

An Investigation of Incipient Melting During Solution Heat Treatment of 7050
Aluminum Forgings and Its Effect on Corrosion Properties

A Senior Project
presented to
the Faculty of the Materials Engineering Department
California Polytechnic State University, San Luis Obispo

In Partial Fulfillment
of the Requirements for the Degree
Bachelor of Science In Materials Engineering

by

Thomas Kilkenny

June 2015

© 2015 Thomas Kilkenny

Table of Contents

I. Abstract	2
II. Acknowledgements	3
III. List of Figures	3
IV. List of Tables	5
V. Introduction	5
a. Problem Statement	5
b. Background	6
i. <i>Aluminum Alloys</i>	6
ii. <i>Forging Processes and Equipment</i>	7
iii. <i>Forging Aluminum</i>	10
iv. <i>Age Hardening</i>	11
c. Overheating and Incipient Melting in Aluminum Alloys	14
i. <i>Microstructure</i>	14
ii. <i>Effect on Mechanical Properties and Corrosion Resistance</i>	16
VI. Experimental Procedure	18
a. Safety	18
b. SS-DTA.....	19
c. Solution Heat Treatment Trials.....	19
d. Corrosion Tests	19
VII. Results	20
a. SS-DTA Data	20
b. Solution Heat Treatments	22
c. Pitting Corrosion.....	28
d. Intergranular Corrosion.....	30
VIII. Discussion	32
IX. Conclusions	34
X. References	35

I. Abstract

7050 alloy (6.2Zn-2.3Mg-2.3Cu-0.12Zr) is a structural aluminum alloy commonly used in the aerospace industry. Its rich chemistry makes it susceptible to incipient melting. Incipient melting is a phenomenon where areas with high local alloy content melt before the rest of the material. This can cause a degradation of mechanical and corrosion properties. A six by four factor solution heat treatment trial was conducted. Six solution heat treatment temperatures, 890-940°F in 10°F increments, and four times, 1, 2, 4, and 6 hours, were used. Incipient melting was observed in the 910°F, 6 hour solution heat treatment and all 920-940°F solution heat treatments. Solution heat treatment temperature was shown to be the more important factor. Little microstructural variance was seen among the different solution heat treatment times at a single temperature. Literature suggested that incipient melting would pit preferentially and increase susceptibility to intergranular attack. All 4 hour solution heat treatments were tested for pitting corrosion susceptibility by immersion of metallographic samples in a 0.1M NaCl solution. Samples with incipient melting present did not show preferential pitting. The same samples were tested for intergranular attack susceptibility according to AMS 2772 and ASTM G110. Samples with incipient melting showed no increase in sensitivity to intergranular attack. Neither of the corrosion properties tested degraded in the expected manner.

Key Words: materials engineering, 7050 aluminum, solution heat treatment, incipient melting, pitting corrosion, intergranular attack

II. Acknowledgements

I would like to deeply thank everyone who went above and beyond to help me complete this project. Scot Forge Company provided abundant resources in the form of equipment, labs, furnaces, and test material. Kyle Rackers, Gabe Lucas, and Dan Boley of Scot Forge provided resources, advice, time, knowledge, and effort in helping this project succeed. The California Poly Technic State University Materials Engineering Department provided labs and resources. Blair London of the Materials Engineering Department went above and beyond the duties of a project advisor to make sure this project was completed to the utmost standard. Thank all of you so much for making this project a success.

III. List of Figures

Figure 1 – Closed-die forging schematic. Closed die forgings often only require a single stroke to obtain a near net shape part. Because of this, closed-die forged parts usually require much more force to form than an equivalent sized open die part. ^[5]	7
Figure 2 – Open-die forging schematic. Open-die forgings are formed with many strokes, one section of the part at a time. Because of this, open-die forgings do not require as much force to form as closed-die parts. ^[7]	8
Figure 3 – A blacksmith forges steel his Nazel 3B power hammer. Even though it is an energy restricted machine, the amount of force can be controlled by the position of the work piece within the stroke length. ^[8]	9
Figure 4 – A Scot Forge crew forges a steel journal down to size on a 3000 ton hydraulic press. A crew of four, including a forge operator, blacksmith, forklift operator, and manipulator operator work on each press at any given time. ^[9]	10
Figure 5 – Flow stresses of commonly forged aluminum alloys as a function of strain percentage, compared with that of 1025 steel. ^[10]	11
Figure 6– Temperature ranges for solution heat treatment, annealing, and aging for the Al-Cu system. The solution heat treatment temperature is kept below the eutectic temperature to avoid eutectic melting. ^[11]	12
Figure 7– Tensile strength aging curve for 6061 sheet. ^[11]	14
Figure 8 – A 7050 forging, W-tempered at 930°F for 2 hours. Al ₂ CuMg (dark gray) is in the process of forming into a rosette (light gray, lacey structure). As-polished condition.....	15

Figure 9- Electron Backscatter Diffraction (EBSD) and Energy Dispersive Spectroscopy (EDS) of grain boundary melting in 7150 plate solution heat treated at 959°F for 1 hour. As expected from the chemical composition, this region is rich in Cu, Mg, and Zn. ^[13] 16

Figure 10– Mechanical properties of various solution heat treatment temperatures (440-490°C) for 7050, aged at 121°C for 6 hours then 163°C for 12 hours. The 490°C solution heat treatment is affected by overheating. ^[14] 17

Figure 11 – Optical Profilometry of 7150 plate solution heat treated for 959°F for 1 hour and immersed in 0.1M NaCl solution for 12 hours. The 2-D profile shows that some pits, the 3-D profile shows intergranular attack along melted grain boundaries. ^[13] 18

Figure 12 - SS-DTA data collected at 5Hz from a 1 in cube of 7050 in an 1100°F furnace. 20

Figure 13 – Analysis of the upper recalescence of the SS-DTA data provided in Figure 12. 21

Figure 14– 1100°F SS-DTA sample. Obvious overheating, rosettes, and grain boundary melting present. Etched with 0.5% HF. 22

Figure 15– 890°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. No signs of overheating, fine distribution of second phase constituents begin to soltuionize or agglomerate as time increases. Etched with 0.5% HF. 23

Figure 16 – 900°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. No signs of overheating, more changes in phase morphology and agglomeration as time increases. Etched with 0.5% HF. 24

Figure 17 – 910°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Continued agglomeration, some possible rosette formation seen in the color and shape change of the Al₂CuMg in the 1 and 6 hour samples. Etched with 0.5% HF. 25

Figure 18 – 920°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Clear rosettes, partially formed rosettes, and grain boundary melting in all samples. Etched with 0.5% HF. 26

Figure 19 – 930°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Rosettes and grain boundary melting present in all samples, similar to 920°F solution heat treatments. Etched with 0.5% HF. 27

Figure 20 – 940°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Rosettes and grain boundary melting present in all samples, intergranular fracture due to incipient melting across 4 and 6 hour samples. Etched with 0.5% HF. 28

Figure 21 – 4 hour solution heat treatments at (a) 890°F, (b) 900°F, (c) 910°F, (d) 920°F, (e) 930°F, and (f) 940°F. Immersed in a 0.1M NaCl solution for 2.5 hours after metallographic preparation. Majority of pitting around MgZn₂ and Mg₂Si precipitates, rosettes and grain boundary melting found un-pitted. 29

Figure 22 – 920°F solution heat treatment for 4 hours. Immersed in a 0.1M NaCl solution for 2.5 hours after metallographic preparation. An intact rosette near other pits. 30

Figure 23– 4 hour solution heat treatments at (a) 890°F, (b) 900°F, (c) 910°F, (d) 920°F, (e) 930°F, and (f) 940°F. Immersed in a H₂O₂ and NaCl solution for 6 hours and then prepared metallographically. Small varied amounts of pitting at the surfaces but no signs of intergranular corrosion. 31

Figure 24 – SS-DTA sample, reached 1100°F for about one hour. Immersed in a H₂O₂ and NaCl solution for 6 hours and then prepared metallographically. Slight intergranular corrosion at surface, extensive eutectic network throughout sample. 32

IV. List of Tables

Table I – Wrought Aluminum Alloy Designation ^[3]6

Table II – Temper Designations of Aluminum Alloys ^[11]13

Table III – Relationship of 2nd Phase Constituents to the Matrix in 7075 ^[17]34

V. Introduction

a. Problem Statement

Aluminum alloy 7050 (6.2Zn-2.3Mg-2.3Cu-0.12Zr) is a prized structural material in the aerospace industry for its high strength, as well as high stress corrosion cracking resistance, fracture toughness, and fatigue resistance ^[1]. However; this alloy produces particularly precarious processing parameters. The solution heat treatment temperature of this alloy is 890°F and the theoretical incipient melting temperature is 910°F ^[1]. A solution heat treatment temperature below 890°F will not sufficiently solutionize the structure while a temperature above 910°F can cause eutectic melting, which is manifested in the formation of rosettes in the microstructure and grain boundary melting. Because of this narrow temperature range, a small error in furnace calibration or heat treatment processes can cause overheating and therefore

incipient melting in a part; an unacceptable condition due to its degradation of mechanical properties and corrosion resistance.

This project will address this problem by furthering the current understanding of rosette formation and grain boundary melting through solution heat treatment trials at various times and temperatures that simulate practical heat treatment errors. Samples from these solution heat treatment trials will be examined metallographically to study the development incipient melting, and then corrosion tests will be conducted to determine the effect of each stage of overheating on corrosion resistance.

b. Background

i. *Aluminum Alloys*

Aluminum is second most abundant metallic element on earth and occurs naturally in its most stable oxide form, alumina (Al₂O₃)^[2]. Alumina is refined by the Hall-Heroult method, which involves dissolving alumina in a cryolite and fluoride salt bath then electrolyzing out pure aluminum. The aluminum is then vacuumed or siphoned into crucibles and send to be cast into fabricating ingots. Due to the high energy and monetary cost of this process, aluminum is nearly always recycled at the end of its life.

Since their advent at the end of the 19th century, aluminum alloys have been sought out for their high specific strength and stiffness, corrosion resistance, as well as electrical and thermal conductivity. It has been the preferred structural material in the aerospace industry since the 1930s, and continues to be today. Aluminum is split into two major categories: cast and wrought compositions. Wrought compositions are represented by four digits while cast are represented by three. In wrought compositions, the first number of the designation denotes the major alloying element, while the next three denote a specific alloy composition range (Table I).

Table I – Wrought Aluminum Alloy Designation^[3]

Four-Digit Series	Main Alloying Element
1xxx	None (99.00% Pure Al minimum)
2xxx	Copper
3xxx	Manganese
4xxx	Silicon
5xxx	Magnesium
6xxx	Magnesium, Silicon
7xxx	Zinc (usually Mg also)
8xxx	Others (e.g. Lithium)
9xxx	Reserved for future use

Wrought aluminum compositions are commonly extruded, rolled, drawn, or forged, depending on the desired geometry. Here forging will be the focus.

ii. Forging Processes and Equipment

Forging is a bulk forming process, meaning that the work piece's shape is significantly altered by large amounts of plastic deformation. Forging methods include die forging, upsetting, orbital forging, spin forging, mandrel forging, ring rolling, and more. Here we will focus on die forging, meaning that the work piece is shaped between tools or dies. Die forging can be divided into two categories, closed die forging and open die forging. In closed die forging the hot metal is shaped within the walls of the dies, closing completely over the work piece and constraining flow of the metal (Figure 1). Closed die forging has heavy reductions and can produce complex, asymmetrical, near net shape parts. These parts can weigh up to several tons, though smaller parts are much more common. ^[4] Due to the fact that each part requires a unique die, closed die forging is more optimal for high volume parts and may have long lead times.

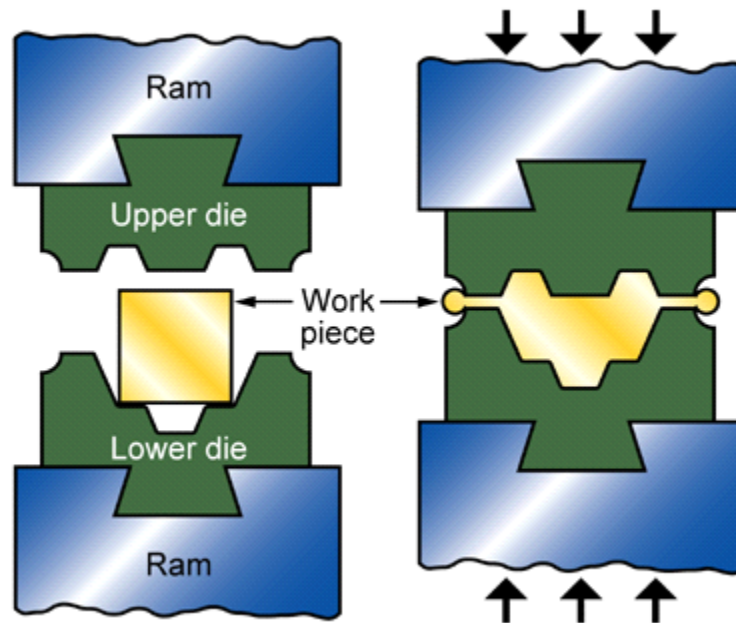


Figure 1 – Closed-die forging schematic. Closed die forgings often only require a single stroke to obtain a near net shape part. Because of this, closed-die forged parts usually require much more force to form than an equivalent sized open die part. ^[5]

In open die forging the hot metal is formed with discontinuous, unconstrained flow, often between flat dies (Figure 2). Parts made by open die forging are geometrically simpler than that of closed die forging; however, open die forging can manufacture parts weighing up to 600 tons.

