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Irradiation-assisted SCC susceptibility of HIPed 316LN-IG stainless steel irradiated at 473 K to 1 dpa

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Abstract

Solid hot-isostatic pressed (solid-HIPed) joint specimens and those with or without thermal-cycled specimens of 316LN-IG were irradiated at about 473 K to 1 dpa in the high flux isotope reactor. Slow strain rate tests were conducted in a high-purity, oxygenated (dissolved oxygen = 10 wt ppm) water at 423, 513 and 573 K with strain rates of $(2\text{–}10) \times 10^{-7}\ s^{-1}$. Tensile tests were also conducted in vacuum at the same temperature range. Fracture surfaces were observed by scanning electron microscopy. No specimen showed irradiation-assisted stress corrosion cracking (IASCC) susceptibility at 423 and 513 K in water. At 573 K, however, intergranular cracks were observed to form in HIPed specimens. It was concluded that the effect of HIPing to IASCC susceptibility is small.

1. Introduction

In the engineering design activity (EDA) of the International Thermonuclear Experimental Reactor, ITER-grade 316LN-IG stainless steel (SS) is proposed for the structural material of the first wall/blanket modules [1]. The solid hot-isostatic pressing (solid-HIPing) is proposed to join the plates to form the modules in which channels for water cooling are located [2,3]. The austenitic SS often fail by stress corrosion cracking (SCC) even in high-purity water after neutron irradiation at elevated temperatures [4]. Tsukada et al. had reported the susceptibility of type 316 SS for the irradiation-assisted stress corrosion cracking (IASCC) after irradiation in the spectral tailoring capsule [5]. Type 316 SS irradiated at temperatures of 333 and 473 K did not show the IASCC susceptibility in water at 333 and 473 K. However, grain boundary separation was observed to form in the specimens irradiated at 603 and 673 K during slow strain rate test (SSRT) at 573 K. Based on these data and knowledge of IASCC, the temperature of cooling water was decided to be 423 K in the ITER EDA [6]. However there are a few data on the effect of HIPing process on the post-irradiation tensile and IASCC behavior of type 316LN SS. This behavior was studied in this work.

2. Experimental

The chemical composition of 316LN-IG SS is listed in Table 1. Two plates (40 mm thick × 300 mm wide × 400 mm long) were HIPed at 1323 K for 2 h at 150 MPa in an Ar environment. The details of solid-HIPing procedure are described elsewhere [7]. Sheet type tensile specimens with 7.86 mm in gage length, 1.54 mm in width and 0.76 mm in thickness were prepared from HIPed interface and a position of 1/4 thickness of the HIPed plate. Hereafter, the former is called HIPed specimen, and the later is called HIP-thermal-cycled specimen. As-received specimens were also prepared from a position of 1/2 thickness of solution-annealed plate.

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

C	Si	Mn	P	S	Ni	Cr	Mo	N	B
0.029	0.44	1.44	0.012	0.009	12.11	17.48	2.56	0.067	3 ppm

Table 2
SSRT conditions

Temperature (K)	Pressure (MPa)	Conductivity (μS/cm)		Dissolved oxygen (wt ppm)	Strain rate (s^{-1})
		Inlet	Outlet		
423	4	0.056	<0.09	10	10×10^{-7}
513	6	0.056	<0.3	10	2×10^{-7}
573	9	0.056	<0.5	10	2×10^{-7}

These specimens were irradiated at nominal temperature of 473 K on the peripheral target position of the high flux isotope reactor (HFIR) to dose level of about 1 dpa. After irradiation, SSRT was conducted in a high-purity, oxygenated (dissolved oxygen = 10 wt ppm) water at temperatures of 423, 513 and 573 K with strain rates of (2–10) $\times 10^{-7}$ s^{-1}. Other test conditions were listed in Table 2. Tensile tests were also conducted in vacuum at the same temperatures with a strain rate of 1×10^{-4} s^{-1}. Fracture surfaces were observed by scanning electron microscopy (SEM). Metallographic examination was carried out on the gage section of tested specimens after chemical etching by aqua regia in order to observe cracking and its morphology at the surface.

3. Results

3.1. Tensile behavior

Fig. 1 shows the temperature dependence of tensile properties of the irradiated specimens. Trend curves of the properties of unirradiated specimens were also indicated in Fig. 1. Irradiation induced the increase of 0.2% offset stress, $\sigma_{0.2}$, and ultimate tensile stress, UTS, and the decrease of uniform strain, ε_u, and total strain, ε_t. The ε_u after the irradiation was larger than 10%. Serration was observed in the stress–strain curves of specimens tested at 573 K. SEM observations showed that all specimens failed in a ductile manner. No fracture was observed at the HIPed interface. There is no distinct difference on the tensile behavior among HIPed, HIP-thermal-cycled and as-received specimens.

