Thermo-mechanical Stress Relief Analysis in PMMA and 6000 Series Aluminum

By

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Submitted to the
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in Partial Fulfillment of the Requirements for the Degree of
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Abstract

Stress relief of materials produced in bulk is a key part of the manufacturing process. The most common kinds are either thermal or mechanical and are commonly applied to commercial metal alloys. A third type, thermo-mechanical, utilizes thermal gradients to induce residual stresses of an equal and opposite nature to balance compressive and tensile stresses existing in the material after solutionizing. The experiment detailed in this work shows the effect of thermal gradients on residual stresses in polymethyl methacrylate (PMMA). A downhill quench from 95°C to 15°C is able to create a deflection of 2.36 millimeters, evidence of residual stress. A subsequent uphill quench from -40 to 100 degrees reduced the deflection by 37 percent. The finite element simulation of a 6000 series aluminum block verifies that under properly controlled processing parameters, it is possible to induce opposite stresses to relieve residual stresses in a quenched material. Additional limitations to the uphill quench technique are detailed in the following work so that thermo-mechanical stress relief may be properly applied to a range of materials.

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Chapter 1: Introduction

High strength aluminum alloys are a key constituent to many engineering designs. From aerospace to computer hardware, manufacturers consistently select these alloys because of the relatively light-weight nature of the metal and its impressive strength to weight ratio. In order to attain the desired mechanical properties, aluminum alloys are subjected to heat treatment and post processing techniques.

One of the most popular post processing techniques, and the particular technique discussed in this paper, is solution heat treatment. In solution heat treatment of an aluminum alloy, the metal system is brought up to its solid solution temperature (in the case of 7000 series aluminum, ca. 500 C) until the alloy reaches a thermodynamically stable, stress free state. It is then quenched to room temperature. The quench is typically carried out in water, but various other quenchents (oil, brine, etc.) can be used to control the severity of the cooling. This processing results in an unstable temper that will age spontaneously at room temperature.

This “natural” aging at room temperature occurs as the alloy system attempts to reach an equilibrium stress state from its current stressed state. As mentioned previously, the as solutionized condition is an unstable temper, and while it is required to reach the mechanical property requirements of high strength aluminum alloys, it results in a distribution of compressive and tensile stresses throughout the bulk of the material. These residual stresses appear upon the quench from the solid solution temperature of the alloy, and are the result of the thermal gradients established by the quench. The cooler surface of the material contracts differently than the hotter bulk, generating compressive stresses and their associated tensile stresses required to balance all stresses to zero. It should be noted that the distribution of these compressive and tensile stresses will be largely dependent on the geometry of the part being heat treated and the severity of the quench.
The residual stress within a metal alloy is of concern because of its direct connection to stress corrosion cracking, distortion during machining, and fatigue in service. Stress corrosion cracking and fatigue reduce the strength of the part and limit the serviceability. Distortion during machining is typically seen when a portion of the part material is removed, say when drilling a hole in a plate, resulting in an unequal balance of forces within the part. As the part is brought back to equilibrium (i.e., net compressive stress is equal to net tensile stress) the material bends.

There are two major stress relieving methodologies used in conventional manufacturing. The first is thermal stress relief. This processing technique gives the unstable aluminum alloy system enough thermal energy to either precipitate a stable phase or overcome diffusive mobility barriers such that residual stress may be relieved. The second is mechanical stress relief. This post processing technique involves applying compressive stresses strong enough to yield the aluminum alloy to strains of one to five percent, relieving residual stress with plastic deformation. While thermal and mechanical are the two commonly utilized stress relief methods, there is an additional thermo-mechanical post processing technique first investigated by Alcoa engineers in the 1950s called the uphill quench.

The idea behind the uphill quench is to subject the metal to a sharp positive thermal gradient in an attempt to induce residual stresses of an opposite nature to those created in the original water quench. In order to be effective, the positive uphill quench needs to be more severe than the initial water quench. According to Croucher's paper in the Journal of ASTM International, this is done most effectively by immersing the heat treated part in a bath of liquid nitrogen until it attains a uniform temperature profile at -196°C, then remove the part from the nitrogen bath and immediately blast the

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part with jets of high velocity steam. This technique developed by the Alcoa engineers H.N. Hill, J.S. Barker, and L.A. Willey in the 1960s and published in their article in the *Transactions of the American Society of Metals* reports efficacy up to 82 percent residual stress reduction. With reported stress reduction this effective, it is strange that this technique has gone largely unrecognized in the manufacturing world.

