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The oxidation of alloy 690 in simulated pressurized water reactor primary water

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ABSTRACT

The oxide film formed on alloy 690 in simulated pressurized water reactor primary water is decorated with NiFe_2O_4 particles which remain epitaxial with matrix. The penetrative inner oxides on both surface and crack wall are composed of Cr-rich $(\text{Fe,Cr,Ni})_3\text{O}_4$ and Cr_2O_3 which were formed by solid state reactions of substrate with inwards diffusing oxygen and have orientation relationships with the substrate. Cr_2O_3 forms first along the widely spaced planes of substrate. Compact Cr_2O_3 layer cannot develop as there is no long-range outward diffusion of Cr in substrate with low defect density at low temperature.

1. Introduction

Nickel base alloy 690 has been widely used in steam generator tubes, vessel head penetrations, and control rod drive mechanism housing in pressurized water reactors (PWR) because of its superior stress corrosion cracking (SCC) resistance to alloy 600 [1–3]. Thus far there have been no reports of cracking of alloy 690 in service. However, laboratory test results have shown that this alloy is not immune to SCC and under some conditions alloy 690 is susceptible to both crack initiation [4] and high rates of crack propagation [5–8]. So it is of importance to further improve the understanding of SCC behavior of this alloy.

Oxidation is a fundamental process in stress corrosion cracking in high temperature water which is typical of a nuclear power plant environment. Significant effort has been made to characterize the oxide films formed on alloy 690. It has been found that the oxidation behavior of alloy 690 can be significantly affected by the dissolved oxygen (DO) concentration [9–12]. In hydrogenated simulated PWR primary water environment, it has been widely reported that the oxide film formed on alloy 690 consists of an outer layer of discrete oxide particles enriched in Ni and Fe and an inner layer enriched in Cr [11,13–19]. It is generally accepted that the outer oxide particle on alloy 690 has a spinel structure and forms by the precipitation of cations dissolved in the solution [9,15,16,19], similar to that on alloy 600 [20,21] and stainless steel [22–25]. Previous studies [11,16,18,19] consistently suggest that the inner layer contains both spinel and corundum Cr_2O_3 which is at the oxide/substrate interface. Tracer experiments by Marchetti et al. [26,27] confirmed that the formation of inner oxide mainly occurs via the inward diffusion of oxygen. The Cr-rich inner layer is believed to be

a barrier that suppresses further oxidation of the matrix.

Although the oxidation behavior of alloy 690 in a hydrogenated environment has been well studied and the results from different labs are generally consistent, there are still some disagreements. The works from Sennour et al. [16,27] and Zhang et al. [11] suggest that the inner oxide is a continuous layer of Cr-rich spinel with some small Cr_2O_3 particles at the oxide/substrate interface. However, the results from Olszta et al. [18,19] and Moss [28] indicate that the inner oxides have filamentary structure with discontinuous Cr_2O_3 platelets surrounded by cubic MO structure. Moreover, their works [19,28] revealed that a compact Cr_2O_3 film formed at the intersection of grain boundaries with the exposed surface.

This work is focused on the oxidation behavior of alloy 690 in 360 °C hydrogenated water. The microstructures of oxides formed on sample surface and an intergranular crack wall were characterized in detail to further improve understanding of the formation of the inner oxide.

2. Experimental

The chemical composition of alloy 690 studied in this work is 57.6 wt.% Ni, 32.7% Cr, 8.64% Fe, 0.25% Mn, 0.315% Al, 0.08% Si and 0.02% C. The material was a forged bar with a diameter of 185 mm. It was hot rolled to an 8 mm thick sheet at around 1100 °C, then solution annealed (SA) at 1100 °C for 1 h and water quenched (designated as SA-00-00). Some of the solution annealed material was cold rolled (CR) to 20% thickness reduction and then aged at 475 °C for 10000 h (designated as CR-475-10000). Materials from both thermo-mechanical conditions were machined into round tensile bars with the sample axis in

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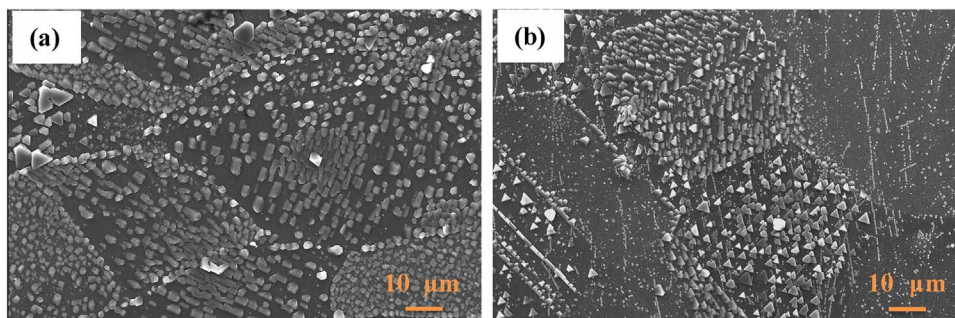


Fig. 1. (a) SEM images of sample surfaces from CR-475-10000 and (b) SA-00-00 after constant extension rate tensile tests in 360 °C high purity water containing 18 cm³ (STP) H₂/kg H₂O. CR-475-10000 was strained to 3.66% during 1270 h exposure, and SA-00-00 was strained to 1.78% during 564 h exposure.

