MECHANICAL PROPERTY EVOLUTION AND THERMAL REMEDIATION OF ALUMINUM MAGNESIUM 5XXX ALLOYS FOLLOWING LOW AND INTERMEDIATE TEMPERATURE THERMAL EXPOSURE

by

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*We also certify that written approval has been obtained for any proprietary material contained therein.
Dedication

To my parents for 27+ years of dedication and support of my dreams.

To Narges my wife who always supports me.
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Mechanical Property Evolution and Thermal Remediation of Aluminum Magnesium 5xxx Alloys Following Low and Intermediate Temperature Thermal Exposure

Abstract

by

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Al-Mg 5xxx alloys are desirable in a wide array of structural applications that require a weldable alloy with good corrosion resistance. However, mechanical property changes may result from intermediate temperature exposures. Commercially available 5xxx alloy plates have been thermally treated up to 10,000 hrs. (13 months) at temperatures of 60°C, 70°C, 80°C, 100°C, and 175°C. Room temperature hardness, tension and fatigue crack growth tests have been completed to determine changes in mechanical properties in the LT orientation. Longitudinal splitting in the short-transverse (ST) direction has been observed during fatigue after sufficient time and temperature exposure combinations. Fracture and fatigue testing has additionally been employed on long-time sensitized material. The evolution of properties (i.e. mechanical/fracture/fatigue, corrosion resistance, etc.) and microstructures (e.g. grain boundary segregation, grain boundary precipitation, etc.) with thermal exposure at these (and lower) temperatures has significant practical implications for various structural applications. The effects of thermal remediation treatments are also described.
Acknowledgements

First, I must give thanks to my God, through whom all good things come to be.

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1 Introduction & Background

1.1 Introduction to Aluminum and Aluminum Alloys

Aluminum is a metallic element having the chemical symbol Al, with atomic number 13 and atomic weight 27. Aluminum is the third most common element in the earth’s crust, coming after oxygen and silicon. About 8% of the crust’s total mass contains Aluminum as oxide and silicate so it is the most abundant metal [1]. Aluminum is the most heavily consumed non-ferrous metal in the world, with current annual consumption at 24 million tons [2]. Furthermore, Aluminum’s unique properties, including light weight, high strength if alloyed, and resistance to corrosion in some conditions, make it an ideal material for use in conventional and novel applications especially automotive, marine industry, construction of buildings, production of defense and aerospace equipment [2]. Unfortunately, commercially pure Aluminum has relatively low yield strength of 20 MPa and an ultimate tensile strength of 30 – 70 MPa, depending on the purity of metal, when tested at room temperature [2].

Aluminum, like all pure metals has a low strength, and cannot be readily used in applications where resistance to deformation and fracture is essential [2]. Therefore, due to the lack of enough mechanical strength, the use of pure Aluminum is limited largely to cladding applications [2]. Therefore, the vast majority of the consumption of Aluminum arises from the use of wrought Al-alloys. These alloys can be divided into families, depending on the alloying elements present, as summarized in Table 1.1. All of the families
of Al-alloys offer superior mechanical properties except 1xxx series due to various strengthening mechanisms (i.e. solid solution strengthening, precipitation hardening, etc.). Although pure Aluminum is very resistant to corrosion because of the presence of an Aluminum oxide film, corrosion resistance generally decreases with increasing alloy content, so tempers have been developed to improve the corrosion resistance of highly alloyed materials [2]. A summary of the different temper designations, as governed by ANSI standards is provided in Figure 1.1.

Although the low strength of pure Aluminum limits its usefulness, Aluminum has a theoretical shear strength of between 0.9 and 2.62 GPa depending on whether the displacement of atoms follow a straight or zig zag path. The significant difference between the strength of commercial alloys and theoretical strength is due to presence of lattice defects, mainly dislocations. Therefore all strengthening mechanisms in commercial alloys are aimed at limiting the movement of dislocations [2].

<table>
<thead>
<tr>
<th>Family</th>
<th>Principal alloying elements present</th>
</tr>
</thead>
<tbody>
<tr>
<td>1xxx</td>
<td>Little alloying present</td>
</tr>
<tr>
<td>2xxx</td>
<td>Cu</td>
</tr>
<tr>
<td>3xxx</td>
<td>Mn</td>
</tr>
<tr>
<td>4xxx</td>
<td>Si</td>
</tr>
<tr>
<td>5xxx</td>
<td>Mg</td>
</tr>
<tr>
<td>6xxx</td>
<td>Mg &amp; Si</td>
</tr>
<tr>
<td>7xxx</td>
<td>Zn</td>
</tr>
<tr>
<td>8xxx</td>
<td>Misc. (Many alloys include Sn &amp; Li)</td>
</tr>
<tr>
<td>9xxx</td>
<td>Reserved for future use</td>
</tr>
</tbody>
</table>

Table 1.1: Major families of Al-alloys. Reproduced from [3]
<table>
<thead>
<tr>
<th>4-digit series</th>
<th>Aluminium content or main alloying elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 xxx</td>
<td>99.0% minimum Copper</td>
</tr>
<tr>
<td>2 xxx</td>
<td>Manganese</td>
</tr>
<tr>
<td>3 xxx</td>
<td>Silicon</td>
</tr>
<tr>
<td>4 xxx</td>
<td>Magnesium</td>
</tr>
<tr>
<td>5 xxx</td>
<td>Magnesium and silicon</td>
</tr>
<tr>
<td>6 xxx</td>
<td>Zinc</td>
</tr>
<tr>
<td>7 xxx</td>
<td>Others</td>
</tr>
</tbody>
</table>

Figure 1.1: Aluminum alloy and temper designation systems (IADS system). Reproduced from [4].

### 1.2 Al-Mg Alloys (5xxx Series)

The major 5xxx structural alloys were developed in the 1950’s and have been used for shipbuilding, armor plate, railroad cars, and tankage for almost 50 years. In most of these applications, strength after welding is a major consideration and higher Mg, Mn and Cr contents are important in maintaining as-welded mechanical properties. The most common
alloys used historically in high-strength applications are 5083, 5454, 5086 and 5456 [5]. Another recent development, alloy 5059, utilizes a Zn addition to a 5.5% Mg alloy to increase the strength and corrosion resistance. Although alloy 5059 has not been thoroughly tested in service, previous Zn-containing alloys (5080 and 5084) were registered and then withdrawn from use because of severe localized corrosion in the (heat affected zones (HAZ) adjacent to welds [5]. Chemical composition limits for high strength Al-Mg-Mn 5xxx alloys is shown in Figure 1.2.

![Figure 1.2: Chemical composition limits for high strength Al-Mg-Mn 5xxx alloys [5].](image)

Binary Aluminum-Magnesium alloys are the basis for an important class of non-heat treatable alloys (5XXX series alloys). Although Magnesium has substantial solubility in
solid Aluminum, binary alloys do not show appreciable precipitation-hardening characteristics with concentration below 7% Magnesium [6]. However, Magnesium provides substantial solid solution strengthening with good ductility as a result of cold work, in addition to excellent corrosion resistance and weldability. As a result, Al-Mg 5xxx series alloys are used in a wide range of marine applications especially for super structures and hulls [4]. Furthermore, these are desirable due to their excellent balance of solid solution strengthening of Mg in the Al matrix. Figure 1.3 shows the binary Al-Mg phase diagram.

Figure 1.3: Al-Mg binary phase diagram. Reproduced from [7]
While most Al 5xxx series alloys have excellent corrosion resistance, even in marine environments, it is possible for high magnesium alloys to suffer a particular type of accelerated corrosion in marine environments. This degradation, known as sensitization, is characterized by Mg migration to the grain boundaries of the material and the formation of Al3Mg2 precipitates (β-phase) [8]. High Mg alloys, including 5059, 5083, 5456, contain 4 to 6 percent Mg, which is above the solubility limit of Mg in Aluminum at room temperature, which is less than one percent as shown in the phase diagram in Figure 1.3 [8]. The nominal chemical compositions of 5059, 5083, 5086, 5383 and 5456 are shown in Table 1.2.
Table 1.2: Nominal chemical compositions of the selected Al-Mg alloys [38]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Silicon</th>
<th>Iron</th>
<th>Copper</th>
<th>Manganese</th>
<th>Magnesium</th>
<th>Chromium</th>
<th>Zinc</th>
<th>Titanium</th>
<th>Other Elements&lt;sup&gt;D&lt;/sup&gt;</th>
<th>Aluminum</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Each</td>
<td>Total&lt;sup&gt;E&lt;/sup&gt;</td>
</tr>
<tr>
<td>5059</td>
<td>0.45</td>
<td>0.50</td>
<td>0.25</td>
<td>0.6 to 1.2</td>
<td>5.0 to 6.0</td>
<td>0.25</td>
<td>0.4</td>
<td>0.9</td>
<td>0.05&lt;sup&gt;F&lt;/sup&gt;</td>
<td>0.15</td>
</tr>
<tr>
<td>5083</td>
<td>0.40</td>
<td>0.40</td>
<td>0.10</td>
<td>0.40 to 1.0</td>
<td>4.0 to 4.9</td>
<td>0.05 to 0.25</td>
<td>0.25</td>
<td>0.15</td>
<td>0.05</td>
<td>0.15</td>
</tr>
<tr>
<td>5086</td>
<td>0.40</td>
<td>0.50</td>
<td>0.01</td>
<td>0.20 to 0.7</td>
<td>3.5 to 4.5</td>
<td>0.05 to 0.25</td>
<td>0.25</td>
<td>0.15</td>
<td>0.05</td>
<td>0.15</td>
</tr>
<tr>
<td>5383</td>
<td>0.25</td>
<td>0.25</td>
<td>0.20</td>
<td>0.7 to 1.0</td>
<td>4.0 to 5.2</td>
<td>0.25</td>
<td>0.40</td>
<td>0.15</td>
<td>0.05&lt;sup&gt;G&lt;/sup&gt;</td>
<td>0.15</td>
</tr>
<tr>
<td>5456</td>
<td>0.25</td>
<td>0.40</td>
<td>0.10</td>
<td>0.50 to 1.0</td>
<td>4.7 to 5.5</td>
<td>0.05 to 0.20</td>
<td>0.25</td>
<td>0.20</td>
<td>0.05</td>
<td>0.15</td>
</tr>
</tbody>
</table>
However, it is additionally desirable that the mechanical properties of these alloys do not degrade during the extended service life of aquatic structures and other components. This can be very problematic as degradation in the mechanical properties can occur in alloys with greater than 3 or 4 wt% Mg in solution [4]. In the high Mg content alloys, Mg can segregate to slip bands and grain boundaries, eventually forming the equilibrium $\beta$-phase as shown by the phase binary diagram of Figure 1.3.

1.2.1 Use of Aluminum alloys in Marine Environments

The initial choice of using an Aluminum alloy for marine applications (fresh water with a pH of 4.0 to 8.5) relates to the protective and passive oxide film which forms on the surface of the material and upon damage, quickly reforms. Adding Mg in order to solid solution strengthen 5xxx series Aluminum alloys produces a stronger yet light weight material with general corrosion resistance. Aluminum alloys considered to be marine grade include either the H116 or H321 temper, and meet the specification of ASTM standard B928 which is the standard for high magnesium Aluminum alloy sheet and plate for marine services and similar environments [9]. Also, Aluminum alloys considered to be armor grade include H131 or H136 and meet the specification of Military standard MIL-DTL-46027K(MR) which is the standard for armor plate, aluminum alloy [10]. The alloys with H temper are considered strain hardened alloys and they are not heat treatable. Their strength is increased through strain hardening at room temperature. The specific tempers for marine grade alloys are defined by the Aluminum Association [11] as follows:
“H116 - Applies to products manufactured from alloys in the 5xxx series, for which the magnesium content is 3% nominal or more. Products are normally strain hardened at the last operation to specified stable tensile property limits and meet specified levels of corrosion resistance in accelerated type corrosion tests. They are suitable for continuous service at temperatures no greater than 150°F (66°C). Corrosion tests include intergranular and exfoliation.”

“H321 - Applies to products from alloys in the 5xxx series, for which the magnesium content is 3% nominal or more. Products are normally thermally stabilized at the last operation to specified stable tensile property limits and meet specified levels of corrosion resistance in accelerated type corrosion tests. They are suitable for continuous service at temperatures no greater than 150°F (66°C). Corrosion tests include intergranular and exfoliation.”

However, recent investigations have revealed that Al 5xxx alloys microstructures may not be stable due to phase evolution under certain conditions. Aluminum alloy 5456 (AA5456) is a good example providing all the advantages and disadvantages. AA5456 is currently used in many superstructures, including US navy CG-47 class cruisers [12]. Cracks in the superstructure have been observed and they were not attributed to stress concentration [12]. Navy investigations revealed cracks were caused by stress corrosion cracking and all cracks were located in 5456-H116 material. Stress corrosion cracking requires three conditions: 1. susceptible material. 2. Tensile loading. 3. Corrosive environment. Although 5xxx alloys are fairly immune to corrosion, microstructural change can impact corrosion
performance. Sea water exposure is sufficient to provide corrosive environment and as result instability can occur [12].

Furthermore, the Marine Aluminum alloys are important because 50 percent of future U.S. Navy structures to be built in the next five years will be fabricated either entirely or partially of Aluminum alloys [8]. Recently, the Office of Naval Research has developed a program to advance the science and technology of ship structures as the ONR Ship Structural Reliability Program [8]. As it is shown in Figure 1.4, ship reliability depends on different factors including prediction methods, structural health monitoring and structural testing. In this study we pursue this approach by following systematic mechanical testings of samples given thermal treatment.

Figure 1. 4: Monitoring ship reliability [8] as taken from the ONR Ship Structural Reliability Program
1.3 Sensitization of AA5XXX Alloys

Although there are desirable properties of 5xxx alloys for marine applications, they have presented a strong susceptibility to stress corrosion cracking. Sensitization is caused in Aluminum alloys with greater than 3 wt% Mg in solid solution undergoing thermal exposure. At elevated temperature Mg segregates to the grain boundary, eventually forming the equilibrium $\beta$-phase ($\text{Mg}_2\text{Al}_3$). After the material undergoes this transformation it is considered sensitized if it fails to pass certain tests [13]. This segregation and eventual formation of $\beta$-phase is a function of time and temperature [4], [14].

1.3.1 Precipitation Behavior in 5xxx Alloys

The precipitation nucleation and growth behavior of the $\beta$-phase in AA5083 has been investigated by Goswami, et al. using Transmission Electron Microscopy (TEM). From this work, which exposed AA5083 to 175°C for 240 hours, Goswami was able to characterize the microstructure and subsequently $\beta$ formation during the annealing process. As presented by Goswami, et al., no $\beta$-phase ($\text{Al}_3\text{Mg}_2$) was observed in the as received sample. The fine scale microstructure of the as received sample showed a number of rod-like and equiaxed precipitates in Al matrix and using EDS technique they were able to map the particles which is shown in Figure 1.5. It is shown that particles contain Mn and Fe beside the Mg and Cr.
Figure 1.5: (a) A bright-field (BF) TEM image showing the rod-like and equiaxed particles in Al-matrix. (b) HAADF image of Mn-rich particle with a bright contrast. (c) Fine probe EDS maps of Al, Mg, Mn, Fe, and Cr from the particle shown in (b). (d) EDS from the Mn-rich, Fe-rich particles and the matrix [15].

On the other hand, the fine scale microstructure of the annealed sample after 175°C for 240 hours was significantly different from the as-received specimen. It was found that precipitation of Mg-rich precipitates occurred on top of Mn-rich rods. This is shown in Figure 1.6 in the TEM bright-field image. Also the high-angle annular dark-field (HAADF)
image showed that the more equiaxed precipitate is Mg-rich (dark) while the rod-like precipitate is Mn-rich (bright).

Figure 1. 6: (a) A bright-field image of the annealed sample showing $\beta$-phase on existing Mn-rich particles. Arrows showing Mn rich particles and Mg rich regions. (b) HAADF image showing a rod-like bright Mn-rich particle, and a dark region partially surrounding the rod-like particle due to Mg enrichment. (c) Fine probe EDS maps of Mg, Al, Mn and Cr [15].
Furthermore, Goswami showed that a large number of very fine faults are present in the \(\beta\) phase that formed atop on Al-Mn type particle as shown in Figure 1.7.

![Multibeam image of \(\beta\)-phase formed on rod-like Al-Mn type particle](image)

**Figure 1.7: A multibeam image of \(\beta\)-phase formed on rod-like Al-Mn type particle** [15].

Based on these observations, it was revealed that most of the grain boundaries in the as-received AA5083 are precipitate free while there is significant grain boundary precipitation of \(\beta\) in the sensitized specimen, as expected. TEM images showed that Mg-rich \(\beta\)-phase formed a continuous film at a number of grain boundaries after annealing. This is shown in the Figure 1.8 as HAADF image of tilted grain boundary which shows a dark 150 nm thick band. EDS line scan shows enrichment of Mg. It is clear also by moving away from
the grain boundary, the amount of Mg decreased. TEM observations in this study, revealed a mostly continuous layer of \( \beta \) formed at the grain boundaries and the thickness of the \( \beta \)-phase is about 50 to 190 nm after sensitization.

