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Stress corrosion cracking susceptibility of a reduced-activation martensitic steel F82H

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A B S T R A C T

In order to examine the stress corrosion cracking (SCC) susceptibility of reduced-activation ferritic/martensitic steel, F82H, slow-strain-rate-test (SSRT) was performed at various temperatures in oxygenated or hydrogenated water. Test specimens of F82H were heat-treated at various temperature conditions, or were cold-worked to simulate radiation hardening and machined to make single edge notch, or were neutron-irradiated at 493 K to 3.4 dpa. It was found that in unirradiated specimen, IGSCC occurred when specimen was normalized only, and TGSCC occurred when cold-worked (over 23%) and notched specimen was tested by SSRT at 573 K in oxygenated water. In irradiated specimen, TGSCC occurred, when SSRT was conducted at 573 K in hydrogenated (DH = 1 ppm) water or when the notched specimen was tested by SSRT at 573K in oxygenated (DO = 10 ppm) water.

1. Introduction

Intergranular stress corrosion cracking (IGSCC) and irradiation-assisted stress corrosion cracking (IASCC) are main fracture modes of structural materials in aged light water reactors [1,2]. Even in fusion reactor, the SCC may become one of the problems for structural materials after long time operation. In the ITER Test Blanket Modules planned by the Japan Atomic Energy Agency, the water cooled solid breeder blanket was selected as the primary candidate [3]. The structural material of the modules is a reduced-activation ferritic/martensitic (RAFM) steel such as F82H and the coolant is water. The temperature and pressure was 553–598 K and 15.5 MPa, respectively.

For SCC susceptibility of RAFM in high temperature water or supercritical water, some data has been reported in Refs. [4–8]. In databases and design rules [9,10], however, corrosion and SCC are not considered yet. In this work, slow-strain-rate-test (SSRT) was carried out on specimens that were heat-treated at various tempering and post welding heat treatment (PWHT) conditions, and on specimens that were cold-worked and notched in order to provide hardness and stress concentration, and on specimens that were neutron-irradiated at 493 K to 3.4 dpa. Then the conditions likely to induce SCC in F82H were examined.

2. Experimental procedure

2.1. Materials and specimen configuration

The RAFM material used in this work was F82H IEA-heat that was heat-treated by normal normalizing and tempering condition, hereinafter called NT-specimen. The weld deposited metal (depo-specimen) fabricated from weld joint was also used. The weld joint was prepared from tungsten–inert-gas (TIG) welded plates with U-groove [11], and the joint was post-welded heat-treated. The chemical compositions of NT-specimen and depo-specimen are listed in Table 1.

Tensile specimen configurations used in this work are shown in Fig. 1(a)–(c). For neutron-irradiation experiment, miniature sheet type specimen (Fig. 1(c)) was used in order to reduce radioactivity. For examination of influence of stress concentration, single edged notch was fabricated at gage center of smooth sheet specimens (example; Fig. 1(b) and (c)).

2.2. Weld heat affect zone simulating condition and radiation hardening simulating condition

In order to examine the effects of unexpected heating during weld process, three kinds of heat treatment condition were applied to NT-specimens. Details of heat treatment conditions are listed in Table 2. In the first condition, only normalizing heat treatment was adapted to NT-specimen, hereinafter called asN-specimen. In the second condition, it was postulated that during PWHT weld deposition metal was over heated to 1073 K near Ac_1 (1093 K [12])

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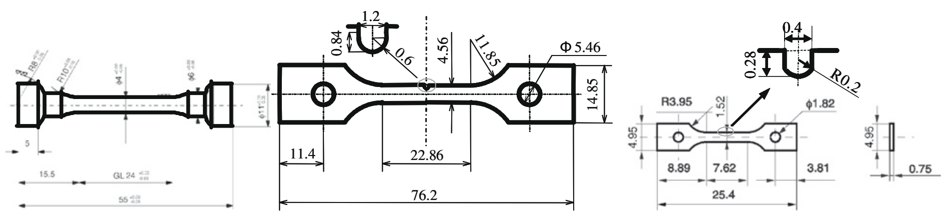
Table 1
Chemical compositions (wt%).

