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Effects of water and irradiation temperatures on IASCC susceptibility of type 316 stainless steel

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Abstract

Effects of water and irradiation temperatures on irradiation-assisted stress corrosion cracking (IASCC) of type 316 stainless steel were investigated. Type 316 stainless steel was irradiated at 333–673 K to a dose level of 16 dpa. Susceptibility to IASCC was evaluated by slow strain rate testing in oxygenated water in the temperature range of 513–573 K. Irradiation at 603 and 673 K caused IASCC in 513 K water, but irradiation below 473 K did not induce IASCC at 513 K. Specimens irradiated at 333 K did not show IASCC susceptibility in 513 K water, but high susceptibility was observed in 573 K water. Effect of irradiation temperature is discussed from the view points of microstructural and microcompositional changes.
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1. Introduction

Type 316(LN) stainless steels will be used as structural materials of first wall/shield blanket modules for the International Thermonuclear Experimental Reactor (ITER). The first wall/shield blanket modules will be exposed to neutron irradiation up to about 5 dpa and cooled by high temperature water ranging from 373 to about 600 K. The cooling water produces a corrosive environment by radiolysis. In addition, neutron irradiation causes the hardening and the segregation at grain boundaries. As a result, austenitic stainless steels lose ductility and corrosion resistance at/near grain boundaries, and become sensitive to irradiation assisted stress corrosion cracking (IASCC), which has been studied extensively in the field of light water reactors. However, there are few studies of IASCC susceptibility in relatively low temperature water like the ITER operating condition [1–4]. During start-up and shut-down of the reactor, plasma disruptions and bake-out operation of ITER, the structural materials will be heated to temperatures that will be different from normal operating temperatures. Therefore, the effect of water temperature on IASCC susceptibility is important.

It was reported that type 316LN stainless steel irradiated at 473 K to 1 dpa did not exhibit any susceptibility to IASCC below 513 K in oxygenated water [3]. Type 316 stainless steel irradiated to 7.4 dpa at 333 and 473 K showed no IASCC susceptibility in oxygenated water at 333 and 473 K, respectively [2], while the stainless steel irradiated at 698 K to 40 dpa showed IASCC susceptibility in 473 K water [1]. In oxygenated water around 573 K, however, IASCC susceptibility was exhibited after the irradiation even to 1 dpa at 333 K [5]. From these previous studies, it is assumed that IASCC is influenced by both of water temperature and irradiation temperature and that irradiation at lower temperature has less influence on the IASCC susceptibility in oxygenated water at lower temperature. On the other hand, thermally-sensitized stainless steels exhibit susceptibility to intergranular stress corrosion cracking (IGSCC) even at 323 K in high purity water [6]. In order to examine this assumption, we studied the effect of water temperature and irradiation temperature on the IASCC susceptibility of type 316 stainless steel irradiated to a higher dose level.

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Table 1
Chemical composition (wt%)

C	Si	Mn	P	S	Cr	Ni	Mo	Ti	B	Fe
0.058	0.61	1.80	0.028	0.003	16.75	13.52	2.46	0.005	0.001	Bal.

2. Experimental procedure

The chemical composition of type 316 stainless steel used in this study is listed in Table 1. The material was solution annealed at 1323 K for 0.5 h followed by a water quench. A sheet type specimen was prepared from the material, and configuration of the specimen is shown in Fig. 1.

The specimens were irradiated at 333, 473, 603 and 673 K to about 16 dpa in the Oak Ridge Research Reactor (ORR) and High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory. A spectrally-tailored irradiation experiment was conducted, and the ratio of He generation to displacement damage was controlled to be about 11 appm/dpa. Details of this irradiation experiments are described in [7]. This He/dpa ratio is equivalent to that expected for a fusion reactor first wall/shield blanket. After the irradiation, a series of slow strain rate tests (SSRT) was performed in order to evaluate IASCC susceptibility at 513 and 573 K in oxygenated water. The concentration of dissolved oxygen (DO) in the inlet-water was controlled at 10 ppm at room temperature. Conductivity of inlet and outlet water was below 0.06 and 0.18 μS/cm, respectively. Elongation of specimen was measured from the deflection of a pull rod. Since the specimen has a large radius of curvature at shoulder (Fig. 1), deformation at the shoulder is not neglected during testing. Nominal strain was calculated as a ratio of the deflection of a pull rod to the specimen's gage length, and the strain rate was $2–4\times10^{-7}$ s^{-1}. After SSRT, the fracture surface was observed using a scanning electron microscope (SEM) in order to examine fracture mode and measure the ratio of IGSCC area on the fracture surface (%IGSCC). Results of tensile testing in vacuum was presented in reference [7].