[6] Open die forging is more ideal for low volume parts and has much shorter lead times than closed die forging for a new part design. Both open and closed die forgings are commonly produced on hammers and presses.

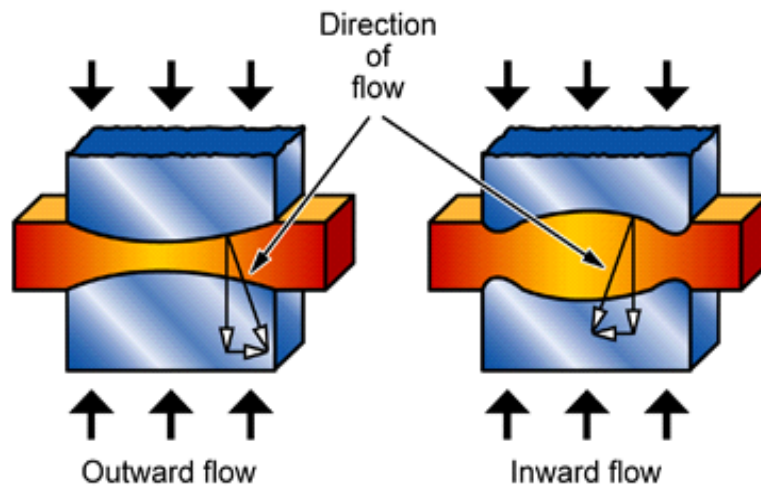


Figure 2 – Open-die forging schematic. Open-die forgings are formed with many strokes, one section of the part at a time. Because of this, open-die forgings do not require as much force to form as closed-die parts. [7]

Forging hammers are characterized by multiple fast impacts on a work piece between contoured dies (Figure 3). The work piece is placed on the lower die, and the ram moves down to deform it. Hammers are energy restricted machines, meaning that the deformation caused by each stroke is limited by the kinetic energy of the ram. Careful control of stroke length, force, and speed must be exercised when forging aluminum due to its strain rate sensitivity. [6]

Hammers are slowly being replaced by forging presses, but are still used for some low volume production of open and closed die aluminum forgings.



Figure 3 – A blacksmith forges steel on his Nazel 3B power hammer. Even though it is an energy restricted machine, the amount of force can be controlled by the position of the work piece within the stroke length. ^[8]

Forging presses are characterized by slower, controlled impacts between various die shapes (Figure 4). Similar to the operation of hammers, the work piece is placed on the lower die while the upper die moves down to deform it. Presses have a variable stroke length and velocity, allowing for close control of metal flow velocities and thus final part dimensions. They are load restricted machines, meaning that deformation of each stroke is limited by the maximum force supplied by the hydraulics or flywheel, depending on the type of press. Presses are well suited to open and closed die forging of aluminum.



Figure 4 – A Scot Forge crew forges a steel journal down to size on a 3000 ton hydraulic press. A crew of four, including a forge operator, blacksmith, forklift operator, and manipulator operator work on each press at any given time. ^[9]

iii. Forging Aluminum

The general processes of producing open die aluminum forgings and closed die aluminum forgings are fairly similar. First, a work piece is cut from cast or wrought stock, usually by a circular saw or band saw with a carbide-tipped blade. The work piece is then deburred before being preheated for forging. Aluminum can be heated with electric furnaces, fully or semi-muffled gas furnaces, oil furnaces, fluidized-bed furnaces, as well as induction and resistance heating units. Gas furnaces are the most common. Since forging temperature ranges for aluminum alloys are narrow, furnace temperature cannot have a greater variance than $\pm 10^{\circ}\text{F}$. Dies are also preheated to help maintain optimal forging temperatures for longer. Forgings that require more die time are started at the upper end of their respective forging temperature range and finished as quickly as possible in an attempt to start and finish the part within the forging temperature range without reheating. For a given forging, force requirements can vary based on forging process, work piece and die temperatures, chemical composition, and strain rate sensitivity. Aluminum can be more difficult to forge than steel, requiring higher flow stresses at forging temperature, especially for high strength alloys such as 7xxx series (Figure 5). After forging, parts are often trimmed, additionally formed, and cleaned. Trimming involves sawing, punching, or machining away the flash from a closed die forging. Forming is further shaping of

the part closer to final tolerance by hot, warm, or cold forging. Cleaning often involves submersion in nitric acid baths and rinsing with water to remove smut from forging. After cleaning, forged parts are ready to undergo heat treatment.

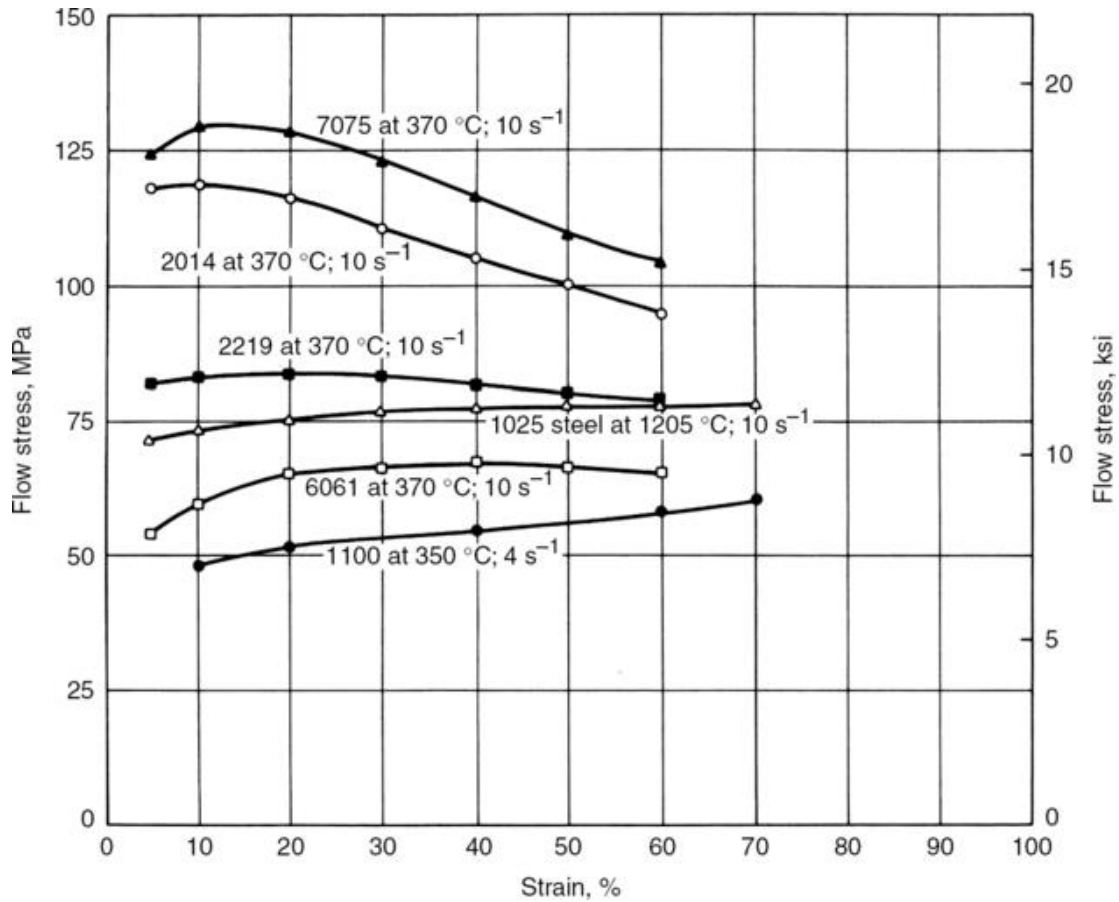


Figure 5 – Flow stresses of commonly forged aluminum alloys as a function of strain percentage, compared with that of 1025 steel. ^[10]

iv. Age Hardening

Wrought aluminum alloys can be divided into two broad categories: heat treatable and non-heat treatable. 1xxx, 3xxx, and 5xxx series are non-heat treatable, meaning that their mechanical properties do not directly benefit from heat treatment. 2xxx, 4xxx, 6xxx, and 7xxx series are heat treatable, which means that their strength can be increased through precipitation strengthening. Precipitation strengthening in aluminum alloys has three basic steps: solution heat treating, quenching, and aging.

The first step of age hardening is to make a super saturated solid solution by heat treatment. This is attained by heating the aluminum alloy through the solvus temperature in an attempt to put as much of the precipitation hardening elements into solution as possible (Figure

6). The part must then be held at this temperature for sufficient time, which depends on part size, geometry, and alloy composition. In addition to solutionizing the structure, solution heat treatment also increases the vacancy concentration, providing favorable sites for heterogeneous nucleation of precipitates. Solution heat treatment temperatures fall into a narrow range. Temperatures that are too low risk not fully solutionizing the structure and thus limiting the potential of age hardening, while temperatures that are too high risk incipient eutectic melting due to overheating, which can degrade mechanical and corrosion properties.

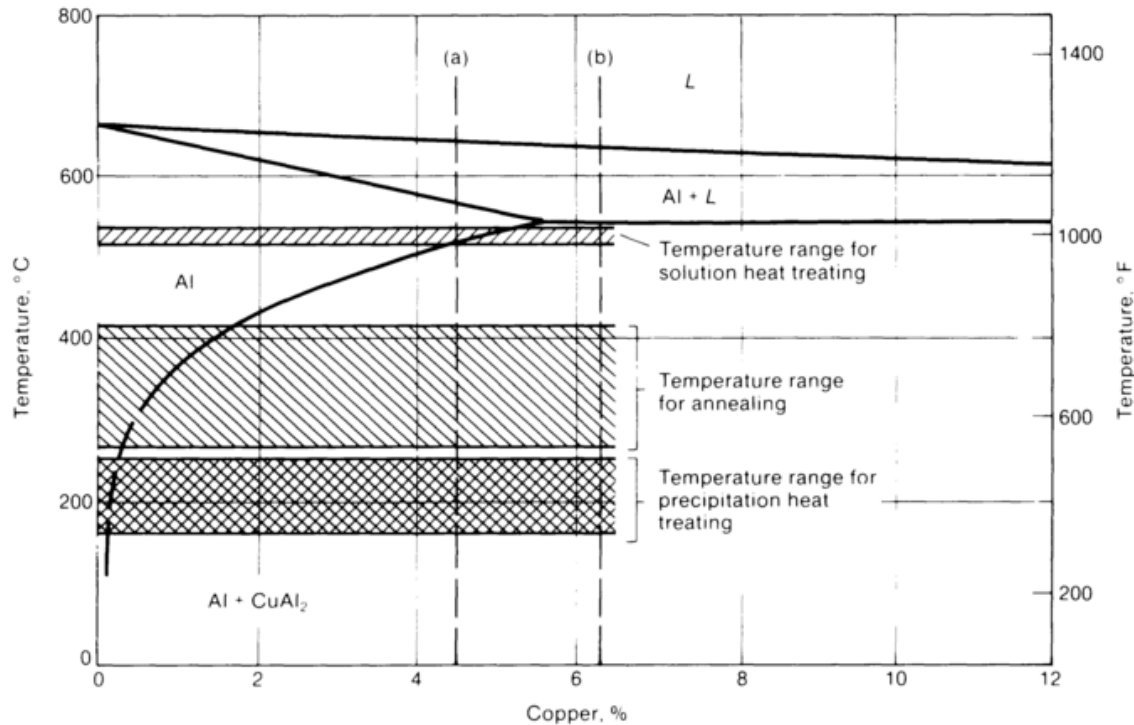


Figure 6– Temperature ranges for solution heat treatment, annealing, and aging for the Al-Cu system. The solution heat treatment temperature is kept below the eutectic temperature to avoid eutectic melting. ^[11]

The goal of quenching is to maintain the solid solution and vacancy concentration produced at higher temperature after cooling to lower temperature. A good quench produces a non-equilibrium super saturated solid solution with a high vacancy concentration, which are both necessary to form the fine dispersion of precipitates that strengthens the alloy. In order to attain this, the quench delay, or time between solution heat treatment and quench, must be minimized to prevent premature precipitation. Precipitates that are clustered around grain boundaries, non-soluble phase constituents, or disordered regions do not contribute to strengthening.

Precipitation strengthening in aluminum alloys is defined as aging. There are two different kinds of aging. In natural aging, precipitation occurs at room temperature, while

artificial aging utilizes a heat treatment to promote precipitation. These tempers have been given a standardized designation of a letter and series of numbers (Table II). For an alloy to be used in the naturally aged condition, T3 or T4 temper, it must become kinetically stable in roughly a week, meaning that further aging does not cause a significant change in properties. Some 2xxx series alloys can be useful in the naturally aged condition and are characterized by high tensile to yield strength ratios, fracture toughness, and fatigue resistance. However, most heat treatable alloys are artificially aged to produce stable properties more quickly.

