Evaluation of Forging Strain for Maximum Grain Size in
Open-Die Forged 6061 Aluminum

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Bachelor of Science

by

Eric Strehl

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Abstract

In an effort to maximize grain growth of open-die forged 6061 aluminum alloy parts, samples from three of Weber Metals’s suppliers (Hidal Co-Almex USA Inc., TST Inc., and Vista Metals Corp.; to be called Supplier H, Supplier T, Supplier V respectively) were cut into cylinders with diameters of 0.75˝ and heights of 2˝, and put through a simulated forging process and heat treatment. It is possible that additional alloying elements may be promoting or inhibiting grain growth in the final part. Maximizing grain growth for aluminum alloy parts results in improved resistance to intergranular corrosion. Samples from each vendor had grain size analysis performed...
prior to running the experiment. Samples were heated to just over 650°F so that when placed in the Instron compression machine for simulated forging they would be between the usual forging temperatures for aluminum of 550 to 650°F. Samples were compressed at a rate of 50 mils/second to final strain values of 0%, 5%, 10%, 20%, and 50%. The samples were then heat treated at 1050°F for a soak of 4 hours. This allows for maximum grain growth to occur before being removed and quenched in water to room temperature. A sectioning, sanding to 600 grit sandpaper, etching with 10% NaOH, and desmut with 33.33% nitric acid allows for grain size analysis to determine which vendor had the largest grain size at each strain value. Grain size measurements were made according to the Heyn Lineal Intercept Procedure from ASTM E0112-12. Supplier H’s alloy with 0% forging strain applied provided the largest average grain size at 1754 µm.

Key Words: Materials Engineering, Forging, 6061, Aluminum, Grains, Grain Size, Corrosion, Intergranular, Grain Growth, Recrystallization, Weber Metals

Problem Statement

Often the aluminum forging industry is attempting to refine the grain size of aluminum alloys to increase the strength of the final product. In applications where strength limitations are not being pushed, other properties must be emphasized. In the example of product use in a corrosive environment, it is advantageous to produce the largest grains possible while achieving the required strength. The grain boundaries in 6061 aluminum are most susceptible to failure due to corrosion, and therefore minimizing the grain boundary fraction will maximize the chemical resistance of the product. The sponsor company, Weber Metals, has customers requesting this additional chemical resistance, and they are therefore attempting to maximize grain size. The forging process will be replicated, adjusting the total strain and subsequently heat treating to allow for maximum grain growth. The goal of this work is to determine the specific supplier and fabrication conditions that provide a final product containing as large a grain size as possible (target average set at 1.5” in diameter). In addition, the relationship between forging strain and grain size is to be explored to better understand how grain size distribution varies within a part.
Background

Wrought Aluminum

Wrought Aluminum is widely used for a variety of mechanical applications due to their workability and relatively high strength-to-weight ratio. These parts are also easy to apply coatings, anodization, or other finishing techniques to (Figure 1). Figure 2 shows how the structural properties of aluminum compare to those of other similar structural alloys. As evident from the specific tensile strength, many aluminum alloys are comparable to steel. Much of this strength is drawn from the alloying elements and heat treatments of the aluminum. When present in the correct proportions, magnesium and silicon form Mg2Si, which is responsible for the alloy’s ability to age harden. Heat treatable aluminum alloys, such as the 6xxx series, allow for the advantage of easy plastic deformation during the shaping process, while retaining the ability to be made significantly stronger and harder through heat treatment and aging. 6xxx series aluminum is comprised mainly of the alloy additions silicon, and magnesium, although it will commonly contain traces of other elements such as copper, chromium, and manganese. The relatively high corrosion resistance of this alloy allowed for its use in applications where it would be constantly exposed to the elements. The strength of 6xxx aluminum compared to other wrought aluminum alloys is displayed in Table I.

Figure 1: Two forged aluminum turbine blades used on Boeing 777.
Figure 2: A comparison of how aluminum alloys rank mechanically relative to other existing structural alloys. Both the tensile strength (a) and the specific tensile strength (b) are shown.

