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# Preparation of Aluminum Oxide Films Under Water Exposure – Preliminary Report on 6061 Series Alloys

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# Preparation of Aluminum Oxide Films Under Water Exposure – Preliminary Report on 6061 Series Alloys

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# EXECUTIVE SUMMARY

Aluminum fuel cladding will undergo general corrosion with resulting formation of adherent oxide films during in-reactor and post-discharge exposures under various water conditions and temperatures. The presence of oxides with chemically-bound water can pose challenges for extended dry storage, and an action plan to identify needed technical and engineering activities and analyses to address the identified knowledge gaps and technical data needs was previously prepared [1]. This report is part of the Task 6 activities of that plan.

This report describes laboratory experiments performed to produce hydroxide (oxide) films on aluminum 6061 series alloy substrates immersed in water at controlled temperatures. The experimental set-up and characterization results to determine the oxide morphology, thickness, and chemical composition are described.

The general observations and findings are as follows:

- Trihydroxide films of gibbsite/bayerite were the predominant types formed in hot-wall exposure at 50°C. The weight gain was approximately 10.8 $\mu g/mm^2$ of corroded specimen area for a 31-day exposure. The observed film thickness ranged from approximately 1 to 2 µm, depending on the local region examined.
- Boehmite film was the predominant type formed in hot-wall exposure at 100°C. The weight gain was approximately 1.14 $\mu g/mm^2$ of corroded specimen area for a 29-day exposure. The observed film thickness ranged from approximately 12 to 15 µm, depending on the local region examined
- The oxide films from the hot-wall tests were subject to additional growth post-hot-wall test during wet storage in room-temperature water. This is attributed to the hot-wall formed oxides being non-protective compared to the intentional "pre-film" oxides and allowing continued corrosion to occur.
- "Pre-filming" exposures in 185°C liquid water were performed to compare to the pre-filming practice used for ATR fuel. The exposures from 9 to 72 hours followed a literature model for oxide formation. Exposure for the ATR pre-film duration of 18 hours yielded an estimated oxide thickness of 2.0 µm, consistent with reported ATR pre-filming results [2].

The full set of data from the aluminum cladding laboratory exposure tests will be compared to future characterization work under Task 6 using specimens from aluminum cladding alloys service-experienced with reactor and post-discharge exposure.

For clarity, this report uses the term "oxide" to refer to all oxidation products of aluminum, including $Al_2O_3$, $Al(OH)_3$, and AlOOH and "hydroxide" to collectively refer to the hydroxyl-containing oxides $Al(OH)_3$ and AlOOH. The specific compositions are referred to as "trihydroxide" for $Al(OH)_3$ (including polymorphs bayerite and gibbsite) and "oxyhydroxide" for AlOOH (with polymorphs including boehmite and diaspore).[a]

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[a] This nomenclature is the authors' preference based on literature usage. However, this usage is not well-established convention. Previous reports in this project have used the term "oxyhydroxides" for all aluminum compounds that have chemically-bound water that could be released at sufficiently high temperature.

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| | |
|---|---|
| ASNF | Aluminum-clad spent nuclear fuel |
| ATR | Advanced Test Reactor |
| SRNL | Savannah River National Laboratory |
| SEM | Scanning electron microscopy |
| XRD | X-ray diffraction |

# 1.0 Introduction

This report describes laboratory experiments performed to produce hydroxide (oxide) films on aluminum 6061 series alloy substrates immersed in water at controlled temperature. The aluminum alloys considered in this program are alloys used in aluminum-clad research reactor fuel, and the water and temperature conditions are selected to produce film structures representative of those formed on fuel cladding as a result of reactor exposure and/or post-discharge wet or dry storage.

This report documents the experimental set-up and characterization results to determine the oxide morphology, thickness, and chemical composition. The results from this work will be compared to oxide characterizations of fuel cladding materials that have been exposed to actual reactor and post-discharge service conditions. The aim is to identify the characteristics of hydroxide films produced under conditions of interest for prefilming, in-reactor and/or wet storage conditions to aid in understanding their response under drying and radiation conditions.

The work in this report comprises interim results and preliminary information prepared under Task 6, Milestone 6.3 in TTQAP SRNL-RP-2018-00610. Additional experimental milestones are in progress and will be reported upon completion.

For clarity, this report uses the term "oxide" to refer to all oxidation products of aluminum, including $Al_2O_3$, $Al(OH)_3$, and AlOOH and "hydroxide" to collectively refer to the hydroxyl-containing oxides $Al(OH)_3$ and AlOOH. The specific compositions are referred to as "trihydroxide" for $Al(OH)_3$ (including polymorphs bayerite and gibbsite) and "oxyhydroxide" for AlOOH (with polymorphs including boehmite and diaspore).

