Post-Print: Structural Analysis of Surface Oxide Films on Electropolished 690TT Alloy in High-Temperature High-Pressure Water under Sequential Hydrogenated and Oxygenated Conditions

Authors: Zhang Zhiming, Wang Jianqiu, Han Enhou, Ke Wei

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Abstract

Electropolished 690TT alloy samples were continuously immersed for 720 h in high-temperature high-pressure water containing 1500 mg/L B, 2.3 mg/L Li, and 2.5 mg/L H2 at 325 °C and 15.6 MPa. After this period, half of the samples were removed for corrosion product analysis, while the remaining samples continued to be immersed for an additional 720 h in the same high-temperature high-pressure water containing 2.0 mg/L O2. The microstructure of the oxide films grown on the sample surfaces under these two conditions was analyzed using SEM, GIXRD, and TEM. The results indicate that the oxide film formed on the surface of electropolished 690TT alloy in hydrogen-only high-temperature high-pressure water exhibits a bilayer structure: the outer layer consists of dispersed large-grained oxides rich in Ni and Fe and loosely packed needle-like oxides rich in Ni; the inner layer is a near-continuous Cr-rich oxide; both the outer and inner layer oxides possess a spinel structure. After continuous immersion in the hydrogen/oxygen-containing solution, the oxide film grown on the sample surface also exhibits a bilayer structure: the morphology, chemical composition, and phase structure of the outer layer are similar to those of the oxide film grown under hydrogen-only conditions, with only the length of the needle-like oxides significantly increased; whereas the inner layer of the oxide film transformed into nanosized NiO. The dissolved oxygen introduced in the later stage expanded the stability region of Ni-containing oxides in the potential-pH diagram, promoting rapid growth of the Ni-rich needle-like oxides in the outer layer; more importantly, the dissolved oxygen increased the corrosion potential of Fe- and Cr-containing oxides, facilitating dissolution of the Cr-rich inner layer oxide formed under hydrogen-only conditions, destroying the protective structure

of the oxide film, and increasing the corrosion rate of electropolished 690TT alloy. During continuous immersion in primary loop hydrogen/oxygen conditions, electropolishing treatment cannot reduce the corrosion rate of 690TT alloy.

Full Text

Preamble

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Abstract

Electropolished (EP) alloy 690TT samples were first oxidized in simulated primary water containing boron and lithium with 2.5 mg/L H_2 at 325 °C and 15.6 MPa for 720 h. Half of the samples were then continuously immersed in the same solution with 2.0 mg/L O_2 for an additional 720 h. The microstructure and chemical composition of the oxide films formed under these two conditions were analyzed using SEM, GIXRD, and TEM. The results show that the dual-layered oxide film formed under single hydrogen water chemistry consists primarily of spinel oxides. The outer layer comprises large oxide particles rich in Ni and Fe, along with underlying loose needle-like oxides rich in Ni. The inner layer consists of continuous Cr-rich oxides. Both layers exhibit a spinel structure. The oxide film formed on EP alloy 690TT under hydrogen/oxygen water chemistry also displays a duplex structure. While the surface morphology and chemical composition of the outer layer are similar to those formed under hydrogen water chemistry, the inner layer transforms into nano-sized NiO. The stable phase region for Ni oxides in the potential-pH diagram is enlarged by the later addition of dissolved oxygen, which promotes rapid growth of the outer Ni-rich needle-like oxides. Furthermore, oxygen increases the corrosion potential of Fe- and Cr-containing oxides, facilitating dissolution of the inner Cr-rich oxide layer formed under hydrogen conditions, destroying the protective structure of the oxide film and increasing the corrosion rate of EP alloy 690TT. Electropolishing treatment does not reduce the corrosion rate of alloy 690TT in simulated primary water with sequentially dissolved hydrogen and oxygen.