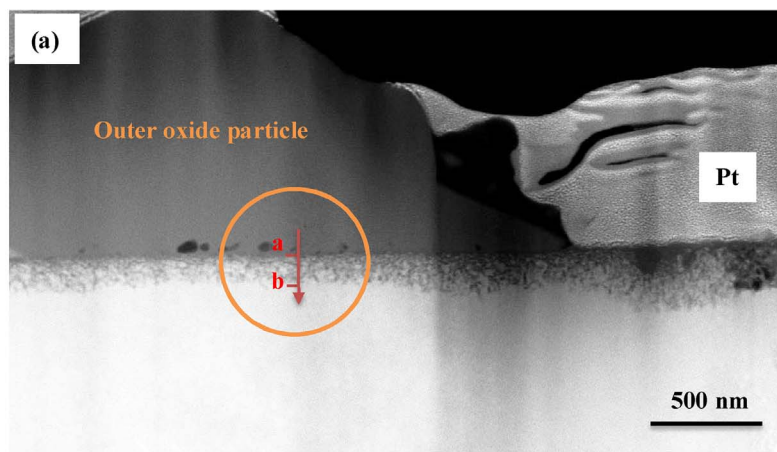
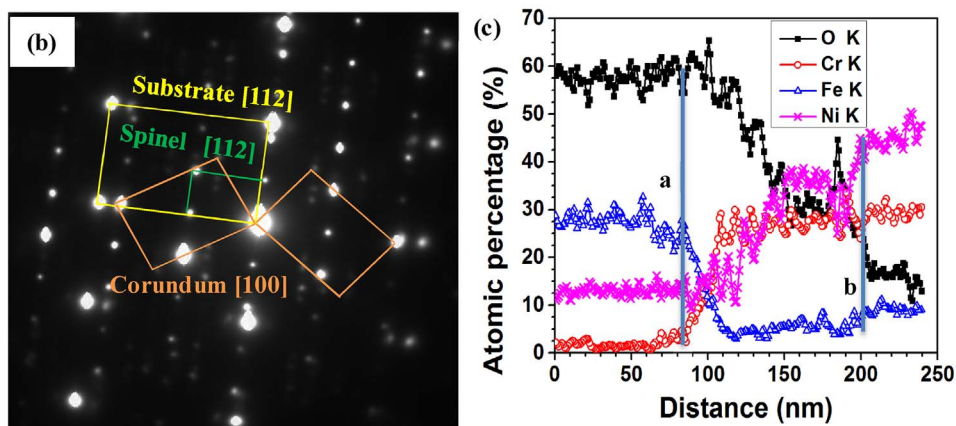


Fig. 2. (a) STEM-HAADF image of oxide formed on surface of CR-475-10000, strained to 3.66% at 1×10^{-8} /s in 360 °C high purity water containing 18 cm³ (STP) H₂/kg H₂O during 1270 h exposure, (b) diffraction pattern from circled area in (a) and (c) element profile along the arrow indicated on (a).



the cold rolling direction as described in a previous study [4]. The tensile bar measures 20 mm in gauge length and 2 mm in diameter. The gauge section of tensile bar was mechanically abraded up to 4000 grit and electropolished for 30 s at 30 V in a solution of 10% (volume fraction) perchloric acid in methanol which was cooled down to -30 °C. The samples were cleaned three times alternately with methanol and acetone immediately after electropolishing. The tensile samples were strained at 1×10^{-8} /s in 360 °C high purity water containing 18 cm³ (STP) H₂/kg H₂O as described in our previous work [4,29]. CR-475-10000 was strained for 1270 h to a uniform plastic strain of 3.66% and SA-00-00 was uniformly strained for 564 h to 1.78%.

Following straining, a cross section of a cracked prior twin boundary from CR-475-10000 and a cross section of oxidized surface from SA-00-00 were made using focused ion beam (FIB) milling with a final beam current of 80 pA on an FEI Helios Nanolab 650. The oxides formed on both the sample surfaces and the crack wall were used for transmission electron microscopy (TEM) analysis on a JEOL 3011 microscope and scanning transmission electron microscopy (STEM)

analysis on a JEOL 2100F microscope. For TEM analysis, selected area electron diffraction (SAED) along with dark field imaging were conducted. For STEM analysis, the samples were characterized with high angle annular dark-field (HAADF) and energy dispersive spectroscopy (EDS) detectors.

3. Results

3.1. Outer oxide particles

Examination of the sample surfaces of CR-475-10000 and SA-00-00 exposed to simulated PWR primary water at 360 °C reveals scattered oxide particles of similar shape and orientation but with a range of sizes on each grain, as shown in Fig. 1. Fig. 2a shows a STEM-HAADF image of oxide formed on surface of CR-475-10000 which contains a large particle. The diffraction pattern from the circled area in Fig. 2a is shown in Fig. 2b and corresponds to the [112] zone axis of the substrate (Fig. 2b). A pattern from [112] zone axis of spinel was also observed. So the spinel particle has a cube-on-cube relationship with matrix and all

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