Table II – Temper Designations of Aluminum Alloys ^[11]

F: As fabricated
O: Annealed
H: Strain-hardened
W: Solution heat treated
T: Heat treated to produce stable temper other than O
T1: Cooled from an elevated-temperature shaping process and naturally aged to a substantially stable condition.
T2: Cooled from an elevated-temperature shaping process, cold worked, and naturally aged to a substantially stable condition.
T3: Solution heat treated, cold worked, and naturally aged to a substantially stable condition.
T4: Solution heat treated and naturally aged to a substantially stable condition.
T5: Cooled from an elevated-temperature shaping process and artificially aged.
T6: Solution heat treated and artificially aged.
T7: Solution heat treated and stabilized, overaged.
T8: Solution heat treated, cold worked, and artificially aged.
T9: Solution heat treated, artificially aged, and cold worked.
T10: cooled from an elevated-temperature shaping process, cold worked, and artificially aged.

Artificial aging is denoted by tempers T5-T10 and involves a long, low temperature heat treatment, usually 240-375°F for 5-48 hours. Different time and temperature cycles are useful for maximizing different properties; commonly used tempers are usually best effort compromises. During the aging of many aluminum alloys, clusters of solute atoms form called Guinier-Preston zones that then form metastable precipitates, which transform to the equilibrium precipitates that strengthen the alloy. A mix of coherent and incoherent small precipitates with short distances between them is optimal for strength but may not be for corrosion properties. If aging is continued beyond the peak value, precipitates and the distances between them grow, resulting in lower strength (Figure 7). T6 and T7 are the most common artificial aging tempers, where T6 is aged to peak strength, and T7 is overaged to improve dimensional stability and corrosion properties.

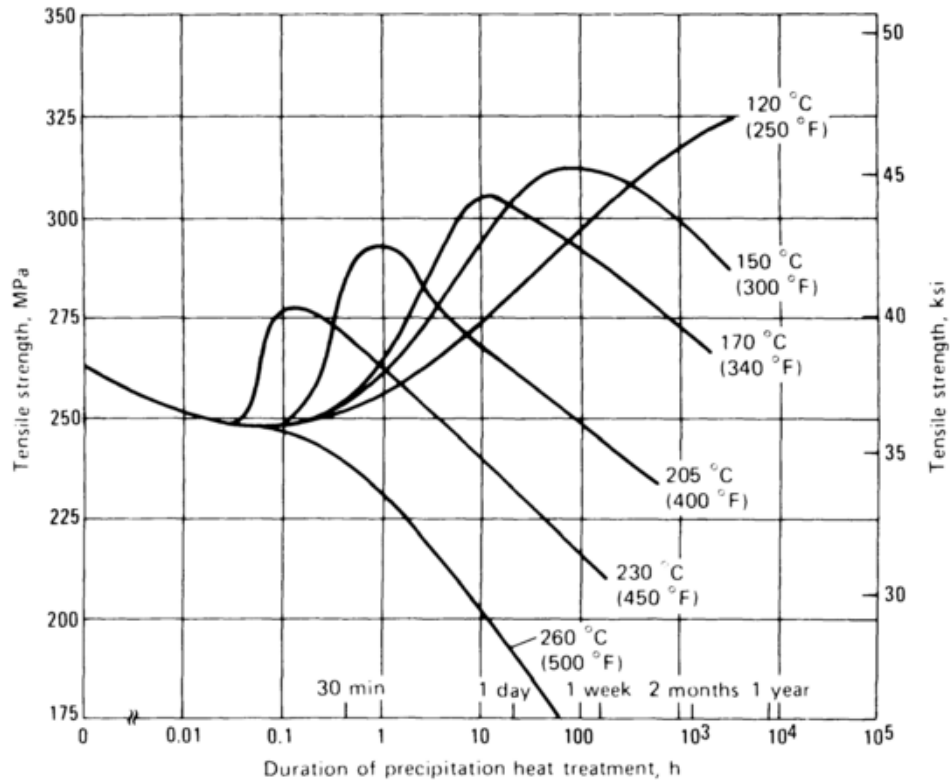


Figure 7– Tensile strength aging curve for 6061 sheet. ^[11]

c. Overheating and Incipient Melting in Aluminum Alloys

i. Microstructure

Due to its chemistry, 7050 is a complicated metallurgical system. The main soluble phase and strengthening precipitate is $MgZn_2$, but Al and Cu can also replace the Zn making $Mg(Zn,Al,Cu)_2$. Another soluble phase, Al_2CuMg , forms upon heating from the $MgZn_2$ that does not go into solution. Mg_2Si is another regular dispersoid that occurs in 7050. $\alpha AlFeSi$ and Al_7Cu_2Fe are the principal-iron bearing phases and are insoluble due to their Fe content. These are the common phases found in 7050 in order of prevalence. ^[12] During solution heat treatment, the transition through the (Al) solvus solutionizes most of the $MgZn_2$, but some transforms into Al_2CuMg . This phase is also soluble in (Al) but an appreciable amount is usually left in the structure. Driven by reduction in surface energy, the phase constituents begin to agglomerate, if given the energy and time to do so. As the Al_2CuMg phase agglomerates, it begins to pull the $MgZn_2$ out of solution, making a low melting eutectic that forms into rosettes (Figure 8). This is the first sign of overheating. The eutectic can be distinguished from the Al_2CuMg phase by its lighter color and lacey structure.

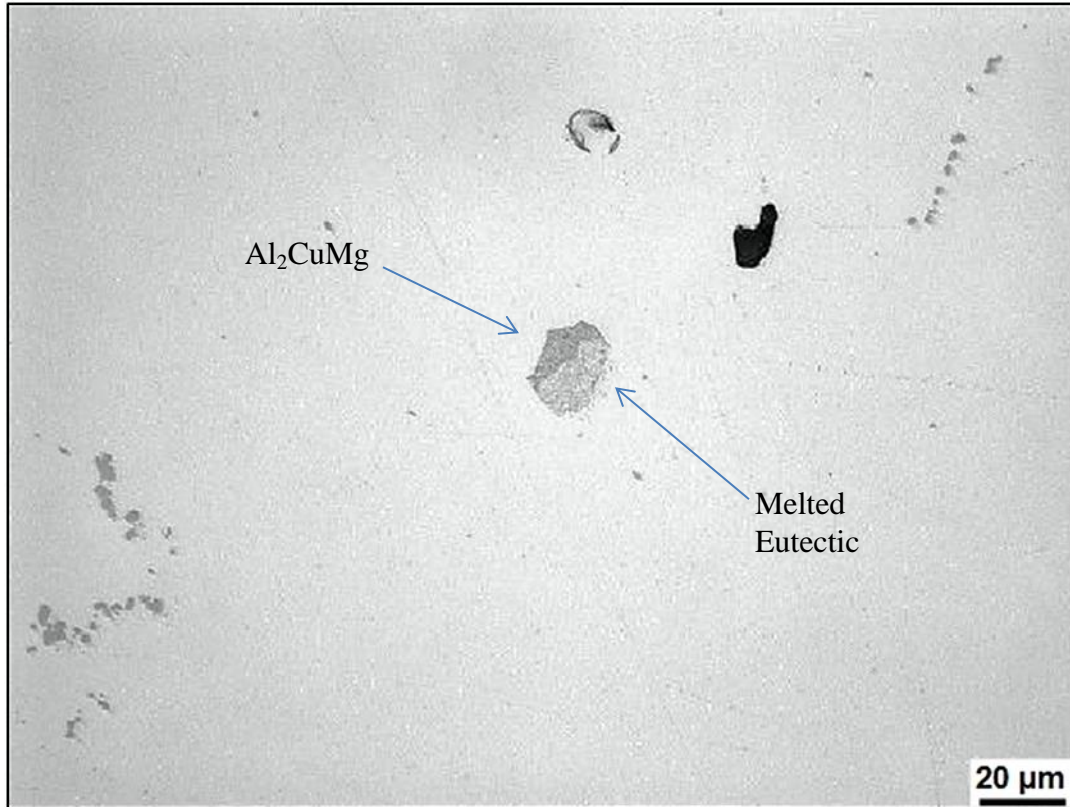


Figure 8 – A 7050 forging, W-tempered at 930°F for 2 hours. Al₂CuMg (dark gray) is in the process of forming into a rosette (light gray, lacey structure). As-polished condition.

As overheating progresses, the rosettes begin to migrate to the grain boundaries, in an attempt to reduce surface energy. Given the energy to diffuse, this process continues until the (Al) grains are depleted and completely cased in melted eutectic. Closer analysis of grain boundary melting in 7150 plate reveals a lamellar structure and a rich chemistry (Figure 9). The chemical composition of this 7150 is nearly identical to that of the 7050 used in this experiment.

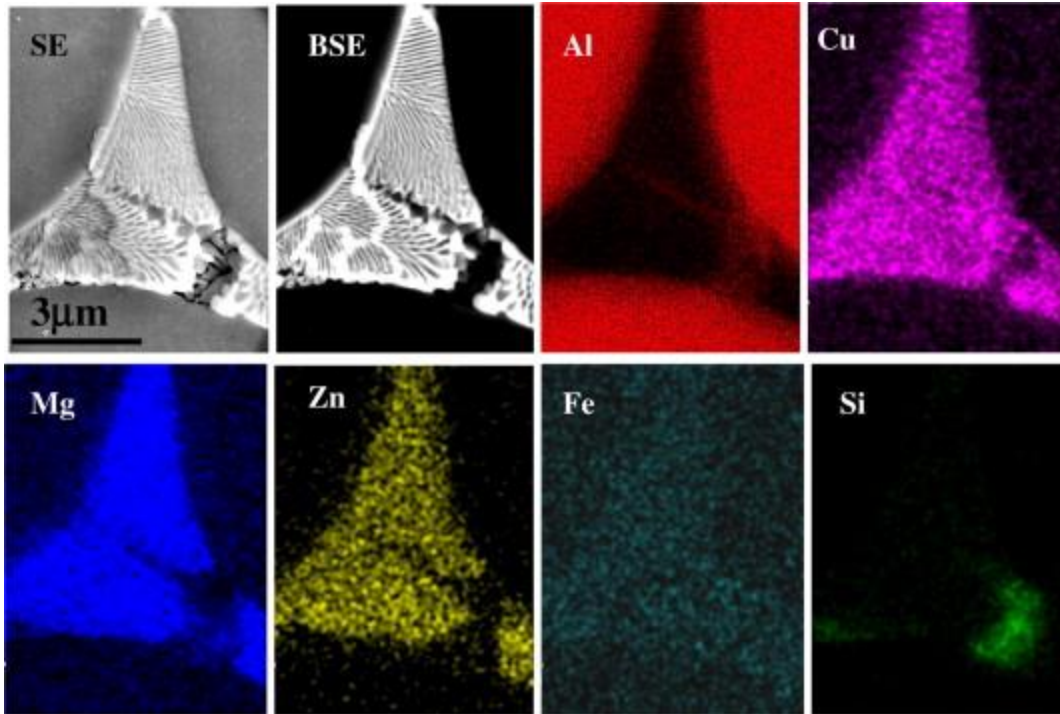


Figure 9- Electron Backscatter Diffraction (EBSD) and Energy Dispersive Spectroscopy (EDS) of grain boundary melting in 7150 plate solution heat treated at 959°F for 1 hour. As expected from the chemical composition, this region is rich in Cu, Mg, and Zn. ^[13]

ii. Effect on Mechanical Properties and Corrosion Resistance

The properties of a heat treatable aluminum alloy are dependent on the microstructure developed during solution heat treatment. Higher solution heat treatment temperatures help to put more of the alloying elements into solution, decreasing the volume fraction of second phase constituents. This increases strength by increasing the effectiveness of subsequent precipitation strengthening. Fracture toughness increases by decreasing the initiation sites related to second phase constituents. ^[14] Conversely, solution heat treatment temperatures past a certain point increase the volume fraction of coarse recrystallized grains, decreasing fracture toughness and strength (Figure 10). These are the mechanical effects of solution heat treatment, without considering overheating.

