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Journal of Nuclear Materials 307–311 (2002) 1568–1572

journal of nuclear materials

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Characterization of 316L(N)-IG SS joint produced by hot isostatic pressing technique

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Abstract

Type 316L(N) stainless steel of the international thermonuclear experimental reactor grade (316L(N)-IG SS) is being considered for the first wall/blanket module. Hot isostatic pressing (HIP) technique is expected for the fabrication of the module. To evaluate the integrity and susceptibility to stress corrosion cracking (SCC) of HIPed 316L(N)-IG SS, tensile tests in vacuum and slow strain rate tests in high temperature water were performed. Specimen with the HIPed joint had similar tensile properties to specimens of 316L(N)-IG SS, and did not show susceptibility to SCC in oxygenated water at 423 K. Thermally sensitized specimen was low susceptible to SCC even in the creviced condition. It is concluded that the tensile properties of HIPed SS are as high as those of the base alloy and the HIP process caused no deleterious effects.

1. Introduction

In the engineering design activity of international thermonuclear experimental reactor (ITER), a module type structure has been proposed for the first wall/shielding blanket component. As a candidate material, type 316L(N) stainless steel ITER grade (316L(N)-IG SS) is being considered. For a fabrication of the module, the hot isostatic pressing (HIP) technique is expected from the view points of high joint strength and dimensional precision, and integrity of the joints. However, deterioration of integrity and susceptibility to stress corrosion cracking (SCC) of the joints during heat treatment in the HIP process has been concerned. Since type 316L SS has low carbon concentration, its susceptibility to intergranular (IG) SCC in high temperature water is expected to be very low. However, for example, Lorenzetto et al. reported a detrimental effect of impurities in water and that 316L(N)-IG SS specimens had failed by SCC at 613 K [1]. Colgeton et al. showed potential ranges for SCC of annealed 316 SS in 5 ppm chloride content water at temperatures from 373 to 673 K [2]. An objective of this study is, therefore, to assess the integrity and susceptibility to SCC of HIPed stainless steel joints through tensile tests in vacuum and slow strain rate tests (SSRT) in high temperature water. In addition, SSRT with a crevice condition were performed to examine effects of crevice on the SCC, because an initiation of SCC would be accelerated by introducing an artificial crevice to the specimen [3].

2. Experimental

2.1. Material and HIP process

A chemical composition of 316L(N)-IG SS used in this study is shown in Table 1. Two plates with 400 mm in length, 300 mm in width and 40 mm in thickness were solution annealed and quenched in water. After polishing of the surface of two plates with abrasive (0.7 μm in diameter), the plate were put together. Then the plates were canned into type 304 SS thin plates and heated for 1 h at 773 K in vacuumed ($<1.33 \times 10^{-4}$ Pa). Then, these plates were pressed isostatically for 2 h in argon

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0022-3115/02/$ - see front matter
PII: S0022-3115(02)00965-0

Table 1
Chemical composition of 316L(N)-IG SS (wt%)

C	Si	Mn	P	S	Ni	Cr	Mo	N	Co	Cu	B	Nb	Ta	Ti
0.029	0.44	1.44	0.012	0.009	12.11	17.48	2.56	0.067	0.02	0.09	3 ppm	<0.01	<0.005	<0.01

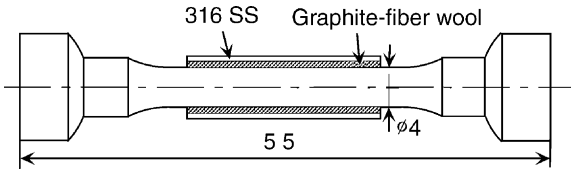

Fig. 1. Configuration of graphite-fiber-covered SSRT specimen (crevice).

gas at 1323 K under pressure of 150 MPa. The HIPed interface was examined by the optical microscope, transmission electron microscope (TEM) and scanning electron microscope (SEM).

2.2. Tensile tests and SSRT

Sheet type specimens with 7.62 mm in gage length, 1.52 mm in width and 0.76 mm in thickness, and round bar type specimens with 24 mm in gage length and 4 mm in diameter were used for tensile tests and SSRT. Specimens with the HIPed joint and without the HIPed joint were machined from the HIPed plates, and specimens without heating history of the HIP process were machined from an as-received plate. These specimens are designated as the HIPed joint, HIP thermal and as-received specimens, respectively. Tensile tests were performed at room temperature (RT), 423, 473, 523 and 573 K in vacuum ($<1.3 \times 10^{-2}$ Pa) with a strain rate of 1×10^{-4} s^{-1}.