To better understand the uphill quench and its applicability to heat treatment, this work aims to offer a theoretical verification of the limits and processing variables of this processing technique. Efficacy ranges for dimensions of heat treated alloy will be evaluated over a range of acceptable metal alloy candidates. A thorough understanding of these factors will shed light on the true applicability of this processing technique and perhaps explain if and under what condition it should be utilized.
Chapter 2: Theory

Section 1: Heat Transfer

Heat transfer in solids is important to materials engineers because of its relevance to understanding and controlling many microstructures. The heat transfer equation is derived from the balance of the heat flux through a representative volume element (RVE). Fourier’s law in Equation 2.1 below shows the relation between heat flux and the temperature gradient.

\[
\dot{q} = -k \nabla T
\]  
(Eqn. 2.1)

From Fourier’s law, it can be seen that heat flux, \( \dot{q} \), is related to the temperature gradient, \( \nabla T \), by the thermal conductivity, \( k \), which has the units Watts/m-K. The sign convention of the gradient is that the heat flux will be in the direction of the negative thermal gradient; in other words heat will flow from hot to cold. When the heat flux into a RVE is set equal to the heat flux out of the RVE, Fourier’s First Law heat conduction equation can be derived. The conduction equation is reproduced in Equation 2.2.

\[
\nabla \cdot (k \nabla T) = \frac{\partial (\rho C_p T)}{\partial t}
\]  
(Eqn. 2.2)

Fourier’s Second Law governs the accumulation of heat in the RVE over time and combines the density, specific heat capacity, and thermal conductivity into a single material constant known as the thermal diffusivity, \( \alpha \). This takes the form as seen in Equation 2.3. The gradient is written out in full for clarity.

\[
\frac{\partial T}{\partial t} = \alpha \nabla^2 T = \alpha \left( \frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right)
\]  
(Eqn. 2.3)

The conduction equation can be used to evaluate systems that fall under three basic boundary conditions. The first and simplest of these boundary conditions is the conduction dominant case. In the conduction dominant case, the assumption is that the thermal gradient within the solid is unchanging with time. Under this assumption, the right hand side of the equation can be equated to zero, allowing
for a relatively simple solution to the differential equation. Thermal profiles can be plotted according to Equation 2.4 for the steady-state (conduction dominant) case. Equation 2.5 shows the non-dimensional form.

\[(\text{Eqn. 2.4}) \quad T = \frac{x}{L} (T_2 - T_1) + T_1 \]

\[(\text{Eqn. 2.5}) \quad \frac{T - T_1}{T_2 - T_1} = \frac{x}{L} \]

It should be noted Equation 2.4 is the 1D solution of an infinite-plate geometry where \(T_1\) is the temperature on one side of the plate, \(T_2\) the temperature on the opposite side, and \(L\) is the thickness of the plate. A graphical schematic from Poirier and Geiger's *Transport Phenomena in Materials Processing* is seen in Figure 2.1. The solution will change slightly depending on the object geometry and coordinate system (Cartesian, cylindrical, etc.).

![Graphical schematic](image)

**Fig 2.1:** Steady-state temperature profile of an infinite sheet of thickness \(L\); Poirier and Geiger [pg.282]

The second case of heat transfer to consider is the convection dominant case. In this case, convection is the rate-limiting process. We can expect negligible thermal gradients in the bulk of the material, as the thermal conductivity is a faster means of transferring heat than the convection of heat.

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into an ambient fluid. Equation 2.6 below describes the rate of heat lost from the plate as equal to the rate of heat transfer into the fluid.

\[
\text{(Eqn. 2.6)} \quad -V \rho C_p \frac{dT}{dt} = hA(T - T_f)
\]

The absence of the thermal conductivity in the above equation should be noted. Instead, the coefficient of convection, \( h \), with units of Watts/m\(^2\)-C, is the relevant processing property to the solution. Integration with separation of variables is provides the solution to the differential equation shown in Equation 2.7.

\[
\text{(Eqn. 2.7)} \quad \frac{T - T_f}{T_i - T_f} = \exp\left(-\frac{hA}{\rho C_p V}t\right)
\]

Thermal profiles with respect to \( x \) are not generated by this solution, rather this provides the cooling history of the object. Figure 2.2 shows the evolution of the thermal profile over time for both thick and thin slabs of material. The thin slab sustains little to no thermal gradient, which is often described as Newtonian cooling.

![Figure 2.2: The cooling history within thick and thin slabs as time goes to infinity; no thermal gradients exist in the thin slab; Poirier and Geiger [pg. 288]](image)

An important consideration for any heat transfer problem is whether the object will be subject to the boundary conditions of the conduction dominant case, the convection dominant case, or a combination of the two. In order to accurately characterize the transport phenomenon behavior, a ratio
of convection versus conduction must be examined. This ratio of resistances is known commonly as the Biot number, seen below in Equation 2.8.

\[
\text{Biot Number} = \frac{hL}{k}
\]

(Eqn. 2.8)

A heat transfer problem can be considered conduction dominant if the convection of heat away from the system is rapid (large \(h\)) and the conductivity is slow (small \(k\)), resulting in a large Biot number. The opposite is true for convection dominant systems, where the coefficient of convection is small and the coefficient of thermal conduction is large. Examining these relative magnitudes of heat transfer coefficients, the following basic rules for heat transfer cases are derived: \(\text{Biot} = \frac{hL}{k} > 10\) means the system is conduction dominant, \(\text{Biot} = \frac{hL}{k} < 0.1\) means the system is convection dominant.