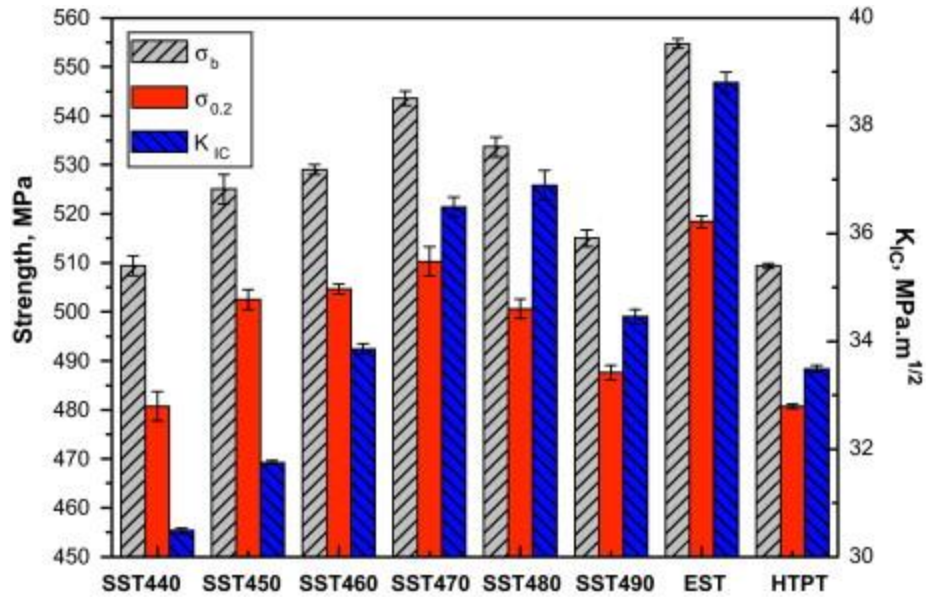


Figure 10– Mechanical properties of various solution heat treatment temperatures (440–490°C) for 7050, aged at 121°C for 6 hours then 163°C for 12 hours. The 490°C solution heat treatment is affected by overheating. ^[14]

The initial danger of overheating lies not in the rosettes themselves but in what they leave behind. Since a rosette is an area of positive segregation, it leaves an area of negative segregation or depletion around it. This depleted region is much weaker than the surrounding material due to its inability to form the $MgZn_2$ precipitates that would strengthen it. These weaker areas have the same effect on mechanical properties as porosity, namely a decrease in ductility and increase in crack initiation sites. The depleted area has little effect on corrosion, while the rosette itself can experience severe pitting corrosion, also providing an initiation site for stress corrosion cracking. As overheating continues, rosettes continue to form, agglomerate, and migrate to the grain boundaries due to the energetic favorability of this position. Melted eutectic material at the grain boundaries provides an easy path for crack propagation causing a significant drop in fatigue strength and fracture toughness, as well as increasing intergranular attack and stress corrosion cracking (Figure 11).

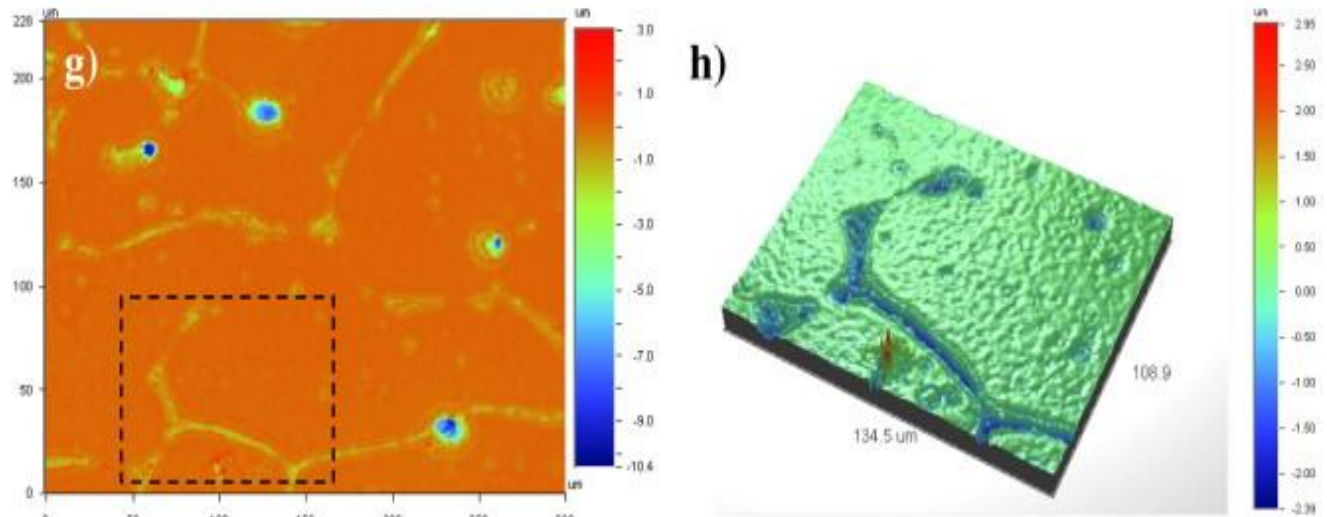


Figure 11 – Optical Profilometry of 7150 plate solution heat treated for 959°F for 1 hour and immersed in 0.1M NaCl solution for 12 hours. The 2-D profile shows that some pits, the 3-D profile shows intergranular attack along melted grain boundaries. ^[13]

The background knowledge provided here on aluminum, 7xxx series, forging, and incipient melting lays the foundation for more investigation of the evolution and effect of overheating in 7050. A deeper understanding of the time-temperature relationship of incipient melting will give aerospace component suppliers a better idea of the consequences of heat treatment errors and what factors of solution heat treatment are the most important in preventing overheating. Studying the corrosion characteristics of these various solution heat treatments will show the relationship between the amount of incipient melting and the severity of corrosion.

VI. Experimental Procedure

a. Safety

There are several major safety concerns in this experiment. Being a project that involves heat treatment, the utmost caution must be exercised when conducting solution heat treatments. Serious burns can occur if the right personal protective equipment is not worn. Thick leather gloves, a reflective apron, and heat resistant face shield must be worn at all times while the furnace is open.

The other major and potentially life threatening safety concern in this experiment is the use of Hydrofluoric Acid (HF) etchants. A quarter-sized drop of full strength HF is enough to be fatal. Extreme caution must be used when using HF. Splash-proof goggles, thick rubber gloves, and an apron must always be worn while etching.

b. SS-DTA

As previously mentioned, the theoretical incipient melting temperature of 7050 is 910°F, however; this can vary significantly based on local chemistry, heating rate, and solution heat treatment soak time. To gain a better understanding of the incipient melting temperature specific to this test material and furnace, a Single Sensor Differential Thermal Analysis (SS-DTA) was performed. A 3/32-inch hole was drilled into a 1-inch cube of test material and a thermocouple was inserted and enclosed in the hole. The sample was then put into an 1100°F resistance furnace. Temperature readings were collected at a rate of 5 Hz until twenty minutes after the temperature of the sample equalized with that of the furnace. As the piece heats up, endothermic phase transformations cause dips in the temperature versus time curve where the energy input is being used to change phases instead of increase temperature. These dips are called recalescences. By fitting a curve to the data points just before the recalescence, the start of the recalescence, and thus the start of the phase transformation, can be more accurately pin pointed.

c. Solution Heat Treatment Trials

The same test piece size and thermocouple procedures were used for the heat treatment trials, but here the thermocouples were only used for temperature regulation not data acquisition. The solution heat treatment temperatures selected to test were 890°F, 900°F, 910°F, 920°F, 930°F, and 940°F. Each of these temperatures was tested for four times: 1, 2, 4, and 6 hours. These times and temperatures were chosen to simulate possible heat treatment errors or furnace malfunctions. The furnace was loaded with four thermocoupled pieces then set to one of the selected temperatures. At each specified time, a piece was removed from the furnace and water quenched.

After solution heat treatment, each piece was sectioned, ground flat, mounted in a compression mounting compound, ground, and polished according to ASTM E3.^[15] All sections were transverse to the grain flow. Samples were viewed and imaged in the as-polished condition as well as etched with 0.5% Hydrofluoric acid (HF). Some samples were also re-polished and etched with a color etchant called Weck's Reagent to help detect the early stages of incipient melting. Weck's Reagent is a mixture of sodium hydroxide, potassium permanganate, and water.

d. Corrosion Tests

All of the work up to this point was done at Scot Forge, while the following was done at Cal Poly. To test for susceptibility to pitting corrosion, the 890-940°F, 4 hour solution heat

treatments were prepared metallographically to a 1 μ m finish and submerged in a 0.1M NaCl solution for 2.5 hours. This procedure was presented in literature and slightly altered to fit this experiment. [13]

Though not required for forgings by the specification that governs the heat treatment of aluminum alloys for aerospace applications (AMS 2772), intergranular attack corrosion tests will be conducted according to ASTM G110. Solution heat treated specimens will be immersed in a NaCl and H₂O₂ solution, prepared metallographically, and examined for the extent of intergranular corrosion. [16]

VII. Results

a. SS-DTA Data

SS-DTA data revealed two recalescences (Figure 12). The lower and more pronounced recalescence, around 525°F, is likely the temperature at which the majority of the alloying elements go into solution in the (Al) phase. The higher and more subtle recalescence, slightly above 900°F, is the temperature at which incipient melting is starting to occur through rosette formation.

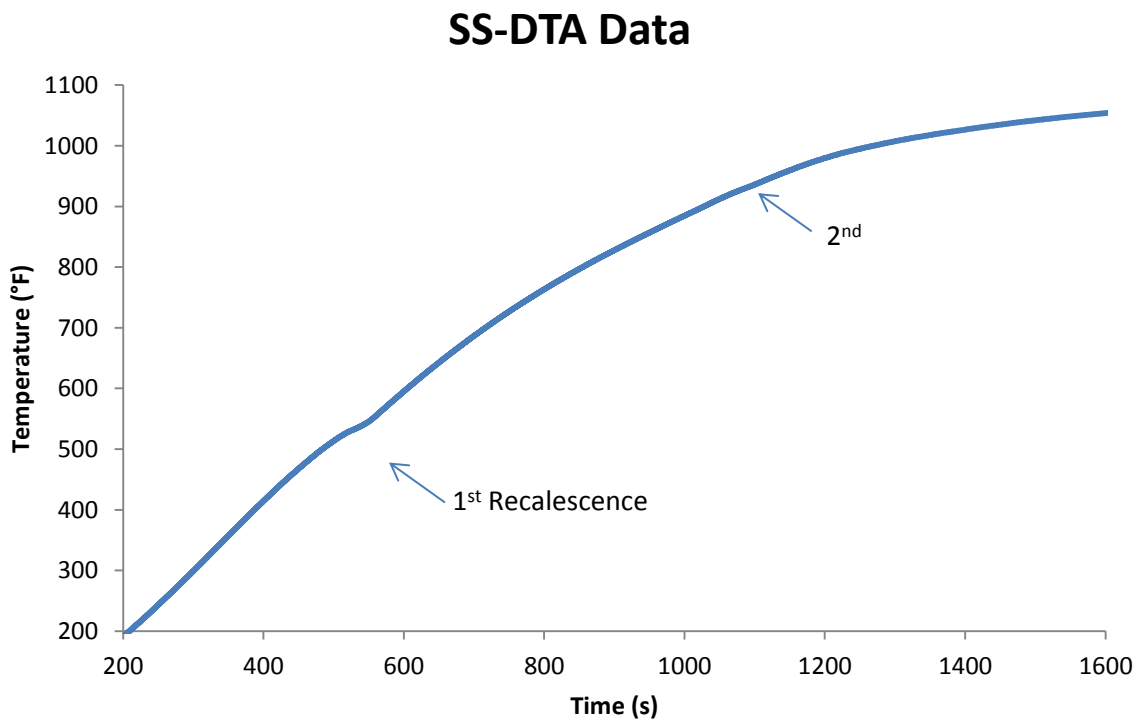


Figure 12 - SS-DTA data collected at 5Hz from a 1 in cube of 7050 in an 1100°F furnace.

Further analysis was conducted on the upper recalescence (Figure 13). The blue line is the collected data, and the thin black line is a trend of the collected data before the recalescence occurs. This trend line equation is then projected and becomes the green line, which is locally compared to the start of the recalescence in the recorded data. The difference of the recorded data (blue line) and expected projection (green line) more precisely pin points the beginning of the transformation. This analysis revealed that the upper recalescence deviated from the expected curve at 924°F.