Figure 1.8: (a) Bright-field TEM image showing the \( \beta \)-phase at the grain boundary for sample aged for 10 h at 448 K (175°C). (b) A HAADF image showing the grain boundary \( \beta \)-phase (appeared dark). (c) and (d) The distribution of Mg and Al from this region, respectively. (e) The EDS line across the grain boundary \( \beta \)-phase obtained from the region (thick arrow), as shown in “b.” The grain boundary position is shown by the dashed line [15].
In another study by Goswami et al. [16] 5083-H131 was annealed at 175°C for 1 hour. In order to characterize this grain boundary, Selected Area Electron Diffraction (SAD) pattern and fine probe EDS were used. Grain boundary presence of $\beta$ is shown in Figure 1.9 (a) in the bright field TEM image. The $\beta$ is not contiguous and formed as allotriomorphs at different locations. Also the Mg content was obtained from the $\beta$–phase precipitate as about 33wt% which is 10 times in respect to the Al matrix between two such allotriomorphs. In the previous work [15] such $\beta$ allotriomorphs were present along the grain boundary after 10 hours of annealing.

Figure 1.9: (a) Bright-field TEM image for sample aged for 1 h at 448 K (175 °C) showing the formation of $\beta$-phase, shown by arrows, at the grain boundary. A dark-field TEM image obtained using (777) reflection of the $\beta$-phase from region (1) is shown as an inset. (b) The fine probe EDS from the $\beta$-phase. (c) The fine probe EDS from the grain boundary between two $\beta$-particles [16]
In addition, Goswami et al. [16] had shown the evolution of the grain boundary β phase as a function of aging time in Figure 1.10. A number of grain boundaries were observed to be fully covered by the β phase for samples aged for greater than 240 hours.

Figure 1.10: (a) through (d) Bright-field TEM images showing the β phase at the grain boundary for samples aged for 10, 25, 240, and 1000 hrs [16].

In other work, Holtz et al. [17] was able to show the evolution of β-phase at different levels of sensitization in AA5083-H131. As is shown in Figure 1.11, (a) the as received specimen exhibits clean grain boundaries without any β-phase. After annealing at 175°C for 1 hour,
β allotriomorphs form as discontinuous sites. By extending the annealing time to 50 hours, ribbon-like β formed which is shown in (c). And finally, the fully sensitized sample at 175°C for 240 hours shows a continuous film of β phase.

Figure 1.11: Basic stages of the microstructure evolution of β-Al3Mg2 in Al 5083-H131 aged at 448 K (175°C). The condition depicted in (d) is considered to be fully sensitized [17].

Holtz et al. [17] were able to show the change in the thickness of the β film as function of temperature and time. This is shown in Figure 1.12 where it can be seen that the β film thickness increased by increasing the annealing temperature from 70°C to 175°C [18].
The works by Searles et al. [19], comprehensively analyzed a 5083 alloy (unspecified temper) and aged at 150°C for up to 333 hours. β-phase formation was found to occur at the grain boundaries in a continuous manner. However, longer exposure times revealed that the grain boundary phase was broken up and discontinuous [19]. Therefore there remains controversy over the continuity or discontinuity of this complex phase at different degrees of sensitization.

It was also shown [19] that the dislocation density in the as-received 5083-H131 sample (which was work hardened at room temperature) was extremely high. Even after annealing and some recovery, the dislocation density is still significantly high as shown in Figure 1.13. The bright field image, and corresponding weak beam image, shows a dislocation network adjacent to the grain boundary in the annealed sample [15]. The importance of dislocations near grain boundary region is that diffusivity can be enhanced by several orders of magnitude [20] through pipe diffusion at low temperature (<0.4 Tm). This may also contribute significant effects on the sensitization of this alloy.
1.3.2 Environmental Effects on the Corrosion, Corrosion Fatigue of 5xxx Alloys

The environmental effects on the corrosion and mechanical behavior of Aluminum alloys have been investigated via different works a number of years ago. Water vapor in air is well known to accelerate the fatigue crack growth rate in Aluminum alloys and this has been confirmed through different works [21–25]. It is believed that hydrogen assists the fatigue crack growth rate upon water vapor exposure. It has been reported that depending on the alloy chemistry and applied stress intensity, fatigue crack growth can be increased up to ten times in ambient air over vacuum [21–25]. Moreover, fatigue crack initiation behavior of welded AA5083 was analyzed by Devries et al. [26] in seawater and it was found that penetration of seawater into surface pores of welded materials can significantly reduce the fatigue life time which is as a result of a stagnant environment. The presence of...
pores in the sub-surface of welded material is shown in the fractograph of the fatigued sample in Figure 1.14.

![Fractograph](image)

Figure 1.14: Fracture surface of bulk material fatigued in seawater: multiple initiation locations (left) and glazed appearance of initiation sites (right) [26].

Donald [27] more recently reported a wide range of fatigue crack growth results for as-received 5383-H116 and 5083-H321 and two more alloys tested in air and seawater. It appeared that the environmental effects were greater with the 5083 alloy based on the fatigue crack growth data. Fatigue crack growth rate in the low $\Delta K$ regime seemed to be increased in seawater. In another attempt, Pao et al. [28] conducted a study on the fatigue crack growth rates in high vacuum and ambient air of ultrafine grained Al-Mg alloy. Fatigue crack growth rates of their materials also were two to ten times higher in ambient air than vacuum which is shown in Figure 1.15.
Figure 1.15: The difference in driving force, $\Delta K$, between vacuum and air at the same crack growth rate is associated with the change in driving force due to environmental effects [28].

Additionally, Menzmer and Sritvatsan [29] were able to get similar results for as-received 5456-H116 alloy tested via fatigue crack growth in three different environments. They used laboratory air, water vapor and oxygen. It was found that at identical stress intensity, the rate of fatigue crack growth in both air and pure water vapor is faster than oxygen and presence of water vapor or moisture almost enhanced crack propagation by factor of three. This comparison is shown in Figure 1.16.
More recently, Holtz et al. [17] studied the fatigue crack growth behavior of AA5083-H131 in different environments, including high vacuum, air and an aqueous solution containing NaCl and an inhibitor at low and high load ratio for both as-received and sensitized conditions. It was found that there is significant environmental enhancement of the crack growth rates in air, for both low and high R ratio, relative to the behavior in vacuum. Also sensitization does not affect the fatigue crack growth behavior dramatically in vacuum or air at low or high ratio as it is shown in Figure 1. 17.

Figure 1. 16: Comparison of the fatigue crack growth response of alloy 5456–H116 in the environments of laboratory air, water vapor and oxygen [29]
Figure 1.17: Fatigue crack growth rates at $R = 0.1$ (a) and $R = 0.85$ (b) in vacuum, air, and 1 pct NaCl + inhibitor. In the legend, “A” refers to as-received or unsensitized condition, and “S” refers to fully sensitized condition. The DOS and the arrow indicate the shift of threshold with increasing sensitization [17].

Holtz et al. also claimed that the change in aging behavior at around 200 hours aging seems to be correlated with the transition from discontinuous or partial $\beta$ phase coverage of the grain boundaries to continuous and full $\beta$ coverage as it is shown in Figure 1.18.
Figure 1.18: ASTM G-67 nitric acid mass loss vs. aging time for Al 5083-H131 aged at 448 K (175°C). The approximate aging time corresponding to the observed transition between partial coverage/discontinuous β grain boundary phase and full coverage/continuous β phase is indicated [17].

Bovard [30] also showed in several thermal exposure treatments of AA5083 the susceptibility of materials under pre-cracked double cantilever beam (SCB) testing by exposure to 2.5% NaCl. It was shown that crack growth rates increased with increasing mass losses. Additionally, cracks tended to grow out of plane and SEM fractography of these specimens shows secondary cracking on the S-L plane meaning perpendicular to the fatigue pre-crack as illustrated in Figure 1.19.
Degradation mechanisms in aluminum include sensitization, corrosion fatigue, and pitting. Degradation occurs to the mechanical properties that measure strength, toughness, fatigue strength, and fatigue crack growth rates. Some data exists on this degradation, but it needs to be better quantified to support forecasting of structural health over time [8].

1.4 Remediation/Reversion or Stabilization of Sensitization in Sensitized Alloys

The manufacturing process of 5xxx plate alloys often occurs by rolling, followed by a stabilization treatment. Although stabilization treatments are often to avoid age-softening, there is another stabilization treatment used in 5xxx alloys to enable magnesium to precipitate at the grain boundary or discontinuously on grain boundaries to reduce the likelihood of future sensitization [5]. However this kind of stabilization may be difficult to apply in practice due to the narrow temperature range where benefits may be observed.
Low temperature treatments can produce $\beta$ precipitation on grain boundaries, and sensitization could be accelerated. While higher temperature treatments can reverse the process and put Mg back in to solution, this will reduce the strain hardening imparted earlier that created the H temper. Kramer et al. [31] investigated similar kinds of heat treatments and reversion processes during the milling process in order to evaluate the reversion of sensitization and restoration of the corrosion performance. In that work [31] they were able to provide a schematic that summarized the stabilization temperature range for Al-Mg alloys, based on the earlier report by Scamans [32] which is shown in Figure 1.20.

![Figure 1.20: Schematic of effect of temperature on stabilization and sensitization of Al-Mg alloys based on the Mg weight% [31]](image)

These alloys can be annealed at temperatures above the $\beta$ phase solid solubility limit.

Kramer et al. [31] used different treatments in order to de-sensitize plate that was known to be susceptible to intergranular corrosion. They used 5456-H116 plate and first sensitized
it at 150°C for 24 hours. Temperatures of 200 to 340°C were then used to determine the best stabilization treatments in addition to evaluating how much softening the remediation treatments could cause via subsequent tensile testing. Based on the results of their tensile testing summarized in Figure 1.21 it was concluded that stabilization below 300°C does not significantly affect strength in this alloy. Kramer et al. [31] suggested that a reasonable temperature range for remediation might be 240°C to 280°C while exposure for 10 minutes would be enough to effectively dissolve the grain boundary $\beta$. Below this range, it was proposed that plates would remain sensitized while above this range the yield strength would be significantly reduced, as shown in Figure 1.21.

![Figure 1.21: Effect of exposure temperature on the Yield strength and Nitric Acid Mass Loss Test (NAMLT) results of 5456 alloy. Increasing temperature decreases the Yield strength and mass loss. [31]](image)

The term stabilization refers to stabilizing both the mechanical and the corrosion properties of AA5xxx alloys [32]. Scamans [32] showed that based on the Magnesium level in the
5xxx alloy, the stabilization treatment temperature may need to be changed. For instance, for AA5083 with 4.5% Mg, the stabilization window could be quite narrow and is between 220°C and 240°C [32]. While stabilization heat treatments at 200°C will initially sensitize AA5083, longer treatment times were shown to eventually provide stabilization due to coarsening of grain boundary precipitate along with some dissolution of the intergranular β phase back into solution [32].

1.5 Purpose of Present Study

The previous researches presented in the preceding sections along with previous work by a former student [33] reveals that the evolution of β phase, corrosion properties and some mechanical properties after high temperature thermal exposures for short times have been well studied. However, there is still lack of information on the mechanical behavior and effects of long time thermal exposures at lower temperatures. Also, there is not much information regarding the strategies for reversion and stabilization of these alloys. Thus, specimens of different alloys including 5083-H116, 5456-H116, 5456-H131 and 5059-H131 were thermally exposed to temperatures of 60°C, 70°C, 80°C, and 175°C for up to 10,000 hours. After exposure and based on the information provided in previous work [33], a series of mechanical tests were conducted in order to more completely understand the evolution of mechanical properties. Also, some stabilization strategies have been investigated. SEM and EDX analysis techniques were used to characterize the changes in the structure and fracture surface behavior of materials. Suggestions for future work, based on the findings are presented as part of larger continuing project.
2 Experimental Methods

2.1 Materials

Five different commercially available 5xxx Al-Mg alloys were used to investigate the effects of low (e.g. 60°C, 70°C) and intermediate (e.g. 80°C, 100°C, 175°C) temperature thermal exposures on the hardness, strength and fatigue crack growth properties. Solid solution strengthened Al-Mg alloys 5083, 5456 with an H116 temper and 5083, 5456, 5059 with H131 temper were obtained. The H116 temper is a standard temper for aquatic applications and is achieved from cold rolling while H131 is a strain hardened temper. Tensile properties reported for H116 temper for all of the alloys investigated are given in Table 2.1 [1, 2]. The nominal chemical compositions of these alloys are provided in Table 2.2, along with the alloy specifications. Appendix A in compact disk (CD) format provides schematics of each plate and dimensions, with details below.

As-received bulk materials were obtained in the form of plate and included 5456-H116 and 5083-H116 plate, 6.35 mm thick, used in the MS thesis of Justin Brosi [33]. Sample designations for 5083-H116 were NSWC1 while that for 5456-H116 was NSWC3. Two additional 12.6 mm thick 5083-H131 and 5456-H116 plates from previous work [33] are identified as Plate C and D, respectively, in Appendix A in compact disk (CD).

6.2 mm thick 5059-H131 plate obtained from Aleris is identified as Plate A in Appendix A in compact disk (CD). Two identical 9.5 mm thick plates of 5083-H131 obtained from the Army Research Laboratory at the Aberdeen Proving Ground, Md are identified as Plate B in Appendix A in compact disk (CD). Two identical 29.2 mm thick plates of 5456-H131
were obtained from the Alcoa Technical Center which are identified as Alcoa Plate in Appendix A.

Table 2.1: Tensile Properties

<table>
<thead>
<tr>
<th>Grade</th>
<th>Min 0.2% σ yield (MPa)</th>
<th>Min UTS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083- H116</td>
<td>215</td>
<td>305</td>
</tr>
<tr>
<td>5456- H116</td>
<td>230</td>
<td>315</td>
</tr>
<tr>
<td>5083- H131</td>
<td>249*</td>
<td>310*</td>
</tr>
<tr>
<td>5456- H131</td>
<td>249*</td>
<td>310*</td>
</tr>
<tr>
<td>5059- H131</td>
<td>272*:^</td>
<td>360*:^</td>
</tr>
</tbody>
</table>

*,^: [35], [36], [10]

Table 2.2: As Received Chemistries

<table>
<thead>
<tr>
<th>Grade</th>
<th>wt% Mn</th>
<th>wt% Mg</th>
<th>wt% Cr</th>
<th>wt% Ti</th>
<th>wt% Cu</th>
<th>Wt% Si</th>
<th>wt% Fe</th>
<th>wt% Zn</th>
<th>wt% Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083- H116^a</td>
<td>0.53</td>
<td>4.40</td>
<td>0.08</td>
<td>0.02</td>
<td>0.03</td>
<td>0.10</td>
<td>0.22</td>
<td>0.03</td>
<td>Bal.</td>
</tr>
<tr>
<td>5456- H116^b</td>
<td>0.64</td>
<td>4.81</td>
<td>0.07</td>
<td>0.02</td>
<td>0.09</td>
<td>0.12</td>
<td>0.32</td>
<td>0.08</td>
<td>Bal.</td>
</tr>
<tr>
<td>5083- H131^c</td>
<td>1.00</td>
<td>4.90</td>
<td>0.25</td>
<td>0.15</td>
<td>0.10</td>
<td>0.40</td>
<td>0.40</td>
<td>0.25</td>
<td>Bal.</td>
</tr>
<tr>
<td>5456- H131^c</td>
<td>1.00</td>
<td>5.50</td>
<td>0.20</td>
<td>0.20</td>
<td>0.1</td>
<td>0.25</td>
<td>0.19</td>
<td>0.25</td>
<td>Bal.</td>
</tr>
<tr>
<td>5059- H131^d</td>
<td>1.20</td>
<td>6.00</td>
<td>0.25</td>
<td>0.20</td>
<td>0.25</td>
<td>0.45</td>
<td>0.50</td>
<td>0.90</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

^a,b: ASTM Standards [37], [38], ^c: [10], ^d: [36]
2.2 Microstructure Evaluation

2.2.1 Metallography

Metallographic evaluation was conducted on the as-received material via optical microscopy at 40x magnification. Small regions of 5059-H131 and 5456-H131 were sectioned, mounted and then polished to a 0.04 μm finish using successive steps of SiC paper, diamond suspension and Al₂O₃ suspension polishing. Following the advice of [39], the polished 5456 and 5059 surfaces were etched at room temperature in modified Keller’s Reagent for 5-7 minutes. Results of Metallography are provided in chapter 3. Two other alloys were characterized in the previous work [33].

2.2.2 Electron Backscatter Diffraction (EBSD) / Orientation Image Mapping (OIM)

Electron backscattered diffraction (EBSD) was performed with scanning electron microscope (SEM) to provide information on crystallographic orientation. After obtaining fine polished sample from the metallographic specimen preparation technique, a sample was polished using the Vibromet machine for three hours and using 1 μm MetaDi Supreme suspension with 5 lbs load to get the best possible fine polished surface. This is crucial for EBSD observation in order to limit the amount of surface deformation and enable formation of diffraction pattern from of the sample. Spot size of 4.5 and high voltage of 15 KeV was used at 10,000 x magnification. Results of EBSD are provided in chapter 3.

2.3 Specimen Geometries

The dimensions of the bend bars are shown in Figure 2.3. All bend bar specimens were removed with the long axis of each specimen parallel to the rolling direction of the plates,
corresponding to the ASTM E-399 LT orientation [40]. This removal configuration is shown schematically in Figure 2.1. The only plate sufficiently thick to remove tension samples from the ST direction was the 5456-H131 plate obtained from Alcoa. The tensile specimen geometry and orientation of tension samples specimens removed from the Alcoa plate in the short transverse direction is schematically shown in Figure 2.2 and Figure 2.3 respectively.