# 2.0 Background

## 2.1 ATR prefilming procedure

Fuel elements for the Advanced Test Reactor (ATR) are prefilmed to produce a layer of boehmite intended to serve as a protective barrier against further corrosion in the reactor and subsequent storage. The fuel elements are immersed in an autoclave filled with distilled water at a temperature of 185°C (±8°C) for 18 h (+2 h/–0 h), as documented in Ref. [2, 3]. This procedure was found to produce boehmite film thicknesses averaging 2.35 μm, measured by SEM [2].

# 3.0 Experimental Procedure

## 3.1 Hot-wall immersion tests

Hydroxide layers were grown on aluminum substrates primarily using a "hot-wall" immersion method, set up as shown in Figure 3-1. This setup supplies heat to the water bath through the aluminum specimen, providing a heat flux across the sample from one face to the other, and it is suitable for replicating conditions in which heat flows in a prescribed direction, such as heat transferred through aluminum fuel cladding in-reactor or during spent fuel pool storage due to decay heat. The magnitude of the heat flux is not imposed at a set value, but instead varies as needed to maintain the surface of the alloy exposed in the bath at the specified temperature. Growth tests spanned temperatures from 50°C to 100°C to grow both low- and high-temperature hydroxides (bayerite or gibbsite and boehmite, respectively).

In the hot-wall setup, only one face of the substrate is exposed to the water, and the outer edge of the specimen disk is protected from water exposure by the specimen holder. The specimen disks are 3 inches in diameter, with a 2.5-inch-diameter area exposed to the water on one face. Therefore, the exposed surface area for oxide formation is taken as $A_{HW} = \pi(2.5\text{ in}/2)^2 = 31.7\text{ cm}^2$.

To assess the effect of the heat flux on hydroxide growth, isothermal immersion tests were conducted for some samples. The isothermal tests were conducted in parallel with the equivalent hot-wall tests by fully immersing a second aluminum substrate of the same geometry in the water-bath portion of the hot-wall apparatus, as shown in Figure 3-1 (right). Thus, a pair of specimens was exposed to the same water bath conditions, one hot-wall specimen with a heat flux through it and a single face exposed to the water and one isothermal specimen with no heat flux through it and all surfaces exposed to the water.

All of the hot-wall and parallel isothermal immersion tests were conducted by immersion in water at atmospheric pressure. The aluminum specimens were weighed prior to immersion testing. Following immersion testing, the specimens were removed from the apparatus, dried, and weighed again. They were then sectioned into multiple samples for further characterization, including scanning electron microscopy (SEM) of both the planar and cross-section views and X-ray diffraction (XRD) analysis to identify the oxide formed.

**Figure 3-1. Experimental setup for hot-wall immersion experiments, showing the locations of (left) the hot-wall samples and (right) the optional isothermal immersion samples.**

### 3.2 Pressurized immersion tests/ATR prefilming replication

Additional isothermal immersion tests were conducted in small pressure vessels (Parr vessels) placed inside a furnace, in order to test liquid-water exposure at higher temperature. The primary motivations were to (1) replicate the ATR prefilming procedure performed in an autoclave at 185°C for 18 h [3] and (2) to compare the hydroxides produced by liquid water immersion at high temperature with those produced by water vapor at similar temperature [4] in order to better understand the oxidation behavior.

The specimens were immersed in water inside the Parr vessels, heated to temperature in a furnace, and removed after a designated exposure time. The samples were loaded into the vessels the day before testing, resulting in approximately 12 hours immersion in room-temperature water prior to the high-temperature exposure. After cooldown, the samples remained immersed at room temperature for between 1 and 24 hours before removal from the vessel. There was no external pressure control imposed; the vessels contained sufficient water to self-pressurize to the saturation pressure of water at the set temperature.

### 3.3 Estimated conversion between weight gain and oxide thickness

Assuming all of the oxidation product remains in the exposed surface layer and the layer is dense and uniform, the expected relationship between sample weight gain per unit area (w) and the surface layer thickness (x) can be estimated as

$$\mathbf{x} = \left[\frac{\mathbf{w}}{(\mathbf{M_{ox}} - \mathbf{M_{Al}})}\right]\left[\frac{\mathbf{M_{ox}}}{\boldsymbol{\rho}_{\mathbf{ox}}}\right] \tag{1}$$

where $\mathrm{M_{Al}} = 27\ \mathrm{g/mol}$ and $\mathrm{M_{ox}}$ are the molar mass of aluminum metal and of the hydroxide ($Al(OH)_3$ or AlOOH), respectively, and $\rho_{\mathrm{ox}}$ is the density of the hydroxide. Here, the ratio on the left calculates the number of moles of hydroxide produced per unit area (in mol/m$^2$) based on the weight gain, and the ratio on the right is the molar volume of the (dense) hydroxide (in m$^3$/mol).