Keywords: alloy 690TT, oxide film, hydrogen water chemistry, oxygen water chemistry, protective microstructure

Introduction

Steam generators (SG) are critical components connecting the primary and secondary circuits in pressurized water reactor nuclear power plants. Serving as the pressure boundary between the two circuits, heat exchange occurs through the SG' s heat-transfer tubes. Nickel-based alloy 690TT, with higher Cr content, has gradually replaced alloy 600 and is now widely used for manufacturing SG tubing. Primary water contacting the inner tube wall operates at 320–330 °C and 15–16 MPa. Prolonged exposure to this high-temperature, high-pressure

water inevitably leads to oxide film formation on the 690TT alloy surface, and the protectiveness of this surface oxide is crucial for preventing further corrosion. Alloy 690TT contains approximately 30 wt% Cr, which ensures formation of a protective oxide film and provides excellent corrosion resistance.

Numerous factors influence corrosion behavior in high-temperature high-pressure water, including surface condition. Studies have shown that electropolishing removes the near-surface deformation layer and reduces high-energy sites that serve as preferential nucleation locations. Consequently, compared with mechanically polished samples, electropolished 304 stainless steel exposed to hydrogenated water at 260 °C exhibits oxide growth characterized by uniform nucleation, finer and more uniform particle size, and relatively better protectiveness. However, ground and electropolished 690TT alloys show completely different corrosion behaviors in simulated primary water. Comparative analysis of cross-sectional microstructures of oxide films formed on these two surface conditions after 15–2160 h exposure revealed that grinding introduces a severely deformed surface layer with nanocrystalline structure, providing numerous short-circuit diffusion paths for outward cation diffusion (particularly Cr) and inward O diffusion, thereby promoting rapid growth of a protective Cr-rich oxide film. In contrast, electropolished samples lack a deformation layer and have lower strain, requiring longer times to form a protective Cr-rich oxide film.

To suppress radiolysis of primary coolant and reduce material corrosion rates, nuclear power plants typically maintain dissolved H_2 concentrations of 25–35 cm^3/kg (STP) during normal operation, ensuring reducing conditions. Nevertheless, oxygen can enter primary water through various pathways, such as dissolved oxygen in makeup water or deliberate O_2 injection during shutdown maintenance. Consequently, actual plant operation involves sequential alternation between hydrogenated and oxygenated water chemistry. Previous studies on ground 690TT alloy exposed to sequential hydrogen/oxygen conditions revealed that later-stage dissolved oxygen completely destroys the oxide film formed under hydrogen conditions, promoting dissolution of the Cr-rich inner oxide and substrate Cr and Fe. When a cold-worked surface is present, this further enhances outward Cr diffusion and dissolution, potentially accelerating corrosion. Thus, grinding—which proves beneficial under hydrogenated conditions—may have the opposite effect under oxygenated conditions. Since electropolished samples provide fewer short-circuit diffusion paths for Cr atoms from the substrate, they might exhibit relatively lower corrosion rates when water chemistry shifts from hydrogenated to oxygenated conditions. Evaluating and comparing the corrosion behavior of 690TT alloy with different surface states under complex water chemistry conditions provides valuable reference for tube manufacturing and long-term service.

This study first immersed electropolished 690TT alloy in simulated primary water with normal hydrogen chemistry, then continued exposure of some pre-corroded samples in oxygenated primary water. The surface morphology, phase

structure, and chemical composition of oxide films formed under these two conditions were compared and analyzed. The influence of later-stage dissolved oxygen on subsequent oxide growth on electropolished samples was discussed and compared with the corrosion behavior of ground samples under identical conditions.

Experimental Methods

The 690TT alloy specimens were taken from commercially used steam generator heat-transfer tubes in nuclear power plants, with chemical composition (wt%) of: Cr 29.02, Fe 10.28, Mn 0.30, Ti 0.33, Si 0.001, P 0.009, C 0.018, N 0.0234, Si 0.31, Cu 0.01, Co 0.015, Al 0.16, and Ni balance. The tubes were first cut longitudinally and flattened using a hydraulic press, then heat-treated at 715 °C for 2 h to remove residual stress. The flattened samples were further cut into 10 mm × 10 mm coupons for oxidation testing. Samples were wet-ground sequentially to 2000-grit, mechanically polished with 2.5 m diamond paste, and electropolished for 3 s in a 30 vol% HNO_3-methanol solution. All specimens were ultrasonically cleaned in acetone and ethanol, dried, and stored in a vacuum desiccator.

