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Depth distribution of Frank loop defects formed in ion-irradiated stainless steel and its dependence on Si addition

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ABSTRACT

Although heavy ion irradiation is a good tool to simulate neutron irradiation-induced damages in light water reactor, it produces inhomogeneous defect distribution. Such difference in defect distribution brings difficulty in comparing the microstructure evolution and mechanical degradation between neutron and heavy ion irradiation, and thus needs to be understood. Stainless steel is the typical structural material used in reactor core, and could be taken as an example to study the inhomogeneous defect depth distribution in heavy ion irradiation and its influence on the tested irradiation hardening by nano-indentation. In this work, solution annealed stainless steel model alloys are irradiated by 3 MeV Fe^{2+} ions at 400 °C to 3 dpa to produce Frank loops that are mainly interstitial in nature. The silicon content of the model alloys is also tuned to change point defect diffusion, so that the loop depth distribution influenced by diffusion along the irradiation beam direction could be discussed. Results show that in low Si (0% Si) and base Si (0.42% Si) samples the depth distribution of Frank loop density quite well matches the dpa profile calculated by the SRIM code, but in high Si sample (0.95% Si), the loop number density in the near-surface region is very low. One possible explanation could be Si's role in enhancing the effective vacancy diffusivity, promoting recombination and thus suppressing interstitial Frank loops, especially in the near-surface region, where vacancies concentrate. By considering the loop depth distribution, the tested irradiation hardening is successfully explained by the Orowan model. A hardening coefficient of around 0.30 is obtained for all the three samples. This attempt in interpreting hardening results may make it easier to compare the mechanical degradation between different irradiation experiments.

1. Introduction

The material degradation behavior under neutron irradiation is a key parameter to be evaluated when selecting suitable materials to be used in light water reactors. But due to the high cost, long irradiation time of neutron irradiation and the complicated procedures to handle specimens with induced radioactivity, heavy ions are often preferred instead of neutron as an alternative tool for the preliminary selection of reactor materials.

Unlike the homogeneous damage introduced by neutron, the damage of heavy ion irradiation varies at different depths. This may result in different microstructure evolution processes at different depths [1–3]. Also, since the irradiation damage region could be close to the top surface in heavy ion irradiation, surface sink may also play a role on defect depth distribution as it can absorb nearby point defects. Thus, the depth distribution of radiation defects is one major difference between heavy ion and neutron irradiation, and needs to be understood before heavy ion irradiation could be confidently used and even be calibrated to neutron irradiation-induced damages.

Stainless steel is the typical structural material used in light water reactor core. Since its degradation process under irradiation is one important issue when considering reactor safety, it could be taken as a typical example to understand the heterogeneous characteristic of heavy ion irradiation damage. Quite many previous works have been done on the depth distribution of cavities in stainless steel irradiated by heavy ions. The depth distribution of vacancy point defects can be directly inferred from observed cavity

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distribution, while the depth distribution of interstitials is usually indirectly reflected only [4,5]. It will be helpful to understand the heavy ion irradiation damage if more depth distribution data about interstitial dislocation defects could be obtained [6]. To achieve this, thin cross-section transmission electron microscopy (TEM) samples covering all irradiation-affected depths are required, and reliable observation technique is also needed for the dense overlapping dislocation defects.

Besides the microstructure evolution during irradiation, the corresponding mechanical property degradation is also the focus when evaluating the behavior of nuclear materials in reactors. In neutron irradiation, tensile strength test, Charpy impact test and hardness test are applicable for the evaluation of mechanical degradation, because the penetration depth of neutron is large in irradiated materials. But in the case of heavy ion irradiation, the irradiation damage layer is roughly around several micrometers thick or even thinner, so nano-indentation test is usually the choice for mechanical property evaluation. However, due to the inhomogeneous distribution of radiation defects formed in heavy ion irradiation, the tested nano-hardness results largely depend on the indentation depth selected [7,8]. This brings difficulties in comparing the hardening results between different research groups and also in building up hardening databases. The quantitative interpretation of the nano-indentation results in heavy ion irradiation needs to be improved.

In neutron irradiation, the irradiation hardening results can be quantitatively explained by the homogeneously-distributed radiation defects through the Orowan model [9,10]. However in heavy ion irradiation, since the defects distribution is inhomogeneous, the defect number density and the average size at any specific depth are not representative for the whole specimen, and thus cannot be directly used in the Orowan model. An algorithm to take the inhomogeneous defect distribution into consideration is needed to apply the Orowan model in heavy ion irradiation.

