EFFECTS OF THERMAL EXPOSURES ON THE
STRESS CORROSION CRACKING OF ALUMINUM ALLOY 5456-H131

by

KEVIN LOUIS MACKE

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Project Advisor: Prof. John J. Lewandowski

Department of Materials Science and Engineering
CASE WESTERN RESERVE UNIVERSITY
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1. Background & Introduction

1.1 Aluminum and Aluminum Alloys

Metallic aluminum is of enormous economic and industrial importance. Many applications benefit from its very high crustal abundance, low density, high thermal conductivity and ductility, compatibility with a range of processing techniques, and relatively good resistance to corrosion in most atmospheric conditions. While commercially pure grades of aluminum suffer from relatively low strengths, with a yield strength of approximately 20 MPa and ultimate tensile strengths of 30 – 70 MPa (dependent on material purity), this drawback is effectively mitigated through the addition of several alloying elements [1, 2].

Several classes, or families, of aluminum alloys are used in a broad array of applications. These families are defined on the basis of the alloying elements found in each and are broken into the categories as indicated in Table 1.1 [1, 2].

<table>
<thead>
<tr>
<th>Alloy Family</th>
<th>Allowing Elements</th>
<th>Principle Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1xxx</td>
<td>Commercially pure</td>
<td>Used chiefly in electrical and chemical industries</td>
</tr>
<tr>
<td>2xxx</td>
<td>Primarily copper, with occasional additions of other elements such as magnesium</td>
<td>Broadly applied in aviation</td>
</tr>
<tr>
<td>3xxx</td>
<td>Manganese</td>
<td>General purpose architectural use, various others</td>
</tr>
<tr>
<td>4xxx</td>
<td>Silicon</td>
<td>Welding rods &amp; brazing sheet</td>
</tr>
<tr>
<td>5xxx</td>
<td>Magnesium</td>
<td>Broad marine application, such as boat hulls, superstructures, and armor plate</td>
</tr>
<tr>
<td>6xxx</td>
<td>Magnesium, silicon</td>
<td>Architectural extrusions</td>
</tr>
<tr>
<td>7xxx</td>
<td>Zinc, plus varying secondary additions of copper, magnesium, chromium, &amp; zirconium</td>
<td>Aircraft structural members and other high strength applications</td>
</tr>
<tr>
<td>8xxx</td>
<td>Tin, lithium</td>
<td>High strength-to-weight applications</td>
</tr>
<tr>
<td>9xxx</td>
<td>Reserved for future use</td>
<td></td>
</tr>
</tbody>
</table>

Table 1.1: Classification of aluminum alloys as defined by [1].
In the alloy designation scheme outlined above, the second digit indicates modifications or impurity limits, and the last two digits indicate the specific alloy or purity. Additionally, casting alloys are designated with a three-digit+decimal system such as 6xx.x, where the first digit corresponds to the families above and the decimal relates to casting specific requirements [1].

Strengthening mechanisms take a variety of forms in aluminum alloys and include solid solution strengthening and precipitation hardening derived from a range of secondary and tertiary phases. Secondary strengthening is achieved through tempering, strain, and age hardening, and is indicated by alphanumeric descriptors following the alloy designation. Basic tempers are as follows [2]:

F: As fabricated
O: Annealed (wrought products only
H: Strain hardened
W: solution heat treated
T: Thermally treated to stable tempers other than F, O, or H, with or without strain hardening

1.2 Aluminum-Magnesium Alloys

The 5xxx series of aluminum alloys generally display high specific strengths, excellent corrosion resistance, and while they are not heat treatable, they benefit from strain hardening [1, 2]. Magnesium concentrations range from 0.8 – 5.1, manganese from 0.08 – 0.7, chromium from 0.12 – 0.25, and titanium from 0.10 – 0.13. The 5xxx series has a long history and the first alloys and applications stem from the 1950’s. Owing to its versatility and desirable combination of attributes, development continues for this class and modern additions may include zinc, as for 5059.

