Anisotropic Oxide Film Growth on Zr-4 Alloy During Corrosion in LiOH Aqueous Solution (Postprint)

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Abstract

Large-grained Zr-4 alloy samples were corroded in 0.01 mol/L LiOH aqueous solution at 360 ℃ and 18.6 MPa for 70 and 160 d. The microstructure and crystal structure of the oxide films, as well as the relationship between oxide film thickness and metal grain surface orientation, were investigated using EBSD, SEM, and HRTEM. The results indicate that after 160 d of corrosion, the oxide film is relatively thick when the metal grain surface orientation is near (0001), and the oxide film growth exhibits pronounced anisotropic characteristics; however, this feature is not evident after 70 d of corrosion. Metal grains with surface orientations near (0001), (1010), and (1120) were selected from the sample corroded for 70 d, and the microstructure at the oxide film/metal interface on their surfaces was examined. The microstructure of oxide films formed on metal grain surfaces with different orientations shows certain variations; the misorientation between m-ZrO2 columnar crystal grains in the oxide film generated on grain surfaces with orientation near (0001) is relatively large. The microstructure and crystal structure of the oxide film are highly complex; in addition to the predominant m-ZrO2, c-ZrO2, t-ZrO2, and sub-oxide Zr3O are also present. The microstructure of the oxide film continuously evolves during the corrosion process, which affects oxygen ion diffusion and consequently influences oxide film growth. This evolution process is further influenced by differences in the initial microstructure of oxide films formed on grain surfaces with different orientations and by the water chemistry conditions during corrosion. This constitutes the primary reason for the anisotropic growth characteristics of oxide films during Zr-4 corrosion in LiOH aqueous solution.

Full Text

Preamble

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Investigation of the Anisotropic Growth of Oxide Layers Formed on Zr-4 Alloys Corroded in LiOH Aqueous Solution

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Abstract

Zr-4 coarse-grained specimens were corroded in static autoclave at 360 ℃, 18.6 MPa in 0.01 mol/L LiOH aqueous solution for 70 and 160 d exposure. EBSD, SEM and HRTEM were used to investigate the microstructures and crystal structures of oxide layers, and the relationships between the oxide thickness and the grain orientation of the metal matrix. The results showed that the oxide layers formed on the grain surfaces with the orientations nearby basal plane (0001) were thicker, and exhibited a prominent anisotropy for the oxide growth when Zr-4 specimens were corroded in LiOH aqueous solution for 160 d, but this was not the case for 70 d. The grains with the surface orientation nearby (0001), (1010) and (1120) were selected from the specimens corroded for 70 d to investigate the effect of metal grain orientation on the microstructure of oxide layers. The results showed that the crystal structure and microstructure of oxide layers formed on different metal grains were obviously different, and the scattering of m-ZrO_2 columnar grain orientations in the oxide layers formed near basal plane (0001) was wider than that near prismatic plane (1010) and (1120). Besides the majority of m-ZrO_2, c-ZrO_2, t-ZrO_2 and sub-oxide phase Zr_3O were also detected at the oxide/metal interface, and it showed that the microstructure and crystal structure of oxide layers were very complex. The microstructural evolution of oxide layers will affect the diffusion of oxygen and subsequently the growth of oxide. Therefore, the microstructural evolution of oxide layers, which was affected by the different microstructure of oxide layers formed initially on grains and the water chemistry of corrosion tests, resulted in the anisotropic growth of oxide layers when Zr-4 specimens were corroded in LiOH aqueous solution in subsequent corrosion tests.

KEY WORDS Zr-4, corrosion resistance, anisotropic growth, oxide layer, microstructure

Introduction

Zirconium alloys are widely used as fuel cladding materials in water-cooled nuclear reactors due to their low thermal neutron absorption cross-section and excellent corrosion resistance and mechanical properties in high-temperature, high-pressure water. In pressurized water reactors, HBO_3 is added to the primary coolant to utilize ^{10}B as a burnable poison, while LiOH is added to adjust the pH value. However, the addition of LiOH adversely affects the corrosion resistance of zirconium alloys, which has attracted particular attention and extensive research [1]. With the development of nuclear technology, deeper fuel burnup and longer refueling cycles are required, necessitating the development of new zirconium alloys with improved corrosion resistance on the water side. Research on the corrosion resistance of zirconium alloys and deeper understanding of the corrosion mechanism can help guide the research and development of new zirconium alloys.

