

The oxidation behavior of 316L in simulated pressurized water reactor environments with cyclically changing concentrations of dissolved oxygen and hydrogen

Xiangyu Zhong^a, Shuang Xia^b, Jian Xu^{a, c, *}, Tetsuo Shoji^a

^a Frontier Research Initiative, New Industry Creation Hatchery Center, Tohoku University, 6-6-10 Aramaki Aoba, Aoba-ku, Sendai, 980-8579, Japan

^b Institute of Materials, Shanghai University, 149 Yanchang Road, Zhabei Qu, Shanghai, 200072, PR China

^c School of Materials, Sun Yat-sen University, No. 135, Xingang Xi Road, Guangzhou, 510275, PR China

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ABSTRACT

The oxidation behavior of 316L stainless steel in the simulated pressurized water reactor primary water with cyclic exposure to hydrogenated and oxygenated water conditions was investigated by contact electric resistance, electrochemical impedance spectroscopy, scanning electron microscopy, Raman spectroscopy and X-ray photoelectron spectroscopy techniques. The electrochemical resistance of the oxide film exhibits significant changes as the environment is shifted from hydrogenated water chemistry to oxygenated water chemistry and vice versa. However, in different hydrogenated periods (or oxygenated periods) with the same water chemistry, the electrochemical resistance of the oxide film is very similar. On the contrary, the oxide morphology and composition do not show obvious changes, and the oxide film thickness has a generally increasing trend. The reason for this phenomenon was also discussed.

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1. Introduction

The corrosion resistance of structural alloys used in nuclear power plants, such as stainless steels and nickel-based alloys, is mainly due to their protective oxide film formed on the surface [1–3]. The water chemistry has substantial effects on the oxide film properties such as composition, structure, and morphology [4–6]. Hydrogen has been introduced to pressurized water reactor (PWR) for reducing the stress corrosion cracking susceptibility and retarding the generation of hydrogen peroxide caused by water radiolysis [7–10].

However, according to “PWR Primary Water Chemistry Guidelines” published by Electric Power Research Institute (EPRI) [11], the addition of reactor make-up water, concentrated boric acid and/or blended boric acid via the chemical and volume control system charging line adds oxygen to the reactor coolant system. Oxygen addition can occur under a variety of circumstances and raises the questions of what parts of the primary circuit are affected by

oxygen ingress and what are the consequences of the addition. For example, 8 conditions of oxygen ingress are given in the document, including oxygen ingress during load following, oxygen ingress at power towards end-of-cycle, oxygen ingress at power, oxygen ingress during boration under hot shutdown conditions etc. Dissolved oxygen concentrations can be controlled during plant heat-up by hydrazine, hydrogen and/or venting. During power operation, the concentrations of oxygen and oxidizing species are controlled by the addition of hydrogen to the reactor coolant. Specific examples exist where loss of hydrogen control led to enhanced crud deposition. It is considered desirable to deaerated makeup water if equipment is presently installed. However, capital expenditure on new deaeration equipment is difficult to justify. More information can be obtained in Ref. [11].

Furthermore, with the motivation of prolonging the service life of the reactors, materials in the reactor have a higher likelihood of exposure to different environments. Recently a systematic study of oxidation behavior of nickel-based alloys and pure Ni in a cyclic

* Corresponding author. Frontier Research Initiative, New Industry Creation Hatchery Center, Tohoku University, 6-6-10 Aza-Aoba Aramaki, Aoba-ku, Sendai, 980-8579, Japan.

E-mail address: xujian@fri.niche.tohoku.ac.jp (J. Xu).

hydrogenated and oxygenated water chemistry in high temperature aqueous environment has been conducted by using various in situ electric/electrochemical methods and ex situ surface analysis techniques [12–14]. It was found that the film resistance in the oxygenated water chemistry is much higher than that in the hydrogenated water chemistry for all the materials. However, the evolution of the oxide film (morphology, composition, thickness, etc.) in a cyclically changing environment is dependent on the material. For Alloy 52 [12] the oxide film morphology and composition seem not changeable while for Alloy 182 [13] and pure Ni [14] some cyclic changes can be found with the cyclically changing environment.

For stainless steels (SSs), which are also commonly used as the structural materials in nuclear power plants, only Kim [15] studied the oxidation behavior of 316 SS in cyclic normal and hydrogen water chemistries. However, he only measured the electrochemical corrosion potential (ECP) in situ, and the cyclic changes between normal water chemistry and hydrogen water chemistry are mainly aimed for boiling water reactors.

In the present work, the corrosion behavior of solution annealed 316L SS exposed to simulated PWR primary water in a cyclically changing dissolved oxygen (DO) and dissolved hydrogen (DH) water chemistry was studied. The electric/electrochemical behavior of 316L SS was investigated by in situ methods such as contact electric resistance (CER) and electrochemical impedance spectroscopy (EIS). Several ex situ surface analysis methods such as scanning electron microscopy (SEM), Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) were used to characterize the oxide film formed during the experiment.

