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SCC behavior of solid-HIPed and irradiated type 316LN-IG stainless steel in oxygenated or hydrogenated water at 423–603 K

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Abstract

Effects of water temperature on the stress corrosion cracking (SCC) susceptibility were studied on a solid hot-isostatic pressed (solid-HIPed) type 316LN-ITER grade (316LN-IG) stainless steel in unirradiated or irradiated condition. Neutron irradiation was conducted at a nominal temperature of 473 K to a dose level of 1 dpa. Slow strain rate testing (SSRT) was performed over a temperature range from 423 to 603 K in oxygenated (DO = 10 ppm) or hydrogenated (DH = 1 ppm) water. In the unirradiated condition, intergranular (IG) type SCC occurred at 603 K in the oxygenated and in the hydrogenated water. In the irradiated condition, IASCC occurred above 573 K in oxygenated water. In the hydrogenated water, IASCC occurred at 513 K, but was suppressed at 573 K.

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1. Introduction

Type 316LN stainless steel (SS) is one of the structural materials of a vacuum vessel and in-vessel components for the International Thermonuclear Experimental Reactor (ITER). The vacuum vessel and in-vessel components such as a shield blanket are irradiated by high energy neutrons and cooled by high temperature water (423–463 K at a normal operation condition and 513 K at a baking condition). In such service conditions, austenitic stainless steels become susceptible to irradiation assisted stress corrosion cracking (IASCC). Stress corrosion cracking (SCC) of unirradiated materials as well as

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IASCC is affected by environmental conditions such as water chemistry and temperature [1-17].

Authors reported in previous works [16,17] that with SSs irradiated below 573 K, IASCC did not occur during a slow strain rate testing (SSRT) below 513 K in oxygenated water. In hydrogenated water from 561 to 593 K, IASCC was suppressed for austenitic SSs irradiated below $\sim 4 \text{ dpa}$ [12,13]. Hydrogen water chemistry (HWC) became, however, insufficient to suppress IASCC susceptibility in specimens irradiated to the higher dose level [14,15]. These IASCC studies have been performed extensively at the operating temperature range of light water reactors. There are few studies on IASCC behavior at temperature range of ITER. The purpose of this study is to examine the effects of water chemistry on stress corrosion cracking (SCC) in the ITER temperature range.

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2. Experimental

The chemical composition of the type 316LN ITER grade (316LN-IG) SS examined in this study is listed in Table 1. Because the in-vessel components, such as a shield blanket, would be fabricated by a solid hot-isostatic pressing (solid-HIPing) technique [18,19], specimens were prepared from the solid-HIPed joints of the type 316LN-IG SS plates. The solid-HIPing condition is described elsewhere [20,21]. Transmission electron microscopy (TEM) and electrochemical potentiokinetic reactivation (EPR) tests revealed that the solid-HIPing process did not cause any thermal sensitization of the materials [21,22]. Sheet-type miniature tensile specimens (Fig. 1) were irradiated at a nominal temperature of 473 K to a dose level of 1 dpa in the high flux isotope reactor (HFIR) [16]. Specimens were mechanically polished by 2400-grit SiC paper and electropolished prior to irradiation.

In order to study the effects of water chemistry, slow strain rate testing (SSRT) was conducted in recirculating water environments containing either 10 ppm dissolved oxygen (DO) or 1 ppm dissolved hydrogen (DH). The DO and DH were controlled by the bubbling of the recirculating water with pure oxygen or hydrogen gas and measured on-line continuously using Toa model DO-32 A DO analyzers and a Toa model DHDI-1 DH analyzer. Water temperature was varied from 423 to 603 K. Other water conditions are listed in Table 2. Strain rates were $1.0 \times 10^{-6} \text{ s}^{-1}$ at 423 K and $2.0-3.0 \times 10^{-7} \text{ s}^{-1}$ at 513-603 K. After SSRT, the fracture surface was examined using a scanning electron microscope (SEM). SCC susceptibility was assessed by calculating the ratio of intergranular (IG) type SCC area (%IGSCC) and transgranular (TG) type SCC area (%TGSCC) to total fractured area.

Single edge notched (SEN) specimens were also used to ease crack initiation [23,24]. The notch in the uniform test section of the sheet-type specimens was machined after irradiation using a low speed diamond wheel saw. The radius of notch tip was 0.18–0.2 mm and the depth of notch was 0.25– 0.45 mm. In these SEN specimens, the nominal stress was calculated by dividing the load by the



Fig. 1. A sheet-type miniature tensile specimen (unit: mm).

Table 2 Water conditions for SSRT on irradiated specimens

Temperature (K)	Pressure (MPa)	Condu (µS/cn	Conductivity (µS/cm)		n) (ppm)		DH (ppm)
		Inlet	Outlet				
423	4	0.056	< 0.09	10	0		
513	6	0.056	< 0.3	10	0		
513	6	0.060	< 0.15	< 0.02	1		
573	9	0.056	< 0.5	10	0		
573	9	0.062	< 0.3	< 0.01	1		
603	15	0.060	<0.5	10	0		

cross-section at the notch root and the nominal strain was calculated by dividing the deflection of a pull rod by the length of the uniform test section.

