The Effects of Dissolved Hydrogen on PWSCC Initiation Behavior and Oxide Characteristics of Alloy 182 Weld

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1. Introduction

There have been a number of incidents of cracking caused by PWSCC in Alloy 82/182 dissimilar metal welds (DMW). Previous studies indicated that the susceptibility of PWSCC is closely related to the oxide characteristics which is dependent on water-chemistry conditions, especially dissolved hydrogen (DH). In this regard, it has been one of the key areas of active research in many countries, and many investigations have been performed by ex-situ and in-situ analysis [1-3]. To provide an important linkage between behaviors of oxide formation and crack initiation, the results of crack initiation test using Alloy 182 specimen and oxide analysis under various DH condition are described. In addition, to investigate the effect of stress, we evaluated characteristics of oxides formed under loaded condition.

2. Experimental

2.1 Test material

The material used in this study was manufactured from a weld deposit of Alloy 182 on a 316L plate with the dimension of T-direction (Transverse) 300mm, L-direction (Longitudinal) 500mm, and S-direction (Thickness) 55mm (Alloy 182 25mm + 316L SS 30mm) The chemical compositions of the test material analyzed by inductively coupled plasma (ICP) method are listed in **Table I**, and the tensile properties are summarized in **Table II**. They meet the requirements of ASME SFA-5.11 [4].

Table I: Chemical composition of Alloy 182 weld [5]

C	Si	Mn	P	S	Fe
0.03	0.34	7.35	0.009	0.001	2.81
Cu	Ni	Ti	Cr	Mo	Cb+Ta
0.01	73.0	0.02	14.2	0.63	1.57

Table II: Mechanical properties of Alloy 182 weld [5]

Alloy 182	RT			325°C		
	YS (MPa)	UTS (MPa)	Elong. (%)	YS (MPa)	UTS (MPa)	Elong. (%)
Upper	369	604	39	320	541	37
Lower	396	623	37	372	560	36

2.2 Test system and conditions

The test environment was simulated PWR water containing dissolved boric acid and lithium hydroxide at 310 °C. The levels of DO, DH, conductivity and pH are monitored and controlled at room temperature. The DO was reduced below 5 ppb, and two dissolved hydrogen (DH) contents, 5cc/kg (low DH), 30cc/kg (close to current operation level) were used. The details of the test conditions are summarized in **Table III**.

Table III: Test environment

Environment		PWR Primary Environment
Temp. & Press.		325 °C, 15.5 MPa
Water chemistry	DO	<5ppb
	DH	5, 30 cc/kg
	Conductivity	~ 20~25 µ S/cm
	pH	6~7

The specimens for oxide analysis were ground using emery papers up to 4000 grit, and U-bend specimens were grounded and polished by 1-µm diamond paste. For crack initiation test, U-bend specimens were fabricated with T-L orientation which is perpendicular to the dendrite main axis. U-bend specimens were either pre-formed to 3% deformation by bending them in one direction, flattening them, and then bending them to 11.3% and 19.2 % in the reverse direction using mandrel. To prevent the relaxation of stress in high temperature, the stress on the U-bend specimen is maintained by spring, as shown in **Fig. 1**. And 10 mm diameter coupon-type specimens were used for ex-situ oxide analysis. All of specimens were exposed to test environment for 24, 100, 500 and 1,000 hours.

Fig. 1. Spring-loaded U-bend specimen for PWSCC initiation test

3. Results and Discussion

3.1 DH effect on PWSCC initiation property

Table IV shows the current status and results of PWSCC initiation tests. From 500 hours exposure, some of cracked specimen were observed in PWR water with 30cc/kg of DH. Finally, cracks are found on the all of specimens exposed to PWR water with 30cc/kg of DH. Meanwhile, it is reasonable that high deflection specimens show higher susceptibility than low deflection specimens and single-bent specimens. The length of some cracks reached hundreds μm on the surface of specimen. More details of the crack size and morphology will be described at the conference.

Table IV: Results of PWSCC initiation test

Environment	*Low DH water (5cc/kg)*		*PWR water (30cc/kg)*		
Deflection level	High	Low	High	Low	Single–bent **
Exposure Times (hrs)	**Cracked specimen ratio (# of cracked specimen / # of exposed specimen)**				
24 hrs	0/4	0/4	0/4	0/4	0/4
100 hrs	0/4	0/4	0/4	0/4	0/4
500 hrs	-	-	3/4	1/4	0/4
1000 hrs	-	-	4/4	4/4	-
1500 hrs	-	-	-	-	2/3

* High deflection ~ 19.2 %, Low deflection ~ 11.3% (calculated by the equation on ASTM G30 [6])
** Single-bent ~ 12% (only in one direction)

3.2 DH effect on oxide film structure

Needle-like oxide (NiO) and Cr-free crystallites ($NiFe_2O_4$) were observed in PWR water (DH : 30cc/kg) and its size was increased with the increase of exposure time. P. Combrade et.al. reported that the external layer's composition for Ni-alloys strongly depend on the nature and surface condition of the base metal and it is generally made of $(Ni_xFe_{1-x})Fe_2O_4$ ferrite [1].

