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High-temperature and high-pressure *in situ* SCC device for synchrotron radiation diffraction experiments and application using an austenitic stainless steel

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Suppressing the stress corrosion cracking (SCC) by reducing the carbon content in austenitic stainless steels is apparently not effective on core shrouds used in boiling water reactors in Japan: trans-granular cracking was found in the shrouds. To clarify the mechanism of the cracking, *in situ* stress measurements on specimens under stretched conditions in hot water have been attempted in the present study. An *in situ* device for diffraction measurements at synchrotron radiation facilities has been developed, and *in situ* experiments have been carried out at SPring-8. The SUS316L steel specimen was solution heat-treated, surface-ground and then placed in the *in situ* device. Sapphire windows were used for the light path in the device. A sufficient diffracted beam intensity was obtained through two sapphire windows and water. The side-inclination method was used for measuring the stress exerted on the specimen. A 2θ–$\sin^2\psi$ plot showed that a tensile stress was induced. The measured stress value is considered to be the summation of stresses owing to pre-straining, *in situ* loading and residual stress owing to surface grinding.

Keywords: *in situ* experiment; SCC; SUS316L; stress measurement; SPring-8.

1. Introduction

It has been reported that stress corrosion cracking (SCC) can be suppressed in austenitic stainless steels by decreasing the carbon content, because chromium-depleted zones owing to carbide formation are not formed. However, many cracks have recently been found in the core shrouds used in an in-service boiling water reactor (BWR) made from low-carbon austenitic stainless steel, SUS316L (Yamamoto, 2003). The cracks were initiated in the grain interiors and propagated in a transgranular mode (Yamamoto, 2003), and cracks were also formed in such regions where the hardness was higher than in other regions, which is attributed to the hand-grinder finishing (Ino, 2003). Chromium carbides at the grain boundaries were not observed in these regions. These facts imply that the conventional SCC mechanism could not be applied to the SCC in SUS316L steel.

It should be mentioned that the reproduction of cracking is important in order to interpret the mechanism of SCC in the core shrouds, *i.e.* the phenomenon that occurred in specimens suffering from high-temperature and high-pressure water conditions have to be clarified. An *in situ* experimental device for use at synchrotron radiation facilities has been developed by the authors in order to investigate the SCC in SUS316L steel. Details of the device and preliminary results are presented here.

2. Experimental procedures

Stress measurements corresponding to changes in the microstructures are suitable for clarifying the SCC mechanism in the core shrouds, because residual stress owing to surface grinding plays an important role in crack formation. However, the phenomena occur under BWR conditions, in water at a temperature of 561 K and pressure of 8 MPa. The high energy and high brilliance of synchrotron radiation can provide an effective beam for measuring the stress of specimens under such conditions.

Schematic illustrations of the specimen chamber for the *in situ* SCC device (Toshin Kogyo) and placement of the chamber on a gonio stage in the synchrotron radiation facility are shown in Figs. 1(*a*) and 1(*b*), respectively. The size of the chamber was ~100 mm long, 50 mm wide and 50 mm high. Windows of about 15 mm in diameter and 10 mm thickness made of sapphire glass were used for a beam path. Other parts of the chamber were made of SUS316 steel. The shape and

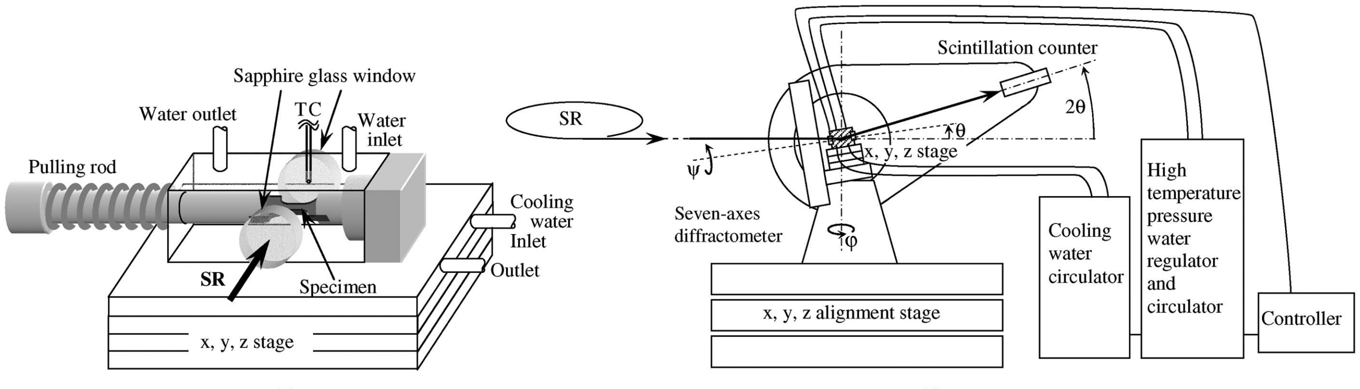

Figure 1
Schematic illustrations of (*a*) the high-temperature and high-pressure *in situ* SCC device and (*b*) its placement on a gonio stage in a synchrotron radiation facility.