While the solutions to the steady-state conduction and Newtonian cooling cases are both relatively simple, the transient case requires a more complex approach. When the Biot number is between 0.1 and 10, the system has a transient solution. We cannot ignore either of the heat transfer modes. The infinite series solution is presented below in Equation 2.9. Like most series solutions, the more terms accounted for in the approximation the more accurate the solution. The first four eigenvalue roots can be seen in Table 2.1, with a more comprehensive table in Carslaw and Jaeger's *Conduction of Heat in Solids*.

\[
\frac{T - T_f}{T_i - T_f} = 2 \sum_{n=1}^{\infty} \frac{\sin \lambda_n L}{\lambda_n L + \sin(\lambda_n L) \cos(\lambda_n L)} \exp(-\lambda_n^2 at) \cos(\lambda_n x)
\]

(Eqn. 2.9)

Table 2.1: First four roots for infinite series solution

<table>
<thead>
<tr>
<th>(\text{Bi})</th>
<th>(k/hL)</th>
<th>(\lambda_1 L)</th>
<th>(\lambda_2 L)</th>
<th>(\lambda_3 L)</th>
<th>(\lambda_4 L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.01</td>
<td>1.5552</td>
<td>4.6658</td>
<td>7.7764</td>
<td>10.8871</td>
</tr>
<tr>
<td>10</td>
<td>0.10</td>
<td>1.4289</td>
<td>4.3058</td>
<td>7.2281</td>
<td>10.2003</td>
</tr>
<tr>
<td>1</td>
<td>1.0</td>
<td>0.8603</td>
<td>3.4256</td>
<td>6.4373</td>
<td>9.5293</td>
</tr>
<tr>
<td>0.1</td>
<td>10.0</td>
<td>0.3111</td>
<td>3.1731</td>
<td>6.2991</td>
<td>9.4354</td>
</tr>
</tbody>
</table>

In addition to the tabulated root charts, solutions for various geometries have been presented in a graphical form. These charts are in terms of the relative temperature as a function of Biot number, Fourier number, and relative position. These dimensionless variables, with the exception of the Biot number which was presented already in Equation 2.8, are presented respectively below in Equations 2.10, 2.11, and 2.12.

(Eqn. 2.10) \( \text{relative temperature } \theta \equiv \frac{T - T_f}{T_i - T_f} \)

(Eqn. 2.11) \( \text{Fourier number } Fo \equiv \frac{at}{L^2} = \frac{kt}{\rho C_p L^2} \)

(Eqn. 2.12) \( \text{relative position } \equiv \frac{x}{L} \)

The earliest of these charts are referred to as the Gurney-Lurie charts. Despite the fact that the temperature response charts were constructed in 1923, they are still in use. The charts relevant to this paper’s investigations have been reproduced in Figure 2.3. The temperature response graphs are critical to evaluating the transient case. They will be important to the heat transfer calculations relevant to the topic of downhill quenching, uphill quenching, relative time scales, and dimension limitations.
Figure 2.3: The temperature response of an infinite cylinder at initial uniform temperature $T_i$ subject to a convective environment at $T_f$; Poirier and Geiger [pg. 295]

Section 2: The Downhill Quench

The cooling rate of a material after heat treatment determines the residual stresses generated. Understanding the relative magnitudes of heat transfer rates upon the downhill quench from hot to cold is the first step in obtaining processing parameters for thermo-mechanical stress relief. In this preliminary analysis of the process, we will look at the heat transfer of water quenched 7000 series aluminum.
First, the appropriate heat transfer mode must be determined. In order to do this, we must evaluate the Biot number given the processing parameters of the water quench. Consider an aluminum cylinder of length 1m and radius 10cm. The thermal conductivity $k$ of 7000 series aluminum is 130 Watts/m-K. Assuming that the water is not subject to forced convection, the coefficient of convection $h$ will be 100 Watts/m$^2$-C. With these material properties, we find that the Biot number is 0.07, which means the heat transfer is convection dominant. Thus, if we are to calculate the heat transfer rate of the water quench, only the convective boundary conditions need to be considered. The solution heat treatment temperature for 7000 series aluminum is approximately 500°C and the temperature of the water is 15°C. The heat transfer rate from the aluminum bar to the water bath can be calculated from Equation 2.13 below.

\[
\text{(Eqn. 2.13) Convective Heat Transfer Rate [Watts] = } hA_{cylinder}(T_f - T_i)\]

For the water quench, a heat transfer rate of about 34 kilowatts can be expected. For the uphill quench to have efficacy, the heat transfer rate back to the cylinder must be of equal or greater magnitude.