Alloy 5456 is a high strength, strain hardenable alloy with additions of 5.1% magnesium, 0.12% chromium, and 0.08% manganese [2]. A high magnesium concentration affords 5456 excellent strength, but the relatively low solubility of magnesium of less than 1% at room
temperature, shown in the binary phase diagram in Figure 1.1, leaves this and similar high magnesium content aluminum alloys susceptible to sensitization after extended exposures at moderate temperatures, whereby magnesium diffusion to the grain boundaries may lead to the eventual formation of the equilibrium β-phase \( \text{Al}_2\text{Mg}_3 \) along the grain boundaries. The formation of Mg-rich precipitates in alloys with similar magnesium concentrations is known to follow a path from GP zones \( \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta \) and has been observed at temperatures well below 200°C [5, 6]. Following the onset of sensitization in these alloys, generally well-tolerated environments can become relatively severe, leading to rapid crack propagation at slow strain rates and even sustained load cracking via stress corrosion cracking. The combination of thermally induced sensitization, followed by environmentally triggered stress corrosion cracking, can lead to rapid brittle failure at strength levels significantly lower than the ultimate tensile strengths. Additionally, the marked decrease in ductility associated with these phenomena does not require contiguous formations of \( \beta \) precipitates[4, 5].

![Aluminum-magnesium binary phase diagram](image)

Figure 1.1: Aluminum-magnesium binary phase diagram. Reproduced from [7].
The environmental dependence of grain boundary fracture in 5456 alloy is indicated by prior studies examining fatigue crack growth in both as-received [8] and sensitized samples. In the case of as-received specimens, dramatic differences have been seen in fatigue crack growth in (dry) environments as compared to laboratory air conditions that may contain anywhere from 10% to 80% humidity. Differences in crack growth rates of up to ten times have been observed [10]. Menzemer and Srivatsan [8] have demonstrated that oxygen-containing environments produced the slowest crack growth rates relative to water or laboratory air. In the case of sensitized specimens subjected to fatigue cracking in the LT orientation, secondary crack growth along grain boundary regions was observed perpendicular to the fatigue pre-crack, in the S-L plane [11].

The impact of stress corrosion cracking is of particular importance to the U.S. Navy which operates 26 CG-47 Ticonderoga Class Aegis cruisers. CG-47 are large ships, 567 feet in length, carrying crews of approximately 364 members, with planned service lives of 40 years [12]. As such they represent substantial investments. 5456 aluminum was used extensively in the superstructures of these ships as a weight saving measure. In 2007, long cracks were noticed in areas that were not associated with known stress concentrations, indicating that their presence was due to causes other than the expected fatigue cracking associated with cyclic stresses. The U.S. Navy concluded that the cause of this cracking was stress corrosion cracking due to exposure to the corrosive marine environment [12]. The fractures were found in significant structural components, including the superstructure weather deck, internal structural members of the gas turbine engine intakes, and in transverse bulkheads [12].
Previous work on aluminum 5456-H116 has indicated that thermally induced sensitization of magnesium-rich aluminum alloys might be mitigated by additional heat treatment [13, 14]. This stabilization must be performed at a high enough temperature to create unstable conditions for the $\beta$ precipitates responsible for the sensitized condition, but low enough to avoid fully annealing the alloy. For alloys with 5.5% magnesium such as 5456, a heat treatment of 10 minutes or longer at 280°C may be sufficient dissolve grain boundary magnesium-rich phases back into solution, but without lowering the high yield strength imparted by the tempering process [14].

1.3 Purpose of Present Study

As detailed above and as shown by previous students [15, 16, 18], previous studies of aluminum-magnesium alloys have described $\beta$ phase formation, corrosion properties, and mechanical properties after short duration high temperature exposures. The mechanical properties of 5456-H131 after long duration low temperature exposures (especially in water) are not fully understood however [15]. Additionally, the mechanical properties of 5456-H131 in both as-received and sensitized conditions tested in saline environments are not well known. This study is intended to characterize the effects of environmental cracking in aluminum alloy 5456-H131 following short and long term low temperature exposures in air and water.
environments. Testing of a stabilization technique for 5456-H131 sensitized at intermediate temperatures for short durations was performed.