Zirconium alloys have an hcp crystal structure, and numerous studies [2-8] have investigated their corrosion anisotropy. Kim et al. [2] studied the corrosion behavior of small pure Zr single crystals in water at 360 ℃ and 18.6 MPa, finding that the oxide film grew significantly faster on the (1120) plane than on the (0001) plane. Sun et al. [5] investigated the growth characteristics of oxide films on three different surfaces of Zr-4 fine-grained samples with rolling texture corroded in LiOH aqueous solution. After 90 days of corrosion, they observed that the oxide growth rate on the rolling plane (SN) with the largest (0001) texture factor increased sharply, showing poor corrosion resistance, while on the cross sections perpendicular to the rolling direction (SR) and transverse direction (ST) with smaller texture factors, the oxide grew more slowly without a transition in growth rate, demonstrating better corrosion resistance. Zhou et al. [8] studied the relationship between oxide growth characteristics and grain surface orientation on large-grained (0.2-0.8 mm) Zr-4 samples under different temperatures and water chemistry conditions, finding significant anisotropy in oxide growth in superheated steam at 500 ℃ and LiOH aqueous solution at 360 ℃, though this anisotropy varied with corrosion conditions. They suggested that oxide growth characteristics are related to the evolution of the oxide’ s microstructure, which is influenced by the microstructural features of the oxide formed early at the oxide/metal (O/M) interface.

When pure Zr oxidizes, monoclinic zirconia (m-ZrO_2) is the only stable oxidation product below approximately 1200 ℃ [9]. However, when zirconium alloys corrode in high-temperature, high-pressure water, the oxide film contains not only large amounts of m-ZrO_2 but also small amounts of tetragonal zirconia (t-ZrO_2), particularly near the O/M interface [10-15]. This phenomenon is related to factors that can stabilize t-ZrO_2 at room temperature, including stress, grain size, oxygen vacancies, and alloying elements [9,16-19]. Additionally, extensive research has been conducted on the crystal orientation of oxide films formed on zirconium alloys [20-22], revealing that the oxide film is textured. During oxidation of zirconium alloys, oxygen ions must diffuse through the oxide film to reach the O/M interface, and the texture of the oxide film can influence oxygen ion diffusion and oxide growth. Therefore, studying the differences in microstructure of oxide films formed on metal grains with different orientations is important for understanding the origin of anisotropic oxide growth.

This work employed large-grained Zr-4 samples to investigate the relationship between oxide growth characteristics and grain surface orientation during corrosion in LiOH aqueous solution at 360 ℃. Three grains with surface orientations near (0001), (1010), and (1120) were selected to observe the microstructure at the O/M interface, aiming to study the effect of metal grain surface orientation on the initial microstructure of the oxide film and further understand the mechanism of anisotropic oxide growth during zirconium alloy corrosion.

1 Experimental Methods

To investigate the relationship between oxide thickness, microstructure, and crystal orientation, large-grained samples were prepared to minimize interference from grain boundaries. This experiment employed β-phase heating and quenching followed by high-temperature annealing in the α-phase region to obtain large grains larger than 0.5 mm. Following the experimental method in reference [8], 0.9 mm thick Zr-4 plates were cut into samples, pickled in a mixed acid solution (10% HF + 45% HNO_3 + 45% H_2O, by volume), rinsed with tap water and deionized water, and dried with a hair dryer. The samples were then vacuum-sealed (10^{-3} Pa) in quartz tubes, heated at 1020 ℃ for 30 minutes, and water-quenched by rapidly immersing the quartz tubes in water while breaking them. The blue oxide film formed on the sample surface during quenching was removed with fine sandpaper, and the samples were pickled, cleaned, dried, and resealed in quartz tubes for vacuum annealing at 800 ℃ for 10 hours to obtain large-grained samples with grain sizes of 0.2-0.8 mm. The samples were then vacuum-annealed at 700 ℃ for 100 hours to reduce the supersaturated solid solution content of alloying elements in α-Zr.

Corrosion experiments were conducted in a static autoclave at 360 ℃ and 18.6 MPa in 0.01 mol/L LiOH aqueous solution for 70 and 160 days. After corrosion, samples were embedded in Zr tubes (30 mm height, 10 mm diameter) using eutectic Pb-Bi alloy. Cross-sectional samples for scanning electron microscopy (SEM) were prepared by low-speed sawing, followed by grinding with sandpaper, mechanical polishing, and final chemical polishing with the aforementioned mixed acid to remove the deformed layer on the cross-section. An Apollo300 SEM equipped with electron backscatter diffraction (EBSD) was used to determine the orientations of different metal grains on the cross-section and measure the corresponding oxide thicknesses.

