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# Prediction and Reduction of Die Soldering.

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# **Die Solder Prediction and Reduction**

by

**Patrick Alan Hogan**

A Thesis

Submitted to the Faculty

of the

WORCESTER POLYTECHNIC INSTITUTE

in partial fulfillment of the requirements for the

Degree of Master of Science

in Materials Science & Engineering

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## **Abstract**

Die Soldering occurs in aluminum permanent mold casting when the cast metal bonds with the die surface and remains stuck upon ejection of the part. Eventually, this layer builds up and production must be stopped for cleaning. It was estimated in a Contech squeeze casting plant in Pierceton, IN, that 1.5% of variable overhead can be directly attributed to die soldering.

Previous work at WPI has focused on developing the mechanism of how soldering occurs. This work focuses on how that knowledge can be applied in an industrial setting. The work has focused on 4 major areas: (1) Using MAGMAsoft to predict die soldering, (2) Using surface metrology to measure die soldering, (3) Documenting the total process effects of using strontium modified casting alloys.

The work has resulted in: (1) Guidelines for using MAGMAsoft to predict die soldering. The results can be incorporated into the existing MAGMA die soldering module, but provide more accurate time and temperature criteria. (2) The results of the study prove that measurement of the surface of the cast part itself can be used as a method for quantifying die soldering. (3) The total process effects of Sr-modification are reported, along with suggestions for immediate use of Sr-modification at the Pierceton, IN casting plant and guidelines for using Strontium in the future.

## **Acknowledgments**

I would like to thank Contech for giving me the opportunity to work as an industrial intern. The experience and knowledge that I have gained in the past two years has been invaluable and should be a contributing factor in any successes I will have in my career. Primarily, Joe Bigelow was a great resource and a wonderful person to work with towards the completion of the project. I would also like to thank Brian Szymanowski, who provided excellent leadership, Chuck Barnes for his help with MAGMA, and the entire staff of the Pierceton casting plant, which was always kind and helpful. Without their cooperation the project could not have been successful. I sincerely hope that my project has yielded significant value for everyone at Contech.

I would also like to give my thanks to my advisor, Diran Apelian, for his unending support and guidance. For the past three years he has inspired me to become a better engineer and person, and has opened many doors for me.

The entire staff at MPI deserves my gratitude for their input and help; it is their contributions to everyone's work that makes MPI a great place to study and enable each student to complete such great work.

Professor Chris Brown and the staff at the Surface Metrology Laboratory deserve thanks for helping me develop and carry out my experimental plans in my efforts to quantify die soldering by measuring the surface of the cast parts.

Finally, I would like to thank my family for always encouraging me to work hard and strive to be my best. Without them, I could not be where I am today, and for that I am very grateful.

# Table Of Contents

Die Solder Prediction and Reduction.....	i
Abstract.....	ii
Acknowledgments.....	iii
Table Of Contents.....	iv
I. Project Motivation.....	1
II. Literature Review.....	3
II.I Die Soldering Mechanism.....	3
II.II Factors Affecting Die Soldering.....	9
II.II.I Temperature.....	9
II.II.II Effect of Alloying Elements.....	12
II.II.III Gate Velocity.....	22
II.II.IV Die Surface Condition.....	23
II.III Die Soldering Measurement.....	23
II.IV Prediction of Die Soldering.....	25
II.IV.I MAGMAsoft Prediction Overview.....	26
II.V Die Coatings and Surface Treatments.....	27
II.V.I Die Coatings.....	27
II.IV.II Coating Failure.....	31
II.IV.III Surface Treatments.....	33
II.IV.IV Coatings and Surface Treatments: Conclusions.....	33
II.V Alternate Die Materials.....	34
II.VI References.....	36
III. Measurement of Die Soldering.....	38
III.I Qualitative Analysis: Surface Rating.....	38
III.I.I Method.....	38
III.I.II Results & Discussion.....	38
III.II Quantitative Analysis: Surface Metrology.....	39
III.II.I Methodology.....	40
III.II.II Results & Discussion.....	42
III.III Conclusions.....	50
IV. Prediction of Die Soldering.....	51
IV.I Methodology.....	52
IV.II Results and Discussion.....	57
IV.III Review of MAGMA Die Soldering Module.....	71
IV.III.I Evaluation Procedure.....	71
IV.III.II Results.....	72
IV.IV Conclusions.....	76
V. Reduction of Die Soldering: Strontium Modification.....	77
V.I Initial Investigation: Method.....	77
V.II Initial Investigation: Results & Discussion.....	79
V.II.I Effect of Strontium Modification on Die Soldering.....	79
V.II.II Effect of Strontium Modification on Scrap Rate.....	81
V.II.III Effect of Strontium Modification on Process Output.....	83
V.III Follow-up Investigation: Method.....	83

V.IV Follow-up Investigation: Results & Discussion .....	85
V.V U251 Carrier: Strontium Modification Investigation.....	88
V.VI Conclusions.....	90
VI. Conclusions & Future Work Suggestions .....	91
V.I Future Work Suggestions .....	92
Appendix 1: Qualitative Solder Rating Images .....	93
Appendix 2: Relative Area Scale Analysis.....	97
Appendix 3: Porosity Severity Rating Scale Examples.....	109

## I. Project Motivation

Die soldering is an unfavorable die casting phenomena which occurs when the cast metal adheres to the die by any of several mechanisms. The problem is particularly apparent in aluminum squeeze casting. Dies for this process are most commonly manufactured from H13 tool steel. The combination of the natural tendency for aluminum to react with iron and the high temperatures and pressures experienced in the die during the squeeze casting process leads to a great deal of die soldering.

Die soldering results in several detrimental effects to the die casting process. A study was carried out on the effects of die soldering on the casting of the U222 Carrier part at the Pierceton, Indiana squeeze casting facility owned by Contech, LLC in November 2006. The results of the study were:

- On average, each machine running the U222 die must be shut down for cleaning at least once per 7.5 hour shift, for a total of 0.5-1 hours. It was estimated that 50% of the need for cleaning can be attributed to soldering. Over a year, this adds up to approximately 85 hours of downtime per machine directly attributable to die soldering.
- Once the die has been shut down, several shots are needed to get the die back to running temperature. Approximately 1,100 warm-up shots per machine are scrapped each year.
- Approximately 200 parts per machine fail a leak test each year due to die soldering. Because of the work flow in the plant, value is lost due to the heat treating, shot blasting and machining performed on each of these parts, which occur before the leak test inspection.
- Each die running the U222 Carrier needs 5-10 new inserts each year, , and an entire core replacement. Most parts besides the Carrier require cores/pins/etc. which need replacing on a regular basis as a result of soldering and erosion. There is additional damage to each die which is impossible to quantify (such as erosion and washout) which results from soldering.
- All things considered, including tooling costs and downtime associated with removal, solder-related costs are a very significant portion of the plant's overall annual operational performance."

Due to the large negative impact of die soldering on the casting process, many studies have investigated the causes of soldering and ways to reduce its impact. Despite this, as can be seen in the above economic analysis, die soldering remains a serious issue in industry today. The objective of this project is to bridge the gap between die soldering research and casting practice by developing practical solutions for reducing soldering and methods for determining whether these solutions are cost effective.

Four specific areas were investigated and studied; these are:

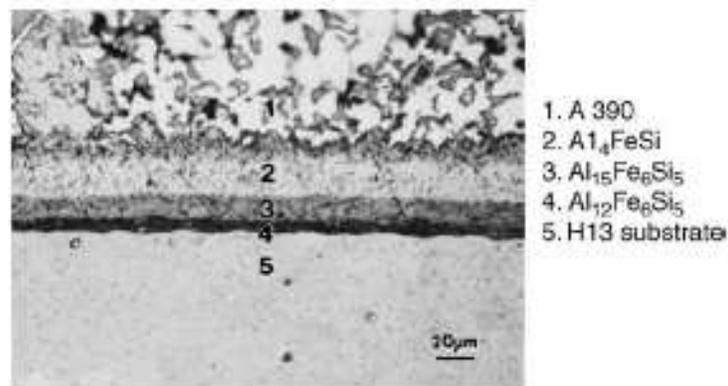
1. Techniques for measuring die soldering. Previous studies have focused either on laboratory studies in which the die casting environment is simulated or involve special dies designed specifically for the purpose of studying soldering. In contrast to these approaches, a method for studying and measuring die soldering on parts currently in production was researched. Without a method for measuring soldering accurately, there is no way to control it. An accurate measurement technique is also needed for validation of predictive models.
2. Prediction of die soldering during the die design phase. Contech currently uses MAGMAsoft to model the design of their new dies in order to minimize porosity, poor fills and other defects as well as to optimize the cooling channel design. If it were possible to predict the location and severity of die soldering before the dies are manufactured, the ability to reduce many of the problems associated with soldering by a die redesign, the use of inserts, selection of alternate material, or any other method which is available or may become available in the future, would be greatly enhanced.
3. Modification of casting alloys with strontium in order to reduce soldering. Strontium, by altering the surface properties of die casting alloys in very small concentrations, reduces the ability of the cast aluminum to wet the die steel, thus reducing the apparent contact area between that cast alloy and the die surface and the interfacial reaction leading to soldering. Research was undertaken to quantify the effects of strontium and to understand under which conditions it is most effective at reducing solder, as well as to document any other effects of strontium to the process.

## II. Literature Review

A background on what is known about soldering, specifically the mechanism, contributing factors, is given here, as well as a review of what has been attempted in the past to prevent it or reduce its effects. A fundamental background is necessary for making accurate decisions and for successful solutions to be developed.

### II.1 Die Soldering Mechanism

An excellent representative image of the type of structure that most commonly results from the interaction between a liquid aluminum alloy and an H13 steel substrate is given by Joshi [1], and displayed in Figure 1. In the image, the aluminum alloy (390) is shown at the top, with layers of different intermetallic phases, progressively higher in iron content, proceeding towards the die steel substrate at the bottom of the image.



**Figure 1: Intermetallic layers formed after dipping H13 steel for 2 hours in liquid aluminum (390) at 680. Joshi [1]**

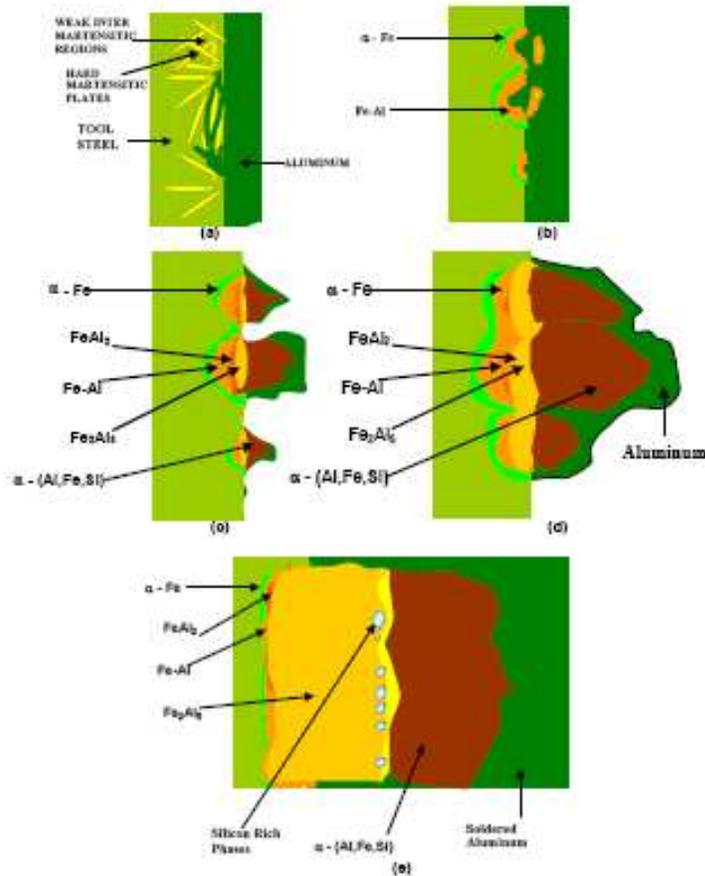
As the compositions of the aluminum alloy and the steel vary, the intermetallic phases can change in both composition and ratio, but the general structure is almost always very similar to that shown above. The structure is similar in the case of other die materials as well, with iron replaced by the predominant component in the die material.

The most comprehensive study of the mechanism of die soldering and the factors that affect it was carried out at WPI by Sumanth Shankar as his Ph.D. dissertation entitled "A Study of the Interface Reaction Mechanism Between Molten Aluminum and Ferrous Die Materials." [2]

Shankar suggested a six-stage mechanism which occurs as die soldering progresses. The mechanism is described as follows:

1. Erosion of grain boundaries at the die surface. Upon introduction to the die, the aluminum melt begins to attack the soft intergranular regions between the martensitic plates and the carbide particles. These areas are eroded, resulting in a solid solution of aluminum and iron atoms.
2. Pitting of the die surface. Hemispherical pits form on the die surface due to the erosion and high drag forces at the surface due to high gate velocity.
3. Formation of iron-aluminum compounds. Compounds such as FeAl, FeAl<sub>2</sub>, Fe<sub>2</sub>Al<sub>5</sub> and FeAl<sub>3</sub> form at the surface of the pits. These phases grow as the aluminum is continuously renewed with each shot.
4. Formation of "pyramid" shaped structures of intermetallic phases. The silicon in the melt reacts here to form  $\alpha$ -(Al,Fe,Si), which has a pyramid-like morphology. This phase grows preferentially, having the largest resulting thickness. The growth of the intermetallic layers is governed by diffusion of iron through the already formed intermetallic phases.
5. Adherence of aluminum onto the "pyramids" of intermetallic phases. The aluminum sticks to this layers due to the arrest of the ongoing reaction between the Al, Si and Fe. Lower thermal conductivity of the intermetallic phases may also contribute to this sticking.
6. Flattening of erosion pits and intermetallic phases. With time, the intermetallic phases widen and join, resulting in a straight interface at the surface. At this point, the  $\eta$ -Fe<sub>2</sub>Al<sub>5</sub> phase grows preferentially. It was noted that most soldered microstructures showed a 1:5 ratio between the thickness of the intermetallic layer and the soldered aluminum.

A schematic picture of the mechanism was also created to visually display the proposed mechanism, shown in Figure 2.



**Figure 3.1:** Schematic of the mechanism of die soldering. (a) Initial attack of the grain boundaries by aluminum to loosen up the hard grains and martensitic plates to cause pitting on the die surface. (b) Formation of the initial iron-aluminum intermetallic phases inside the pits and around the broken grains close the die surface. (c) "Pyramid" growth of the ternary  $\alpha$ -(Al,Fe,Si) phase on the pits over the  $\eta$ -Fe<sub>2</sub>Al<sub>5</sub>. In addition, the pits expand laterally and in depth. Aluminum begins to stick after this layer structure is formed resulting in the beginning of soldering. (d) Shows the growth of intermetallic layers and merging of neighboring pits. Molten aluminum encounters the die surface.

**Figure 2:** Schematic illustration of the die soldering mechanism proposed by Sumanth Shankar in his Ph.D. dissertation [2].

In an alternate form, Shankar also created a flow chart to illustrate the stages that occur in die soldering of H13 steel by aluminum alloy 390, shown in Figure 2.

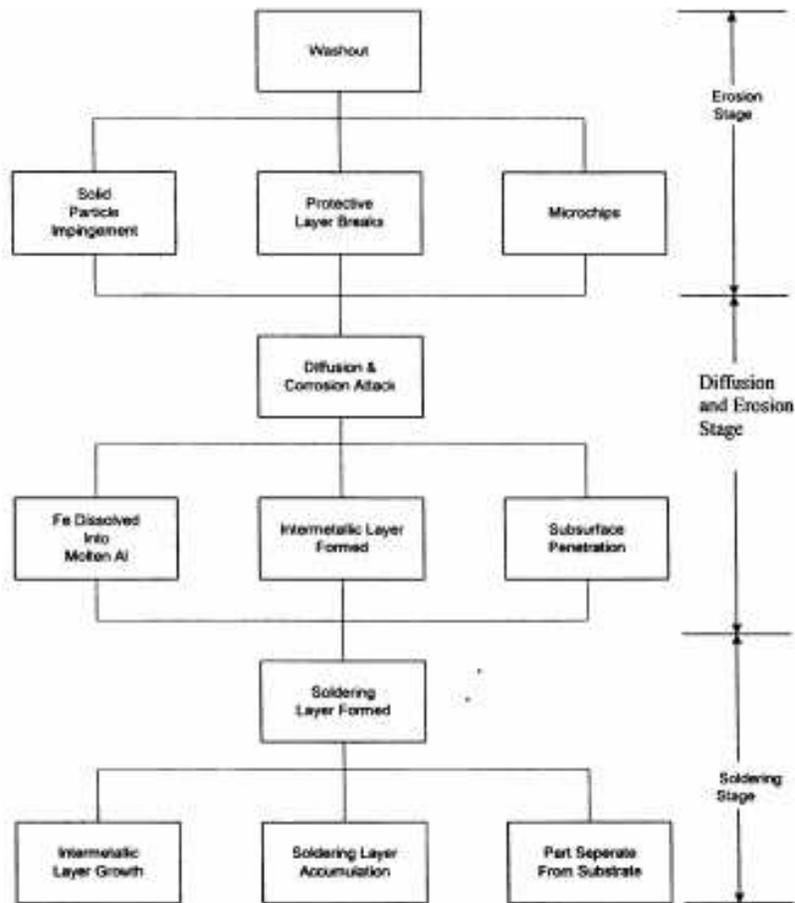


Figure 3: Stages of die soldering of H13 steel by aluminum 390 alloy created by Shankar [2].