In this work, 316 model alloys are irradiated at 400 °C, because at this relatively high temperature, dislocation defects are mainly interstitial in nature [11,12]. Focused ion beam (FIB) technique is applied, and reciprocal lattice rod (relrod) technique purposed by D.J. Edwards et al. is used to distinguish the depth distribution of dense Frank loops [13]. Theoretically, the depth distribution of dislocation loops should be determined by the damage profile of irradiation and the diffusion of point defects along the irradiation beam direction. As the alloying element silicon is believed to substantially change the point defect diffusion [14–16], silicon content in model alloys is tuned to study the role of point defect diffusion along the irradiation beam direction on loop depth distribution. Attempts are made to apply the Orowan model to heavy ion irradiation by utilizing the depth distribution of Frank loops observed.

2. Experimental

2.1. Samples for irradiation

Three high purity 316L austenite stainless steel model alloys are selected in this work. Table 1 shows their composition, and their major difference is the Si content. These model alloys are solution annealed before irradiation, and are then cut into small pieces of ~10 × 2 × 0.5 mm. The surface for irradiation is polished by emery papers, and is then buff polished. Finally electrochemically polishing is performed by electrolyte of 10% perchloric acid and 90% acetic acid at around 10 °C to obtain a smooth surface free of possible mechanical hardening.

The heavy ion irradiation is performed in beam line 5 in High Fluence Irradiation Facility, The University of Tokyo. Samples are irradiated by 3 MeV Fe^{2+} at 400 °C, and the irradiation beam is perpendicular to the sample surface. Irradiation flux is 5.9×10^{11} cm^{-2} s^{-1}, and the fluence is 4.8×10^{15} cm^{-2}. The damage profile and Fe ion implantation profile are calculated by the SRIM 2013 code [17] with displacement energy of 40 eV [18,19], as shown in Fig. 1. The corresponding irradiation dose is ~3 dpa, which means the average dpa from the sample top surface to the peak dpa depth.

A total number of 20 Faraday cups are installed at the specimen chamber end to monitor the actual irradiation beam current. Each Faraday cup is 2 mm in diameter. The distance between the centers of each two faradays cup is kept at 4 mm, and the 20 Faraday cups are placed in a 5 × 4 array to ensure a uniform current distribution on the specimen stage. Beam current is checked at every 30 min to confirm its stability. Dose rate is controlled to be around 4×10^{-4} dpa/s. Irradiation temperature is controlled by two thermal couples, and their fluctuation in reading number during irradiation is less than ±2 °C.

The irradiation hardening of the three samples has been confirmed by nano-indentation test before and after irradiation. The indentation is performed by Shimadzu DUH-211 with a Berkovich indenter at a constant indentation depth of 200 nm. 200 indents are performed for each test, and the distance between each two indents is kept at 30 µm. The tested irradiation hardening for low Si, base Si and high Si samples are 0.78 GPa, 0.55 GPa and 0.36 GPa respectively, and the indentation details are described elsewhere [20]. The tested hardening is quite large in value, so dense radiation defects should have formed in all the three samples.

2.2. FIB sampling

Transmission electron microscopy (TEM) cross-section specimens are prepared by focused ion beam (FIB) technique. The FIB

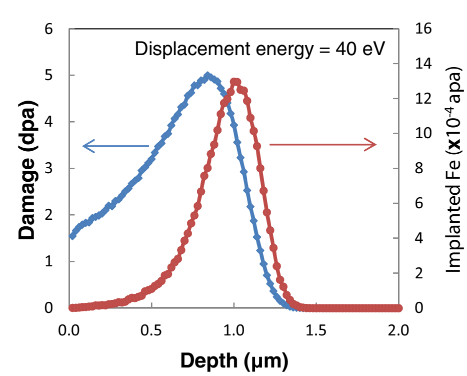

Fig. 1. Damage and ion implantation profile of 3 MeV Fe^{2+} irradiation calculated by SRIM code. The profile of induced damage is shown in dpa (displacement per atom) and implanted ion concentration in apa (atoms per atom).

Table 1
Chemical composition of high purity 316L stainless steel model alloys.

	Alloying elements (wt.%)						Impurity (wt. ppm)				
	Fe	Cr	Ni	Mo	Mn	Si	P	S	Ca	C	N
Low Si (LS)	Bal.	16.9	13.0	2.29	1.00	<0.001	–	<10	–	82	<10
Base Si (BA)	Bal.	17.3	13.2	2.36	0.88	0.42	–	<10	–	111	<10
High Si (HS)	Bal.	17.0	13.5	2.27	0.99	0.95	–	<10	–	97	<10