3.2. SSRT behavior

Fig. 2(a)–(c) show the stress–strain curves of irradiated specimens after SSRT. The curves after tensile tests were also indicated in the figures. As seen in Fig. 2(a) and (b), stress–strain curves of all specimens after SSRT in 423 and 513 K water were similar to those from tensile tests. In 573 K water, however, the curves after SSRT were different from those after the tensile tests. The $\sigma_{0.2}$ and work hardening behavior after SSRT were equivalent to those after the tensile tests, but ε_t after SSRT is smaller than that after tensile test. HIPed specimen failed nearly at the same strain where as-received specimens failed.

It was revealed by SEM observations that all specimens after SSRT in 423 and 513 K water failed in a fully ductile manner. Fig. 3(a) and (b) show the fracture surfaces of HIPed and as-received specimens after SSRT in 573 K water. On the surfaces of HIPed specimens, intergranular (IG) and transgranular (TG) cracking

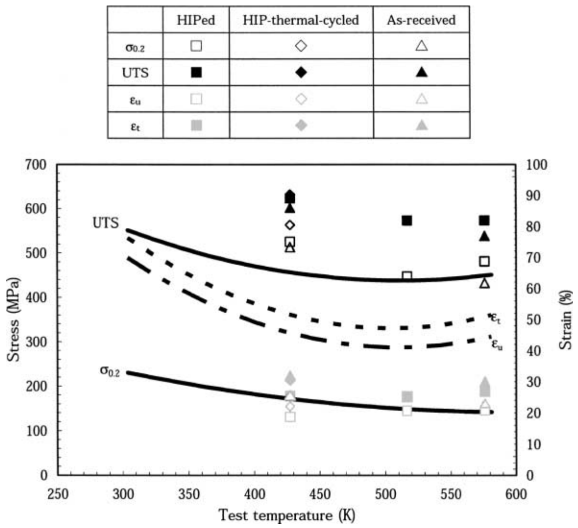


Fig. 1. Test temperature dependence of tensile properties in 316LN-IG irradiated at 473 K to 1.2 dpa. Lines indicate trend curves of tensile properties of unirradiated 316LN-IG.

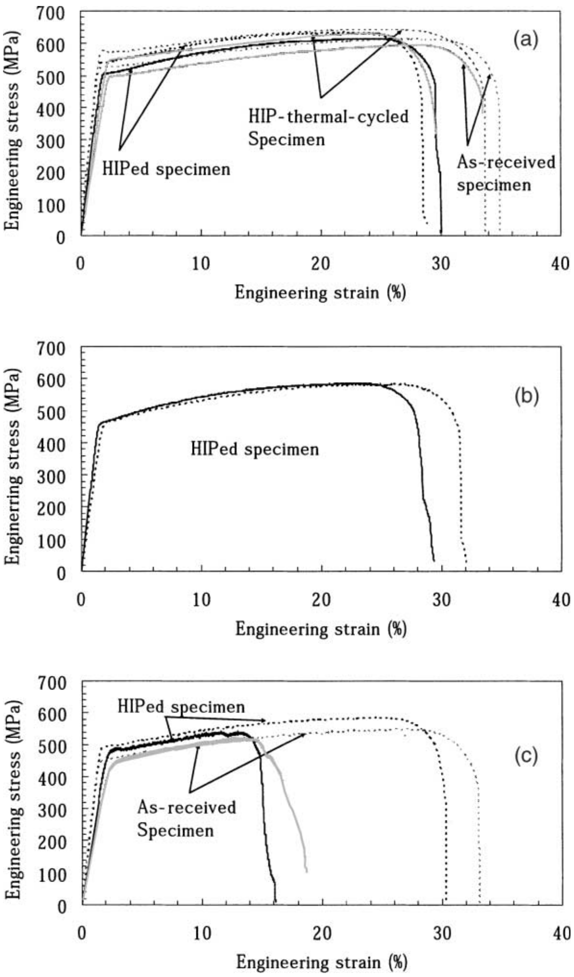


Fig. 2. Engineering stress–strain curves of irradiated specimens after SSRT in a high-purity, oxygenated (dissolved oxygen = 10 wt ppm) water (solid line). Broken lines indicate the curves after tensile tests in vacuum. (a) 423 K. (b) 513 K. (c) 573 K.