**Figure 2.1: Bend Bar Specimen Geometry.**

**Figure 2.2: Tensile Specimen Geometry.**
2.4 Thermal Exposure Treatments

After precision machining of all samples from the plate stock, samples were thermally exposed in separate Blue M furnaces maintained at temperatures of 60°C, 70°C, 80°C, 175°C for times of 100 hrs, 500 hrs, 2000 hrs, 4000 hrs, 10000 hrs, 15000 hrs and 20000 hrs in order to simulate the low and intermediate temperature sensitization for short, intermediate and long time exposures. The long exposure times were selected to provide information relevant to the potentially long service thermal exposure experienced by such materials.

The details of thermal exposure time and temperatures for each of the alloys are provided in the Table 2.3.
Table 2.3: Thermal Exposure time and temperatures

<table>
<thead>
<tr>
<th>Temp</th>
<th>As Received</th>
<th>100 hrs (4 days)</th>
<th>500 hrs (17 days)</th>
<th>2000 hrs (83 days)</th>
<th>4000 hrs (166 days)</th>
<th>5000 hrs (208 days)</th>
<th>10,000 hrs (415 days)</th>
<th>15,000 hrs (622 days)</th>
<th>20,000 hrs (830 days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60°C</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>ongoing</td>
<td>ongoing</td>
<td>ongoing</td>
<td>ongoing</td>
</tr>
<tr>
<td>70°C</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>ongoing</td>
<td>ongoing</td>
<td>ongoing</td>
<td>ongoing</td>
</tr>
<tr>
<td>80°C</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>175°C</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
</tbody>
</table>

2.5 Possible Remediation Treatments for Sensitized Material

Various techniques were explored to reverse the detrimental effects of sensitization. These included both mechanical and thermal treatments described below.

2.5.1 Mechanical Remediation Treatments

Sensitized specimens were deformed at room temperature to further reduce the plate thickness using an MTS 50 KIP servo hydraulic testing machine. Sensitized samples were deformed at room temperature to the following strain levels: 1.7%, 3.7%. Subsequent mechanical testing was conducted in a manner similar to the tests described below.

2.5.2 Thermal Stabilization/Reversion Treatments

Thermal remediation treatments were also conducted on separate samples to stabilize/reverse the sensitization based on the results of Kramer et al [31]. Initial attempts used 240°C, 260°C or 280°C exposures for 15 minutes conducted in Blue M ovens. Other work by Scamans [41] showed that the amount of Magnesium in the alloy would affect the thermal stabilization treatment. For instance the stabilization window for AA5083 is quite small and is between 220 °C and 240°C. For AA5456 this range is between 210°C and
260°C. For 5059 this range is between 240°C and 280°C. In order to accommodate the Scamans [41] work, a limited number of AA5456 and AA5083 specimens were given thermal treatments for longer times, ranging from 1-5 hours.

2.6 Mechanical Testing

The effects of thermal exposure on the mechanical behavior of these alloys and the effectiveness of remediation strategies after thermal exposure/aging were evaluated by hardness testing, tension testing and fatigue crack growth testing. All tests were carried out in the Advanced Manufacturing and Mechanical Reliability Center (AMMRC) at Case Western Reserve University (CWRU).

2.6.1 Hardness Testing

Rockwell B hardness testing was conducted following ASTM E18 standard [42] using 100 kg force onto the flat surface of a specimen using 1/16” steel ball. Average results were calculated from at least five indentations on the samples.

2.6.2 Tension Testing

Tension testing was conducted in the short transverse direction using Instron 1361 screw driven machine on a limited number of 5456-H131 specimens. In some cases specimens were immersed in water for specific amount of time prior to testing. Test Works 4 testing software was used for controlling the machine and data acquisition. Displacement rates of 0.0036 and 0.804 mm/min provided strain rates of 5 x 10^{-6}/s and 1 x 10^{-3}/s respectively. Crosshead displacement was recorded at rate of 0.05 Hz. All specimens were tested to failure based on the ASTM E8/E8M standard [43].
2.6.3 Fatigue Crack Growth Testing in Air

Fatigue crack growth testing was conducted using DCPD (Direct Current Potential Drop) technique following ASTM E647 standard [44]. Small holes were first drilled on the surface of bend bars in order to subsequently attach the current and voltage leads to the specimen by peening. The current and voltage lead holes were drilled with diameter of 0.60 mm and 0.83 mm in order to accommodate wires of 0.50 mm and 0.81 mm respectively. Also, a 500 μm diameter notch was introduced in the bend bars using a 20 HC diamond wafer blade at 3000 rpm on a low speed wafering saw. In all cases, the notch was created in the long-transverse (LT) direction as shown in the Figure 2.4 except for 5456-H131 which the notch was created in LS direction as shown in Figure 2.3. The nominal notch length was about 2.75 mm and 7.75mm for LT and LS direction respectively, and before each test a careful measurement was conducted using a digital caliper. The fatigue setup is shown in Figure 2.5.

Fatigue testing was conducted on the MTS 50 kip, 20 kip and Instron 1331 servo-hydraulic machines. The specimens were loaded into the machines in three point bending configuration (3PB) as shown in Figure 2.5. The loading span for long bars (i.e. l=60 mm) was 38.1 mm and for the short bars (i.e. l=30 mm) was 18.1 mm. In all testing an R-ratio which is defined as:

\[
R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} = \frac{K_{\text{min}}}{K_{\text{max}}}
\]  

(2.1)

An R of 0.3 was used to minimize the movement of the samples during testing. The cyclic frequency was set at 20 Hz and all tests were conducted based on the ASTM E647 standard
The FTA, Inc. FCGR (Fatigue Crack Growth Rate) testing software was used for controlling the test, data acquisition and data analysis purposes. The software automatically adjusts the data collection rate depending on the crack growth observed from the DCPD measurements. The resulting data was smoothed using the modified-secant method [44].

Data is presented as differential crack growth per cycle ($\frac{da}{dN}$) versus $\Delta K$ on a log-log scale.

Fatigue crack growth tests were designed to load shed from an intermediate $\Delta K$ until the fatigue threshold was established. This threshold test acquisition was continued until a sizable amount of data existed below the ASTM E647 defined threshold limit of $10^{-7}$ mm/cycle. Once threshold was established, the fatigue test was stopped and then restarted at an intermediate $\Delta K$ until fast fracture failure. For tests conducted without load shedding, the $\Delta K$ increased with crack growth. Thus, the second half of the fatigue crack growth tests can be defined as “Rising $\Delta K$” testing. This two test approach to fatigue crack growth testing is shown schematically in Figure 2. 6.

![Figure 2.4 Three point bending configuration was used for fatigue crack growth testing. Schematic shows location of DCPD leads.](image)
Figure 2. 5 DCPD connection setup. Leads were fixed by peening the leads into holes previously drilled.

Figure 2. 6 Schematic diagram of fatigue crack growth testing showing threshold test (red) followed by rising ΔK test (blue). Fatigue threshold (ΔK_{th}), Paris Law slope (m), and overload toughness (K_c) were obtained from this testing regime.
2.6.4 Fatigue Crack Growth Testing in Dry Environment

In order to investigate the environmental effects on the fatigue crack growth behavior of specimens, selective samples were prepared for testing in a Dry environment using Magnesium Perchlorate [45]. Mg (ClO₄)₂ can provide a locally dry environment as others have shown [45]. In these tests identical samples were fatigued in the presence of Mg (ClO₄)₂. This was done by placing adhesive tape around the sides of the sample in order to hold the Mg (ClO₄)₂ powders around the sample. After that, a similar fatigue crack growth test was conducted as described in section 2.6.3 that first started at intermediate ΔK and was conducted under rising ΔK conditions until fast fracture failure.

2.6.5 Fatigue Crack Growth Testing in Cold Environment

Fatigue crack growth testing also was conducted at low temperature in order to investigate the effects of a cold and dry environment on the fracture surface morphology of fatigued samples. In this case three different sensitized samples from different alloys were tested at -100 °C. An MTS model 409.80 controlled temperature cabinet resident on the 20 KIP MTS machine was utilized. Injection of liquid Nitrogen (LN₂) in to the chamber at regular intervals enabled temperature control to ± 1°C. In a typical test, the bend sample was placed into the 3PB fixture at room temperature under a slight compressive load in load control. Then liquid Nitrogen was injected in to the chamber until the chamber stabilized and reached -100 °C. After that, a similar fatigue crack growth test was conducted as described in section 2.6.3 that first started at intermediate ΔK until fast fracture failure under rising ΔK conditions.
2.6.6 Anticlastic Testing

Anticlastic testing is a severe fatigue testing technique designed to directly test the ST plate direction and to simulate the types of loading encountered by wing/fuselage structures at critical locations and pressure vessels for cryogenic containment [46]. Utilizing opposite bending moments, reversed normal stresses at the surface of the plate and reversed shear stresses at the midplane or neutral surface are produced. This results in maximum normal stresses at the plate surface and maximum shear stresses at the mid-plane as illustrated in Figure 2.7. This test has been utilized to give a qualitative determination of the inherent S-T weakness often exhibited by aluminum lithium alloys [46]. Sample dimensions were provided based on previous work [46]. All anticlastic testing was conducted in air at room temperature using an MTS 50 KIP servo-hydraulic testing machine. The anticlastic test apparatus was provided by Prof. Terry McNelley from the Naval Post Graduate School in Monterey, CA.

Specimens were mounted in the testing system as shown in the Figure 2.8. Testing was conducted under displacement control set to cycle under either ±750, ±1000 or ±1500 pound load at a frequency of 1 Hz until failure occurred.

The importance of conducting anticlastic fatigue tests relates to the difficulty of testing the S-T direction in thin plate using conventional test techniques. This technique directly probes the S-T direction.
2.7 Fracture Surface Analysis

2.7.1 Scanning Electron Microscopy (SEM)

In order to correlate microstructural observations to mechanical property changes, scanning electron microscopy (SEM) of specimen fracture surfaces was employed. Most SEM work
was conducted on a state-of-the-art Quanta 200i 3D system manufactured by FEI and located in the Swagelok Center for the Surface Analysis of Materials (SCSAM) on the campus of CWRU. This microscope is capable of Dual beam Focused-Ion Bean (FIB) operation and electron imaging is accomplished via tungsten filament emission. Limited high-resolution SEM imaging was completed on a Nova with a field emission tip and also located in SCSAM. In the vast majority of cases, an accelerating voltage of 15 keV and a working distance near 20 mm from the microscope aperture were used for both microscopes. In selected instances, the analysis of fracture surfaces also required elemental composition information. In these situations, an X-Ray Energy-Dispersive Spectrometry (XEDS) system manufactured by Noran was employed at high voltage of 15 KeV and dwell time of 30 ms.

2.7.2 Metallographic Cross-Sections

Selected samples were metallographically sectioned to examine the details of delamination/splitting on the fracture surfaces and subsequently imaged via scanning electron microscopy at different magnifications. Small regions of the fracture surface of the samples exhibiting such delamination/splitting were sectioned and mounted using copper-impregnated conductive powder in order to improve imaging near the specimen-mount interface. The mounted samples were polished to 0.03 μm finish using SiC paper, diamond suspension, and Al₂O₃ suspension polishing.
3 Experimental Results

3.1 Microstructure observation

The resulting optical microscope images of metallographic evaluation were stitched together to create a three dimensional representation of the as-received microstructure. These are provided as Figure 3.1 for 5059-H131 and Figure 3.2 for 5456-H31. In these figures the directions are denoted as RD for rolling direction, TD for transverse direction and ND for normal direction.

Figure 3.1: 5059-H131 as-received microstructure
3.2 Hardness Testing

Results from the Rockwell B hardness testing were averaged for each of the specimens and summarized in separate tables. The hardness values are also plotted as a function of thermal exposure time (in hours) on a logarithmic scale and separated by thermal exposure temperatures. Tabular results for 5083-H116 are provided in Table 3.1 and shown graphically in Figure 3.3. Results for 5456-H116 are provided in Table 3.2 and shown graphically in Figure 3.4. Results for 5059-H131 are provided in Table 3.3 and shown graphically in Figure 3.5. Finally results for 5456-H131 are provided in Table 3.4 and shown graphically in Figure 3.6.
Table 3.1: 5083-H116 Hardness Data

<table>
<thead>
<tr>
<th>Thermal Exposure Time (hrs)</th>
<th>60°C</th>
<th>70°C</th>
<th>80°C</th>
<th>100°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>52±1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2000</td>
<td>--</td>
<td>50±0.8</td>
<td>51.5±0.5</td>
<td>47±1</td>
</tr>
<tr>
<td>4000</td>
<td>43±2.1</td>
<td>41±2.5</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

Figure 3.3 Plot of hardness averages from 5083-H116 thermal exposures
Table 3. 2: 5456-H116 Hardness Data

<table>
<thead>
<tr>
<th>Thermal Exposure Time (hrs)</th>
<th>60°C</th>
<th>70°C</th>
<th>100°C</th>
<th>175°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td></td>
<td></td>
<td>54±0.9</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>48.3±1</td>
<td>47.3±1</td>
<td>--</td>
<td>54±0.9</td>
</tr>
<tr>
<td>1000</td>
<td>46.9±1</td>
<td>48.3±1</td>
<td>43.8±1</td>
<td>54±0.9</td>
</tr>
<tr>
<td>2000</td>
<td>46.9±1</td>
<td>48.3±1</td>
<td>43.8±1</td>
<td>54±0.9</td>
</tr>
<tr>
<td>4000</td>
<td>50.8±0.5</td>
<td>50.5±1</td>
<td>50.5±1</td>
<td>48.3±1</td>
</tr>
<tr>
<td>5000</td>
<td>51.3±0.6</td>
<td>49.5±0.6</td>
<td>49.5±0.6</td>
<td>48.3±1</td>
</tr>
</tbody>
</table>

Figure 3. 4 Plot of hardness averages from 5456-H116 thermal exposures
### Table 3.3: 5059-H131 Hardness Data

<table>
<thead>
<tr>
<th>Thermal Exposure Time (hrs)</th>
<th>Thermal Exposure Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>60°C</td>
</tr>
<tr>
<td>0</td>
<td>--</td>
</tr>
<tr>
<td>100</td>
<td>--</td>
</tr>
<tr>
<td>4000</td>
<td>67.7±0.6</td>
</tr>
</tbody>
</table>

**Figure 3.5** Plot of hardness averages from 5059-H131 thermal exposures
Table 3.4: 5456-H131 Hardness Data

<table>
<thead>
<tr>
<th>Thermal Exposure Time (hrs)</th>
<th>Thermal Exposure Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>60°C</td>
</tr>
<tr>
<td>0</td>
<td>60.8±0.5</td>
</tr>
<tr>
<td>100</td>
<td>--</td>
</tr>
<tr>
<td>4000</td>
<td>57.5±1.7</td>
</tr>
</tbody>
</table>

Figure 3.6 Plot of hardness averages from 5456-H131 thermal exposures

3.3 Tension Testing in Short Transverse Direction

Tension testing in short transverse direction was completed as described in section 2.6.2. Only a limited number of heat treatments of 5456-H131 thermal exposures were tested in tension as the focus of the present work was concentrated on the fatigue crack growth behavior of the alloys. All of the results are listed in Table 3.5. Testing was conducted
according to the procedure of section 2.6.2. Since the lengths of samples were too short to use an extensometer, Young’s Modulus was not calculated. Based upon the load and displacement measurements as recorded by the Test Works 4 Testing software, the stress and strain were calculated. From the individual stress-strain curves for each specimen, 0.2% Yield Strength, and Ultimate Tensile Stress were determined. This determination was done using Excel 2010. The 0.2% Offset Yield Strength was determined by manually identifying the intercept between the stress-strain curve for each tension test and the 0.2% offset fit line generated in the Excel. The Ultimate Tensile Strength (UTS) for each of the tension tests was determined using Excel by searching for the maximum load in each data file and then calculating the UTS from this load. The percent elongation and percent reduction in area were calculated based on the final diameter and length of each sample. In order to calculate the percent reduction in area at the neck, the minimum diameter at the neck must be determined. Knowing the final diameter, \(d_f\), and the initial diameter, \(d_0\), the percent reduction in area, \(\% RA\), can be calculated from the initial area, \(A_0\), and the final area, \(A_f\), as:

\[
\% RA = \frac{A_0 - A_f}{A_0} \times 100 = \frac{d_0^2 - d_f^2}{d_0^2} \quad (3.1)
\]

Final diameter measurements were taken using a caliper by finding the region of greatest necking in each tension specimen while viewing the broken tension specimen in a side view on binocular microscope at 10x magnification. Also for confirmation, several specimen diameters were measured via Scanning Electron Microscopy.
The results of all Yield Strength, UTS, and elongation along with percent in reduction area from each tension test are provided in Table 3.5 for all of the samples. The strain rates, immersion time in water (in hour) are also listed in the table. Immersion times in water ranged from 0 to 669 hours.