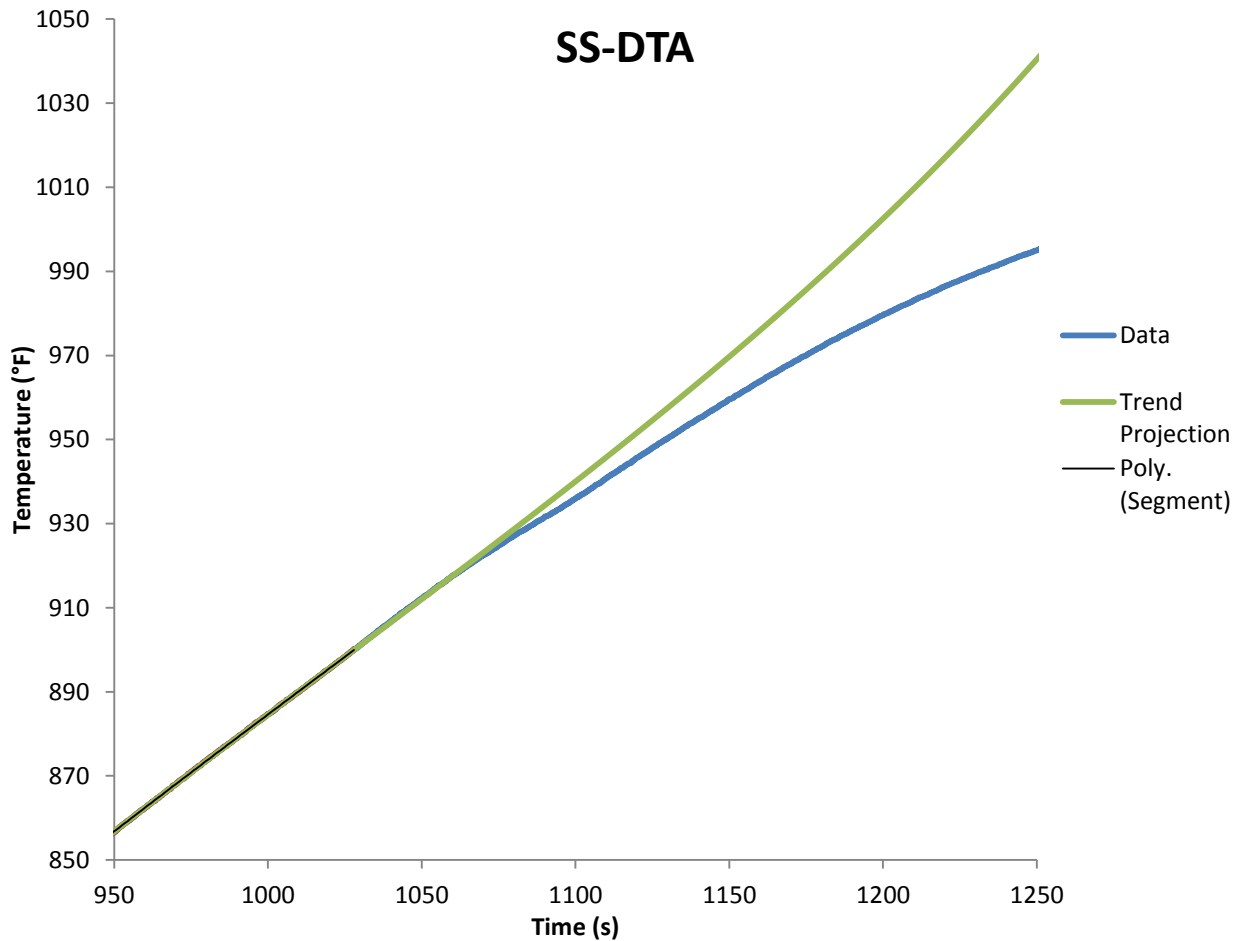


Figure 13 – Analysis of the upper recalescence of the SS-DTA data provided in Figure 12.

This sample showed no obvious visual signs of melting when it was removed from the furnace and quenched, so a metallographic sample was prepared from it to confirm that the aforementioned recalescence actually represented melting. The sample showed extensive rosettes and grain boundary melting, forming a eutectic network around the grains (Figure 14).

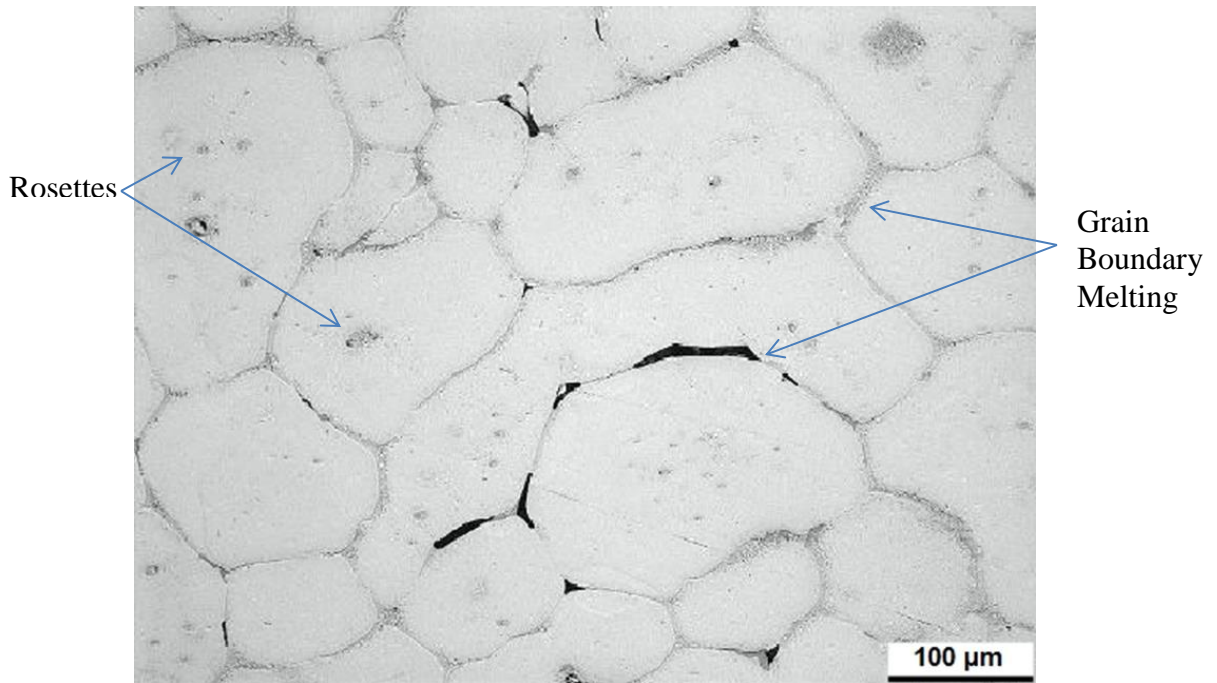


Figure 14– 1100°F SS-DTA sample. Obvious overheating, rosettes, and grain boundary melting present. Etched with 0.5% HF.

b. Solution Heat Treatments

As expected, the 890°F solution heat treatment trial samples showed no signs of overheating, though the longer times showed a more solutionized structure, while the phase constituents that remained were more agglomerated (Figure 15).

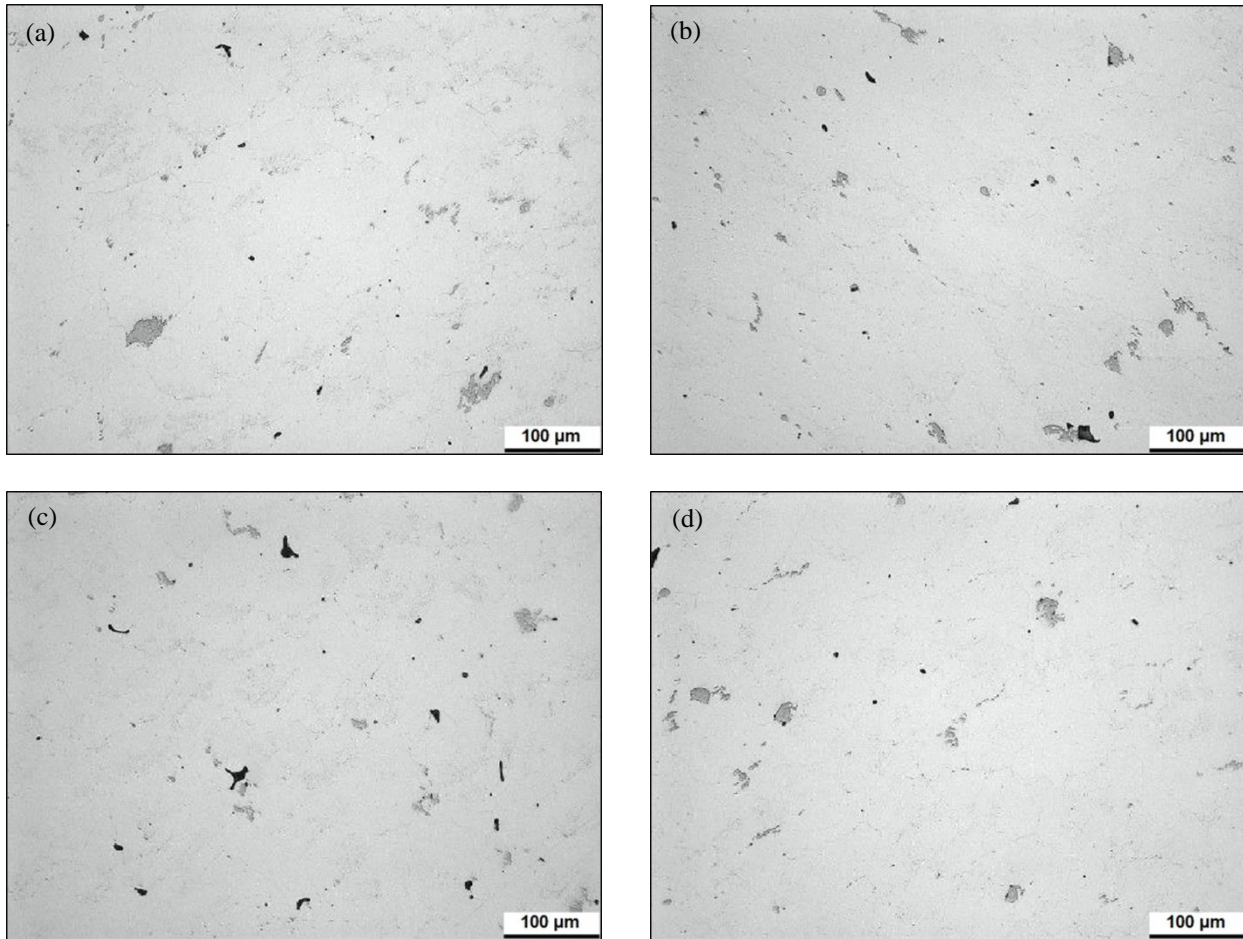


Figure 15– 890°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. No signs of overheating, fine distribution of second phase constituents begin to soltuionize or agglomerate as time increases. Etched with 0.5% HF.

The 900°F solution heat treatment showed more overall change in phase morphology, including the same trend of agglomeration over time of undissolved phases as the 890°F trial. No apparent signs of melting were observed in any of these specimens (Figure 16).

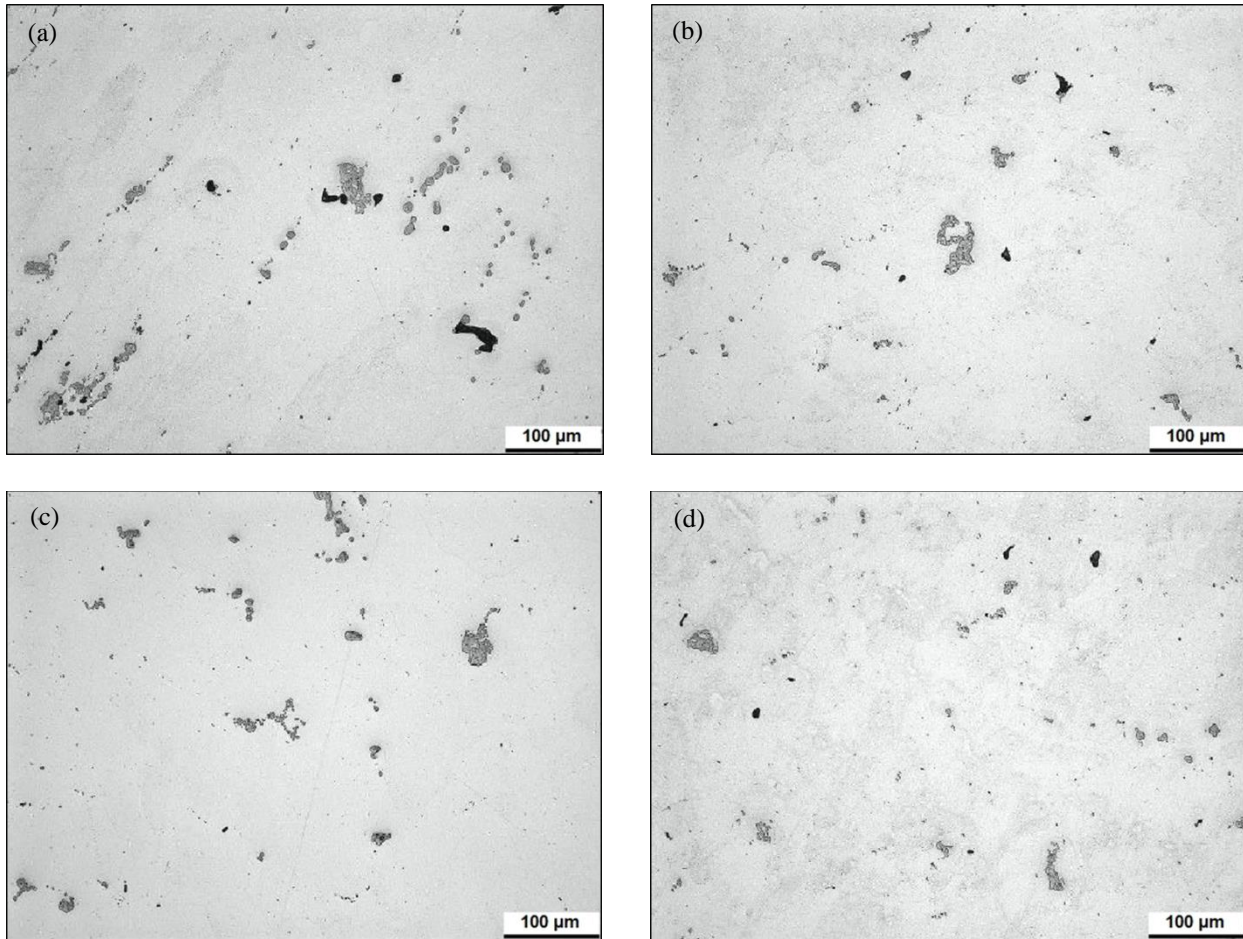


Figure 16 – 900°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. No signs of overheating, more changes in phase morphology and agglomeration as time increases. Etched with 0.5% HF.