Table I: Comparison of Strength Ranges for Wrought Aluminum Alloys

<table>
<thead>
<tr>
<th>Aluminum Association series</th>
<th>Type of alloy composition</th>
<th>Strengthening method</th>
<th>Tensile strength range</th>
</tr>
</thead>
<tbody>
<tr>
<td>1xxx</td>
<td>Al</td>
<td>Cold work</td>
<td>70-175</td>
</tr>
<tr>
<td>2xxx</td>
<td>Al-Cu-Mg (1-2.5% Cu)</td>
<td>Heat treat</td>
<td>170-310</td>
</tr>
<tr>
<td>2xxx</td>
<td>Al-Cu-Mg-Si (3-6% Cu)</td>
<td>Heat treat</td>
<td>380-520</td>
</tr>
<tr>
<td>3xxx</td>
<td>Al-Mn-Mg</td>
<td>Cold work</td>
<td>140-280</td>
</tr>
<tr>
<td>4xxx</td>
<td>Al-Si</td>
<td>Cold work (some heat treat(a))</td>
<td>105-330</td>
</tr>
<tr>
<td>5xxx</td>
<td>Al-Mg (1-2.5% Mg)</td>
<td>Cold work</td>
<td>140-280</td>
</tr>
<tr>
<td>5xxx</td>
<td>Al-Mg-Mn (1-6% Mg)</td>
<td>Cold work</td>
<td>280-380</td>
</tr>
<tr>
<td>6xxx</td>
<td>Al-Mg-Si</td>
<td>Heat treat</td>
<td>150-380</td>
</tr>
<tr>
<td>7xxx</td>
<td>Al-Zn-Mg</td>
<td>Heat treat</td>
<td>380-520</td>
</tr>
<tr>
<td>7xxx</td>
<td>Al-Zn-Mg-Cu</td>
<td>Heat treat</td>
<td>520-620</td>
</tr>
<tr>
<td>8xxx</td>
<td>Al-Li-Cu-Mg</td>
<td>Heat treat</td>
<td>280-560</td>
</tr>
</tbody>
</table>

(a) Alloy 4032 is heat treatable.
Corrosion

While corrosion resistance is a strength of 6061, weaknesses can arise within the alloy causing corrosion to occur. When 6xxx series has more silicon than is required to form $\text{Mg}_2\text{Si}$ (i.e., a ratio smaller than 1.73:1 wt%), corrosion will increase because of the strong cathodic nature of the insoluble silicon. In addition, if there is excess $\text{Mg}_2\text{Si}$ when at the solutionizing temperature, it may reprecipitate upon cooling the part, showing up as dark precipitates (Figure 3).

![Figure 3: 6061 ingot showing the precipitation of $\text{Mg}_2\text{Si}$ (dark) on cooling after heat treatment due to the presence of more $\text{Mg}_2\text{Si}$ than could stay in solution.](image)

It is well documented that the addition of copper to 6061 aluminum reduces its resistance to intergranular corrosion. Intergranular attack is a localized corrosion that occurs at the grain boundaries when they are more easily corroded than the interior of grains. This selective corrosion can be caused by either the precipitation of a second phase or segregation that occurs at the grain boundaries. Regardless of whether the precipitate is noble or active, the presence of a precipitate near the precipitate free zone (PFZ) of the grain boundary is to be avoided. The chemical difference between the precipitate and the PFZ will cause microgalvanic corrosion to occur. Examples of a noble particle and an active particle are shown in Figure 4.
Figure 4: A depiction of how microgalvanic corrosion could occur from either a noble precipitate (a) or an active precipitate (b) in the PFZ.\textsuperscript{4}

The PFZs, which commonly occur at the grain boundaries, will accelerate intergranular corrosion as demonstrated above. An image from a transmission electron microscope (TEM) of the PFZ located on either side of the grain boundary is shown in Figure 5. Because there is a lack of solutes in the PFZs, the electrochemical potential of these zones is lower than that of the rest of the grain. This potential difference exposes the PFZs as weak points where galvanic corrosion may occur.\textsuperscript{8}