Table 3.5: Effect of thermal exposure on tensile testing results for 5456-H131

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Strain Rate</th>
<th>Time immersed in Water (Hrs)</th>
<th>0.2% Yield Stress (MPa)</th>
<th>UTS (MPa)</th>
<th>%Elongation</th>
<th>%RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456- as received</td>
<td>1x10^{-3} /sec</td>
<td>0</td>
<td>284</td>
<td>373</td>
<td>8.2</td>
<td>3.2</td>
</tr>
<tr>
<td>5456-as received</td>
<td>5x10^{-6} /sec</td>
<td>0</td>
<td>273</td>
<td>386</td>
<td>8.0</td>
<td>16.2</td>
</tr>
<tr>
<td>5456- as received</td>
<td>5x10^{-6} /sec</td>
<td>89</td>
<td>272</td>
<td>370</td>
<td>8.5</td>
<td>12.8</td>
</tr>
<tr>
<td>5456- as received</td>
<td>5x10^{-6} /sec</td>
<td>640</td>
<td>288</td>
<td>395</td>
<td>7.2</td>
<td>13.9</td>
</tr>
<tr>
<td>5456-AR-(80°C in Water)</td>
<td>5x10^{-6} /sec</td>
<td>111</td>
<td>263</td>
<td>369</td>
<td>8.8</td>
<td>23.2</td>
</tr>
<tr>
<td>5456-AR-(80°C in Water)</td>
<td>5x10^{-6} /sec</td>
<td>618</td>
<td>261</td>
<td>376</td>
<td>9.7</td>
<td>23.5</td>
</tr>
<tr>
<td>5456-AR-(80°C in Water)</td>
<td>5x10^{-6} /sec</td>
<td>191</td>
<td>262</td>
<td>378</td>
<td>10.9</td>
<td>15.3</td>
</tr>
<tr>
<td>5456-60°C-4000Hrs</td>
<td>5x10^{-6} /sec</td>
<td>143</td>
<td>274</td>
<td>394</td>
<td>9.2</td>
<td>9.4</td>
</tr>
<tr>
<td>5456-60°C-4000Hrs</td>
<td>5x10^{-6} /sec</td>
<td>687</td>
<td>270</td>
<td>387</td>
<td>7.5</td>
<td>27.9</td>
</tr>
<tr>
<td>5456-70°C-4000Hrs</td>
<td>1x10^{-3} /sec</td>
<td>0</td>
<td>257</td>
<td>362</td>
<td>10.3</td>
<td>11.1</td>
</tr>
<tr>
<td>5456-70°C-4000Hrs</td>
<td>5x10^{-6} /sec</td>
<td>0</td>
<td>260</td>
<td>378</td>
<td>10.6</td>
<td>11.1</td>
</tr>
<tr>
<td>5456-70°C-4000Hrs</td>
<td>5x10^{-6} /sec</td>
<td>135</td>
<td>260</td>
<td>379</td>
<td>9.2</td>
<td>11.7</td>
</tr>
<tr>
<td>5456-70°C-4000Hrs</td>
<td>5x10^{-6} /sec</td>
<td>669</td>
<td>267</td>
<td>384</td>
<td>9.6</td>
<td>18.9</td>
</tr>
<tr>
<td>5456-175°C-100Hrs</td>
<td>1x10^{-3} /sec</td>
<td>0</td>
<td>207</td>
<td>330</td>
<td>11.0</td>
<td>15.2</td>
</tr>
<tr>
<td>5456-175°C-100Hrs</td>
<td>5x10^{-6} /sec</td>
<td>0</td>
<td>216</td>
<td>354</td>
<td>13.6</td>
<td>15.9</td>
</tr>
<tr>
<td>5456-175°C-100Hrs</td>
<td>5x10^{-6} /sec</td>
<td>119</td>
<td>215</td>
<td>361</td>
<td>12.5</td>
<td>13.6</td>
</tr>
<tr>
<td>5456-175°C-100Hrs</td>
<td>5x10^{-6} /sec</td>
<td>662</td>
<td>213</td>
<td>351</td>
<td>8.9</td>
<td>20.6</td>
</tr>
</tbody>
</table>

Stress strain curves were calculated for each condition and are provided in Figures 3.7 to Figure 3.17. The effects of water immersion are shown by comparison of immersion times of 0, 120 and 650 hours. For the low and high strain rate tests, individual stress strain plots
are provided. Furthermore, Yield strength, UTS, elongation and percent reduction in area data are plotted as a function of thermal exposure time and temperature on a logarithmic scale in Figures 3.18 to Figure 3.21 respectively. Serrated flow is exhibited in many of the stress strain curves.

Figure 3. 7: Representative engineering stress-strain curves on 5456-H131 after different thermal exposures. Tension tests conducted at room temperature at $5 \times 10^{-6}$/sec
Figure 3.8: Representative engineering stress-strain curves on 5456-H131 after different thermal exposures. Prior to testing in air at $5 \times 10^{-6}$ /sec at RT, samples were exposed to water at the temperatures and times shown.

Figure 3.9: Representative engineering stress-strain curves on 5456-H131 after different thermal exposures. Prior to testing in air at $5 \times 10^{-6}$ /sec at RT, samples were exposed to water at the temperatures and times shown.
Figure 3.10: Representative engineering stress-strain curves on 5456-H131 after different thermal exposures. Tension tests conducted at room temperature at $1 \times 10^{-3}$ /sec.

Figure 3.11: Representative engineering stress-strain curves comparing 5456-H131 As-Received samples tested at room temperature at different strain rates.
Figure 3. 12: Representative engineering stress-strain curves comparing 5456-H131 given thermal exposure of 70°C /4000hrs prior to testing at room temperature at different strain rates.

Figure 3. 13: Representative engineering stress-strain curves comparing 5456-H131 As-Received samples exposed to water for 89 hours and 640 hours, then tested at room temperature in air at 5x10⁻⁶ /sec.
Figure 3.14: Representative engineering stress-strain curves comparing 5456-H131 As-Received samples exposed to water at 80°C for 111 hours and 618 hours, then tested in room temperature air at 5x10^-6 /sec.

Figure 3.15: Representative engineering stress-strain curves comparing 5456-H131 given a thermal exposure of 60°C /4000hrs prior to exposure to water for 143 hours and 687 hours, then testing in air at room temperature at 5x10^-6 /sec.
Figure 3. 16: Representative engineering stress-strain curve comparing 5456-H131 given a thermal exposure of 70°C /4000hrs prior to exposure to water for 135 hours and 669 hours. Tension testing conducted at room temperature in air at 5x10^-6 /sec.

Figure 3. 17: Representative engineering stress-strain curve comparing 5456-H131 given a thermal exposure of 175°C /100hrs prior to exposure to water for 119 hours and 662 hours. Tension testing conducted at room in air temperature at 5x10^-6 /sec.
Figure 3.18: Effect of thermal exposure and exposure to water at RT on 0.2% Offset Yield Strength at 5x10^-6 /sec.

Figure 3.19: Effect of thermal exposure and exposure to water at RT on Ultimate Tensile Strength.
Figure 3.20: Effect of thermal exposure and exposure to water at RT on Percent Elongation for 5456-H131 tension tests.

Figure 3.21: Effect of thermal exposure and exposure to water at RT on Percent Reduction in Area for 5456-H131 tension tests.
3.4 Fatigue Crack Growth

3.4.1 Fatigue Crack Growth Curves

Fatigue crack growth data are plotted in terms of \( \frac{da}{dN} \) versus \( \Delta K \) in accordance with the ASTM E647 standard [44]. Results for 5456-H116 are provided in Figure 3.23; 5083-H116 are provided in Figure 3.23; 5059-H131 are provided in Figure 3.25; 5456-H131 are provided in Figure 3.26. The effects of water exposure for various times on the fatigue crack growth behavior of all 5456-H116 and 5083-H116 are provided in Figure 3.26. The effects of various remediation treatments on 5456-H116 are provided in Figure 3.27; results for Remediated 5083-H116, H131 are provided in Figure 3.28; results for Remediated 5059-H131, 5456-H131 are provided in Figure 3.29. The effects of pre-strain on sensitized 5456-H116 are provided in Figure 3.30.

In some cases, fatigue curves from different tests were combined to enable direct comparison and discussion later in the discussion section.
Figure 3.22: Effects of thermal exposure on Fatigue Crack Growth curves for 5456-H116
Figure 3.23: Effects of thermal exposure on Fatigue Crack Growth curves for 5083-H116
Figure 3.24: Effects of thermal exposure on Fatigue Crack Growth curves for 5059-H131
Figure 3.25: Effects of thermal exposure on fatigue crack growth curves for 5456-H131
Figure 3.26: Effects of thermal exposure on Fatigue Crack Growth curves for 5456-H116, 5083-H116 immersed in water and then tested in air
Figure 3.27: Effects of various remediation conditions on Fatigue Crack Growth curves for 5456-H116
Figure 3. 28: Effects of various remediation conditions on Fatigue Crack Growth curves for 5083-H116, H131
Figure 3.9: Effects of various remediation conditions on Fatigue Crack Growth curves for 5059-H131, 5456-H131.
Figure 3.30: Effects of pre-strain at levels shown on Fatigue Crack Growth curves for sensitized 5456-H116
3.4.2 Fatigue Threshold

For a limited number of samples, the fatigue threshold, $\Delta K_{th}$ was measured as described in section 2.6.3. These were combined with previous measurements of fatigue threshold from previous work [33] and are provided in Tables 3.6 to 3.15.

3.4.3 Paris Law Slopes

The Paris Law slope can be determined from the crack growth rate, $\left( \frac{da}{dN} \right)$, at intermediate $\Delta K$ values, where the crack growth behavior is approximately linear on a log-log sale. In this regime, the crack growth can be related to the stress intensity range, $\Delta K$, via the Paris Law equation [44]:

\[
\frac{da}{dN} = C \left( \Delta K \right)^m
\]

where $C$ and $m$ are the fatigue crack growth constants. Linear regression was used to fit fatigue crack growth data and $m$, the Paris slope, was determined using slope of curve. Log$_{10}(C)$ is given as y-intercept of the fit line. For all of the $\left( \frac{da}{dN} \right)$ versus $\Delta K$ plots, the linear regression was performed with Origin software. Results of the linear regression of equation 3.2, reported as $m$ are presented in Tables 3.6 to 3.15 for all the different specimens and thermal exposure conditions.
3.4.4 Critical K at Overload (K_c)

In tests conducted under rising ∆K conditions, the specimen catastrophically failed as shown in Figure 2.6. Thus, each specimen exhibited a critical ∆K at which overload occurred, termed ∆K_c. Although not identical to the plane strain fracture toughness, K_{IC}, determined from a fracture toughness test, ∆K_c can be converted to a maximum K_c at failure when the R-ratio is known. Therefore, considering K_{max} at overload in the rising ∆K portion of the (\frac{da}{dN}) and based on Equation 2.1, K_{max} at overload is given as:

\[
K_{max} = \frac{\Delta K_c}{1 - R}
\]  

(3.3)

In all Tables, K_{max} at overload is reported as K_c. Tables 3.6 to 3.15 summarize ∆K_{th}, C and K_c. Fractographic observations summarized shortly, revealed the presence of splitting and delamination in the ST direction. The tables provide the range of K_{max} values where splitting is observed.
<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>$\Delta K_{th}$ (MPa√m)</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-F60T4000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>2.9</td>
<td>N/A</td>
<td>19.5</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F60T4000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>2.6</td>
<td>24.8</td>
<td>Yes</td>
<td>10-14</td>
</tr>
<tr>
<td>5456-F60T5000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>2.8</td>
<td>N/A</td>
<td>21.8</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F60T5000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>3</td>
<td>26</td>
<td>Yes</td>
<td>9-13.5</td>
</tr>
<tr>
<td>5456-F70T2000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>3.5</td>
<td>23</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F70T4000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>3.9</td>
<td>3.3</td>
<td>38</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F70T4000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>2.5</td>
<td>23.6</td>
<td>Yes</td>
<td>11.5-13.5</td>
</tr>
<tr>
<td>5456-F100T1000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>4.5</td>
<td>28</td>
<td>Yes</td>
<td>9-19</td>
</tr>
<tr>
<td>5456-F100T1000</td>
<td>Rising K (Dry)</td>
<td>Not measured</td>
<td>2.9</td>
<td>25</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F100T5000</td>
<td>Constant $\Delta K$</td>
<td>Not measured</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>13</td>
</tr>
<tr>
<td>5456-F100T5000</td>
<td>Constant $\Delta K$ (1Hz)</td>
<td>Not measured</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>13</td>
</tr>
<tr>
<td>5456-F100T5000</td>
<td>Constant $\Delta K$ (Dry)</td>
<td>Not measured</td>
<td>N/A</td>
<td>N/A</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F175T100</td>
<td>Rising K (1Hz)</td>
<td>Not measured</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>8-25</td>
</tr>
</tbody>
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Table 3.7: Effects of thermal exposures on Fatigue Crack Growth results for 5083-H116

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>∆Kth (MPa√m)</th>
<th>Paris Slope, m</th>
<th>Overload Kc (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of Kinf for Delamination (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083-F60T4000</td>
<td>Threshold, Rising ∆K</td>
<td>2.9</td>
<td>N/A</td>
<td>17.6</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5083-F60T4000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>4.5</td>
<td>39.2</td>
<td>Yes</td>
<td>11-21</td>
</tr>
<tr>
<td>5083-F70T2000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>3.5</td>
<td>23</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5083-F70T4000</td>
<td>Threshold, Rising ΔK</td>
<td>2.9</td>
<td>N/A</td>
<td>22</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5083-F70T4000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>2.1</td>
<td>33.8</td>
<td>Yes</td>
<td>11-13</td>
</tr>
<tr>
<td>5083-F80T2000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>4</td>
<td>27</td>
<td>Yes</td>
<td>8-10</td>
</tr>
<tr>
<td>5083-F100T2000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>5</td>
<td>28</td>
<td>Yes</td>
<td>8-11</td>
</tr>
<tr>
<td>5083-F100T2000</td>
<td>Rising ΔK</td>
<td>Not measured</td>
<td>N/A</td>
<td>Not measured</td>
<td>Yes</td>
<td>Not measured</td>
</tr>
<tr>
<td>5083-F100T2000</td>
<td>Rising ΔK (1Hz)</td>
<td>Not measured</td>
<td>N/A</td>
<td>Not measured</td>
<td>Yes</td>
<td>Not measured</td>
</tr>
<tr>
<td>5083-F100T2000</td>
<td>Rising ΔK (Dry)</td>
<td>Not measured</td>
<td>2</td>
<td>34</td>
<td>No</td>
<td>None</td>
</tr>
</tbody>
</table>
Table 3. 8: Effects of thermal exposures on Fatigue Crack Growth results for 5456-H116, 5083-H116 immersed in water and tested in water

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>∆K∞ (MPa/√m)</th>
<th>Paris Slope, m</th>
<th>Overload Kc (MPa/√m)</th>
<th>Delamination in S-T</th>
<th>Range of Kmax for Delamination (MPa/√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-AR (Immersed in water for 3 days at RT)</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>5.3</td>
<td>24</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F70T2000 (Immersed in water for 3 days at RT)</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>5.5</td>
<td>29</td>
<td>Yes</td>
<td>10</td>
</tr>
<tr>
<td>5456-F70T2000 (Immersed in water for 3 days at RT)-Aged 6 month at RT</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>2.7</td>
<td>33</td>
<td>Yes</td>
<td>9-26</td>
</tr>
<tr>
<td>5456- (Immersed in Water at 80°C for 500 Hrs)</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>2.9</td>
<td>36</td>
<td>Yes</td>
<td>8-16</td>
</tr>
<tr>
<td>5456- (Immersed in Water at 80°C for 500 Hrs) (Dry)</td>
<td>Rising ∆K (Dry)</td>
<td>Not measured</td>
<td>3</td>
<td>33.3</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456- (Immersed in Water at 80°C for 500 hrs) + (Baked 65 Hrs at 80°C)</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>2.9</td>
<td>33.9</td>
<td>Yes</td>
<td>10-22</td>
</tr>
<tr>
<td>5083-F70T2000 (Immersed in water for 3 days at RT)</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>2.5</td>
<td>32</td>
<td>Yes</td>
<td>10.5</td>
</tr>
<tr>
<td>5083-F70T2000 (Immersed in water for 3 days at RT)-Aged 6 month at RT</td>
<td>Rising ∆K</td>
<td>Not measured</td>
<td>2.9</td>
<td>33.6</td>
<td>Yes</td>
<td>9.5-24</td>
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</table>
Table 3. 9: Effects of thermal exposures on Fatigue Crack Growth results for 5059-H131

<table>
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<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>$\Delta K_{th}$ (MPa$\sqrt{m}$)</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa$\sqrt{m}$)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa$\sqrt{m}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5059-AR</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>2.7</td>
<td>26</td>
<td>Yes</td>
<td>9-15</td>
</tr>
<tr>
<td>5059-AR</td>
<td>Rising $\Delta K$</td>
<td>3.3</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>5059-F60T4000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>2.6</td>
<td>37</td>
<td>Yes</td>
<td>10-12</td>
</tr>
<tr>
<td>5059-F70T4000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>3.4</td>
<td>2.9</td>
<td>24</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5059-F70T4000</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>2.3</td>
<td>35</td>
<td>Yes</td>
<td>10-13</td>
</tr>
<tr>
<td>5059-F175T100</td>
<td>Rising $\Delta K$</td>
<td>Not measured</td>
<td>4.9</td>
<td>23</td>
<td>Yes</td>
<td>9-20</td>
</tr>
<tr>
<td>5059-F175T100</td>
<td>Rising $\Delta K$ (Dry)</td>
<td>Not measured</td>
<td>N/A</td>
<td>N/A</td>
<td>No</td>
<td>None</td>
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</table>
Table 3.10: Effects of thermal exposures on Fatigue Crack Growth results for 5456-H131

