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Proton-irradiation induced defects in modified 310S steels characterized with positron annihilation spectroscopy and transmission electron microscopy

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ABSTRACT

An effective method to improve the irradiation resistance of austenitic stainless steels is adding oversized solutes into steels. In this work, the irradiation resistances of two type of modified 310S steels, in one of which Zr was added and in another Nb, Ta, and W were added, were investigated by proton irradiations at 563 K. Irradiation induced vacancy-type defects was characterized with positron annihilation spectroscopy (PAS), while dislocation loops and bubbles whose size are greater than 1 nm are characterized with transmission electron microscopy (TEM). It is found that the relative S parameter ΔS/S extracted from PAS is more effective than S parameter in evaluating the quantity of vacancy-type defects. It was revealed from ΔS/S that more vacancy-type defects produced in (Nb, Ta, W)-added steels than that in Zr-added steels, and this trend became more obvious with the dose increasing. S-W curves reveal that proton irradiation induced two kinds of vacancy-type defects, i.e. vacancy clusters and proton-vacancy clusters. TEM observation shows that the density of small bubbles induced by proton in (Nb, Ta, W)-added steels is much higher than that in Zr-added steels. Both $\frac{1}{3}\langle 1\,1\,1\rangle$ and $\frac{1}{2}\langle 1\,1\,0\rangle$ dislocation loops were observed with TEM in all of the specimens. The mean size and number density of dislocation loops in (Nb, Ta, W)-added steels are slightly larger than that in Zr-added steels, and increased with increasing irradiation dose. Both PAS and TEM observations shows that irradiation damage in Zr-added steels is less serious than that (Nb, Ta, W)-added steels, and the possible mechanisms are discussed through the enhancement of point defect recombination by oversized solute atoms.

1. Introduction

Austenitic stainless steels are considered to be one of candidate structure materials for Gen-IV nuclear reactor due to their high creep resistance and reasonable corrosion/oxidation resistance [1,2]. Studies of corrosion tests imply that materials with high concentration of chromium exhibit significant effect on corrosion tolerance, such as 310S and HR3C which possessing not only prominent tolerance of steam oxidation and high-temperature corrosion because of high chromium content but also better creep strength than martensitic steels [3,4]. However, at the irradiation conditions of reactors, one of severe problems for austenitic stainless steels is irradiation induced swelling. One of the earliest research by Wolfer and .Garner in the early 1980s [5] found that irradiation swelling together with other irradiation defects such as dislocation loops have a strong impact on the physical properties of materials, especially on embrittlement and hardening [6–8]. Previous studies have suggested that addition of component elements is an effective method to improve the irradiation resistance of materials [9–13]. For example, Gan et al. found that the addition of solute atom Hf in 316SS austenitic stainless steels can significantly improve its radiation resistance [13].

In present study, the investigated 310S steels were modified through adding two kinds of solutes: Zr was added (the specimen was named as SC-1) or (Nb, Ta, W) was added (the specimen was named as SC-2). Proton irradiations was employed to compare the effects of two kinds of added solutes on irradiation induced defects. The defects in irradiated specimens were characterized with positron annihilation spectroscopy (PAS) and transmission electron microscope (TEM).

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Table 1
Compositions (wt%) of the investigated modified 310S steels.

Alloy	Fe	Cr	Ni	Mo	Si	Mn	C	V	P	S	Zr	Nb	W	Ta
SC-1	Bal.	24.46	21.25	0.37	0.061	0.06	0.035	0.011	0.005	0.004	0.19	–	–	–
SC-2	Bal.	24.13	21.48	0.65	0.071	0.045	0.032	0.01	0.005	0.004	–	0.18	0.73	0.31

Table 2
Irradiation conditions and the results of bubbles and dislocation loops observed by TEM.

Materials	50 keV H^+ at 563 K		TEM results of bubbles			TEM results of dislocation loops		
	Fluence (ions m^{-2})	Time of Irradiation (s)	Mean size (nm)	Number density (m^{-3})	Number of measured	Mean size (nm)	Number density (m^{-3})	Number of measured
SC-1	4×10^{20}	5302	–	–	–	4.8	1.06×10^{22}	264
SC-2	4×10^{20}	5302	–	–	–	5.7	1.05×10^{22}	288
SC-1	1.2×10^{21}	17,223	1.5	5.37×10^{20}	42	5.6	2.65×10^{22}	270
SC-2	1.2×10^{21}	17,223	1.5	1.34×10^{21}	72	6.4	3.79×10^{22}	284

Note: "–" refers to no bubbles were observed.