The 910°F solution heat treatment, as suggested by the ASM Handbook, seems to be the “tipping point” of rosette formation. Some samples showed the Al_2CuMg phase beginning to melt by a slight change in color and transition to a more lacey structure, while some just showed more agglomeration (Figures 17).

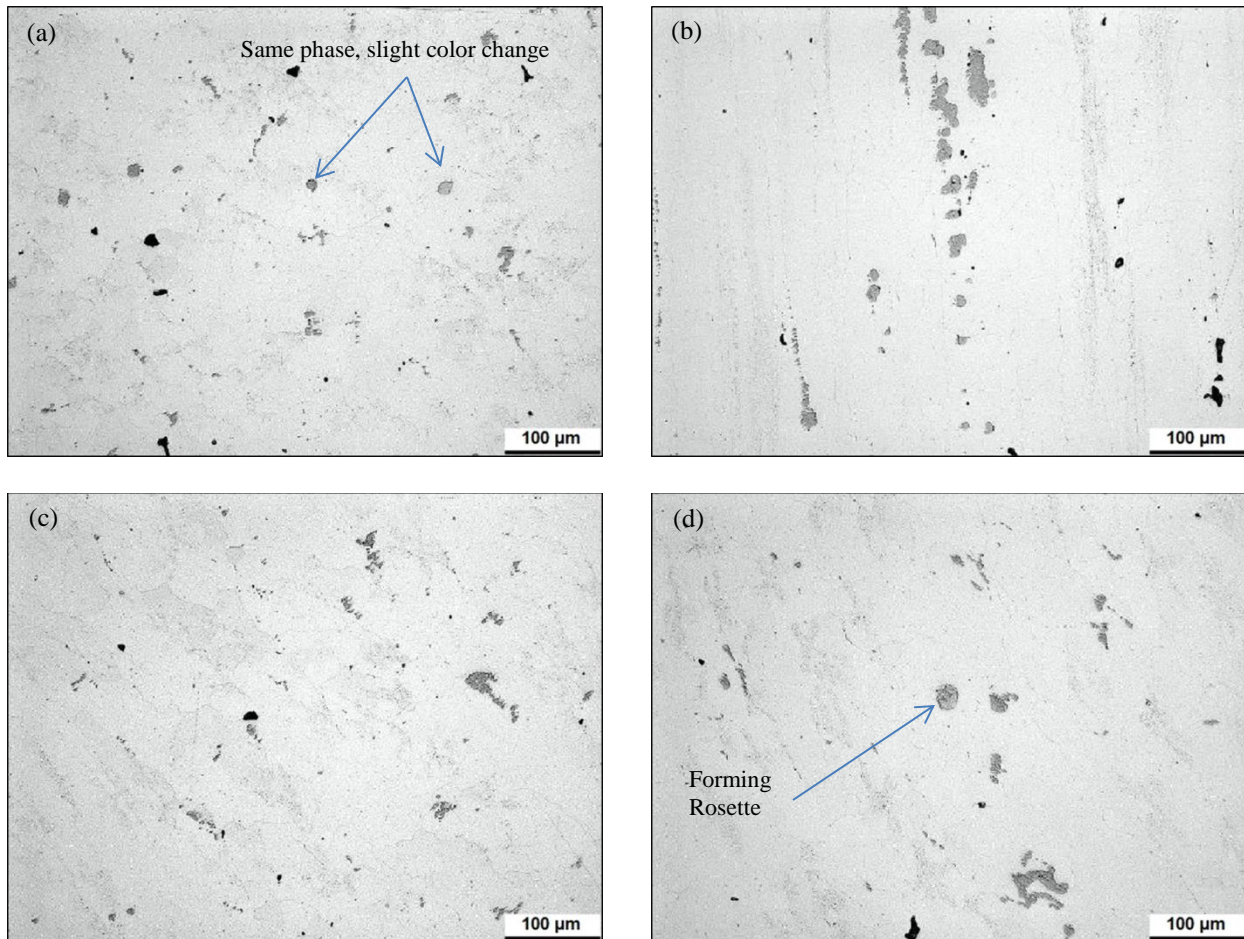


Figure 17 – 910°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Continued agglomeration, some possible rosette formation seen in the color and shape change of the Al_2CuMg in the 1 and 6 hour samples. Etched with 0.5% HF.

The 920°F solution heat treatment shows obvious signs of melting: rosettes, rosettes still in formation, as well as the beginning of grain boundary melting (Figures 18).

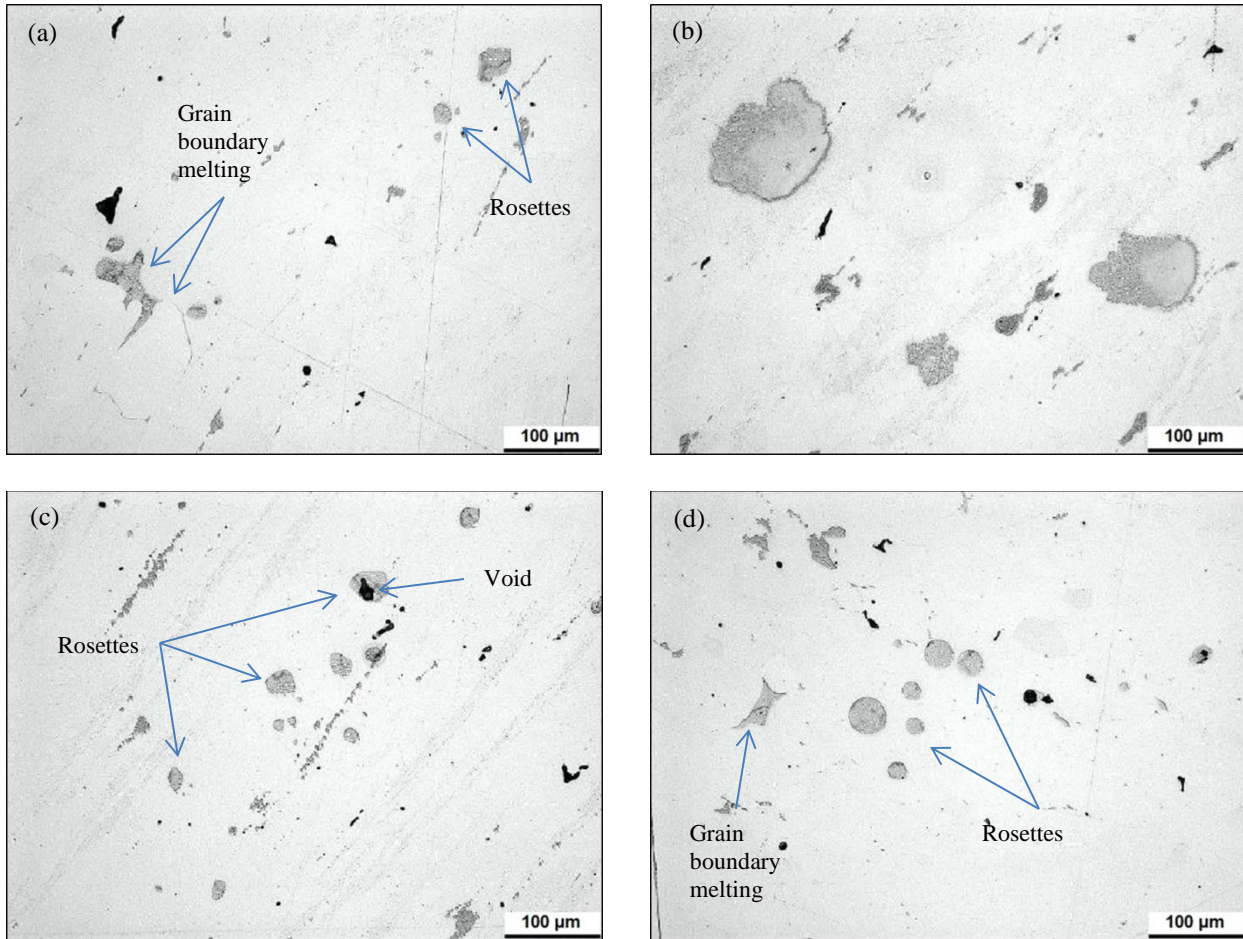


Figure 18 – 920°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Clear rosettes, partially formed rosettes, and grain boundary melting in all samples. Etched with 0.5% HF.

The same signs of melting that were apparent in the 920°F solution heat treatment are slightly more common and prevalent in the 930°F solution heat treatment, but the overheating has not progressed further than that (Figures 19).

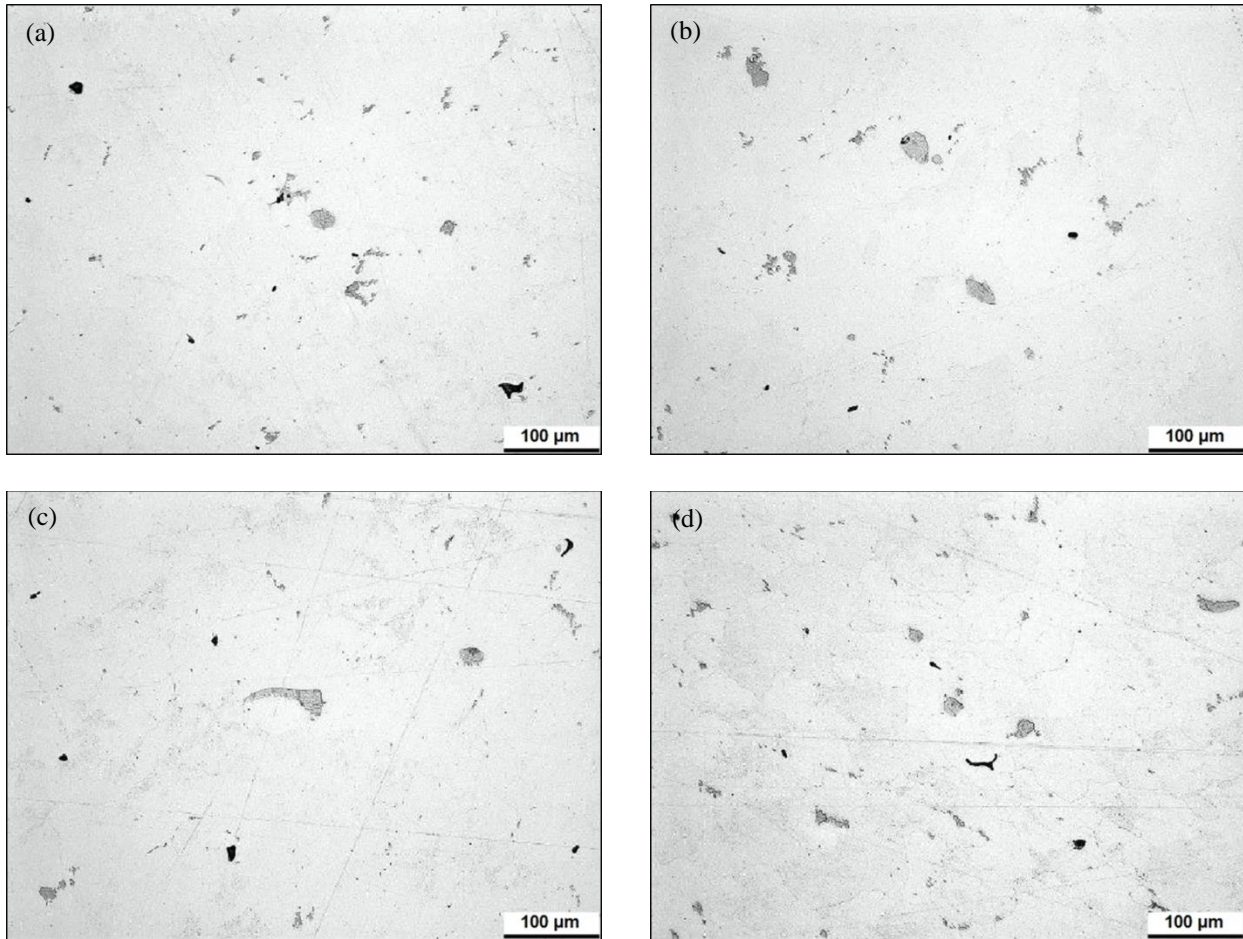


Figure 19 – 930°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Rosettes and grain boundary melting present in all samples, similar to 920°F solution heat treatments. Etched with 0.5% HF.

The 940°F solution heat treatment showed rosettes and not only grain boundary melting but also fracture that looks to be caused by grain boundary melting (Figure 20).

