Transactions of the Korean Nuclear Society Spring Meeting
Jeju, Korea, May 23-24, 2019

Electrochemical Potentiodynamic Repassivation Behavior of Type 304L Stainless Steel

H. M. Jang [a, b*], D. J. Kim [a], J. H. Lee [b] H. P. Kim [a],
[a]*Nuclear Materials Research Division, Korea Atomic Energy Research Institute (KAERI)*
[b]*Department of Advanced Materials Engineering, Chungnam National University*

1. Introduction

Stainless steel may be susceptible to intergranular corrosion (IGC) and stress corrosion cracking in harsh environments even though it shows generally good resistance to corrosion in most environments. Extensive researches on IGC of stainless steel have been performed to understand IGC mechanism and to predict IGC rate in nuclear industry. Stainless steel can be sensitized during welding process or during exposure to high neutron dose at high temperature. Most of works on IGC were performed for type 304 stainless steel containing only austenite phase. But type 304 stainless steel used for baffle of reactor vessel in OPR and APR contains delta ferrite as well as austenite phase. The literature review shows that the delta ferrite is intentionally added to austenite to prevent hot cracking during hot working. A small amount of d-ferrite ranging from 1% to 6% in austenitic stainless steel is effective in preventing hot cracking [1-4]. The presence of delta phase may change sensitization kinetics. In this study, IGC of type 304 stainless steel containing delta ferrite were evaluated by electrochemical potentiokinetic reacting (EPR) method in order to understand the grain boundary sensitization kinetics and to clarify the role of delta ferrite on IGC.

2. Experimental Methods

Chemical composition of the specimen used in this work was shown in Table 1. It is Type 304L stainless steel with carbon content of 0.024 wt. %. The sensitization heat treatment was performed using a 3 zone tube heating furnace in an Ar atmosphere. Sensitization heat treatment was performed at 500, 600, and 700°C for 1 hour to 96 hours. The degree of sensitization was evaluated by an electrochemical method.[5-6] Specimens were prepared according to ASTM and JIS standards. EPR test specimen size was 10mm x 10mm x 10mm, and one side of this specimen was welded with nickel wire and the nickel wire was encapsulated with Teflon tube. The specimen was mounted with resin except test surface. The surface of the mounted specimen was grinded and polished with alumina powder up to 1 μm. The EPR test solution was prepared with 0.5M sulfuric acid and 0.01M KSCN. I_r/I_a was obtained for sensitization evaluation. After EPR test, the surface was observed by SEM.

Table 1: Chemical composition of the specimen (wt. %).

C	Cr	Ni	Mn	Cu	P
0.024	18.35	8.11	1.43	0.28	0.033

3. Results and Discussion

3.1 Microstructure of as received specimen

Fig. 1. showed microstructure at plane perpendicular to rolling direction of the as received specimen etched oxalic acid. Microstructure was composed of austenite with elongated delta ferrite. Chromium content of the austenite phase measured with EDS was about 19 % while that of delta ferrite was about 27 %. Because of lower chromium content in austenite phase, austenite phase etched well compared to delta ferrite phase as shown in Fig. 1.

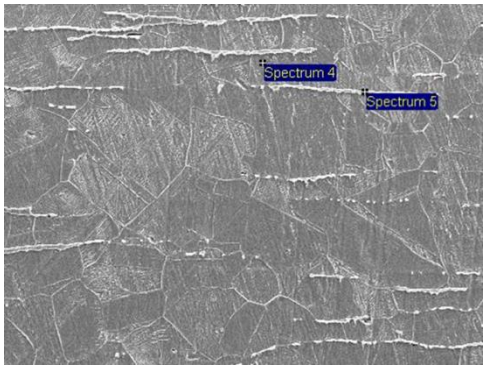

Fig. 1. Microstructure at plane perpendicular to rolling direction of the as received specimen etched oxalic acid.

Table 2 :Chemical composition measured with EDS (wt.%).

c	Si	Cr	Mn	Fe	Ni
Spectrum 4	0.88	18.92	2.25	70.03	7.92
Spectrum 5		26.55	1.87	67.09	4.47

3.2 Double loop EPR curve

Fig. 2. showed double loop EPR curves for specimens heat treated at 700°C. Activation current density was about 5.9×10^{-2} A/cm^2 for specimens heat treated at 700°C from 0 hour to 96 hours. It did not depend on heat treatment time at 700°C. Passive current density was about 1×10^{-5} A/cm^2 which is consistent with value published work[x]. Reactivation current density gradually increased with heat treatment time. Fig. 3. showed ratio of reactivation current density to activation current density (I_r/I_a) as a function of heat treatment time at 500°C, 600°C and 700°C, respectively. For specimen heat treated at 500°C and 700°C, I_r/I_a increased with

heat treatment time up to 98 hours. But for specimen heat treated at 600°C, I_r/I_a increased with heat treatment time up to 48 hours and then decreased with further heat treatment time.