A sample was cut from the specimen corroded for 70 days using a low-speed saw. The oxide film on one side was ground away with sandpaper until the thickness reached approximately 0.1 mm. The metal substrate side was chemically polished with the mixed acid to obtain a relatively flat metal surface with sufficient thickness. After cleaning with deionized water and drying, EBSD was used to determine the orientations of grains on the surface. Three grains with surface orientations near (0001), (1010), and (1120) were selected, and Helios 600i dual-beam (ion and electron) focused ion beam (FIB) was used to prepare thin cross-sectional samples of the oxide film on these three large grains. A JEM-2010F high-resolution transmission electron microscope (HRTEM) was employed to study the microstructure and crystal structure of the oxide films.

2 Results and Discussion

2.1 Relationship Between Oxide Film Thickness and Metal Grain Surface Orientation

After corroding Zr-4 large-grained samples in 0.01 mol/L LiOH aqueous solution at 360 ℃ and 18.6 MPa for 70 days, the thicknesses of oxide films on 88 metal grains were measured, ranging from 1.5 to 3.0 m. The oxide thicknesses were divided into two categories: greater than and less than 2.5 m, and their relationship with metal grain surface orientation is shown in the inverse pole figure in [Figure 1: see original paper]a. The figure reveals that due to the small variation in oxide thickness, the anisotropic growth characteristic was not yet obvious, though the oxide film was slightly thicker on grains with surface orientations near (0001). After 160 days of corrosion, oxide thicknesses on 87 metal grains were measured, ranging from 3.0 to 50 m. The thicknesses were categorized into three levels: 20.0–50.0 m, 5.0–7.0 m, and less than 4.5 m. [Figure 1: see original paper]b shows the relationship between oxide thickness and metal grain orientation. After 160 days of corrosion, oxide films were relatively thicker on metal grains with surface orientations near (0001), with some grains showing oxide films that had changed color from black to earthy yellow, as shown in [Figure 2: see original paper]a. [Figure 2: see original paper]b shows a cross-sectional SEM image of this earthy yellow oxide film, with a measured thickness of approximately 50 m, about ten times thicker than the oxide film on adjacent grains.

Sun et al. [5] observed that for fine-grained Zr-4 samples with rolling texture corroded in LiOH aqueous solution at 360 ℃, the oxide growth rates on the three different surfaces were similar before the transition occurred at approximately

90 days. However, after the transition, the oxide growth rate on the rolling plane (SN) with the largest (0001) texture factor increased sharply, showing poor corrosion resistance. Before the transition, there was little difference in oxide thickness among SN, SR, and ST surfaces, and the anisotropic growth characteristic was not evident, consistent with the results obtained after 70 days of corrosion ([Figure 1: see original paper]a). After the transition, the anisotropic characteristic became increasingly pronounced, as shown in [Figure 1: see original paper]b after 160 days of corrosion, where oxide growth rates increased on metal grains with surface orientations near (0001). Consequently, the oxide growth rate on SN with a larger texture orientation factor increased, and the difference in oxide thickness between SN and SR/ST surfaces gradually increased, demonstrating prominent anisotropic oxide growth.

2.2 Effect of Metal Grain Surface Orientation on Oxide Film Microstructure

Based on the differences in oxide film thickness on grains with different orientations after 160 days of corrosion, three grains with surface orientations near (0001), (1010), and (1210) were selected from the sample corroded for 70 days. Cross-sectional TEM thin samples of the oxide films were prepared by FIB to observe the microstructure, as shown in [Figure 3: see original paper]. Comparing the selected area electron diffraction (SAED) patterns in [Figure 3: see original paper]a–c reveals that the oxide film formed on the (0001) plane showed relatively large misorientation between m-ZrO_2 columnar grains, forming an almost continuous “eyebrow-shaped” diffraction ring in the SAED pattern ([Figure 3: see original paper]a). In contrast, the oxide films formed on (1010) and (1210) planes showed more consistent orientations among m-ZrO_2 columnar grains, producing only intermittent “eyebrow-shaped” diffraction spots in the SAED patterns. [Figure 3: see original paper]d–f show dark-field TEM images of m-ZrO_2 columnar grains in oxide films formed on (0001), (1010), and (1210) planes, respectively. The columnar grains in [Figure 3: see original paper]d are more scattered and disconnected, while those in [Figure 3: see original paper]e and f form a continuous layer with consistent orientation. These observations agree well with the results of Du et al. [23], who studied the microstructure of thin oxide film samples from Zr-4 large-grained specimens corroded in LiOH aqueous solution for 5 hours. The results demonstrate that even before the corrosion transition, the microstructure of oxide films formed on metal grains with different orientations showed significant differences.

