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Effect of temperature and dose on vacancy-defect evolution in 304L stainless steel irradiated by triple ion beam

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ABSTRACT

This study aims to investigate the effects of temperature and dose on the vacancy-defect evolution in 304L stainless steel irradiated by the triple beam of Ni, H, and He ions below 673 K, which is relevant to water-cooled nuclear energy facilities. The irradiation experiments were performed simultaneously using 150-MeV Ni ions whose energy was degraded by passing through a tantalum foil of 8.5-μm thick, 100-keV H ions, and 200-keV He ions at 473, 523, 573, 623, and 673 K, respectively. The irradiation-induced microstructures in the steel were probed by the slow positron beam technique. The results show that the S parameter of the irradiated sample decreases as the temperature increases, and the S parameter of the high-dose irradiated sample decreases further. We explained the results by analyzing the structure of the defects in the irradiated samples at various temperatures and the effects of the annealing effect. In addition, the analysis of the S–W plots reveals the transition of the helium-vacancy cluster to the overpressured He_mV_n $(m > n)$ cluster under the influence of temperature and irradiation dose.

1. Introduction

The real irradiation environments for structural materials in nuclear energy facilities would be characterized by a high displacement damage rate, and a large amount of hydrogen and helium atoms induced by nuclear transmutation reactions. The heavy ion accelerated by a high-energy accelerator, which can generate substantial displacement damages on structural materials, could simulate high fluence neutron irradiations and the simultaneous triple-beam irradiations with heavy ions, hydrogen and helium, to simulate the real environments of nuclear reactors. The simultaneous irradiation of Ni, H, and He ions has been reported to promote cavity formation and swelling [1,2]. Lee et al. revealed that synergistic effects of displacement damages and hydrogen/helium atoms have a significant impact on the mechanical property of the ferritic steels [3]. In fact, in triple beam irradiation of steels at elevated temperatures beyond 723 K, the synergistic effects of dpa and hydrogen/helium on the microstructural evolution have been reported to be modest [4–10]. However, these studies primarily focus on the synergy effect between ion species at high temperatures, neglecting the effects of irradiation conditions. This work aims to investigate the effects of irradiation conditions on the vacancy-like defect evolution in 304L stainless steel irradiated by the triple beam of Ni, H, and He ions below 673 K, which is relevant to water-cooled nuclear reactors, using the Doppler broadening spectroscopy of slow positron.

2. Experimental procedures

The chemical composition of the 304Lstainless steel (SS) investigated in this work is listed in Table 1. As-received bulks were treated in solution at 1080 °C for 1 h followed by water quenching. The bulk specimens were fabricated into square sheets of

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Table 1
Chemical composition of the 304Lstainless steel.

Materials	Fe	C	Si	Mn	P	S	Ni	Cr	Mo
304L	Bal	0.015	0.54	0.97	0.027	0.006	10.1	18.6	0.3

Table 2
The accumulated fluences of hydrogen, helium, and nickel for each dpa value at the peak.

Peak dpa value	*H (ion/cm^2)*	*He (ion/cm^2)*	*Ni (ion/cm^2)*
0.5	0.7×10^{13}	1.1×10^{14}	4.2×10^{14}
2	2.3×10^{13}	3.8×10^{14}	3.2×10^{15}
6	4.6×10^{13}	7.6×10^{14}	1.0×10^{16}
15	1.1×10^{14}	1.9×10^{15}	2.5×10^{16}

1 mm × 10 mm × 10 mm. All specimens were mechanically polished before the irradiation experiment, initially with sandpaper of varying grits and finally, to a mirror-like finish with 1-μm diamond paste.

The triple simultaneous irradiation (Ni + He + H) was performed at the HI-13 tandem accelerator located at the China Institute of Atomic Energy. The details of the triple ion beam irradiation facility can be found in Ref. [11]. In our study, a 150-MeV Ni ion has been utilized to produce bulk damages, and the energy degrader of a tantalum foil of 8.5-μm thick was added such that the damage peak can be shifted to ~1-μm from sample surface. The 200-keV He-ion and 100-keV H-ion beams from the implanters were implanted into the steels. To simulate the production ratio of hydrogen, helium, and the displacement damages in the reactors, the fluxes of hydrogen, helium, and nickel ions were fixed at 1.3×10^{13}, 2.1×10^{14}, and 8.4×10^{14} ions/(cm^2 s), respectively. The SRIM [12] calculated damage rate is 0.016 dpa/s at peak, where the contribution values of hydrogen and helium are 6.6×10^{-7} dpa/s and 1.1×10^{-4} dpa/s, respectively. The H/He distribution ranged from ~200 nm to ~700 nm, as shown in Fig. 1. Two sets of samples were irradiated to 0.5 dpa and 6 dpa separately at 473, 523, 623, and 673 K. Another set of samples was irradiated at 573 K to 0.5, 2, 6, and 15 dpa, respectively. All implanted fluence were accumulative and the detailed fluences of implantation are shown in Table 2. The peak dpa value is also shown in the table for each fluence.