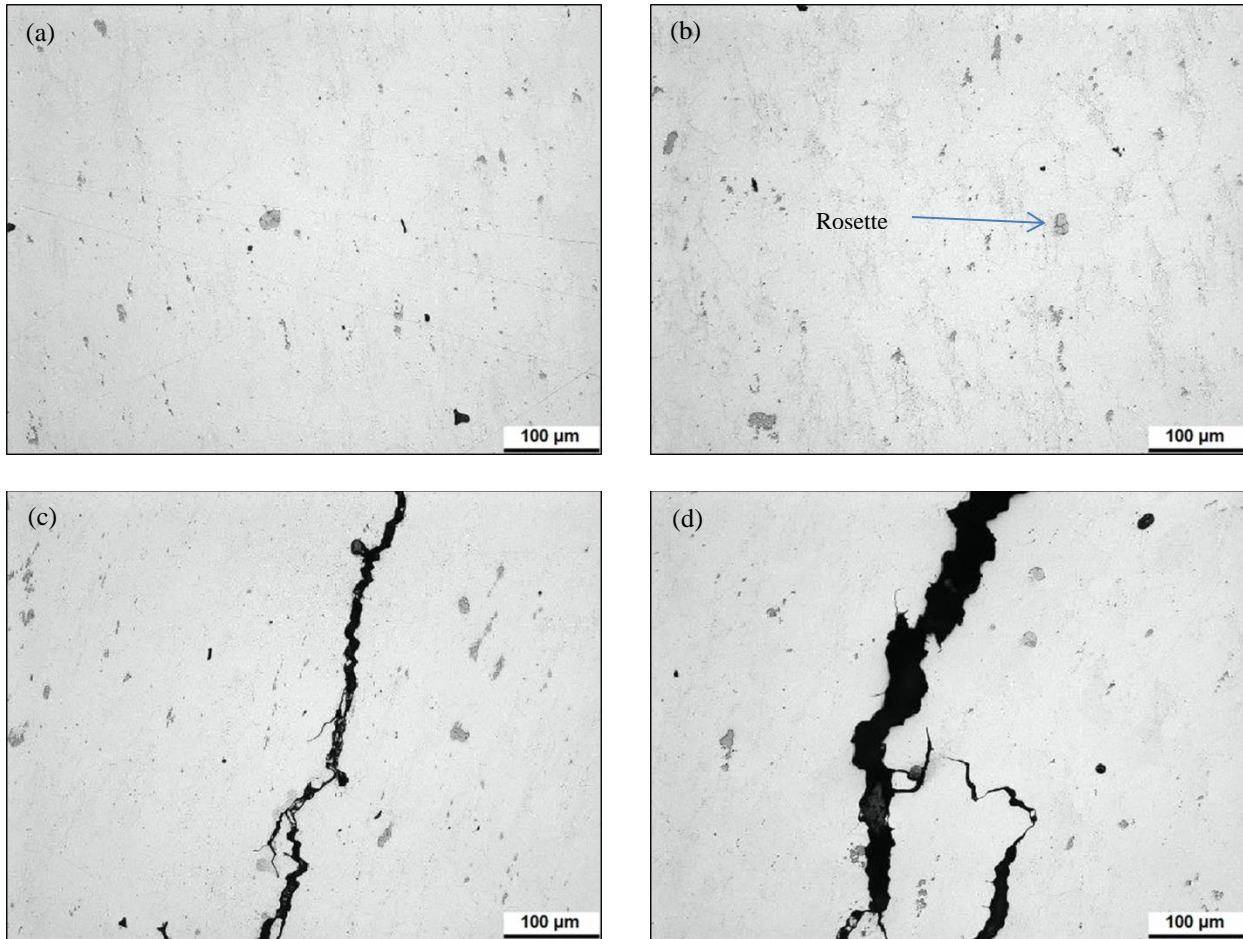


Figure 20 – 940°F solution heat treatment for (a) 1, (b) 2, (c) 4, and (d) 6 hours. Rosettes and grain boundary melting present in all samples, intergranular fracture due to incipient melting across 4 and 6 hour samples. Etched with 0.5% HF.

c. Pitting Corrosion

The severity pitting corrosion caused by the NaCl solution was roughly the same across all solution heat treatment trials (Figure 21). There are different densities of pitting across each surface. Some anodic regions are more heavily pitted (Figure 21c), while other regions are more cathodic and show lower pitting density (Figure 21d), but all microstructures showed both regions. In the 920°F-940°F solution heat treatments, rosettes and grain boundary melting were found un-pitted in the cathodic region (Figures 21d-f). A closer look shows a clearly formed rosette, identified by its lamellar structure, intact near other pits, implying that it is in general cathodic to the matrix (Figure 22).

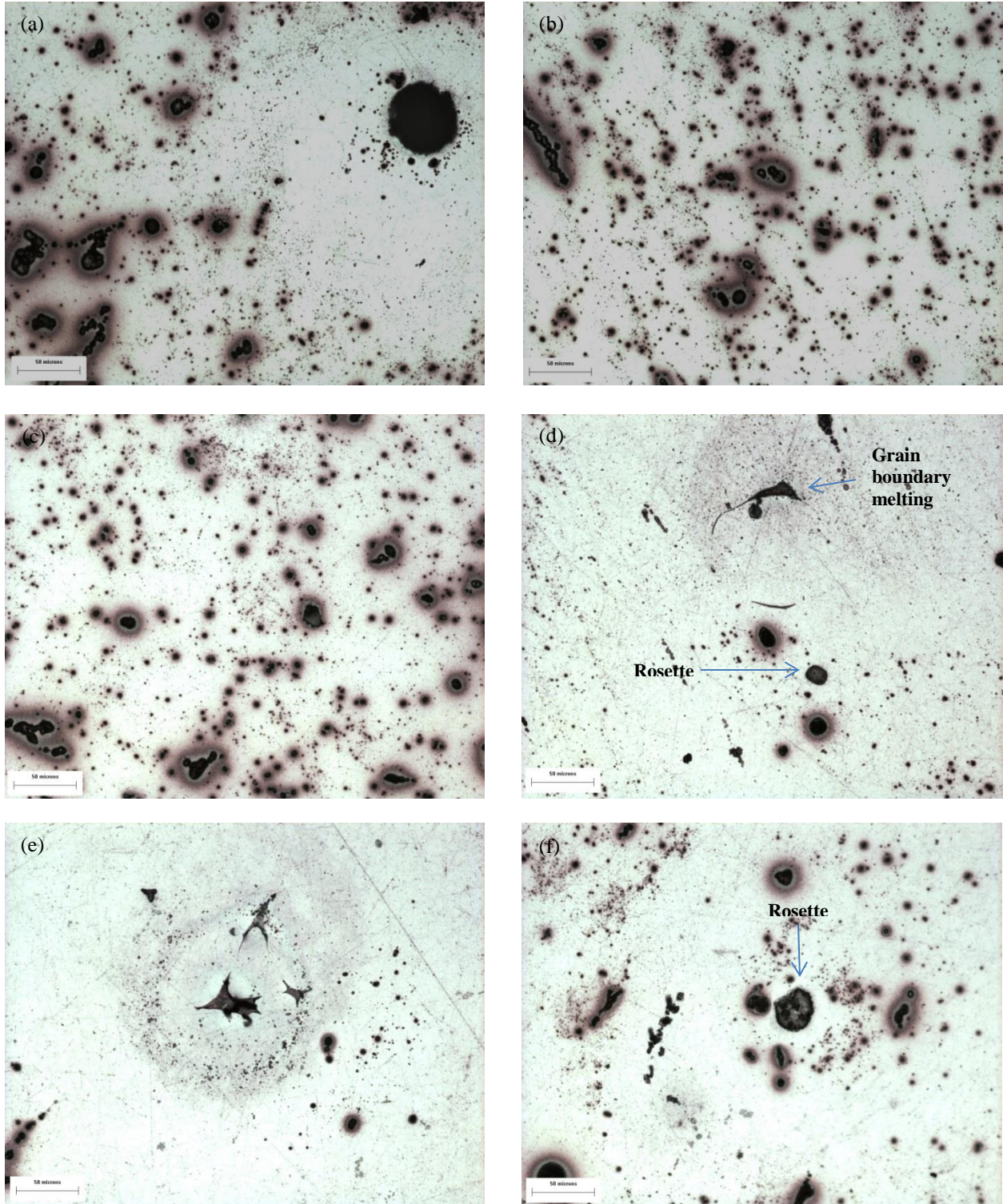


Figure 21 – 4 hour solution heat treatments at (a) 890°F, (b) 900°F, (c) 910°F, (d) 920°F, (e) 930°F, and (f) 940°F. Immersed in a 0.1M NaCl solution for 2.5 hours after metallographic preparation. Majority of pitting around $MgZn_2$ and Mg_2Si precipitates, rosettes and grain boundary melting found un-pitted.

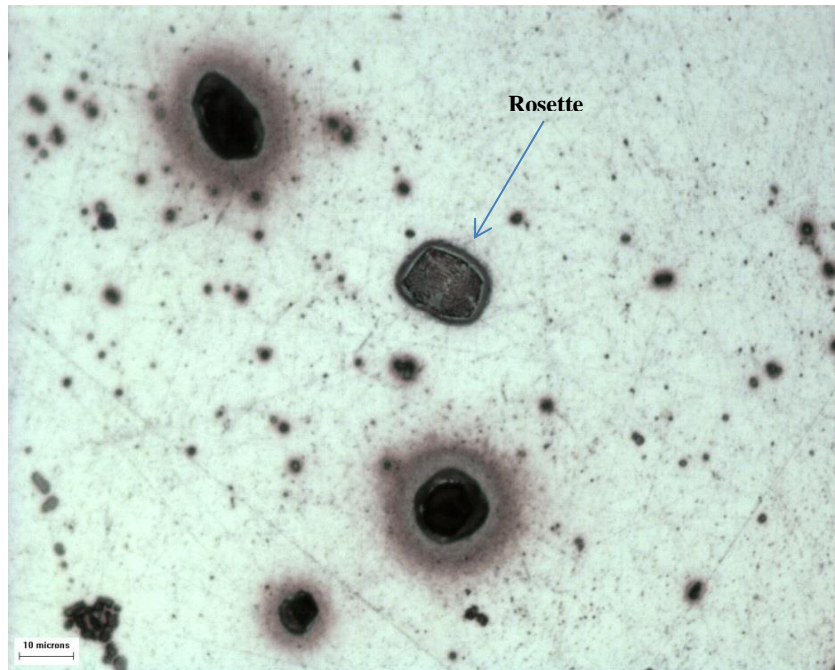


Figure 22 – 920°F solution heat treatment for 4 hours. Immersed in a 0.1M NaCl solution for 2.5 hours after metallographic preparation. An intact rosette near other pits.

d. Intergranular Corrosion

Similarly to the pitting corrosion tests, little variance in corrosion sensitivity was seen across all solution heat treatment trials (Figure 23). All samples showed some pitting at the surface but no intergranular attack. To see the effect of extreme incipient melting on intergranular attack susceptibility, the same intergranular corrosion test was conducted on test material from the SS-DTA, which reached 1100°F. This sample, which has an extensive melted eutectic network at the grain boundaries, showed only slight susceptibility to intergranular attack (Figure 24).