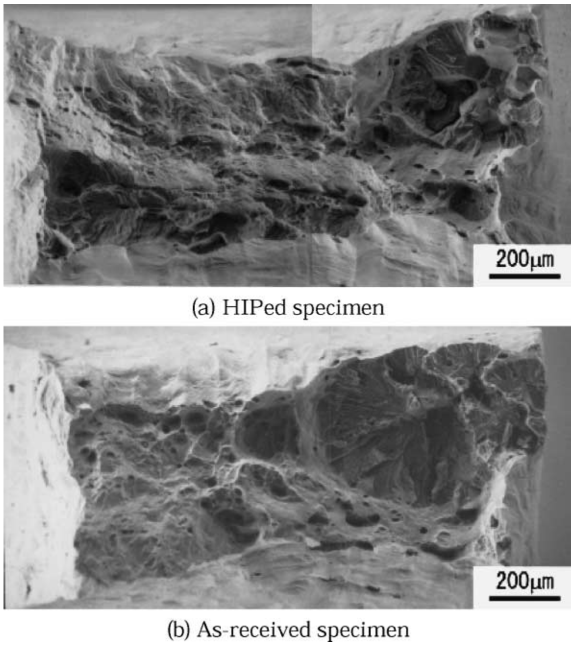


Fig. 3. Fracture surfaces of HIPed and as-received specimens after tensile test in vacuum and SSRT in oxygenated water (dissolved oxygen = 10 wt ppm) at 573 K.

were observed. On the fracture surface of as-received specimens, only TG cracking was observed. The percentages of IGSCC and TGSCC on fracture surface of HIPed specimens were 8% and 22%, respectively. The percentage of TGSCC of as-received specimens was 36%.

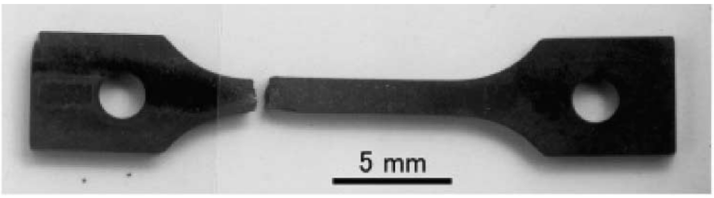
On the HIPed specimen after SSRT in 573 K water, fracture occurred away from the HIPed interface (Fig. 4(a)). At the HIPed interface, no crack was observed (Fig. 4(b)). As seen in Fig. 4(c), a small crack initiated transgranularly, and then propagated along grain boundaries or transgranularly. Fig. 5 shows a small crack observed on the as-received specimen after SSRT in 573 K water. Not only TG cracking but also IG cracking were observed in the small crack, though only TG cracking was observed on fracture surface.

4. Discussion

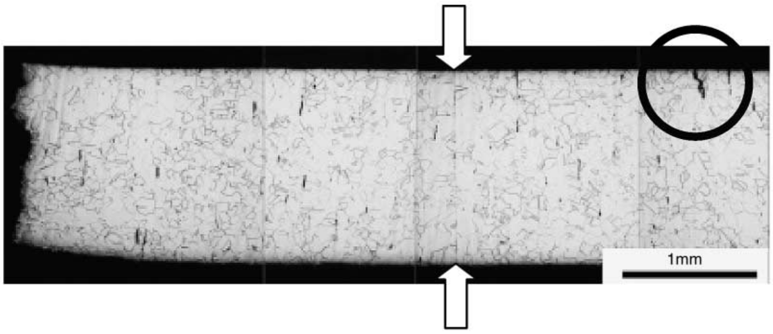
Nakano et al. [7] reported that there was no difference on the tensile behavior of unirradiated specimens of present alloys, although a higher density of inclusions was observed at the HIPed interface. Even after irradiation, there are no distinct differences on tensile properties among the alloys. No fracture occurred at HIPed interfaces. It is concluded that these inclusions at the HIPed interface do not affect the tensile behavior even after the neutron irradiation.

Both HIPed and as-received specimens showed the same temperature dependence on IASCC susceptibility. HIPed specimens ruptured in a fully ductile manner at 423 and 513 K, but failed in a brittle manner of IG and TG SCC at 573 K. As-received specimens also ruptured in a ductile manner at 423 K, but failed in a brittle manner of IG and TG SCC at 573 K. The percentage of SCC on fracture surface of HIPed specimen was equivalent to that of as-received specimen. At 573 K, the strain to maximum stress of HIPed specimens was the same as that of as-received specimens. No SCC was observed at the HIPed interface. These results indicate that solid-HIP procedure has no influence on IASCC behavior of 316LN-IG SS irradiated at 473 K to about 1 dpa.

