## **PWSCC Crack Tip Observation of Alloy 600**

Yun Soo Lim<sup>\*</sup>, Hong Pyo Kim, Hai Dong Cho, Han Hee Lee Nuclear Materials Research Division, Korea Atomic Energy Research Institute 1045 Daeduk-daero, Yuseong, Daejeon, 305-353, Korea \*Corresponding author: yslim@kaeri.re.kr

## 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 characterized by microscopic equipments. 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 325 °C, 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 $\sqrt{}$  m. Major experimental parameters such as the temperature, load, displacement, pH, electric conductivity, ECP and dissolved oxygen concentration were being monitored and collected by PC through an A/D converter.



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

#### 2.2 Characterization of cracks

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/EDS examinations in the plane section. Fig. 2 shows an SEM/EBSD image of advancing cracks. From the results, it was found that the cracks propagated along the random high angle grain boundaries, which had misorientation angles over 15 degrees between the adjacent grains without any coincidence site lattice (CSL) specialty. It is well known that the random high angle grain boundaries do. FIB TEM specimens for observation of crack tips were taken from the circled regions in the figure.



Fig. 2 SEM/EBSD result on the PWSCC cracks of Alloy 600

# 2.3 Identification of corrosion products inside the cracks

A bright field image of a crack tip taken from an FIB TEM specimen is shown in Fig. 3. In the figure, the white region was identified as a crack. The crack propagated along a grain boundary, and finally stopped in front of an intergranular Cr carbide ( $Cr_7C_3$ ). Coarse intergranular Cr carbides, as shown in the figure, were significantly precipitated on the grain boundaries in this alloy due to a heat treatment at high temperature (930 °C) for a long time (3 hours). From the above

result, it is believed that intergranular Cr carbides have an blocking effect on the crack propagation.



Fig. 3 TEM bright field image of a PWSCC crack in Alloy 600

Various kinds of corrosion products were found inside a crack, and all of them were identified as oxides. Fig. 4 shows some corrosion products in a crack and their related selected area diffraction pattern (SADP). From SADP analysis, the coarse and faceted particles shown in Fig. 4 were identified as spinels with a lattice constant of 8.33 Å. They had Cr, Fe and Ni as major metallic components. The ring patterns in the inset of Fig. 4 were originated from an fcc structure with a lattice constant of 4.18 Å, and the major elements of chemical composition were Ni and O. From the above facts, it was confirmed that the ring patterns were originated from NiO. Nickel oxides had a needle-like shape. Two kinds of oxides, spinels and NiO, existed on the outer layer from a crack wall. On the inner layer, on the other hand, there were Cr<sub>2</sub>O<sub>3</sub> type oxides.



Fig. 4 TEM bright field image of corrosion products and the related selected area diffraction pattern

Fig. 5 shows a high resolution image of oxides in a PWSCC crack. Amorphous with high oxygen content was also found adjacent to a crack wall, as shown in the figure. The lattice images of spinel structure are clearly seen.



Fig. 5 High resolution image of oxides in a PWSCC crack of Alloy 600

#### **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., inner and outer layers. In the outer layer, NiO and spinels were found. In the inner layer, on the other hand,  $Cr_2O_3$  existed. Amorphous with high oxygen content was also found adjacent to a crack wall.

### REFERENCES

 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.
P.M. Scott, Proc. 9<sup>th</sup> 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. 11<sup>th</sup> International Conf. on Environmental Degradation of Materials in Nuclear Power System – Water Reactors, NACE/TMS, Stevenson, WA, 2003, p. 29