SSRT and creviced SSRT were performed at 423 and 513 K in oxygenated high temperature water. The applied strain rates were 1×10^{-6} and $2 - 10 \times 10^{-7}$ s^{-1}, respectively. Schematic configuration of the creviced SSRT specimen is shown in Fig. 1. To make a crevice condition, the specimen was wound by graphite-fiber wool and covered with 316 SS. After the tests, specimens were examined by optical microscope, SEM and TEM.

3. Results and discussion

3.1. Microstructure of HIP interface

As shown in the optical micrograph of the HIPed joint (Fig. 2), no defective portion of the HIPed interface and anomalous grain growth by the HIP process were observed. Fig. 3 shows a TEM micrograph of the HIPed interface. Dislocation density near the HIPed joint was

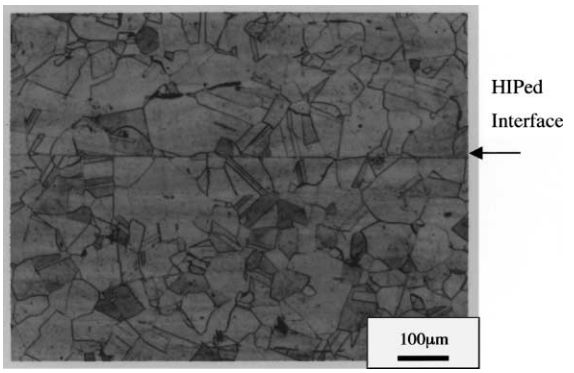

Fig. 2. Optical micrograph of the HIPed joint.

as low as that in the matrix of the alloy. Any voids such as reported for the interface of HIPed Cu/SS316L joint materials [4] were not observed in this study. Small precipitates were, however, observed at the HIPed interface. From the results of analyses of the energy dispersive X-ray spectrum and X-ray diffraction pattern obtained by TEM, it is clarified that these precipitates are $M_{23}C_6$. The other types of small particles were observed not only at the HIPed interface but also in the grain. TEM analyses revealed that these particles are aluminum oxide and silicon oxide. A density of the oxides at the HIPed interface was comparable with those in a grain and at the grain boundary.

3.2. Tensile properties and susceptibility to SCC

In Fig. 4, the ultimate tensile stresses (UTS) from the present study and from the literature [5] are compared. As seen in this figure, UTS obtained in this study are within a scatter band of the data. For all of tensile test conditions in this study, fractured surfaces showed only ductile fracture. For the HIPed joint specimens, no large cavity at the HIPed interface and no fracture initiated from the HIPed interface were observed. Therefore, it is concluded that the HIPed interface does not form a region where the fracture easily initiates, and that tensile properties of the HIPed joint specimens are as high as those of the base alloy.

SSRT of the HIPed joint specimen performed in 423 K water (DO = 32 wt ppm) with a strain rate of 1×10^{-6} s^{-1} shows that a stress–strain behavior during SSRT was similar to that from tensile tests, because no SCC occurred by the SSRT. Only a dimple pattern was

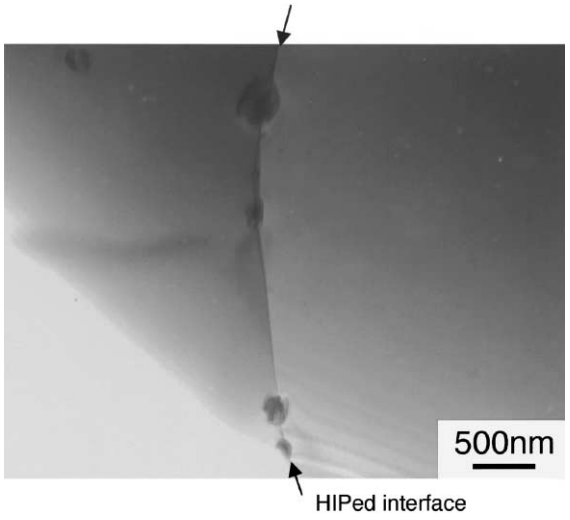

Fig. 3. TEM photograph of precipitates on the HIPed interface.

observed on the fracture surface after SSRT and the fracture did not occur at the HIPed interface. Therefore it can be concluded that 316L(N)-IG SS has no SCC susceptibility in oxygenated 423 K water even after HIP process.