**Section 3: The Uphill Quench**

As noted in the previous section, the water quench of a 7000 series aluminum cylinder yields a heat transfer rate of about 34 kilowatts. Knowing this relative magnitude, it is possible to check the feasibility of the uphill quench. An investigation by H.N. Hill and R.S. Barker published in the *Transactions of the American Society of Metals* in 1960 details what they believe to be the most effective method for thermo-mechanical stress relief. Hill and Barker’s hypothesis was that residual stresses can be counteracted by inducing residual stresses of an equal and opposite nature with a sharp positive thermal gradient or uphill quench. In an attempt to improve upon the “deep freeze” stress relief technique, which utilizes dry ice and boiling water, Hill et al maximized the temperature
differential by using liquid nitrogen (LN2) and high velocity steam. Their steam chamber for reheating the cooled aluminum slab is shown in Figure 2.4.

According to Hill et al's method, the aluminum must supercool in a liquid nitrogen bath at -196°C. The cylinder must then immediately be transferred to a high velocity steam chamber. For the sake of simplicity, assume that the steam has a temperature of 100°C. With the previously mentioned processing temperatures and sample geometry, it is now possible to solve for the minimum coefficient of convection necessary. Setting Equation 2.13 equal to the downhill quench heat transfer rate of 34 kilowatts, the coefficient of convection $h$ is found to be 166 Watts/m^2-K. This coefficient of convection $h$ is not completely ridiculous since high velocity steam can have $h$ values on the order of kilowatts/m^2-K. The details of how $h$ is derived experimentally can be found in Groeneveld et al's work with film-
boiling heat transfer. While high velocity steam is convenient and economic, it is not the only option for delivering heat flux to the sample. The uphill quench can be achieved with a variety of quenchants and is not limited to boiling water, brine, molten salt or metal. Extreme care must be taken, however, when considering molten salt or metal reheating strategies, as any condensation of air moisture on the supercooled part during the transfer from the LN2 bath to the reheating bath could result in violent spatter upon uphill quenching.

From these simple calculations, a basic understanding of the necessary reheating specifications can be deduced. It is important to understand the severity of the original downhill quench, the conductivity of the material, and the characteristic thickness of the part being processed because each plays a role in the minimum coefficient of convection required for the uphill quench. The same analysis conducted in this chapter for 7000 series aluminum can be applied to other metals and plastics to determine the optimal reheating strategy.

Section 4: Processing Time Scales and Dimension Limitations

Hill et al's work with uphill quenching sheds some light on processing time scales and effective sample geometry for successful thermo-mechanical stress relief. However, the technique is still ambiguous. Croucher et al's technical paper in the *Journal of ASTM International* offers insight to some of the pitfalls of the uphill quench, but no hard guidelines to sample dimensions or process schedule for optimal stress relief. This is likely because appropriate dimensions and heating schedules depend largely on the material being treated. Both Hill et al and Croucher et al focus their papers on high strength aluminum, a popular alloy for aerospace materials. Dimensional guidelines and heating schedules prescribed in these papers are unique to the aluminum alloy with which they experiment.

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However, it is possible, with simple Biot number and Fourier diagram analysis, to determine appropriate dimensions and heat schedules for various materials.

For example, let us analyze the dimension limits for an infinite steel plate. Assume that $h$ for water is 100 Watts/m$^2$-K and $k$ in steel is 40 Watts/m-K. Remember that in order to obtain stress relief thermo-mechanically, it is imperative to induce thermal gradients of equal or greater magnitude than that of the downhill quench. A steel plate with characteristic length of 1mm has a Biot number of 0.0025, indicating Newtonian heating. An even larger thickness of 10cm further pushes the system into the transient regime with a Biot number of 0.25. Any radius larger than 4 meters will fall into the steady-state regime. Because we are looking to obtain the sharpest thermal gradients possible, an ideal length scale for uphill quenching is the length at which the Biot number is equal to 1. Table 2.2 details the ideal length scales for Al 6061-T6, Type 501 stainless steel, and PMMA for the three different heat transfer cases (Newtonian, transient, steady-state). From the table it is seen that ideal length scales for aluminum, steel, and PMMA all vary quite significantly with differences in the thermal conductivity.

