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Preparation of Aluminum Oxide Films Under Water Exposure – Preliminary Report on 5052 Series Alloys

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November 2018

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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 due to their potential to decompose and release free water and/or generate gases (e.g., oxygen, hydrogen) due to radiolysis, which may cause corrosion, pressurization, or flammability conditions within the storage container. Therefore, 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 5052 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:

- The hydroxide phases observed were predominantly bayerite for 50°C oxide growth and boehmite for 100°C oxide growth, which are consistent with the literature [2] and with previous results for other aluminum alloys [3, 4].
- The calculated dense-film thicknesses corresponding to the measured weight gains were close to those measured from SEM images of the cross-section for both samples. For oxide grown at 50°C for 112 days, the calculated thickness of 2.4 μm fell slightly below the measured range of 2.7–5.8 μm , suggesting that the average film density may be lower than that of bulk bayerite, e.g., due to porosity. For the oxide grown at 100°C for 12 days, the calculated thickness of 0.85 μm fell within the measured thickness range of 0.4–2 μm (with thickness up to 5.6 μm in a localized pit), suggesting that the film may be nearly dense boehmite.
- The oxide grown at 50°C for 112 days featured a varied range of oxide morphologies. The surface was largely covered by dense-looking, blocky oxide particles, which appear to have formed on top of a “needle-like” oxide layer similar to that observed in previous tests at both 50 and 100°C [3, 4]. In some regions, these blocky particles are closely packed together with no “needle-like” morphology visible, while in other regions, isolated blocks are completely surrounded by the “needle-like” structure. SEM of the cross-section of the 50°C sample showed a two-layer oxide structure, supporting the hypothesis that the blocks sit on top of the “needle-like” layer instead of replacing it. This is consistent with the two-layer film observed for a previous sample that grew a dense-looking oxide film at 50°C [3].

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 , $\text{Al}(\text{OH})_3$, and AlOOH and “hydroxide” to collectively refer to the hydroxyl-containing oxides $\text{Al}(\text{OH})_3$ and AlOOH . The specific compositions are referred to as “trihydroxide” for $\text{Al}(\text{OH})_3$ (including polymorphs bayerite and gibbsite) and “oxyhydroxide” for AlOOH (with polymorphs including boehmite and diaspore).^a

^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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LIST OF ABBREVIATIONS

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

1.0 Introduction

This report describes laboratory experiments performed to produce hydroxide (oxide) films on aluminum 5052 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 6.4 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 , $\text{Al}(\text{OH})_3$, and AlOOH and “hydroxide” to collectively refer to the hydroxyl-containing oxides $\text{Al}(\text{OH})_3$ and AlOOH . The specific compositions are referred to as “trihydroxide” for $\text{Al}(\text{OH})_3$ (including polymorphs bayerite and gibbsite) and “oxyhydroxide” for AlOOH (with polymorphs including boehmite and diasporite).

2.0 Experimental Procedure

2.1 Hot-wall immersion tests

Hydroxide layers were grown on aluminum substrates using a “hot-wall” immersion method, set up as shown in Figure 2-1. 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 below about 80°C and boehmite above 80°C [2, 5]).

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 $A_{\text{HW}} = \pi(2.5 \text{ in}/2)^2 = 31.7 \text{ cm}^2$.

All of the hot-wall 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.

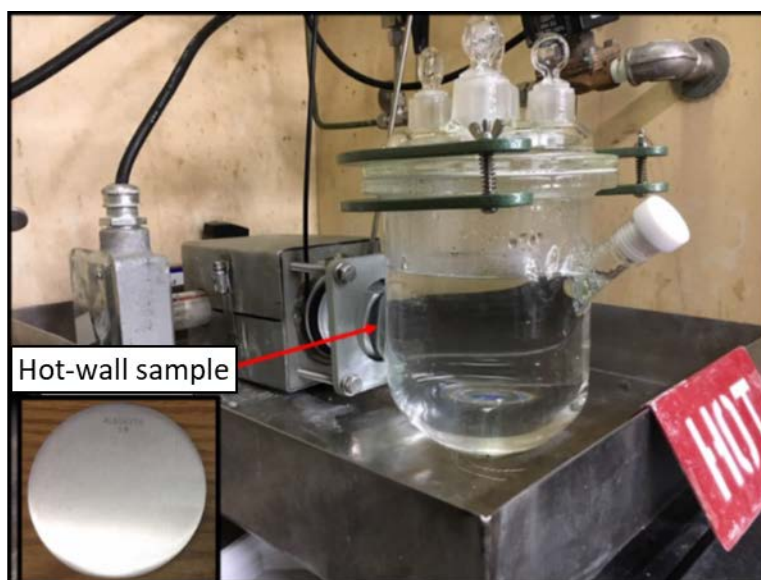


Figure 2-1. Experimental setup for hot-wall immersion experiments, showing the locations of a hot-wall specimen. Inset picture shows one of the discs used as samples.

