can be attributed to the formation of defects such as dislocation loops and cavities. The synergistic effects of He and H on the defect evolution and irradiation hardening in Fe-10Cr were discussed.

## 1. Introduction

Reduced activation ferritic/martensitic (RAFM) steels with Cr contents ranging between 9% and 12% are among the most promising candidates for structural materials in future fusion and advanced fission reactors because of their low swelling, high thermal conductivity, low helium (He) embrittlement and thermal shock resistance [1–5]. The physical and mechanical properties of these steels in non-irradiated form are reasonably well understood due to their large-scale use, e.g. in chemical and petrochemical industries, and coal-fired power plants [6]. However, the key to success for ensuring safe and satisfactory operation in a high-temperature radiative environment lies in a basic understanding of the effects of radiation on these materials. Thus, many basic researches have been focused on binary Fe-Cr model alloys, which are representative of RAFM steels.

Exposure of metals to irradiation results in a production of numbers of vacancies and interstitials during the collision between energetic particles and target atoms. These point defects (vacancies and interstitials) will experience migration and accumulation, giving rise to the formation of dislocation loops and cavities [7]. These defects agglomerates increase the stress required for yield and subsequent plastic flow and lead to irradiation hardening, embrittlement and swelling.

Production of He by nuclear transmutation in components of nuclear power reactors has been recognized for some time to lead to detrimental effects on the materials properties [8]. He generation rates in steels increase in the order of fast reactors, fusion devices, light water reactors, and accelerator-driven spallation devices [9]. Hydrogen (H) generation also increases in the same order, but usually at higher levels than He [9]. Interstitial He atoms will rapidly precipitate out at nearby sinks (vacancies, vacancy complexes, and grain boundaries), where they are strongly trapped [10]. As He-atom clusters grow, they eventually eject lattice atoms to form cavities. The stabilization of cavities by He results in void swelling and blister formation [11,12]. H was thought to play a secondary role compared with He due to its easy desorption from the steel because of its high mobility. However, recently H is known to be strongly captured in He-nucleated voids or bubbles, thereby contributing to cavity stabilization [13]. Furthermore, in some alloy systems (vanadium alloys [14] and RAFM alloy [15,16]) He and H appear to interact synergistically to strongly promote swelling induced by cavity although the synergetic mechanism need to be further revealed. The synergetic effects on dislocation loop evolution also need to be concerned.

Meanwhile, the potentially synergistic effects of He and H on mechanical property including irradiation hardening and modulus change need to be well studied. He atoms can induce extra component of radiation hardening and the synergistic effects of He and Fe ion have been reported for F82H steel [17]. More recently, the effect of $He^+$ and sequential $Fe^+/He^+$ ions irradiation on irradiation hardening of Fe-Cr

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**Table 1**
Composition of carbon and nitrogen in Fe-Cr specimen.

| Specimen | C (wt.%) | N (wt.%) |
|---|---|---|
| Fe-10Cr | 0.0028 | 0.0012 |

based Eurofer-steels at 573 K have been reported [18]. The hardening effect of simultaneous $Fe^+/He^+$ implantation is larger while sequential $Fe^+/He^+$ irradiation is smaller than single $Fe^+$ irradiation. Specifically, the single-beam He-ion implantations do not give rise to significant hardening in Eurofer97 even may cause softening in Fe-9%Cr alloy have been reported also [18]. However, for CLAM steel, single-($He^+$) ions irradiation can induce vacancy-type defects and give rise to obvious hardening [19,20]. Effects of He and sequential He and Fe/H ions on the hardening of RAFM steels need to be further studied. The study of sequential-ion irradiation is beneficial to identify the individual role of He and H in the synergistic effects. Furthermore, less is reported about the effect of irradiation on the elastic modulus change in RAFMs, let alone the synergistic effects of He and H [21,22].

The present paper investigates the synergistic effects of He and H on the mechanical properties (hardness and elastic modulus) and microstructure change of Fe-Cr model alloy by using single He, H and sequential (He + H)-ions irradiation. Irradiation hardening was measured by nano-indentation and correlated with microstructure. Modulus changes have been tested and analyzed also. Microstructure changes before and after ion irradiation has been observed and analysed by transmission electron microscopy (TEM). The effect of He and H synergy on the irradiation hardening and elastic modulus change in Fe-10Cr is discussed in detail.