Thermal conductivity of different materials will affect the heating schedule in much the same way. In Figure 2.5, Hill et al's thermal profiles versus time are plotted. According to their results, the 2 inch thick aluminum sheets took about 20 seconds to reach a uniform temperature. It is expected that because of the differences in density, heat capacity, and conductivity, time-scales for establishing uniform temperature profiles will be different.

**Table 2.2:** Length-scale calculations based on Biot number analysis under water quench conditions (ie $h=100$ Watts/m$^2$-K)

<table>
<thead>
<tr>
<th>Material</th>
<th>Steady-State Case (Bi=0.1)</th>
<th>Transient Case (Bi=1)</th>
<th>Newtonian Case (Bi=0.1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al 6061-T6</td>
<td>13 meters</td>
<td>1.3 meters</td>
<td>13 centimeters</td>
</tr>
<tr>
<td>Type 501 Stainless Steel</td>
<td>4 meters</td>
<td>40 centimeters</td>
<td>4 centimeters</td>
</tr>
<tr>
<td>PMMA</td>
<td>2 centimeters</td>
<td>2 millimeters</td>
<td>0.2 millimeters</td>
</tr>
</tbody>
</table>
Figure 2.5: Temperature profiles of aluminum slabs during the uphill quench;
Chapter 3: Experimental Methods

Section 1: Material Selection and Equipment List

As mentioned in Chapter 2, thermo-mechanical stress relief was originally applied and analyzed in 6000 and 7000 series aluminum alloys by Alcoa. Aluminum alloy, in particular 6061-T6, has very high thermal conductivity among all commercial alloys, on the order of 170 Watts/m-K. From Biot number and Fourier analysis, the ideal length scales to obtain a transient condition for heat transfer in these highly conductive alloys is on the order of a meter. Additionally, 6061-T6 aluminum has a solution heat-treatment temperature on the order of 500 C. In order to make experimental evaluation of the uphill quench more convenient and well controlled, it was decided that a more appropriate material analog to test would be poly-methyl methacrylate (PMMA), more commonly known as acrylic. According to Table 2.2 in Chapter 2, the characteristic thickness to obtain a transient heat transfer problem in PMMA is on the order of 2 mm.

PMMA has many physical properties that make it an attractive candidate for evaluation of thermo-mechanical stress relief. In the theory, it was discussed that thermo-mechanical stress relief relies on the concept of inducing residual stresses of equal and opposite nature to those which result from the downhill quench during solutionizing. This idea demands that thermal gradients, and thus differences in thermal expansion, must be generated in the sample upon uphill quenching. Acrylic is ideal as a material because of its low thermal conductivity (circa 0.2 Watts/m-K). With a low thermal conductivity, the thickness dimensions of an acrylic sample coupon required to see gradients is reduced to the order of millimeters. Additionally, acrylic can be annealed at a much lower temperature, around 95 C. This fact compared to the 500 C annealing temperature of 6061-T6 aluminum eliminates the need for large, bulky aluminum coupons and high power furnaces.

The equipment utilized in the uphill quench evaluation is as follows:
- PMMA 3mm thick strips, 1”x8”
- Pyrex dish for water quenching
- Foodsaver Vacuum Sealing System
- Breville Toaster Oven with digital control interface
- Poly-Science Anti-griddle
- 6061 Al block (for conductive cooling on anti-griddle)
- Heat plate
- Omega AWG 36, k-type thermocouples, PicoLog Recorder

Section 2: Thermo-mechanical Stress Relief Processing Overview

The residual stress relief technique known as the uphill quench follows a progression of the following thermal steps: 1.) High Temperature Annealing, 2.) Downhill Quench, 3.) Low Temperature Annealing, and 4.) Uphill Quench. Each of these steps will be covered in more detail in the rest of the chapter. Figure 3.1 is an illustration of coupon preparation and thermocouple placement. PMMA strips of 3mm thickness and of dimensions 1”x8” are sandwiched flat together, with a thermocouple contained in a groove in between (at the center) the two strips and at both surfaces. Figure 3.1 shows the thermocouple arrangement in the vacuum sealed PMMA strips. The thermocouples and PMMA strips are then vacuum sealed together using the FoodSaver system. The intention behind vacuum sealing the coupons and thermocouples together is two-fold: first to hold the coupons flat against each other during thermal processing, and second to create an interface at the center, in between the PMMA, strips that will change temperature much slower than the surface, enabling necessary thermal gradients.
**Figure 3.1:** The fine gage thermocouples are placed in the vacuum sealed bag and held to the surface and center of the PMMA strips throughout thermal processing.