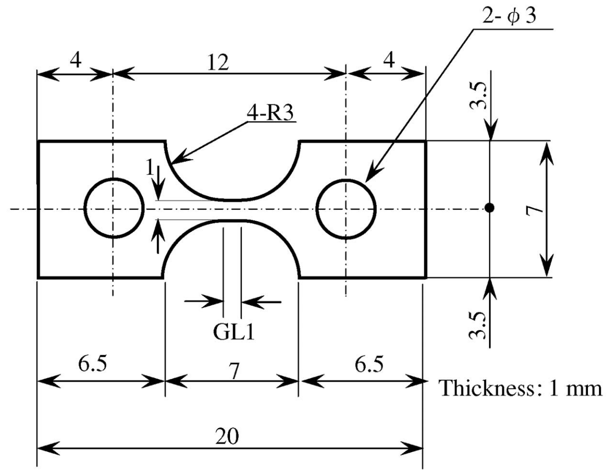

Figure 2
Shape and dimensions of the specimens for the *in situ* experiments.

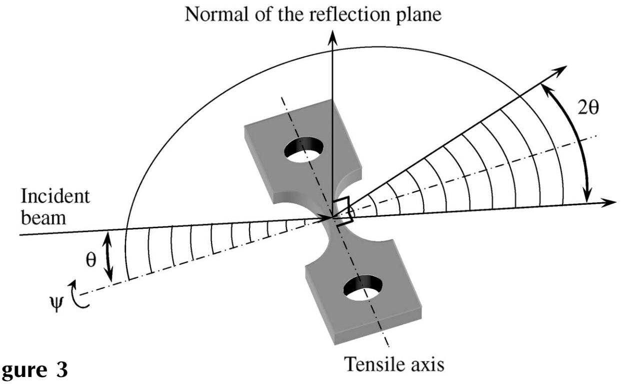

Figure 3
Geometry for stress measurements.

dimensions of the specimen are shown in Fig. 2. The gauge length of the specimen was 1 mm, and the size of the cross section in the gauge part was 1 mm wide and 1 mm thick. The specimen was horizontally placed in the chamber as shown in Fig. 1(*a*). The geometry for the stress measurements is shown in Fig. 3; the axis of inclination (ψ) is perpendicular to the tensile axis. The specimen chamber enables measurement of the diffracted beam in the range 0–15°. One end of the specimen was placed in a fixed chuck and the other end was fixed on the pulling rod. When the chamber is filled with high-pressure water, the pulling rod is pushed out in order to pull the specimen. The pulling force induced by the water at a temperature of 561 K and pressure 8 MPa is about 240 N. An additional force of ∼120 N can be applied to the specimen using a spring. A view of the entire specimen chamber is shown in Fig. 4(*a*), and the specimen observed through the sapphire windows is shown in Fig. 4(*b*).

The specimens were prepared from a low-carbon austenitic stainless steel, SUS316L, the chemical composition of which is listed in Table 1. The specimens were solution heat-treated at 1223 K for 900 s, then quenched in ice water. The surface of the specimen was mechanically polished using #2000 emery paper, and then one of the surfaces was ground using a grinding disc with a #100 grit size. The grinding direction was parallel to the longitudinal direction, the tensile direction, of the specimen. The specimen was pre-strained by slow strain-rate testing ($1\ \mu\text{m min}^{-1}$) in hot water under the same conditions mentioned above to up to 80% of the endurance limit in strain, and then provided for the *in situ* experiment.

The *in situ* SCC experiment was carried out on beamline BL02B1 at the synchrotron radiation facility SPring-8, at a beam energy of 72 keV. The specimen chamber, set on the gonio stage in BL02B1, is shown in Fig. 5(*a*), and the control units together with the chamber are shown in Fig. 5(*b*).