3.3. SCC susceptibility under creviced condition

For the HIPed joint, HIP thermal and as-received specimens, SSRT was performed in 513 K water under creviced condition. Creviced condition was obtained by introducing artificial crevice with graphite-fiber wool [3]. All specimens were thermally sensitized at 1033 K for 100 h in vacuum ($<4 \times 10^{-4}$ Pa) in order to increase the IGSCC susceptibility especially at the HIPed interface. The stress–strain behavior of these specimens after SSRT was similar for all specimens. As described in the above section, tensile tests of the thermally sensitized specimen were carried out. However, elongation data between SSRT and tensile tests cannot be compared directly because of the difference of specimen shape and size. For all specimens, the maximum stresses after SSRT were equivalent to the UTS from tensile tests. Therefore, it seems that these specimens were elongated near to the uniform elongation during SSRT. Fig. 5(a) shows the fracture surface of thermally sensitized, HIPed joint specimen. Transgranular (TG) SCC and ductile fracture were observed at the edge and in the center region, respectively (Fig. 5(b)). For all specimens, the area ratio of TGSCC indicated approximately 10–30%. As seen in Fig. 5(c), a lot of TG cracks were observed on a region covered by graphite-fiber wool and it suggested that the initiation of SCC was accelerated under creviced condition. Fig. 6 indicates that fracture did not occur at the HIP interface, and even small TG cracking did not initiate at the interface.

Although it is well known that IGSCC occurs easily for the thermally sensitized stainless steel in oxygenated water, no IGSCC was observed after the creviced SSRT in 513 K water. In addition it is noted that IG cracking was not initiated at the tip of TG cracks, i.e. natural crevice. Since no IGSCC was observed in all specimens, we concluded that initiation of IGSCC under creviced condition was difficult for 316L(N)-IG SS even after HIP process.

On the other hand, it is known that TGSCC is promoted by the existence of impurities such as Cl^- in water [6]. The concentration of Cl^- in feeding water of this

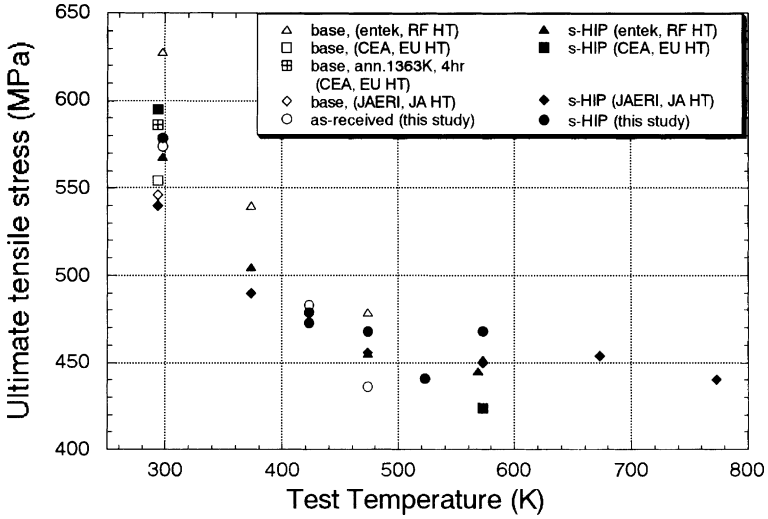

Fig. 4. Temperature dependence of UTS of the HIPed joints and as-received 316L(N)-IG SSs.