	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	N	Ti	Ta	W
F82H	0.09	0.07	0.1	0.003	0.001	0.01	0.02	7.87	0.003	0.19	0.007	0.004	0.03	1.98
Weld depo[a]	0.07	0.09	0.46	0.003	0.003	0.005	–	7.41	–	0.18	0.005	–	0.023	2.02

Normalizing condition: heat treat at 1313 K for 38 min and then air cooled.
Tempering condition: heat treat at 1023 K for 60 min and then air cooled.
PWHT condition: heat treat at 993 K for 60 min and then air cooled.
[a] Chemical composition of TIG weld wire.



(a) Round bar type specimen (b) Notched sheet type specimen (c) Miniature notched sheet type specimen

Fig. 1. Specimens' configurations (unit = mm).

Table 2
Welding process simulating heat treatment condition.

	Normalizing			Tempering		
	Temp. (K)	Time (min)	Quenching	Temp. (K)	Time (min)	Quenching
asN	1313	38	Water	–	–	–
Ac_1	1313	38	Water	1073	60	Water
HAZ	Partial austenitization (at 1123 K for 10 min)		Water	Post welding heat treatment (at 993 K for 60 min)		Water

where asN-specimen begun to form γ phase. This specimen was called Ac_1-specimen. In the third condition, heat affected zone (HAZ) was simulated for NT-specimen by partial austenitization and following PWHT. This specimen was called HAZ-specimen.

Since the macroscopic true stress-true strain behavior after irradiation could be modeled [13] by Swift type equation [14], NT-specimens were cold-worked to the rolling reduction of 23% and 47% [15]. For the sake of acceleration of SCC initiation by stress concentration [16–18], single edge notch (SEN) was machined in gage center of the cold-worked specimens.

In these heat-treated and cold-worked specimens, micro Vickers hardness tests were performed to evaluate strength property change. The test weight was 5 N. The Vickers hardness was average value of over 8 point measurements.

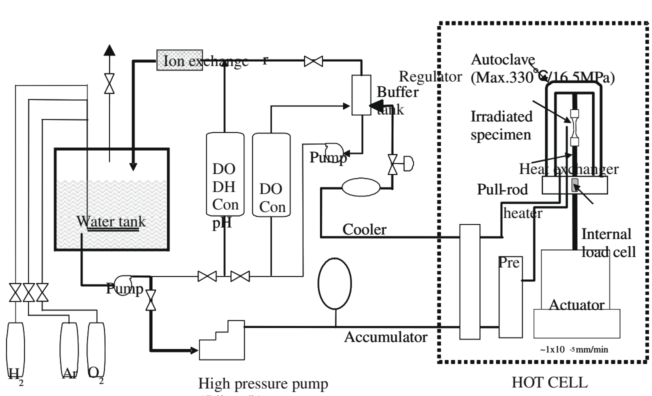


Fig. 2. Illustration of water flow of SSRT machine for irradiated materials.

2.3. Neutron-irradiation experiment

Neutron irradiation was conducted at 493 K to dose level of 3.4 dpa (dose rate = 1.0×10^{-7} dpa/s) in Japan Research Reactor No. 3. Thermal ($E < 0.68$ eV) and fast ($E > 1.0$ MeV) neutron fluences were 6.5×10^{25} and 2.4×10^{25} n/m^2, respectively, and the irradiation time was 9306.6 h.

2.4. SSRT condition

Fig. 2 shows the schematic diagram of SSRT and high temperature water loop. In this work, testing parameter such as water temperature, concentration of dissolved oxygen (DO) and dissolved hydrogen (DH), and strain rate were varied ranging from 513–603 K, DO = 40–10000 ppb or DH = 0–1 ppmn, and 2×10^{-7} to 1×10^{-6} s^{-1}, respectively. The water conductivity was below 0.1 μS/cm at the inlet of the test autoclave.