2. Experimental Procedures

2.1 Materials

Commercially manufactured 5456-H131 plate with a thickness of 29.2 mm was obtained from the ALCOA Technical Center. Multiple tensile specimens with the geometry specified in Figure 1.1 and oriented in the ST direction as specified in Figure 1.2 were removed from this plate stock. All specimens were polished by hand in a rotating chuck at 3100 rpm with 600-, 800-, and 1200-grit silicon carbide metallographic papers and white kerosene as a lubricant. Post-polishing cleaning was done in dry ethanol in an ultrasonic bath for two minutes.

Specified mechanical properties for the plate are 0.2% yield stress = 249 MPa (minimum) and UTS = 310 MPa (minimum). Alloy composition was as follows:

\[
\begin{align*}
\text{Mn} & = 1.00 \text{ wt}\% \\
\text{Mg} & = 5.50 \text{ wt}\% \\
\text{Cr} & = 0.20 \text{ wt}\% \\
\text{Ti} & = 0.20 \text{ wt}\% \\
\text{Cu} & = 0.1 \text{ wt}\% \\
\text{Si} & = 0.25 \text{ wt}\% \\
\text{Fe} & = 0.19 \text{ wt}\% \\
\text{Zn} & = 0.25 \text{ wt}\% \\
\text{Al} & = \text{ balance}
\end{align*}
\]

Figure 2.1: Tensile specimen geometry. Reproduced from [15].
2.2 Metallography

Metallographic specimens were prepared from samples removed from the plate stock corresponding to the three principal planes. The samples were polished with a series of silicon carbide coated papers of decreasing grit sizes with water lubrication, followed by diamond paste on cloths using an Allied High Tech Products 8” MultiPrep Polishing System. Final polishing was performed with colloidal silica on a Buehler VibroMet 2 vibratory polisher. Samples were washed for two minutes in an ultrasonic bath in a 1% solution of Liquinox detergent in deionized water before advancing to finer grits. Polishing media and times are listed in Table 2.1.

<table>
<thead>
<tr>
<th>Abrasive</th>
<th>Backing Medium</th>
<th>Platen Action</th>
<th>Rotation</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>120-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>Until coplanar</td>
</tr>
<tr>
<td>240-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>5 min.</td>
</tr>
<tr>
<td>400-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>5 min.</td>
</tr>
<tr>
<td>600-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>10 min.</td>
</tr>
<tr>
<td>800-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>10 min.</td>
</tr>
<tr>
<td>1200-Grit SiC</td>
<td>Paper</td>
<td>120 rpm</td>
<td>Stationary</td>
<td>10 min.</td>
</tr>
<tr>
<td>3 μm Diamond Paste</td>
<td>Allied PLAN-Cloth</td>
<td>100 rpm</td>
<td>co-rotation</td>
<td>15 min</td>
</tr>
<tr>
<td>1 μm Diamond Paste</td>
<td>Allied TECH-Cloth</td>
<td>100 rpm</td>
<td>co-rotation</td>
<td>30 min.</td>
</tr>
<tr>
<td>Colloidal Silica</td>
<td>Micronap cloth</td>
<td>50% Amplitude</td>
<td>NA</td>
<td>60 min.</td>
</tr>
</tbody>
</table>

Table 2.1: Metallographic polishing conditions. Times noted are approximate as each grit was applied until the scratch pattern from the preceding grit was completely removed.
Etching was performed with Barker’s anodic reagent (2% fluoroboric acid in deionized water). A DC current was applied to the polished samples with 316L stainless steel electrodes at a potential of 20 V. Etching was performed in a polyethylene basin on a stir plate and the solution was agitated continuously for the two minute duration. Samples were rinsed with deionized water and air dried after etching and the solution was allowed to cool to room temperature before etching the next sample.