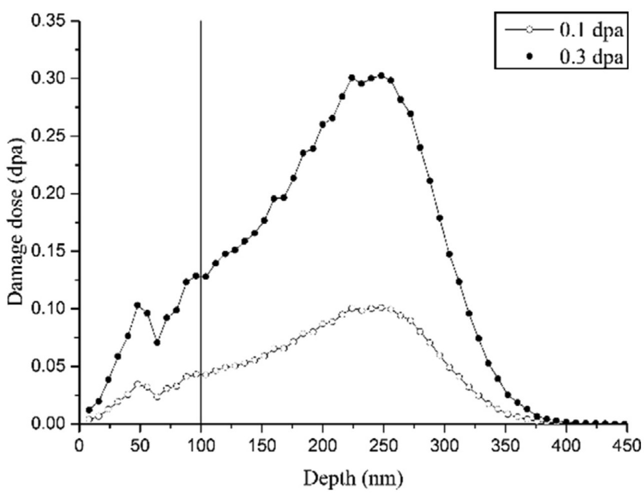

Fig. 1. Depth profile of damage events for 50 keV H^+ irradiations.

2. Experimental

The chemical compositions of the two modified 310S steels used in this study are given in Table 1. The preparation of the materials has been described in the previous literature [14]. The sheets of 10 mm × 10 mm × 0.5 mm were cut from the bulk. The surface of the sheets were mechanically polished with the silicon carbide sandpapers up to the grade of 5000 and then electropolished using a solution of 5% perchloric acid and 95% ethanol electrolyte at 243 K. For prepare TEM samples, the sheet was mechanically polished to the thickness of about 0.1 mm. 3 mm diameter disks were punched and then further milled to final thickness of 40–50 μm. Finally these disks were polished by twin-jet electropolisher using a solution of 10% perchloric acid and 90% acetic acid.

Ion irradiations were performed at a 200 kV ion implanter in the Accelerator Laboratory of Wuhan University. The prepared polished sheets and TEM specimens were simultaneously irradiated with 50 keV proton to the fluence of 4×10^{20} $H^+ \cdot m^{-2}$ and 1.2×10^{21} $H^+ \cdot m^{-2}$. The irradiation conditions are list in Table 2. The irradiations were performed at the temperature of 563 ± 5 K. The damage profiles calculated by SRIM are shown in Fig. 1. The displacement energy is 40 eV [15] and the model used to calculate damage dose was quick calculation of damage [16]. The peak damage dose is 0.1 and 0.3 displacements per atom (dpa) and the dose at 100 nm depth is 0.04 dpa and 0.1 dpa, corresponding to the fluence of 4×10^{20} $H^+ \cdot m^{-2}$ and 1.2×10^{21} $H^+ \cdot m^{-2}$, respectively.

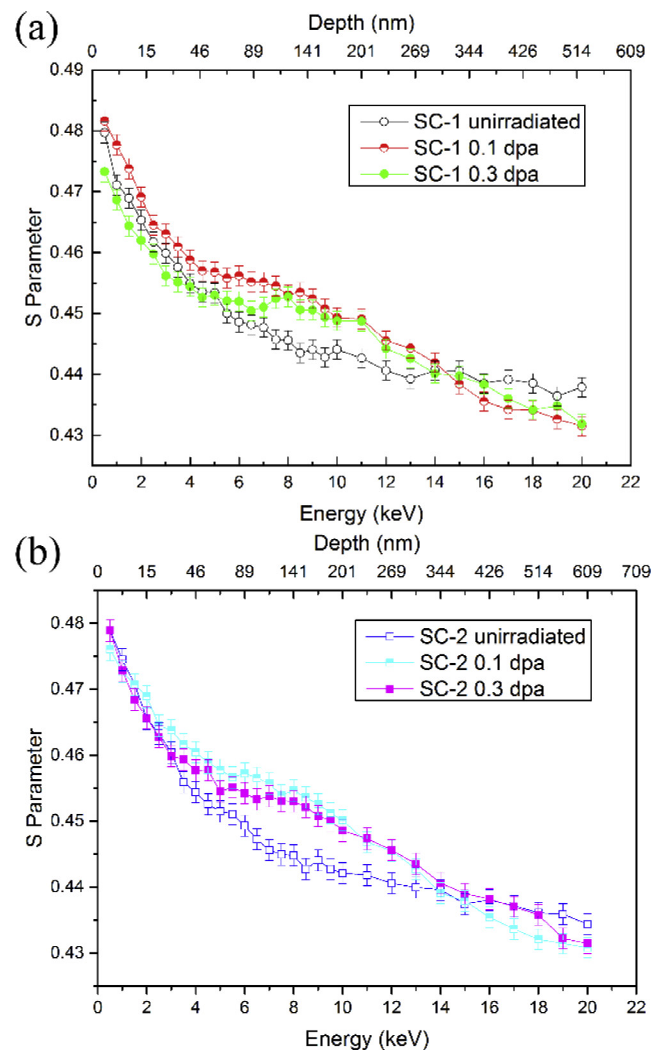

Fig. 2. S-E curves for (a) SC-1 and (b) SC-2 irradiated with different fluence at 563 K.

After irradiation, positron annihilation Doppler Broadening (DB) measurements were performed at slow positron beam facility in Institute High Energy Physics. The generated intensity of the slow positron beam is about 1×10^5 $e^+ \cdot s^{-1}$. The number of slow positrons entering in the specimen is about 5×10^5 e^+. The energy resolution of