Immersion tests were conducted using a dynamic high-temperature high-pressure corrosion testing system with a recirculating water loop. The test solution simulated primary water chemistry containing boron and lithium. High-purity deionized water and analytical-grade H_3BO_3 and $LiOH \cdot H_2O$ were used to prepare a solution containing 1500 mg/L B and 2.3 mg/L Li. High-purity N_2 and H_2 were first used to control the dissolved H_2 content at 2.5 mg/L (O_2 < 1 mg/L). The autoclave pressure was raised to 15.6 MPa and temperature to 325 °C, and the electropolished samples were immersed in this hydrogenated solution for 720 h. After this stage, half the samples were removed for surface morphology observation and microstructural analysis. High-purity N_2 and O_2 were then used to control dissolved O_2 content at 2.0 mg/L (H_2 < 2 mg/L), and the remaining samples were continuously immersed in this oxygenated solution for another 720 h. Thus, two exposure conditions were investigated: single hydrogen water chemistry for 720 h (DH) and sequential hydrogen water chemistry for 720 h followed by oxygen water chemistry for 720 h (DH/DO).

After immersion testing, surface morphology and local chemical composition were examined using a FEI XL 30 environmental scanning electron microscope (ESEM) equipped with energy-dispersive spectroscopy (EDS). Grazing incidence X-ray diffraction (GIXRD) was performed at the BL14B1 beamline of a synchrotron radiation facility to analyze the phase composition of the corrosion product film formed under single DH conditions, using 10 keV photons at incident angles of 0.1°–0.5°. Conventional Cu Kα X-ray diffraction (XRD) using an X' Pert Pro Panalytical diffractometer was employed to analyze the oxide film formed under DH/DO conditions. Cross-sectional samples of the oxide films formed under both DH and DH/DO conditions were prepared using FEI

QUANTA 200 3D and Helios 600 focused ion beam (FIB) systems. The cross-sections were analyzed using a 200 kV JEOL 2100F high-resolution transmission electron microscope (HRTEM) equipped with EDS.

Results

[Figure 1: see original paper] shows SEM images of oxide films grown on electropolished samples under the two immersion conditions. After 720 h in single DH solution, the surface was covered with dispersed large oxide particles (1–2 m) and loose needle-like oxides (Fig. 1a). Following sequential DH/DO exposure, the surface still exhibited large particles and needle-like oxides, but the particle size increased significantly to 2–4 m and the needle-like oxides grew substantially longer (Fig. 1b).

SEM-EDS analysis of the large particles in Fig. 1 was performed. To compare chemical composition differences, O content was subtracted and Ni, Cr, and Fe contents were recalculated as absolute values, as shown in . The large particles formed under both conditions had similar compositions, being rich in Ni and Fe with very low Cr content.

[Figure 2: see original paper]a presents GIXRD results for the corrosion product film on electropolished samples after 720 h in single DH solution, with diffraction peak intensities normalized. Based on the 2 diffraction angles, the oxide film consisted primarily of a spinel-structured oxide and minor metallic Ni. Since diffraction peaks for spinel oxides such as $NiFe_2O_4$, $NiCr_2O_4$, and $NiCrFeO_4$ differ by only 0.3°, the exact chemical formula could not be determined. After sequential DH/DO exposure, the corrosion products consisted mainly of spinel-structured oxides and NiO, with a small amount of $Ni(OH)_2$ (Fig. 2b).

Comparing morphology and phase composition between the two conditions, later-stage dissolved oxygen had relatively minor effects on surface morphology but caused significant changes in phase structure.

