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A study on microstructural changes of austenitic stainless steel after proton irradiation

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1. Introduction

Irradiation assisted stress corrosion cracking (IASCC) in reactor internals under high neutron irradiation exposure is considered as one of detrimental degradations against integrity of pressurized water reactor (PWR) for the long term operation. Numerous studies regarding IASCC in reactor internals have been progressed for the understanding of IASCC mechanism and the control of IASCC [1-3]. Particle irradiation method has been used for the IASCC study. Especially, proton irradiation technique has been known to be a strong method for emulating of the neutron irradiation effect under light water nuclear reactor system [4]. IASCC initiation susceptibility of reactor internal material in PWR simulated condition has been investigating using proton irradiation as part of R&D project. The objective of the project is to verify the effect of dissolved hydrogen concentration on IASCC initiation behavior of austenitic stainless steels in PWR water. In this work, we focus on microstructural changes of the proton irradiated austenitic stainless steels with different doses.

2. Experimental

2.1 Materials

Type 316L stainless steel was used for this work. The composition of the experimental steel is given in Table 1. Before the proton irradiation, electro-chemical surface treatment was conducted for damage-free surface.

Table 1 Chemical compositions of the test alloy (wt%)

Material	Cr	Ni	P	Mo	Mn
316 SS	16.7	10.8	0.1	2.0	1.3
	Si	S	C	Fe	
	0.59	0.001	0.047	Bal.	

2.2 Proton irradiation

Proton irradiations were conducted at the Michigan Ion beam Laboratory in the University of Michigan. The General Ionex Tandetron accelerator was used for the proton irradiation experiments. The proton irradiation was to produce four level irradiated samples (1, 3, 5, 10 dpa). The calculated radiation damage (dpa) is shown in Fig. 1. The calculation was performed with full cascade mode in the SRIM code [5]. In the calculation, the displacement energy was set to be 40 eV [6]. According to Fig. 1 of SRIM calculation result, the radiation damage remained constant up to around 15 μm in depth. There was a steep rise at 18 ~ 20 μm in depth. The damage and implantation of hydrogen (proton) drop suddenly after 20 μm in depth.

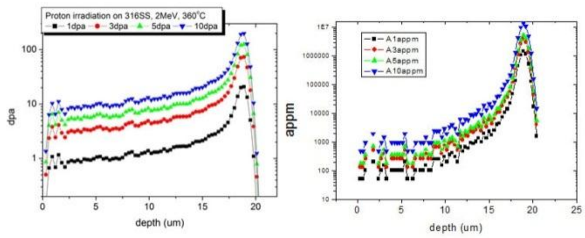

Fig. 1 Radiation damage profiles and implanted H (Proton) by calculation of SRIM code

2.3. The preparation of TEM sample

TEM samples were extracted at a depth of ~ 15 μm from sample surface and at a depth of 15 ~ 20 μm. Focused ion beam milling method was used for the preparation. Additional Ar ion milling with energy of 300V was conducted for the reduction and elimination of surface damage by the previous FIB milling.

TEM analysis was conducted for the analysis of typical microstructural changes such as the formation of radiation induced segregation at grain boundary and the formation of radiation defects including frank loop and voids. Grain boundary composition profiles were measured via scanning transmission electron microscopy (STEM) using the JEM 2100F at Korea Atomic Energy Research Institute, which is equipped with energy dispersive X-ray spectroscopy (EDS).

3. Result and discussion

3.1 Radiation induced segregation at grain boundary

The changes of microchemistry at grain boundary at a depth of about 10 μm were measured by STEM-EDS. Fig. 2 shows chromium and nickel concentration profiles at the grain boundaries. At 1 dpa, the Cr profile showed no depletion behavior at the grain boundary. At 3, 5 and 10 dpa, the Cr depletion and Ni enrichment were clearly observed at the grain boundaries. It was found that the segregation of Ni and depletion of Cr was saturated at the doses.

Fig. 2 also shows the grain boundary chromium and nickel concentration profiles at a grain boundary at a depth of about 16 μm. There was strong radiation induced segregation behavior at the grain boundary. Both the grain boundaries were depleted in Cr to levels of 11-12 wt%. The Ni composition exhibited the largest change among the alloying elements, segregating at both boundaries to levels of 20-22 wt%.

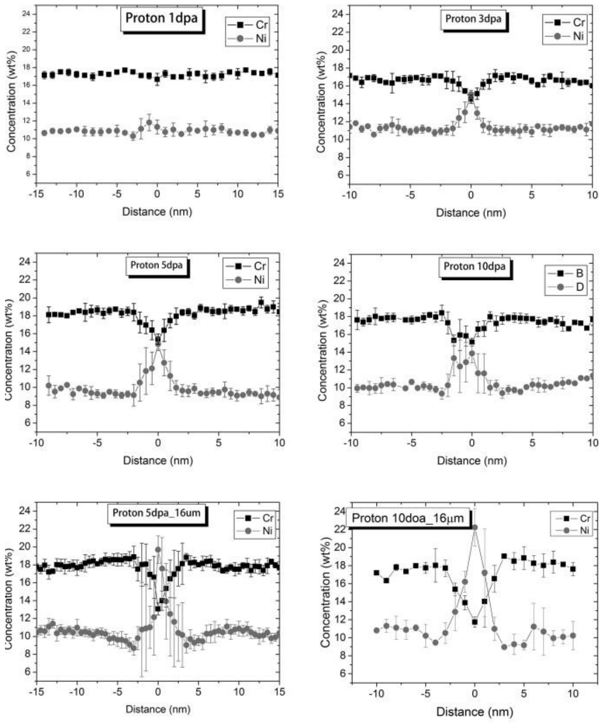

Fig. 2 Cr and Ni concentration profiles at a grain boundary

3.3. The observation of cavity microstructure

The formation of cavity was observed at the depth ranged from 18 μm and to 20 μm as shown in Fig. 3. According to Fig. 1 showing calculated implantation of hydrogen (proton), hydrogen was concentrated at the depth region (18 ~ 20 μm). It was concluded that the cavities are due to proton implantation effect rather than radiation damage effect.

4. Conclusions

Microstructural changes in the proton irradiated 316 stainless steels were investigated.

(1) Dose and implanted hydrogen(proton) were calculated with the irradiation conditions by using SRIM code.
(2) RIS behavior was clearly developed and saturated above 3 dpa in the proton irradiated austenitic stainless steels.
(3) Strong RIS was observed at the depth of ~ 16 μm.
(4) Fine cavities of which size is less 5 nm in diameter were only seen at a depth of ~ 19 μm.

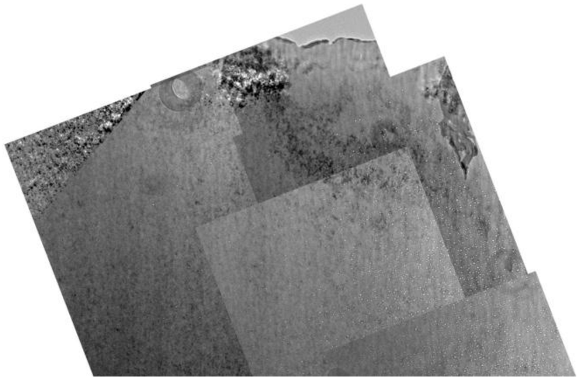

Fig. 3 The formation of cavity in the proton irradiated austenitic stainless steel

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