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In-situ SCC observation of thermally-sensitized and cold-worked type 304 stainless steel irradiated to a neutron fluence of 1×10^{25} n/m^2

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ABSTRACT

Crack initiation and crack growth processes of irradiation assisted stress corrosion cracking of stainless steels were studied by slow strain rate testing (SSRT) in oxygenated high temperature 561 K water. In-situ observation was carried out during SSRT for type 304 stainless steel irradiated to a neutron fluence of 1.0×10^{25} n/m^2 ($E > 1$ MeV) at 323 K in the Japan material testing reactor. The specimens were subjected to solution annealing, thermal sensitization, or cold working prior to neutron irradiation. The solution annealed material exhibited a combination of transgranular stress corrosion cracking (TGSCC) and ductile fracture, and almost all intergranular stress corrosion crackings were observed in the thermally-sensitized material. In the cold-worked material, cracking was introduced before the maximum stress was reached, and the fracture mode changed from TGSCC to ductile fracture to transgranular cracking together with the progress of crack growth in one direction.

1. Introduction

Austenitic stainless steels (SSs) will be used as major structural materials for the first wall/shield blanket modules and the vacuum vessel in the international thermonuclear experimental reactor (ITER). In the ITER R&D program [1], irradiation assisted stress corrosion cracking (IASCC) is considered as one of the specific problems of the application of SSs to fusion. To examine crack initiation and crack growth processes of IASCC in SSs, in-situ observation of the gage length of irradiated specimens was carried out during slow strain rate testing (SSRT) in oxygenated high temperature water. By in-situ observation during SSRT, it is possible to obtain visual information, e.g. the timing and location of crack initiation, the number of introduced cracks and the crack growth rate. Moreover, the combination of the visual information, the stress–strain curve and the fracture surface examination is useful to understand IASCC behavior. Results from in-situ observation on type 304 SS irradiated to a neutron fluence of 1.0×10^{26} n/m^2 ($E > 1$ MeV) have been reported already [2]. Irradiated type 304 SS was used for in-situ observation, though the first wall tube and the shield block of the ITER would be made from type 316L(N)-IG SS which had good resistance to stress corrosion cracking (SCC). Since SCC susceptibility in type 304 SS is higher than that in type 316L(N)-IG SS, it is effective to investigate IASCC behavior in type 304 SS for detection effects of heat treatment and cold working (CW). The results from in-situ observation during SSRT of type 304 SS irradiated to a fluence of 1.0×10^{25} n/m^2 ($E > 1$ MeV) which is around the threshold fluence for IASCC susceptibility, are presented comprehensively in this paper.

2. Experimental

2.1. Materials

Commercial purity type 304 SS was used for this study. The chemical composition expressed in wt% is Fe–0.063C–0.50Si–0.98Mn–0.026P–0.015S–9.99Ni–18.65Cr–0.036N–<0.01Co. Type 304 SS was subjected to solution annealing (SA) at 1373 K for 60 min, thermal sensitization (TS) for 1023 K for 100 min followed by aging at 773 K for 24 h, or CW (20%). The specimens, with gage length of 20 mm, width of 4 mm and thickness of 1 mm, were machined and loaded in an irradiation assembly and irradiated at 323 K in the coolant water of the Japan material testing reactor (JMTR) for a total of 69 days. The fast neutron fluence was estimated to be 1.0×10^{25} n/m^2 ($E > 1$ MeV).

2.2. SSRT and in-situ observation

SSRT was conducted in oxygenated high purity water at 561 K and 9.0 MPa. The applied strain rate was 1.7×10^{-7} s^{-1}. Dissolved oxygen (DO) concentration was controlled at 8 ppm. The gage

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length section of the specimen was observed through a window equipped in the autoclave with a charge-coupled device (CCD) camera system during SSRT. Detailed conditions regarding SSRT and in-situ observation have been described previously [2]. Subsequently, fracture surface examination was performed using a scanning electron microscope (SEM).

3. Results

The results of SSRT are summarized in Table 1. In-situ observation of the TS and CW materials was successfully carried out. Stress–strain curves during SSRT of the SA, TS and CW materials are shown in Fig. 1. The SA material exhibited the highest elongation and the CW material exhibited the highest maximum stress of the all specimens. Although stress–strain behavior at around the yield stress of the TS material was similar to that of the SA material, stress decreased after reaching 476 MPa.