2.2 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

$$x = \left[\frac{w}{(M_{ox} - M_{Al})} \right] \left[\frac{M_{ox}}{\rho_{ox}} \right] \quad (1)$$

where $M_{Al} = 27$ g/mol and M_{ox} are the molar mass of aluminum metal and of the hydroxide ($Al(OH)_3$ or $AlOOH$), respectively, and ρ_{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/m²) based on the weight gain, and the ratio on the right is the molar volume of the (dense) hydroxide (in m³/mol).

3.0 Results and Discussion

Table 3-1 summarizes the weight gain data for 5052 hot-wall tests. The post-hot-wall weights were taken immediately after removal from the hot-wall apparatus and preliminary drying, prior to storage and additional characterization. The weight gain per unit area was computed based on the area exposed to the water, i.e., $A_{HW} = \pi(2.5 \text{ in}/2)^2 = 31.7 \text{ cm}^2$ for hot-wall tests.

Table 3-1. 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)
5052-50-31518	48–53	112	34.9688	34.9813	0.0125	3.95
5052-100-31518	99–101	12	35.6025	35.6068	0.0043	1.4

3.1 Hot-wall immersion: 5052 at 50°C for 112 days

Figure 3-1, Figure 3-2, and Figure 3-3 show characterization results for a 5052-alloy hot-wall specimen exposed to water at a nominal surface temperature of 50°C (actual variation 48–53°C) for 16 weeks (112 days). This was the longest-duration hot-wall test to date, providing evidence of how the oxide formation process evolves with time (albeit still substantially shorter exposure than that of actual SNF). The specimen

was weighed immediately after removal from the water bath and preliminary drying of the sample and was subsequently stored bagged until further characterization.

The XRD spectrum shown in Figure 3-1 identifies the oxide as mostly bayerite with some minor peaks indicating the presence of some boehmite. The intensity of the bayerite peaks suggest the presence of a relatively thick bayerite structure. Trihydroxide (either gibbsite or bayerite) is the expected phase for 50°C exposure [2].

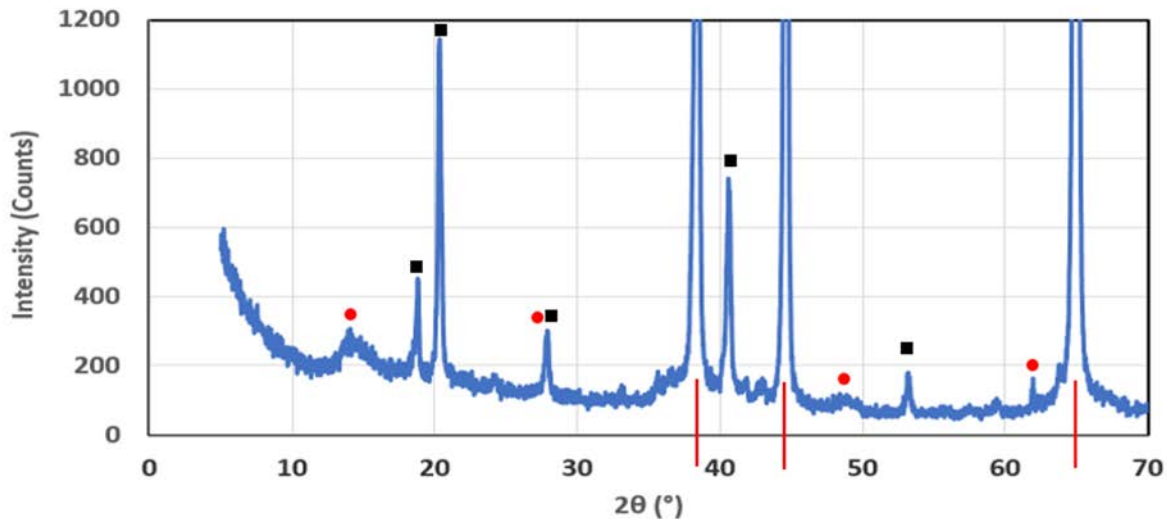


Figure 3-1. XRD spectrum of 112-day hot-wall growth on Al-5052 at 50°C. Red lines indicate locations of aluminum peaks, red circles indicate boehmite peaks, and black squares indicate bayerite peaks.