The thermal gradients are monitored with the thermocouples that are placed at the surface and in between the PMMA strips. Thermal gradients from high temperature annealing to downhill quenching in water are compared to the thermal gradients from the low temperature annealing on the anti-griddle to the boiling water quench. The difference in temperature at the surface and the center of the coupons will provide quantitative evidence on the magnitude of thermal gradients, and thus relative magnitude of residual stress relief. In addition, the deflection of the PMMA strips provides another, although less precise, tangible observable for evidence of residual stresses in the material. The thermal
data is recorded using a thermocouple Picoscope and the software PicoLog. Samples are collected at a rate on the order of milliseconds to capture the rapid quench behavior.

In addition to logging the thermal data from fine gage thermocouples, deflection of the PMMA strips is measured. The deflection measured following the downhill quench, and subsequent uphill quench are to be used as a surrogate means of the degree of residual stress relief. Because the internal compressive and tensile stresses cause yielding in thin sections of PMMA, we can measure the yield and correlate it to the residual stresses inside the material. The comparison of the yielding after the downhill quench and the yielding after the uphill quench gives a demonstration of stress relief efficacy. Diminished deflection following the uphill quench offers concrete physical evidence that the residual stresses have been reduced. Although not as sophisticated as XRD analysis, residual stress analysis via comparison deflection before and after the process will give a macroscopic evaluation of stress relief.

Section 3: Process Details

The first step of the processing technique is to bring the vacuum sealed PMMA strips to a temperature sufficient to thermally alleviate residual stresses in the material. The annealing temperature of acrylic is 95 C. In order to ensure a temperature profile absent of gradients in the coupons, the PMMA is annealed for 10 minutes at 95 C in the Breville oven. The convection settings ensure a uniform heating environment.

Following the annealing just below the softening temperature of acrylic, the sample is immediately quenched in cold water at a temperature around 15 C. Fourier number analysis of the downhill quench conditions require that the sample be submerged in the water on the order of 15 seconds to obtain a temperature differential of 70 degrees between the center and surface of the coupon. Specific calculations for the time scale of the downhill quench can be found in Appendix A.
Following the 15 second water quench, the coupon is dried and immediately transferred to the anti-griddle for low temperature annealing.

The low temperature annealing step of the process is meant to bring the PMMA strips to the lowest possible temperature. As mentioned earlier in Section 2, a 6061 aluminum block is left on the anti-griddle to stabilize at -40 C. The PMMA sample is placed on the anti-griddle, underneath the conducting block heat sink. For 10 minutes, the sample is cooled to -40 C. Once a uniform temperature profile is established, the sample is removed from the anti-griddle and immediately uphill quenched in a Pyrex dish filled with boiling water at 100 C. The heat transfer calculations using Fourier number analysis dictate that the sample be submerged for 30 seconds, for a temperature gradient of 100 degrees in magnitude (center of coupon ca. 0 C, surface ca. 100 C). Following the full process, the PMMA strips are removed from the vacuum sealing and the deflection of the sample coupons is measured. Thermal data is also scrutinized to determine the relative efficacy of the thermo-mechanical stress relief technique.

Section 4: Finite Element Analysis Simulation

In addition to the physical experiment, a Finite Element Analysis (FEA) simulation is conducted to offer a qualitative look at the behavior of materials through the processing technique. A simple 2D-geometry was selected to represent a 6061-T6 aluminum block of dimensions 8"x4". The FEA simulation utilizes 3 surface film conditions, with coefficient of convection, $h$, modified to encapsulate the processing environments (ie convection oven, water quench, etc.) at each step in the process. Coupled thermal-displacement analysis and stress analysis was conducted by Simula’s Abaqus. The simulation provides a tool to shed more light on the heat transfer behavior on the original material proposed in Hill et al’s paper from Alcoa. From this simulation, important verification of experimental proceedings will be produced.
Figure 4.1: The thermal data characterizing the downhill quench from solution treatment temperature for PMMA into cold water is seen above. The PMMA coupon was quenched for 30 seconds.

Thermal data were collected using 3 different fine gage (36 AWG) type-k thermocouples from Omega. At a sampling rate on the order of milliseconds, thermal gradients between the surface of the PMMA sample coupons were captured. Figure 4.1 represents the data collected during the downhill quench step of the thermo-mechanical stress relief process. Two thermocouples (Surface Temperature 1 and 2) were responsible for gathering temperatures at the two surfaces of the PMMA sample. Both were necessary to get an understanding of the realistic uniform heat flux to the system. The third thermocouple, located at the center of the PMMA coupon, provided crucial thermal data for determining the magnitude of thermal gradients inside the sample. From the two surface
thermocouples, it can be seen that the sample had reached virtual uniform temperature in the solution heat treatment, and saw minor thermal differentials upon quenching. This may be caused by human error in the quenching technique, such as bottom side of the PMMA sample being submerged in cold water before the top side.