A series of chemical reactions which may potentially occur between the cast aluminum and the die steel to cause soldering to occur are listed as follows [2]:

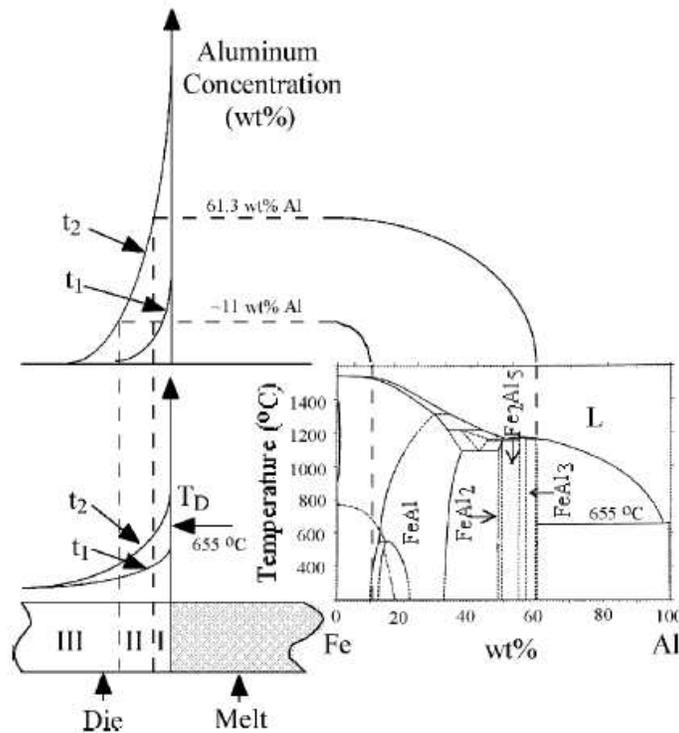
1.  $\text{Fe(s)} + \text{Al(l)} \rightarrow \alpha\text{-Fe(s)}$
2.  $\alpha\text{-Fe(s)} + \text{Al(l)} \rightarrow \beta\text{-FeAl(s)}$
3.  $\beta\text{-FeAl(s)} + \text{Al(l)} \rightarrow \zeta\text{-FeAl}_2\text{(s)}$
4.  $\zeta\text{-FeAl}_2\text{(s)} + \text{Al(l)} \rightarrow \eta\text{-Fe}_2\text{Al}_5\text{(s)}$
5.  $\eta\text{-Fe}_2\text{Al}_5\text{(s)} + \text{Al(l)} \rightarrow \theta\text{-FeAl}_3\text{(s)}$
6.  $\theta\text{-FeAl}_3\text{(s)} + \text{Al-Si(l)} \rightarrow \alpha\text{-FeAlSi(s)} + \text{Al(l)}$

The thermodynamic quantities associated with the formation of these compounds at 700C, as given by Richards [3] and compiled by Shankar:

**Table 1: Thermodynamic Quantities associated with the formation of various Fe-Al intermetallic phases\***

PHASE	$\Delta H_{273K}^{\circ}, J.mol^{-1}$	$\Delta S_{273K}^{\circ}, K^{-1}.mol^{-1}$	$\Delta G_{973K}^{\circ}, J.mol^{-1}$
$\alpha$ -FeAl	-51240	51.0	-11090
$\zeta$ -FeAl <sub>2</sub>	-81900	-73.3	-16999
$\eta$ -Fe <sub>2</sub> Al <sub>5</sub>	-194040	-166.7	-19636
$\theta$ -FeAl <sub>3</sub>	-112560	95.6	-22.869

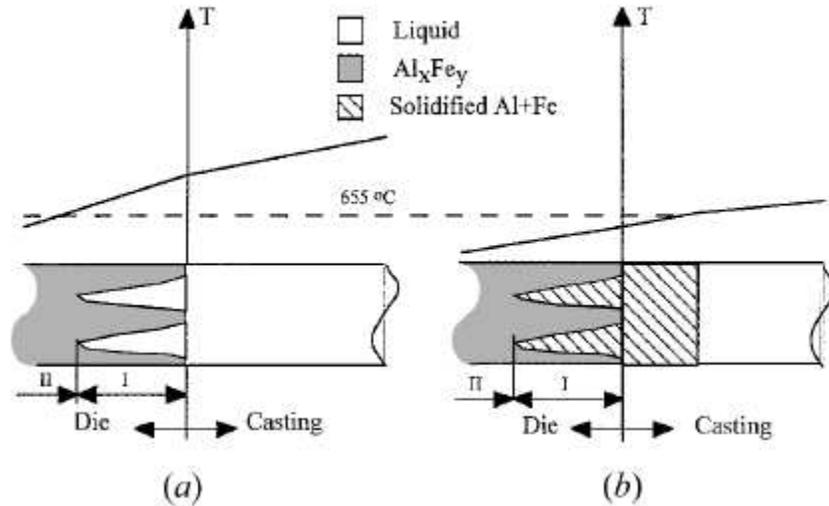
Another detailed analysis of the mechanism of die soldering was conducted by Q. Han and S. Viswanathan at the Oak Ridge National Laboratory in Oak Ridge, TN [4]. They suggest that as an aluminum gradient becomes present at the surface of the die, various intermetallic phases form, depending upon the iron-aluminum phase diagram. This results in three distinct regions at the surface, shown in Figure 4 below.



**Figure 4: Schematic illustration showing possible aluminum composition and temperature profiles at the die surface, and the resulting phases for the case of pure aluminum cast in an iron die. Three regions at the die surface can be identified. Soldering occurs in region I. [4]**

Of the three regions, region I, directly situated at the interface, is the critical region, as it contains Al-rich intermetallic phases, largely FeAl<sub>3</sub>. FeAl<sub>3</sub> has a melting temperature of 655C, often lower than the casting temperature of the aluminum alloy. When the temperature in Region I exceeds 655C during the cycle, the intermetallic

phases melt and liquid is present at the surface. When these phases melt again, they can grow out into the melt and join the casting to the die or, as shown in Figure 5.



**Figure 5: Schematic Illustration of the mechanism of die soldering for the case of pure aluminum in a pure iron die, indication (a) the presence of liquid at the die surface when the temperature at the surface is higher than a critical temperature, 655C and (b) the formation of a joint between the casting and the die on solidification of this liquid phase. [4]**

The authors also propose the concept of a critical soldering temperature, above which die soldering will occur. This critical temperature is the die-surface temperature above which a liquid phase can form. In the case of pure aluminum solidifying in pure iron, this would be 655C, or the temperature of the eutectic reaction between Al and FeAl<sub>3</sub>. This temperature varies, however, as the composition of the aluminum alloy and the die steel changes as other components are introduced into the system.

Hanliang [5], et al. proposed an energy criterion for soldering:

$$\frac{A_r}{A_a} \geq \frac{2\gamma_\alpha}{\gamma_\alpha + \gamma_\beta - \gamma_{\alpha\beta}}$$

Where  $A_r$  = real contact area between die and casting

$A_a$  = apparent contact area between die and casting

$\gamma_\alpha$  = surface energy of the casting

$\gamma_\beta$  = surface energy of the die

$\gamma_{\alpha\beta}$  = interfacial energy between the casting and the die

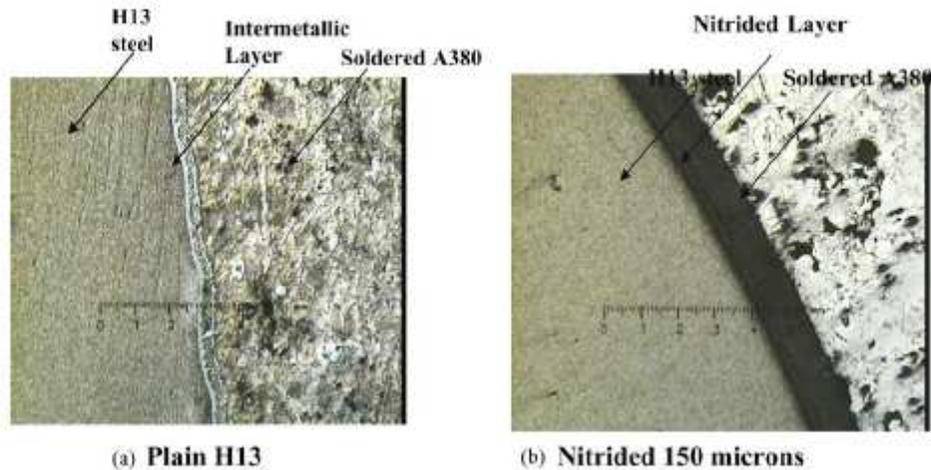
This is based on the theory that the energy required to separate the soldering interface is:

$$W_1 = A_r (\gamma_\alpha + \gamma_\beta - 2\gamma_{\alpha\beta}).$$

And the work required to divide the aluminum casting into two parts is:

$$W_2 = 2A_a\gamma_\alpha.$$

Soldering has been found in some studies in the absence of the formation of intermetallic layers. For example, Figure 6 shows an image of a soldered sample on the left with visible intermetallic layers. However, on the right is an image of nitrided H13 with soldered aluminum. The nitrided band is visible, but no intermetallic phases can be seen.



**Figure 6: Example of two forms of soldering. On left, soldering with intermetallic layers. On the right, soldering has occurred in the absence of intermetallic layers. [1]**

## ***II.II Factors Affecting Die Soldering***

### **II.II.I Temperature**

Temperature at the surface of the die is among the most important variables promoting die soldering. Higher temperatures increase the activity of surface atoms, increase diffusion coefficients and increase reaction rates, all of which will cause die soldering to occur sooner and progress faster. High temperatures can also have a tempering effect on the die surface and promote washout and soldering by softening the surface.

Anecdotally, die temperature is a very important factor contributing to die soldering. Regardless of other steps taken, if the die is inadequately cooled, soldering will likely be a significant problem. Therefore, care should be taken to design the cooling system in a die very carefully so that the temperature can be minimized and solder reduced.

Tsuchiya et al. [6] recorded the temperature in a 7-pin experimental cavity and showed that the pins which experienced the highest temperatures during a cycle were also the first pins on which soldering was observed. This behavior can be noted in Figure 7 and Figure 8.

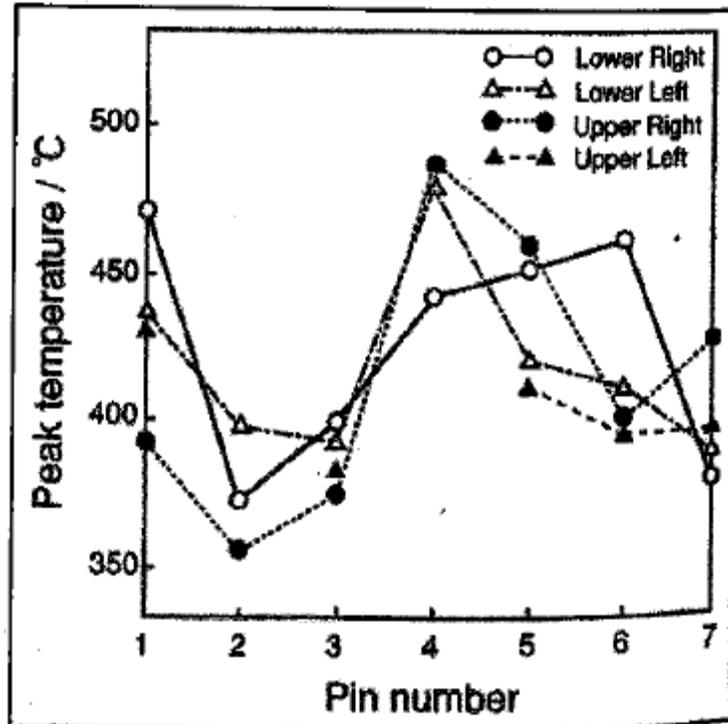


Figure 7 - Maximum temperatures seen at each of seven pins in the experimental die used by Tsuchiya et al. [6]

Soldering	Pin#	Numbers of shots				
		50	100	200	400	2000
Film-like	1	N, N-S, TiN, CrN, TiC	VC, CrC			
	2&3		N	H, TiN, TiC, VC, CrC, CrN		
	4	H, N, N-S, TiN, TiC, VC, CrC, CrN				
	5&6	H, N	TiN, TiC, VC, CrC	CrC		
	7		H, N, TiN, TiC, CrC, CrN	VC		
Massive	1	H, N, N-S, TiN, TiC, VC, CrN			CrC	
	2&3				H, N, CrN	VC
	4	H, TiN, TiC, CrC	N	N-S, CrN	VC	
	5&6			H, N	TiN, TiC, VC, CrC	N-S
	7			H, TiN, TiC, CrC	N, VC	

*Remarks: N: Nitriding, N-S: Nitrosulphuring, TiN: CVD-TiN, TiC: CVD-TiC,  
VC: TRD-VC, CrC: TRD-CrC, CrN: TRD-CrN, H: Hardened*

Figure 8 - Number of shots to observation of soldering in pins with maximum temperatures shown in Figure 7. Note that pins 2&3 which show the lowest maximum temperature are the last to develop soldering and vice versa for pin 4. [6]

In an accelerated erosion test, Shivpuri, et al. [7] examined the difference in soldering and erosion behavior at two casting temperatures, 650 and 700C. As expected, much more soldering was observed at the higher temperature. However, somewhat counter-intuitively, erosive wear was greater at the lower temperature. Chu, et al. [8] found similar results where erosive wear was greater at a casting temperature of 621C than at a casting temperature of 677C or 732C. Shivpuri explains this interesting result by noting that at 650C there is a considerable solid fraction at the time of casting, and the solid particles in the melt will lead to enhanced erosive wear.

One of the leading factors contributing to localized hot spots is the geometry of the cast part. In locations where it is necessary to have a piece of steel surrounded by cast metal, soldering will likely be a problem. These locations will get very hot due to the liquid aluminum transferring heat into the localized area from many directions, and also because cooling cannot be placed close to the surface due to thermal stress considerations. In the following work on the U222 Carrier, it will become apparent that any convex region in the die with a small radius of curvature typically will experience the significant soldering during a long cycle. Unfortunately, the part design is typically not something that can be altered in order to minimize soldering.

### II.II.II Effect of Alloying Elements

It is known that different aluminum alloys have different soldering behavior due to the effects of alloying elements. Kajoch et al. [9] tested the die soldering tendencies of several die casting alloys using a friction welding set-up which tested the load necessary to join an aluminum ball with a steel ring. The more load required to join the two indicates that the aluminum alloy is more resistant to soldering, since the reaction between the two metals occurs less easily.

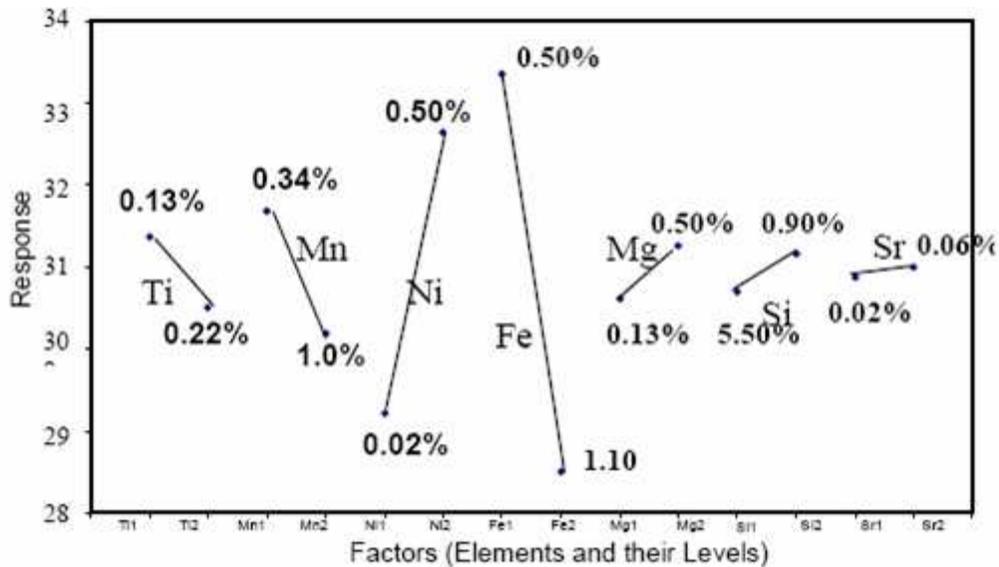
The results of this experiment showed that primary aluminum has the highest tendency for soldering, followed by Al-Mg alloys, hypoeutectic Al-Si, Al-Si-Cu alloys and finally eutectic Al-Si has the least tendency for soldering. The results are given in Table 2.

**Table 2 - Results of the friction welding experiment performed by Kajoch [9]. Higher loads represent a higher resistance to soldering.**

Alloy Group	Alloy	Load (N)
Al	Al99.6	2600
	Al98	2650
Al-Mg	AlMg5Si (515)	2675
	AlMg10 (520)	2850
Al-Si-Cu	AlSi12Cu2 (319)	2775
	AlSi12Cu2 (383)	3550
Al-Si	AlSi9 (408)	3250
	AlSi12 (413)	3650

However, since die soldering is generally not a contributing factor to the selection of an alloy (mechanical properties and cost are much more important factors), the most interesting work for practical applications is in studying the effect of individual elements. Knowledge developed in this area can help to optimize individual alloys within specifications.

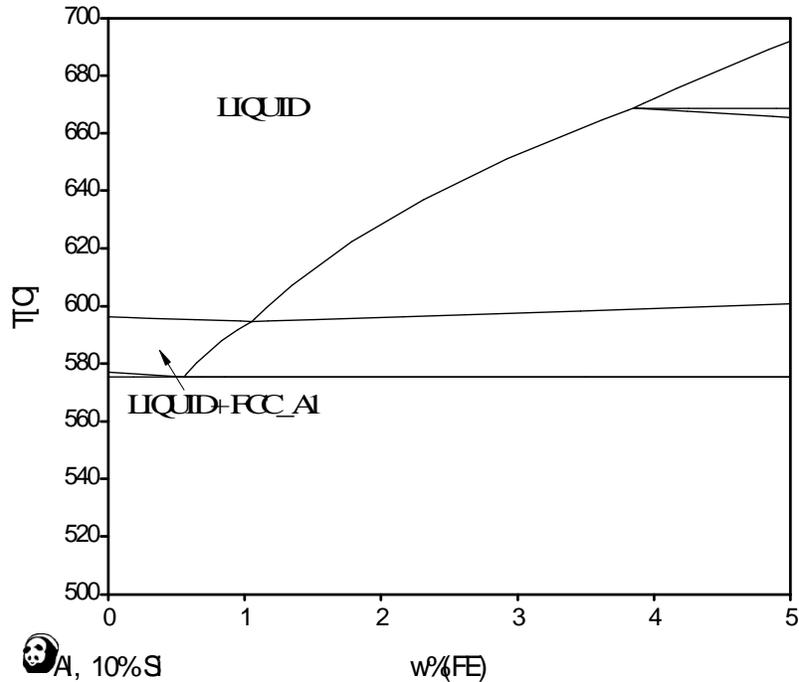
In addition to studying the mechanism of soldering, Shankar [2] also conducted a study regarding the influence of a set of alloying elements over a set range of values. The results are summarized in a main effects plot in Figure 9.



**Figure 9: Effects of various elements on the thickness of the intermetallic layer in the reaction between H13 and molten aluminum. [2]**

The results show that the elements that most strongly affect the development of the intermetallic layer are iron, which slows the growth of the intermetallic layer, and nickel, which promotes the growth of the intermetallic phases.