After the SSRT, SEM observation was carried out to measure the area of transgranular type SCC (TGSCC) and IGSCC on fracture surface. The side surface of the specimens was also examined using SEM. SCC susceptibility was evaluated from the ratio of SCC area to fracture surface area (%SCC).

3. Results and discussion

3.1. Unirradiated materials

3.1.1. NT-specimen and depo-specimen

SCC susceptibility of NT-specimen was examined at 513, 573 and 603 K in oxygenated (DO = 10 ppm or 40 ppb) water. The results are given in Table 3. In all NT-specimens, fracture surface

Table 3
SCC susceptibility of NT and depo-specimens.

DO	Temp. (K)	%SCC
NT-specimen		
10 ppm	513	0
	573	0
	603	0
40 ppb	573	0
Depo-specimen		
10 ppm	573	0

showed a typical martensitic mixed quasi-cleavage and ductile–dimple fracture. Depo-specimen showed higher yield stress and lower fracture strain than NT-specimen during SSRT. Only dimple was observed on the fracture surface of the depo-specimen. Some small cracks were observed at side surface of all specimens. Tensile properties of NT and depo-specimens tested by SSRT are equal to those tensile tested in vacuum.

It is reported that low alloy steels become sensitive to the strain-induced corrosion cracking (SICC) at faster strain rate of $1 \times 10^{-6}\ s^{-1}$ [19]. A NT-specimen was tested by SSRT at 573 K with a faster strain rate of $1 \times 10^{-6}\ s^{-1}$ in oxygenated (DO = 10 ppm) water. Yield stress and UTS were equivalent to those tested by slower strain rate. Fracture strain was 3% smaller than that tested by a slower strain rate. Only dimple pattern was observed on the fracture surface. SCC dose not occur in NT-specimen of F82H even in the SICC initiation strain rate.

Table 4
SCC susceptibility of welding process simulating heat-treated specimens.

	$Hv_{0.5}$	%SCC
HAZ-specimen	191 ± 3.1	0
Ac_1-specimen	195 ± 3.8	0
NT-specimen	221 ± 7.2	0
asN-specimen	397 ± 4.8	29.9 (IGSCC)

Table 5
SCC susceptibility of cold-worked specimen with single edge notch.

	$Hv_{0.5}$	%SCC
NT-specimen	221 ± 7.2	0
23%CW-specimen	242 ± 5.1	5.8 (quasi-TGSCC)
47%CW-specimen	254 ± 4.3	8.7 (quasi-TGSCC)
asN-specimen	397 ± 4.8	91.1 (IGSCC)

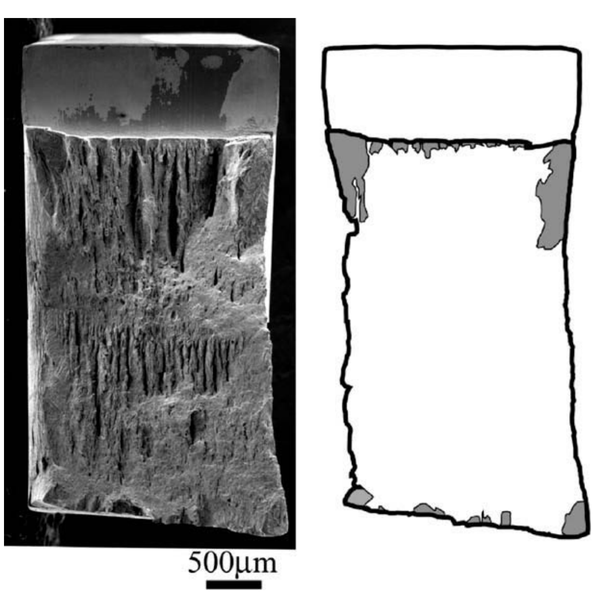
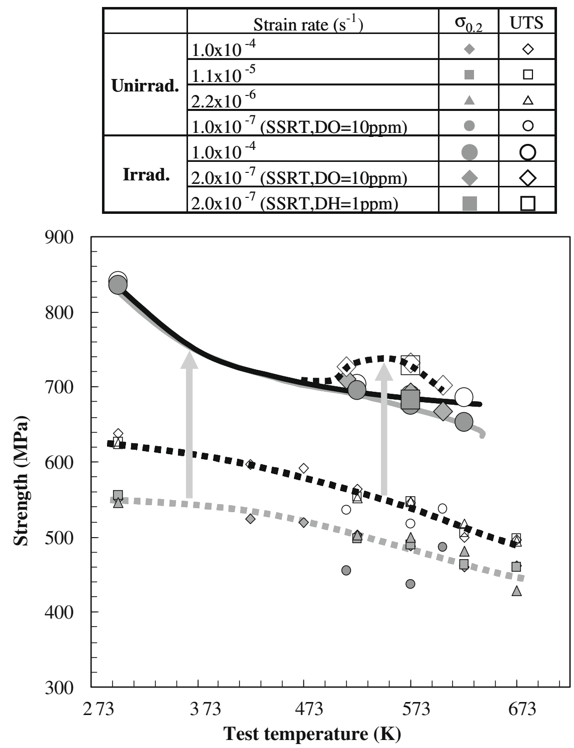