SEM images of the surface (Figure 3-2) show substantial variations in morphology over the surface. A “needle-like” morphology was observed in previous experimental results for Al-1100 [3] and Al-6061 [4] resulting from ~30-day exposure at 50°C and from 7 to 41-day exposures at 100°C. A similar structure appeared on patches of the present specimen, but with the addition of isolated, roughly cylindrical particles either on top of or embedded in the needle-like structure (Figure 3-2(a)–(c)). Other areas featured densely packed blocky particles of oxide with no needle-like structure visible (Figure 3-2(d)–(e)). There were also regions where irregularly shaped particles were surrounded by needle-like structure (Figure 3-2(f)).

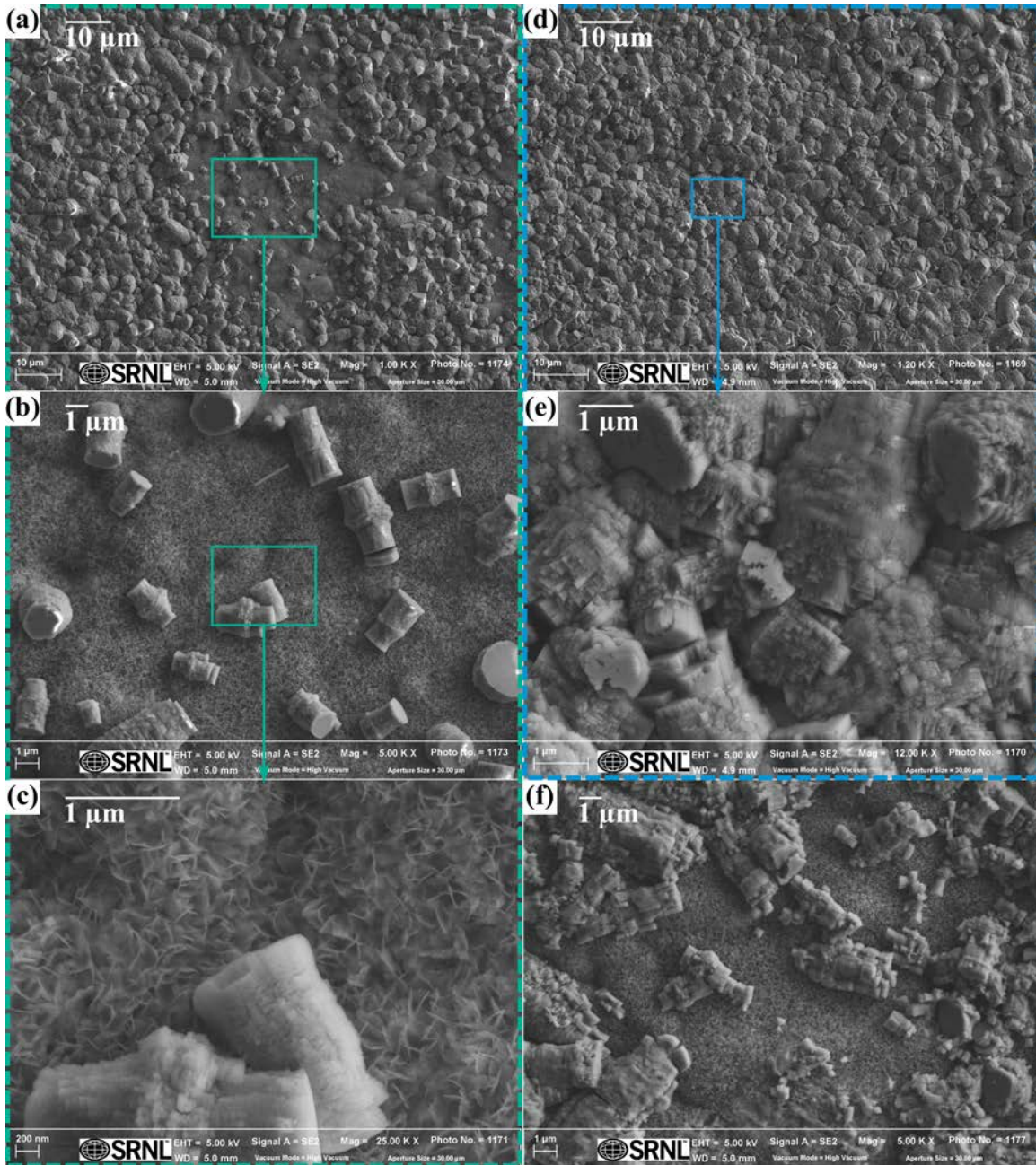


Figure 3-2. Plan-view SEM images for a 5052-alloy hot-wall sample exposed to 50°C water for 16 weeks (112 days), followed by 43 days bagged storage prior to imaging. Images (a)–(c) show the same location at increasing magnification ($\times 1k$, $\times 5k$, and $\times 25$), revealing a “needle-like” appearance surrounding isolated, roughly cylindrical particles. Similarly, (d) and (e) show two levels of magnification ($\times 1.2k$ and $\times 12k$) of a region of densely packed particles with no “needle-like” structure visible. Finally, (f) shows a region with irregularly shaped particles over “needle-like” structure.