## 4.0 Results and Discussion

### 4.1 Atmospheric pressure immersion tests

Table 4-1 summarizes the weight gain data for 6061 hot-wall tests. The post-hot-wall weights were taken immediately after removal from the hot-wall apparatus, prior to storage and additional characterization. The weight gain per unit area was computed based on the area exposed to the water, i.e., $\mathrm{A_{HW}} = \pi(2.5\ \mathrm{in}/2)^2 = 31.7\ \mathrm{cm}^2$ for hot-wall tests.

**Table 4-1. Weight gain data for hot-wall specimens.**

| Specimen | Temperature (°C) | Hot-wall exposure (days) | Initial weight (g) | Post-hot-wall weight (g) | Weight gain (g) | Weight gain per unit corrosion area *w* (μg/mm$^2$) |
|---|---|---|---|---|---|---|
| 6061-50-121717 | 47-52 | 31 | 40.1456 | 40.1798 | 0.0342 | 10.8 |
| 6061-100-111118 | 98-101 | 20 | 40.1560 | 40.1596 | 0.0036 | 1.14 |

#### *4.1.1 Hot-wall immersion: 6061 at 50°C for 31 days*

Figure 4-1, Figure 4-2, and Figure 4-3 show characterization results for a 6061-alloy hot-wall specimen exposed at 50°C for 31 days. The specimen was stored bagged in a desiccator between the immersion test and characterization. XRD indicated that the film consisted primarily of bayerite (Figure 4-1), with possible gibbsite present as well. These trihydroxides are the expected phases for 50°C exposure [5].

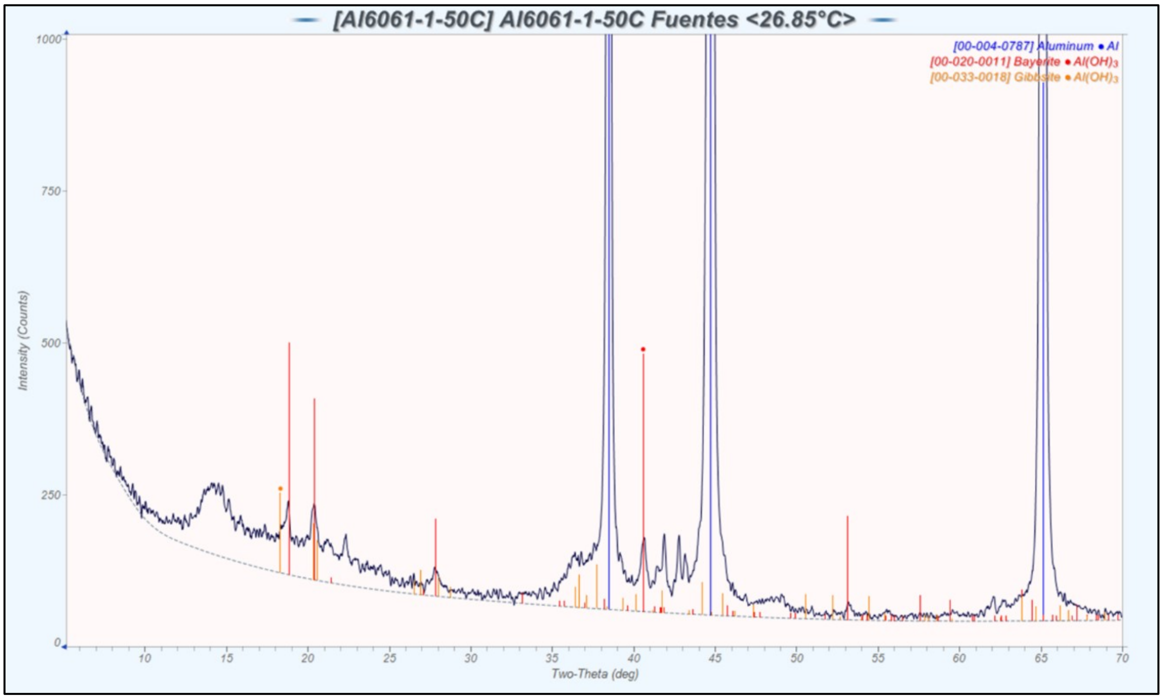

**Figure 4-1. XRD spectrum for a hot-wall sample of 6061 alloy exposed to 50°C water for 31 days, followed by 23 days bagged storage inside a desiccator prior to characterization. The hydroxide layer primarily consists of bayerite, with possible gibbsite.**

SEM images of the surface morphology (Figure 4-2) show an overall indistinct and “cloudy”-looking appearance. The sample displays some “needle-like” features, similar to those observed for 100°C water exposure in literature studies [6] and previous experimental results for 1100 alloy [7] but much less distinct. The morphology shown in Figure 4-2 may reflect a lower-magnification view of a “spongelike” morphology similar to that observed for shorter-term aqueous corrosion of nominally pure aluminum at 40–50°C (Figures 12 and 13 of Ref. [8]). Images of the film cross-section (Figure 4-3) indicate a total film thickness ranging from about 1 to 2 μm. The film appeared to consist of a single layer.