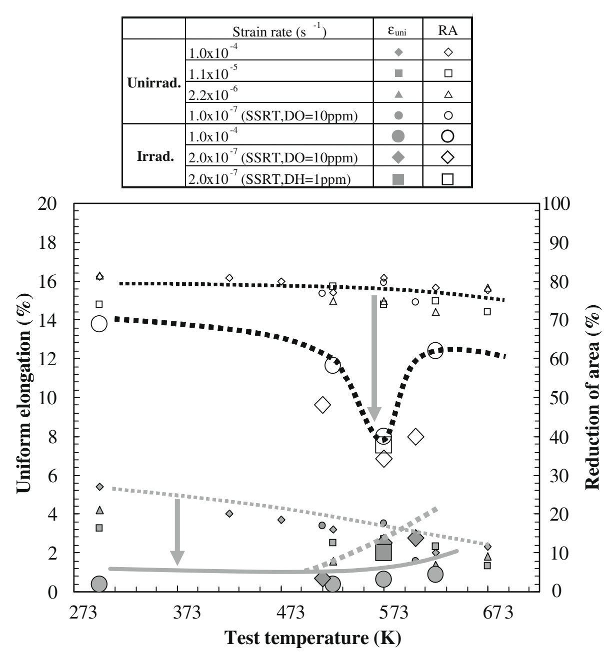
Fig. 3. Quasi-TGSCC on fracture surface in 47%CW-specimen (gray area means quasi-TGSCC).

3.1.2. Welding process simulating heat-treated specimens

SCC test results of three kinds of heat-treated specimens are given in Table 4, and in the table micro Vickers hardness data are



(a) Yield stress ($\sigma_{0.2}$) and ultimate tensile stress (UTS)



(b) Uniform elongation (ε_{uni}) and Reduction of area (RA)

Fig. 4. Tensile properties of neutron-irradiated F82H.

also indicated. IGSCC occurred only in asN-specimens at crevice between chucking jigs and the specimen, although SSRT was performed four times with round bar type and sheet type asN-specimens. In order to initiate SCC, more harmful water condition rather than inlet water is needed. As seen in Table 4, hardness of asN-specimen is the highest among other specimens. The hardness of Ac_1-specimen and HAZ-specimen are lower than that of NT-specimen. This dependence of hardness on heat history is the same to that during weld and PWHT [12].

One kind of low alloy steel, SFVC2B, with lower hardness due to heat treatment has higher SCC susceptibility in higher temperature water [20]. In this work, SCC dose not occur in the HAZ-specimen and the Ac_1-specimen that have lower hardness. Martensitic stainless steels are thermally sensitized when the steels are heat-treated at high temperature (~1000 K) in short time (~ a few minute) and then tempered al lower temperature [21]. In this work, both Ac_1-specimen and HAZ-specimen were tempered at about 1000 K for 1 h followed by water quenching, so that the thermal sensitization did not occur in these specimens.