SEM of the film cross-section (Figure 3-3) indicates a total film thickness ranging from about 2.7 to 5.8 μm . In addition, the film appears to consist of two layers of oxide of roughly equal thickness separated by a dark line. The outer layer consists of bumps at a length scale similar to that of the blocks observed on the plan-view SEM, suggesting that the blocks form an outer layer sitting on top of another oxide layer, possibly the

“needle-like” structure. A similar two-layer oxide structure was previously observed for an Al-1100 specimen exposed at 50°C for 29 days [3]. The Al-1100 sample with the two-layer oxide featured a dense-looking morphology over the entire surface imaged in plan-view with no “needle-like” structure apparent [3]. This correlation between dense-looking surface morphologies observed in plan-view and two-layer oxides observed in cross-section is consistent with the hypothesis that the dense oxide structures form as a second (outer) layer.

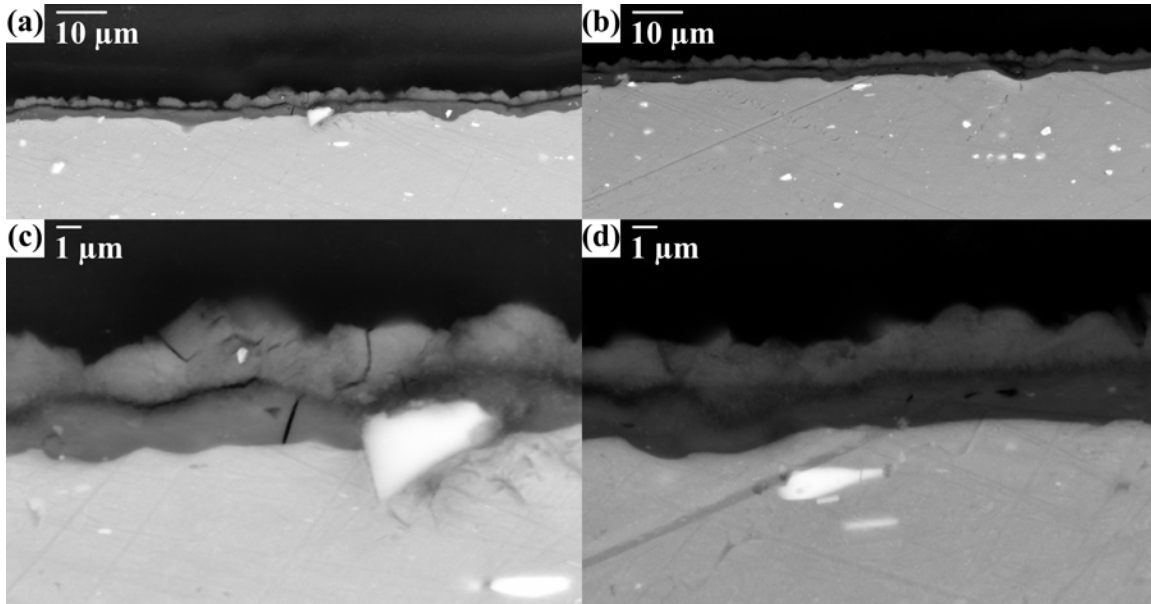


Figure 3-3. SEM images of the cross-section for a 5052-alloy hot-wall sample exposed to 50°C water for 112 days, followed by 85 days bagged storage prior to imaging. Total hydroxide layer thickness ranges from approximately 2.7 to 5.8 μm and appears to consist of two layers.