It is widely known in die casting practice that the level of iron plays an important role in die soldering. As Figure 10 indicates, Iron has solubility in Al+10%Si of ~2-4% at casting temperatures. This solubility drops to ~1-2% in the temperature range where the alloy is in contact with the die surface and the reaction can occur.



**Figure 10: Phase diagram of Aluminum + 10% Silicon as Iron is increased.**

Thus, even small additions of iron reduce the chemical potential gradient for iron from the die material to the liquid alloy, slowing the rate of any soldering reaction. Chu [8] notes that the Al-Fe-Si eutectic reaction occurs at about 0.8% iron. Additions of iron at this level or above can significantly reduce the occurrence of soldering.

The drawback of iron additions to alloys is the deterioration of mechanical properties associated with iron intermetallic phases which form during solidification. As Holz [10] shows in Figure 11, the strength, and especially the elongation, of aluminum alloy 380 deteriorate with increasing iron content.

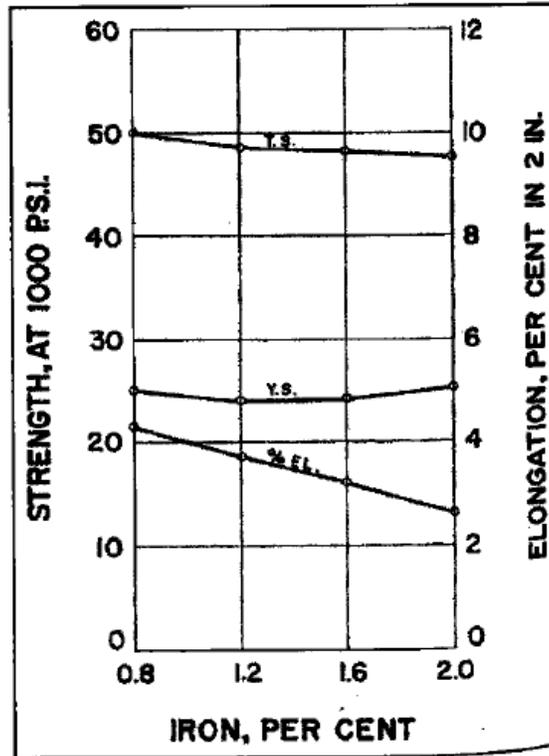
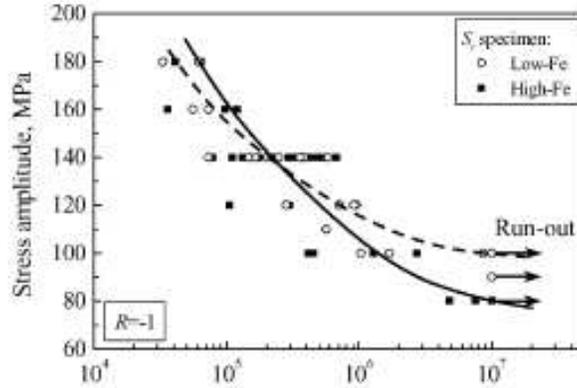
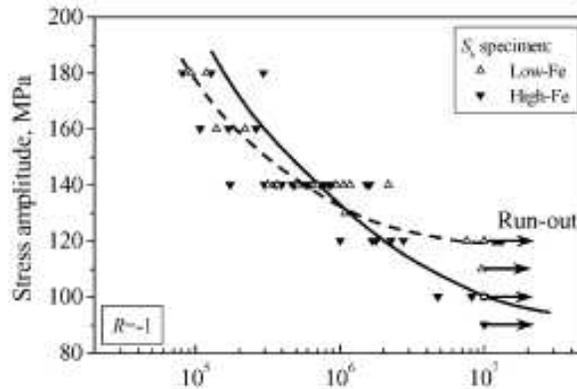


Figure 11: Loss of mechanical properties with increasing iron content of aluminum alloy 380. [10]

Iron additions can also have an effect on the fatigue life of alloys. Yi [11] studied this and made the conclusions that iron (at 0.57% rather than 0.06%) reduces the fatigue life in the long lifetime regime, while slightly increasing the fatigue life in the short lifetime regime. It was also found that large plate-like Fe-rich intermetallic phases promote crack initiation. Graphs depicting these results are shown in Figure 12 and Figure 13.



(a)



(b)

Figure 12: S/N curves for Low-Fe (0.06%) and High-Fe (0.57%) samples. [11]

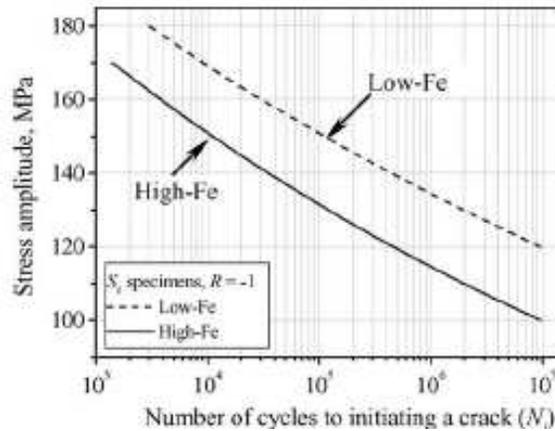
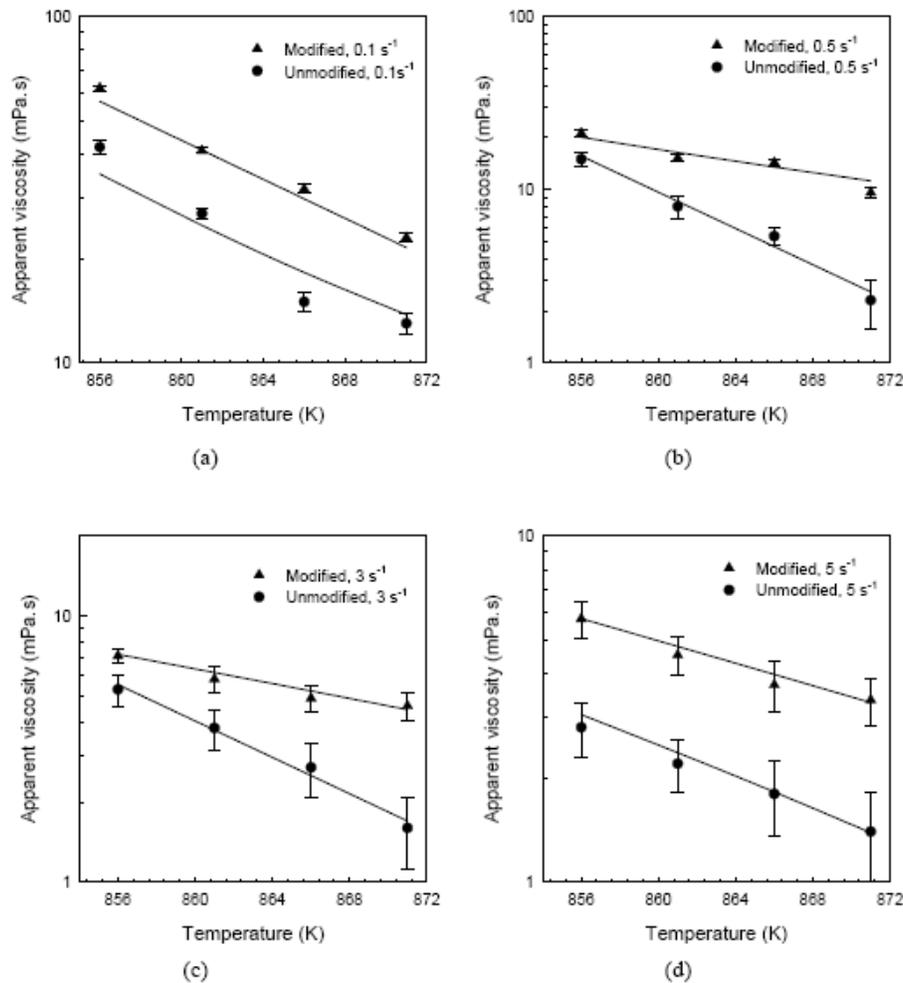


Figure 13: Crack initiation life for Low-Fe (0.06%) and High-Fe (0.57%) calculated from an FEA initiation model. [11]

Due to these factors, a good deal of current work, led by Mercury Marine, is focused on developing alloys that can resist die soldering while having low iron content. This work has been focused on the use of strontium as an alloying element.

As was studied and documented by Shankar and Makhlouf at MPI, Strontium significantly alters the apparent viscosity of the aluminum alloy, as seen in Figure 14, and consequently changes the surface tension and the interfacial energy of the system. According to the theory, these changes reduce the ability of the alloy to wet the die steel and thus less interaction (and subsequently, less soldering) occurs at the surface.

It is interesting that in the experiments that Sumanth Shankar performed, the effects of Strontium were minimal (see Figure 9). This is likely because there is a practical upper limit to the effectiveness of strontium at reducing soldering. In practice, a Sr level of 0.02 wt% is adequate. Therefore, at the two levels that Shankar chose (0.02 and 0.06 wt %), the difference is not apparent. However, had he chosen 0% as one of the tested levels, the effects likely would have been much more significant.



**Figure 14: Apparent viscosity of unmodified and Sr-modified (0.023 wt%) Al-Si alloys as a function of temperature. (a) At a shear rate of  $0.1 s^{-1}$  (b) At a shear rate of  $0.5 s^{-1}$  (c) At a shear rate of  $3 s^{-1}$  (d) At a shear rate of  $5 s^{-1}$ . Makhlouf, Shankar [12]**

The surface energy change also affects how the eutectic structure forms. The typically long plates of silicon are refined to smaller particles. This refinement is beneficial in terms of mechanical properties. Makhlouf and Shankar [12] attribute this to the change in interfacial energy, which does not allow the eutectic structure to nucleate on the primary Al dendrite. Instead, aluminum grains nucleate in the eutectic liquid, and the silicon is required to grow between these grains and acquire the fibrous morphology observed in modified alloys. This modification can greatly improve mechanical properties.

This phenomenon has been shown to alter the eutectic nucleation temperature of alloys. The following data collected by Shankar and Makhlouf [12] graphically represent this phenomenon (Figure 15).

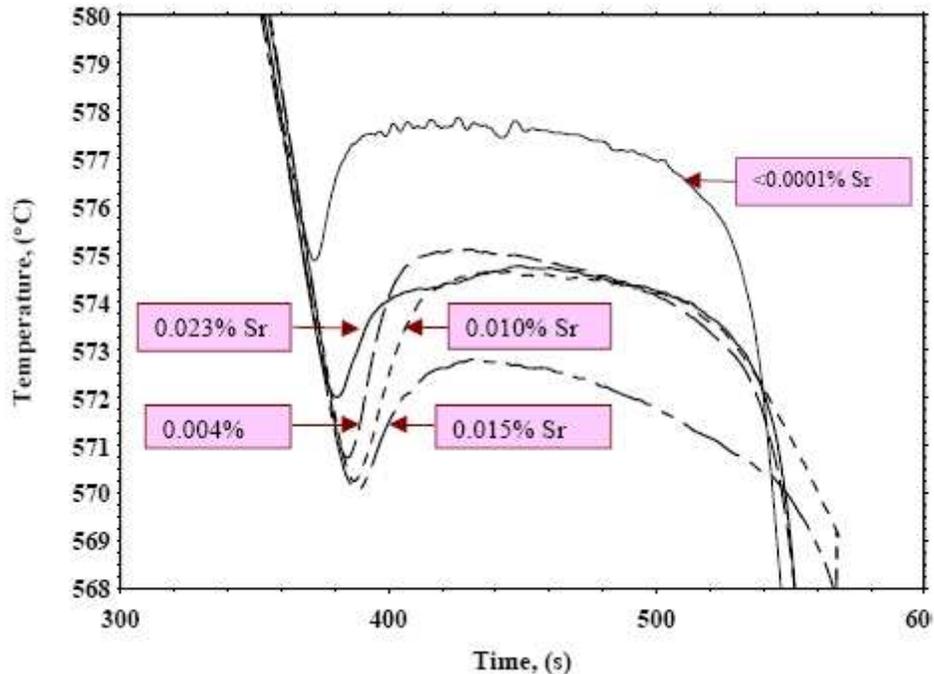


Figure 15: Effect of Sr concentration on the eutectic nucleation temperature of Al-Si alloys.\*

Kopper and Donahue [13] give some theoretical analysis to this surface energy change by analyzing the thermodynamics of the two surfaces in contact by using the Gibbs adsorption equation, which gives an excess surface concentration of a solute in a two-component system.

Their study found that at a concentration of 230 ppm, an imperfect monolayer with 83.4% coverage is formed at the surface. Mercury Marine is currently developing two new alloys, named Mercalloy 366 and 367, which have strontium concentrations in

the range of 500-1000 ppm. At these concentrations, there is potential for a significant reduction in iron content since the strontium content can reduce soldering and the need for iron is therefore reduced. Indeed, the new alloys have iron concentrations of 0.2 and 0.25 wt%, respectively. These levels are much lower than typical die casting alloys with iron contents in the range of 0.6-1%.

However, strontium also potentially can increase or alter the formation of porosity in the resulting structure. Anson, et al. [14] performed a detailed study of the effects of strontium on porosity in A356. The authors' data and findings are as follow.

An increasing amount of strontium results in increasing percent porosity, as shown in Figure 16. This increase continues beyond the limit of any modifying effect on the microstructure.

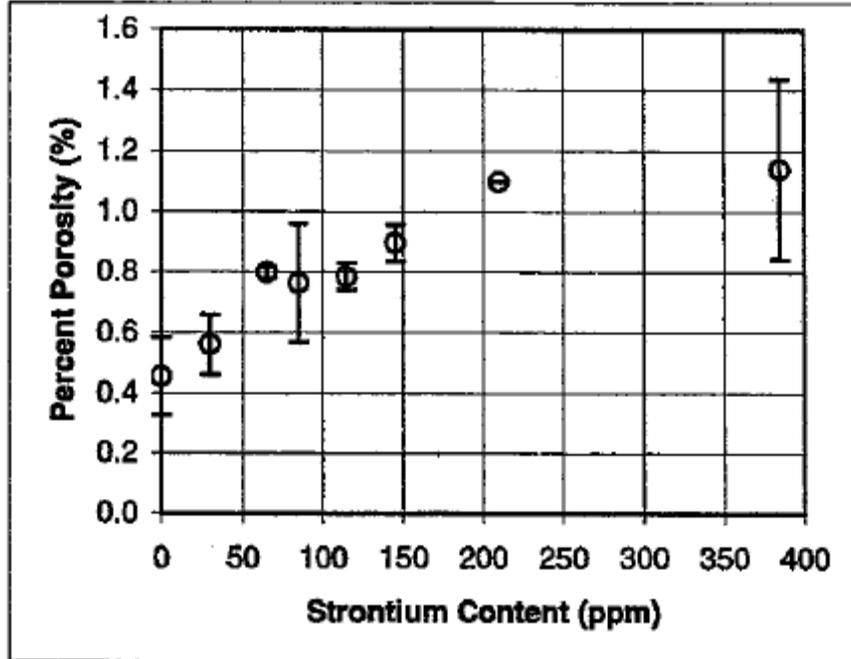


Figure 16 - Effect of strontium content on percent porosity. [14]

As the percent porosity increases, the pore density (number of pores) falls, as seen in Figure 17. This can be explained by a reduction in shrinkage porosity since strontium helps to enable feeding through modifying the eutectic structure, as it is clear that the reduction in pore size only occurs up to ~100 ppm, after which an additional Strontium addition has no modifying effect.

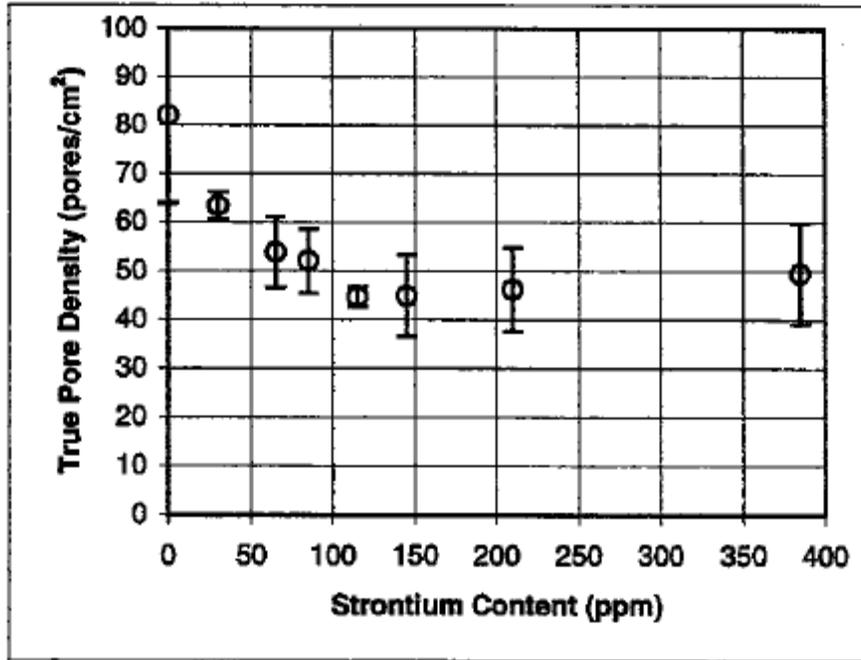


Figure 17 - Effect of strontium content on pore density. [14]

This means that the average pore size must increase to accommodate these other changes (Figure 18).