3. Results and discussion

3.1. Unirradiated specimens

Effects of water temperature on SCC susceptibility was examined by SSRT in oxygenated water (DO = 10 ppm). SEM observation of the fracture surfaces indicated that specimens failed in a fully ductile mode at 423 and 513 K, while TGSCC was the dominant mode above 553 K. A small area of IGSCC was observed on the specimen tested at 603 K. Fig. 2(a)–(d) show the SEM photographs and illustrations of SCC area on the fracture surfaces of specimens tested at 573 and 603 K. A larger area of TGSCC was observed at the position where local necking had developed.

In order to examine the effects of water chemistry on SCC susceptibility at 603 K, a specimen was

Table 1 Chemical composition of type 316LN-IG stainless steel (wt%)

Chemical composition of type 510LN-1G stamess steel (wt%)											
С	Si	Mn	Р	S	Cr	Ni	Мо	Ν	В		
0.029	0.44	1.44	0.012	0.009	17.48	12.11	2.56	0.067	0.0003		



Fig. 2. Fracture surfaces of unirradiated specimens after SSRT. In (e), area of IGSCC was enlarged in picture.

tested in hydrogenated water (DH = 1 ppm). As seen in Fig. 2(e) and (f), IGSCC occurred even in the hydrogenated water. TGSCC was the primary failure mode with small areas of IGSCC observed around the edge of fracture surface.

Fig. 3 summarizes effects of water temperature on SCC susceptibility of unirradiated, solid-HIPed specimens. IGSCC occurred at 603 K on both specimens during SSRT in oxygenated and hydrogenated water. Although thermally sensitized stainless steels showed IGSCC susceptibility during SSRT in the a temperature range 353–593 K in oxygenated water [1–6], there are few reports on the IGSCC initiation on solution-annealed stainless steels during SSRT in oxygenated water.

In hydrogenated water, on the other hand, occurrence of IGSCC on solution-annealed stainless steels during SSRT was observed by some researchers [7–10]. At a temperature range from 593 to 633 K, the IGSCC susceptibility of solutionannealed stainless steels increased with increasing water temperature during SSRT in the simulated PWR primary water containing ~2820 ppm H₃BO₃, ~7 ppm LiOH and ~1–4 ppm DH [10]. In this study, IGSCC occurred during SSRT at 603 K in high purity (deionized) water containing



Fig. 3. Effects of water temperature on SCC susceptibility of unirradiated specimens.

only about 1 ppm DH (Fig. 2(e)). The DH might cause the IGSCC in hydrogenated water.

3.2. Irradiated specimens

In previous papers, we reported that the solid-HIPed specimens did not show IGSCC susceptibility below 513 K in oxygenated water and did at 573 K [16]. In order to study the water temperature dependence of IASCC susceptibility in oxygenated water, a specimen was tested at 603 K. %IGSCC and %TGSCC was 16.6% and 60.1%, respectively. The %IGSCC of the specimen tested at 603 K was larger than that at 573 K. Above 573 K, IGSCC susceptibility of irradiated specimens seemed to increase with increasing water temperature.

Since IASCC did not occur at 513 K in a smooth bar specimen [16], the SEN specimen was tested at 513 K in oxygenated water. As seen in Fig. 4(a), the SEN specimen failed in a completely ductile mode after SSRT. Therefore, the occurrence of IASCC was difficult at 513 K in oxygenated water.

In hydrogenated water at 573 K, IASCC was not observed (Fig. 4(d)). At 513 K, TGSCC occurred at the notch root of the SEN specimen (Fig. 4(c)). Many small cracks were formed on the sides of the SEN specimen.

Fig. 5 summarizes the effects of DO, DH and water temperature on %SCC (=%IGSCC + %TGSCC) of irradiated specimens. In oxygenated water, %SCC seemed to increase with increasing water temperature. In hydrogenated water, on the other hand, %SCC seemed to decrease with increas-



Fig. 5. Effects of water chemistry on IASCC susceptibility of a solid-HIPed 316LN-IG stainless steel irradiated at 473 K to 1 dpa. Number of parenthesis is %IGSCC.

ing water temperature. It is well known that for stainless steels irradiated below a dose level of about 4 dpa, hydrogen water chemistry mitigates IASCC in the temperature range from 561 to 593 K [12,13]. However, TGSCC was observed at 513 K in hydrogenated water (Fig. 4(c)). Hydrogen embrittlement or H-induced cracking usually occurs below 373 K [25–29]. The temperature range to initiate hydrogen embrittlement might be raised by irradiation. In this study, a limited number of tests were conducted, so that further study is necessary to elucidate the IASCC mechanism in the HWC condition.



Fig. 4. Fracture surfaces of irradiated specimens after SSRT at 513 and 573 K in oxygenated (DO = 10 ppm) and hydrogenated (DH = 1 ppm) water.

4. Conclusions

SCC susceptibility of a solid-HIPed type 316LN-IG stainless steel in unirradiated or irradiated condition was studied by SSRT in the temperature range from 423 to 603 K in oxygenated (DO = 10 ppm) or hydrogenated (DH = 1 ppm) water. Neutron irradiation was conducted at 473 K to dose level of 1 dpa.

- Unirradiated type 316LN-IG showed IGSCC susceptibility at 603 K in both oxygenated and hydrogenated water.
- (2) Irradiated type 316LN-IG showed IASCC susceptibility above 573 K in oxygenated water. The susceptibility increased with increasing water temperature. At 513 K, IASCC did not occur on smooth bar specimen or notched specimen. In hydrogenated water, IASCC was suppressed at 573 K. However, TGSCC occurred at 513 K.

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