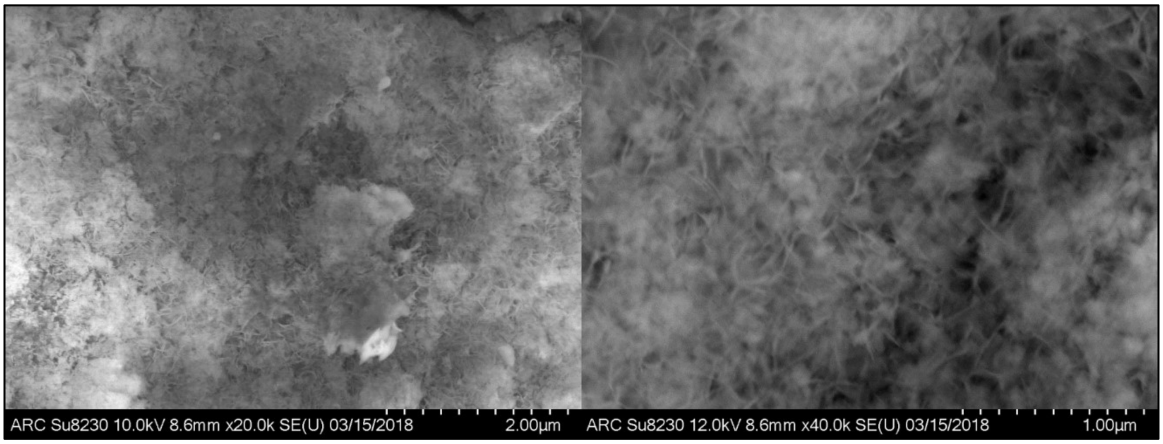

**Figure 4-2. SEM images of the surface for a hot-wall sample of 6061 alloy exposed to 50°C water for 31 days, followed by 57 days bagged storage in a desiccator prior to imaging, at two different levels of magnification (×20k on the left, and ×40k on the right). The hydroxide morphology includes some “needle-like” features interspersed with “cloudy”-looking indistinct regions.**

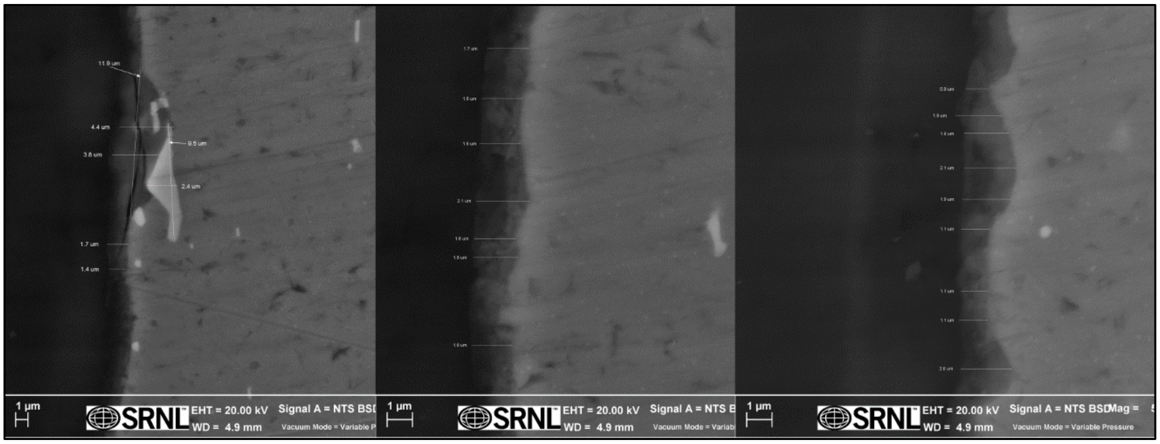

**Figure 4-3. SEM images of the cross-section for a hot-wall sample of 6061 alloy exposed to 50°C water for 31 days, followed by 77 days bagged storage in a desiccator prior to imaging. Total hydroxide layer thickness ranges from approximately 1 to 2 μm and appears to be a single layer.**

The surface morphology observed for the 6061 alloy at 50°C was very different than that for the 1100 alloy exposed under similar conditions (Figure 3 of Ref. [7]), which featured a scaly surface appearance and a thicker, apparently 2-layer film ranging from about 3–6 μm thick rather than 1–2 μm. The cause of the different structures is currently unknown.