Stresses in the specimens were measured based on the 2θ–$\sin^2\psi$ method (Macherauch & Müller, 1961) by the side-inclination technique on the diffraction of the {113} planes in the SUS316L steel, where θ and ψ are the diffraction angle and side-inclination angle, respectively. The diffraction peaks were measured in the 2θ angle range from 8.95 to 9.2° in steps of 0.01°. The measuring time for each step was 40 s. The initial angle between the incident beam and the specimen surface was 4.51°. The side-inclination angles, ψ, were in the range 0–56.8°, resulting in 0–0.7 for $\sin^2\psi$.

The geometry of the beam alignment is shown in Fig. 6. The vertical and horizontal sizes of the beam were about 0.2 and 1.0 mm, respectively. The specimen surface was firstly irradiated with a half-width beam in the horizontal direction as

Table 1
Chemical composition of the specimen (mass %).

C	Si	Mn	P	S	Ni	Cr	Mo	N
0.011	0.89	1.05	0.024	0.006	12.09	17.60	2.03	0.026

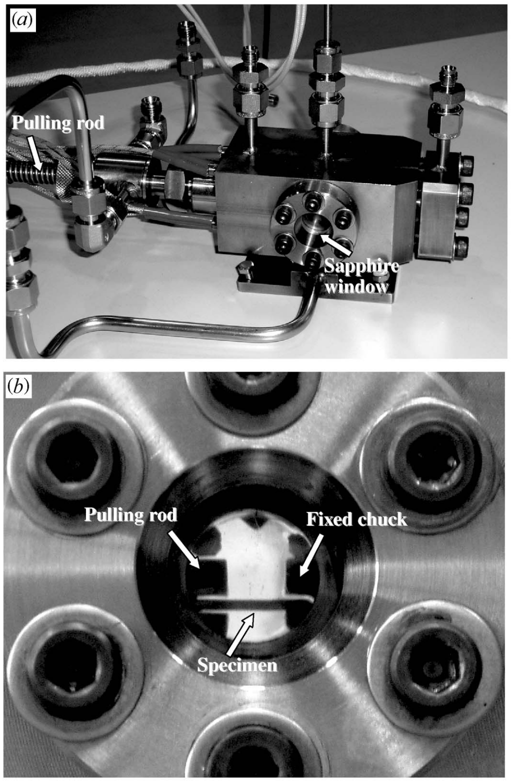

Figure 4
(*a*) View of the whole specimen chamber and (*b*) the specimen in the chamber.

shown in Fig. 6(*a*), then the specimen was raised by 0.1 mm. The initial angle between the incident beam and the specimen surface was 4.51°, so the maximum depth of the irradiated volume was about 0.28 mm, as shown in Fig. 6(*b*). However, double slits of vertical width 0.2 mm were used for collimating the diffracted beam, so the maximum depth of the gauge volume was about 0.24 mm.

The intensity of the direct beam through two sapphire windows and water with and without the specimen was about 6.8 and 9.8 kcounts s^{-1}, respectively, without the double slits, while the diffracted beam intensity for the $\{113\}_\gamma$ planes through the sapphire windows and water was about 50 counts s^{-1} at angle $\psi = 0°$.

3. Results and discussion

Results of the *in situ* measurements of the 113_γ diffraction peak in hot and high-pressure water are shown in Fig. 7, in which the solid circles and curves show the measured data and the fitted results using a Lorentzian curve, respectively. The peak intensities varied depending on the inclination angles in the range ~1500–2500 counts s^{-1}, which is considered to be

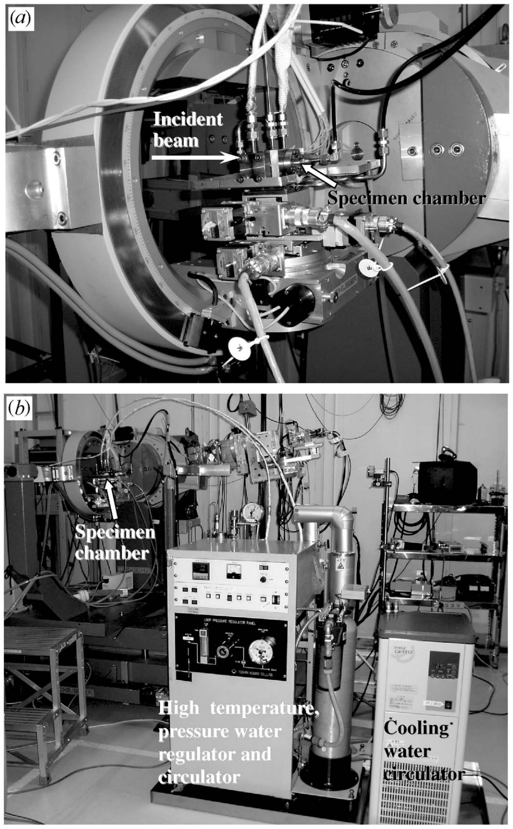

Figure 5
(*a*) Specimen chamber on the gonio stage of BL02B1 in SPring-8 and (*b*) set-up of the chamber and other instruments at BL02B1.