Figure 5: TEM image of a 6061-T6 grain boundary. Note the presence of a PFZ on both sides of the boundary.\textsuperscript{7}

Relationship of Grain Size and Corrosion

There is some debate amongst researchers regarding the effect of grain size on corrosion in aluminum.\textsuperscript{10} Some believe that the focus should be on grain refinement, as that may result in a decrease in a product’s galvanic reaction kinetics as well as its
susceptibility to pitting and stress corrosion cracking. These results are achieved by two different effects: the ease of passivation of fine-grained materials, and the physical breaking down of second phase particles below their critical size through the grain refinement process, preventing them from acting as localized cathodes. Other authors focus instead on the grain boundaries. They note that by increasing the size of the grains, the total area of grain boundaries is decreased, causing a reduction in the number of initiation sites for intergranular corrosion. Both sides of the argument have numerous papers detailing how they arrived at their separate conclusions. For the purpose of this report, the argument of increased corrosion resistance with increased grain size will be followed, as Weber’s clients asked for larger grain size due to corrosion issues.

Recrystallization

One of the most important variables affecting grain size is the amount of cold work in the part. In order for a metal to recrystallize on heating, there is a minimum amount of work that must take place. By altering the amount of work placed into the part, the resulting grain size will vary (Figure 6). For most metals, this critical work requirement is approximately 5-7%. The metal is then heated to recrystallize, as recrystallization is a thermally activated process. While the minimum recrystallization temperature is highly variable with changes in alloy chemistry, a commonly accepted approximation states that it is around 40% of the alloy’s melting temperature in Kelvin.

![Figure 6: Difference in the amount of work put into a part results in differences in maximum achievable grain size. With 5% forging strain (left) the grains are much larger after recrystallization than those in the sample with 50% forging strain (right).](image)

Recrystallization is a function of temperature, time, work, and solute atoms. The temperature determines the rate of recrystallization, while time, work, and solute atoms all determine the degree of recrystallization possible by controlling the migration of grain boundaries. The ability of the boundaries to move depends on the strain in the microstructure and any dislocation or solutes that may impede the movement. The basic equation that describes the migration of grain boundaries resolves the velocity $v$ by taking into account the mobility $M$ and the driving force $P$ through the expression:

$$v = M P$$  \((1)\)

In reality, the driving force $P$ is a complicated value that depends on direction of growth as well as a volumetric driving force, which accounts for the elimination of dislocations as the recrystallization occurs. However, these details are beyond the scope of this project.
A fully recrystallized structure is almost entirely strain free, with few dislocations located inside of the grains. Recrystallization will occur more quickly with higher amounts of cold working, but the added nucleation sites from the dislocations will result in smaller grains. If a significant improvement in the supplied material and control of the fabrication process, ultrafine dispersions of any impurities or dislocations can be formed. By decreasing their size, their negative influence on grain growth could lead to higher recrystallization temperatures and significantly larger grain sizes. However, having this sort of control on large sized full production parts is unrealistic, as creating these ultrafine dispersions is time intensive.

The rate of heating to the annealing temperature has a substantial effect on the final grain size as well. If there is a gradual ramping up to the annealing temperature, it will allow for the maximum amount of diffusion to occur, and in turn create larger grains. It is also possible for additional grain growth to occur on a much smaller scale following recrystallization; additional annealing leads to the gradual purging of small grains that are positioned unfavorably relative to their neighbors.

Disruptions to Grain Growth

Common alloying elements and impurities (Cu, Fe, Mg, Mn, Cr) are detrimental to large grain growth, and are commonly distributed in the ingot in such a way as to favor a finer grain size for improved strength. Having control of the casting and preheating conditions of the ingot would allow for the elimination of this difficulty. The shape of the grains are determined largely by Mn, Cr, Zr, as these are present in the form of fine dispersoid particles which form bands of high and low concentrations. These bands can disrupt the recrystallization of the grains, altering the final shape and size of the grains.

