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Effect of temperature on corrosion behavior of alloy 690 in high temperature hydrogenated water

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ABSTRACT

The influence of temperature on the corrosion behavior of Alloy 690 is evaluated using potentiodynamic polarization curves, electrochemical impedance spectra (EIS), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM). The corrosion rate of Alloy 690 reaches a local maximum at 250 °C. The kinetic control step of the growth of oxide film changes from the diffusion process of aqueous-phase ions to the growth of Cr-rich barrier layer in the temperature range of 200–300 °C. A modified double-layer model is proposed to describe the effect of temperature on the structure and composition of the oxide film.

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1. Introduction

Nickel-based alloys are usually used as structural materials in high temperature pressurized water due to their high resistance to stress corrosion cracking and corrosion [1–3]. Alloy 690 has been widely used as steam generator heat transfer tubing material in nuclear power plants as a modification material for Alloy 600 [4–10]. It is generally accepted that corrosion and stress corrosion in high temperature aqueous environments are basically electrochemical processes [11–13]. Using electrochemical measurement techniques, many studies have investigated the effects of temperature [3,6,14–19] and different water chemistry parameters, such as dissolved oxygen (DO) [6,20], dissolved hydrogen (DH) [11,21,22], and pH [16,23,24], on the electrochemical behavior of nickel-based alloys and stainless steels (SSs) in high temperature water.

Wu et al. [3,15] reported that the corrosion rates of Alloy 625 and 304 SS increased with the increase of temperature, and the growth of the oxide film at 25 °C and 100 °C was controlled by the diffusion process, while the growth within the temperature range of 200–300 °C was controlled by the chemical precipitation

of dissolved metal ions. Kim et al. [16] studied the influence of temperature on the electrochemical behavior of Alloy 600 and found that the passive current increased with the increase of temperature. Additionally, Kim et al. [16] suggested that the passivation of Alloy 600 was a thermally activated process. Maekawa and Warzee et al. [25–28] concluded that the corrosion rate of A304 SS in high temperature water experienced a local maximum around 250–300 °C, particularly for mechanical polished surfaces.

However, few studies have investigated the influence of temperature on the corrosion behavior of Alloy 690 in simulated hydrogenated primary water. In the present work, the aim is to study the effect of temperature on the electrochemical properties of the oxide film on Alloy 690 in high temperature hydrogenated water and to discuss the related influence mechanism of temperature on the structure and composition of the oxide film.

2. Experimental

2.1. Samples and solutions

The material used in this study was commercial steam generator tubing material Alloy 690 provided by the Electric Power Research Institute (EPRI, Palo Alto, California). The chemical composition of Alloy 690 tubes is given in Table 1. The arc angles and area of electrochemical samples were 72° and 3.25 cm², respectively. The samples used for electrochemical experiments were gradually ground with

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Table 1
Chemical composition of Alloy 690 tubes (wt%).

Ni	Cr	Fe	Mn	Ti	S	P	C	N	Si	Cu	Co	Al
59.50	29.02	10.28	0.30	0.33	0.001	0.009	0.018	0.0234	0.31	0.010	0.015	0.16

successive silicon papers up to 2000 grit. Then the specimens were point-welded to pure Ni wires which had already been shielded by heat-shrinkable polytetrafluoroethylene tubes. Every set of tests included five samples, three were used as parallel samples during electrochemical measurements to ensure the repeatability and another two parallel samples for SEM, XPS and TEM analysis. The samples used for SEM, XPS and TEM analysis were polished using 2.5 μm diamond paste before they were immersed in high temperature water.

The composition of the electrolyte was 2.3 ppm (by weight) Li as $\text{LiOH}\cdot\text{H}_2\text{O}$ and 1500 ppm B as H_3BO_3 solution. In the present experiments, an autoclave with a re-circulated water loop was used, and relevant information about the water loop was detailed in our previous work [6]. The concentrations of DO and DH were measured at the inlet of the loop and controlled at less than 0.01 ppm and 2.3 ppm (about $26\text{ cm}^3\text{ H}_2/\text{kg H}_2\text{O}$), respectively. The testing temperatures were chosen at 200 °C, 225 °C, 250 °C, 275 °C, 288 °C and 300 °C with a constant pressure of 10.0 MPa in the re-circulated water loop. Alloy 690 was immersed at each temperature for 288 h.