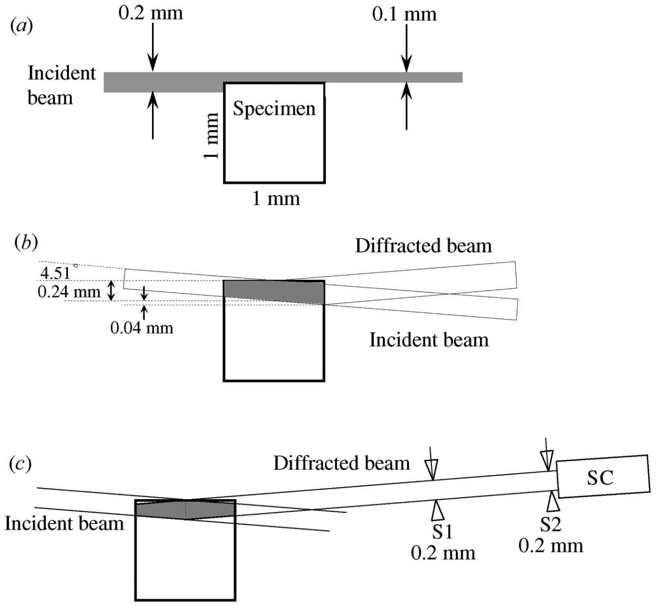

Figure 6
Geometry of beam alignment for stress measurements of specimens. (*a*) The specimen surface is irradiated with half of the incident beam. (*b*) The θ–2θ condition for {113} planes in SUS316L steel after the specimens are raised by 0.2 mm. (*c*) The diffracted beam is collimated with double slits of width 0.2 mm. Dark areas in (*b*) and (*c*) show gauge volumes contributing to diffraction intensity. SC = scintillation counter.

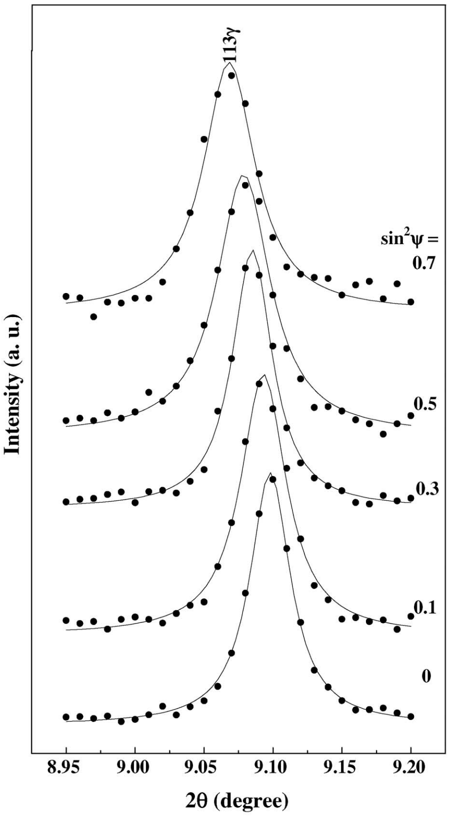

Figure 7
Diffraction peaks for 113_γ planes measured with $\sin^2\psi$ in the range 0–0.7 using the high-temperature and high-pressure *in situ* SCC experiment.

due to the texture of the specimen, and therefore the intensity was normalized in Fig. 7. Peaks of the 113_γ diffraction shifted to lower angle with increasing inclination angle, ψ, meaning that a tensile stress was exerted on the specimen.