Fracture surfaces and schematic illustrations of the specimens are shown in Fig. 2. The SA material exhibited a combination of transgranular (TG) SCC (TGSCC) and ductile fracture mode, and almost all intergranular SCC (IGSCC) were observed in the TS material. Since the SA material exhibited no IGSCC, to induce IGSCC, it is not sufficient to be irradiated to a neutron fluence causing IASCC susceptibility. For this reason, IGSCC was not introduced by IASCC in the TS material. The CW material exhibited TGSCC, a dimple pattern and TG cracking consisting of some isolated cracks without a river pattern (see Fig. 2(e)).

4. Discussion

In Fig. 3, crack initiation and growth behavior are shown in a stress–strain curve, fracture surfaces and the images of in-situ observation during SSRT for the CW material. On the CW material irradiated to a fluence of 1.0×10^{25} n/m^2, crack initiation was observed before the maximum stress was reached, though crack initiation in the CW material irradiated to a fluence of 1.0×10^{26} n/m^2 was observed immediately after the maximum stress was reached [2]. In unirradiated thermally-sensitized type 304 SS, the initiation of the first crack was observed when the stress reached 80–90% of the maximum stress [3]. Stress at the initiation of the first crack increased with neutron fluence. Fukuya et al. [4] found that both dislocation channels and twins caused microscopic strain accumulation at interactions at grain boundaries and that strain accumulation was likely to cause IG crack initiation in irradiated SSs. Onchi et al. [5] conducted SSRT in irradiated thermally-sensitized 304 SS in inert gas and water and reported that the initiation of IG cracks in water would occur so as to relieve stress and strain concentration at grain boundaries. They inferred that TG cracking might be initiated at deformation twin boundaries. It is reasonable considering stress/strain accumulation at grain boundaries that the stress at the initiation of the first crack increases with neutron fluence.

The initiated crack propagated in one direction, from the left side to the right side in images of in-situ observation. TGSCC changed to ductile fracture at a strain of 4.1%, after which stress decreased rapidly from 730 MPa to 330 MPa. Subsequently, TG cracking was introduced at a strain of 4.2% and the rate of stress reduction slowed down. Since displacement induced by SSRT was small, deformation during a ductile fracture could not reach the quantity of deformation required for failure. Instead, the deformation rate decreased, and TG cracking was introduced. Therefore it is expected that the CW material exhibits progressive change in fracture mode from TGSCC to ductile fracture to TG cracking with time.

Table 1
Results of SSRT after neutron irradiation to 1×10^{25} n/m^2 ($E > 1$ MeV).

Specimen ID	Treatment	Yield stress (MPa)	Maximum stress (MPa)	Total elongation (%)	Fraction of IGSCC (%)	Fraction of TGSCC (%)	Reduction in area (%)	In situ observation	Number of introduced cracks
F5	SA	463	568	11.5	0	51.2	51.0	Not successful	–
F6		478	584	13.1	0	88.9	54.4	Not successful	–
H4	TS	450	476	3.7	98.1	0	24.1	Successful	1
H5		462	497	3.7	100	0	12.8	Not successful	–
G6	CW	806	840	4.9	0	22.9	18.0	Successful	1

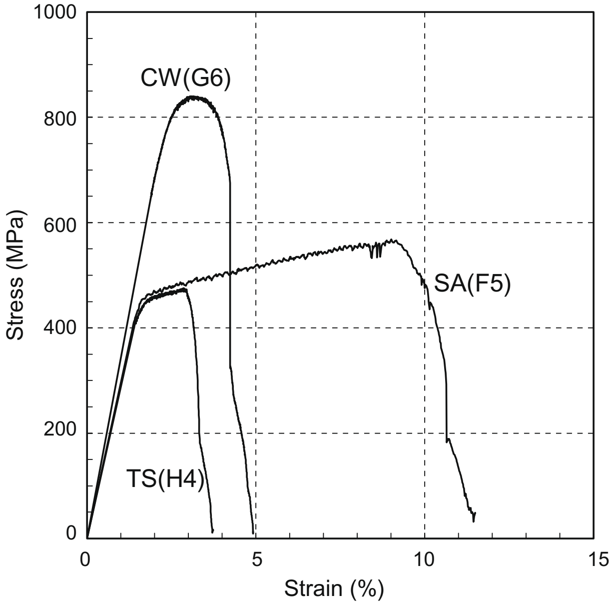

Fig. 1. Stress–strain curves from SSRT for SA, TS and CW materials irradiated to a neutron fluence of 1×10^{25} n/m^2.