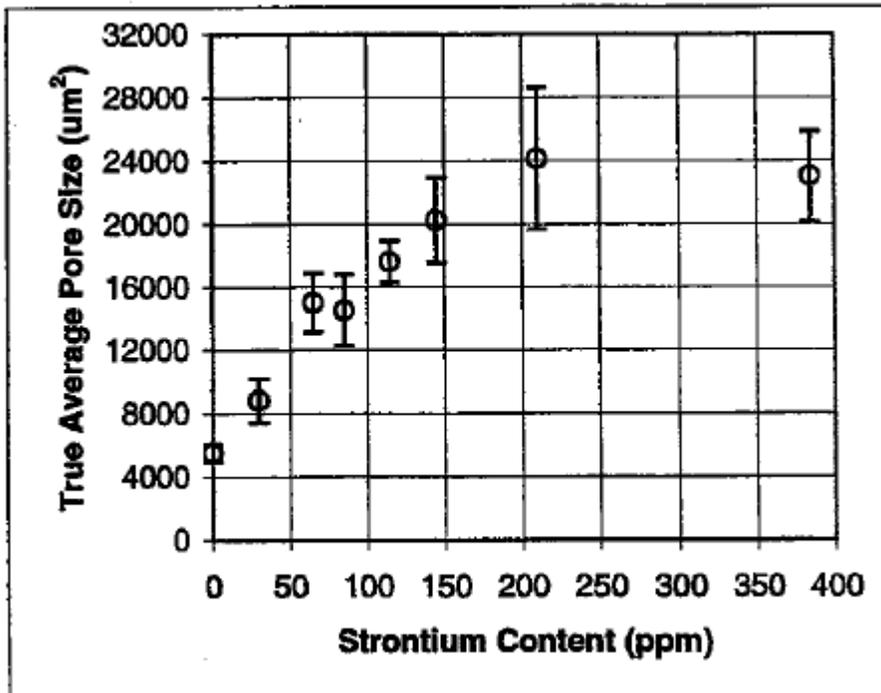


Figure 18 -The effect of strontium content on the average pore size. [14]

This increased pore content may potentially have a negative effect on the fatigue life of any component cast with strontium modification at the strontium levels required for any decrease in soldering behavior. One mitigating factor, however, may be that the pores become much more spherical with a strontium addition [14].

Contech has prior data that supports that Strontium modification may actually result in improved part quality. The benefits of the modified structure outweigh the negative effects of increased and/or modified porosity. Due to the existence of this prior data, no mechanical testing was performed on any parts that were modified with strontium during this thesis, so no data are presented.

The effect of strontium in creating more gas porosity may change with the nature with which the strontium addition is made. Guthy et al. [15] showed that the amount of hydrogen soluble in an aluminum melt drops significantly upon immediate addition of Sr to the melt. However, after some time, the hydrogen levels rise back to their normal levels. Therefore, to take advantage of this phenomenon the authors suggest that castings be made with the modified alloy shortly after the strontium addition. An example of this behavior is given in Figure 19.

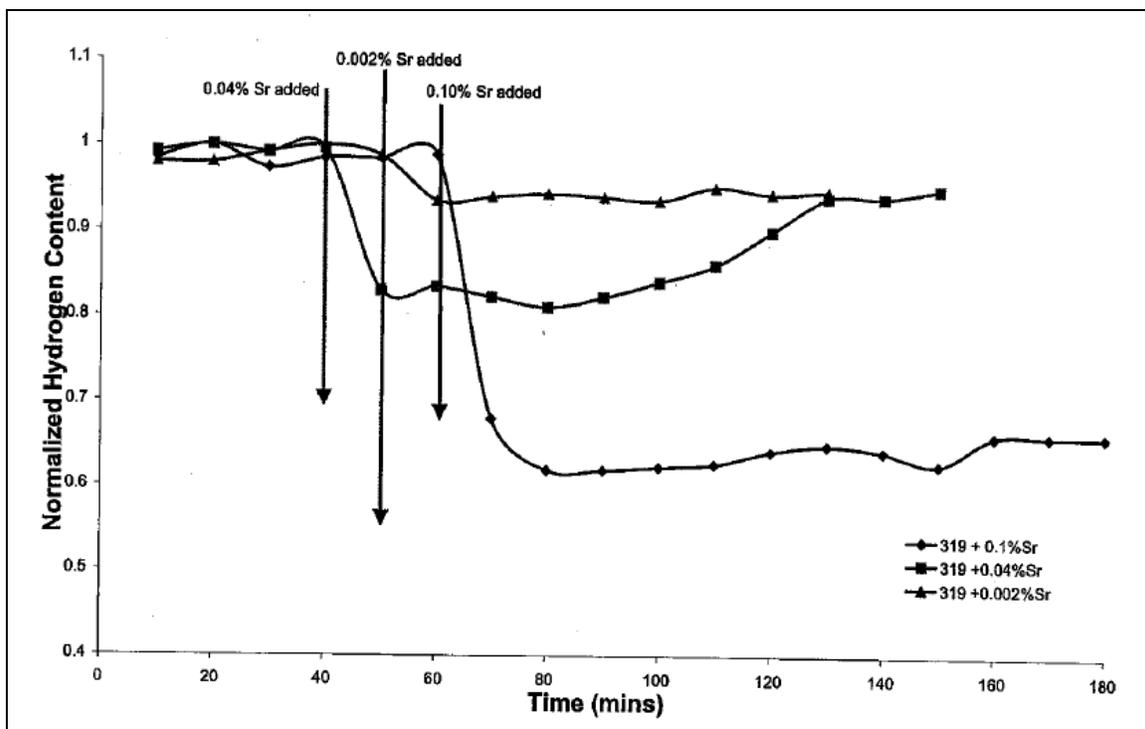


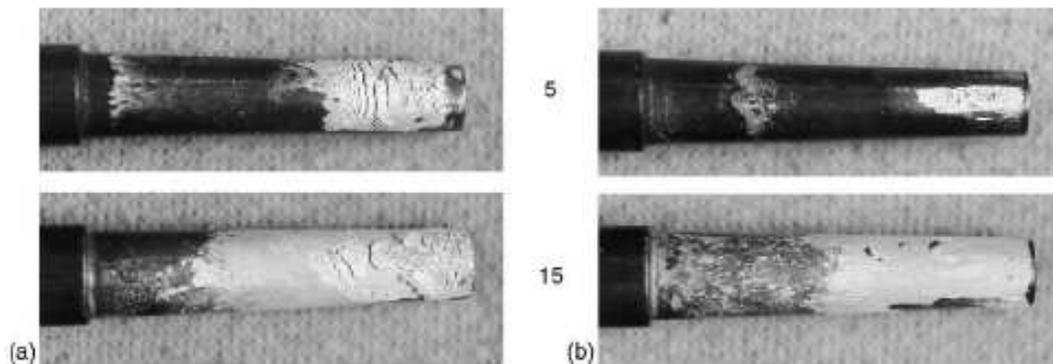
Figure 19 - Variation of hydrogen level in aluminum A356.2 alloy upon addition of strontium. [15]

The authors give several potential reasons for their discovery. They postulate that the increase in strontium may cause strontium hydride to form. Alternately, the decrease in surface tension which is a result of strontium modification may encourage the development of hydrogen gas bubbles over dissolved hydrogen.

### II.II.III Gate Velocity

As noted in Shankar's [2] mechanism, die erosion and pitting is one of the first steps leading to die soldering behavior. A high gate velocity accelerates this erosion and subsequently leads to soldering. Chu et al. [8] found that wash out of die pins is the primary factor in the initiation of die soldering in the case of 390 Alloy.

Besides simply the overall die design, another way that gate velocity manifests itself in the case of pins is the side of the pin which faces the gate. Chen [16] has shown a significant difference in the amount of soldered material built up on the surface of pins depending on which side of the pin is facing the gate. This can be seen in Figure 20. This image is also a good example of the appearance of a soldered pin at the macro scale.



**Figure 20: Influence of pin fixturing in the die. The side of the pin facing the gate (a) shows significantly more soldering than the side diametrically opposite the gate (b). Chen [16]**

This is typically a less significant factor in squeeze casting where the gate velocity is much lower than in high-pressure die casting, but it is still a consideration. Locations that the cast metal impinges upon will still erode faster than other areas of the die. However, this erosion will be less than is seen in the HPDC process. These areas will also experience greater heat transfer due to increased turbulence in the flow around the die.

#### **II.II.IV Die Surface Condition**

The surface condition of the die is a critical factor in the prevention of soldering. It is known that on a large scale, areas of the die which protrude into the cast alloy such as cores and pins are much more likely to solder due to their higher temperature. At a micro scale the same phenomenon will occur. On a rough surface, the small protrusions will experience a higher temperature than any local area on a smooth surface. Thus, the surface should be smooth, but not below the limit after which lubricant will not adhere to the surface.

The surface condition can be preserved by good cleaning techniques and by surface modifications and coatings which harden the steel at the surface and slow erosion from occurring or which can serve as a physical barrier separating the melt from the die.

The surface roughness of the die is also an important factor in the application of lubricants to the surface. If the surface is too smooth, the lubricant may not adhere, and any benefits from the lubricant will not be realized.

#### ***II.III Die Soldering Measurement***

One of the primary obstacles to conducting research relating to die soldering is the fact that it is essentially a concept rather than a specific property or event. There are no agreed upon methods for testing it, as there are for example in testing mechanical properties of a material, which can be easily studied and reported by following ASTM standards.

In designing any experiments to study die soldering, the researcher must first decide what the primary condition is that he/she is trying to prevent. Is it the thickness of the soldered aluminum that is most detrimental or the area that is covered with solder, for example; and how is it that this condition can be measured?

Additional complications arise from the fact that soldering is very closely related to other phenomena such as corrosion, erosion and oxidation. Therefore, it is difficult to decouple the effect of soldering from these to ensure that the measurement method is truly measuring the effects of soldering, not the effects of any these other events. Practically, it is probably impossible to entirely separate these events since, as was shown in the mechanism studies above, erosion and washout contribute significantly to

the onset and progression of die soldering, so it is unlikely that soldering will occur in the absence of these other phenomena.

The early part of the thesis work focuses on attempting to develop a measurement technique that could be applied to later studies. It is salient to first examine the previous measures used in studies to document die soldering behavior in order to discover their benefits and drawbacks.

One of the best quantitative approximations of die soldering behavior is the thickness of the intermetallic layers formed at the interface of the cast aluminum and the steel die material. These layers can be studied by cross-sectioning the interface and examining the layers present. The thickness of these layers should indicate the likelihood for the two materials to react under the specific local conditions (temperature, compositions, pressure) Assuming that this reaction is directly proportional to the severity of soldering, this thickness can be used directly as a measurement for soldering. Indeed, Shankar [2] noted that there was a typically a 1:5 ratio of the thickness of intermetallic layers and the thickness of the soldered aluminum in the samples he examined. There is some evidence that die soldering can occur in the absence of the formation of intermetallic layers [1] (see Figure 6), but this is a rare occurrence and typically not the way in which soldering occurs in industry.

A visual inspection of the die or pin in question can be used as a measure of soldering behavior. For example, some studies document the number of cycles before some benchmark like the visual appearance of soldering on the surface of the die appears. Other times a ranking system has been used in an attempt to introduce more resolution into the system. For example, Lakare [17] used the following ranking system to evaluate the surface of cores in production trials:

1. No visually noticeable soldering
2. Perceivable soldering
3. Significant soldering
4. Excessive soldering

Physical measurement of the stuck aluminum on the die surface is sometimes used in experiments and in the industrial setting in order to quantify an instance of soldering. For example, Tsuchiya [6] used the area percentage of a pin covered with stuck aluminum as well as the maximum height of soldered aluminum as a measure of soldering. Indeed, soldering is sometimes roughly characterized at the Pierceton, IN

plant by a measurement of the area covered by the stuck aluminum or the depth/height of stuck aluminum.

However, Tsuchiya [6] noted that different forms of soldering manifest themselves differently in regards to this measure. As he noted, “massive soldering” typically covers a much smaller area than “film-like soldering”, yet has a much higher maximum height, making it arguably more detrimental to the process, despite covering less area. Additionally a self-cleaning effect has been observed, where the maximum height will fluctuate over a series of shots as the soldering builds up and is then removed by subsequent shots. This makes measurements of soldering height uncertain, since the effect of self-cleaning cannot be known at any one specific shot unless the sampling rate is very frequent.

Some common linear surface metrology parameters have been used to evaluate the extent of soldering and washout (it is very difficult to distinguish between the two in many cases). Chellapilla [18] showed that the Ra value of core pins increased over the course of testing during a production evaluation test. This data is displayed in Table 3.

**Table 3 - Roughness values of pins before and after testing. Adapted from [18]**

Core Pin	Ra Value ( $\mu\text{m}$ )	
	Before Testing	After Testing
Chromium Carbide	0.3521	0.7112
Chromium Nitride	0.4572	1.0000
Titanium Nitride	0.2556	6.0000

As aluminum sticks to the H13 pins, there is a corresponding weight increase of an H13 pin or core, due to the extra aluminum stuck to the surface. Some experimenters have used the weight change of a pin after a series of shots as a measure of soldering [6]. Similarly, in this case it is difficult to isolate the increase in weight due to soldered aluminum with the decrease in weight due to washout. This can be done if the soldered aluminum is removed from the pins by a KOH solution or by hydrochloric acid and the change in weight from washout determined.

#### ***II.IV Prediction of Die Soldering***

Methods for reducing die soldering inevitably require more capital investment in the design and manufacturing of the die. In order to justify this increased cost and to

make the correct decision regarding when to implement expensive solutions, engineers need to have an idea of when solder will occur and its severity.

The industry uses MAGMAsoft casting simulation software to simulate the casting process for each of their new dies before they are manufactured. Using MAGMAsoft, hot spots can be identified and attempts made to minimize these hot spots with alternate cooling channel designs, part configurations, etc. If this does not work, inserts can be designed into the die in order to minimize the economic costs of replacing worn sections of the die. The limitation of this approach is that it is uncertain what the critical conditions are which will lead to soldering, so it is difficult, if not impossible to know when the more expensive countermeasures are justified.

### II.IV.I MAGMAsoft Prediction Overview

MAGMAsoft is a comprehensive simulation tool used for the production of castings. It is used as a way to predict poor fills, porosity, thermal stresses, etc. in order to optimize the design of a part for manufacturability and to optimize the die that the part will be cast in.

A requirement for modeling die soldering in MAGMA is a mathematical die soldering criterion description of soldering that the code can use to generate a prediction. For example, the Niyama criterion is used in order to predict porosity:

$$N = \frac{\sqrt{G}}{\dot{T}}$$

Where G=temperature gradient and  $\dot{T}$ =cooling rate.

Some work has been performed to try and arrive at a die soldering criterion that can be expressed in a mathematical formula. Hianliang, et al. [5] proposed an energy criterion for soldering:

$$\frac{A_r}{A_a} \geq \frac{2\gamma_\alpha}{\gamma_\alpha + \gamma_\beta - \gamma_{\alpha\beta}}$$

Where  $A_r$  = real contact area between die and casting

$A_a$  = apparent contact area between die and casting

$\gamma_\alpha$  = surface energy of the casting

$\gamma_\beta$  = surface energy of the die

$\gamma_{\alpha\beta}$  = interfacial energy between the casting and the die

This is based on the theory that the energy required to separate the soldering interface:

$$W_1 = A_r (\gamma_{\alpha} + \gamma_{\beta} - 2\gamma_{\alpha\beta}).$$

And the work required to divide the aluminum casting into two parts is:

$$W_2 = 2A_a\gamma_{\alpha}.$$

Alternately, the criterion can be expressed as:

$$\frac{A_r}{A_a} = \exp \left[ - \frac{2c\rho(b_M + b_m)\Delta U}{2c\rho R(b_M T_M + b_m T_m) + R b_M c_d^2 \cos^2 \beta P} + \frac{\Delta U}{RT_0} \right]$$

Where:

- $A_r/A_a$  = ratio of real to apparent contact area between the die and the cast alloy
- $\Delta U$  = activation energy of interaction
- $R$  = gas constant
- $T_m, T_M$  = temperature of the melt and mold
- $T_0$  = critical temperature at which the bond is complete
- $\mu, \beta$  = casting velocity and angle to die surface
- $c$  = specific heat of cast aluminum
- $c_d$  = Darcy equation constant ( $\sim 0.8$ )
- $P$  = injection pressure
- $\rho$  = aluminum density
- $b_m, b_M$  = heat accumulation coefficient of the melt and mold

The complexity of this equation would certainly lead to questions about the nature of the results, especially with the number of difficult constants to determine. A simpler solution may be sought, but one was not found in a search of the literature.

## ***II.V Die Coatings and Surface Treatments***

Since soldering results from a reaction between the iron in the die steel and the aluminum and silicon in the cast alloy, one promising method for reducing the occurrence of soldering is to apply coatings or surface treatments to the die that prevent or deter these reactions from occurring.

### **II.V.I Die Coatings**

Die coatings, generally ceramic in nature, are used to physically separate the cast metal from the die surface. Lakare [17] listed the requirements for a successful coating or surface treatment for soldering resistance. The required properties are:

- The coating should be chemically stable in molten aluminum and at the die casting temperatures.
- The coating should be thick enough to prevent direct diffusion of aluminum into the die steel [or iron into the cast aluminum].

- The coating should have high oxidation resistance and should maintain its adhesion and properties at the die casting temperatures.
- The coating should have as little porosity and defects as possible.

Lin, et al. [19] lists another set of requirements:

- The coating should be non-wetting with liquid aluminum.
- The coating should be wear and oxidation resistant.
- The coating should be able to accommodate the thermal residual stresses induced by shot cycling (temperature and pressure) during the pressure die casting process.
- The coating should be adherent to the die material – an engineered interface
- The coating should be able to delay the onset of thermal fatigue cracking (heat checking).

Many studies have analyzed the effectiveness of coatings to the die, due to their excellent promise in mitigating soldering. Common methods for applying a coating to a surface include PVD, CVD, PCVD, TRD and Thermal Spray.

Selection of a coating is not an easy process, as the best coating for a situation strongly depends on the conditions the coating is subjected to, as well as the method with which it is applied. The following is a summary of some of that research.

Chellapilla et al. [18] created a table (Table 4) showing the effectiveness of various coatings at resisting some die casting failure modes which are commonly associated with soldering. Note that virtually all of the coatings perform better than plain H13 steel in most applications.

**Table 4 - Relative Performance of coatings in die casting resistance applications. H-High Resistance; M-Medium Resistance; L-Low Resistance; NC-Not Conclusive; NA-Not Available.**

	CrV (PVD)	VC (TRD)	CrC (CVD)	TiN (PVD)	Metal Life (SuTr)	W (Ion Impl)	Pt (Ion Impl)	Mo (Ion Impl)	H-13
Corrosion	H	H	H	M	L	NA	NA	NA	L
Erosion	H	H	H	H	M	M	L	L	L
Heat Checking	M	NC	M	M	M	H	H	H	L

A simple way to apply a surface coating is through oxidation of the die steel itself. Jahedi and Fraser [20] studied this method by oxidizing H13 steel and Incoloy MA 956. The H13 steel formed a layer of iron oxide, while the MA956 formed a layer of alumina due to the presence of 4.5 wt% Al in the alloy. Both oxide layers prevented the formation of intermetallic layers on pins after 20 casting cycles, although some build-up of soldered aluminum was observed.