Doppler broadening spectroscopy (DBS) measurements were performed at ~ 295 K using an energy-variable slow positron beam facility established within the Institute of High Energy Physics (IHEP), CAS. The energy range of the mono-energetic positron beams are 0.18 eV–20 keV, which covers the depth up to ~652 nm in the 304L SS. The experimental details have been published elsewhere [13–15]. The DBS is characterized by an S (for shape) parameter, defined as the ratio of counts in a fixed central area (of width 1.6 keV and centered around 511 keV) of the DB spectra to the total number of counts in the spectra. When the positrons annihilate after being localized near the vacancy-like defect sites, annihilation occurs predominantly with free electrons as opposed to the defectless crystal or impurity atom-vacancy complex where positrons are more likely to annihilate with the core electrons. Consequently, in the presence of vacancy-like defects, the annihilation photo peak in the DB spectra is sharp and produces a higher S parameter. For the positron annihilation in the defect-free crystal or impurity atom-vacancy complex, the annihilation photo peak is flatter, and the W parameter is larger.

3. Results and discussion

The high temperature irradiation process is also an annealing process, so we perform a positron Doppler broadening measurement after annealing the raw sample at the corresponding temperature as a reference for the irradiated sample. Fig. 2 shows the S parameters as a function of positron implantation energy for increasing annealing temperature. For the depths larger than typically 120 nm the S parameters show no variation with depth. However, a decrease of S value with increasing temperature is evident and is ascribed to a decrease in the concentration of vacancy type defects. Figs. 3 and 5 show the dependence of the S parameter on the implanted positron energy (S-E curve) for the triple-beam specimen irradiated to 0.5 dpa and 6 dpa at different temperatures. As shown, the S parameter of the irradiated sample decreases as the temperature increases, and the S parameter of the high-dose irradiated sample decreases further. The S parameters of the samples irradiated by the two doses differ with the temperature, because the defect structures change with the irradiation time. It is well-known that samples irradiated by high-energy nickel ions produce vacancy defects, but these generated defects have different structures at different temperatures [16]. In order to eliminate the effects of non-irradiation effects such as surface effects, the fitting program VEPFIT [17] based on the multi-layered

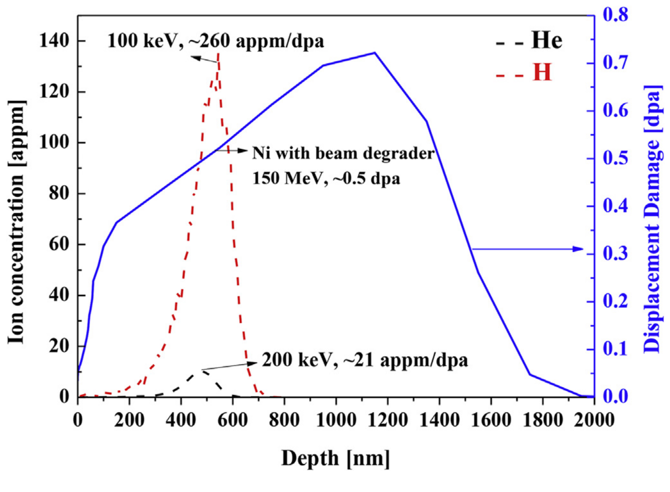

Fig. 1. Profiles of ion concentration in 304L SS irradiated with 100-keV H, 200- keV He, 150-MeV Ni ion calculated with SRIM-2013.

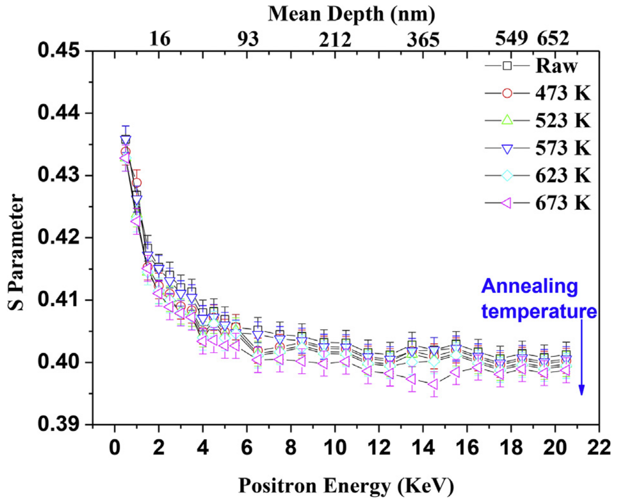

Fig. 2. Annealing study of 304L SS: S parameters obtained with the DBS.