Experimental Procedure

Weber Metals provided 6061 sample cubes from three different suppliers (Hidal Co-Almex USA Inc., TST Inc., and Vista Metals Corp.; called Supplier H, Supplier T, Supplier V respectively). Weber Metals then machined these cubes into cylinders with an approximate diameter of 0.75" and a height of 2". A SentroTech ST-1100-666 furnace was utilized for the forging simulation and heat treatment of the samples. A 5584 series Instron Tensile and Compression Test machine was used to apply the specific strain percentages to the samples. Weber provided wool insulation in an attempt to minimize the heat exchange between the heated platens and the room temperature platens during testing. Images of each supplier’s material microstructure were taken in the as received condition (Figure 7).
These cylindrical samples underwent a simulated forging process where they were brought to 670ºF along with a set of small, hardened steel platens. These were all then quickly transported to the compression tester to have a specific strain percentage introduced at a strain rate of 0.05 inches/second while remaining at forging temperature (550-650ºF). The heated platens were placed on the top and bottom of the cylindrical sample to minimize heat loss (Figure 8). Following compression, the parts were then allowed to air cool to ambient temperature before going into the furnace again for annealing. They were then soaked at 1075ºF for four hours, and quenched in water upon removal (Figure 7). The strain values analyzed included 0%, 5%, 10%, 20%, and 50%. The compressive extension required to achieve these strains was calculated based on the height of each sample. A minimum load value was set to ensure that all slack between the crosshead platen and the heated platen was removed.

These samples were bisected about their vertical axis, and readied for analysis following standard metallography preparation techniques (Figure 9). Following this, each sample was etched using a 10% NaOH etchant for 15 minutes and rinsed with DI water before the application of the desmut. The desmut was a 33.33% nitric acid, used to remove any intermetallic compounds or other reaction products which were insoluble in the NaOH solution. The grain size was measured utilizing the Heyn Lineal Intercept Procedure from ASTM E0112-12. This involved the overlaying of measured horizontal, vertical, and two intersecting diagonal lines over a photomicrograph (Figure 10). This method includes counting the number of grains that each line intersects, and using this to approximate the average grain size.
Figure 8: Schematic showing the experimental procedure, starting with the simulated forging, followed by the heat treatment and grain size analysis.

Figure 9: A 2” long sample that is sectioned, sanded, etched, desmut, and ready for grain size analysis.
Realistic Constraints

Ensuring the safety of both the compression equipment and those using it for this project was an important portion of this project. Whenever performing compression tests on samples with diameter to length ratios above 1.0, the danger always exists that the part may be misaligned, causing it to kick out during the test. While this is reason enough for use of protective shields, this project also involved these samples being heated, adding an additional element of danger to what could happen if the heated part were to be ejected from the machine. Because of this risk, two ½˝ thick Plexiglas shields were placed at the openings of the Instron while the compressions were being performed.

During the initial trials, it was found that at higher strain values the samples were cooling much more quickly as the surface area in contact with the platens increased. This is a realistic constraint in the large-scale forging world, where stronger metals need large amounts of deformation. The manufacturability of these parts can be improved by increasing the temperature, but there will always be limitations to the size and depth to which parts can be forged. In the case of this study, this issue caused the load cell to initially reach its limit of 30,000 lbs before some of the larger strain values could be achieved. A two-fold solution was implemented, involving an improved heated platen method to reduce heat dissipation from the part, and a limiting of the forging strains to a 50% maximum. This was done largely for the safety of the equipment being used, and for the accuracy of the results.

Results

Looking at the results from the three different suppliers, it was clear that supplier H had the most substantial grain growth across all strains. The average grain sizes for each supplier are shown in Table II, and graphically displayed in Figure II relative to the strain values.

As expected, an increase in the forging strain corresponds to a decrease in average grain size. The maximum achieved grain size occurred at 0% forging strain for all suppliers, but supplier H had the largest average grain size of 1754µm in diameter (Figure 12).