2.3 Thermal Exposures to Produce Sensitization

Tensile samples were tested in the as-received condition as well as in sensitized conditions resulting from thermal exposure in varied environments. Polished and cleaned tensile specimens were placed in Blue-M convection ovens at temperatures of 80°C and 175°C in environments of air, deionized water, and an aqueous solution of 0.03 M NaCl. Air-exposed samples were placed on watch glasses without contact between gauge length surfaces. Water-exposures were conducted in glass beakers sealed with aluminum foil and gauge lengths were not permitted to contact adjacent samples. Exposure times of 75, 100, 125, 250, 500, 750, 1,000, and 4,000 hours were chosen to produce a range of degrees of sensitization, from mild to severe. Following sensitization, several specimens exposed at 175°C in air were subjected to an additional thermal stabilization treatment in an effort to remediate the induced sensitization. Stabilization consisted of 15 minutes at 280°C in air, followed by cooling in ambient laboratory air.
Table 2.2: Sensitization conditions and the numbers of tensile specimens produced in each condition.

<table>
<thead>
<tr>
<th>Exposure Time (hr.)</th>
<th>60°C Air</th>
<th>70°C Air</th>
<th>80°C Air</th>
<th>80°C DI Water</th>
<th>80°C 0.03M NaCl\textsubscript{aq} Solution</th>
<th>175°C Air</th>
<th>175°C DI Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>-</td>
<td>-</td>
<td>6</td>
<td>3</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>100</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16</td>
<td>2</td>
<td>-</td>
</tr>
<tr>
<td>125</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>-</td>
<td>4</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>250</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>500</td>
<td>-</td>
<td>-</td>
<td>6</td>
<td>6</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>750</td>
<td>-</td>
<td>-</td>
<td>8</td>
<td>12</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1,000</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>13</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4,000</td>
<td>3</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

2.4 Mechanical Testing

All tensile specimens were tested on an Instron Model 1361 screw-driven machine, with data collection performed by MTS TestWorks software. Testing ran from mid-summer to late winter and ambient laboratory temperatures ranged from 20-29°C. Humidity levels were not controlled. Nominal strain rates of $5 \times 10^{-5}$/sec were used for all tests and data was collected from a 10 kN load cell at a sample rate of 0.5 Hz. Prior to testing, specimen gauge length diameters were measured with Mitutoyo digital calipers.
2.4.1 Dry Environment Testing

Dry air conditions were achieved by packing anhydrous magnesium perchlorate desiccant around the test specimen after fixturing was completed. Containment of the desiccant was provided by foil-backed tape and later by Tygon tubing as shown in Figure 2.4.

Figure 2.4: Mounted tensile test specimen with anhydrous magnesium perchlorate contained by a Tygon sleeve.

2.4.2 Wet Environment Testing

Tensile specimens were tested in four wet conditions consisting of deionized water, 0.6 M NaCl\textsubscript{aq} solution, 0.6 M NaCl\textsubscript{aq} + 0.3% H\textsubscript{2}O\textsubscript{2} solution, and 0.03 M NaCl\textsubscript{aq} solution. Initial tests were performed in an approximately 40 ml chamber formed by a Tygon sleeve placed around the test fixture as shown in Figure 2.5. Later testing was done in an acrylic chamber with a solution volume of approximately 200 ml. For all wet tests, the grip portions of the specimens and metallic portions the test fixtures were sealed with white petroleum jelly to prevent galvanic corrosion and of the test fixture in the saline environment. A portion of tests were subject to measurement of galvanic potential between the 5456-H131 specimen and a silver chloride reference electrode.
Results & Discussion

3.1 Tensile Testing

All thermally sensitized specimens listed in Table 2.2, in addition to as-received specimens were tested to failure in varied environments. For each specimen, the data recorded in TestWorks was plotted and fitted by hand to determine the 0.2% offset yield stress. Ultimate tensile stresses were determined by and exported from TestWorks, and elongation was measured from TestWorks displacement data. Post-failure final diameters were measured optically with calibrated micrographs of the fracture surfaces in the ImageJ image processing package. Analysis of the fracture surfaces for selected specimens was conducted by SEM and the resulting micrographs were analyzed for percent-flat fracture vs. ductile failure in ImageJ as well.