For bayerite, $M_{\text{Al}(\text{OH})_3} = 78 \text{ g/mol}$ and $\rho_{\text{bayerite}} = 2.53 \text{ g/cm}^3$ [5]. Thus, Equation (1) computes the thickness of a uniform, dense bayerite film corresponding to the post-hot-wall weight gain (Table 3-1) as 2.4 μm. This is slightly less than, but quite close to, the minimum thickness of ~2.7 μm measured from the cross-sectional SEM images. The actual morphology of the oxide film was quite non-uniform (Figure 3-2 and Figure 3-3). If the film was completely dense, the calculated thickness would be expected to fall between the maximum (5.8 μm) and minimum (2.7 μm) thicknesses observed from the cross-section. The fact that the observed thickness range lies entirely above the calculated value suggests that the film density may be lower than that of dense bayerite, e.g., due to porosity in the film.

3.2 Hot-wall immersion: 5052 at 100°C for 12 days

Figure 3-4, Figure 3-5, and Figure 3-6 show characterization results for a 5052-alloy hot-wall specimen exposed at 100°C for 12 days. The specimen was weighed immediately after removal from the water bath and preliminary drying of the sample and was subsequently stored bagged until further characterization.

The XRD spectrum shown in Figure 3-4 identifies the oxide as mostly boehmite, with some minor peaks indicating the presence of SiC, which may be a contaminant. Boehmite is the expected phase for 100°C exposure [2, 5].