For bayerite, $\mathrm{M}_{\mathrm{Al(OH)_3}} = 78$ and $\rho_{\mathrm{bayerite}} = 2.53\ \mathrm{g/cm^3}$ [9], and the estimated thickness from Equation (1) is 6.5 μm, significantly larger than even the thickest regions of the film observed in Figure 4-3. This is unexpected, since the apparent porosity of the film in the surface view (Figure 4-2) would be expected to lead to a thicker film for the same mass than the dense film assumed in the calculation. The cause of this discrepancy is not immediately apparent, but there are several possibilities. If the hydroxide was not completely composed of stoichiometric bayerite, then the calculation may underestimate the film density $\rho_{ox}$ and/or overestimate the ratio $M_{ox}/(M_{ox} - M_{Al})$, either of which would tend to overpredict the film thickness. Since the specimen was weighed immediately after immersion in water and featured an apparently porous film, it is also possible that some water may have remained on the film during weighing without being removed by the wipes, increasing the apparent weight gain.

### *4.1.2 Hot-wall immersion: 6061 at 100°C for 20 days, plus 29 days room-temperature immersion*

Figure 4-4, Figure 4-5, and Figure 4-6 show characterization results for a 6061-alloy hot-wall specimen exposed in 100°C water for 20 days, followed by immersion in room-temperature water for 29 days. It was expected that the boehmite film formed during the initial 20-day exposure would be protective against further corrosion in low-temperature water, and that corrosion in room-temperature water of even fresh metal surface would be slow compared to that at 50°C. XRD taken after 9 days of immersion in room-temperature water (Figure 4-4) indicate that the film consisted of boehmite.

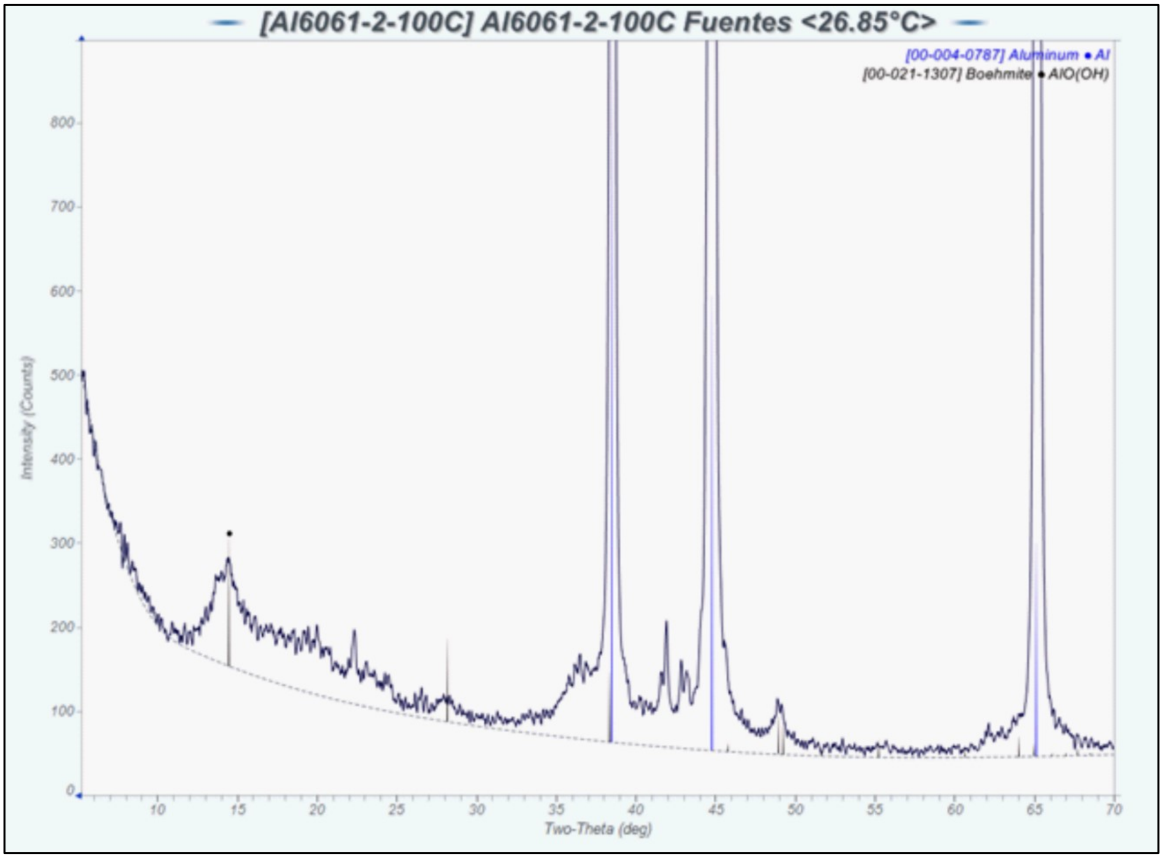

**Figure 4-4. XRD spectrum for a hot-wall sample of 6061 alloy exposed to 100°C water for 20 days, followed by 9 days in room-temperature water prior to characterization. The hydroxide layer primarily consists of boehmite.**

SEM of the surface morphology (Figure 4-5) showed a primarily needle-like appearance with a few isolated squarish, rather than elongated particles. These isolated squares could be either block-like particles of hydroxide or platelets oriented parallel rather than perpendicular to the surface. There also appear to be some relatively large fissures in the hydroxide (Figure 4-6, left).