Sensitized specimens were generally found to exhibit lower ultimate strengths, as well as decreased ductility compared to as-received specimens in identical test environments (see Figure 3.1). Serrated flow was observed in as-received samples as well as specimens with a range of thermal exposures. In both as-received and thermally exposed samples tested in air or
dry air, these serrations (e.g. Figure 3.3) are generally agreed to be related to the association of Mg with dislocations and the repeated locking/unlocking of dislocations by the mobile Mg [17]. This typically only occurs after significant deformation as the increased strain and dislocation density is required to increase the mobility of the Mg enough to eventually pin the dislocations and thereby produce serrated flow at relatively high strain levels. These serrations are distinctly different than the feature observed past the UTS in Figures 3.4 and 3.5 which is related to repeated cracking of the material and loss in cross sectional area at a much lower total elongation/strain. These different test conditions have produced five distinctly different patterns of stress-strain curves, regarding both the onset of serrated flow and the type of additional deformation/elongation after the UTS was exceeded.

Generally smooth curves of the type shown in Figure 3.2 were produced by specimens sensitized in both air and water for durations from 250-4,000 hours, tested in all environments. The lack of serrated flow in Figure 3.2 compared to that shown in Figure 3.3 results from the much lower strains achieved in these samples, thereby limiting the mobility of the Mg and preventing it from pinning the dislocations in the manner shown in Figure 3.3. The type of serrations shown in Figure 3.3 were only produced by the dry air test environment, regardless of prior thermal exposure. All of these samples exhibited high elongations/strains, consistent with the argument that pinning of dislocations by Mg requires sufficient mobility of the Mg and that this mobility can only be obtained at high dislocation densities since Mg is a substitutional atom with limited mobility it Al unless a high dislocation density enhances the diffusion rate.

Curves with a multi-stage reduction in strength past the UTS, with sharp drops in stress followed by continued extension and intermittent slight increases in stress (Figure 3.4) were seen for all thermal exposures but were only observed for specimens tested in saline environments. This results from cracking of the samples and the associated loss of cross sectional area that produces the load/stress drops observed. The fourth type of pattern observed is similar to the previous multi-stage curve, but without the serrated flow past the UTS.
Specimens with long and short thermal exposure in air tested in 0.6 M NaCl + H₂O₂ environments produced this curve type, shown in Figure 3.5. The final curve observed is shown in Figure 3.6 and is characterized by abrupt failure with negligible yielding. Specimens sensitized in both air and water for long and short durations produced this type of curve, but almost exclusively in 0.6 M NaCl + H₂O₂ test environments. After failure, sensitized specimens were observed to have several fracture initiation sites at the surface of the gauge length, always oriented perpendicularly to the strain axis.

Figure 3.1: Representative stress-strain curves for tensile specimens sensitized in 80°C air and tested in 0.6 M NaCl + 0.3% H₂O₂.
Figure 3.2: Representative stress-strain curves showing mildly serrated flow produced by all thermal exposures and test environments.

Figure 3.3: Representative stress-strain curves showing mildly serrated flow followed by pronounced serration. A wide range of thermal exposures and test environments produced this type of curve, but dry air test environments produced these curves exclusively.
Figure 3.4: Representative stress-strain curves showing microcracking beyond UTS. All exposures produced curves of this type but only in saline test environments.

Figure 3.5: Representative stress-strain curves similar to those of Figure 3.4 but without serrated flow beyond UTS. All exposures produced curves of this type but were observed most commonly in saline test environments with hydrogen peroxide.
Figure 3.6: Representative stress-strain curves demonstrating the abrupt failure observed in saline test environments with hydrogen peroxide.

Figure 3.7: 0.2% offset yield stresses for tensile specimens sensitized at 80°C in deionized water (a) and air (b).
Figure 3.8: Ultimate tensile stresses for tensile specimens sensitized at 80°C in deionized water (a) and air (b).