2.3 Crystal Structure, Defects, and Strain in Oxide Films

[Figure 4: see original paper] shows HRTEM images and fast Fourier transform (FFT) patterns at and near the O/M interface of the oxide film formed on the (1010) plane. The microstructure and crystal structure at the O/M interface are extremely complex, with most grains lacking clear grain boundaries. Dashed lines in the figure roughly delineate different grains or zirconia with different crystal structures. FFT analysis revealed that besides m-ZrO_2, cubic zirconia (c-ZrO_2), t-ZrO_2, and sub-oxide Zr_3O were also present at the O/M interface. Both t-ZrO_2 and c-ZrO_2 are unstable at room temperature and represent metastable phases. The Pilling-Bedworth (P.B.) ratio for zirconium oxidation to zirconia is 1.56, generating substantial compressive stress in the oxide film. To partially relax this stress, numerous defects—including vacancies, interstitial atoms, and dislocations—form in the zirconia crystals, and the large compressive stress can also stabilize these metastable phases [24]. The Zr_3O in [Figure 4: see original paper]a can be divided into two regions, I-Zr_3O and II-Zr_3O, which should belong to the same grain. Due to large compressive stress at the interface and the influence of Moiré fringes from crystal stacking, the lattice fringe images in these two regions show obvious differences. Some originally extinct spots reappear in the FFT patterns due to lattice distortion, resulting in different spot distributions between the two regions.

Inverse FFT (IFFT) of the (200) and (111) planes of c-ZrO_2 in the boxed area in [Figure 4: see original paper]b is shown in [Figure 5: see original paper]a and b. The lattice fringe images reveal numerous edge dislocations (indicated by arrows) and severe crystal distortion. Geometric phase analysis (GPA) was used to analyze the strain state in [Figure 5: see original paper]a and b, yielding [Figure 5: see original paper]c and d, where blue (green) and red represent compressive and tensile strain regions, respectively. Regions where color changes abruptly from blue (green) to red typically correspond to dislocation cores, matching the positions indicated by arrows in [Figure 5: see original paper]a and b. When edge dislocations exist in a crystal, the surrounding lattice undergoes elastic distortion, with atoms on the side of the extra half-plane experiencing compressive strain and those on the opposite side experiencing tensile strain. Overall, the strain field analysis shows that the [111] direction experiences greater strain and more severe distortion than the [100] direction in this region. These IFFT and GPA results demonstrate that the oxide film has not only complex microstructure and crystal structure but also intricate defect, stress, and strain states. These complex factors interact and cause continuous evolution of the oxide microstructure during corrosion, directly affecting oxygen diffusion and thus influencing the oxide growth rate.

2.4 Influence of Oxide Film Microstructural Evolution on Oxide Growth Characteristics

The oxidation process of Zr always occurs at the O/M interface, and the rate of oxygen ion diffusion through the oxide film to the O/M interface becomes the determining factor for oxide growth in the later stages of corrosion. Therefore, the evolution of oxide microstructure with increasing corrosion time plays a crucial role in the alloy’ s corrosion resistance. This evolution is influenced not only by temperature and water chemistry conditions but also by the microstructural characteristics of the oxide formed early at the O/M interface [25,26]. When metallic Zr oxidizes to zirconia, substantial compressive stress is generated in the oxide film, while the metal substrate in contact with the oxide experiences tensile stress [27]. Under these conditions, the zirconia crystals contain numerous defects, as confirmed by the experimental results above ([Figure 4: see original paper] and [Figure 5: see original paper]). These defects can diffuse and coalesce under temperature and stress, easily forming pores at zirconia grain boundaries. Under stress, these pores further develop into microcracks, accelerating oxygen diffusion and relaxing compressive stress in the oxide film, ultimately affecting oxide growth and the alloy' s corrosion resistance [24]. Geng et al. [28] studied the stress changes in oxide films 0.5–2.5 m thick during zirconium alloy corrosion under different temperatures and water chemistry conditions, observing that compressive stress in the oxide film decreased with increasing oxide thickness, confirming that the oxide microstructure indeed evolves during growth.