In a thermodynamic analysis of several coatings, Hianliang et al. [5] found that a laser melted Mo coating will reduce the soldering tendency of a die, while a WC-Co coating will make the formation of physicochemical soldering impossible. Experimental results supported these theoretical findings, with the die with Mo coating showing a soldering resistance five times that of a typical H13 pin, and the resistance of the WC-Co coated die was 15 times that of a normal H13 pin. Additionally, it was shown that only mechanical soldering occurred on the WC-Co coated die.

Tsuchiya [6] studied the soldering tendencies of H13 pins with various coatings and surface treatments. Most pins coated with TRD-VC, CVD-TiN, TRD-CrC and CVD-TiC were more resistant to soldering than nitrided pins, and in most cases, there was no damage to the underlying pins, even after 10,000 shots.

Shivpuri et al. [7] found that thin wear resistant TiN coatings and carbides of vanadium, boron and chromium significantly improve the erosion resistance of H13.

Chellapilla et al. [18] found that a VC coating was the most successful at reducing corrosion and soldering in a dip-test and also suffered the least wear in an accelerated erosion test, with TiN also performing well. In thermal cycling tests, a CrC coating was found to perform best.

In contact angle studies, Chu [8] studied the work of adhesion between A380 alloy and various coatings and surface treatment. Work of adhesion is defined as

$$W_{ad} = \gamma_{lv} + \gamma_{sv} - \gamma_{sl} = \gamma_{lv}(1 + \cos \theta).$$

The results are given in Table 5.

**Table 5 - Work of adhesion between A380 alloy and various surface coatings/treatments. A higher work of adhesion means a higher tendency for soldering due to better interaction between the two at the surface. [8]**

Surface Treatment	Work of Adhesion
TiN (PVD)	0.384 $\gamma_{lv}$
TiN (CVD)	0.171 $\gamma_{lv}$
VC (TD)	0.268 $\gamma_{lv}$
Nitriding	0.318 $\gamma_{lv}$
CrC (PVD)	0.343 $\gamma_{lv}$
Heat Treated- HRC46	0.344 $\gamma_{lv}$
Heat Treated- HRC52	0.318 $\gamma_{lv}$

Chu also measured the coefficient of friction between A380 and H13 steel treated with various conditions, shown in Figure 21.

Surface Condition	Coating Method	Friction Coefficient @1002g	Friction Coefficient @295g
TiN	PVD	0.45	0.48
TiN	CVD	0.48	0.51
CrC	PVD	0.52	0.55
VC	TD	0.42	0.43
Nitriding	Gas	0.51	0.53
H-13 (HRC46)	Heat Treated	0.49	0.52

Figure 21 - Coefficient of friction between cast aluminum and H13 steel with various surface treatments (see experimental setup below). Results were similar for a hypereutectic alloy. [8]

Carrera et al. [21] studied the wetting angle between three aluminum alloys (360, 383 and 390) and 9 substrates (H13, CrN, TiAlN, Al<sub>2</sub>O<sub>3</sub>, MoZrN, MoN<sub>x</sub>, ZrN, TiN-TiCN and CrC). The results are displayed below in Table 6. Almost any coating can improve the wetting behavior of aluminum over that of plain H13. However, the authors note that it was difficult to make more than general observations with the sessile drop test, so the best coating cannot be identified this way.

Table 6 - Wetting angles between three aluminum alloys and various potential coatings for die casting dies. [21]

Coating or Material	360 Aluminum			383 Aluminum			390 Aluminum		
	Beginning Angle (deg)	Finishing Angle (deg)	Force to Remove Droplet (lbs)	Beginning Angle (deg)	Finishing Angle (deg)	Force to Remove Droplet (lbs)	Beginning Angle (deg)	Finishing Angle (deg)	Force to Remove Droplet (lbs)
H13	80	65	53	111	116	9.5	96	97	110
CrN (P)	126	124	40	126	95	0	122	123	89
CrN (IB)	113	123	58	104	111	2.3	113	120	22
CrC	119	109	0	95	107	11	106	114	8
TiAlN	111	113	0	120	118	1.5	121	122	0
ZrN	113	107	11.5	119	114	93	113	108	20
MoZrN	117	120	0	118	115	0	123	118	2.25
MoN <sub>x</sub>	110	114	0	111	112	0	120	111	0
Al <sub>2</sub> O <sub>3</sub>	95	107	0	115	117	0	114	115	8
TiN-TiCN	96	114	0	118	115	0	111	121	0

Ludtka, et al [22] showed that a boron coating on H13 steel pins, deposited with the Cathodic Arc Technique, reduced the wetting of the pins by molten aluminum, and reduced soldering was seen in a dip test.

Some work is being put into engineering an optimized "coating system" for aluminum pressure die casting. Carrera [21] has defined the architecture of such a coating:

1. A 'working layer' that offers a non-wetting, wear and oxidation resistant surface that interferes with the liquid aluminum.

2. An intermediate multi-layer or functionally graded layer that will minimize the thermal stresses.
3. A thin adhesion layer that improves the adhesion of the coating system to the H13 die surface.

In order for coatings to be widely accepted throughout the die casting industry, some practical issues need to be addressed. These include:

1. Robustness of coating to varying process conditions
2. Ease of coating application and cost
3. Repairability of coatings and coated dies.
4. Susceptibility of coating to nicking, scoring, etc.
5. Coating-lubricant interaction.

### II.IV.II Coating Failure

There are several ways in which a coating may fail while in service. If any defects are present in the coating, as shown in Figure 22, aluminum may come in contact with the H13 substrate, with intermetallics growing under the coating. Eventually, the intermetallics will grow and cause the coating to rupture, as seen in Figure 23.

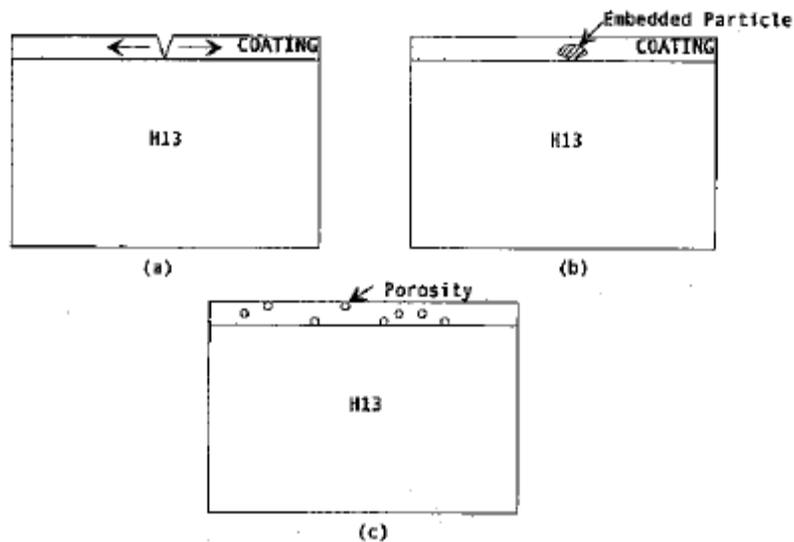


Figure 22 - Defects in a die coating which may lead to coating failure. These include cracks, embedded particles and porosity. [17]

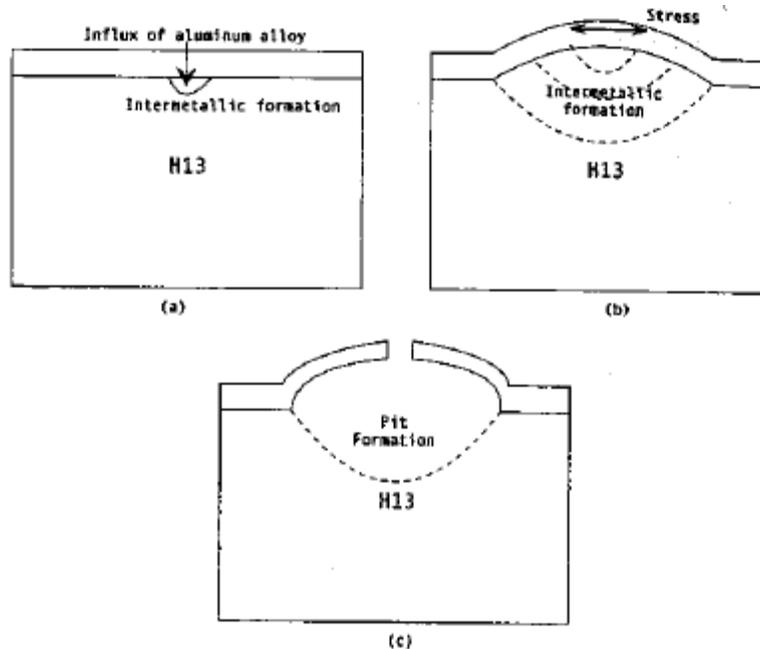


Figure 23 - Schematic of failure of a coating due to intermetallic formation. [17]

A second mechanism of coating failure is through thermal cycling. Due to differences in coefficients of thermal expansion, different stresses in the coating and substrate can cause delamination of the coating. A crack will often form at the delamination site, which penetrates into the substrate and subsequently widens to allow contact between the H13 steel and cast aluminum, where the soldering reaction begins. A schematic of this failure mode is seen in Figure 24.

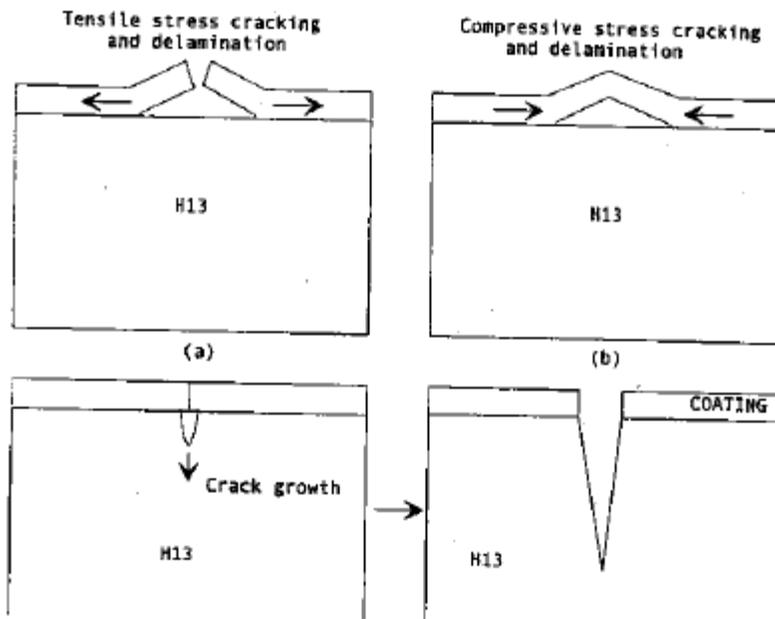


Figure 24 - Schematic of coating failure due to delamination and cracking of the coating. [17]

In four-point bending cyclic fatigue tests, Challapilla [21] found that the cracks which led to failure originated in the substrate below the coating, rather than in the coating at the surface, which would be expected. The authors explain this phenomenon by concluding that the high Young's modulus of the coating shifts the neutral axis of the substrate and leads to higher strains at the bottom of the specimen than at the top.

### **II.IV.III Surface Treatments**

Surface treatments alter the composition of the surface of the die in order to make the surface harder and more resistant to wear so that washout and the initial stages of soldering are prevented or delayed. Common surface treatments include carburizing, nitriding, cold working and work hardening. Currently, the Pierceton, IN plant uses such treatments on the surfaces of many dies.

Kajoch et al. [9] showed that a black oxide treatment applied to the die surface can reduce die soldering tendency. It was shown that a black oxidized die insert withstood 36-40 shots before soldering occurred, whereas plain H13 inserts withstood only 5-7 shots.

Xiaoxia et al. [23] tested the effect of inoculating H13 steel samples with silicon. The study found that inoculation of H13 steel with 0.5% silicon decreases the diffusion rate of Al atoms in the Fe matrix and thus restrains the formation of intermetallic layer. This is attributed to a decreased activity coefficient for aluminum in the Fe-Al-Si phase, which prevents aluminum atoms from moving from the molten aluminum to the steel substrate.

### **II.IV.IV Coatings and Surface Treatments: Conclusions**

In addition to the testing performed in all of these studies, a variety of coatings and surface treatments have been tested at the Pierceton, IN casting plant. Similar to the results of the studies cited above, the results were inconsistent and unconvincing to the tooling engineers. At present, no coatings are used in any of the programs worked on in the plant in the course of this project. The die surfaces are treated with a ferritic nitro-carburizing process, but beyond this no special coatings are used.

## II.V Alternate Die Materials

Despite the fact that H13 steel, and steels in general, have poor soldering resistance, these materials are still predominantly used in aluminum die casting operations. The advantage of using H13 steel is ease of fabrication, toughness, low cost and familiarity.

There is some potential for other materials to be used in casting applications. This replaces the need to coat the steel surface with a material that will not react as readily with the molten aluminum, by replacing the entire die material in a problem area with a material that is less likely to solder.

Shankar [2] studied the material QRO90 extensively in his Ph. D. research in comparison to H13 steel. QRO90 is a modified H13 steel developed and sold by Uddeholm steel. As Figure 25 suggests, QRO90 resists soldering better than H13 steel does.

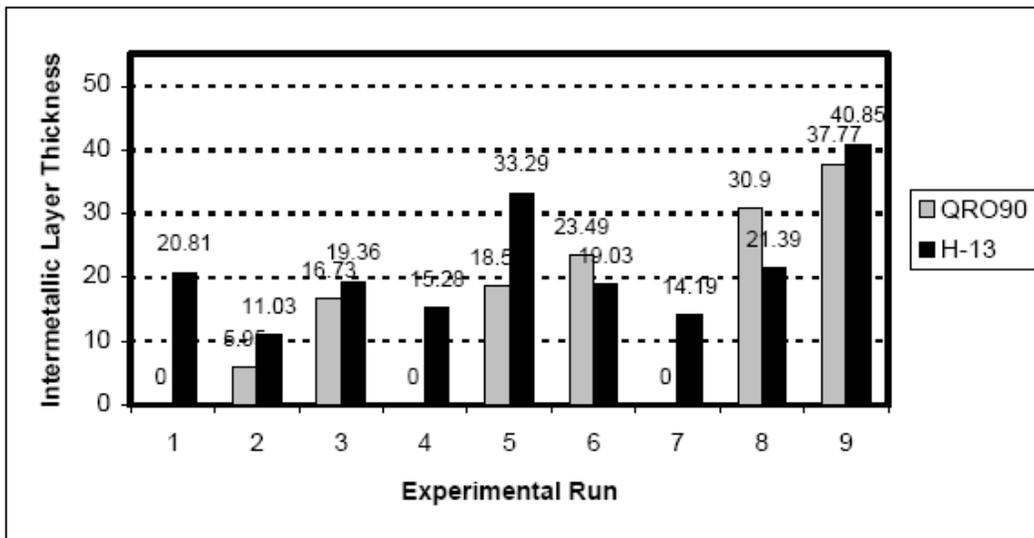


Figure 25: Comparison of intermetallic layer thickness of two die materials: QRO90 and H13. [2]

In addition to QRO90, Shankar lists several other materials that may be used in order to resist soldering:

- CermeTi – Titanium based powder metallurgy composite material.
- ANVILOY – Tungsten based powder metallurgy composite material.
- TZM

Zhu et al. [24] tested a series of advanced metal materials for aluminum die-casting dies in terms of their soldering, washout and thermal fatigue resistance. These materials were: Anviloy1150, H13, Mo-785, Ti-6Al-4V, Ni-718, cast iron and copper based alloys.

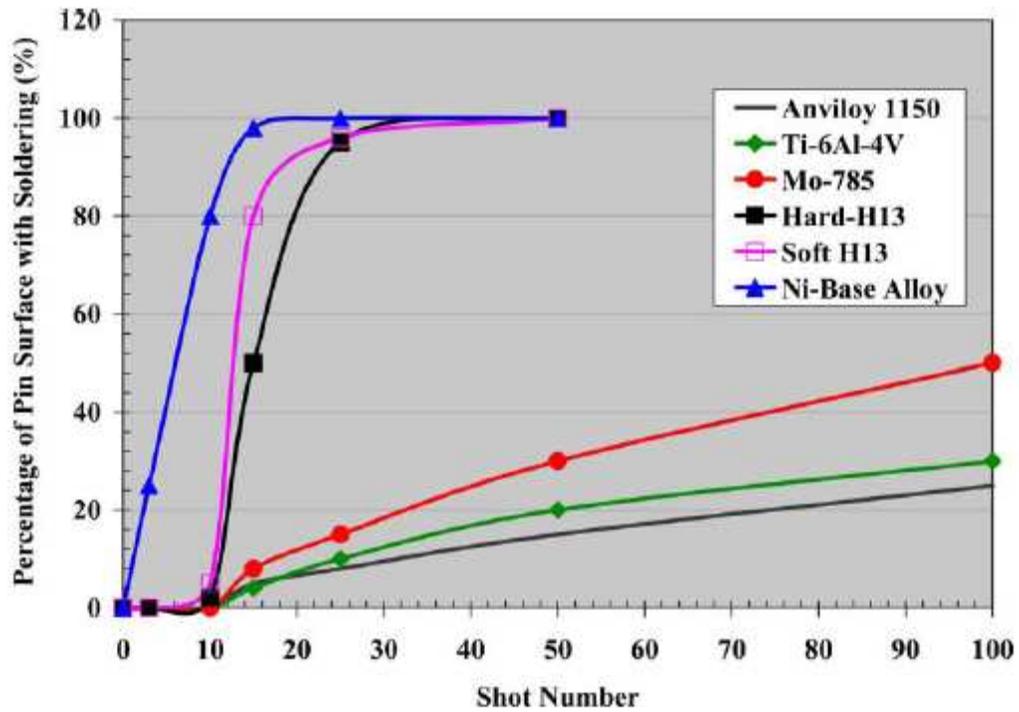


Figure 26: Soldering tendency of several advanced metal materials tested by Zhu [24] for solder resistance. Percentage of pin covered by solder measured.