Elongation measurements from TestWorks were used to calculate ductility indices for sensitized specimens in varied environments after Holroyd and Scamans [4]. Percent elongation for as-received samples in a given test environment serve as the unity value for sensitized specimens tested in identical environments, with the elongation ratio expressed as a percent of unity. Increasingly severe environments generally decreased the % elongation ratio as did thermal exposures with increased time/temperature as shown in Figure 3.9. Values of % elongation ratio > 1 likely resulted from the multiple microcracking observed in samples tested in certain environments. This explanation is consistent with the fact that samples with % elongation ratios > 1 were generally those tested in the most severe environments (0.6 M NaCl\text{aq} and 0.6 M NaCl\text{aq} + 0.3\% \text{H}_2\text{O}_2) which were most likely to produce significant microfracturing past the UTS. For samples thermally exposed in water and tested in dry air that produced % elongation ratios > 1, actual values were 1.09 and this may be attributable to microcracking associated with hydrogen diffusion into the sample during thermal exposure.
A similar ductility ratio was calculated for percent reduction in area, with the same method as was used for the elongation ratio. A general trend of reduced ductility was displayed for most test environments but inaccuracies in the measurements preclude meaningful inferences from the data. Sources of variation include differing measurement techniques for initial and final diameters with different degrees of accuracy and precision, slight variation of the gauge length diameter that may have resulted in the location at which \( d_i \) was measured being a poor representation of the true \( d_i \) at the fracture site, and small relative changes in diameter due to the brittle failure observed in sensitized failures. In many cases, these factors likely combined to produce random variation in measurements that were greater in magnitude than the measured changes in diameter.

A final ductility index was calculated as the difference between the 0.2% offset yield stress and the UTS (Figure 3.10). Specimens sensitized in water were found to exhibit a generally small decrease in ductility as measured by this value, with the most corrosive environments producing the greatest reduction in ductility. Dry test environments showed no decrease in ductility in even highly sensitized specimens relative to the as-received condition. For specimens sensitized in air, the trend of decreasing ductility with increasing sensitization times
was observed with noticeably less scatter in the data as compared to the specimens sensitized in deionized water.

Figure 3.10: (UTS) – (0.2% \( \sigma_y \)) for tensile specimens sensitized at 80°C in deionized water (a) and air (b).

Increased sensitization times were found to increase the prevalence of flat fracture patterns on the fracture surfaces of the failed tensile specimens as shown in Figures 3.11 and 3.12. For any given specimen, the fracture surface was divided between flat, relatively featureless surfaces, and dimpled ductile tearing. As a group, specimens sensitized in air rather than water show less scatter in the results for this measure.

Figure 3.11: Percentage of the total fracture surface identified as flat fracture for tensile specimens sensitized at 80°C in deionized water (a) and air (b).
Figure 3.12: Fracture surfaces of failed tensile specimens tested in an aqueous solution of 0.6 M NaCl + 0.3% H₂O₂ with regions of flat fracture denoted by green coloration. Specimens exhibit a general increase in flat fracture with longer exposure times. The as-received condition (a) produced 43% flat fracture. The remainder of specimens presented were sensitized in 80°C air for 75 hr. (b, 30%), 125 hr. (c, 79%), 500 hr. (d, 63%), 750 hr. (e, 71%), and 1,000 hr (f, 71%).

Tensile specimens sensitized in air and deionized water at 175°C for 100 hours were subjected to a stabilization treatment of 280°C for 15 minutes and tested to failure in a variety of environments. Measured values of the 0.2% offset yield and ultimate tensile stresses shown in Figure 3.13 indicate that sensitization in these samples was mitigated to a significant degree. The extent of loss of strength in the sensitized samples was impacted by the corrosiveness of the testing environment. As such, the saline environments showed the largest changes for the stabilized specimens relative to sensitized specimens, while the deionized water and laboratory air test environments showed smaller increases in strength following stabilization.
Figure 3.13: Effects of stabilization treatment on sensitized tensile specimens in varying test environments. 0.2% σy stress and UTS are shown for as-received, sensitized, and sensitized and stabilized specimens.
3.2 Metallography

3.2.1 As-Received Grain Structure

The as-received material was found to have a grain structure with significant elongation in the rolling and long-transverse planes, and an intermediate degree of elongation in the short transverse plane, as shown in Figures 3.14 and 3.15. Numerous intermetallic particles were spread uniformly throughout the matrix.