The maximum thermal gradient upon the downhill quench was found to be 53.96°C. This resulted in a deflection between the two strips of PMMA of 2.36 millimeters. The generation of a significant thermal gradient in addition to the resultant deflection is evidence of residual stresses. Although not quantified, the warping of PMMA strip geometry is indicative of residual stresses that cause yielding in the material.

![Uphill Quench Temperature vs. Time](image)

**Figure 4.2:** The above thermal data captures the uphill quench from -40°C to boiling water at 100°C for the center and both surfaces of the PMMA sample. The sample was quenched in boiling water for 30 seconds.
The same sampling techniques were utilized for the collection of thermal data in the uphill quench step of the process. Figure 4.2 illustrates the temperature versus relative time for the uphill quench. The PMMA sample was left to stabilize at -40°C on an anti-griddle with a conducting aluminum block serving as a heat sink for the top side of the sample. From the thermocouple data, it is seen that a uniform temperature was not completely obtained in the low temperature annealing step. This is likely caused by the unequal heat sink potential between the stage of the anti-griddle and the conducting block. The non-uniformity of low temperature annealing could be a source of inefficacy when considering the goal of thermo-mechanical stress relief. The relief of residual stresses comes from the ability to induce stresses of an equal and opposite nature. If there is an unequal heat flux on symmetric boundaries, stress relief will be less effective. Despite the minor temperature differential before the uphill quench, the deflection between the PMMA strips of the sample was reduced to 0.87 mm.

Table 4.1 shows the maximum temperature differentials obtained through each step of the thermo-mechanical stress relief technique. The uphill quench itself was capable of reducing the deflection of the PMMA strips by 37 percent. This is indicative of relief of the residual stresses that originally cause yielding, and by extension, deflection of the acrylic.

<table>
<thead>
<tr>
<th>Process Step</th>
<th>Maximum Delta T [°C]</th>
<th>Deflection between Coupons [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Downhill Quench</td>
<td>53.96</td>
<td>2.36</td>
</tr>
<tr>
<td>Uphill Quench</td>
<td>86.24</td>
<td>0.87</td>
</tr>
<tr>
<td>% Stress Relief:</td>
<td></td>
<td>37%</td>
</tr>
</tbody>
</table>

**Table 4.1**: The maximum thermal gradient and measured deflection for both the downhill and uphill quench. Percentage stress relief is deduced from the reduced deflection between sample coupons.
Section 2: Finite Element Model

Figure 4.3: The principle stress contours (left) and the nodal temperature contours (right) from the FEA simulation of 6061-T6 aluminum alloy for the downhill quench. Max and min observables are noted in the legend.

The finite element software Abaqus provides a cross-sectional view of the residual stresses and thermal gradients due to thermo-mechanical stress relief in 6061-T6 commercial aluminum alloy. By incorporating the mechanical and thermal properties of 6000 series aluminum into the simulation and analyzing the field output, we see verification of stress behavior inside the block of material due to thermal gradients. Figure 4.3 above shows the stress distribution and thermal gradients from the downhill quench. The downhill quench is characterized by a film coefficient of 100 Watts/meter-K, and water quench conditions to an ambient temperature of 15°C from 500°C (6000 series solution treatment temperature). From the simulation contours, it can be seen that tensile stresses on the order of thousands of Pascals build up in the middle of the block of material, and compressive stresses on the order of tens of thousands Pascals from the thermal contraction are seen at the edge of the block.
Attention to the legend of the right FEA contour shows that a thermal gradient of only 24°C was sustained during the quench. This is not surprising, as 6061-T6 aluminum has the comparatively high thermal conductivity for commercial alloys, making thermal gradients difficult to sustain.

Figure 4.4: The maximum principle stress contours (left) and the nodal temperature contours (right) for the uphill quench step of thermo-mechanical stress relief in 6061-T6 aluminum alloy. Max and min observables are noted in the legend.

Upon analysis of the uphill quench, clear differences can be noticed in contrast to Figure 4.3. The first of these differences is the replacement of tensile stresses with compressive stresses in the middle of the system. The second difference, which captures the essence of the uphill quench, is seen in the right part of Figure 4.4 concerning the nodal temperatures. The opposite nature of both the contour plot of max principle stress and nodal temperature indicates the theoretical possibility that a thermo-mechanical stress relief technique is plausible. Thermal gradients induce residual stresses on the basis of the thermal contraction or expansion that the system experiences. Both the thermal gradients and
resultant residual stresses are seen in Figures 4.3 and 4.4. The magnitude of the stresses and gradients is realistic, and the contours of these observables verify the stress relief seen in physical experiment.
Chapter 5: Conclusions and Future Work

Section 1: Relevant Conclusions

The thermo-mechanical stress relief technique known as the uphill quench can be used effectively with proper knowledge of the heat transfer and processing parameters involved. Before any physical experiments were conducted, hand calculations of the heat transfer phenomenon defined the limitations to realistic application of the uphill quench. After checking the feasibility of the technique that Croucher et al suggests (i.e., liquid nitrogen bath to high-velocity steam), it was found that the amount of heat flux required to generate thermal gradients in highly conductive aluminum alloy was reasonable assuming a convection coefficient on the order of thousands of Watts per meter Kelvin. Croucher et al., however, neglects to define parameters on the relative length scales in which this technique will be effective, as well as the scope of its application.