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>$\Delta K_{th}$ (MPa$\sqrt{m}$)</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa$\sqrt{m}$)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa$\sqrt{m}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-AR</td>
<td>Rising $\Delta K$</td>
<td>2.99</td>
<td>3</td>
<td>26.5</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F60T4000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>3.3</td>
<td>2.9</td>
<td>30.4</td>
<td>Yes</td>
<td>9.5-15</td>
</tr>
<tr>
<td>5456-F70T4000</td>
<td>Threshold, Rising $\Delta K$</td>
<td>3.4</td>
<td>2.3</td>
<td>32.3</td>
<td>Yes</td>
<td>10-16</td>
</tr>
<tr>
<td>5456-F175T100</td>
<td>Threshold, Rising $\Delta K$</td>
<td>3.5</td>
<td>2.9</td>
<td>27.2</td>
<td>Yes</td>
<td>11-23</td>
</tr>
<tr>
<td>5456-F175T100</td>
<td>Rising $\Delta K$ (Dry)</td>
<td>Not measured</td>
<td>2.6</td>
<td>29</td>
<td>No</td>
<td>None</td>
</tr>
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</table>
### Table 3.11: Effects of thermal remediation treatments on Fatigue Crack Growth of 5456-H116

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-F175T100+240°C /15min</td>
<td>Rising $\Delta K$</td>
<td>2.1</td>
<td>39</td>
<td>Yes</td>
<td>12-30</td>
</tr>
<tr>
<td>5456-F175T100+260°C /15min</td>
<td>Rising $\Delta K$</td>
<td>1.8</td>
<td>41</td>
<td>Yes</td>
<td>12-15</td>
</tr>
<tr>
<td>5456-F175T100+260°C /30min</td>
<td>Rising $\Delta K$</td>
<td>2.5</td>
<td>33</td>
<td>Yes</td>
<td>10-30</td>
</tr>
<tr>
<td>5456-F175T100+260°C /60min</td>
<td>Rising $\Delta K$</td>
<td>4.5</td>
<td>26</td>
<td>Yes</td>
<td>12</td>
</tr>
<tr>
<td>5456-F175T100+260°C /300min</td>
<td>Rising $\Delta K$</td>
<td>2.8</td>
<td>34</td>
<td>Yes</td>
<td>11-15</td>
</tr>
<tr>
<td>5456-F175T100+280°C /10min</td>
<td>Rising $\Delta K$</td>
<td>2.8</td>
<td>24</td>
<td>Yes</td>
<td>8-24</td>
</tr>
<tr>
<td>5456-F175T100+280°C /15min +175°C /100hrs</td>
<td>Rising $\Delta K$</td>
<td>1.5</td>
<td>39</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-F175T100+280°C /15min</td>
<td>Rising $\Delta K$</td>
<td>2.2</td>
<td>34</td>
<td>Yes</td>
<td>9-31</td>
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Table 3.12: Effects of thermal remediation treatments on Fatigue Crack Growth of 5083-H116, 5083-H131

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>Paris Slope, m</th>
<th>Overload Kc (MPa*m)</th>
<th>Delamination in S-T</th>
<th>Range of Kmax for Delamination (MPa*m)</th>
</tr>
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<tbody>
<tr>
<td>5083-F175T100+240°C /15min</td>
<td>Rising ΔK</td>
<td>3.1</td>
<td>25</td>
<td>Yes</td>
<td>9-20</td>
</tr>
<tr>
<td>5083-F175T100+260°C /15min</td>
<td>Rising ΔK</td>
<td>2.9</td>
<td>17</td>
<td>Yes</td>
<td>12-15</td>
</tr>
<tr>
<td>5083-F175T100+280°C /15min</td>
<td>Rising ΔK</td>
<td>2.7</td>
<td>32</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5083-F175T100+280°C /15min +175°C /100hrs</td>
<td>Rising ΔK</td>
<td>3.0</td>
<td>35</td>
<td>Yes</td>
<td>10-23</td>
</tr>
<tr>
<td>5083-H131-F175T100+240°C /60min</td>
<td>Rising ΔK</td>
<td>3.6</td>
<td>27</td>
<td>Yes</td>
<td>11-18</td>
</tr>
<tr>
<td>5083-H131-F175T100+ 240°C /300min</td>
<td>Rising ΔK</td>
<td>2.4</td>
<td>39</td>
<td>Yes</td>
<td>12-24</td>
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Table 3. 13: Effects of thermal remediation treatments on Fatigue Crack Growth of 5059, 5456-H131

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa√m)</th>
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<tbody>
<tr>
<td>5059-F175T100+280°C/15min</td>
<td>Rising ΔK</td>
<td>2.9</td>
<td>28</td>
<td>Yes</td>
<td>8-24</td>
</tr>
<tr>
<td>5059-F175T100+280°C/15min +175°C/100hrs</td>
<td>Rising ΔK</td>
<td>2.3</td>
<td>31.5</td>
<td>Yes</td>
<td>8-24</td>
</tr>
<tr>
<td>5059-F175T100+280°C/30min</td>
<td>Rising ΔK</td>
<td>2.3</td>
<td>28</td>
<td>Yes</td>
<td>8-24</td>
</tr>
<tr>
<td>5456-F175T100+280°C/15min</td>
<td>Rising ΔK</td>
<td>4.6</td>
<td>28.6</td>
<td>Yes</td>
<td>10-25</td>
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Table 3. 14: Effects of pre-strain on Fatigue Crack Growth of sensitized 5456-H116

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>Paris Slope, m</th>
<th>Overload $K_c$ (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of $K_{max}$ for Delamination (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-F175T100- (1.7% Strain)</td>
<td>Rising ΔK</td>
<td>1.5</td>
<td>39</td>
<td>Yes</td>
<td>9-28</td>
</tr>
<tr>
<td>5456-F175T100- (3.7% Strain)</td>
<td>Rising ΔK</td>
<td>2.3</td>
<td>36</td>
<td>Yes</td>
<td>10-20</td>
</tr>
</tbody>
</table>
Table 3.15: Effects of testing at -100°C on Fatigue Crack Growth of sensitized 5456-H116

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Test Type</th>
<th>Paris Slope, m</th>
<th>Overload K_c (MPa√m)</th>
<th>Delamination in S-T</th>
<th>Range of K_max for Delamination (MPa√m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5456-H116-F175T100</td>
<td>Rising ΔK</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>None*</td>
</tr>
<tr>
<td>5083-H131-F175T100</td>
<td>Rising ΔK</td>
<td>N/A</td>
<td>N/A</td>
<td>No</td>
<td>None</td>
</tr>
<tr>
<td>5456-H131-F175T100</td>
<td>Rising ΔK</td>
<td>N/A</td>
<td>N/A</td>
<td>Yes</td>
<td>None*</td>
</tr>
</tbody>
</table>

*: Due to Liquid Nitrogen ran out, it was not possible to determine when LN2 was in chamber.

3.5 Anticlastic Testing

Trial samples of 6061-T6 Aluminum alloys were first tested to failure at various loads including ±750, ±1000 or ±1500 lbs at a frequency of 1 Hz. Then, specimens of 5456-H116 given exposure to 175°C/100hrs were tested at either ±750 or ±1000 lbs to failure. In all cases fracture occurred in the plane normal to the plate surface in the short transverse direction. Anticlastic bend testing results are listed in Table 3.16 for both 6061-T6 and 5456-H116.
Table 3. 16: Anticlastic fatigue testing results for 6061-T6 and 5456-H116

<table>
<thead>
<tr>
<th>Specimen/Exposure</th>
<th>Cyclic load</th>
<th>Nf cycles to failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061- as-received</td>
<td>750</td>
<td>692,530</td>
</tr>
<tr>
<td>6061-F175T100</td>
<td>1000</td>
<td>1,777,782</td>
</tr>
<tr>
<td>6061-F175T100</td>
<td>1500</td>
<td>1,368</td>
</tr>
<tr>
<td>5456- as-received</td>
<td>1000</td>
<td>13,880</td>
</tr>
<tr>
<td>5456-F175T100</td>
<td>1000</td>
<td>12,560</td>
</tr>
<tr>
<td>5456-F175100</td>
<td>750</td>
<td>104,765</td>
</tr>
</tbody>
</table>

3.6 Fracture Surface Analyses

After completion of the tension tests, fatigue crack growth tests and anticlastic tests, selected specimen fracture surfaces were analyzed under Scanning Electron Microscopy (SEM). In most cases, the orientation of the sample while imaging was maintained, so as to provide a one to one comparison between fracture surfaces from different thermal exposures and testing conditions.

3.6.1 Tensile Specimen Fracture Surface Features

Different specimens of 5456-H131 tension tests were selected in order to provide a representative sample of fracture surface features. In order to provide a representative sample of fracture surface features, SEM images from the as-received condition were compared to several different thermal exposure that are provided in Figure 3.31 and 3.32 for 5456-H131 materials. Specifically, the as-received samples were compared to samples
given thermal exposures of 60°C, 70°C for 4000 hours that were tested at slow strain rate (i.e. 5x10^{-6} /sec) in air. Also an as-received 5456-H131 sample immersed in water at 80°C for 500 hours and tested in air at 5x10^{-6} /sec is included to enable comparison to other sensitized samples in Figure 3.31 and 3.32.

Moreover, SEM images of the specimen 5456-H131 given thermal exposure to 175°C for 100 hours and tested at slow strain rate (i.e. 5x10^{-6}/sec) were compared to high strain rate (i.e. 1x10^{-3}/sec) in Figure 3.33.

All of the testing conditions are summarized in Table 3.5. A complete list of SEM images along with all of the pictures is included in the Appendix B on a Compact Disk (CD) for future reference.
Figure 3. 31: Low magnification SEM images of fracture surfaces of 5456-H131 specimens thermally exposed for time/temperature combinations shown and tensile tested in air at $5 \times 10^{-6}$/sec.
Figure 3.32: Higher magnification SEM images of fracture surfaces of 5456-H131 specimens thermally exposed for time/temperature combinations shown and tensile tested in air at 5x10^-6/sec.
Figure 3.33: Low and high magnification SEM images of fracture surfaces of 5456-H131 175°C 100 hrs specimens tensile tested at either $5 \times 10^{-6}$ /sec or $1 \times 10^{-3}$ /sec rate after thermal exposures indicated.

In order to show the mode of fracture (i.e. Flat vs. Slant), side view SEM images were taken and are shown in Figure 3.34. Some of the samples exhibited sub-critical cracking in the short transverse direction. The extent of these cracks increased with increasing thermal exposure, exposure to water, or decrease in strain rate.
Figure 3.34: Low magnification SEM images of side view of all samples tested in air at $5 \times 10^{-6}$/sec
Previous results [33] obtained in the L-T direction showed short transverse splitting exhibited on the fracture surfaces of fatigue crack growth tests of 5456-H131 samples exposed to 175°C /100 hrs. The present tension specimens were machined from the S-T direction. As such, splitting is expected on the surface of sensitized tension samples, as shown in Figure 3.35. Lateral splitting was also observed.

Figure 3.35: SEM images of side view and fracture surface of 5456-H131 175°C 100 hr specimens tested at 1x10⁻³ /sec. Arrows show the opening of splits in the S-T direction.
Figure 3.36 illustrates a ductile fracture mode in the short transverse splits.

Figure 3.36: SEM images of fracture surface of 5456-H131 175°C 100 hr specimens tested at 1x10⁻³ sec showing ductile features.
3.6.2 Fracture Surface Features of Anticlastic Samples

A low magnification optical view of a fractured anticlastic sample is provided in Figure 3.37. In addition to fracture evident perpendicular to the plane of the plate, more significant fracture occurs in the short transverse direction.

Figure 3.37: Side and top view of optical macrograph of failed Anticlastic sample of 5456-H116 175°C 100 hrs tested in air at ± 1000 lbs. at 1Hz

Figure 3.38 and 3.39 show SEM top views of the plate surface in the 5456-H116 and 6061-T6 samples, respectively.
Figure 3.38: Representative SEM images of top surfaces of 5456-H116 vs. 5456-H116 175°C 100 hr samples tested in Anticlastic Testing.
Figure 3. 39: Representative SEM images of top views of 6061 sample tested via Anticlastic Testing that failed after 1368 cycles when tested at ±1500 lbs.

Figure 3. 40 provides low and high magnification SEM images of the fractured 5456-H116 and reveals fracture in the short transverse direction parallel to the top surface of the plate.
Figure 3. 40: SEM image of 5456-H116 175°C 100 hr Anticlastic test showing intergranular fracture in the short transverse direction, arrowed.

3.6.3 Fracture Surface observation of Fatigue Crack Growth Samples

Macroscopically, the appearance of the fracture surfaces of the bend bars tested for the fatigue crack growth were similar regardless of the thermal exposures. Specifically, three distinct regions of fracture surface features can be identified: region of fatigue crack growth, fatigue threshold, and a region of overload fast fracture, Figure 3.41. A complete list of SEM images along with all of the pictures are included in the Appendix C as a Compact Disk (CD) for future references.
In order to systematically study the fractographic observations, specific regions of the fracture surface were examined, as shown in Figure 3.42. High magnification imaging was used at different regions of the fracture surface and different images were recorded. Then, using the DCPD fatigue data, each images was assigned to a specific $K_{\text{max}}$ value in order to enable comparison at equivalent stress in intensity levels. Fracture surface images were categorized into three different regions of the fracture surface: Low $\Delta K$, Medium $\Delta K$ and High $\Delta K$/overload.
3.6.3.1 SEM observations for 5456-H116 alloy

As described in section 2.6, fatigue crack growth testing was conducted in different environments. In most cases testing was done in laboratory air. Additional tests were conducted in a Dry environment as described earlier. SEM images of 5456-H116 100°C/5000 hour specimens tested in fatigue at constant $\Delta K$ of 13 MPa$\sqrt{\text{m}}$ in Air are compared with Dry environment in Figure 3.43. Splits in the S-T direction are clearly evident in the air test, but are not present in the identical test conducted Dry. Similar behavior was observed in 5456-H116 100°C/1000 hour sample as shown in Figure 3.44. SEM images of 5456-H116 exposed to either 60°C, 70°C for 4000 hours and tested in Air...
are shown in Figure 3.45. Splitting in these two cases observed at low ΔK regime. In all SEM images, arrows indicate splits.

Figure 3.43: SEM images of 5456-H116 100°C 5000 hr specimen tested at constant ΔK of 13 MPa√m in Air vs. Dry environment. Splits (arrowed) present in Air test, absent in Dry test.
Figure 3. 44: SEM images of 5456-H116 100°C 1000 hr specimen tested in Rising $\Delta K$ in Air vs. Dry environment. Splits present in Air test (arrowed) are absent in Dry test. (SEM images were taken at $\Delta K = 15$ MPa$\sqrt{m}$)

Figure 3. 45: SEM images of 5456-H116 60°C, 70°C 4000 hr specimen tested in Rising $\Delta K$ in Air environment. Splits present in Air test arrowed (SEM images were taken at $\Delta K = 12$ MPa$\sqrt{m}$)
In order to determine the combined effects of environment and thermal exposure, one 5456-H116 sample was immersed in distilled water held at 80°C for 500 hours. This sample was also tested in fatigue crack growth at rising $\Delta K$ in Air and Dry conditions. Splits present in the air test were not present in the Dry test. This is shown in Figure 3.46. In addition, one of the two halves of this sample was baked 65 hours at 80°C and again tested at same testing condition in Air to evaluate the effects of such bake out on the splitting behavior during fatigue crack growth. Splitting still occurs as the SEM image of this sample shows in Figure 3.47.