Figure 3.14: As-received microstructure of the 5456-H131 plate.
3.2.2 Fracture Characterization in Sensitized Tensile Test Specimens

Two failed tensile specimens were sectioned longitudinally, polished, and etched to reveal grain boundaries. The specimens were selected to represent moderate sensitization (80°C in air for 250 hours) as well as a high degree of sensitization (80°C in water for 1,000 hours). Tension data for these samples are shown in Table 3.1 and Figure 3.16 and low-magnification SEM micrographs shown in Figure 3.17 indicate flat fracture outlined in green. Both samples failed with flat (rather than slanted) fracture surfaces when they were tested in identical 0.6 M NaCl<sub>aq</sub> environments.

<table>
<thead>
<tr>
<th>Sensitization</th>
<th>0.2% σ&lt;sub&gt;y&lt;/sub&gt; (MPa)</th>
<th>UTS (MPa)</th>
<th>% Elongation Ratio</th>
<th>% Flat Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 (air)</td>
<td>247</td>
<td>260</td>
<td>0.6</td>
<td>57</td>
</tr>
<tr>
<td>1,000 (water)</td>
<td>160</td>
<td>165</td>
<td>0.4</td>
<td>61</td>
</tr>
</tbody>
</table>

Table 3.1: Yield and ultimate strengths and ductility measures for selected failed tensile specimens.
Figure 3.16: Stress-strain curves for selected tensile specimens.

Figure 3.17: Fracture surfaces of selected tensile specimens with regions of flat fracture indicated by green coloration. Specimens were sensitized for 250 hours in air (a) and 1,000 hours in water (b).
On analysis of the polished and etched longitudinal surface shown in Figure 3.18, the highly sensitized specimen was found to have numerous secondary intergranular cracks penetrating the gauge length. The secondary cracks were found to extend up to a millimeter into the depth of the gauge length perpendicular to the principle strain axis, with cracks appearing several millimeters away from the fracture surface. The moderately sensitized sample was not observed to have any secondary crack formation outside the zone affected by the primary failure surface. Within the moderately branched network of the crack at the failure surface, the primary mode of failure was intergranular, with some individual grains showing evidence of transgranular fracture propagation to adjacent grain boundaries where the crack continued to propagate intergranularly.
Figure 3.18: Longitudinal sections of failed tensile specimens showing extensive secondary intergranular fracture in a highly sensitized specimen (a-d) and primarily intergranular fracture constrained to the failure surface in a moderately sensitized specimen (e-g).
Conclusions

Aluminum 5456-H131 has been shown to behave in a manner consistent with the understanding that magnesium rich aluminum alloys are highly susceptible to sensitization and environmentally enhanced failure. The present work has shown that this sensitization behavior is thermally induced and occurs at temperatures as low as 80°C at durations as short as 75 hours. At short durations, sensitization produces modest reductions in tensile strength but noticeable decreases in ductility. At moderate durations (as short as 250 hours), significant reductions in yield and ultimate strengths as well as ductility is observed. These effects become progressively larger as sensitization times increase, up to the tested limit of 1,000 hours.

Sensitization environments (i.e. thermal exposure in air vs. water) were only shown to have a modest effect on the degree of environmental cracking. Testing environments, however, have a pronounced effect on the degree of environmentally enhanced cracking (i.e. stress corrosion cracking). Corrosive environments such as saline solutions, with or without the addition of H₂O₂, produce dramatically lowered strength and ductility in sensitized samples. Sensitized 5456-H131 specimens are shown to be significantly affected by very small amounts of moisture, with laboratory air yielding reductions in strength and ductility while dry, desiccated environments produce nearly identical properties in even moderately to highly sensitized samples.

Attempts to remediate sensitized specimens through thermal stabilization show significant increases in ductility and UTS. While samples did not regain their as-received ductility/UTS after stabilization, the reversal in the degree of embrittlement is consistent with resolution of the Mg-rich grain boundary phases. This should be checked with careful TEM measurements. The increased prevalence of flat cracking at the failure surface, combined with increased secondary cracking in highly sensitized specimens lends evidence in support of β phase precipitates as the primary factor in thermally induced environmentally assisted cracking in 5456 aluminum.
References