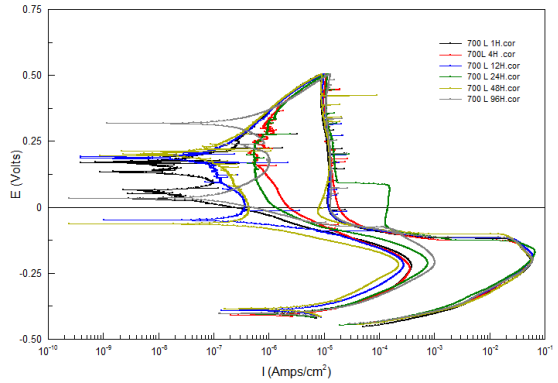

Fig. 2. Double loop EPR curves for specimens heat treated at 700°C.

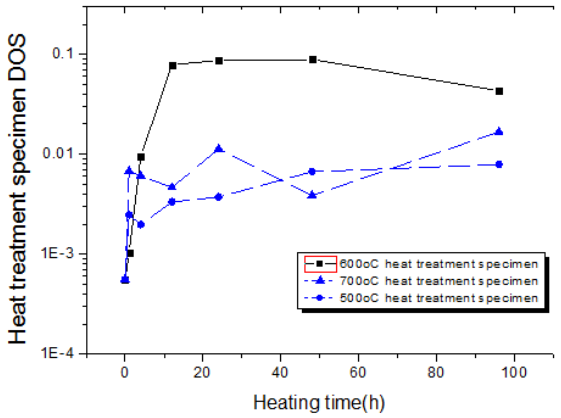

Fig. 3. Ratio of reactivation current density to activation current density (I_r/I_a) as a function of heat treatment time

3.3 Corrosion morphologies observation after EPR test

Fig. 4. showed corrosion morphologies after EPR test for specimen heat treated at 600°C. At very short heat treatment time of 1hr, both Υ-Υ grain boundary corrosion and Υ-δ phase boundary corrosion were not observed. As heat treatment time increases to 12hours, Υ-Υ grain boundary corrosion still was not observed but severe Υ-δ phase boundary corrosion was apparent. As heat treatment time increases further to 48 hours Υ-Υ grain boundary corrosion occurred slightly and severe Υ-δ phase boundary corrosion was apparent. Υ-δ phase boundary corrosion occurred at early heat treatment time compared to Υ-Υ grain boundary corrosion. Preferential phase boundary corrosion seems to be related with higher chromium diffusivity in delta ferrite phase. Delta ferrite phase is relatively open structure because BCC structure is less close packed compared to FCC.

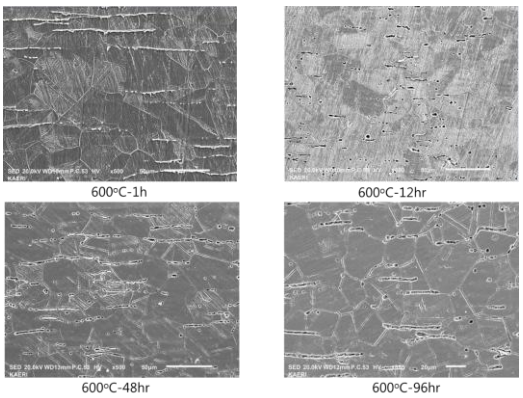

Fig. 4. Microstructure observation after EPRtest.

4. Summary

Microstructure of the specimen used in this work was composed of austenite with elongated delta ferrite. Chromium content of the austenite phase measured with EDS was about 19 % while that of delta ferrite was about 27 %. For specimen heat treated at 500°C and 700°C, I_r/I_a increased with heat treatment time up to 98hours. But for specimen heat treated at 600°C, I_r/I_a increased with heat treatment time up to 48 hours and then decreased with further heat treatment time. Υ-δ phase boundary corrosion was occurred at early time compared to Υ-Υ grain boundary corrosion. Preferential phase boundary corrosion seems to be related with higher chromium diffusivity in delta ferrite phase. Delta ferrite phase is relatively open structure because BCC structure is less close packed compared to FCC.

REFERENCES

[1] H. P. Kim, D. J. Kim, Corrosion and Protection Vol. 17 No. 1. (2018)
[2] T. Ogawa, E. Tsunetomi, Hot cracking susceptibility of austenitic stainless steels, Weld. Res. Suppl. 61 (1982) 82s-93s.
[3] J.C. Lippold, W.F. Savage, Characterization of weld solidification cracking in a duplex stainless steel weldments: Part III-the effect of solidification behavior on hot cracking susceptibility, Weld. Res. Suppl. 61 (1982) 88S-96S.
[4] C.L. Lai, L.W. Tsay, W. Kai, C. Chen, The effects of cold rolling and sensitization on hydrogen embrittlement of AISI 304L welds, Corros. Sci. 52 (2010) 1187-1193.

[5]Guanshun Bai, Shanping Lu, Dianzhong Li, Yiyi Li Corrosion Science 90 347-358 (2015)
[6]F.Ruel, P. Volovitch, L. Peguet, A.Gaugain, K. Ogle, Corrosion Vol. 69, No. 6 (2013)