In Fig. 4, the fraction of IGSCC and TGSCC is plotted as a function of yield stress using literature data [2,5–7]. The fraction of IGSCC and TGSCC in the SA and TS materials increases with yield stress while that of the CW materials decreases at high yield stress (>750 MPa). Hide et al. [6] performed side surface observation and suggested deformation characteristics of the irradiated specimens after SSRT. It was difficult for the irradiated CW material to deform homogeneously through the gage section because of its high hardness. Since the true strain rate at a localized deformation region increased, the time for deformation was not sufficient to propagate IGSCC. Consequently, TGSCC or ductile fracture was induced and the fraction of IGSCC to TGSCC decreased at high yield stress.

5. Conclusions

To examine IASCC initiation and growth processes in SSs, in-situ observation during SSRT of type 304 SS irradiated to a fluence of 1.0×10^{25} n/m^2 ($E > 1$ MeV) was conducted in oxygenated high purity water at 561 K. The material was subjected to SA, TS or CW. Subsequently, SEM examination of the fracture surface was performed. The following conclusions were drawn from the this study;

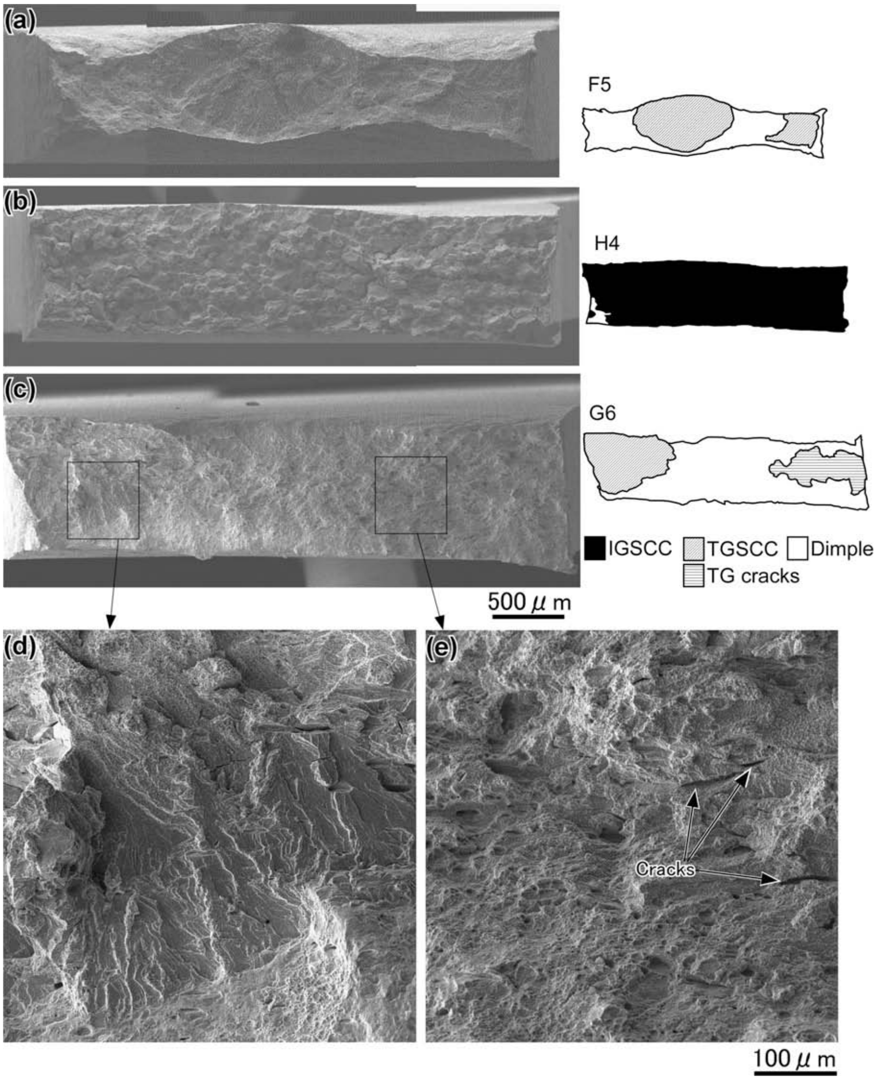

Fig. 2. SEM images and schematic illustration of the specimens, (a) SA, (b) TS, (c) CW material, (d) TGSCC of and (e) TG cracks of CW material.