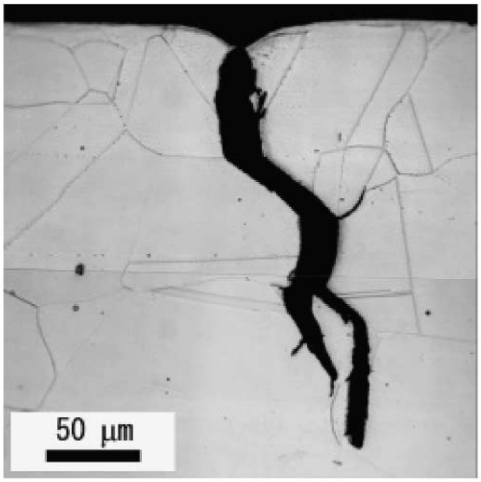
Jenssen et al. [8] reported that type 316L-NG SS showed the IASCC susceptibility after irradiation to about 1 dpa at 561 K and SSRT in 548 K water. Chung et al. [9] reported that high-purity type 316L SS showed



(a) Fractured specimen.



(b) HIPed interface. Arrow indicate HIPed interface.



(c) Magnification of crack observed in upper right side of (b).

Fig. 4. Optical photographs at HIPed interface and cracking in HIPed specimen after SSRT in 573 K water.

the IASCC susceptibility after irradiation to about 1 dpa at 562 K and SSRT in 562 K water. The dose levels reported by these authors are comparable to that in this work, although the irradiation temperature in this work was lower than reported conditions. It is considered that the dose level of about 1 dpa at 473 K is enough for type 316L SS to induce the IASCC in 573 K water.

The influence of heat treatment during the HIPing process on IASCC susceptibility was considered especially a possibility of thermal sensitization during furnace cooling. Okada et al. [10] reported that Cr depletion and Ni enrichment by radiation-induced segregation at grain boundaries were accelerated on type 304 SS that was thermally sensitized before irradiation. Nakano et al. [7] reported that $M_{23}C_6$ type precipitates were observed at the HIPed interface of the present alloys. These precipitates could give rise to IASCC at the HIPed interface. However, no SCC was observed at the interface. It was concluded that the influence of heat

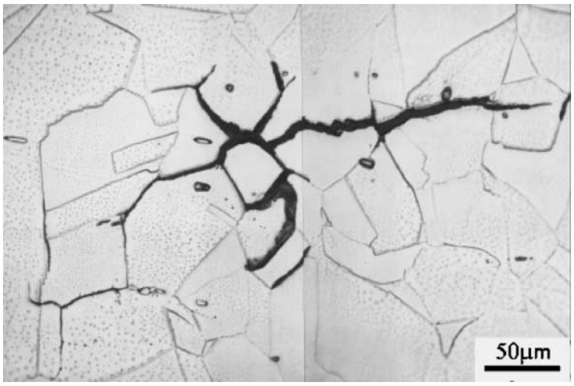


Fig. 5. Surface cracking on gage section in as-received specimen after SSRT in 573 K water.

treatment during the HIP process on IASCC susceptibility was negligible on the 316LN-IG SS irradiated to about 1 dpa at 473 K.

The results of this work indicate that 316LN-IG SS and its HIPed alloy have no IASCC susceptibility at 423 K under ITER operating conditions, and 513 K of baking conditions of the ITER vacuum vessel, up to a dose level of about 1 dpa. Further studies are necessary for an extraordinary operation above 573 K, such as the case of plasma disruption, to minimize a possibility of IASCC failure.

5. Summary

Solid-HIPed joint specimens and those with or without thermal-cycled specimens of 316LN-IG were irradiated to 1 dpa at about 473 K in the HFIR. The IASCC susceptibility was evaluated with SSRT in high-temperature water and tensile tests in vacuum. The following results were obtained:

(1) There is no difference of tensile properties among solid-HIPed joint specimens and those with or without thermal-cycled specimens of 316LN-IG at 423, 513 and 573 K. No fracture was observed to occur at the interface of HIPed specimens.
(2) Solid-HIPed joint specimens tested by SSRT at 423 and 513 K ruptured in a ductile manner. However, IASCC occurred at 573 K. 316LN-IG also failed in a ductile manner at 423 K, but by brittle manner of IASCC at 573 K.
(3) Solid-HIPing procedure had no obvious influence on tensile properties and IASCC susceptibility.

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