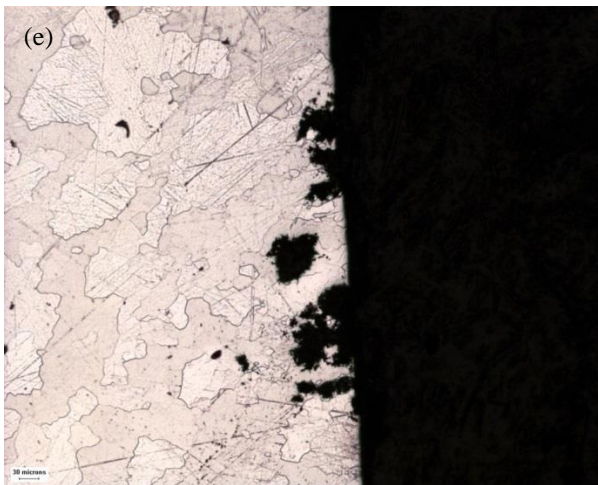
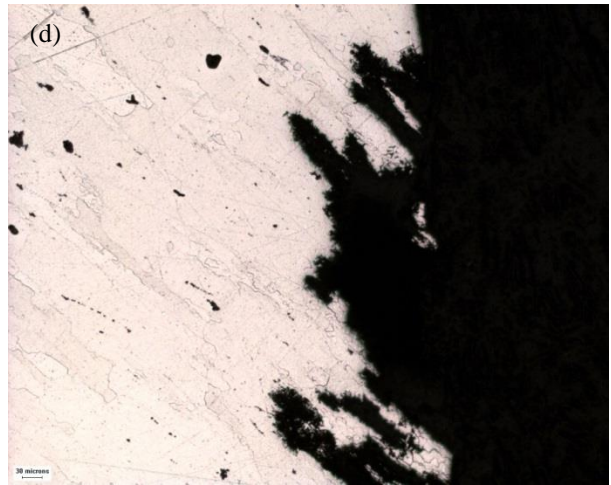
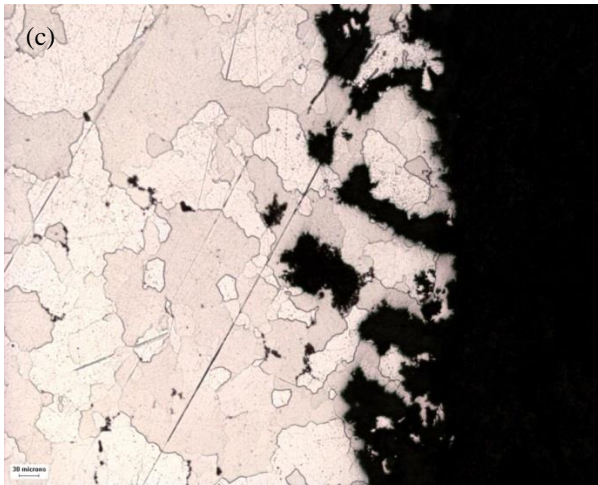
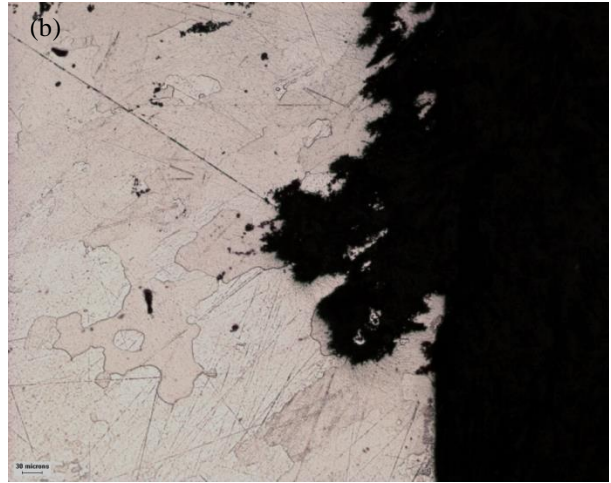
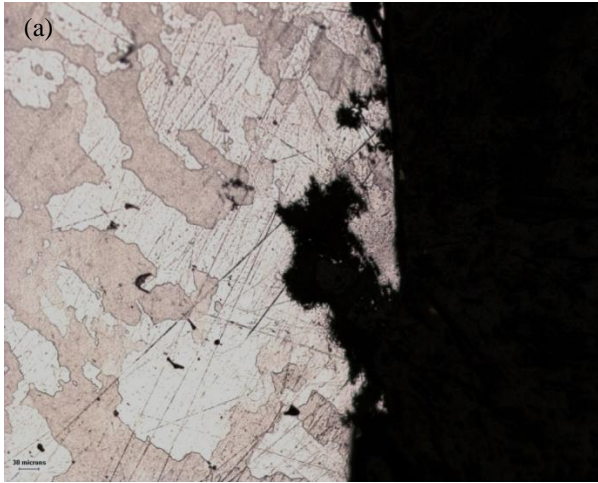


Figure 23– 4 hour solution heat treatments at (a) 890°F, (b) 900°F, (c) 910°F, (d) 920°F, (e) 930°F, and (f) 940°F. Immersed in a H₂O₂ and NaCl solution for 6 hours and then prepared metallographically. Small varied amounts of pitting at the surfaces but no signs of intergranular corrosion.

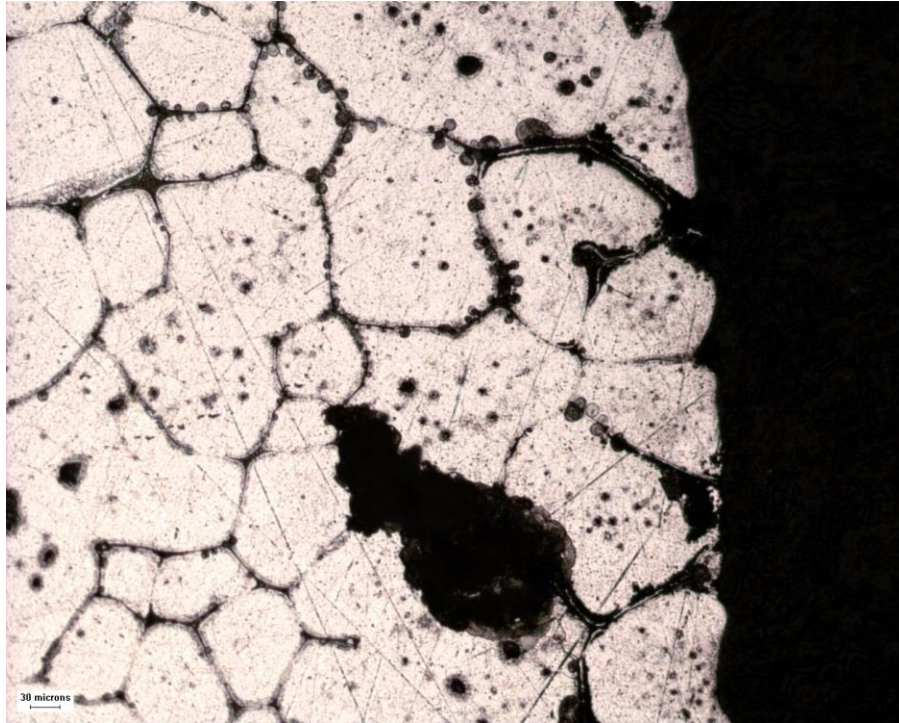


Figure 24 – SS-DTA sample, reached 1100°F for about one hour. Immersed in a H₂O₂ and NaCl solution for 6 hours and then prepared metallographically. Slight intergranular corrosion at surface, extensive eutectic network throughout sample.

VIII. Discussion

The two recalescences in the SS-DTA data represent the (Al) solvus and the incipient melting of the remaining Al₂CuMg. The experimental incipient melting temperature of 924°F is slightly different than the theoretical value of 910°F. This difference could be due to several factors. The higher heating rate during SS-DTA, due to putting room temperature material into an 1100°F furnace rather than turning the furnace on after its loaded, would generate a need for more superheat than equilibrium conditions. Variation in the chemistry of the test material could also cause a change in incipient melting conditions. This difference could also be due to the physical parameters of the SS-DTA test such as test piece size and thermocouple wire diameter. Less test material and larger thermocouple wires both decrease the sensitivity of the test, increasing the chance that the SS-DTA would not detect a significant difference in the time-temperature curve until the incipient melting is more severe. Whatever the reason for this difference between theoretical and experimental incipient melting temperatures, SS-DTA confirmed that the selected temperature range would span from un-melted to melted structures and gave an estimate where incipient melting should occur.

Microstructurally, the expected overall trends from these solution heat treatment trials were observed. Both higher temperatures and longer times lead to a more solutionized structure, with more agglomeration in the remaining second phase constituents that did not go into solution. The occurrence of incipient melting fell in between the theoretical and experimentally determined incipient melting temperatures. There were no signs of melting any of the 910°F samples except for the 6 hour trial, which showed only a few instances of early rosette formation. The 920°F samples, on the other hand, each showed distinct and obvious signs of melting, which fits the 924°F recalescence. Solely from this microstructural observation, temperature seems to have a much greater effect on incipient melting than time, since the 910°F, 6 hour sample showed only the earliest stages of melting and the 920°F, 1 hour sample showed clear signs of melting in the form of rosettes and grain boundary liquation. Thus, qualitatively an extra 10°F made a much larger difference than an extra 5 hours at temperature. This conclusion is consistent with what is expected for incipient melting, being a diffusion-based, thermally activated process. From a production standpoint, a part accidentally left at solution heat treating temperatures for twice as long as normal is much better off than a part that exceeded those temperatures by 10-20°F, even for a short amount of time. In the case of this 7050 aluminum, the incipient melting boundary seems to be excessive times at 910°F. Any heat treatment hotter than this, for any significant amount of time, is likely to cause melting.

With respect to severity, the higher temperature and longer times showed more frequent and more significant melting, once again with temperature having the greater effect. Rosettes as well as grain boundary melting occurred in all samples 920°F and above, with fracture occurring in the longer 940°F trials. These fractures appear to be intergranular by their jagged crack path and widely branching nature. Originating at the surface of the piece, initiation likely occurred during quenching and then propagated through the material along grain boundaries weakened by eutectic melting.

Literature suggested that the incipient melted eutectic phase would corrode in pitting preferentially before the rest of the structure. This claim was refuted by the results which showed solution heat treatment temperature and incipient melting to have no effect on pitting corrosion susceptibility. Additional literature showed that the Al_2CuMg phase from which the rosettes form is normally cathodic to the matrix in 7075, a similar alloy (Table III). Assuming that these phases have the same relationship to the matrix in 7050 as in 7075, the Al_2CuMg phase should

not corrode in any type of galvanic corrosion. The incipient melting phase; however, is not just Al_2CuMg . During overheating Mg and Zn, which are highly active elements in a galvanic series, are pulled out of solution and form a different phase when added to Al_2CuMg . Since the incipient melted eutectic phase was found un-corroded, it can be inferred that this increased Mg and Zn segregation does not change the relationship of the phase to the matrix. This implies that incipient melting in 7050 does not have an effect on galvanic corrosion.

Table III – Relationship of 2nd Phase Constituents to the Matrix in 7075^[17]

Second Phase Constituent	Relation to Matrix
MgZn_2	Anodic
$\text{Al}_7\text{Cu}_2\text{Fe}$	Cathodic
Al_3Fe	Cathodic
Mg_2Si	Anodic
Al_2CuMg	Cathodic

Intergranular attack, being another form of local galvanic corrosion, was not affected by incipient melting. Little to no intergranular attack susceptibility was observed in any of the solution heat treatment temperatures. This can be justified by the same reasoning as the pitting corrosion, namely a cathodic incipient melted phase. The SS-DTA sample, however; showed some susceptibility to intergranular corrosion. Assuming the melted eutectic phase has a fixed composition, this can be explained by a change in composition of the matrix. A more pure (Al) matrix will be less likely to be anodic than one saturated with Mg and Zn, shifting the galvanic series to be more likely to corrode the eutectic phase compared to the matrix.

IX. Conclusions

1. No signs of overheating were found in any of the 890-900°F solution heat treatments. First signs of rosette formation were found in the 910°F samples, with progressively more formation with increasing time. Obvious incipient melting was found in all 920-940°F solution heat treatments.

2. Solution heat treatment time has much less of an effect on incipient melting than temperature. Little microstructural variance was observed between one hour and six hour solution heat treatments.
3. The incipient melting eutectic phase in 7050 is cathodic to the matrix, making incipient melting have no significant effect on pitting corrosion or intergranular attack because they are forms of galvanic corrosion.

X. References

- [1] J. Davis, ASM Specialty Handbook: Aluminum and Aluminum Alloys, Materials Park: ASM International, 2007.
- [2] E. L. Rooy, Introduction to Aluminum and Aluminum Alloys, Volume 2, ASM International, 1990.
- [3] E. Starke, Application of Modern Aluminum Alloys to Aircraft, London: Elsevier Science, 1996.
- [4] ASM International, Closed-Die Forging in Hammers and Presses, Volume 14A, ASM International, 2005.
- [5] CES, *Closed-Die Forging Schematic*. [Art]. Granta Design, 2014.
- [6] ASM International, Open-Die Forging, Volume 14A, ASM International, 2005.
- [7] CES, *Open-Die Forging Schematic*. [Art]. Granta Design, 2014.
- [8] <http://www.phillipsmetalworks.com/shop.htm>.
- [9] <http://www.scotforge.com/Learning-Center/Forging-101/Open-die-forging#490124-3-drawing-down-the-first-step-to-size>.
- [10] G. Kuhlman, Forging of Aluminum Alloys, Volume 14A, ASM International, 2005.
- [11] ASM International, Heat Treating of Aluminum Alloys, Volume 4, ASM International, 1991.
- [12] Kaiser Aluminum & Chemical Corporation, 7050 Metallography Handbook, Pleasanton.
- [13] D. K. Xu, "Effect of solution treatment on the corrosion behaviour of aluminium alloy AA7150: Optimisation for corrosion resistance," *Corrosion Science*, vol. 53, no. 1, pp. 217-225, 2011.
- [14] N. Han, "Effect of solution treatment on the strength and fracture toughness of aluminum alloy 7050," *Journal of Alloys and Compounds*, vol. 509, no. 10, pp. 4138-4145, 2011.
- [15] ASTM International, "ASTM E3: Standard Practices for Preparation of Metallographic Specimens," ASTM International, West Conshohocken, 2011.
- [16] ASTM International, *ASTM G110: Standard Practice for Evaluating Intergranular Corrosion Resistance of Heat Treatable Aluminum Alloys by Immersion in Sodium Chloride + Hydrogen Peroxide Solution*, West Conshohocken, 2009.
- [17] M. Cavanaugh, "Evaluation of a Simple Microstructural-electrochemical Model for Corrosion Damage Accumulation in Microstructurally Complex Aluminum Alloys," *Engineering Fracture Mechanics*, vol. 76, no. 5, pp. 641-650, 2009.