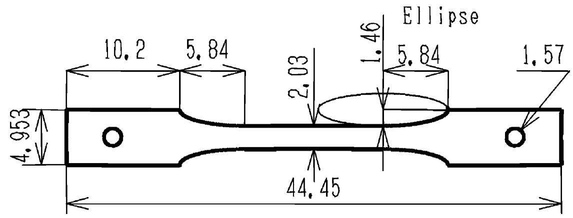


Fig. 1. Specimen configuration (thickness: 0.76 mm).

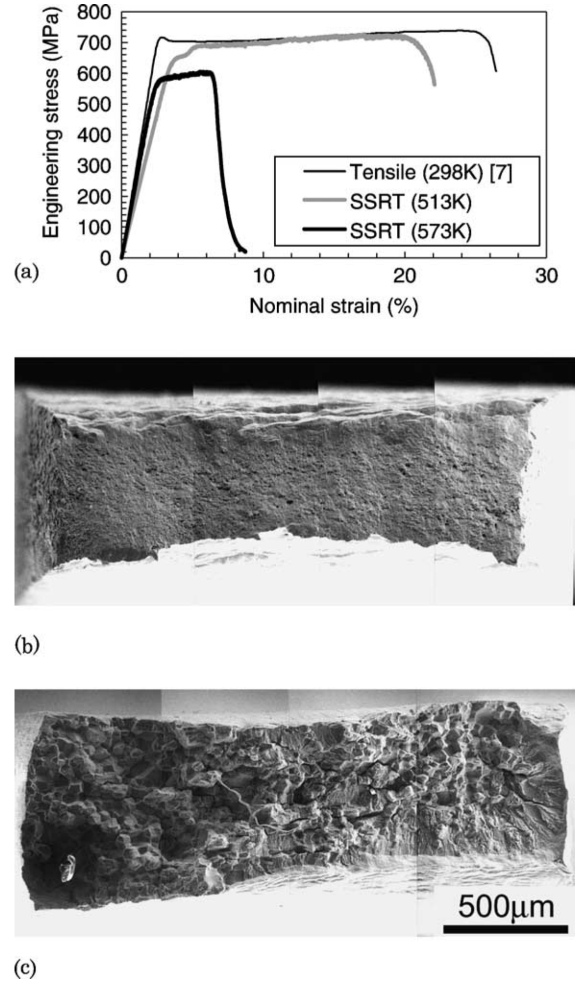


Fig. 2. Stress–strain curves and fracture surface of specimen irradiated at 333 K: (a) stress–strain curves; (b) fracture surface after SSRT at 513 K; (c) fracture surface after SSRT at 573 K.

3. Results

3.1. Irradiation at 333 K

Fig. 2(a) shows the stress–strain curves after SSRT at 513 and 573 K in oxygenated water. The stress–strain curve after a tensile test at 298 K in vacuum was also shown in the figure [7]. The stress–strain behavior in 513 K water was similar to that at 333 K. In 573 K water, total elongation was smaller than in 513 K water. Fig. 2(b) and (c) shows fracture surfaces after SSRT in 513 and 573 K water, respectively. Dimple ductile rupture occurred in 513 K water. However, intergranular fracture was observed on a specimen tested at 573 K with the %IGSCC of the specimen about 54%.

3.2. Irradiation at 473 K

Fig. 3(a) shows the stress–strain curve after SSRT at 513 K in oxygenated water. For comparison, the stress–strain curve after tensile testing at 473 K in vacuum is also shown in the figure [7]. Tensile strength in 513 K water was equivalent to that at 473 K. The elongation after SSRT was larger than after the tensile test. This may be caused by an incorrect measurement of the elongation, as mentioned in experimental procedure. The SEM photograph shows complete ductile fracture after SSRT at 513 K in oxygenated water (Fig. 3(b)).

3.3. Irradiation at 603 K

Fig. 4(a) shows a stress–strain curve after SSRT at 513 K in oxygenated water. The stress–strain curve after a tensile test at 603 K in vacuum is also plotted in the figure [7]. The specimen tested by SSRT exhibited a steep decrease of stress near the yield stress of specimen tensile-tested at 603 K. The fracture surface is shown in Fig. 4(b). IGSCC was observed and %IGSCC was about 82%.