[Figure 3: see original paper] shows cross-sectional TEM images of the oxide film formed on electropolished 690TT alloy after 720 h in single DH solution. [Figure 3: see original paper]b is a magnified view of the rectangular region in Fig. 3a. The oxide film exhibited a typical duplex structure: the outer layer consisted of the large particles and needle-like oxides observed by SEM, with the needle-like oxide layer measuring 500-600 nm thick. During FIB thinning, some needle-like oxides were lost due to Ga ion beam bombardment, so only a few needle-like oxides were retained for TEM observation. Beneath this layer, near the substrate, was the inner oxide layer with non-uniform thickness, reaching approximately 90 nm at its thickest point. A clear interface existed between the inner and outer layers; needle-like oxides grew outward while inner oxides grew inward from this interface, suggesting it represents the original sample surface. TEM-EDS analysis of positions 1–4 in Fig. 3a is presented in , showing only absolute Ni, Cr, and Fe contents. Similar to SEM-EDS results, the large particles were rich in Fe and Ni (with Fe > Ni) and very low in Cr. The needle-

like oxides were also primarily Ni and Fe oxides, with Ni content much higher than Fe and only about 15.87% Cr. Compared with the substrate composition (position 4), the inner layer showed significant Cr enrichment up to 73.80 wt%, far exceeding Ni and Fe contents.

[Figure 4: see original paper] shows TEM images of the oxide film formed on electropolished 690TT alloy after sequential DH/DO exposure. [Figure 4: see original paper]b is a magnified view of the rectangular region in Fig. 4a. Note that the outermost large particles observed in SEM were also lost during sample preparation due to excessive Ga ion beam bombardment. The oxide film again exhibited a duplex structure: an outer layer of large particles and loose needle-like oxides, and a continuous inner layer near the substrate, though parts of the inner layer were also missing due to ion beam damage. [Figure 4: see original paper]c and d show high-magnification TEM and HRTEM images of the inner layer, revealing it consists primarily of 5–10 nm oxide particles with amorphous oxide distributed between them. A clear interface separated the inner and outer layers, with needle-like and inner oxides growing outward and inward, respectively. Unlike the oxide film formed in single DH solution, growth was significantly faster under DH/DO conditions, with the needle-like oxide layer reaching 3.0–4.5 m and the inner layer averaging 300 nm thick.

Selected-area electron diffraction (SAED) analysis of the needle-like oxides, inner oxides, and substrate (positions 1–3 in Fig. 4a) is shown in [Figure 5: see original paper]a–c. The loose needle-like oxides exhibited a spinel crystal structure. SEM-EDS analysis of multiple needle-like oxides in Fig. 1b yielded an absolute composition (wt%) of: Ni 70.60, Fe 17.43, Cr 11.96. The inner layer diffraction rings corresponded to nano-sized NiO, consistent with the morphology in Fig. 4c and d. TEM-EDS analysis of the inner layer composition confirmed high Ni content (87.11 wt%) with very low Cr (8.63%) and Fe (4.27%), indicating the inner layer is primarily NiO. [Figure 5: see original paper]c shows the diffraction pattern of the 690TT alloy substrate. Based on electron diffraction results, the spinel oxide and NiO identified by GIXRD are distributed in the outer and inner layers, respectively.

Discussion

In the results for electropolished 690TT alloy after 720 h in single DH solution, few needle-like oxides were retained (Fig. 3) and the inner oxide layer was too thin for electron diffraction analysis. However, literature [12–15] has confirmed that for nickel-based alloys like 690TT, the outer large particles, needle-like oxides, and inner Cr-rich oxides all possess spinel structures, consistent with the GIXRD results in Fig. 2a showing the oxide film is dominated by spinel oxides. Since both outer and inner layers contain Ni, Cr, and Fe (Table 2), the spinel oxides are likely non-stoichiometric with the formula $(Ni\ Fe_{1\ \ })(Fe\ Cr_{1\ \ })_2O_4$ (x, $y < 1$). Minor metallic Ni was also detected. Ziemniak and Hanson [16] found that oxidation of nickel-based alloy 600 in hydrogenated high-temperature water is non-selective. Given that alloy 690 contains ~60% Ni, besides Ni participating