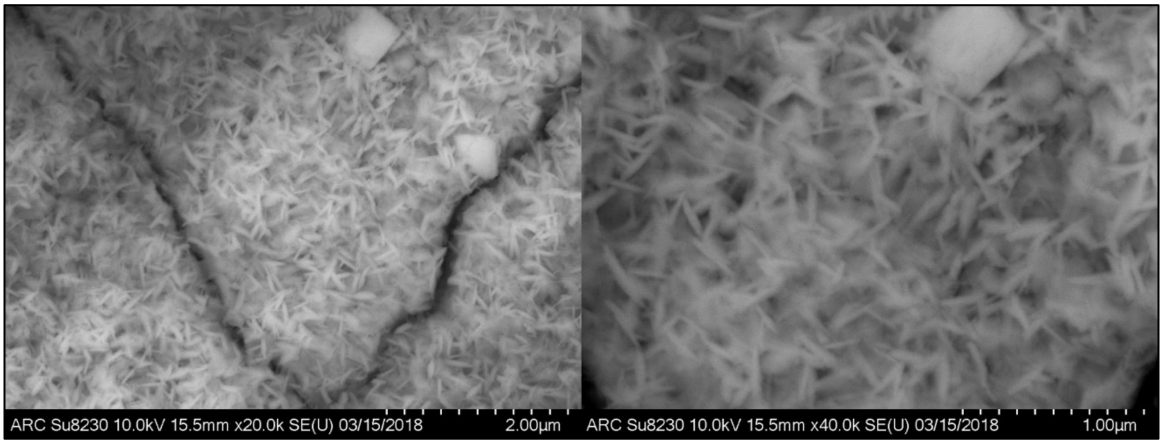

**Figure 4-5. SEM images of the surface for a hot-wall sample of 6061 alloy exposed to 100°C water for 20 days, followed by 29 days in room-temperature water and 14 days in bagged storage prior to imaging, at two different levels of magnification (×20k on the left, and ×40k on the right). The hydroxide has a predominantly ``needle-like'' appearance with scattered ``blocks'' of oxide.**

SEM images of the film cross-section (Figure 4-6) shows that this film is multi-layered and much thicker than those obtained in other experiments on 1100 [7] and 6061. The total hydroxide layer thickness ranges from approximately 10 to 15 µm. There appear to be two or three layers of oxide: a dense-looking, dark gray inner layer next to the metal; a highly porous, lighter-colored outer layer, and a region in between that appears dense but similar in color to the porous outer layer. This is significantly thicker than the previously observed film on 1100 at 100°C, which was approximately 0.5–2 µm thick after 41 days hot-wall exposure and appeared to be a single layer [7]. The middle and right-hand image both show dark cracks that appear to go completely through the outer layer at an angle and may correspond to the fissures visible on the surface. The middle image also shows cracks within the inner layer, approximately perpendicular to the plane of the surface, and apparent separation between the layers in the lower half of the image.

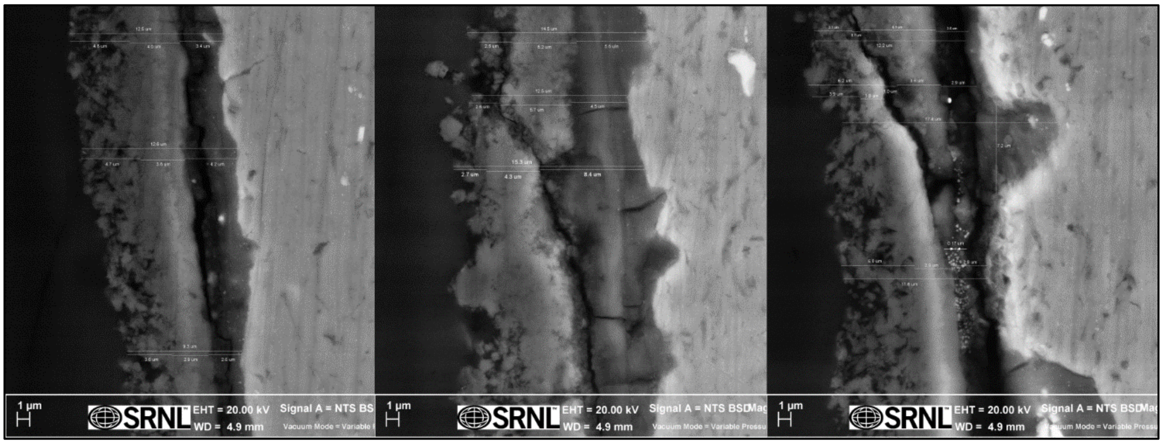

**Figure 4-6. SEM images of the cross-section for a hot-wall sample of 6061 alloy exposed to 50°C water for 20 days, followed by 29 days in room-temperature water and 34 days bagged storage prior to imaging. Total hydroxide layer thickness ranges from approximately 10 to 15 µm and appears to be two or three different layers.**