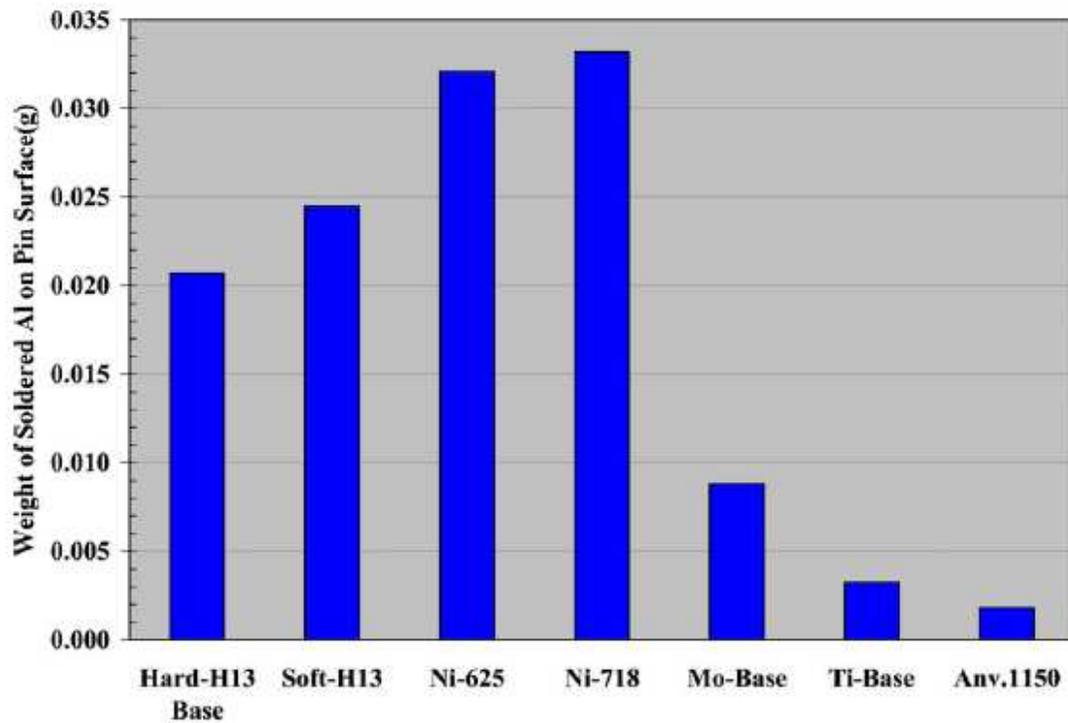


Figure 27: Soldering tendency of several advanced metal materials tested by Zhu for solder resistance. Weight of soldered aluminum measured. [24]

As Figure 26 and Figure 27 suggest, Anviloy1150 has the best resistance to soldering, followed by Mo-785, Ti-6Al-4V, H13, Ni-718, cast iron and copper based alloys.

Yan et al. [25] discuss some alternate ceramic materials that might have the potential for an effective defense against soldering. These include AlN, Si<sub>3</sub>N<sub>4</sub> and sialons, which in addition to being resistant to dissolution by aluminum alloys, also have favorable mechanical properties. It is important that any material resists infiltration by aluminum, so the composition must be free of any element that will dissolve readily in aluminum and with minimal porosity.

## **II. VI References**

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### **III. Measurement of Die Soldering**

In order to achieve the goals of the project, an effective way to measure die soldering is needed. Without a way to measure a phenomenon, there is no way to control it. A measurement technique is necessary for developing and testing a predictive model, and for use in experiments testing various methods for reducing soldering.

Several methods used previously by researchers were discussed in the literature review section. Unfortunately many of these methods are not applicable to the product discussed in this project, the U222 Carrier, since there are no pins or small cores in the die that can be interchanged easily and studied. Thus, many methods such as weight gain/loss of the die or intermetallic thickness were not applicable to the study.

#### ***III.I Qualitative Analysis: Surface Rating***

The first method attempted to rate soldering in the U222 Carrier was a simple visual analysis of the surface.

##### **III.I.I Method**

Several U222 Carrier castings were collected from a production run. The surface of the castings were visually analyzed and photographed from several angles. Using the castings and these photographs, the surface of the casting (and thus the corresponding surface of the die where the solder would be visible) was rated green (no soldering; solder rating = 1), yellow (moderate soldering; solder rating = 2) or red (severe soldering; solder rating = 3). These ratings were confirmed by process engineers at the Pierceton plant.

##### **III.I.II Results & Discussion**

Figure 28 shows a representative image of the rating system devised (the green, no solder areas are not colored). A series of images showing the entire surface of the casting can be found in Appendix 1.



**Figure 28: Green (no solder), yellow (moderate soldering), red (severe soldering) scale.**

This initial analysis gives an indication of where the soldering occurs on the surface. The locations of interest that should be studied specifically in future modeling, and trial runs can be identified through these images.

The solder rating system works for initial analysis and for classification for developing the predictive model, but it is not effective for measuring soldering in a trial run. While it does show an accurate representation of where soldering is occurring, the resolution is very poor, as there are only three levels of behavior. It is also possible for any biases on the part of the observer to affect the results. For these reasons, another approach is needed to quantify soldering.

### ***III.II Quantitative Analysis: Surface Metrology***

It was recognized during an initial investigation into die soldering at the Pierceton plant that the surface condition of the casting is a good indicator of the level of soldering on the die surface. The operator of each die casting machine cannot see the die surface from his/her station, but needs to know when to shut down the die for cleaning when it is necessary. In order to achieve this, a set of visual standards for maximum acceptable solder is placed at each machine, which the operator compares to the parts as they come off the line. When the part reaches the degraded condition shown in the standard, the machine is shut down for cleaning.

From this, we know that the surface condition of the casting can potentially be a measure for soldering. The high pressures used in squeeze casting cause the casting to

fit very closely to the surface of the die, so any solder buildup on the die will show up in a series of pits on the surface of the cast part, theoretically making the surface rougher. The hypothesis is that a surface metrology method can measure and quantify the level of soldering. This was tested at the WPI Surface Metrology Laboratory.

### III.II.I Methodology

19 locations were selected for study on the surface of the U222 Carrier casting. With the aid of the red, yellow, green ranking system, these locations were selected with a variety of solder severities, as well as a variety of geometric features. Of the 19 locations, 10 came from severe solder areas, 5 from moderate solder areas and 4 from no solder areas. Some of these locations are shown in Figure 29.



**Figure 29: Location of several surface metrology locations.**

With these locations selected, a series of cast parts were collected from a run at the Pierceton, IN squeeze casting plant. In this, the first casting after a die cleaning was collected, along with approximately every 40<sup>th</sup> (1, 41, 81, 123, 163, 203, 243 and 283) casting up to and including the last casting made before the die was shut down for cleaning. From these castings, measurements were made on the first casting after cleaning and the last casting before cleaning, in order to get an idea of how soldering over a series of shots affects the surface roughness.

At each of the 19 locations on each of the castings, a surface scan was taken using the scanning laser profiler or microscope (SLM) at the WPI Surface Metrology

Laboratory. The output of these scans was a 6mm x 6mm grid with a spot size (resolution) of 0.0254 mm x 0.0254 mm, with the height of the surface given at each of these points.

Two of the locations were discarded due to problems with the scan data. These omissions left 17 locations for use in data analysis, 4 each at solder ratings 1 and 2 and 9 at solder rating 3.

Using SFrax software developed at WPI and Mountains, commercially available surface metrology software, the surfaces were analyzed. They were each first leveled by removing the best fit 3<sup>rd</sup>-order surface function and filtered to remove any unreasonably high slopes, and then subsequently quantified using a variety of surface parameters.

These included:

- Ra (filtered average roughness) with a Gaussian filter of 0.8 mm
- Pa (unfiltered average roughness)
- Pq
- Relative Area Scale Analysis (see Appendix 2)

For each of the profile parameters, every 10<sup>th</sup> profile was selected, for a total of 23 profiles, with the results averaged. Pq was selected instead of Rq because the results of the analysis showed that the Pa values were better at distinguishing among groups than the Ra values. This will be shown below in the results section.

For each of the parameters tested, four responses were reviewed:

1. The value of each parameter on the first casting after cleaning.
2. The value on the last casting before cleaning.
3. Raw change in the response from the first casting to the last casting.
4. Percent change from the first casting to the last casting.

Each combination of the parameters and the responses were tested for significant differences among groups, in an attempt to identify a roughness parameter that can differentiate between various levels of soldering. Preliminarily, an ANOVA test was run to test for differences between each of the three groups, with subsequent t-tests run between individual groups. For the area scale analysis, the tests were run at each at each scale.

Where the data did not meet the assumptions of a traditional ANOVA or t-test (normal distribution, equal variances), the applicable non-parametric test was used to test the differences in group means. These were the Kruskal-Wallis ANOVA on Ranks (replacing the standard one-way ANOVA) and the Mann-Whitney Rank Sum Test

(replacing the t-test). The cases where these tests were used are marked in the charts below.

Since the data violated the assumptions at many area scales for the area-scale analysis, the non-parametric tests were used for all tests at each scale.

### III.II.II Results & Discussion

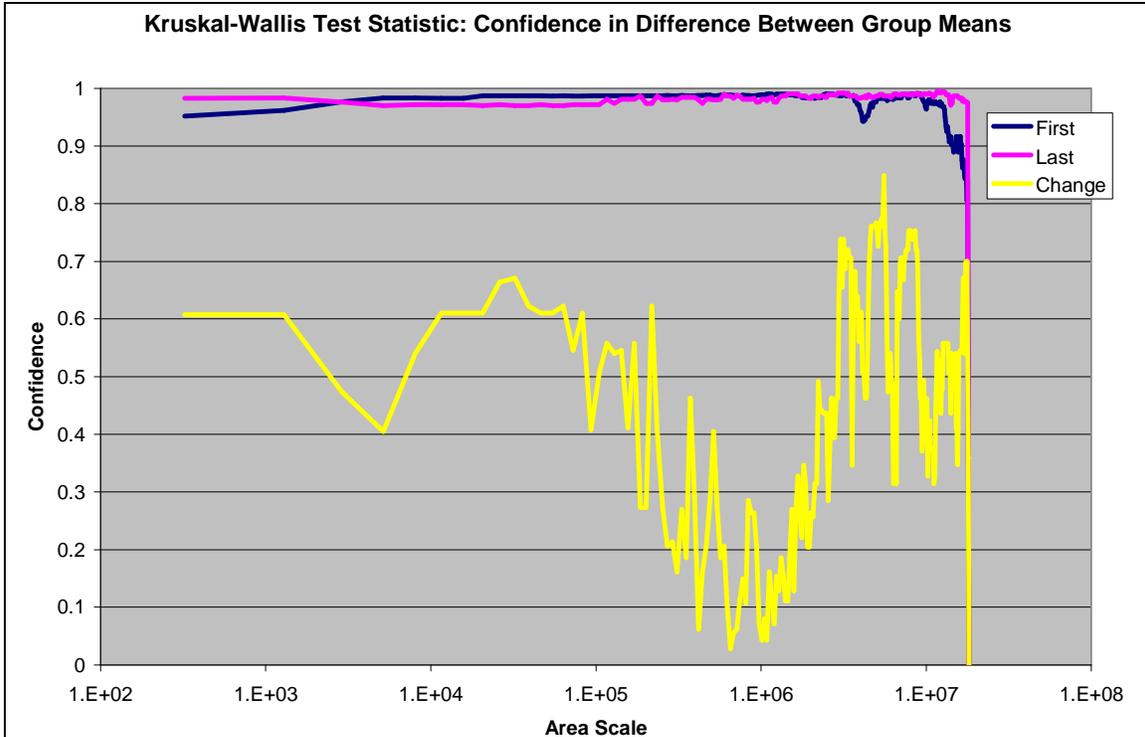
#### ANOVA Results

Table 7 displays the results of the ANOVA test between the three solder rating groups for each variable and parameter. At the 95% confidence level, the differences between group means are significant for all three parameters from the measurements on both the first casting after cleaning and the last casting before cleaning. The change variables do not seem to be a strong indicator of soldering, as indicated by the very high p values.

Table 7: ANOVA results (p values) (solder ratings 1, 2, 3).

ANOVA				
	First	Last	Change	%Change
Ra	0.023	0.044	0.613	0.916*
Pa	0.010	0.012*	0.333	0.404
Pq	0.007	0.013*	0.358	0.750*
*Kruskal-Wallis ANOVA on Ranks				

Figure 30 shows the results of the Kruskal-Wallis test at each area scale from the area scale analysis. Similarly to the profile parameter test, the group means for the first and last casting are significantly different at most area scales, while the change in area scale does not seem to be a strong indication of soldering.



**Figure 30: Confidence results (p value) of the K-W test at each area scale for differences among group means.**

Because of the lack of significance shown by the change in relative area with area scale, these variables were henceforth disregarded to reduce analysis time.

Figure 31 shows a portion of the plot displayed in Figure 30. The maximum significance between groups from the measurements on the first casting after cleaning is  $p=0.009276$  at a scale of approximately  $2,500,000 \mu\text{m}^2$ . This compares well to the values found in the profile parameter tests.

The maximum significance between groups from measurements on the last casting after cleaning is  $p=0.004825$  at an area scale of approximately  $11,750,000 \mu\text{m}^2$ . This compares very well to values found in the tests run on the profile parameters, in fact it is the only test that shows significance at the 99.5% level.

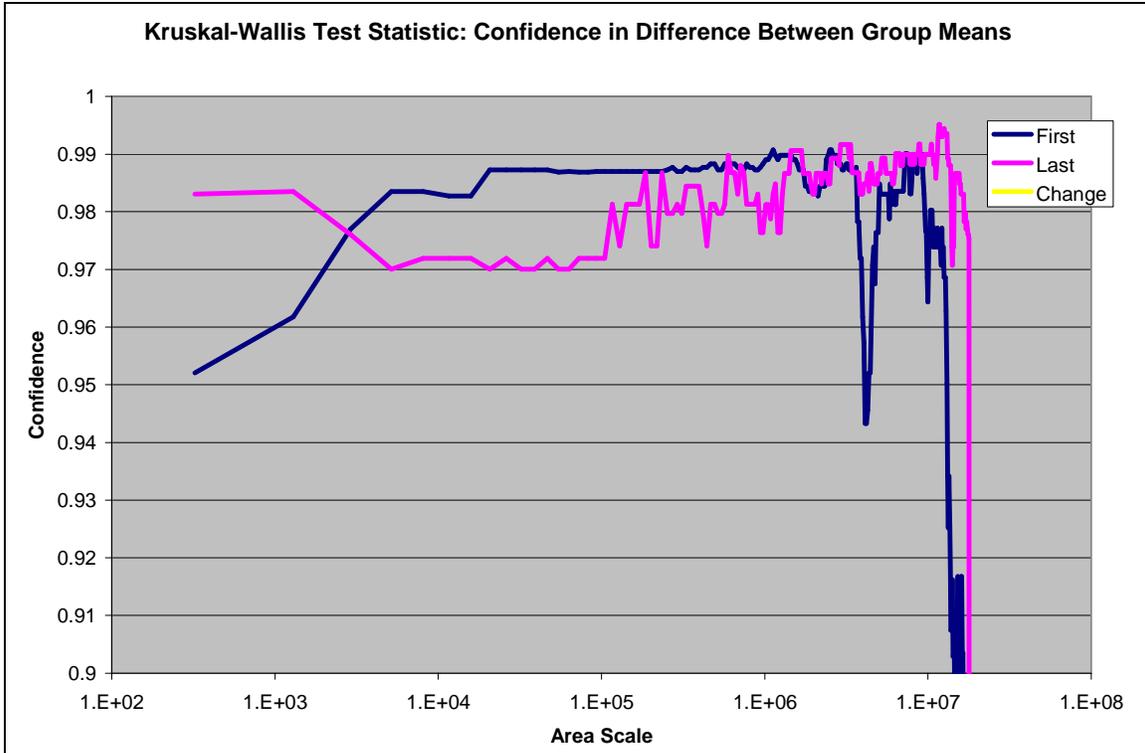


Figure 31: Zoomed in portion of Figure 30, showing scales with differences among groups at >90% confidence.

**t-Tests: Solder Ratings 1v3**

Table 8: t-test results (p-values) (solder ratings 1&2).

1v3				
	First	Last	Change	%Change
Ra	0.007	0.018	0.371	0.817*
Pa	0.006	0.007*	0.205	0.271
Pq	0.004	0.007*	0.220	0.294
*Mann-Whitney Rank Sum Test				

As with the ANOVA tests between all groups, significant differences are found between groups means for all three profile parameters on both the first casting after cleaning and the last casting before cleaning. This is expected, since groups 1 and 3 (no soldering, severe soldering; respectively) should be very different. The change variables again do not show any ability to differentiate between the groups.

In Figure 32, the plot of significance between groups by area scale is plotted. The highest confidence is  $p=0.005479$  at various area scales. This compares very well with the profile parameter test results. The significance is generally higher on measurements made on the first casting after cleaning.

Pq on the first casting after cleaning is the greatest indicator of soldering behavior, where  $p=0.004$ .

