Transmission Electron Microscopy Characterization of PWSCC Cracks in Alloy 600

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1. Introduction

Primary water stress corrosion cracking (PWSCC) in reactor pressure vessel head penetration nozzles, their welded parts, and steam generator tubes at pressurized water reactors have been found in many countries [1]. Several models have been reported for the PWSCC phenomena [2,3], however, the exact failure mechanisms have not been fully understood up to now. In the present study, PWSCC cracking properties of Alloy 600 used as the CRDM nozzle material were investigated through microscopic equipments. Especially, the microstructural and chemical changes around a crack tip during PWSCC were studied using TEM specimens fabricated by a focused ion beam (FIB) method.

2. Methods and Results

2.1 PWSCC experiment

In the test, a 1/2 CT (compact tension) specimen was used, and its dimensions are shown in Fig. 1. Before the PWSCC test, the CT specimen was fatigue pre-cracked in a length of about 2 mm in the air. The PWSCC test was conducted under the simulated primary water environmental conditions, that is, 1200 ppm B + 2 ppm Li containing pure water at 340 ℃, dissolved oxygen contents below 5 ppb, hydrogen partial pressure of 14.3 psi, and an internal pressure of 2300 psi. The maximum stress intensity factor at a crack tip was maintained at 30 MPa√ m. Major experimental parameters such as the temperature, load, displacement, pH, electric conductivity, ECP and D.O. were being monitored and collected by PC through an A/D converter.

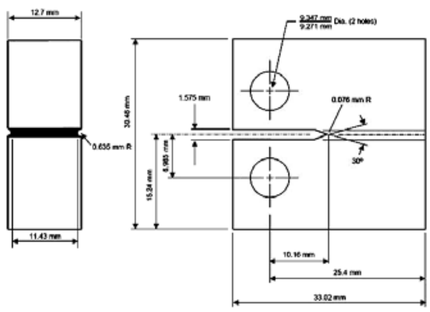

Fig. 1. Dimensions of a 1/2 CT specimen.

2.2 Cracking morphologies

After the PWSCC test, the cracked CT specimen was sliced into two pieces, one for the observation of the fractured surface in the cross section, and the other for the OM, SEM/EBSD and TEM examinations in the plane section. Fig. 2 shows the OM image around the crack tips. From the SEM/EBSD analysis, it was found that the cracks propagated along the random high angle grain boundaries, which have a higher energy than the low angle and/or CSL special grain boundaries do.

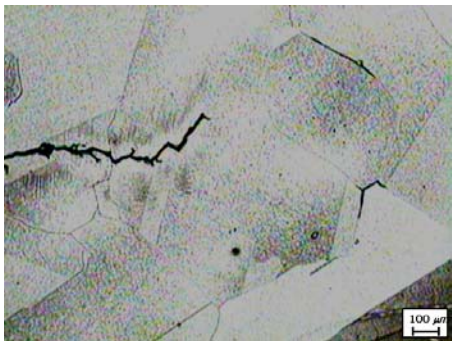

Fig. 2 OM image around the crack tips of the CT specimen after the PWSCC test.

Fig. 3 shows an overall fracture surface of the Alloy 600. In the figure, the pre-fatigue and the PWSCC areas could be identified. The fracture morphology of the Alloy 600 showed that the cracks grew along the grain boundaries. Therefore, it is confirmed that the cracking mode was completely intergranular in this case. The predominant failure mode of Alloy 600 is well known to be intergranular in the primary and secondary water environments [4].

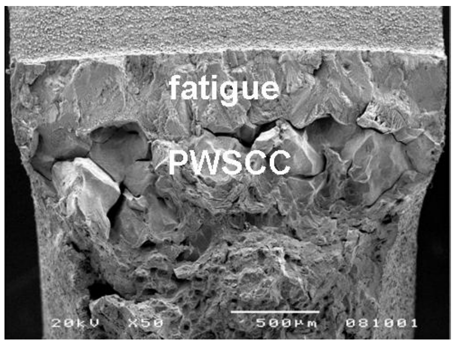

Fig. 3 Fracture surface of the CT specimen

2.3 FIB TEM results

A TEM bright field image around a crack tip of the Alloy 600 after the PWSCC test is shown in Fid. 4. In the figure, the white region was identified as the cracking region. A strong oxygen peak was detected by TEM/EDS, and some types of oxides were filled inside the region. It is also clearly seen in the figure that a crack propagated along a grain boundary.