For boehmite, $M_{AlOOH} = 60$ and $\rho_{boehmite} = 3.01\ g/cm^3$ [9]. The estimated film thickness calculated from the post-hot-wall weight gain using Equation (1) is 0.69 µm, about 15–20 times thinner than the thickness observed by cross-section after the additional 29 days in room-temperature water. The total oxide thickness in Figure 4-6 was also much larger than that for roughly 30 days hot-wall exposure at 50°C for either 6061 (1–2 µm after 31 days, Figure 4-3) or 1100 (3–6 µm after 29 days [7]) and featured an apparent layered structure with partial separation between oxide layers, creating voids. In combination, these facts suggest that significant additional hydroxide growth may have occurred during room-temperature immersion, implying that the boehmite film formed during hot-wall exposure was not protective and may even have accelerated additional corrosion.

## 4.2 Pressurized immersion tests

Parr vessels were used to expose 4 samples each of 6061 and 1100 alloys to water at 185°C (130 psi) for four different times (i.e., 9, 18, 36 and 72 hours). Results for weight gain per unit area as a function of time are plotted in Figure 4-7 and listed in Table 4-2. Estimated dense oxide film thicknesses were calculated and reported in Table 4-2 using Equation (1) for boehmite (with $\mathrm{M_{AlOOH}} = 60$ and $\rho_{\mathrm{boehmite}} = 3.01$ $\mathrm{g/cm^3}$ [9]).

A power-law model from Ref. [4] was fitted to the data with model parameters chosen by minimizing the sum of squares using Solver in Excel. The model is expressed as

$$\boldsymbol{w = b \exp\left(-\frac{c}{T}\right) t^z} \tag{2}$$

where $w$ is the weight gain per unit area in µg/dm$^2$, $T$ is the temperature in K, $t$ is the time in h, and $b$, $c$, and z are fitted constants. The curve fits are shown in Figure 4-7 and the corresponding fitted model parameters and R$^2$ values are given in Table 4-3. For the four data points, the model appeared to fit the 6061 data well. The 1100 data were scattered and did not follow the parabolic trend; no immediate reason for the scatter is apparent. More data points, either additional exposure times or replications of the same conditions, would improve the model parameters.