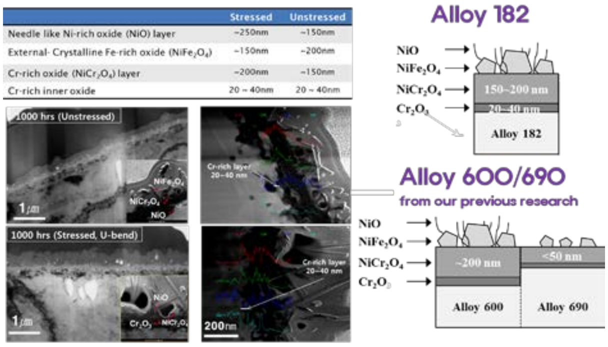

Fig. 2. Oxide analysis of Alloy 182 exposed to PWR water (DH : 30cc/kg)

In addition, internal Cr-rich oxide ($NiCr_2O_4$) layer was observed under needle-like and crystallites oxide layer. It is reported that the Cr-rich inner layer comprises two "sub-layers" [1,2]. It was analysed that on the metal side the sub-layer is made of Cr_2O_3 (or $(Cr,Fe)_2O_3$) oxide. On the solution side, the sub-layer is Cr rich spinel type oxide. These behaviors are consistent with the previous results [1,2]. Analysis of oxides formed at other condition is on-going and the results will be available at the conference. Meanwhile, Ni-rich and Cr-rich oxide thickness of stressed specimen is slightly thicker than that of unstressed specimen, as shown in **Fig. 2**. In summary, Alloy 182 exposed to PWR water has multi-layered oxide structure which is needle-like Ni-rich oxide layer and external Fe-rich crystalline oxide on the Cr-rich oxide layers regardless of applied stress, as shown in **Fig. 2**.

3. Conclusions

The results of crack initiation test using Alloy 182 specimen and oxide analysis under various DH condition are described. Under loaded condition, the thickness of Ni-rich and Cr-rich oxide layer were slightly thick, but oxide structure was multi-layered regardless of applied stress. Still, no clear tendency between stress and oxide structures was observed. As additional tests and analyses are underway, such results will provide more information to reveal the DH effect on crack initiation and oxide structure.

ACKNOWLEDGMENTS

This study was mainly supported by the KETEP-International Collaborative Energy Technology R&D Program (No. 20128540010010) of MOTIE of the Republic of Korea. Financial support for three of the authors was provided by the BK-Plus Program of the MSIP of the Republic of Korea.

REFERENCES

[1] P. Combrade, P.M. Scott, M. Foucault, E. Andrieu and P. Marcus, Oxidation of Ni Base Alloys in PWR Water: Oxide Layers and Associated Damage to the Base Metal, Proceedings of the 12th Int. Conf. on Environmental Degradation of Materials in Nuclear Power System -Water Reactors (2005), 883-890.
[2] M. Sennour, L. Marchetti, F. Martin, S. Perrin, R. Molins and M. Pijolat, A detailed TEM and SEM study of Ni-base alloys oxide scales formed in primary conditions of pressurized water reactor, Journal of Nuclear Materials, 402 (2-3) (2010), 147-156.
[3] L. Marchetti, S. Perrin, O. Raquet and M. Pijolat, Corrosion Mechanisms of Ni-Base Alloys in Pressurized Water Reactor Primary Conditions, Materials Science Forum 595-598 (2008), 529-537.
[4] ASME Sec II Part C - SFA-5.11/SFA-5.11M, Nickel and Nickel-Alloy Welding Electrodes for Shielded Metal Arc Welding, ASME, New York, NY, 2010.
[5] J.-D. Hong, J. Lee, C. Jang, J.H. Kim, T. Shoji, Oxide Characterization for the Mechanistic Understanding of the Initiation Behaviors of Alloy 182 Weld In Various Water Chemistry Conditions, Present at NPC 2014, October 26-31 (2014), Sapporo, Japan.
[6] ASTM G30-97, Standard Practice for Making and Using U-Bend Stress-Corrosion Test Specimens, ASTM, Philadelphia, Pennsylvania, 2009.