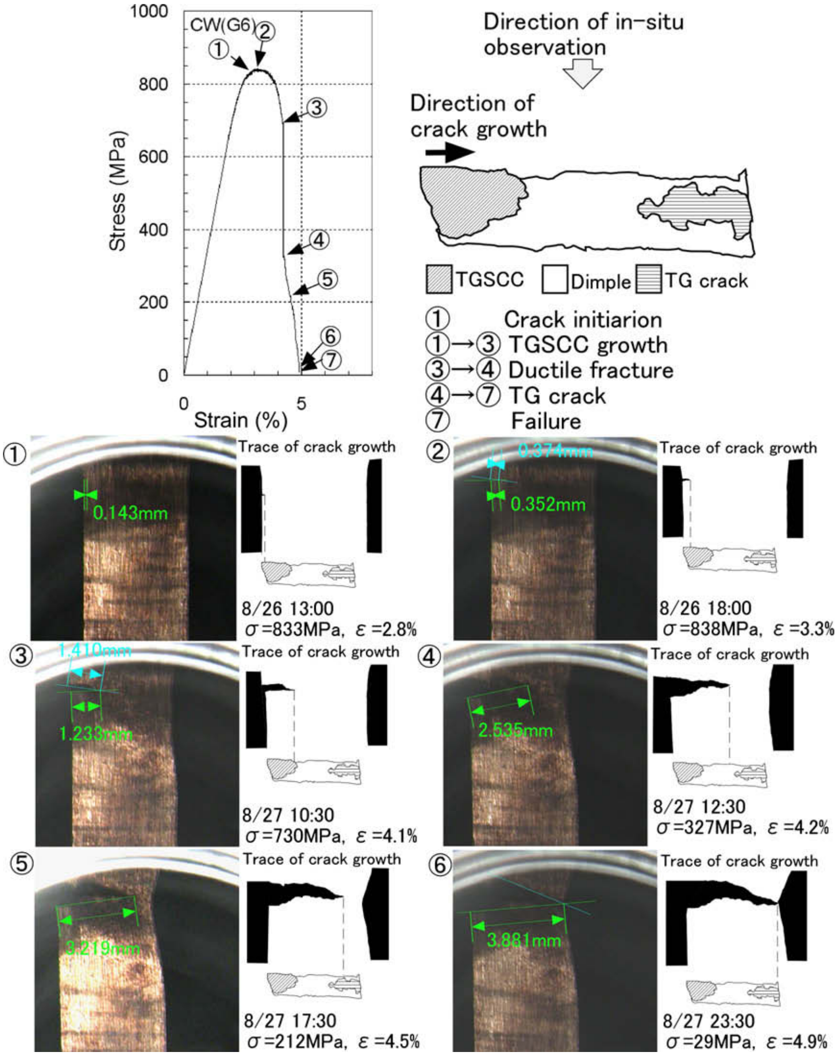

Fig. 3. Crack initiation and growth behaviors based on the stress–strain curve, fracture surfaces and the images of in-situ observation during SSRT for the CW material.

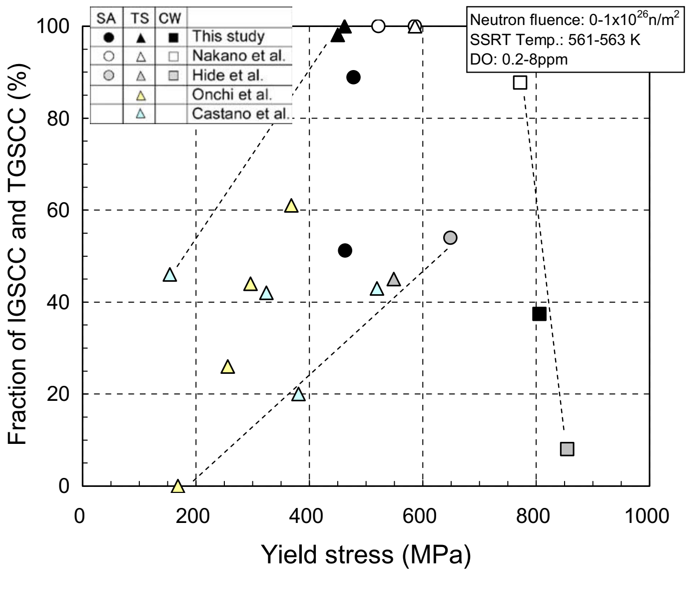

Fig. 4. Fraction of IGSCC and TGSCC as a function of yield stress [2,5–7].

1. The SA material exhibited a combination of TGSCC and ductile fracture, and almost all IGSCCs were observed in the TS material. TGSCCs, a dimple pattern and TG cracking were observed in the CW materials.
2. In the CW material, cracking was introduced before the maximum stress was reached and the fracture mode changed from TGSCC to ductile fracture to TG cracking together with the progress of crack growth in one direction.

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