## 2. Experimental

### 2.1. Materials and ion irradiation

The material analyzed in this study is Fe-10wt%Cr, a model alloy of RAFM steels. Chemical compositions of samples were listed in Table 1. The Fe-Cr alloy was melted in a vacuum induction furnace into 5 kg ingots and subsequently hot-forged at 1473 K into 150 mm × 35 mm × 35 mm bars. The surfaces of the samples were mechanically polished to remove the oxide layer. The samples were normalized at 1253 K for 30 min followed by quenching in water at room temperature. Single- and sequential-ion irradiations with energy of 100 keV were performed in an ion accelerator under a vacuum of $\sim 10^{-4}$ Pa at 773 K with the accuracy of ± 10 K. The detailed irradiation parameters are given in Table 2. Single-$He^+$ and $H^+$ were implanted into the Fe-Cr alloy with the dose of $3 \times 10^{16}$ $He^+/cm^{-2}$ and $7 \times 10^{16}$ $H^+/cm^{-2}$, respectively. Sequential-ion irradiation refers to He ($3 \times 10^{16}$ $cm^{-2}$) immediately followed by H ($7 \times 10^{16}$ $cm^{-2}$) ion. The dose rate was $2.57 \times 10^{11}$ ions/$cm^2$/s. Displacement damage profile and He and H concentration profile in Fe-Cr model alloy are shown in Fig. 1. It was calculated by SRIM 2013 with displacement threshold energy 40 eV and based on the Kinchin-Pease calculation, as recommended by Stoller et al. [23,24]. The displacement depth and ions range are approximately < 600 nm according to the calculation.

### 2.2. Mechanical property test

Given the limitation on ion irradiation depth and sample size, conducting conventional tensile tests was not practical. The mechanical properties (hardness (*H*) and elastic modulus (*E*)) of the irradiated and unirradiated samples were tested by nano-indentation which is a widely used technique for the study on mechanical properties of materials at nanoscale [25–27]. Nanoindentation hardness and modulus were measured by using depth-control mode in ambient atmosphere with an MTS Nanoindentation XP system. As the nucleation of dislocation within the plastic zone, the indentation hardness of materials is always observed to increase with decreasing the depth of penetration, known as the indentation size effect (ISE) [25]. For irradiated materials, there is not only the ISE but also damage gradient effect (DGE) which means inhomogeneous damage in the sample. Here we evaluate the indentation hardness (*H*) in terms of the instinct hardness ($H_L$) of irradiated area and hardness ($H_S$) of un-irradiated area of the materials by using a new nano-indentation model, which is described as follow:

$$H = \frac{H_L - H_S}{1 + k\left(\frac{h}{t}\right)^2} + H_s + \frac{A}{h} \tag{1}$$

where H is the composite hardness, $H_L$ and $H_S$ represent the irradiation harden layer and substrate (unirradiated layer) hardness respectively, and k is a constant that characterize the change in hardness as the indenter passes from the layer to the substrate and therefore constitute an important feature of the film-substrate system. h/t denotes the indentation depth (h) normalized with respect to the harden layer thickness and has been termed the relative indentation depth. Based on the Nix-Gao model and a film/substrate system model [28,29], the new model was proposed to describe the composite hardness of the ion-irradiated materials and explained the ISE and in homogeneous damage, and described in another reference in detail [30].

The tip truncation was calibrated using fused silica as a reference specimen. The allowable thermal drift rate was limited to 0.05 nm/s. For each sample, eight measurements were taken randomly to obtain typical results. Continuous stiffness measurement (CSM) method was applied to obtain the *H* and *E* vs. depth (*h*) profile continuously up to a depth of about 1000 nm [31]. The calibration of the bluntness of the indentation tip is based on the Oliver-Pharr method [32]. The continuous stiffness was measured with a harmonic displacement of 2 nm and 45 Hz frequency.

### 2.3. Microstructural characterization

The microstructural observation and structural evolution of Fe-Cr (unirradiated, single- and sequential-ion irradiated samples) were conducted using common TEM. Specimens suitable for TEM studies were prepared by standard techniques [33,34]. Specimens of 3 mm disc were punched out from a 0.3 mm thick strip which was prepared from the model alloy bar by electro discharge machining and mechanically milling to a final thickness of 0.08 mm. Then, samples were polished by diamond paper to obtain good surface. Before irradiation, samples were heated at 1023 K for 90 min and followed by air cooling to remove strain effect. The final TEM specimens were electro-polished in 5% $HClO_4$ + 95% $C_2H_5OH$ solution.

TEM observation including dislocation loops and voids were performed using JEOL-2010 and FEI Tecnai F20. The accelerating voltage

**Table 2**
Irradiation condition of sequential ion-beam in this study.

| Condition ID | Kinds of ions | Energy of ions (keV) | Displacement damage (dpa) | He concentration (at.%) | H concentration (at.%) |
|---|---|---|---|---|---|
| Single (He) | $He^+$ | 100 | 1.88 | 1.95 | 0 |
| Single (H) | $H^+$ | 100 | 0.54 | 0 | 5.43 |
| Sequential (He + H) | $He^+ + H^+$ | 100 | 2.07 | 1.95 | 5.43 |