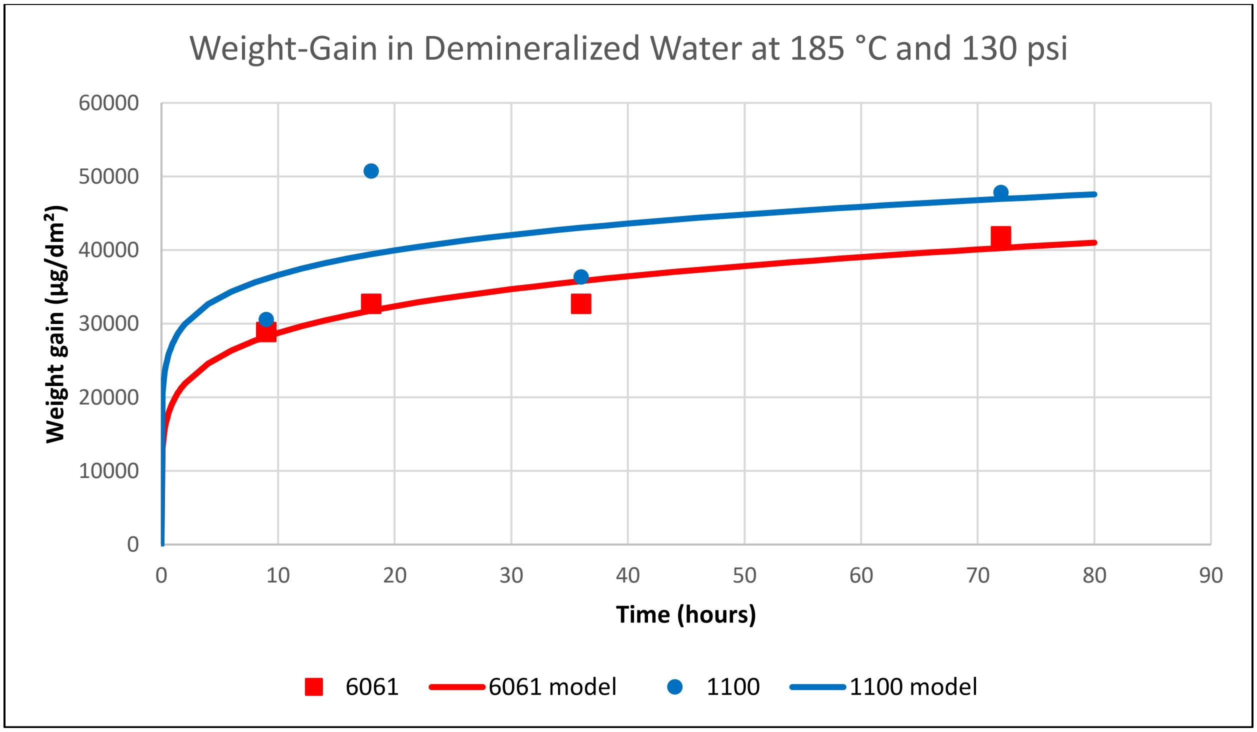

**Figure 4-7. Weight gain per unit area for specimens immersed in 185°C liquid water as a function of exposure time.**

**Table 4-2. Weight gain per unit area for specimens immersed in 185°C liquid water as a function of exposure time.**

| | **6061** | | **1100** | |
|---|---|---|---|---|
| **Time (hr)** | **Wt. Gain ($\mu g/mm^2$)** | **Calculated oxide film thickness (μm)** | **Wt. Gain ($\mu g/mm^2$)** | **Calculated oxide film thickness (μm)** |
| 9 | 2.89 | 1.75 | 3.06 | 1.85 |
| 18 | 3.27 | 1.98 | 5.08 | 3.07 |
| 36 | 3.27 | 1.98 | 3.64 | 2.20 |
| 72 | 4.19 | 2.53 | 4.78 | 2.89 |

**Table 4-3. Fitted parameters and $R^2$ values for the weight gain data in Figure 4-7 according to Equation (2) with temperature $T = 458$ K.**

| | **6061** | **1100** |
|---|---|---|
| b | $3.301\times10^6$ | $3.301\times10^6$ |
| c | 2352 | 2194 |
| z | 0.1707 | 0.1256 |
| $R^2$ | 0.85 | 0.25 |

From Table 4-2, the calculated thickness of a dense boehmite film based on the weight gain of 6061 after 18 h exposure time, corresponding to the ATR pre-filming duration, is 1.98 μm. This is 84% of the average film thickness of 2.35 μm reported for the ATR prefilming procedure and measured by SEM [2]. Direct observation of the oxide film from the Parr tests would aid in comparison. In particular, it is currently unknown whether there was any significant porosity in the ATR film, and porosity would tend to increase the film thickness for a given weight gain compared with the dense film assumed in the calculation.

## 5.0 Conclusions

Laboratory experiments were performed to produce and characterize hydroxide (oxide) films on aluminum 6061 series alloy substrates immersed in water at controlled temperatures. The experimental set-up and characterization results to determine the oxide morphology, thickness, and chemical composition are described in this report.

The general observations and findings are as follows:

- Trihydroxide films of gibbsite/bayerite were the predominant types formed in hot-wall exposure at 50°C. The weight gain was approximately 10.8 $\mu g/mm^2$ of corroded specimen area for a 31-day exposure. The observed film thickness ranged from about 1 to 2 μm, depending on the local region examined.
- The dense-film thickness estimated from the weight gain at 50°C significantly exceeded the film thickness observed by SEM, suggesting that excess water trapped in the film immediately after immersion testing may have skewed the weight measurement. As a result, drying to remove free and adsorbed water without thermally decomposing the hydroxide film water is being investigated to ensure accurate weight measurements.
- Boehmite was the predominant film type formed in hot-wall exposure at 100°C. The weight gain was approximately 1.14 $\mu g/mm^2$ of corroded specimen area for a 29-day exposure. The observed film thickness ranged from about 12 to 15 μm, depending on the local region examined.

- The hydroxide films from the hot-wall tests were subject to additional growth post-hot-wall exposure during wet storage in a room-temperature. This is assumed to be due to the hydroxide film being non-protective compared to the intentional “pre-film” oxides used in, e.g., ATR fuel.
- “Pre-filming” exposures to 185°C liquid water in pressure vessels were performed to compare to the pre-filming practice used for ATR fuel. The weight gain of 6061 samples from 9 to 72 hours were well fitted by a power-law model proportional to $t^{0.17}$. Exposure to the ATR pre-film time of 18 hours yielded an estimated oxide thickness (computed from weight gain) of 2.0 µm on the 6061 sample, consistent with ATR reported results of 2.35 µm average thickness (observed by SEM) [2].

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