2.2. Electrochemical measurements

A Gamry Reference 600 potentiostat and related softwares were employed in all the electrochemical measurements. A three-electrode system including an external Ag/AgCl reference electrode, working electrode, and platinum (Pt) counter electrode was used. The concentration of KCl in the Ag/AgCl reference electrode was 0.1 M, and the size of the Pt foil was $20 \times 20\text{ mm}$. The open circuit potential (OCP) and EIS measurements at different temperatures were conducted at intervals. The EIS measurements were performed at the OCP with a signal amplitude of 10 mV rms. Due to the complexity of the testing electrolyte at high temperatures (e.g., high solution resistance), the potentiostat would make an artifact when conducting the EIS measurement in high frequency range. In order to exclude the artifact of the measurement device in high frequency range, the selected measurement frequency range was from 10^2 Hz to 10^{-2} Hz . Measurements of the potentiodynamic polarization were conducted at a scan rate of 0.5 mV/s. Tests under different temperatures were repeated three times and the repeatability for every set of tests was very good due to the high stability of re-circulated water loop. Gamry Echem Analyst software was used to fit the results of the EIS data.

All the electrode potentials measured in the present work were calibrated to standard hydrogen electrode (SHE) using the following relationship [29]:

$$E_{\text{SHE}} = E_{\text{obs}} + 0.2866 - 0.001 \times (T - t) + 1.745 \times 10^{-7} \times (T - t)^2 - 3.03 \times 10^{-9} \times (T - t)^3 \quad (1)$$

where T is the testing temperature, and t is the room temperature (25 °C), E_{obs} is the measured electrode potential, and E_{SHE} is the electrode potential vs. SHE.

2.3. SEM, XPS and TEM analysis

To observe the microstructure of corrosion products formed at different temperatures, a FEI XL30 field emission environmental scanning electron microscopy (ESEM) was used. A VG ESCALAB 250 X-ray photoelectron spectrometer was used for XPS analysis. Composition-depth profiles were obtained with 2.0 keV Ar ions at

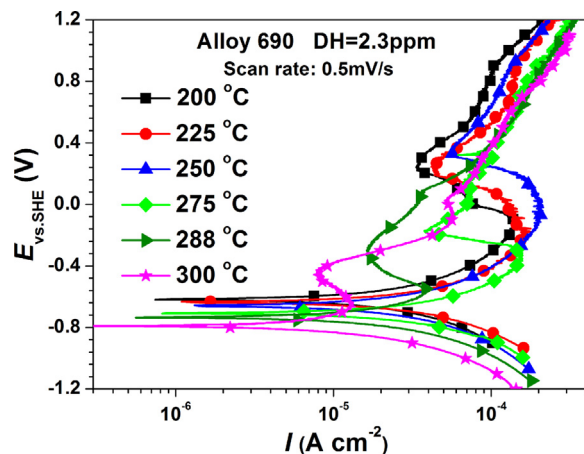


Fig. 1. Potentiodynamic polarization curves for Alloy 690 after 288 h immersion at different temperatures.

a target current of $2.0\text{ }\mu\text{A}/\text{cm}^2$. The sputtering rate was estimated to be about 0.1 nm/s (vs. Ta_2O_5). Thin-foil samples for TEM observation were prepared using a FEI Helios 600 focus ion beam (FIB) equipment with Ga ion sputtering after coating the samples with a thin layer of Pt. The microstructure and chemical composition of the cross-section of the oxide film for Alloy 690 were analyzed using a FEI 300 kV Tecnai G2 F30 high-resolution TEM, equipped with an energy-dispersive spectroscopy (EDS) system.

3. Results

3.1. Potentiodynamic polarization curves

Fig. 1 shows the potentiodynamic polarization curves for Alloy 690 after 288 h immersion at different temperatures. The corrosion potential E_{corr} for Alloy 690 decreases with increasing temperature. Both the peak activation current density and minimum passivation current density for Alloy 690 increase first at 200–250 °C and then decrease rapidly at 275–300 °C.

3.2. Open circuit potential and electrochemical impedance spectroscopy

Fig. 2 shows the EIS curves for Alloy 690 after 288 h immersion at different temperatures. The solution resistance (R_s) corresponding to the impedance modulus $|Z|$ at 10^2 Hz decreases with increasing temperature. The difference in low-frequency impedance and solution resistance can be used to compare the protectiveness of the oxide film formed in different environments qualitatively. The difference in $|Z|$ between 10^{-2} Hz and 10^2 Hz decreases slowly in the temperature range of 200–250 °C and increases dramatically at 275–300 °C. The approximate frequency ranges, in which two time constants arise, are indicated with red arrows in **Fig. 2b**.

Fig. 3 shows the evolution of OCP values with immersion time for Alloy 690 at 250 °C and 288 °C. The OCP values for Alloy 690 remain stable on the whole during the testing time at 250 °C and 288 °C.

Fig. 4 shows the EIS plots for Alloy 690 after different exposure time at 250 °C. It can be seen that the EIS impedance for Alloy 690 at 250 °C changes slightly with increasing immersion time after

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