3.4. Irradiation at 673 K

Fig. 5(a) shows the stress–strain curve after SSRT at 513 K in oxygenated water. The stress–strain curve after

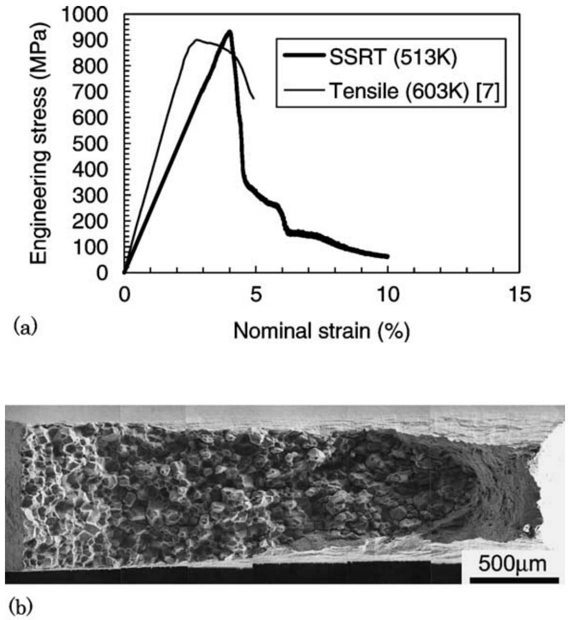


Fig. 4. Stress–strain curves and fracture surface of specimen irradiated at 603 K: (a) stress–strain curves; (b) fracture surface after SSRT at 513 K.

tensile test at 673 K in vacuum is also shown in the figure [7]. In 513 K water, stress rapidly decreased when the stress reached around 500 MPa. By comparing the elastic deformation behavior of the SSRT and tensile tests, the specimen tested by SSRT seemed to fail without distinct plastic deformation (before yielding). SEM observation of fracture surface revealed that the specimen failed completely by IGSCC (Fig. 5(b)).

3.5. Effect of water temperature and irradiation temperature

The dose dependence of IASCC susceptibility in oxygenated high-temperature water is shown in Fig. 6 for type 316(L) stainless steels irradiated around 473 K [2–4,8]. The results of this study indicated that the specimens heavily irradiated around 473 K did not exhibit IASCC susceptibility below 513 K in oxygenated water. In 573 K water, on the other hand, type 316L stainless steel showed IASCC even after irradiation to 1 dpa [3].

Fig. 7 summarizes the effect of water temperature and irradiation temperature on IASCC susceptibility of type 316(L) stainless steels. Data from previous studies [1–3] is also plotted in the figure. In oxygenated water below 513 K, specimens irradiated below 473 K shows no IASCC susceptibility. In 573 K water, on the other hand, IASCC occurred in all specimens irradiated at 333–698 K. Specimens irradiated above 603 K to higher

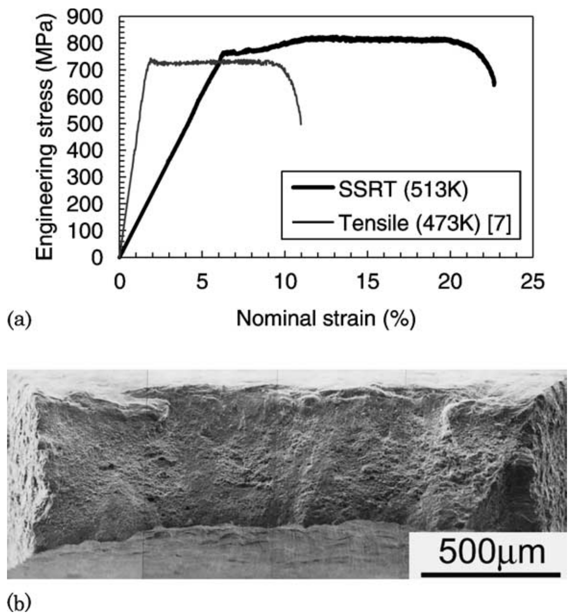


Fig. 3. Stress–strain curves and fracture surface of specimen irradiated at 473 K: (a) stress–strain curves; (b) fracture surface after SSRT at 513 K.