Furthermore, oxide evolution is related to water chemistry conditions. Zhou et al. [8] found that Zr-4 large-grained samples showed obvious anisotropic oxide growth when corroded in 0.01 mol/L LiOH aqueous solution at 360 ℃ and 18.6 MPa, but not in deionized water under the same conditions. By studying the surface undulations of oxide films and the development of pores and microcracks during zirconium alloy corrosion in LiOH aqueous solution, they proposed that Li^+ ions from the solution penetrate the oxide film and adsorb on pore walls, reducing the surface free energy of zirconia [24]. This promotes pore formation and microcrack development, accelerating the evolution of oxide microstructure and oxide growth. Geng et al. [28] also found that the compressive stress in oxide films formed on Zr-4 samples corroded in 0.01 mol/L LiOH aqueous solution at 360 ℃ and 18.6 MPa was much lower than that in deionized water under the same temperature, indicating that the oxide microstructure evolves more readily during corrosion in LiOH aqueous solution, resulting in more pronounced stress relaxation.

The above discussion indicates that the evolution of oxide microstructure during corrosion is inevitable due to the numerous defects and large internal stresses generated during oxide formation. If the microstructural characteristics of the oxide formed at the O/M interface also differ due to the orientation of the substrate metal grains, these differences can be superimposed on the evolution process, potentially leading to anisotropic oxide growth. The oxide film formed on the (0001) plane showed relatively large misorientation between m-ZrO_2 columnar grains, while those on (1010) and (1210) planes showed more consistent orientations ([Figure 3: see original paper]). Consequently, when the oxide microstructure evolves, more pathways favorable for oxygen ion diffusion will develop in the oxide formed on the (0001) plane, accelerating oxide growth. This effect becomes more pronounced when samples are corroded in LiOH aqueous solution, as Li^+ penetration accelerates microstructural evolution. This explains why anisotropic oxide growth is more evident in LiOH aqueous solution than in deionized water, and why this process only becomes apparent after the oxide reaches a certain thickness and internal stress accumulates to a certain level. [Figure 6: see original paper] shows fracture morphologies of oxide films with different thicknesses after 160 days of corrosion. The thicker oxide film (~35 m) in [Figure 6: see original paper]a contains numerous pores and microcracks ([Figure 6: see original paper]b), while the thinner oxide film (~4.4 m) in [Figure 6: see original paper]c has fewer pores and microcracks and remains relatively dense ([Figure 6: see original paper]d). Combining the results from [Figure 1: see original paper] and [Figure 3: see original paper], it is evident that during long-term corrosion in LiOH aqueous solution, the evolution and its consequences are influenced by the initial microstructural characteristics of the oxide formed on grains with different orientations. Oxide films with columnar grains having small orientation differences and fewer defects evolve slowly, hindering oxygen diffusion and resulting in slower oxide growth. Conversely, oxide films with large orientation differences and more defects among columnar grains develop more pores and microcracks, facilitating oxygen diffusion and accelerating oxide growth. This accelerated growth, in turn, promotes further stress accumulation, driving additional microstructural evolution and creating more pores and microcracks, leading to even faster oxide growth. In this process, Li^+ penetration and adsorption on pore and microcrack surfaces reduces the surface free energy of zirconia, further accelerating microstructural evolution and amplifying the differences between oxide films with initially different microstructures. Ultimately, with increasing corrosion time, the evolution of oxide microstructure leads to pronounced anisotropic oxide growth.

Conclusions

1. When Zr-4 large-grained samples were corroded in 0.01 mol/L LiOH aqueous solution at 360 ℃ and 18.6 MPa, the anisotropic growth characteristic was not obvious after 70 days, but became prominent after 160 days, with oxide films being relatively thicker on metal grains with surface orientations near (0001).
2. After 70 days of corrosion, differences existed in the microstructure of oxide films formed on metal grains with different orientations. The oxide film formed on the (0001) plane showed relatively large misorientation between m-ZrO_2 columnar grains, forming an almost continuous "eyebrow-shaped" diffraction ring in SAED patterns, while those on (1010) and (1210) planes showed more consistent orientations among m-ZrO_2 columnar grains, producing only intermittent "eyebrow-shaped" diffraction spots.
3. The microstructure and crystal structure near the oxide/metal interface were very complex. Although m-ZrO_2 was the main phase, c-ZrO_2, t-ZrO_2, and sub-oxide Zr_3O were also present. The zirconia crystals contained numerous dislocations, severe lattice distortion, and complex strain distribution.
4. The continuous evolution of oxide microstructure during corrosion affects oxygen diffusion and thus influences oxide growth. This evolution is af-

fected by both the initial microstructural differences of oxide films formed on grains with different orientations and the water chemistry conditions during corrosion, which is the main reason for the anisotropic oxide growth observed when Zr-4 alloy is corroded in LiOH aqueous solution.

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