Figure 3. 46: SEM images of 5456-H116 immersed in water at 80°C for 500 hr, and tested at Rising $\Delta K$ in Air vs. Dry environment. Splits evident in Air test (arrowed) are not present in Dry test. (SEM images were taken at $\Delta K = 12$ MPa√m)
Figure 3. 47: SEM image of 5456-H116 sample immersed in water at 80°C for 500 hr followed by baking at 80°C for 65 hr and fatigue testing at Rising $\Delta K$ in Air. SEM image shows splitting (arrowed). (SEM image was taken at $\Delta K = 13.3$ MPa$\sqrt{m}$)

3.6.3.2 SEM observations for 5083-H116 alloy

Representative SEM images of the fracture surfaces for the 5083-H116 alloy tested in Air vs. Dry are shown in Figure 3.48. 5083-H116 exposed to 100°C/2000 hrs was tested in fatigue crack growth in Air and Dry environments. Similar to the 5456 results, splitting observed in Air tests was not exhibited in the dry tests, Figure 3.48. The effects of RT water immersion for short time on the fatigue crack growth behavior of this alloy is shown by the SEM images of 5083-H116 70°C sample exposed to 2000 hours and a 5083-H116 sample exposed to 70°C for 2000 hours tested after three days exposure to distilled water prior to fatigue testing under Rising $\Delta K$ in Air, Figure 3.49. S-T Splitting in fatigue was exhibited after three days exposure to distilled water, in addition to samples held at RT for 6 months, Figure 3.49 and 3.50.
Figure 3. 48: SEM images of 5083-H116 100°C 2000 hr specimen tested in Rising $\Delta K$ in Air vs. Dry environment. Splits present in the Air test (arrowed) are not exhibited in the Dry test. (SEM images were taken at $\Delta K = 10.6$ MPa$\sqrt{m}$)

Figure 3. 49: SEM images of 5083-H116 sample exposed to 70°C for 2000 hr and 5083-H116 70°C 2000 hr with three days immersion in water tested at Rising $\Delta K$ in Air environment. (SEM images were taken at $\Delta K = 16$ MPa$\sqrt{m}$). Splits (arrowed) are evident in the sample tested after water exposure.
Figure 3. 50: SEM images of 5083-H116 sample exposed to 70°C 2000 hr and given three days exposure to water followed by 6 month aging at room temperature in Air. Sample was tested in Rising $\Delta K$ in Air environment and revealed splits (arrowed). (SEM images were taken at $\Delta K = 15$ MPa$\sqrt{m}$)

\[ \text{SEM observations for 5059-H131 alloy} \]

SEM images for 5059-H131 alloy tested in fatigue in Air shows splitting even in the as-received condition, Figure 3.51, although these were not always observed in Dry tests. 5059-H131 sample exposed to 175°C for 100 hours tested at Rising $\Delta K$ in Air vs. Dry environments. SEM images of both Air and Dry tests shown in Figure 3.52 reveal that the number and lateral opening of S-T splits is higher in comparison to other alloys. Such splits also are evident in overload, Figure 3.53, often visible by eye. Significantly, Dry testing does not reduce the extent of splits in overload in this alloy condition.
Figure 3. 51: SEM image of 5059-H131 as received specimen tested at Rising $\Delta K$ in Air. (Splits (arrowed) are obvious at low $\Delta K$ regime) $\Delta K = 10 \text{ MPa} / \text{m}$
Figure 3. 52: SEM images of 5059-H131 sample exposed to 175°C for 100 hr and tested at Rising \( \Delta K \) in Air vs. Dry environment. Splits (arrowed) present in the Air test are not evident in Dry test. (SEM images were taken at \( \Delta K = 10 \text{ MPa}\sqrt{m} \))

Figure 3. 53: SEM images of 5059-H131 sample exposed to 175°C for 100 hr and tested in Rising \( \Delta K \) in Air vs. Dry environment splits (arrowed) are evident in overload region in all cases.
3.6.3.4 SEM observations for 5456-H131 alloy

5456-H131 alloy does not exhibit splits in the as received condition, Figure 3. 54. Splitting present in the 5456-H131 sample exposed to 175°C for 100 hours and tested in air was not evident in a Dry test, Figure 3. 55. The major difference in this case relates to the orientation of splitting, which is rotated by 90 degrees with respect to the all previous alloys. Also, splits were observed in overload similar to 5059-H131, shown in Figure 3.56. The similarity between the size of splits in 5059-H131 and 5456-H131 is also evident.

Figure 3. 54: SEM image of 5456-H131 as received specimen tested at Rising ΔK in Air. No Splits evident. (SEM image was taken at ΔK= 8.1 MPa√m)
Figure 3. 55: SEM image of 5456-H131 sample exposed to 175°C for 100 hrs and tested at Rising $\Delta K$ in Air vs. Dry environment. Splits (arrowed) present in the Air test are not evident in Dry test. (SEM images were taken at $\Delta K = 9$ MPa$\sqrt{m}$)

Figure 3. 56: SEM images of 5456-H131 sample exposed to 175°C for 100 hrs and tested at Rising $\Delta K$ in Air vs. Dry environment. Splits (arrowed) are evident in overload region in both cases.
3.6.4 Effectiveness of Remediation Strategies after Thermal Exposure/Aging

As described in section 2.5, thermal stabilization treatments were conducted in order to eliminate splitting. Thermal remediation treatments of 240°C and 260°C for 5456-H116, and 5083-H116 previously sensitized at 175°C/100 hours were not successful in eliminating splitting during fatigue in Air. However, Figures 3.57- 3.59, show that the number of splits decreased by increasing the remediation temperature and the splits are not evident after remediation at 280°C, shown in Figure 3.58 and Figure 3.60. Rockwell B hardness also decreased after this thermal treatment. In order to evaluate the effectiveness of this thermal treatment, successfully remediated samples of 5456-H116, 5083-H116 at 280°C/15 minutes were thermally exposed at 175°C for 100 hours. Unfortunately, the splits re-appeared during fatigue as shown in Figure 3.58 and Figure 3.60.
Figure 3. 57: SEM images of 5456-H116 sample exposed to 175°C for 100 hrs then remediated at either 240°C or 260°C /15min and tested in fatigue in Air. Splits (arrowed) are still exhibited but the amount decreased compared to 5456-H116 175°C for 100 hrs sample.
Figure 3. 58: SEM images of remediated sample of 5456-H116 exposed to 175°C for 100 hrs then remediated at 280°C /15Min followed by re-exposure to 175°C/100hrs. Splits are not evident in remediated sample but then re-appear (arrowed) in thermally exposed sample.
Figure 3. 59: SEM images of 5083-H116 sample exposed to 175°C for 100 hrs remediated at either 240°C or 260°C /15min. Splits (arrowed) still evident although the extent of splits decreased in comparison to the 5083-H116 sample exposed to 175°C for 100 hours.
Figure 3. 60: SEM images of remediated sample of 5083-H116 exposed to 175°C for 100 hrs then
remediated at 280°C /15Min followed by re-exposure to 175°C/100hrs. Splits are not evident in
remediated sample but then re-appear (arrowed) in thermally exposed sample.

SEM images of 5059-H131 thermally exposed at 175°C and for 100 hours and then fatigue
tested, shows that splits appear at various locations, including overload, Figure 3.61. The

\[
\Delta K = 7.9 \text{ MPa√m}, K_{\text{max}} = 10.8 \text{ MPa√m} \\
175°C/100hrs (Splits) \\
\text{Hardness}=51 \text{ HRB}
\]

\[
\Delta K = 8.1 \text{ MPa√m}, K_{\text{max}} = 11.6 \text{ MPa√m} \\
175°C/100hrs + 280°C /15min \\
(No S-T Splits) \\
\text{Hardness}=45 \text{ HRB}
\]

\[
\Delta K = 11.9 \text{ MPa√m}, K_{\text{max}} = 17 \text{ MPa√m} \\
175°C/100hrs + 280°C /15min + 175°C/100hrs \\
(Splits re-appear) \\
\text{Hardness}=44 \text{ HRB}
\]
splits present in overload were visible without magnification. Thermal remediation treatment of 240°C, 260°C and 280°C for 15 minutes did not eliminate the splits in fatigue and overload, Figure 3.56.

Figure 3.61: SEM images of 5059-H131 sample exposed to 175°C for 100 hr which exhibits splits (arrowed) at various ΔK, including overload.
Figure 3.62: SEM image of a thermally remediated sample of 5059-H131 previously exposed to 175°C for 100 hrs and remediated at 280°C /15Min. Splits (arrowed) are present at various $\Delta K$.

Similar attempts were made to thermally remediate 5456-H131 and results similar to that on 5059-H131 were obtained. Splits continue to be exhibited in overload and are not eliminated after thermal remediation as shown in Figure 3.62 and 3.63. However, the splits are now perpendicular to the crack growth direction in these cases.

Figure 3.63: SEM images of 5456-H131 sample exposed to 175°C for 100 hrs which exhibits splits (arrowed) in overload. Crack growth direction is top to bottom.
Figure 3. 64: SEM images of remediated sample of 5456-H131 sample exposed to 175°C for 100 hrs then remediated at 280°C /15Min. Splits (arrowed) are still present perpendicular to crack growth direction. (SEM image was taken at $\Delta K = 11.8$ MPa$\sqrt{m}$)

As described in section 2.5, mechanical remediation treatments were also conducted on 5456-H116 samples exposed to 175°C for 100 hr. The specimens were compressed to different strain levels (e.g. 1.7% and 3.7%) prior to fatigue testing in air. SEM images shown in Figure 3.65 still reveal extensive splitting.
3.6.5 SEM Imaging with Back-Scatter Electron Detector (BSE)

In order to more fully characterize the lateral splits, SEM imaging was conducted on metallographically sectioned and polished samples taken perpendicular to the fracture surfaces. One fatigue sample of 5456-H116 thermally exposed to 175°C for 100 hours obtained from previous work [33], sectioned and examined in the SEM. This particular specimen was chosen because SEM views of the lateral splits revealed they were wider than in other thermally exposed specimens. Figure 3.66 shows back scatter electron (BSE) images revealing splits that are in range of 25 to 75 μm deep. Another examination was conducted on the 5456-H116 sample thermally exposed to 100°C for 5000 hours and tested at constant ΔK of 13 MPa√m and 5456-H116 thermally exposed to 70°C for 4000 hours, shown in Figures 3.67 and Figure 3.68, respectively.
Figure 3. 66: SEM images of lateral splits (arrowed) shown in cross sections of 5456-H116 specimen thermally exposed to 175°C for 100hrs and tested in Rising $\Delta K$. The $K_{\text{max}}$ for splits in this region of the fracture surface is approximately 25 MPa$\sqrt{m}$. Fracture surface is at the top of each image. Crack growth direction is into the page.

Figure 3. 67: SEM images of lateral splits (arrowed) in cross sections of 5456-H116 exposed to 100°C for 5000 hrs and tested in Constant $\Delta K= 13$ MPa$\sqrt{m}$. Fracture surface is at the right of each image. Crack growth direction is into the page.
Figure 3.68: SEM images of lateral splits (arrowed) in cross sections of 5083-H116 exposed to 70°C for 4000 hrs and tested in Rising AK. The $K_{\text{max}}$ for splits in this region of fracture surface is between 11-13 MPa$\sqrt{\text{m}}$. Fracture surface is at the right of each image. Crack growth direction is into the page.

3.6.6 Focused Ion Beam (FIB) Imaging of Splits

Observation of lateral splitting can also be obtained via Focused Ion Beam technique. A fatigued sample of 5456-H116 thermally exposed to 175°C for 100 hours was sectioned
via FIB. SEM imaging was first used to image the splits and then a region of splitting was selected a certain $K_{max}$. FIB was used in order to remove material from the fracture surface and reveal the cross section of lateral splitting. At several points the split depth was measured and recorded. Similar results were obtained as that shown in the metallography and conventional cross sectioning, Figures 3.69 and 3.70.

Figure 3.69: SEM images of 5456-H116 sample exposed to 175°C for 100 hrs and fatigue tested.

Splits (arrowed) sectioned via FIB examination.
3.6.7 Electron Back-Scatter Diffraction (EBSD)

Another fracture surface examination technique was used in order to investigate the dependency of splitting to the orientation of grains. A cross-sectioned sample of 5456-H116 thermally exposed to 175°C for 100 hrs and fatigue tested was fine polished to prepare a very smooth surface as shown in Figure 3.71. EBSD technique was used to characterize the orientation relationship of grains around the splits. In the first trial, orientation mapping was not successful so FIB technique was used to remove very thin layer of polished surface. The orientation mapping subsequently obtained was much better. This is shown in Figure 3.72 and Figure 3.73.

Figure 3. 70: SEM images of 5456-H116 sample exposed to 175°C for 100 hrs showing the depth of splitting revealed by FIB.
Figure 3. 71: SEM image of 5456-H116 sample exposed to 175°C for 100 hrs showing splits (arrowed) on cross section of polished surface before EBSD.

Figure 3. 72: SEM image of 5456-H116 sample exposed to175°C for 100 hrs, sectioned, and then additionally polished via FIB.
Figure 3.73: SEM image of 5456-H116 sample exposed to 175°C for 100 hrs after orientation mapping.
4 Discussion

4.1 Hardness Testing Trends

As it can be seen from Figure 3.3 to Figure 3.6 and Table 4.1, 5059-H131 exhibits a greater hardness than 5456-H131 by about 10%, and 5456-H131 exhibits greater hardness than 5456-H116 and 5083-H116 by about 11%. Thermal exposure of 5059-H131 specimens do not show any decrease in hardness for 60°C and 70°C thermal exposures for times up to 4000 hours. However, exposure of 5059-H131 to 175°C/100hrs causes a decrease of about 10% in Rockwell B hardness. The hardness changes after thermal exposure of 5456-H131 specimens were similar in that they did not show a dramatic decrease in hardness with increasing thermal exposure time and temperature for 60°C and 70°C thermal exposure for up to 4000 hours, with more significant changes after exposure to 175°C /100hrs.

Table 4.1: Summary of Hardness Rockwell B Hardness values for as-received alloys

<table>
<thead>
<tr>
<th>Alloy Type</th>
<th>Rockwell B Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083-H116</td>
<td>52±1</td>
</tr>
<tr>
<td>5456-H116</td>
<td>54±0.9</td>
</tr>
<tr>
<td>5456-H131</td>
<td>60.8±0.5</td>
</tr>
<tr>
<td>5059-H131</td>
<td>66.5±0.6</td>
</tr>
</tbody>
</table>

As it was explained in section 1.2, 5083, 5059 and 5456 alloys, have high strength in the annealed condition due to their high Mg content ranging from 4 to 6 %. These alloys
exhibit comparable elongation characteristics to alloys with much lower Mg content, as Mg concentration below 7% do not exhibit appreciable precipitation hardening [6] as it can be seen from Figure 4.1. In addition to the solid solution strengthening by Mg which occurs up to 17.4 wt% Mg [6], minor addition of Cr, Mn and Ti provided for grain refinement and raise the recrystallization temperature for these alloys [4], [6]. Addition of Fe and Si form dispersed intermetallic particles that provide another strengthening mechanism in 5xxx series alloys [6]. Also, small Cu additions are added to pin dislocations with small strains, leading to work hardening. Zinc is added primarily for strengthening purposes by tying up some amount of Mg in MgZn2 particles [6]. As it was summarized in Table 2.2, 5059 has more alloying elements like Fe, Si, and Zn. These elements are the main ones to increase the strength of these alloys. Therefore, as it is expected and also listed in Table 2.1, 5059 has better nominal mechanical properties. Generally, 5059 is stronger than 5456 and 5083.
On the other hand, two of the alloys, 5083 and 5456, have H116 temper and two other alloys, 5059 and 5456, have H131 temper. H116 is a cold rolling temper while H131 is a strain hardened temper. This could affect significantly the mechanical properties like Rockwell B hardness. Generally, in the present work, H131 temper alloys, shown to have higher Rockwell B Hardness than H116. This could be due to the sequence of treatments that could affect the microstructure and dislocation density.
In addition to the differences in alloy composition and temper affecting hardness, the 175°C/100hrs thermal exposure produces the lowest hardness for all specimens. Such observations are consistent with various other studies in the literature [48], [49] where similar tests were conducted on Al-Mg alloys. Thermally-induced degradation of Rockwell B hardness can be produced by various factors. It could be a function of alloy and temper type, both of which are intrinsic characteristics of the materials. Such degradation could also be a function of annealing-induced changes to the microstructure and/or Mg segregation to the grain boundaries as a result of sensitization. The two most likely culprits for the degradation of hardness are annealing and sensitization as discussed previously [33].

Exposure of cold- or warm-worked materials to elevated temperatures may cause the processes of recovery, re-crystallization, and/or grain growth to occur, depending on the time/temperature combinations. However, the temperatures investigated presently (60°C, 70°C, 80°C, 100°C and 175°C) are unlikely to produce recrystallization and grain growth based on previous work on cold-worked Al-Mg alloys that illustrated temperatures in excess of 190°C are required to produce such changes in microstructure for cold-rolled Al-Mg alloys [50], [51]. Therefore, it is unlikely that the matrix in the present alloys re-crystallized during thermal exposures below 175°C. Related work on 5083 alloys [52] that was designed to induce recrystallization was carried out at 400°C, suggesting that the commercial alloys exhibit a higher recrystallization temperature in comparison to the binary Al-Mg alloys. Moreover, as it was concluded in other previous work [33], it is also unlikely that grain growth contributed to the mechanical property evolution. Examination
of the microstructures after thermal exposure did not reveal any significant changes in
grain size from that shown in the as-received 3D images shown earlier in previous work
[33].

Thus, it is likely that the decrease in mechanical properties observed presently
results from recovery due to annealing. An additional source of hardness reduction could
relate to the process of sensitization, whereby Mg segregation to the grain boundaries and
the eventual formation of $\beta$ phase depletes the matrix of some of the solid solution
strengthening provided by Mg. This phenomena correlates with the work of Oguocha et
al. [48] that reported similar observations of hardness/strength reduction for higher
temperature but shorter time mechanical tests on 5083-H116. In order to distinguish
between which observed effects are caused by recovery and which by sensitization,
substantial TEM and DSC testing needs to be conducted in future work.