Our aim is to study the corrosion behavior (including the electric/electrochemical properties of oxide film and oxide morphology/composition/thickness) of solution annealed 316L SS exposed to simulated PWR primary water in a cyclically changing dissolved oxygen (DO) and dissolved hydrogen (DH) water chemistry. This water chemistry is to simulate the behavior when aerated water is added from the chemical and volume control system at full power. It should be noted that the test condition was not to fully simulate the PWR primary water, but rather excessive conditions were chosen from the scientific viewpoint.

On the other hand, from the scientific viewpoint, the oxidation/reduction ability of the alloy/oxide system and the correlation between oxide composition/thickness and oxide resistance are also worthy to studying. From our previous studies [12–14], oxide composition/thickness and oxide resistance seem not change synchronously for some alloys during cyclically changing dissolved oxygen (DO) and dissolved hydrogen (DH). As a follow-up study, our concern moves to 316L SS as shown in the present paper.

2. Experimental

2.1. Material and specimens

The material used for this experiment was a solution-annealed 316L SS with the composition of (in wt.%): C: 0.018, Si: 0.47, P: 0.28, Cr: 17.68, Mn: 0.82, Ni: 12.1, Mo: 2.2, the balance being Fe. For CER measurements, a pair of 2-mm diameter cylindrical specimens, which is specially designed, was used with flat surfaces polished with emery paper up to 1500 grit. The geometry is given in Fig. 1. Other coupons for the characterization of oxide film were exposed in a bypass tube between the preheater and the autoclave during experiments. The size of the coupons was 8 mm (length) \times 4 mm (width) \times 2 mm (thickness). The surfaces of the coupons were finished with 1500 grit emery paper. For the retrieval of the coupons from the bypass tube, the entire procedure was finished

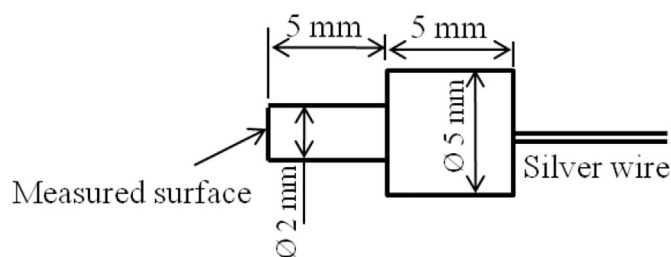


Fig. 1. Schematic drawing showing the geometry and dimension of the specimen used for the CER measurements.

within 5 min, including cooling down, pressure release and sample retrieval. All specimens were cleaned ultrasonically using ethanol and pure water before the experiments.

2.2. Water chemistry control

A simulated PWR primary water chemistry with 1200 ppm (by weight) B^{3+} as H_3BO_3 and 2 ppm Li^+ as $LiOH$ was used. The temperature of the autoclave and preheater was set to 290 °C. A circulated loop was used with a rate of 180 ml/min through the autoclave. Pure nitrogen gas, pure hydrogen gas, and a mixture of nitrogen and oxygen gases were used to control the inlet DO and DH. Cyclic changes in DH and DO were used. The details of each cycle including time and DH/DO concentration are given in Fig. 2. Each cycle contains four periods: DH and DO periods indicate the oxygenated water chemistry period and the hydrogenated water chemistry period respectively, during which DH or DO concentration is constant. Transition I period is the transition period from a DH period to a DO period, and transition II period is from a DO period to a DH period. The number ahead of DH or DO indicates the number of cycle, and the number following DH or DO indicates the day in each period. For example, 2DH2 means the second day of the second DH period. In the DH periods, DH was about 4500 ppb, and DO was less than 10 ppb. In the DO periods, DO was 3000 ppb, and DH was less than 20 ppb.

2.3. CER measurements

The CER technique was used to measure the electric resistance of the passive film directly. This technique was developed by VTT Technical Research Centre of Finland Ltd. The principle was introduced as follows. The electric resistance of a solid-solid contact between two specimens of similar material immersed to the test environment is measured by applying a direct current of known value through the two specimens and measuring the corresponding voltage drop. The two specimens were periodically connected and separated. When the two specimens were separated, these specimen surfaces were exposed to the test environment for oxidation.

2.4. Electrochemical measurements

For ECP and EIS measurements, a Solartron 1260 impedance/gain-phase analyzer coupled with a Solartron 1287 electrochemical interface were used. A 0.025 mA/cm² ac signal over the range of 1 mHz–30 kHz on a sample with a 2 cm² surface area at open circuit potential was used for the EIS measurements. A platinum counter electrode with an approximate surface area of 5 cm² was used. A pressure-balanced external Ag–AgCl (0.1 mol/L KCl) reference electrode was used for the ECP and EIS measurements. EIS data were collected every 24 h and analyzed by ZSimpWin software. All electrode potentials in the present work have been converted to the standard hydrogen electrode (SHE) by using the

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