in spinel formation, excess NiO should also form. With 2.5 mg/L H_2 in the solution, this metallic Ni likely results from reduction of NiO or $NiFe_2O_4$ by H_2, stabilizing it in the oxide film [6,16,17]. Sennour et al. [13] also identified Cr_2O_3 nodules at the inner oxide/substrate interface using high-resolution analytical techniques.

Therefore, the oxide film formed on electropolished 690TT alloy in single DH solution has a duplex structure: an outer layer of Ni- and Fe-rich large particles and Ni-rich needle-like oxides; an inner layer of Cr-rich oxides; with both layers having spinel structures. The average corrosion rate of the inner layer is 1.10 mm/a (Fig. 3b), similar to oxide structures formed on 690TT alloy after 1440 and 2160 h in hydrogenated water [5].

In contrast, the oxide film formed on electropolished 690TT alloy under DH/DO conditions also exhibits a duplex structure. The outer layer consists mainly of Ni- and Fe-rich large particles and Ni-rich needle-like oxides with spinel structure, similar to hydrogen-only conditions. The minor $Ni(OH)_2$ identified by GIXRD primarily forms from deposition of Ni ions from solution onto the sample surface [10]. However, the inner layer consists of nano-sized NiO particles, with an average corrosion rate of 3.65 mm/a (Fig. 4b).

Thus, later-stage dissolved oxygen not only promotes rapid growth of outer Ni-rich needle-like oxides but, more importantly, transforms the inner Cr-rich oxide formed under hydrogen conditions into NiO and increases the inner layer corrosion rate. The effect of dissolved oxygen on the inner oxide microstructure of electropolished samples is similar to that observed on ground samples [11].

The influence of dissolved oxygen on oxide film microstructure can be explained thermodynamically [11]. According to potential-pH diagrams for Ni-H_2O, Cr-H_2O, and Fe-H_2O at 300 °C [18], the stable forms of the main alloying elements under reducing hydrogen conditions are Ni, Cr_2O_3, and Fe_2O_3, respectively, while under oxidizing conditions they are NiO, CrO_4^{2-}, and FeO_4^{2-}. High-temperature potential-pH diagrams for oxides [19] also indicate that Cr_2O_3 and Cr-containing spinels like $NiCr_2O_4$ and $FeCr_2O_4$ are stable under hydrogenated conditions, whereas only $NiFe_2O_4$ is stable under oxygenated conditions. Kim [20] found that oxygen significantly increases the corrosion potential of oxides and alloy elements, causing selective oxidation of Cr in Cr-containing oxides to soluble CrO_4^{2-}, resulting in greater Cr dissolution than Fe and Ni. Therefore, in hydrogenated solution, 690TT alloy can form Cr-rich spinel oxides and Cr_2O_3. Literature [21] indicates that spinel oxides like $NiCr_2O_4$ and continuous Cr_2O_3 layers reduce cation diffusion rates, making the inner oxide layer formed under hydrogen conditions protective.

Due to low surface strain and fewer nucleation sites, electropolished samples facilitate rapid surface diffusion along screw dislocation cores for needle-like oxide growth in both hydrogenated and oxygenated water [10,12]. When exposure conditions shift from hydrogenated to oxygenated water chemistry, the potential-pH diagram thermodynamically expands the stable phase region for

Ni oxides while shrinking that for Cr oxides. Consequently, outer Ni-rich needle-like oxides grow rapidly first (Fig. 1b). The porous nature of needle-like oxides provides no barrier to inward oxygen diffusion, allowing the previously formed Cr-rich inner oxide and any interfacial Cr_2O_3 to be oxidized to higher-valency soluble CrO_4^{2-} ions that enter solution. After this oxide dissolution, most Fe and Cr atoms from the fresh substrate are similarly oxidized to high-valency ions and dissolve, leaving a Ni-enriched dealloyed layer on the substrate surface. Oxidation of this layer leads to formation of inner NiO, with only small amounts of Fe and Cr atoms being oxidized and retained in the inner oxide layer.