Simple Biot number analysis of 6000 series aluminum, steel, and poly-methyl methacrylate allow for the deduction of relevant length scales under which the uphill quench will be effective. It was found that in order to sustain sharp thermal gradients in aluminum with an uphill quench, the aluminum part being treated must have a characteristic thickness of around 1 meter. Steel, because of its lower thermal conductivity, must have a characteristic length around 40 centimeters. PMMA was found to have a characteristic length on the order of millimeters for the uphill quench to be effective. Because of PMMA's convenient thermal properties and dimensions, the physical experiment was carried out using this material.

PMMA strips were utilized to understand the details of thermo-mechanical stress relief. The experiment proved that residual stresses are a result of thermal gradients in the material. The thermal gradients cause differences in thermal expansion and material mobility, manifesting in residual stresses. In the event that the residual stresses are of larger magnitude than the yield strength of the material, a deflection is observed. In the experiment with PMMA, it was found that upon downhill quenching from
95 C in a water bath produces a deflection of 2.36 mm between the sample coupons. Thermal data logging of the same step of the process shows that a thermal gradient of 54 degrees C is sufficient to produce the observed deflection.

Despite the severity of the downhill quench, the uphill quench from the low temperature annealing step reduced deflection of the PMMA strips by 37 percent. The reduction of deflection from 2.36 mm to 0.87 mm is evidence that a sharp positive thermal gradient does induce residual stresses of an opposite nature to balance the tensile and compressive stresses inside the material. The maximum thermal gradient logged during the uphill quench is 86 degrees C.

These findings in the physical experiment are verified by the finite element analysis simulation. Although the material simulated was intended to mirror the thermo-physical properties of 6000 series aluminum, the observed stresses and thermal gradients support the thermo-mechanical stress relief theory. The experiment using PMMA was able to correlate thermal gradients to residual stresses and resultant deflection of the material. What is seen in the FEA simulation is that the thermal processing of the downhill and subsequent uphill quench creates thermal gradients that are responsible for internal stresses. Moreover, the uphill quench step does, in fact, induce residual stresses of an equal and opposite magnitude if the coefficient of convection allows for sufficient transport of heat. Because the simulation allows us to prescribe the surface film coefficient, fine control over the process was established. It is concluded that under the proper processing conditions, it is possible to utilize an uphill quench as a form of effective thermo-mechanical stress relief.

**Section 2: Sources of Error and Future Work**

The physical experiment exploring the uphill quench technique has a few sources of error. Upon observing the thermal log of the downhill and uphill quench data, it can be seen that there are minor temperature differentials between the two surface thermocouples. These differences in the
temperature on the surfaces of the PMMA are indicative of unequal heat flux into the system. If this is the case, then the thermal gradients and induced residual stresses from either side are not equal. This compromises the efficacy in the thermo-mechanical stress relief.

Another difficult aspect of the physical experiment to control is the precise temperature of the anti-griddle. Unlike the Breville oven utilized in the high temperature annealing step, the anti-griddle has no digital display or control interface. The anti-griddle is simply switched on, and a conducting block is laid on the stage to cool down to roughly the same temperature as the cold stage. In the thermal log of the uphill quench, it can be seen that the surface thermocouples show a thermal gradient through the sample. At this point in the technique, the sample is intended to have a uniform temperature profile at around -40°C. Because the uniformity is never met, it is expected that the uphill quench again loses efficacy due to this lack of control.

In order to move the exploration of thermo-mechanical stress relief forward, it is recommended that the length scales for efficacy be evaluated with physical experiment. Because of the lack of time and ample resources, it was not possible to conduct the physical experiment with a 1 meter thick section of 6000 series aluminum or a 40 centimeter thick section of steel.

Additionally, it is important to explore the effect of various quenchants. The effectiveness of a downhill water quench will be different from an oil quench, just as liquid nitrogen to high velocity steam quench will be different from a dry ice to boiling water quench. By comparing the thermal gradients of these different quench techniques, a more effective form of thermo-mechanical stress relief can be analyzed. Finding the optimal thermal gradient magnitude for the uphill quench of a material will shed light on details of the process that can be specified and controlled precisely for steel, aluminum, and other materials.
Chapter 6: Works Cited


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