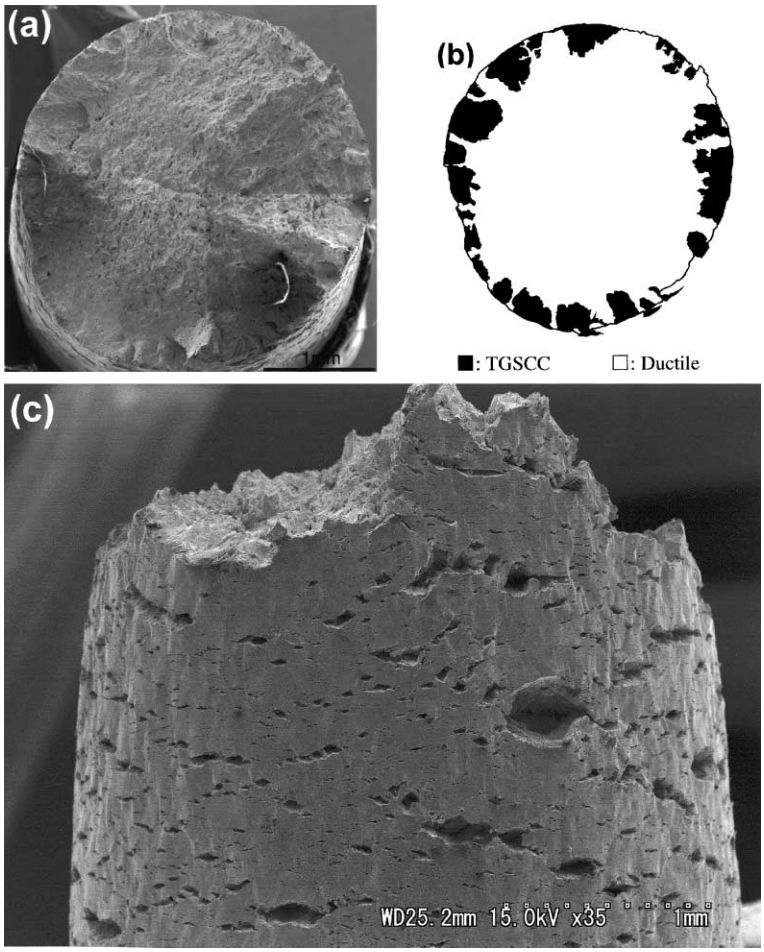

Fig. 5. SEM photographs of thermally sensitized HIPed joint specimen after creviced SSRT in oxygenated (DO = 20 wt ppm), high purity water at 513 K: (a) whole fractured surface, (b) schematic illustration of TGSCC area, and (c) TG crackings introduced by an artificial crevice.

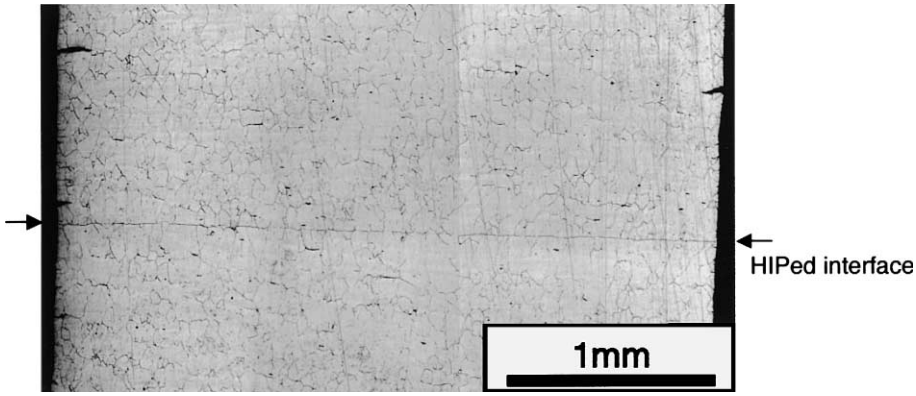

Fig. 6. Position of the HIPed interface in the HIPed joints after creviced SSRT in oxygenated (DO = 20 wt ppm), high-purity water at 513 K.

study was 0.6 wt ppb, which was low enough to prevent the occurrence of TGSCC. Akashi [3] suggested that the graphite-fiber wool used in this study could simulate the configuration of crevice and also promote chemical reactions, resulting to acceleration of SCC. Therefore, crevice may introduce TGSCC even in the water containing very low concentration of impurities. For all specimens, however, the propagation of TGSCC was not significant, and IGSCC did not occur and the specimens ruptured in 513 K water after elongation near to uniform elongation. Therefore, the effect of TGSCC seems to be not important for the fracture of thermally sensitized 316L(N)-IG SS under creviced condition. It is also inferred that if a quenching time from 1073 to 823 K during HIP process is shortened like in this study, a susceptibility to SCC can be ignored even under creviced condition.

4. Conclusions

To evaluate the integrity and susceptibility to SCC of a joint of 316L(N)-IG SS fabricated by the HIP technique, tensile tests in vacuum and SSRT in high temperature water were performed. From these experiments, the following conclusions were drawn:

1. HIPed joint specimens indicated no deterioration of tensile properties at RT-573 K and the HIP interface did not cause a fracture of specimen. Tensile properties of the specimen with the joint were comparable to those of base alloys.
2. No susceptibility to SCC of HIPed joint specimens was observed from SSRT in oxygenated water at 423 K, although some precipitates of $M_{23}C_6$ formed during the HIP process were detected at the interface.
3. Under creviced condition, thermally sensitized 316L(N)-IG SS showed a low susceptibility to TGSCC in oxygenated 523 K water. However, the HIP interface showed no initiation of TGSCC and the fracture mode of the specimen was ductile.

Acknowledgements

A part of this work is founded by ITER task (TASK no. T511-2JA). The authors gratefully acknowledge support by Dr Kalinin of joint central team task officer and Dr Ando of home team of Japan.

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