Marchetti et al. [14] used O^{18} isotope tracing in simulated nuclear primary water corrosion experiments, calculating oxygen grain boundary diffusion coefficients in spinel oxide (Ni Fe_1)(Cr Fe_1)$_2O_4$ as 2\$×10^{\{-18\}-1×10}\{-17\}\$ cm^2/s. At 330 °C, the oxygen grain boundary diffusion coefficient in Ni is approximately 8.25\$×10^\{-11\}\$ cm^2/s [22]. Thus, oxygen diffuses much faster in Ni than in Cr-containing spinels. Consequently, the newly formed inner NiO cannot protect the substrate in oxygenated solution, leading to higher corrosion rates for electropolished 690TT alloy under later-stage oxygenated conditions compared with hydrogenated conditions.

Compared with ground 690TT alloy exposed to identical DH/DO sequential conditions for the same duration [11], dissolved oxygen also transforms outer granular oxides formed under hydrogen conditions into platelet oxides and minor needle-like oxides on ground surfaces, completely altering outer oxide morphology. More significantly, the growth rates of inner NiO after dissolution of Cr-rich oxides must be compared between the two surface finishes. Grinding 690TT alloy to 400-grit creates a severely deformed surface layer (~470 nm thick) [7] much thicker than the inner oxide formed under hydrogen conditions (25–40 nm) [6,11]. After this oxide dissolves, new inner oxide growth occurs within the deformed layer. However, the high defect density in the deformed layer does not apparently accelerate inner NiO growth, as the inner NiO growth rates are comparable for ground and electropolished surfaces. This may be attributed to the dense Cr-rich granular outer oxide formed on ground samples under hydrogen conditions, which (or its residual after dissolution) inhibits inward oxygen diffusion, thereby affecting subsequent NiO growth. In contrast, the porous needle-like outer oxide on electropolished samples provides no barrier to oxygen ingress, allowing continuous inner NiO growth.

During complex sequential hydrogen/oxygen water chemistry transients in nuclear power plants, electropolished and ground 690TT alloys exhibit different oxide growth characteristics. When primary water chemistry shifts from normal hydrogenated conditions to oxygenated conditions or localized oxygenated zones, initial electropolishing treatment of 690TT alloy does not reduce further material corrosion.

Conclusions

1. Under both single hydrogenated and sequential hydrogenated/oxygenated immersion conditions, electropolished 690TT alloy surfaces are covered with dispersed large oxide particles and loose needle-like oxides.
2. After 720 h in single hydrogenated solution, the oxide film on electropolished 690TT alloy exhibits a duplex structure: an outer layer of Ni- and Fe-rich large particles and Ni-rich needle-like oxides; an inner layer of dense Cr-rich oxides. Both layers have spinel structures, and the inner oxide layer provides substrate protection.
3. After 720 h in hydrogenated solution followed by 720 h in oxygenated solution, the oxide film on electropolished 690TT alloy also shows a duplex structure. The outer layer structure and composition are similar to that formed in single hydrogenated solution, but the inner layer consists primarily of nano-sized NiO. This oxide film provides no substrate protection.
4. Later-stage dissolved oxygen promotes rapid growth of outer Ni-rich needle-like oxides and dissolution of the inner Cr-rich oxide formed under hydrogen conditions, as well as dissolution of substrate Cr and Fe. This destroys the protective oxide structure and increases the corrosion rate of electropolished 690TT alloy. Electropolishing treatment does not reduce the corrosion rate of 690TT alloy during simulated primary water exposure with sequential hydrogen/oxygen chemistry.

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Note: Figure translations are in progress. See original paper for figures.