It is also important to recognize that a hardness indentation produces a large strain
at the indent (e.g. 8%-[53], [54]), thus direct comparison to tensile test data for the 0.2%
offset yield strength may not be appropriate. Previous work [33] has shown that both
hardness and yield strength decrease up to 100hrs for 80°C, 100°C and 175°C. However,
for thermal exposures greater than 100 hours, the yield strength continues to decrease
while the hardness returns to roughly the as-received levels. This discrepancy likely arises
because of the vastly different strains present in the hardness indent in comparison to the
0.2% offset yield strength, as suggested above. In effect, the hardness indent strains the
material to some strain (stress) well up on the stress-strain curve. In that regard, the
hardness indent not only captures yielding, but it also captures work hardening since it
induces about 8% strain in to the material [53], [54] as it is shown in Figure 4.2. The previous data [33] shows that while the 0.2% offset yield stresses reduce significantly with aging in different samples, the stress-strain curves show that the strengths at about 8% strain are similar. For example, specimens of 5456-H116 thermally exposed to 80°C for 100, 1000 and 2000 hours have values for 0.2% offset yield stresses of 260, 245 and 236 MPa respectively, but the values for UTS are 363, 362 and 360 MPa respectively. Thus, while the yield stress reduction appears to occur by a combination of annealing reducing the effects of cold work and removal of Mg from the matrix, the hardness recovery at long time exposures produces roughly as-received hardness levels. Similar behavior was also reported by Oguocha et al. [48].

![Figure 4.2: Showing plastic zone under the indenter][53]
The present work on 5456-H116 and 5083-H116 similarly showed hardness reductions up to 100hrs for 175°C and then a return to as-received hardness values. These observations could not be confirmed for 5059-H131 and 5456-H131 as only limited sensitization treatments and specimens of these materials were available.

4.2 Tension Testing Trends of 5456-H131

Tension testing was only conducted in the short transverse direction in order to probe this potentially weak direction that was detected as splitting in the S-T direction for 5456-H131 alloy tested in fatigue. As it can be seen from Figure 3.18, the 0.2% offset yield stress and ultimate tensile stress depend on the thermal exposure temperature and time. It is clear that by increasing the thermal exposure temperature up to 175°C for 100 hours, the yield stress and ultimate tensile stress decreased about 30% from the as-received condition. Additional exposure in water does not produce a dramatic change in the yield stress and ultimate tensile stress (less than 5%) for different thermal exposure time/temperature.

However, specimens of 5456-H131 thermally exposed to 175°C for 100 hours showed delamination behavior in ST direction along the circumference of tension samples tested in air without previous exposure to water. This can be related to the S-T splitting observed previously [33] and presently in fatigue crack growth tests conducted in the L-T orientation. These grain boundary failures can be exacerbated by tension testing directly in the weak (i.e. S-T) direction. As was found in fatigue crack growth testing of 5456-H131, the S-T splits obtained in fatigue testing in the L-T orientation behave differently
in this alloy. Figure 3.55 and 3.56 showed the direction of S-T splitting obtained in fatigue which will be explained in section 4.4.

As mentioned earlier, the reason for conducting S-T tension testing was to directly evaluate the weakness of this direction by taking tension samples to directly test this weak plane in tension. As it can be seen in figure 3.35, splitting is clear along the circumference of tension sample of tested in air. This is driven by stresses (i.e. $\sigma_{11}$) that develop in this direction. Mechanistically what appears to have happened during the deformation, in the material with coarse grain boundary precipitates as well as matrix precipitation, is shown schematically in Figure 4.3 [55]. This schematic is taken from work conducted on precipitation hardened aluminum alloys where grain boundary precipitation creates a precipitate free zone (PFZ) that substantially lowers the strength of the grain boundary as compared to the matrix. The application of a tensile stress in such a heterogeneous microstructure has been proposed to localize strain in the weaker PFZ, thereby producing eventual ductile fracture near the grain boundary, but at a significantly reduced strain (and stress) in comparison to a material where no PFZ exists. Figure 4.3 (a) shows the likely deformation/fracture events in the absence of a PFZ, assuming the presence of gb precipitates. In such a microstructure, there would be incompatibility between stress and strain due to the difference in the elastic modulus of the grain boundary precipitate with respect to the matrix due to the presence of nondeformable grain boundary precipitates. Fracture would likely intervene at the gb precipitates but crack growth into the stronger matrix would be more difficult than crack growth into a weaker PFZ as shown in Figure 4.3 (b). In this case, if there is a PFZ present, Figure 4.3 (b), where the matrix precipitate...
is not the most stable phase [55], then the strain will localize in the PFZ region and strongly promote the formation of microvoids at the grain boundary precipitates and their subsequent growth into the weaker PFZ. The main point here is that the absence of a PFZ region requires a higher void nucleation and growth strain in the grain boundary regions. The presence of gb precipitates and weaker PFZ will localize strain into the PFZ and thereby promote localized deformation and fracture in this region, eventually producing intergranular fracture (i.e. fracture along the gb regions) at much lower ductility than that required in materials obtained in the as-received (i.e. non-sensitized) condition.

Figure 4.3: Schematic representation of the deformation processes when (a) no pfz is present, and when (b) wide pfz present [55].
Thermal exposure of 175°C for 100 hours degrades the 0.2% offset yield stress and ultimate tensile stress dramatically (about 40%) for 5456-H131 for tests conducted in air as well as those exposed to water.

The present work is consistent with previous work [33] that showed that thermal exposure time and temperature, over a more limited range of conditions than tested presently, can produce a significant effect on many mechanical properties, including tensile properties. Specially, reduction in 0.2% offset yield strength and ultimate tensile strength were found to correlate well with an increase in thermal exposure time and temperature.

The trend of decreasing strength can be explained by considering the combination of sensitization and recovery effects, which was suggested in Section 4.1 for the hardness reductions observed. Additionally, sensitization involves the mass transfer of chemical species which is discussed in detail in Section 1.3. Briefly, in 5xxx alloys, chemical constituents migrate to form a constituents-rich second phase at the primary-phase grain boundaries [4], [53], which is shown in Figures 1.7 and 1.10. This migration, which is governed by mass transfer, is a function of temperature and time and would result in the generation of depleted zone surrounding the grain boundaries, where the concentration of such minor constituents has migrated to the grain boundaries [56], [57]. As the depleted zone is no longer strengthened by the presence of the minor constituent atoms, the tensile properties decrease [53]. Therefore, sensitization is proposed to be a major contributor in the mechanical property evolution observed for thermal exposures of 5xxx series alloys. This degradation in properties can also be linked to a low temperature annealing of the
H116 temper, which would be expected to cause a decrease in yield strength and ultimate tensile strength and hardness. After short time periods, while annealing is likely still occurring, sensitization is also predicted to be occurring in conjunction with the effects of the recovery process. Also since the H116 temper is produced by cold working [38], annealing would still be expected to occur at longer period of times.

Various techniques have been used previously [16], [19], [52], [57–59] in order to provide evidence for sensitization of 5083 and 5456 alloys with thermal exposure. Positioning of the precipitate particles along the grain boundaries and the growth of the particles with thermal exposure have been confirmed with TEM in those works and are well documented. The presence of a PFZ is also evident in some works [55], [60–63].

4.3 Anticlastic testing

In the anticlastic tests, fracture occurred perpendicular to the plane of plate in the short transverse direction in all tests for 5456-H116 as it can be seen from Figure 3.37. SEM observations revealed intergranular fracture in the short transverse direction. In the anticlastic tests, specimen failure did not occur instantaneously and required numerous cycles and fracture paths to develop. Failure was mainly intergranular separation in short transverse direction. This could be due to the severe plastic deformation induced by opposite bending moments, reversed normal stress at the surface of the plate and reversed shear stresses at the midplane or neutral surface are produced. This produces a maximum normal stress at the plate surface and maximum shear stresses at the midplane which was shown previously in Figure 2.7. Unfortunately, only a few samples were available for testing and a range of thermal exposures and cyclic loads could not be tested in order to
directly show the effects of sensitization on the fatigue life in these tests. More testing is required with different thermal exposures in order to determine the fatigue life in Anticlastic specimens. This is proposed as future work that should be conducted in order to directly test the S-T direction in fatigue.

4.3 Fatigue Crack Growth Testing

After testing under three point bending, fatigue crack growth parameters like Paris slope, m, and overload $K_c$ were determined. There does not appear to be a big effect of thermal exposure time or temperature for testing in the long transverse (LT) direction. This is consistent with previous work [33]. However, SEM images presented in Section 3.6.3 indicate that lateral splitting can develop in the short-transverse direction (ST) during the LT and LS sample testing, and the details of this splitting depend on both thermal exposure time and temperature. It is important to also note that there is no evidence of splitting in the as-received specimens of 5083-H116, 5456-H116 and 5456-H131, although some lateral splitting in a different direction is evident in as-received specimens of 5059-H131. In order to distinguish the different type of splits observed in the S-T direction, Figure 4.4 provides a schematic of the observations along with 3-D representations of the test direction. As will be summarized shortly, splitting can occur in different ways depending on the material type, thermal exposure, level of $K_{\text{max}}$, and the type of environment tested (e.g. Air vs. Dry condition).
Figure 4.4 indicates that splitting may occur in sensitized materials in two different ways, denoted as Type A and Type B and these are typically not present in unsensitized materials. A summary of the types of splits observed and their location are provided in Table 4.2. It appears that the splits of Type A are not often present in overload except in 5059-H131, while 5456-H131 exhibits Type B splits in fatigue as well as in overload.

It should also be noted that splits are not exhibited near the outside surfaces of the samples, as schematically shown in Figure 4.4.
Table 4.2: Summary of splitting type that occurs in fatigue or overload for different alloys in AR vs. Sensitized conditions

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Sample Thickness</th>
<th>Type A Split</th>
<th>Type B Split</th>
<th>Splitting in overload?</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AR Sensitized</td>
<td>AR Sensitized</td>
<td>AR Sensitized</td>
<td></td>
</tr>
<tr>
<td>5083-H116</td>
<td>6.20 mm</td>
<td>Few</td>
<td>Extensive</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td>None</td>
<td>None</td>
<td>No</td>
</tr>
<tr>
<td>5456-H116</td>
<td>6.25 mm</td>
<td>None</td>
<td>Extensive</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td>None</td>
<td>None</td>
<td>No</td>
</tr>
<tr>
<td>5059-H131</td>
<td>6.25 mm</td>
<td>None</td>
<td>Very Extensive</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Few</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Yes (Type A)</td>
</tr>
<tr>
<td>5456-H131</td>
<td>25.5 mm</td>
<td>None</td>
<td>None</td>
<td>Extensive</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>No</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Yes (Type B)</td>
</tr>
</tbody>
</table>

The stresses and stress states that drive Type A splits are different than those that drive Type B splits, as discussed later. There are also some differences in the depth (i.e. into the sample) of the splits as well as their width (i.e. opening). The former could be related to the plastic zone size ($r_y$), which is different in the different regions of the fatigue fracture surface exhibiting splits for each of the alloys and thermal exposure conditions, as the plastic zone size is controlled by the yield strength, level of stress intensity factor, and whether the stress state is plane stress or plane strain. In plane stress, the plastic zone size is calculated from Equation 4.1, while that in plane strain is calculated from Equation 4.2.

\[
\begin{align*}
    r_y &= \frac{1}{2\pi} \left( \frac{K_I}{\sigma_{YS}} \right)^2 \\
    r_y &= \frac{1}{6\pi} \left( \frac{K_I}{\sigma_{YS}} \right)^2
\end{align*}
\]

Equation (4.1)

Equation (4.2)
A schematic of the plastic zone around the crack tip is shown in Figure 4.5 in both plane strain and plane stress conditions. As illustrated by both Equations 4.1 and 4.2 and Figure 4.5, the plastic zone size in plane stress is three times greater than plane strain.

![Figure 4.5: Schematic of plastic zone around a crack tip in plane strain (Eq. 4.2) vs. plane stress (Eq. 4.1)](image)

Table 4.3 summarizes the magnitude of the plastic zone sizes in both plane stress and plane strain for alloys where Type A and Type B splitting was observed. Included in Table 4.3 is the 0.2% offset yield strength in the as-received condition along with a range of different $K_{\text{max}}$ in the fatigue cycle where splits were observed on the fracture surface of sensitized materials. The highest $K_{\text{max}}$ in Table 4.3 for each sample listed provides the maximum $K_{\text{max}}$ where splits were observed, as summarized later in Figures 4.9, 4.10, 4.11 and 4.12. These calculations were done for as-received materials because of availability of yield strength values. As it was confirmed previously in section 4.2, the values of yield strength in sensitized materials are lower than as-received, so the value of plastic zone size would be larger than that listed in Table 4.3 in sensitized materials. Thus, since the plastic zone size is dependent on the ratio of $(K/\sigma_y)^2$, increases in $K$ and/or decreases in $\sigma_y$ can
significantly change the plastic zone size. Furthermore, the stresses inside the plastic zone, are very dependent on whether plane stress or plane strain conditions dominate.

It was also observed that lateral splits occurred more frequently, propagated more deeply into the sample, and exhibited a wider opening with increasing thermal exposure time and temperature. For example, specimens of 5456-H116 given exposure to 175°C/100hrs exhibited splits of about 60 μm width and 50 μm depth at $K_{\text{max}}$ of about 25 MPa√m. In contrast, values for split width and depth are about 30% to 50% less, respectively, for the low temperature/longer time exposures.

**Table 4.3: Summary of plastic zone size calculation at different $K_{\text{max}}$ where splitting was observed**

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Sample Thickness</th>
<th>Yield Stress (MPa) of AR Material</th>
<th>$K_{\text{max}}$ MPa√m</th>
<th>$r_y$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Plane σ</td>
</tr>
<tr>
<td>5083-H116</td>
<td>6.20 mm</td>
<td>215</td>
<td>8</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>13</td>
<td>0.44</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19</td>
<td>0.65</td>
</tr>
<tr>
<td>5456-H116</td>
<td>6.25 mm</td>
<td>230</td>
<td>8</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>13</td>
<td>0.39</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19</td>
<td>0.56</td>
</tr>
<tr>
<td>5456-H131</td>
<td>6.25 mm</td>
<td>285</td>
<td>8</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>13</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19</td>
<td>0.37</td>
</tr>
<tr>
<td>5059-H131</td>
<td>25.5 mm</td>
<td>303</td>
<td>8</td>
<td>0.14</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>13</td>
<td>0.22</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>19</td>
<td>0.33</td>
</tr>
</tbody>
</table>
Thus, both the presence and appearance (i.e. depth, width) of splits is dependent on a number of factors (e.g. plane stress/strain, sensitization, K, strength). The effect of strength level and K directly affect the magnitude of plastic zone size in both plane stress and plane strain, as shown by Equations 4.1 and 4.2 and the calculations shown in Table 4.3. The importance of stress state (plane strain vs. plane stress) on the stresses in the crack tip region are shown by the schematics in Figures 4.6 and 4.7. Since there is no $\sigma_{33}$ in plane stress, it is unlikely that splits can be promoted, assuming that the thru-thickness stress $\sigma_{33}$ is contributing to the observation.

The importance of sensitization is also somewhat complicated and must be linked with the observations made on the S-T tension samples where grain boundary (gb) failure was observed in sensitized samples. This clearly indicates that regions of grain boundary weakness evolve with increasing sensitization, and these will fail if a stress (and strain) of sufficient magnitude is applied across such grain boundaries. However, in the fatigue experiments, this relates to the requirement of a sufficient level of $\sigma_{33}$ (and strain) in order to cause such splitting. Thus, as explained in Section 4.1 and 4.2, the loss of cold work combined with loss of Mg in the matrix due to its migration to the gb will have multiple effects. On the one hand, the yield strength will be reduced, thereby decreasing the maximum value of $\sigma_{33}$ possible in plane strain. If the yield strength reduction is great enough, the sample may transition to plane stress with increasing K, thereby removing $\sigma_{33}$ completely, and prohibiting splitting completely (assuming $\sigma_{33}$ is important). On the other hand, the PFZ will localize flow near the grain boundary and promote gb failure, as long as $\sigma_{33}$ is high enough.
This concept can be invoked as one of the main factors contributing to the observations of higher levels (and different types) of splitting in 5059-H131. As discussed earlier, this alloy has higher strength, and since the crack tip stresses scale with the yield strength, this promotes more splitting compared to the other alloys. Figure 4.6 shows the stress distribution ahead of a crack in plane stress. As illustrated, yielding at the crack tip limits the peak stress to the yield stress in plane stress. Since there is no $\sigma_{33}$ stress in plane stress, it is not possible to produce Type A splits unless the gbs are very weak. As pointed out earlier, plastic zone size is the distance ahead of the crack tip where the elastic stress distribution exceeds the yield criterion for plane stress, as illustrated in Figure 4.6. Due to yielding within the plastic zone, the stresses are lower than the values from the elastic stress field equations. The yielded material thus offers less resistance than expected, and large deformations occur, and there is no thru thickness stress, $\sigma_{33}$. As pointed out, the plastic zone size increases if the stress (and K) is increased, and it is smaller for the same K for material for higher yield strength. Since 5059-H131 is stronger, it will experience higher stresses in the three different directions in plane strain. This may promote greater splitting in this alloy. In overload, the plastic zone size is still within the plane strain regime, although it is much bigger than that obtained during fatigue which means that this material can exhibit larger splitting in overload as shown in Figure 3.53.
Now, considering that the thickness is much larger compared to the plastic zone size, the material outside the plastic zone is subjected to relatively low stresses $\sigma_{11}$ and $\sigma_{22}$, thus relatively small Poisson contraction in the z-direction, as its thickness in the z-direction is held nearly constant by the surrounding material. Hence, as a result, a tensile stress develops in the z-direction, which elevates the value of $\sigma_{11} - \sigma_{22}$ necessary to cause yielding, in turn decreasing the plastic zone size relative to that for plane stress. This stress state, known as plane strain, has higher $\sigma_{22}$ and $\sigma_{33}$ stresses than exhibited in plane $\sigma$.