3.1.3. Radiation hardening simulating specimen

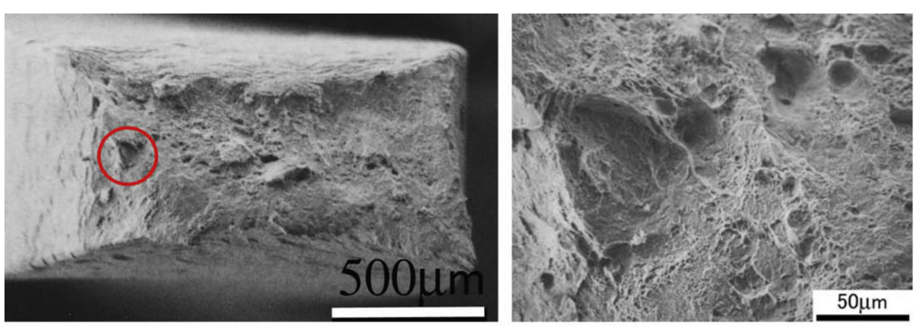
SCC susceptibility of cold-worked specimens with SEN was evaluated at 573 K in oxygenated (DO = 10 ppm) water. SCC susceptibility of each hardened specimen is compared in Table 5. The Vickers hardness data are also listed in the table. IGSCC occurred only in asN-specimen with SEN. The IGSCC took place in the crevice of chucking jig, and no SCC was observed in the SEN. In CW-specimens, small area of quasi-TGSCC was observed at SEN tip and at the back surface against the notch, as shown in Fig. 3. The stress condition near back surface side seemed to undergo compressive stress. The area of quasi-TGSCC area at the tip is larger than that at the back surface. NT-specimen was completely broken in the ductile mode. The hardness of cold-worked specimens is higher than NT-specimen and smaller than asN-specimen. The level of cold work over 23% is much higher than total elongation of F82H at RT. This level of hardening may affect the SCC susceptibility in F82H.

3.2. Neutron-irradiated material

3.2.1. Effects of test temperature and environment on tensile and SSRT properties and on IASCC susceptibility

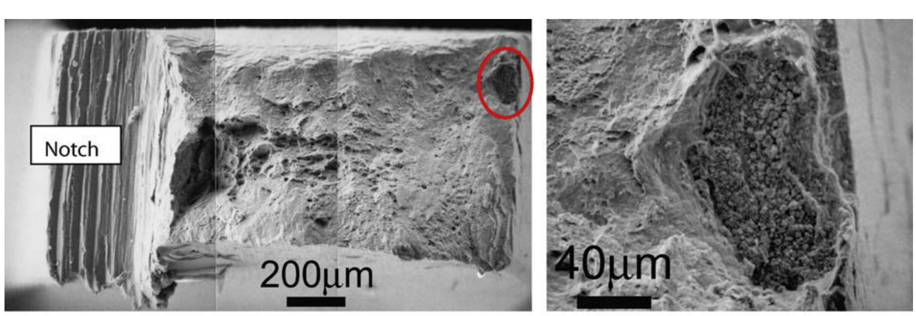
A few miniature sheet type specimens irradiated 493 K to 3.4 dpa were tensile-tested in air at temperature range from 298 to 623 K with a strain rate of $1.0 \times 10^{-4}\ s^{-1}$. Dependences of test temperature on tensile strength and ductility are shown in Fig. 4(a) and (b), respectively. The strain rate dependence of unirradiated F82H steel tensile-tested with various strain rates of 2.2×10^{-6} to $1.0 \times 10^{-4}\ s^{-1}$ is also indicated in the figure. Unirradiated F82H steel dose not have a strain rate dependence on tensile properties in the strain rate and temperature range conducted in this work. After irradiation, the increase of yield strength and UTS accompanies the large decrease of uniform elongation and reduction of area (RA). The uniform elongation of irradiated specimens shows a recovery tendency above 573 K, and the recovery tendency is pronounced in specimens tested by SSRT. RA showed unexpected temperature dependence around 573 K.