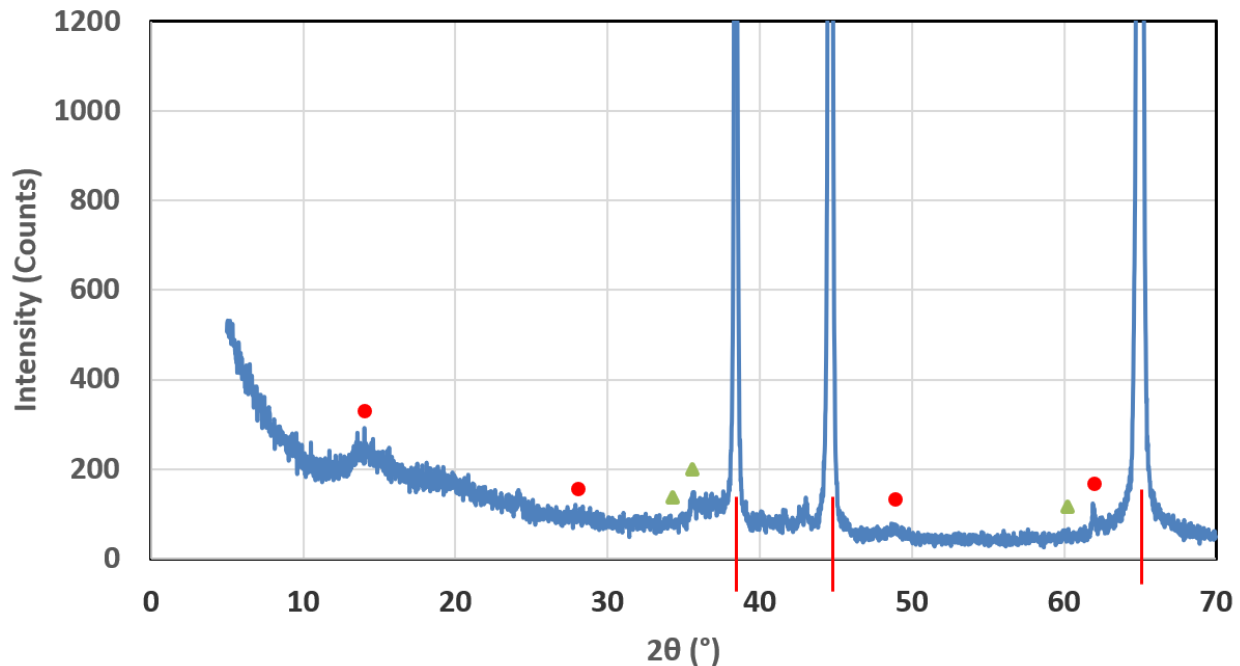


Figure 3-4. XRD spectrum for 12-day hot-wall growth on Al-5052 at 100°C. Red lines indicate locations of aluminum peaks, red circles indicate boehmite peaks, and green triangles indicate peaks of SiC, which may be a contaminant.

SEM images of the surface (Figure 3-5) show a “needle-like” morphology similar to that observed in previous experimental results for Al-1100 [3] and Al-6061 [4] over portions of the surface. However, there were also regions where the oxide layer appears to have a denser structure (e.g., lower-left of Figure 3-5(a)). In contrast to the 50°C sample in Figure 3-2, the dense-looking regions of the film did not appear to form as distinct blocky structures, but instead appeared as if oxide had started to fill in the pores in the “needle-like” structure. Previously [3], an Al-1100 sample exposed at 100°C for 41 days was observed to feature isolated flat blocks or “scales” on top of a “needle-like” structure, similar to Figure 3-2(b) except for the shape of the blocks. Figure 3-5(a) appears to show a couple of flat, blocky structures near the top right, resembling those previously observed on the Al-1100 sample [3], with the largest having a hexagonal shape. Since the sample in Figure 3-5 was exposed for only 12 days while that in Ref. [3] was exposed for 41 days, it is possible that additional blocks would develop given extended exposure time.

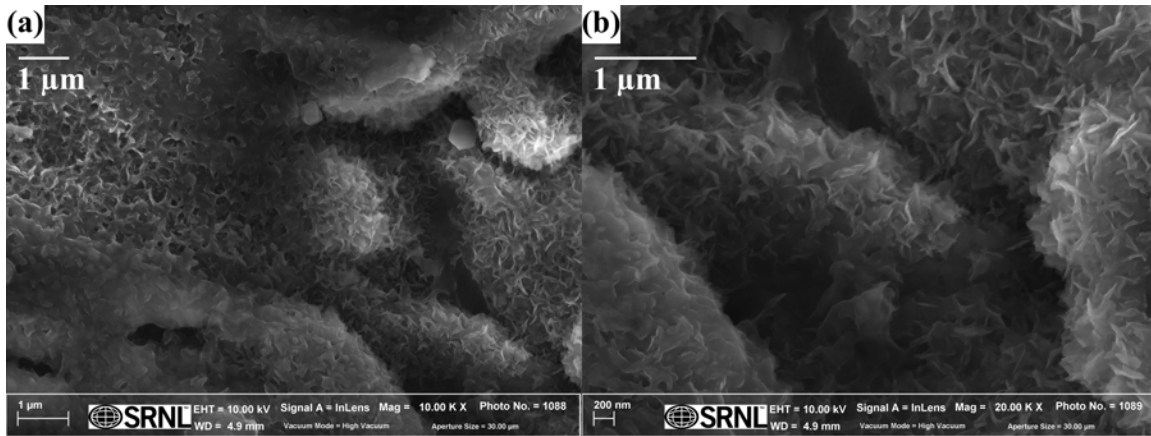


Figure 3-5. Plan-view SEM images for a 5052-alloy hot-wall sample exposed to 100°C water for 12 days, followed by 90 days bagged storage prior to imaging, at (a) $\times 10k$ and (b) $\times 20k$ magnifications. A “needle-like” structure is apparent over a significant fraction of the surface, but there are extensive areas of denser-looking oxide.

SEM of the film cross-section (Figure 3-6) indicates a total film thickness ranging from about 0.4 to 5.6 μm . The maximum thickness occurred in the small, ~ 5 – 10 - μm -wide region of thick oxide in Figure 3-6(b) and (d), which appears to correspond to localized penetration into the metal. Outside of this localized pit, the oxide thickness appeared to be fairly uniform with a maximum thickness of about 2 μm . The film appears to consist of a single layer.