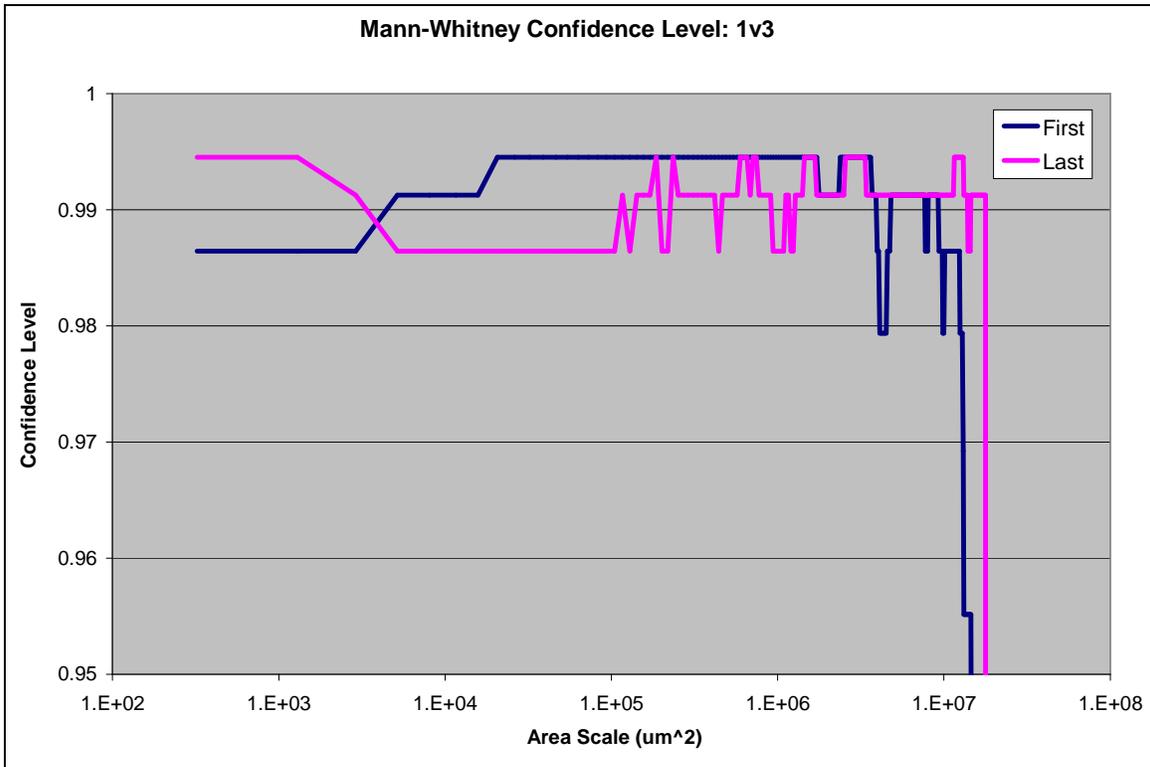


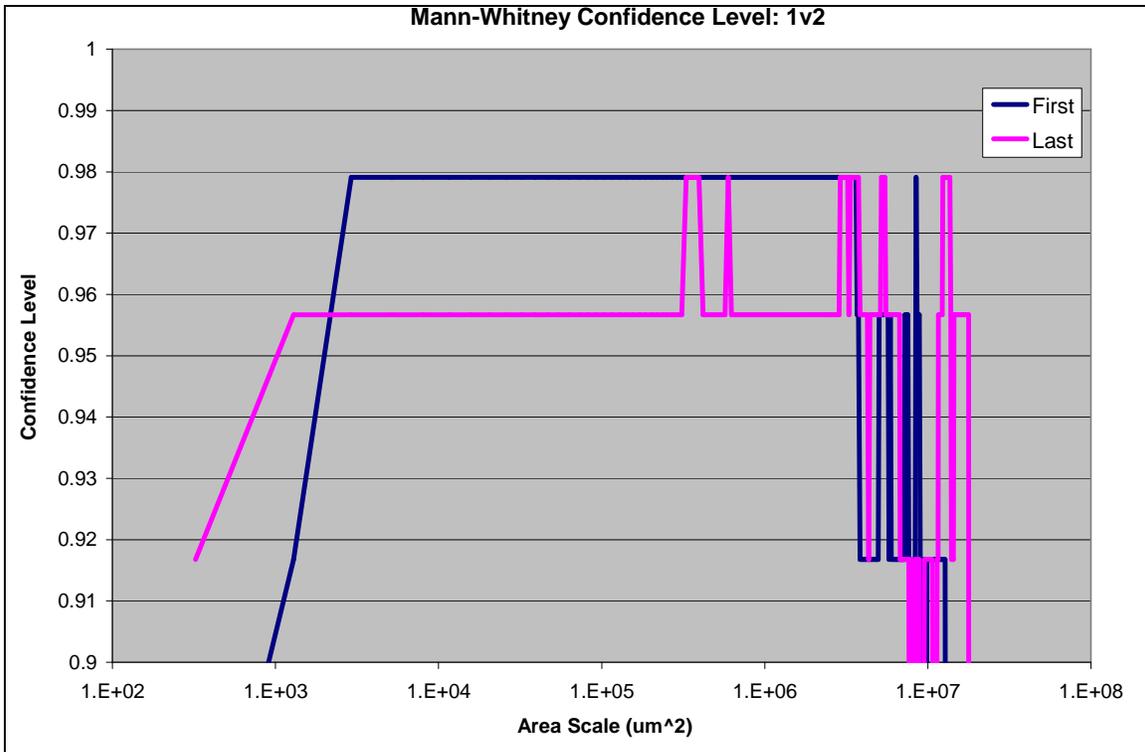
Figure 32: Confidence results of the MW test at each area scale for differences between ratings 1&3.

### t-Tests: Ratings 1v2

Table 9: t-test results (p values) (solder ratings 1&2).

	1v2			
	First	Last	Change	%Change
Ra	0.057*	0.049	0.234	0.518
Pa	0.009	0.036	0.441	0.589
Pq	0.011	0.035	0.438	0.612
*Mann-Whitney Rank Sum Test				

For groups 1 and 2 (no soldering, moderate soldering; respectively), the group means are different at a 95% confidence level for each of the three parameters on both the first and last casting, except for Ra on the first casting. As expected, the p values are higher, indicating that it is more difficult to differentiate between groups 1 and 2 than groups 1 and 3.



**Figure 33: Confidence results of the MW test at each area scale for differences between ratings 1&2.**

In Figure 33, the results of the MW tests are plotted. The maximum significance for both the first and last castings is 0.020921, which compares well to the data for the profile parameter tests, although it is hard to compare the values for different tests since the non-parametric tests are less powerful. The maximum confidence is seen at a variety of scales, and is generally higher for the first casting.

Pa on the first casting is the strongest indicator of soldering behavior, with  $p=0.009$ .

### **t-Tests: Ratings 2v3**

**Table 10: t-test results (p values) (solder ratings 2&3).**

<b>2v3</b>				
	<b>First</b>	<b>Last</b>	<b>Change</b>	<b>%Change</b>
Ra	0.763	0.722	0.861	0.817*
Pa	0.590	0.239	0.757*	0.589*
Pq	0.556	0.262	0.817*	0.817*
<i>*Mann-Whitney Rank Sum Test</i>				

As is shown in Table 10, the differences between group means of groups 2 and 3 (moderate soldering, severe soldering) are not significant when comparing the profile parameter values. However, in Figure 34, the plot of the Mann-Whitney tests by area-

scale, there are a few scales at which locations from the two groups are differentiable at a 90% or even 95% confidence level.

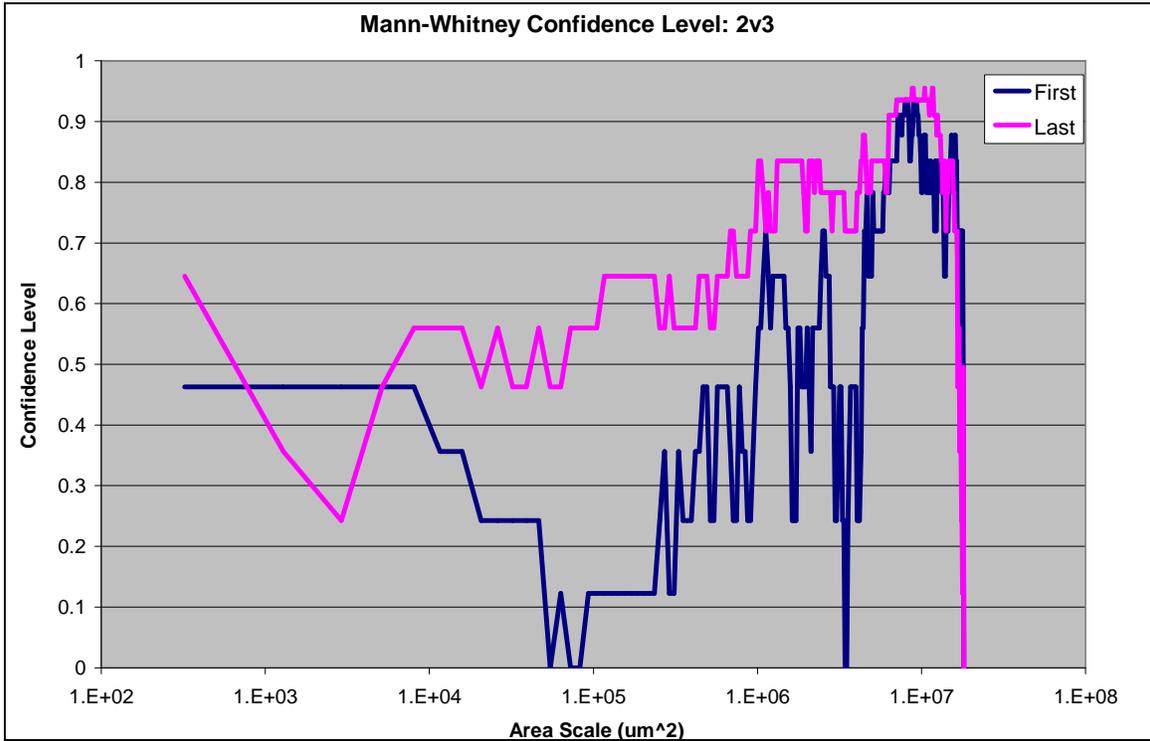


Figure 34: Confidence results of the MW test at each area scale for differences between ratings 2&3.

The maximum confidence between group means found in the area scale analysis is on the last casting at several area scales (interestingly, rather large scales), where  $p=.044862$ .

**t-Tests: Ratings SvNS**

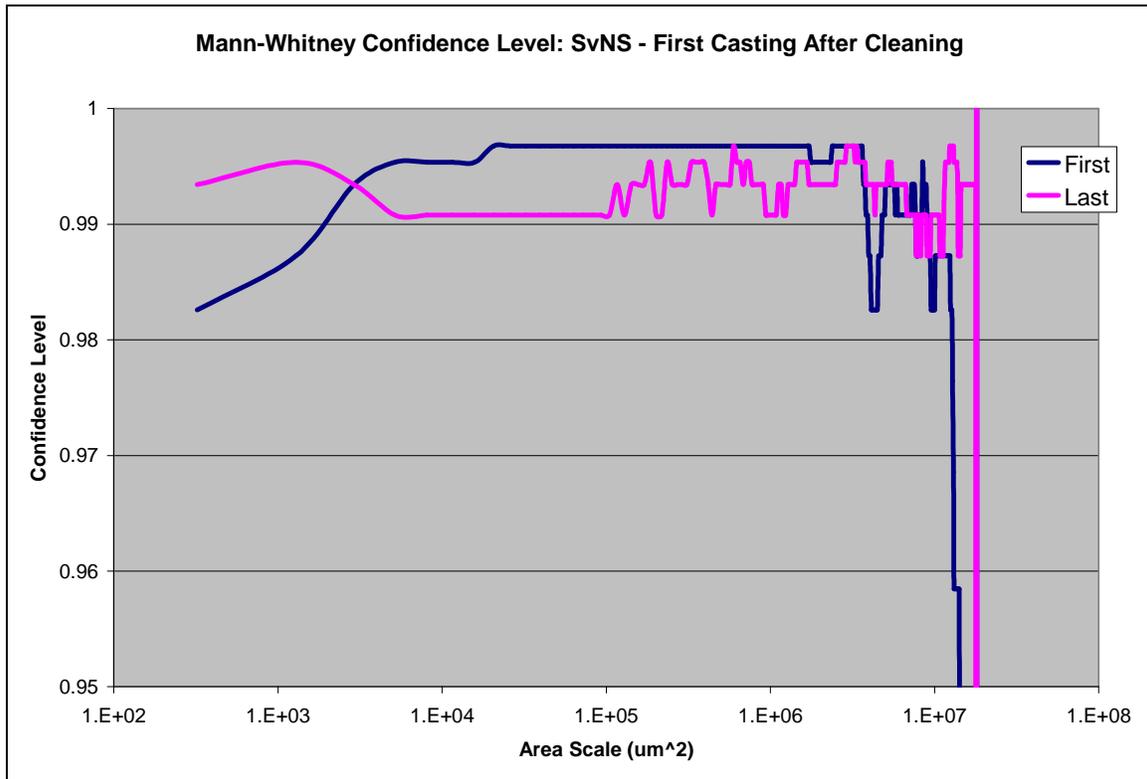
Two further groups were created due to the somewhat inconclusive results of the comparison between groups 2 and 3. These groups consisted of the locations which experience soldering (groups 2 or 3, here denoted as group S – solder), and those that do not experience soldering (group 1, denoted as group NS – no solder).

Table 11: t-test results (p values) (solder ratings S&NS).

SvNS				
	First	Last	Change	%Change
Ra	0.006	0.012	0.324	0.777*
Pa	0.002	0.010	0.246	0.322
Pq	0.002	0.012	0.257	0.533*
*Mann-Whitney Rank Sum Test				

The tests show (see Table 11) a significant difference between group means for all parameters on the first and last castings. The maximum significance is seen for Pa and Pq on the first casting after cleaning, with >99% significance. Thus, it does not seem difficult to determine which groups experience soldering, but differentiating between the groups seems to be difficult.

In Figure 35, the plot of significance between group means by area scale, the maximum significance between the two groups is 0.003241, at various area scales, which compares well to the profile parameter results.



**Figure 35: Confidence results of the MW test at each area scale for differences between ratings S&NS.**

The maximum confidence, where  $p=0.002$  is found in the case of both Pa and Pq on the first casting after cleaning.

### **Change from First to Last Casting within groups**

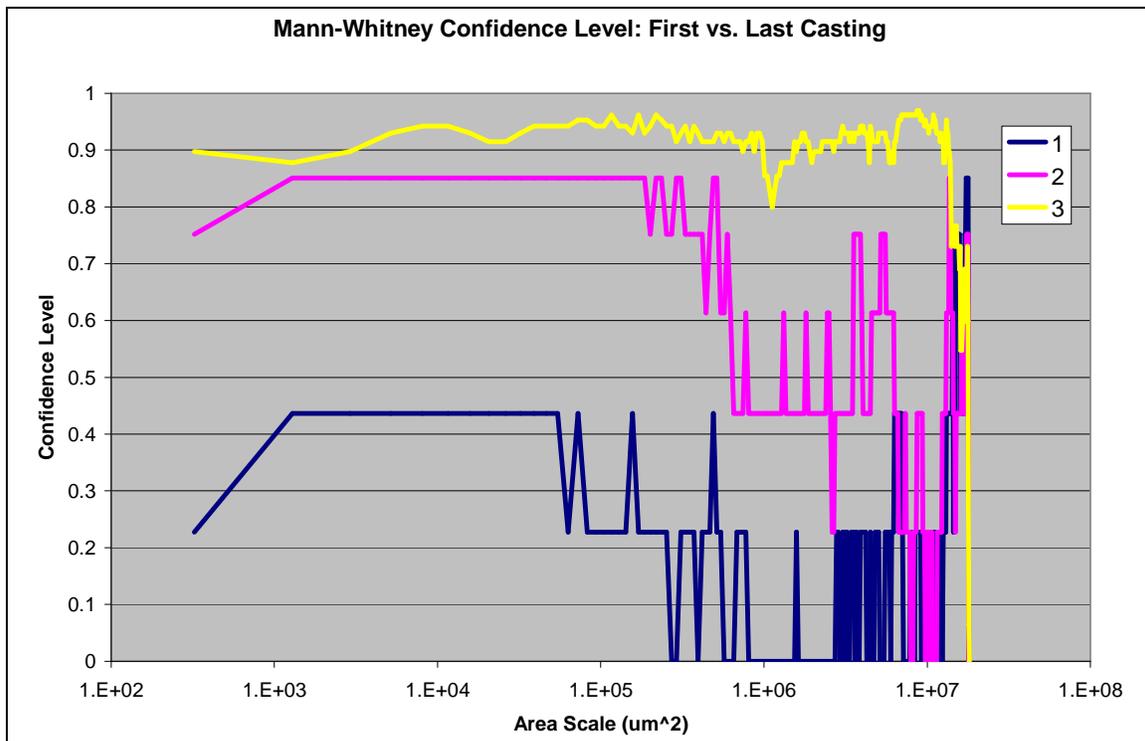
Despite the fact that the change in roughness parameters from the first casting after cleaning to the last casting before cleaning is not an effective way to differentiate between groups, it seems that it is possible to differentiate between the measurements made on the first casting and those made on the last casting within the solder rating groups. Figure 36 shows a plot of statistical tests on the area scale analysis within each

solder rating group on the measurements taken on both the first casting after cleaning and the last casting before cleaning.

**Table 12: t-test results (p values) first vs. last casting.**

	FvL		
	1	2	3
Ra	0.435	0.248	0.044
Pa	0.804	0.377	0.070*
Pq	0.747	0.359	0.077*

\*Mann-Whitney Rank Sum Test



**Figure 36: Mann-Whitney confidence in a difference between groups. First casting after cleaning vs. last casting before cleaning within solder rating groups.**

The results of the test indicate that in cases of severe soldering, the group means of the relative area of the locations on the first casting after cleaning is significantly different from the same locations on the last casting before cleaning at many area scales.

### Summary

To summarize the most significant statistical test for each comparison:

- ANOVA: Area Scale, last casting after cleaning;  $p=0.004825$  at an area scale of approximately  $11,750,000 \mu\text{m}^2$ .
- 1v3: Pq, first casting after cleaning;  $p=0.004$ .
- 1v2: Pa, first casting after cleaning;  $p=0.009$ .
- 2v3: Area Scale, last casting before cleaning;  $p=.044862$  at several area scales (interestingly, large scales).

- SvNS: Pa & Pq, first casting after cleaning;  $p=0.002$ .
- FvL: Ra, group 3 (severe soldering)

### **III.III Conclusions**

- The statistical tests prove that surface metrology can be used to measure the surface of the cast part in order to quantify soldering in the absence of other alternatives.
- It appears that the effect of soldering on the roughness of the surface is more of a lasting effect than an effect that causes change over several hundred shots. However, the fact that the Ra group means of solder ratings 3 from the first and last casting are significantly different shows that there is promise in using change to quantify soldering over a smaller series of shots. Further study is needed to prove or disprove this point.
- This method may be valuable in tests to evaluate how process parameter changes effect soldering in the future. With the methods used later in the project, it takes several days to acquire a large enough sample size of data using a cleaning time method. With the surface metrology method explored in the section, the roughness can be measured before and after a set period of say 100 parts and these measurements used to quantify soldering, saving large amounts of time. Further study under different conditions are necessary to prove that the repeatability of these measurements is good enough to draw confident results from these tests, but this work shows that the method holds promise.
- At this point, it is difficult to declare that one parameter is more effective than others. A follow-up study with more samples and taken over several runs from cleaning to cleaning (to determine the variability of the surface roughness) can help to further refine the method.
- It is unlikely that a surface measurement method would be used on the floor of a manufacturing plant as an indicator of when soldering is occurring. Visual inspection of the part is sufficient for determining when cleaning should take place. Instead, the method can be used as a way to study the response of the system during trials, in order to bring soldering under control and to determine the best methods for reducing soldering.

## IV. Prediction of Die Soldering

As the financial analysis at the beginning of the thesis suggests, die soldering is an expensive problem in the aluminum die casting process. However, any actions taken to reduce soldering and reduce losses in the process inevitably add cost to the design and manufacture of the die. In order to justify these increased costs, it would be beneficial to have a method for accurately predicting soldering and its severity during the initial design process, so that an estimate of the cost of soldering can be determined.