IASCC susceptibility was examined by SSRT with a strain rate of $1 \times 10^{-7}\ s^{-1}$ in oxygenated (DO = 10 ppm) water at temperature of 513, 573, and 603 K. In these conditions, SCC was not formed in irradiated specimen. In another RAFM steel, Eurofer 97, no SCC is reported in smooth round bar specimen after irradiation at 573 K up to 2.3 dpa and SSRT at 573 K in oxygenated (200 ppb) or hydrogenated (DH = 650 ppb) water [5]. However, quasi-TGSCC was observed in irradiated NT-specimen at 573 K in hydrogenated (DH = 1 ppm and DO = 40 ppb) water (Fig. 5). Area of the quasi-TGSCC



(a) Fracture surface (b) Quasi-TGSCC area in circle in (a)

Fig. 5. Quasi-TGSCC occurrence at smooth sheet type specimen irradiated at 497 K to 3.4 dpa and tested by SSRT at 573 K in hydrogenated (DH = 1 ppm) water.



(a) Fracture surface of notched specimen (b) Quasi-TGSCC area in circle in (a)

Fig. 6. Quasi-TGSCC occurrence at notched specimen irradiated at 497 K to 3.4 dpa and tested by SSRT at 573 K in hydrogenated (DO = 10 ppm) water.

is very small. Authors reported that in irradiated austenitic stainless steel (type 316L-IG), DH-induced TGSCC occurred at lower temperature [22]. Influence of DH on IASCC of RAFM must be studied in further detail.

3.2.2. Influence of stress concentration

In an irradiated miniature sheet type specimen, SEN was machined by slow speed diamond saw after irradiation. This specimen was tested by SSRT at 573 K in oxygenated (D = 10 ppm) water with a strain rate of 1×10^{-7}. Fig. 6 shows fracture surface with a small area of quasi-TGSCC against the SEN side. As seen in Fig. 6(b), the quasi-TGSCC was covered by large oxide crystals. In unirradiated 23% and 47%CW-specimen, quasi-TGSCC was observed mainly at SEN tips (Fig. 6), however in irradiated specimens the quasi-TGSCC was not observed on the fracture surface at SEN tip. The quasi-TGSCC in irradiated specimen may be caused easily by compressive strain. The yield stress of irradiated specimen was intermediate between that of 23%CW-specimen and that of 47%CW-specimen. The mechanism of quasi-TGSCC initiation in irradiated specimen may be different from that in cold-worked specimen.

4. Conclusions

The SCC susceptibility of a reduced-activation ferritic/martensitic (RAFM) F82H was evaluated by slow stain rate test (SSRT) in high temperature, oxygenated or hydrogenated water. The F82H specimens were heat-treated to simulate the material condition after TIG-welding, or were cold-worked to simulate the hardening after irradiation, or were machined to provide stress concentration, or were neutron-irradiated. Following results were obtained:

(1) IGSCC occurred in only normalized specimens, but influence of stress concentration on IGSCC initiation was weaker than that of water condition in crevice.
(2) The quasi-TGSCC occurred in cold-worked (23–47%) specimens with single edge notch (SEN). The quasi-TGSCC was observed on the fracture surface at tip and near the back side against the notch. In normalized and tempered specimens, no SCC was observed.
(3) In irradiated specimens, TGSCC did not occurred in smooth sheet type specimen tested by SSRT at 573 K in oxygenated (DO = 10 ppm) water. However, the quasi-TGSCC was observed on the fracture surface near back side against notch even in the oxygenated water. In hydrogenated (DH = 1 ppm and DO = 40 ppb) water, the quasi-TGSCC was observed even in the smooth sheet type specimen.

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