A plot of the peak angles, 2θ, *versus* the values of $\sin^2\psi$ is shown in Fig. 8. The slope of the fitted line is about −0.041. By using the slope value, Young's modulus of 197 GPa and Poisson's ratio of 0.29 (Shankar *et al.*, 2001), the stress in the specimen was calculated to be about 690 MPa. In this case, the spring for adding an additional force was not used, so the applied load on the specimen was 240 N. The cross section of the gauge part of the specimen was 1 mm^2, and the applied stress was 240 MPa. Subtracting the applied stress from the measured value produces a 450 MPa stress. In a previous paper (Yamamoto *et al.*, 2004), the residual stress in a specimen of SUS316L steel induced by surface grinding using a disc of #30 grit size was reported to be about 430 MPa in the grinding direction. Since a grinding disc with a finer grit size (#100) was used in the present study, the residual stress owing to surface grinding is considered to be less than that in the previous study. It should be mentioned that the specimen was pre-strained by the slow-strain-rate testing and plastically deformed with slip traces and micro-cracks being observed in the specimen. Although the qualitative value of the stress owing to the pre-straining was not estimated, the measured value of the stress, 690 MPa, would be the summation of the residual stress owing to surface grinding, pre-straining and the applied stress. The deformed layer formed by surface grinding is considered to be about 10–20 µm in depth, while the gauge volume in the present study was about 240 µm in depth. The stress in the surface layer would be higher than the measured value.

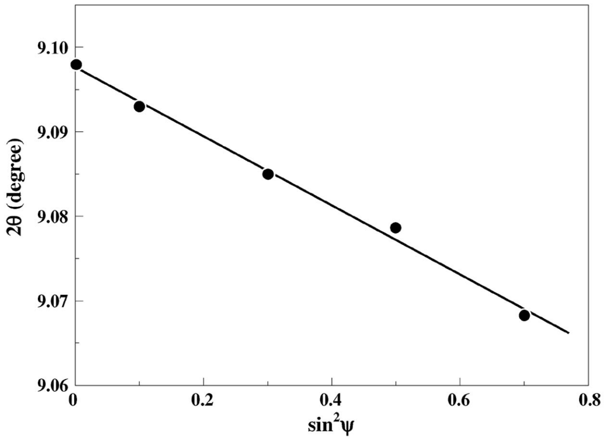

Figure 8
2θ–$\sin^2\psi$ plot obtained by the *in situ* high-temperature and high-pressure SCC experiment.

It is well known that cold working has deleterious effects on the SCC behaviour (Muraleedharan *et al.*, 1985; Gigada *et al.*, 1982). Muraleedharan *et al.* (1985) reported that the total time to fracture in the SCC tests on SUS304 and 316 steels decreased with increasing cold work. For low-carbon austenitic stainless steels, SUS304L and 316L, the same results were reported by Gigada *et al.* (1982). Also, the higher the applied stress, the shorter the time to failure (Muraleedharan *et al.*, 1985). Cold workings in their studies were applied by tensile deformation (Muraleedharan *et al.*, 1985), and by cold-rolling or cold-drawing (Gigada *et al.*, 1982). Although they did not refer to the senses of residual stress, compression or tension, deformation deteriorates the SCC properties. In the present study, it has been clarified that surface grinding causes residual stress in tension, so the surface-ground portions suffered from higher deformation and higher tensile stress compared with the other unground portions. This is considered to be a reason for the crack nucleation in the core shrouds.

4. Summary

A device for high-temperature and high-pressure *in situ* SCC experiments has been developed for use with synchrotron radiation diffraction measurements. A load of 240 N can be applied to specimens when the specimen chamber is filled with hot water at a temperature of 561 K and pressure 8 MPa. In order to clarify the mechanism of the SCC phenomenon that occurred in the SUS316L steel core shrouds, a preliminary experiment has been successfully carried out using a device in which stress exerted in the specimen can be measured. This device enables measurement of the stress by the side-inclination method with inclination angles, ψ, in the range 0–56.8°. From a 2θ–$\sin^2\psi$ plot, the stress was calculated as ~690 MPa, which is considered to be the total of the stresses owing to prestraining, *in situ* loading and the residual stress induced by surface grinding.

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References

Gigada, A., Mazza, B., Pedeerri, P., Salvago, G., Sinigaglia, D. & Zanni, G. (1982). *Corr. Sci.* **22**, 559–578.
Ino, H. (2003). *Mater. Sci. Technol.* **73**, 1094–1104.
Macherauch, E. & Müller, P. (1961). *Z. Angew. Phys.* **13**, 305–312.
Muraleedharan, P., Khatak, H. S., Gnanamoorthy, J. B. & Rodriguez, P. (1985). *Metall. Trans. A*, **16**, 285–289.
Shankar, P., Palanichamy, P., Jayakumar, T., Raj, B. & Ranbanathan, S. (2001). *Metall. Mater. Trans. A*, **32**, 2959–2968.
Yamamoto, A., Yamada, T., Nakahigashi, S., Liu, L., Terasawa, M. & Tsubakino, H. (2004). *ISIJ Int.* **44**, 1780–1782.
Yamamoto, M. (2003). *Mater. Sci. Technol.* **73**, 731–734.