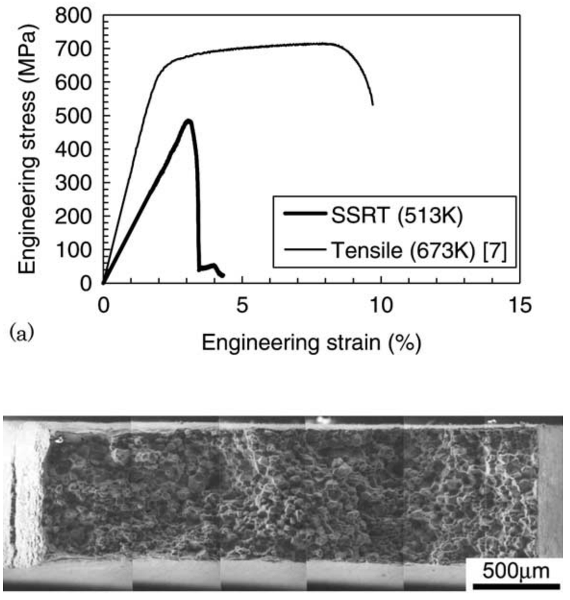
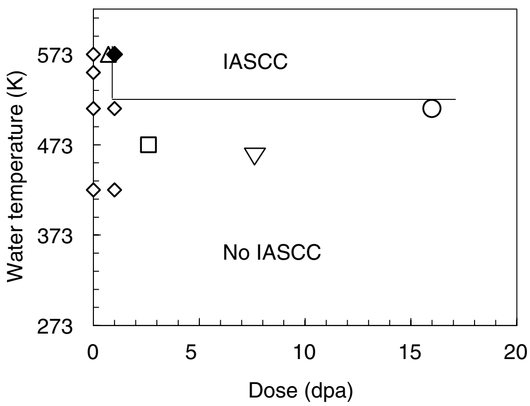


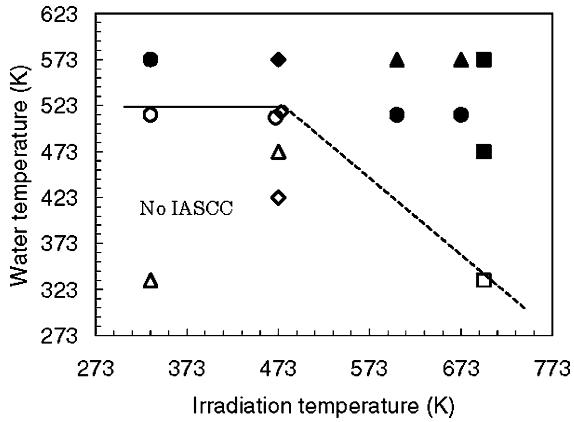
Fig. 5. Stress–strain curves and fracture surface of specimen irradiated at 603 K: (a) stress–strain curves; (b) fracture surface after SSRT at 513 K.



Materials	IASCC	Ductile	Refernce
316	—	▽	[2]
316LN	◆	◇	[3]
316LN	—	□	[4]
316L	—	△	[8]
316	—	○	This study

Fig. 6. Dose dependence of IASCC susceptibility for type 316 stainless steels irradiated around 473 K.

dose level exhibited IASCC even at 513 K in oxygenated water.



Materials	Dose (dpa)	IASCC	Ductile	Reference
316LN	1	◆	◇	[3]
316	7	▲	△	[2]
	16	●	○	This study
20%CW 316	40	■	□	[1]

Fig. 7. Water temperature and irradiation temperature dependence of IASCC susceptibility for type 316 stainless steel.

4. Discussion

Irradiation temperature strongly influences the microstructural and microcompositional changes, and which could affect on mechanical properties. An effect of irradiation temperature was detected on IASCC susceptibility in oxygenated water below 513 K (Fig. 7).

Formation of dislocation loops, precipitates (including solute segregation), cavities and phase transformations from γ to α were induced by irradiation [9,10]. The irradiation above 643 K, enhances carbide precipitation [9,10]. The irradiation above 673 K showed IASCC susceptibility at 473–513 K in oxygenated water, as shown in Fig. 6. For unirradiated austenitic stainless steels, the sensitization occurs due to Cr depletion accompanied with carbide formation on grain boundaries. Sensitized stainless steels showed IGSCC susceptibility at lower temperature such as about 323 K [6]. It was speculated that the formation of radiation-assisted carbides might have an influence on the IASCC susceptibility. However the carbide formation was occasionally observed at grain boundaries of austenitic stainless steels irradiated around 643–673 K [9,10]. Therefore, other microstructural features may influence the IASCC susceptibility of specimens irradiated above 673 K.