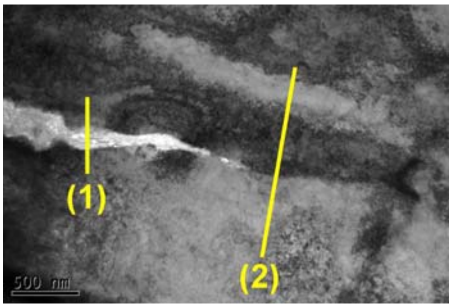

Fig. 4 TEM bright field image around a crack tip of Alloy 600.

The compositional changes of Ni, Cr, Fe, Ni and O across the grain boundary containing the cracking region, denoted as (1) in Fig. 4, are shown in Fig. 5. From the graph, it can be expected that the cracking region has two oxide layers. In the layer close to the bare metal, the oxides have high Cr and low Ni contents, which means that the layer consists mainly of the chromium oxide. On the contrary for the layer far from the bare metal (i.e., outer layer), the major metallic element of the oxide is Ni, which means that the main oxide is nickel oxide in this case.

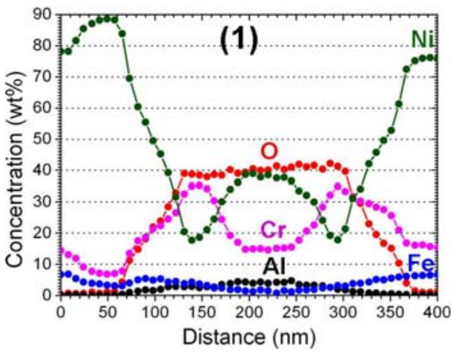

Fig. 5 Compositional changes across the grain boundary (1) in Fig. 4.

The compositional changes of Ni, Cr, Fe and O across the grain boundary several tens of nm away from the crack tip, denoted as (2) in Fig. 4., are shown in Fig. 6. Oxygen was detected around the grain boundary. Since the bare metal had no oxygen content in the beginning, it is reasonable to assume that the oxygen diffused into the grain boundary/matrix from the environment during the test. This experimental result strongly supports that the internal oxidation model [2] is valid to some extent as the PWSCC failure mechanism.

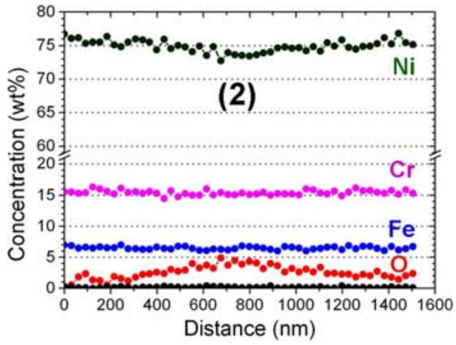

Fig. 6 Compositional changes across the grain boundary (2) in Fig. 4.

3. Conclusions

The cracking mode of Alloy 600 in a simulated primary water environment was completely intergranular, and the cracks were propagated along the random high angle grain boundaries. The cracking region around the crack tip consisted of two oxide layers, i.e., chromium-rich and nickel-rich oxides. In the base metal away from the crack tip, oxygen was detected. The existence of oxygen in the bare metal supports the fact that the internal oxidation model is valid to some extent as a PWSCC mechanism.

REFERENCES

[1] E. S. Hunt and D. J. Gross, PWSCC of Alloy 600 Materials in PWR Primary System Penetrations, TR-103696, Electric Power Research Institute, Palo Alto, Calif., 1994.
[2] P.M. Scott, Proc. 9th International Conf. on Environmental Degradation of Materials in Nuclear Power System – Water Reactors, NACE/TMS, Warrendale, Penn., 1999, p. 3
[3] P. M. Scott and P. Combrade, Proc. 11th International Conf. on Environmental Degradation of Materials in Nuclear Power System – Water Reactors, NACE/TMS, Stevenson, WA, 2003, p. 29
[4] G.S. Was, Corrosion, 1990, 46, 319.