<table>
<thead>
<tr>
<th>Forging Strain (%)</th>
<th>Average Grain Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Supplier T</td>
</tr>
<tr>
<td>0</td>
<td>1019</td>
</tr>
<tr>
<td>5</td>
<td>656</td>
</tr>
<tr>
<td>10</td>
<td>653</td>
</tr>
<tr>
<td>20</td>
<td>527</td>
</tr>
<tr>
<td>50</td>
<td>321</td>
</tr>
</tbody>
</table>

The most substantial finding was that, across all suppliers, grain size was maximized when no simulated forging was performed on the part. It can therefore be concluded that there is sufficient stress in the microstructure of the raw supplied aluminum for recrystallization to occur. While this may seem detrimental to the goal of maximizing grain size, it may lead to design improvements.
for these improved corrosion resistance parts. If the raw material being used to make the part already has passed the critical work value to allow recrystallization, minimizing any additional work will maximize the potential for grain growth.

![Graph comparing the average grain size for each supplier at each measured forging strain value. Supplier H yielded the highest grain sizes at all measured forging strains.](image)

**Figure 11:** Graph comparing the average grain size for each supplier at each measured forging strain value. Supplier H yielded the highest grain sizes at all measured forging strains.

![Microstructure of 0% forging strain samples from supplier T, H, and V. (left to right)](image)

**Figure 12:** Microstructure of 0% forging strain samples from supplier T, H, and V. (left to right)

## Discussion

The correlation between forging work and grain size has been shown through the trend across all three suppliers, but it can also be seen within single samples as well. Sample V9, for example, has a wide variation in grain size within a single photomicrograph. This is due to the specific mode of deformation in compression during the forging simulation. V9 was compressed to 50% strain, which
caused it to deform in a double barrel mode. This mode of deformation causes a stress concentration at the middle of the part. An example of this correlation of stress distribution and grain size is shown in Figure 13.

![Figure 13: Correlation of stress and grain size is exemplified in V9, with the small grain portions being areas of large applied strain, and the large grain portions being areas of lesser applied strain.](image)

As anticipated, it appears that the composition of the alloy has a profound effect on grain size, as even at 20% forging strain for supplier H, the achieved grain size is larger than even the maximum grain size achieved by supplier V. The surprising point about the variations in these chemistries is that the H supplier has the highest values of Fe, Mn, Mg, and Ti. The only substantial alloying element which supplier H does not contain the most of the three suppliers is Cr. While Cr is known to be an additive introduced into 6061 for grain refinement, it would be expected that the larger values of Fe, Mn, Mg, and Ti in this sample would be more than enough to make up this difference. This is not the case however, as sample H12 was capable of reaching the largest grain size of all tested samples. A possible explanation for this could be variation in the processing that goes into the billet at the supplier. If the supplier was able to keep more of these alloying elements in solution, their effect as nucleation sites for new grains would be minimized.

One potential variable in this experiment that may have affected the results is the impact of dynamic recrystallization. This has the possibility of occurring when large enough forces are applied to a heated object that some recrystallization can occur without surpassing the recrystallization temperature. Its impact was beyond the scope of this project however, and therefore it was disregarded.

**Conclusions**

When a company buys bulk material for a specialized application, it is crucial that they are aware of the processing that has gone into the supplied material. For the specific application which requires a highly corrosion resistant 6061 aluminum, the amount of work in the billet when received and the composition of the alloy are both crucial to making the highest quality and therefore most
effective part. Of the samples tested, supplier H’s 0% strained sample provided the largest average grain size. Because the other two suppliers also had their largest grain sizes with 0% forging strain, it can be concluded that these samples were received with more than the critical amount of work for recrystallization.

Future studies should be looking for variations in the heat treatment ramp, as well as the actual heat treatment temperature used. Knowing a range of strain values present in the specific part requiring the improved corrosion resistance would allow for an improved focus on the project. Corrosion testing on the samples would be beneficial to the final customer as well, so that they could see the advantage of conducting research on this topic.

References


**Acknowledgements**

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