Microcompositional changes at/near grain boundaries are caused by the radiation-induced segregation (RIS). The concentration of Cr decreases at/near grain boundaries, but concentration of Ni and Si increases. The temperature dependence of segregation of major alloying elements was predicted by a model based on the inverse-Kirkendall mechanism [11]. The amount of Cr depletion increased with increasing irradiation temperature [11]. Therefore the increased amount of Cr depletion at higher temperature may contribute to the observed IASCC susceptibility in lower temperature water (Fig. 6). It was calculated, on the other hand, that irradiation up to 10 dpa causes Cr depletion (less than 14%) even at 373 K [11]. In this study, the specimen irradiated at 333 K to 16 dpa exhibited IASCC susceptibility in 573 K water (Fig. 2). Type 316LN-IG irradiated at 473 K to 1 dpa also showed IASCC susceptibility in 573 K water [3]. Type 304L stainless steels become susceptible to IASCC at 573 K in oxygenated water after irradiation at 323 K to 1 dpa [5]. Irradiation to about 1 dpa above 323 K seems to induce enough Cr depletion to cause IASCC in 573 K water. In 513 K water, however, IASCC was not observed in the specimen that was irradiated at 473 K up to 16 dpa. For this specimen, it is predicted that Cr depletion at/near grain boundaries is enough to cause IGSCC. If the occurrence of IASCC in 573 K water is assumed to be mainly caused by Cr depletion at grain boundaries due to RIS, it is speculated from the results of no IASCC susceptibility below 513 K water that the IASCC mechanism is different from the SCC mechanism of thermally-sensitized materials. Therefore Cr depletion alone seemed not to be key material parameter for IASCC in lower temperature water. Microstructural observations and microchemical analyses at grain boundaries may help to understand the contribution of precipitates and RIS to IASCC susceptibility in lower-temperature water.

Dislocation structures were also influenced by irradiation temperature [7]. Small black dot defect clusters are a dominant structure at temperatures from 323 to 523 K. At 523–673 K, faulted Frank loops dominate the microstructure. Above an irradiation temperature of about 673 K, the density of Frank loops decreased rapidly and the density of other defects such as cavities and precipitates increased. These defects affected micromechanical deformation mechanisms [7,12–14], especially the changes in flow properties such as strain to necking (STN) which is favored by localized deformation, depending on the size and density of defects, mainly Frank loops, and test temperature [12,14]. The STN remains high at temperatures below 473 K or above 673 K after the irradiation to 16 dpa [7]. At temperatures around 573 K, a significant reduction of the STN was observed after irradiation at 7.4 dpa and the stress rapidly decreased by mechanical instability just after yielding [9]. In this study, STN greater than 5% was observed on the specimens irradiated below 473 K. The specimens did not show any IASCC susceptibility in 513 K water. It was speculated that the lower susceptibility to localized deformation may be related to the reduced susceptibility to IASCC in 513 K water.

Type 316 stainless steel irradiated at 673 K up to 16 dpa showed high STN after tensile testing at 673 K [9]. The stainless steel showed IASCC susceptibility in 513 K water and the IASCC seemed to initiate before yielding (Fig. 5). Similar results were observed on the specimen irradiated at 673 K to 7.4 dpa and tested by SSRT in 573 K water [2]. Type 304 stainless steel irradiated at 673 K to 50 dpa also showed high STN after tensile testing in air at 562 K and IASCC susceptibility in 562 K water [15]. It appeared that IASCC initiated before yielding in type 304 stainless steel [15]. Irradiation above 643 K is prone to initiate IASCC before yielding and to enhance IASCC susceptibility, although irradiation above 673 K caused less deterioration of flow properties than around 573 K. Therefore, a flaw property such as STN is also not the only key material parameter.

5. Conclusions

Effect of water temperature and irradiation temperature on IASCC susceptibility of type 316 stainless steel was examined using slow strain rate testing in oxygenated water at temperatures of 513 and 573 K. The type 316 stainless steel was irradiated to 16 dpa at temperatures ranging from 333 to 673 K in spectrally-tailored irradiation experiments. A strong effect of irradiation temperature on IASCC susceptibility was observed in 513 K water. Specimens irradiated below 473 K did not show IASCC susceptibility, but specimens irradiated above 603 K did. In 573 K water, on the other hand, IASCC susceptibility did not depend on the irradiation temperature.

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