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TEM Study on the Proton Irradiated Type 316 Stainless Steel

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1. Introduction

Irradiation assisted stress corrosion cracking (IASCC) has been recognized to be a potentially critical phenomenon for core internals in pressurized water reactors over decades. Though the IASCC mechanism is not fully understood because of its complexity, it is widely accepted that IASCC can be affected by the water environment and the irradiation-induced material properties [1]. The compositional change at grain boundary, especially the depletion of Cr, radiation-induced segregation, and/or localized deformation due to irradiation [2,3] can considerably contribute to IASCC. In the present study, microscopic examination on the type 316 stainless steel (SS) using TEM equipment was performed to find out the microstructural changes due to a proton irradiation.

2. Methods and Results

2.1 Proton Irradiation

In the experiment, type 316 SS plates were used in the solution-annealed condition, and their chemical composition is shown in Table 1. The specimens with dimensions of 2 mm x 15 mm x 1.5 mm were ground/polished to a final finish of 0.3 ㎛ alumina powder, and then electropolished in a 10 % perchloric acid + 90 % methanol for about 10 seconds at 50 V at –50 ℃ to get clean surfaces prior to the proton irradiation. 2 MeV protons were irradiated on the front surface of the specimen at 1, 3, 5 displacements per atom (DPA), respectively. The specimen temperature was maintained at 360 ℃ during the irradiation.

Table 1 Chemical composition of the type 316 SS

Fe	Cr	Ni	C	Si	Mn	Mo	P	S
bal.	16.93	10.44	0.57	0.53	1.03	2.07	0.0026	0.001

2.1 Specimen Preparation for Microscopic Study

The specimens for the optical microscope (OM) and scanning electron microscope (SEM) were prepared by a chemical etching in a solution of 2 % bromine + 98 % methanol. The proton irradiation depth is very small, normally 30 ㎛ order under the present irradiation conditions [4]. Therefore, thin foil specimens for the TEM/ATEM were prepared by grinding slabs to an approximately 30 ㎛ thickness from the un-irradiated surfaces. They were then electro-jet polished in a 7 % perchloric acid + 93 % methanol solution at -40 ℃ with a current of approximately 80 mA. Scanning electron microscopy was performed using a JEOL 5200 (operating voltage 25 kV) and a JEOL 6300 (operating voltage 20 kV). Transmission electron microscopy was carried out with a JEOL 2000 FXII (operating voltage 200 kV), and a JEOL JEM-2100F (operating voltage 200 kV).

2.3 Microstructure of the un-irradiated type 316 SS

The specimen used in this study was identified to be an austenitic phase having an fcc structure with a lattice constant of 0.3597 nm. The OM image of the un-irradiated type 316 SS is shown in Fig. 1. The microstructure was homogeneous without any abnormal grain growth and texture. Any intergranular and intragranular precipitations were hardly found. String-like ferrites were often found in the austenitic matrix.

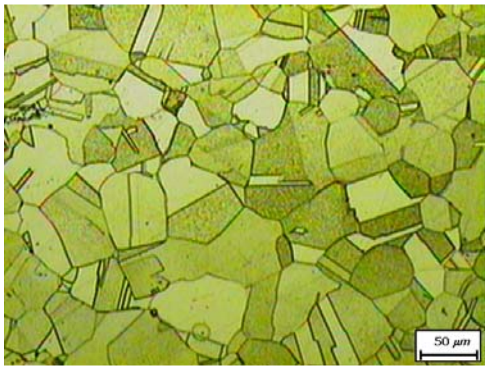

Fig. 1 OM image of the un-irradiated specimen.

A TEM image of the defects taken under a two-beam condition in the un-irradiated specimen is shown in Fig. 2. The number density of dislocation was relatively low, and they were mainly arranged on the {111} slip planes. Stacking fault images were frequently found due to its low stacking fault energy [5]. However, any other defects except dislocations and stacking faults were hardly found in these specimens.

Fig. 2 TEM bright image of the un-irradiated specimen

2.4 TEM results of the irradiated type 316 SS

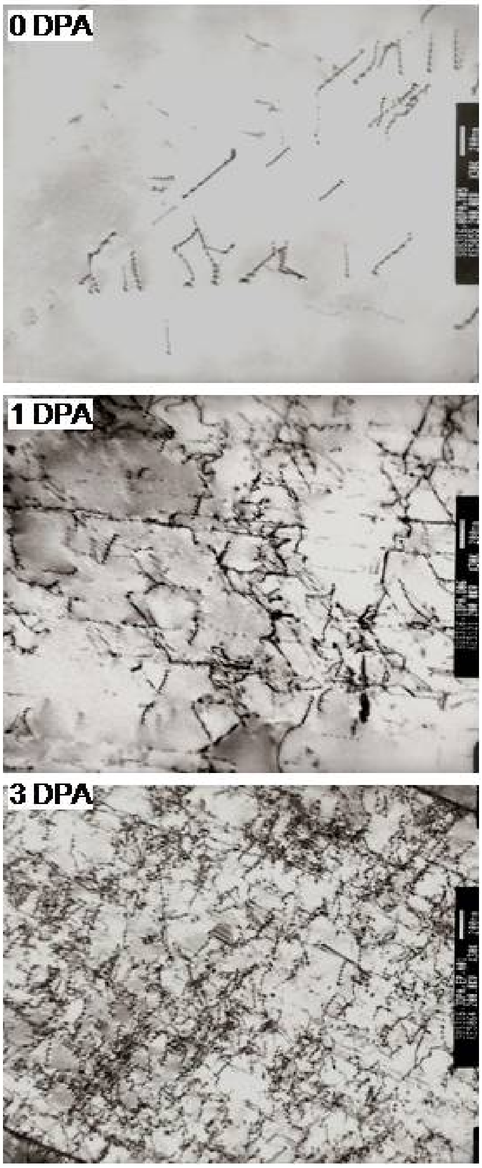

Fig. 2 Variations of the dislocation density depending on the proton irradiation dose.

Fig. 4 shows the proton irradiation effect on the dislocation density in type 316 stainless steel. As the irradiation dose increased, the dislocation density was increased. It was also found that point defects such as vacancies/interstitials and/or small dislocation loops were produced by the proton irradiation. Finally, microtwins evolving from the stacking faults were easily found in the irradiated specimens. From the above results, it is confirmed that the proton irradiation produced many crystal defects such as point defects, dislocations and microtwins in type 316 stainless steels.

3. Conclusions

The microstructure of the un-irradiated type 316 SS was a homogeneous austenitic phase with some string-like delta ferrites, and no other intergranular/ intragranular precipitates were found. The number density of dislocation was relatively small, and stacking fault images were frequently observed due to its low stacking fault energy.

Crystal defects such as dislocations, points defects, and microtwins were easily produced in type 316 stainless steels due to a proton irradiation. The number densities of defects were also increased as the proton irradiation dose increased.

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