Figure 4. 6: Small-Scale Yield Model for Restricted Crack Tip Plastic Deformation [64].

Figure 4. 7: Type A Delamination caused by $\sigma_{33}$ at crack tip plane Strain ($r_1<<$thickness), while Type B Delamination caused by $\sigma_{22}$ at crack tip in either plane strain (left) or plane Stress ($r_1 \sim$ thickness).
Considering the above explanation, the dependence of lateral splitting of Type A and splitting of Type B on \( K_{\text{max}} \) is dependent on the stress state of the material along the fracture surface plane during fatigue crack growth in addition to the weaknesses of gb’s. As shown in Figure 4.7, it is possible that split development is the result of a Mode I driven fracture event that is promoted by transverse stresses (i.e. \( \sigma_{33} \)) for type A and \( \sigma_{22} \) stresses in the plane of the crack for Type B that develop. While the \( \sigma_{33} \) stresses can only exist in plane strain, \( \sigma_{22} \) stresses are always present, except at the crack tip where they must be zero at the free surface. In other words, high tensile stress at the crack tip and the high constraint provided by the surrounding elastic material in plane strain produces the triaxial stress state shown on the left in Figure 4.7 and 4.8 while the stresses in plane stress as shown on the right of Figure 4.7.

To begin to quantitatively analyze the Type A lateral splits, the range of \( K_{\text{max}} \) where splitting was observed was summarized in Section 3.4. The dependence of Type A lateral splitting on thermal exposure time and temperature is evident for all of the alloys tested. Also, in all the alloys, the appearance of large amounts of lateral splitting occurs in a narrow range at lower thermal exposure temperatures. But the time onset of splitting appears to be highly temperature dependent. It is beneficial to consider graphical representation of the conditions required for splitting, as is provided in Figure 4.9 to Figure 4.12. These plots show the \( K_{\text{max}} \) range in which splitting is observed as a function of thermal exposure time and temperatures. Therefore, these figures can be viewed as a guide to providing a map of thermal exposure and testing conditions where Type A lateral
splitting in 5083-H116, 5456-H116 and 5059-H131 and Type B splitting in 5456-H131 has been found to occur in the different alloys tested.

Figure 4. 8: Cracked plate in which the plastic zone size is of the same order of magnitude as the plate thickness. The plastic zone at mid thickness has a plane stress shape, but there is a zone of high triaxiality close to the crack tip [64].
Figure 4. 9: Summary figure of $K_{\text{max}}$ in fatigue where lateral splitting (Type A) is observed in 5456-H116 in Air

Figure 4. 10: Summary figure of $K_{\text{max}}$ in fatigue where lateral splitting (Type A) is observed in 5083-H116 in Air
Figure 4. 11: Summary figure of $K_{\text{max}}$ in fatigue where lateral splitting (Type A) is observed in 5059-H131 in Air

Figure 4. 12: Summary figure of $K_{\text{max}}$ in fatigue where splitting (Type B) is observed in 5456-H131 in Air
SEM images presented in Section 3.6.3 indicate that the characteristics of splits developed in the S-T direction during the L-T direction testing do exhibit dependency on thermal exposure time and temperature. In most cases, lateral splitting occurred above a $K_{\text{max}}$ of 8 MPa√m and typically higher. At this stress intensity level, the plastic zone size is different in the different alloys as provided in Table 4.3 for as-received alloys. In sensitized alloys, the yield strength is somewhat lower, thereby limiting the peak $\sigma_{33}$ stresses, but the plastic zone size is bigger at certain $K_{\text{max}}$ and the grain boundary regions are somewhat weaker, suggesting that the material can split easier as long as the sample remains in plane strain.

Also, for a given sensitized material and strength level, increasing $K_{\text{max}}$ causes an increase in plastic zone size. Low K values produce small plastic zone sizes, thereby limiting the depth of split as well as width. Increasing the K, in contrast, increases the plastic zone size, producing the potential of deeper and wider splits if the grain boundary strength is reduced sufficiently.

Additionally, the amount of splitting is different from alloy to alloy. SEM images showed that for a thermal exposure of 175°C for 100 hours, the highest number of splits occurred in 5059-H131. This can be explained by also considering the difference in the alloy and Magnesium content and also difference in temper type which is H131 rather than H116. Another factor could be plastic zone size ($r_3$) which is governed by yield strength ($\sigma_y$) and level of stress intensity factor ($K_{\text{max}}$). As it is summarized in table 4.3, depending on these factors, plastic zone size is different and the stress state could change significantly. In this table low, medium and high $K_{\text{max}}$ values were considered for the calculation of
plastic zone size. As it can be seen, increasing the level of $K_{max}$ increases plastic zone size. Also in most cases, splits do not seem to appear at the edges of samples. This can be explained considering the stress state change from the middle of the fracture plane to the edge as it is shown in Figure 4.13. It is clear that plastic zone size is much bigger in the edges of sample which is in plane stress due to the fact that $\sigma_{22}$ is zero at free surface. While in the middle of fracture plane, material feels plane strain which is much more severe than plane stress condition.

![Figure 4.13: Through-thickness plastic zone schematic](image)

**4.4 Remediation Treatments to eliminate splitting**

Two different strategies were used in order to eliminate lateral splitting. Thermal remediation treatments and mechanical remediation. In some cases, Type A splits were
eliminated in fatigue crack growth. Type A and Type B overload splits were not eliminated using thermal remediation of the types attempted presently.

The ability to eliminate splitting over a range of $K_{\text{max}}$ in the thermally remediated specimens tested in Fatigue Crack Growth appears to be a function of remediation temperature and time. Increasing the remediation temperature in 5083-H116 and 5456-H116 from 240°C to 260°C and 280°C for 15 minutes appeared to narrow the range of $K_{\text{max}}$ for splitting, while the use of 280°C /15 minutes completely eliminated splitting. This can be explained based on the earlier discussion that reviewed the effects of sensitization on yield strength, segregation of the Mg to the grain boundary, as well as the formation of a PFZ that could accompany the presence of $\beta$ phase. It was observed that increasing the remediation treatment temperature from 240°C to 280°C decreased the yield strength.

The yield strength reduction after such remediation treatments could have more than one source. On the one hand, such high temperatures could produce additional recovery of the cold worked structure. There was no evidence of recrystallization at these higher temperatures. In addition, high enough thermal remediation treatments could dissolve some of the $\beta$ or other Mg-rich phases at the grain boundary. This would effectively put more Mg back into the matrix and increase the strength by solid solution strengthening, while also potentially eliminating or minimizing the PFZ. Although this awaits further analysis, it is likely that the strength drop is due to additional annealing of the material. The amount of the strength drop was 10%.
This remediation-induced loss of strength will produce a larger plastic zone size for a given $K_{\text{max}}$. While the stresses inside the plastic zone will be somewhat lower since they scale with the yield strength, the increased plastic zone size will produce deeper splits, providing the gb regions are sufficiently weakened to enable this to occur. Sufficient thermal remediation to eliminate the splits likely arises due to the dissolution of some \( \beta \) and enrichment of the PFZ with Mg to levels that prevent splitting at the $K$’s that previously caused splitting. The drop in strength is not sufficient to change the stress state from plane strain to plane stress as it was calculated for the as-received condition in Table 4.3, even with reduction of 10% in strength in remediated samples, and thus there will remain $\sigma_{33}$ stresses that could cause splitting of gbs that were weak enough.

None of the thermal remediation treatments utilized presently produced any effects on the delamination behavior of 5059-H131 and 5456-H131. A summary of the various remediation treatments along with their success is provided in Table 4.4. Thermal remediation treatments of 240°C, 260°C and 280°C for 15 minutes did not eliminate the splits in 5059-H131 and 5456-H131 previously exposed to 175°C for 100 hours. The reason could be due to the state of the stress and plastic zone size as discussed earlier. Also the alloy temper in these two alloys is different than the others. Due to the lack of information about this temper, it is not possible to relate temper treatment to this behavior at this time.
Table 4.4: Summary of results for thermal remediation treatments

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Remediation condition</th>
<th>Successful?</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083-H116</td>
<td>240°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>280°C/15min</td>
<td>Yes (type A)</td>
</tr>
<tr>
<td>5456-H116</td>
<td>240°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/30min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/60min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/300min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>280°C/10min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>280°C/15min</td>
<td>Yes (type A)</td>
</tr>
<tr>
<td>5059-H131</td>
<td>240°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>280°C/15min</td>
<td>No</td>
</tr>
<tr>
<td>5456-H131</td>
<td>240°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>260°C/15min</td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>280°C/15min</td>
<td>No</td>
</tr>
</tbody>
</table>

In attempting to explain the remediation treatments, it is also useful to discuss the potential effects of what are termed ‘stabilization treatments’ to the material. Stabilization treatments are used both to stabilize the mechanical and the corrosion properties of AA5xxx alloys. It refers to heat treatment process that provide resistance to sensitization.
This resistance to sensitization is achieved by heat treatment at a temperature just below where the $\beta$-phase precipitate will dissolve back into Aluminum solid solution and the precise temperature chosen depends on the Magnesium level in the alloy. As it was reported a number of years ago by Dix et al. [66] the stabilization temperature is a function of Magnesium level. This is shown in the Al-Mg phase diagram in Figure 4.14 [32] which was developed later by Scamans [32]. The shaded region of the figure shows the Magnesium level and temperature regime where sensitization can occur. At temperatures above the sensitization range but below the limit of solid solubility curve, precipitation of $\beta$ phase promotes stabilization. However, at temperatures below the stabilization temperature range, precipitation of $\beta$ phase occurs mainly on grain boundaries and sensitization occurs as result. It should be noted that stabilization treatments for mechanical property stabilization in Al 5xxx alloys are invariably sensitization treatments, although the temperatures and times involved are usually such that only low levels of sensitization to corrosion are developed [32]. As it can be seen from the Figure 4.14, for an alloy like 5083 the stabilization window is quite small and is between 220°C and 250°C. So this figure could be used as a guide to determine the appropriate range of thermal treatment in order to reverse the sensitization. It means that depend on the Mg wt%, one can determine what temperature should be used in order to stabilize Aluminum 5xxx alloy.
Mechanical remediation treatments were not successful in eliminating splitting in 5456-H116. The plastic strains utilized cause an increase in strength, thereby increasing the local stresses near the crack tip without affecting the near gb regions. Thus, there was no reduction in splitting in any condition as the stress state was still plane strain, the plastic zone size was reduced, and the gb regions were still weakened by the sensitization.

4.5 Environmental Effects

In all four alloys, Type A and Type B splits in the S-T direction are clearly evident in the air test, but are not present in the identical test conducted Dry. However, this is not the
case in the overload region for 5059-H131 and 5456-H131 which has Type A and Type B splitting respectively. In other words, even in the Dry test for these two alloys, splitting is evident in overload. These observations are summarized in Table 4.5 for all of the alloys.

The results indicate that environmental effects may contribute to the observation of splitting, but are not solely responsible, as overload regions exhibit crack growth at high velocity where environmental effects are usually not possible.

Table 4.5: Summary of environmental effects on elimination of splitting in sensitized alloys tested in Dry condition

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Type A Split</th>
<th>Type B Split</th>
<th>Splitting in Fatigue?</th>
<th>Overload Splitting?</th>
</tr>
</thead>
<tbody>
<tr>
<td>5083-H116</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>5456-H116</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>5059-H131</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>Yes (Type A)</td>
</tr>
<tr>
<td>5456-H131</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
<td>Yes (Type B)</td>
</tr>
</tbody>
</table>
5 Conclusions

After conducting a series of hardness tests, tension tests, fatigue crack growth experiments and thermal remediation treatments, the mechanical properties evolution after thermal exposures at temperatures of 60°C, 70°C, 80°C, 100°C and 175°C for up to 5000 hours has been catalogued for 5083-H116, 5456-H116, 5059-H131 and 5456-H131 materials. Based on the observations of this project, a number of conclusions can be made:


2. S-T direction tension testing indicates that increasing the time and temperature of thermal exposure for 5456-H131 decreases the yield strength and ultimate tensile strength. The two main factors contributing to this degradation are recovery and the removal of Mg due to sensitization.

3. The fatigue Paris constant, and overload $K_c$ are not measurably affected by the thermal exposures presently employed when testing is conducted in the L-T orientation.

4. Material separation in the S-T direction (lateral splitting) was observed on the fracture surfaces of all four 5058-H116, 5456-H116, 5059-H131 and 5456-H131 after sufficient thermal exposures. These features were observed at specific ranges
of $\Delta K$ (and $K_{\text{max}}$) including overload in some cases in 5059-H131 and 5456-H131. Splits occur in two different types, designated Type A and Type B. Type B splits only occur in 5456-H131. Similar splitting was observed in tension samples of 5456-H131 thermally exposed to 175°C for 100 hours and tested in the S-T orientation.

5. The splitting appears to be associated with the fracture of the material at or near the grain boundaries. This happens due to the evolution of segregation/precipitation of a phase at the grain boundaries that evolves as a result of thermal exposure, along with the local differences in strength that results.

6. Thermal remediation treatments of 5083-H116 and 5456-H116 at 280°C for 15 minutes eliminate the Type A splits that occur in fatigue crack growth.

7. Environmental conditions could have a major role on the splitting behavior of all four alloys tested in the fatigue crack growth tests. Splits evident in the Air test are not present in Dry test.

8. 5059-H131 and 5456-H131 alloys may be more prone to sensitization and splitting behavior. The split widths are much larger and they are present even in the overload region.

9. Thermal remediation treatments did not have any effects on the splitting appearance of 5059-H131 and 5456-H131. This may be due to their initial sequence of temper or treatment which is different than H116.
6 Suggestions for Future Work

Based on the results and discussion of Chapter 3 and 4, a number of possibilities for future work are suggested and summarized below.

6.1 Testing of thermal exposure times in excess of 10,000 hours

Many tension and bend bar specimens have been left in the temperature chambers for further thermal exposure. It is suggested that these specimens be removed after longer periods and tested in accordance with the Procedure of Chapter 2. To add to the bulk of work presented in the present work, this future work should allow for specimen removal at times that are in excess of 20,000 hours, which was the maximum thermal exposure studied in the present work. This will enable investigation of the effects of additional Mg segregation at lower temperatures for longer times.

6.2 Toughness testing of thermally exposed material of 5456-H131 in the S-T direction

As previous work along with the current work showed that the S-T direction is the weakest direction, toughness and fatigue testing in the S-T direction could fully probe the weakness of this direction by comparing S-T testing results with those obtained in the L-T direction after different time and temperature exposures.
6.3 Tensile testing of thermally exposed 5456-H131 alloy after aging in water at 80°C

As the 80°C thermal exposure tests have shown clear indications of sensitization, including the occurrence of lateral splitting in sufficiently long thermal exposures, some tension samples have been immersed in water while thermally exposed to 80°C. This may affect the grain boundary fracture behavior under certain situations. Exposure to water may induce corrosion as well as diffusion of hydrogen into the material. This could induce additional splitting.

6.4 Characterization of material via Isothermal DSC

Since the evolution of Mg-rich phases at different temperatures has been not been fully characterized, it would be beneficial to investigate such phase formation using Isothermal DSC. This work would be even more useful considering a series of very slow (less than 1°C per minute heating) DSC tracing of a range of thermally exposed conditions. Such DSC would allow more accurate identification of precipitate phase growth and dissolution than was possible in the previous work. It would be an excellent addition to the literature for a description of Mg-rich phase evolution in the 5xxx series, which, based on literature reviews, is found to be lacking.

6.5 Fatigue crack growth testing in wet/water environment

In order to investigate environmental effects on the fatigue crack growth behavior of 5xxx series alloys, fatigue testing in a humid environment as well as in a water environment would be beneficial.
6.6 Electrical Conductivity measurements of sensitized specimens

It has been shown that the electrical conductivity of AA5083 is largely controlled by the free magnesium level once the formation of Mg$_2$Si based on the silicon level in the alloy has been taken into account [32]. So, comparative conductivity measurements for the same thermal exposure treatments could reveal how much precipitation of Mg-rich phases occur during sensitization, as this will increase the conductivity. However, very little work has been published that evaluates the correlation between the effects of stabilization treatments and conductivity. Stabilization of a sensitized alloy is considered beneficial since it returns some of the Mg to the matrix. The use of conductivity measurements to confirm that a stabilization practice has been effective deserves thorough investigation. Decreases in conductivity would reveal reversion of some $\beta$ and return of some Mg to the matrix. However, decreases in conductivity to the solid solution levels would indicate that the material was inadvertently stabilized above the $\beta$-phase solvus and could still become sensitized in service.
Appendices

Appendix A- Schematics of plates along with dimensions
Schematics attached as a compact disk.

Appendix B- Tensile Specimen Fracture Surface images
SEM images attached as a compact disk.

Appendix C- Fatigue Crack Growth Specimen Fracture Surface images
SEM images attached as a compact disk.
References


Afterword

“What appears to be the END

may really be a new BEGINNING…”