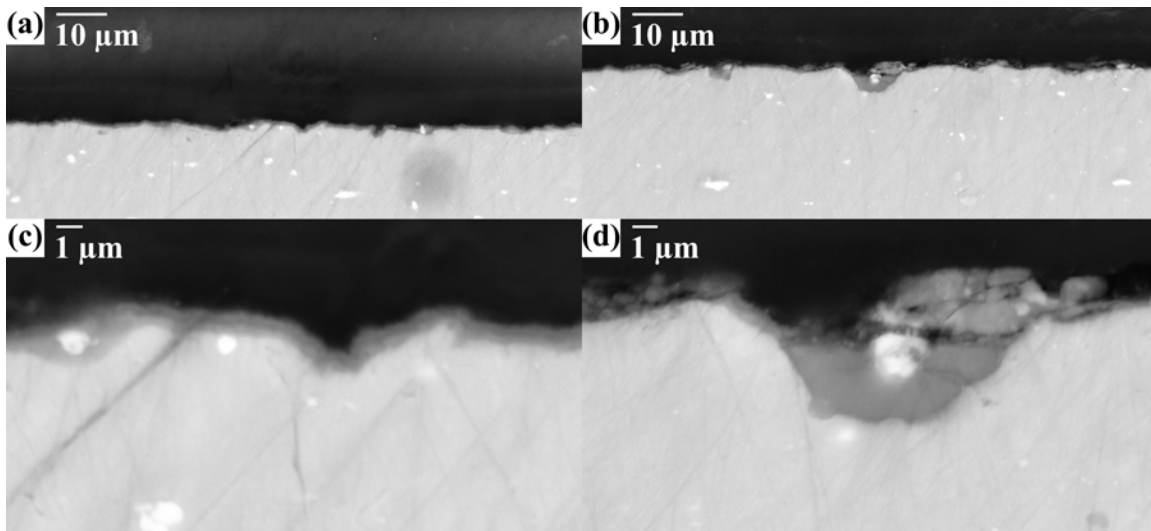


Figure 3-6. SEM images of the cross-section for a 5052-alloy hot-wall sample exposed to 100°C water for 12 days, followed by 170 days bagged storage prior to imaging. Total hydroxide layer thickness ranges from approximately 0.4 to 5.6 μm and appears to consist of a single layer.

For boehmite, $M_{Al(OH)_3} = 60$ g/mol and $\rho_{boehmite} = 3.01$ g/cm³ [5]. Thus, Equation (1) computes the thickness of a uniform, dense boehmite film corresponding to the post-hot-wall weight gain (Table 3-1) as 0.85 μm . This falls inside the thickness range of 0.4–2 μm observed from the cross-section (excluding the pit in Figure 3-6(b) and (d)).

4.0 Conclusions

Laboratory experiments were performed to produce and characterize hydroxide (oxide) films on aluminum 5052 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:

- The hydroxide phases observed were predominantly bayerite for 50°C oxide growth and boehmite for 100°C oxide growth, which are consistent with the literature [2] and with previous results for other aluminum alloys [3, 4].
- The calculated dense-film thickness corresponding to the measured weight gain was close to that measured from SEM images of the cross-section for both samples. For the oxide grown at 50°C for 112 days, the calculated thickness of 2.4 μm fell slightly below the measured range of 2.7–5.8 μm , suggesting that the average film density may be lower than that of bulk bayerite, e.g., due to porosity. For the oxide grown at 100°C for 12 days, the calculated thickness of 0.85 μm fell within the measured thickness range of 0.4–2 μm (with thickness up to 5.6 μm in a localized pit), suggesting that the film may be nearly dense boehmite.
- The oxide grown at 50°C for 112 days featured a varied range of oxide morphologies. The surface was largely covered by dense-looking, blocky oxide particles, which appear to have formed on top of a “needle-like” oxide layer similar to that observed in previous tests at both 50 and 100°C [3, 4]. In some regions, these blocky particles are closely packed together with no “needle-like” morphology visible, while in other regions, isolated blocks are completely surrounded by the “needle-like” structure. SEM of the cross-section for the 50°C sample showed a two-layer oxide structure, supporting the hypothesis that the blocks sit on top of the “needle-like” layer instead of replacing it. This observation was consistent with the two-layer film observed for the previous Al-1100 sample that grew a dense-looking oxide film at 50°C [3].

5.0 References

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