Currently, an idea of where soldering will occur can be determined by analyzing the results of a simulation in MAGMASoft and looking for locations where the die becomes hot. Soldering will likely occur in these areas. However, there is not an easy way to translate from the temperature in these locations to the extent of soldering, and then to an estimated cost of soldering, necessary to justify any expenses to mitigate the soldering. One objective of the project was to determine how to relate a given time-temperature curve from a location at the part-die interface to an extent of soldering that that location will experience.

Despite many years of research on the topic, there is still some disagreement as to how soldering occurs at the micro-scale, due to the complexity of the phenomenon which sometimes manifests itself in different ways. There is disagreement on whether Fe or Al is the diffusing species and how large a role die erosion and intermetallic layer growth play in the formation of soldering. Thus, a model developed from first principles, as is now being explored by some modelers for micro-porosity or hot tearing, is not an attractive method, at least in the scope of one part of this project.

Instead, a 'data mining' method was explored. The idea behind this method is to examine parts currently in production and analyze which locations on the die-casting interface experience soldering and then correlate the conditions at these locations to the soldering behavior. This should determine which types of conditions promote soldering. These conditions can then be looked for in later simulations for an initial warning against soldering.

## IV.1 Methodology

41 locations at the interface of the die and the casting were selected, using the solder rating system developed in this project as a guide. These points were selected over a range of areas on the surface and comprised:

- 14 points in green (no soldering) areas
- 12 points in yellow (moderate soldering) areas
- 15 points in red (severe soldering) areas

After these points were selected, the next step was to use MAGMA to see what the conditions at these points are. To do this, two cooling curve control points were inserted into the model for each location, one at the surface of the die and one at the surface of the cast part. An image showing some of these points in the geometry is displayed in Figure 37. The two control points at each location are located directly next to each other, but in different materials.

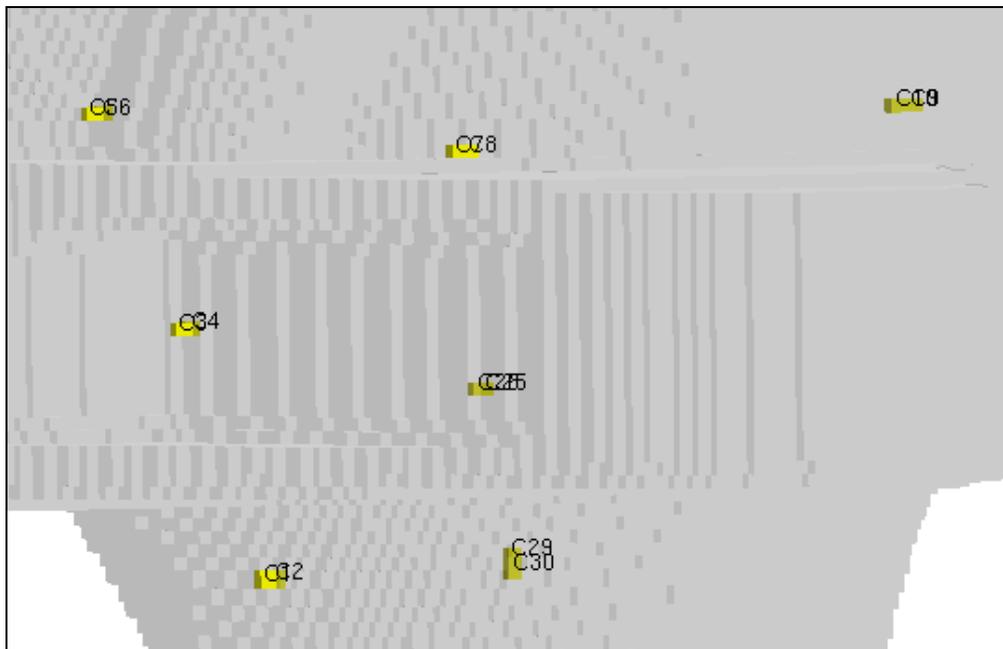


Figure 37: Several pairs of cooling curves in the MAGMA geometry for generating data.

Using these control points, the following data can be obtained for each point:

- The temperature at the steel surface over the casting cycle
- The temperature at the surface of the cast aluminum over the casting cycle
- The velocity of the metal in the cavity during filling of the part

Statistical techniques were used to analyze the data more thoroughly and in order to draw more concrete insights. A series of variables were calculated from the raw data. These were:

- The maximum die and cast metal temperature and velocity at each point.
- The average die and cast metal temperature and velocity at each point.
- An average soldering potential at each location (discussed below).
- 'Bearing Curve' Analysis. The time each location spends above a series of temperatures during the cycle (discussed below).

### **Soldering Potential**

It was noted that the average temperature calculations above treat an increase in temperature as a linear increase in terms of soldering potential or rate. This is probably an incorrect way of looking at the situation. The soldering potential (S) at any temperature probably follows more of an Arrhenius type curve, rather than a linear increase, due to the increases in diffusion, atom activity at the surface, etc. which should exhibit an Arrhenius type relationship with temperature.

To find the activation energy, the results of an experiment run by Sumanth Shankar for his Ph.D. research were analyzed. These results are shown in the following tables.

**Table 5.1:** Experimental Design of Exploratory Experiments to Determine Characteristics of the Soldered Interface.

<b>L9 Taguchi Matrix</b>					
	<i>Factors</i>	<i>Time (s)</i>	<i>Temperature (F)</i>	<i>Coating</i>	<i>Alloy</i>
	Level 3 (3)	75	1250		380 + Ti + Sr
	Level 2 (2)	45	1200	Coating	380 + Ti
	Level 1 (1)	30	1150	No Coating	380
<i>Experiment Number</i>	<i>DESIGN OF EXPERIMENTAL RUNS</i>				
1		30	1150	No	380
2		30	1200	Yes	380
3		30	1250	No	380+ Ti +Sr
4		45	1150	Yes	380+ Ti +Sr
5		45	1200	Yes	380
6		45	1250	No	380+ Ti
7		75	1150	No Coating	380+ Ti
8		75	1200	No	380+ Ti +Sr
9		75	1250	Yes	380

**Table 6.2:** Intermetallic Layer Thickness from the Runs #1 and #2 of L-9 Experimental Matrix and the S/N Values

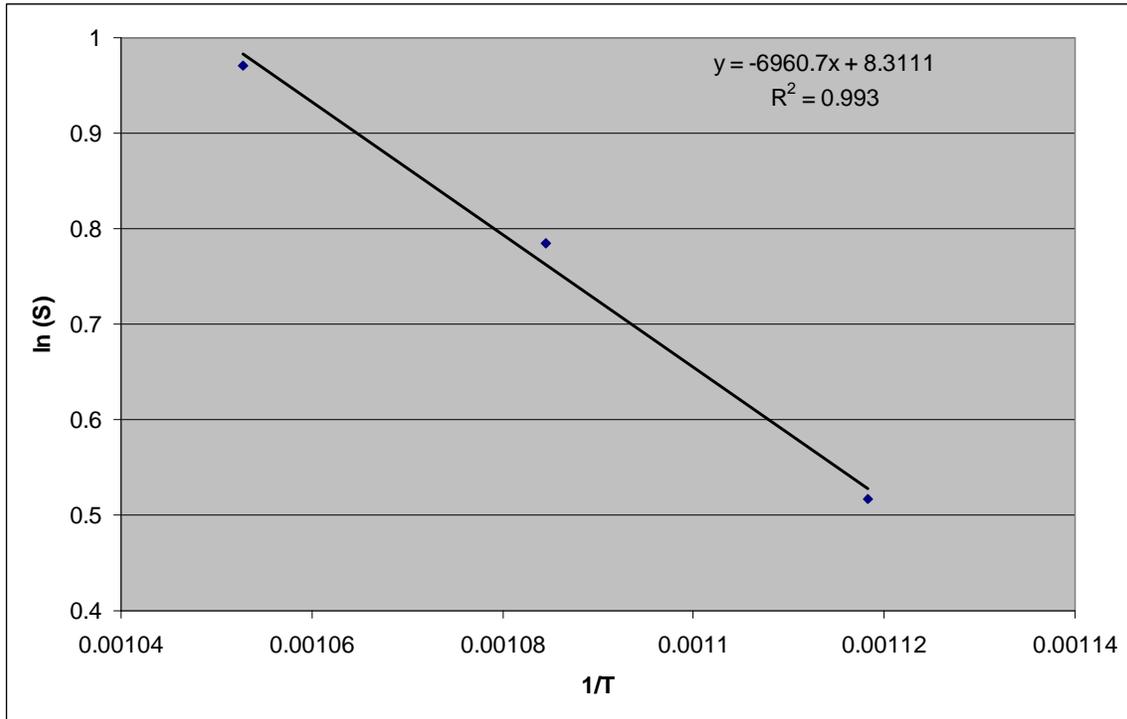
<i>Trials/ Repetitions</i>	<i>R1 (microns)</i>	<i>R2 (microns)</i>	<i>S/N</i>
1	1.773125	2.388571	11.808191
2	1.16423	1.0431	1.5832901
3	2.00764	1.864371	11.416926
4	1.423192	1.63423	7.167547
5	3.1534	3.504	20.771033
6	2.147033	1.659455	10.483545
7	1.593857	1.245	5.4426188
8	2.2341	2.0432	13.119337
9	4.389	3.780071	24.206361

In his research, he tested the depth of intermetallic layers formed between aluminum alloy 380 and H13 steel (the alloy in this case in ADC12, but it is assumed the results should be fairly close). The experiment in question looked at three temperatures: 1150, 1200 and 1250 F. The main effects, in terms of intermetallic layer thickness were 1.694429, 2.14226 and 2.67155  $\mu\text{m}$ , respectively, at these three temperatures.

By converting the Arrhenius equation  $S = S_o \exp\left(\frac{-Q}{RT}\right)$  to  $\ln(S) = \ln(S_o) - \frac{Q}{RT}$ ,

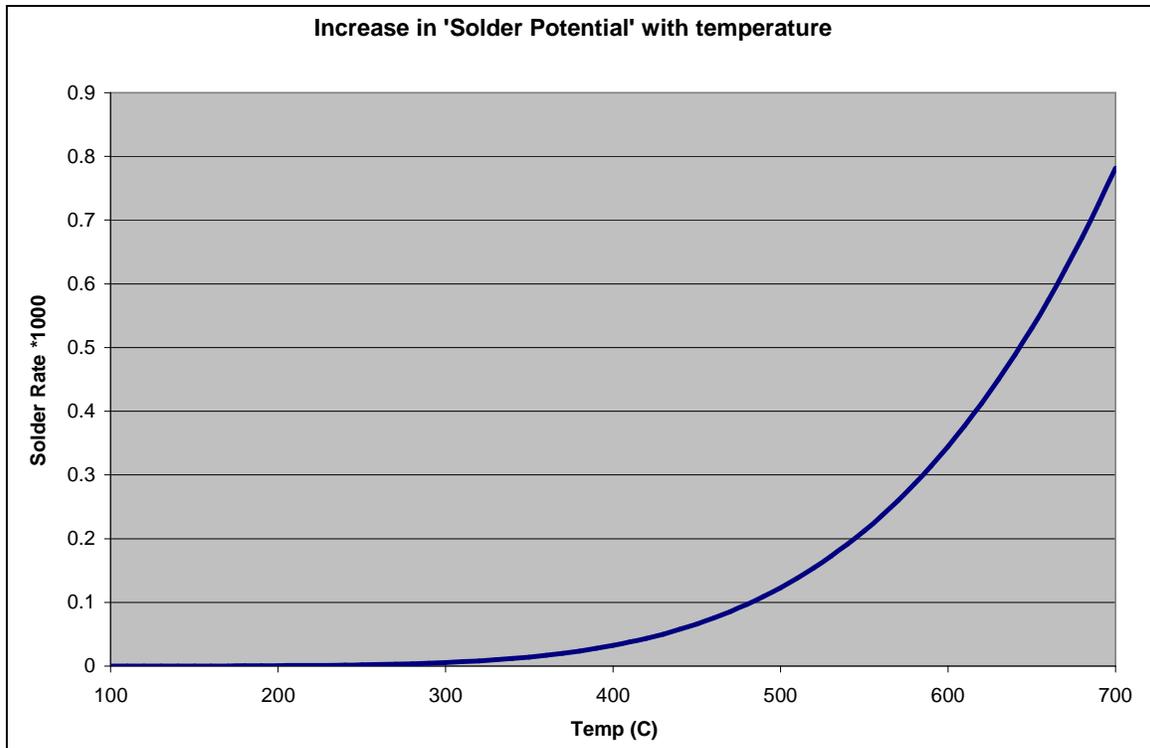
the results can be plotted, as in Figure 38, and  $S_o$  and  $Q$  determined from the equation for the best fit line. This was done, with the results:

$S_o = 4068.786$  microns  
 $Q = 57871$  joules



**Figure 38: Plot of main effects from L9 matrix in Sumanth Shankar's Ph.D. research, plotted in the form of an Arrhenius equation to find the activation energy of soldering.**

The values determined above were plugged back into the Arrhenius equation and used to calculate a 'soldering potential' at each temperature that would more closely match the actual effect of an increase in temperature where an increase from 300 to 600 degrees more than doubles the likelihood that soldering will occur. Figure 39 shows how the 'soldering potential' increases with temperature due to the Arrhenius equation.



**Figure 39: Increase in soldering rate with temperature, as predicted by Arrhenius curve. This treatment should be more realistic than a liner treatment of temperature's effect on soldering.**

### **'Bearing Curve' Analysis**

In the literature, a critical soldering temperature has been suggested, above which soldering will occur, and below which soldering will not occur. The dendrite coherency temperature is suggested as the critical temperature, since below this temperature new aluminum will not be able to feed to the surface to be used in the reaction. This is probably oversimplifying the problem, but it does suggest an interesting way to look at the data. If there is a critical temperature, the amount of time spent above this temperature should be an excellent predictor of soldering.

To test this, a Matlab program was written to determine how much time was spent above each temperature from 100C to 700C, in intervals of 0.5 degrees, at each location at both the surface of the steel and the surface of the aluminum. This method was termed a 'bearing curve' analysis from the surface metrology method which tests how much of a surface is above a given height, in order to correlate to friction coefficients and other performance metrics and to develop engineered surfaces.

### **Statistical Analysis**

As a preliminary test for the variables, each was tested for significant differences between the three groups (solder rating 1, 2 and 3). To do this, an ANOVA test was performed for differences between the three groups and a t-test for differences between each two groups individually. Where the data does not meet the underlying assumptions for an ANOVA or t-test (normally distributed data, equal variances between groups), a non-parametric test, the Kruskal-Wallis ANOVA on Ranks (for all three groups) or the Mann-Whitney test (for differences two groups) were performed. Additionally, two new groups were created: S (any location experiencing solder, groups 2 and 3 combined) and NS (locations not experiencing solder, group 1).

For the bearing curve analysis results, the test was performed at each temperature. Non-parametric tests were performed on the bearing curve analysis data at all temperatures, since the data did not meet the assumptions for a parametric test at each temperature, for ease of comparison.

Due to the proprietary nature of the results, specific times and temperatures from the results are not given in the results section below.

#### ***IV.II Results and Discussion***

Figure 40 and Figure 41 plot the time vs. temperature curves generated by MAGMA for each of the 41 locations at both the surface of the steel and the surface of the aluminum, respectively. Figure 42 plots the velocity in the cavity during filling. In each plot the curves are colored based on their solder rating: green for locations with no soldering, yellow for moderate soldering and red for severe soldering.

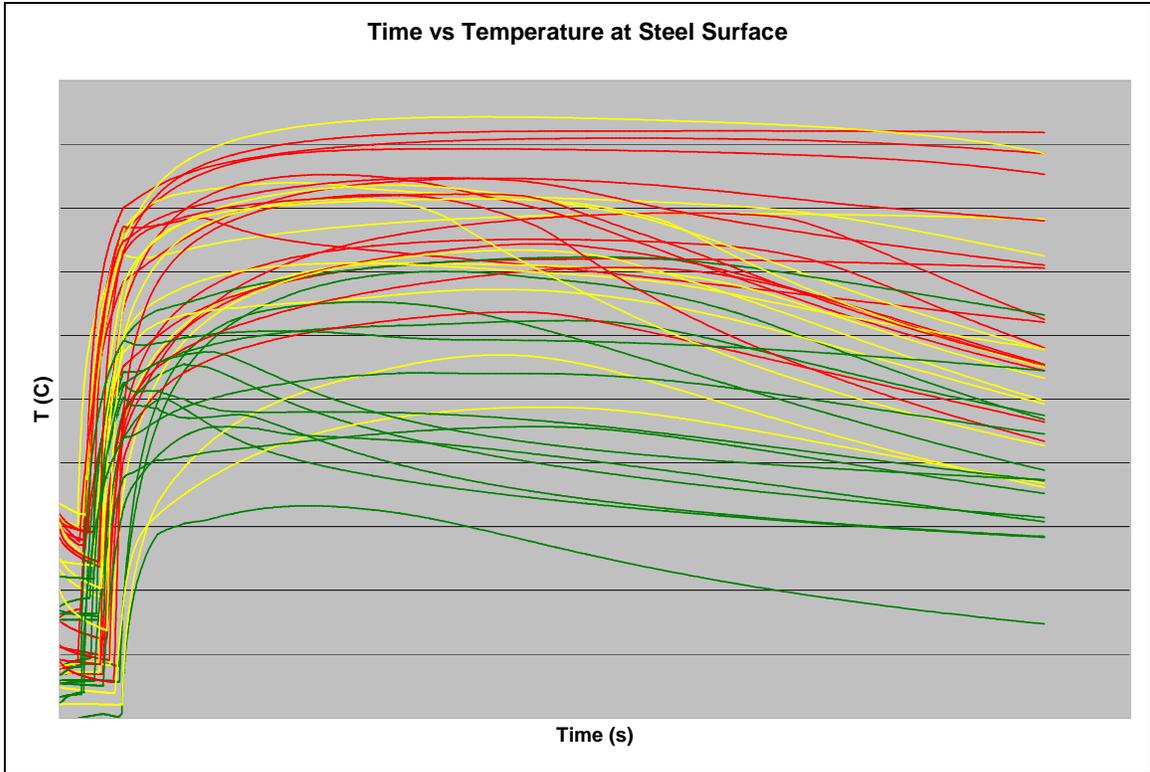


Figure 40: Time vs. temperature curves for each of the 41 locations in the surface of the steel. Green curves indicate no soldering; yellow moderate soldering; red severe soldering.

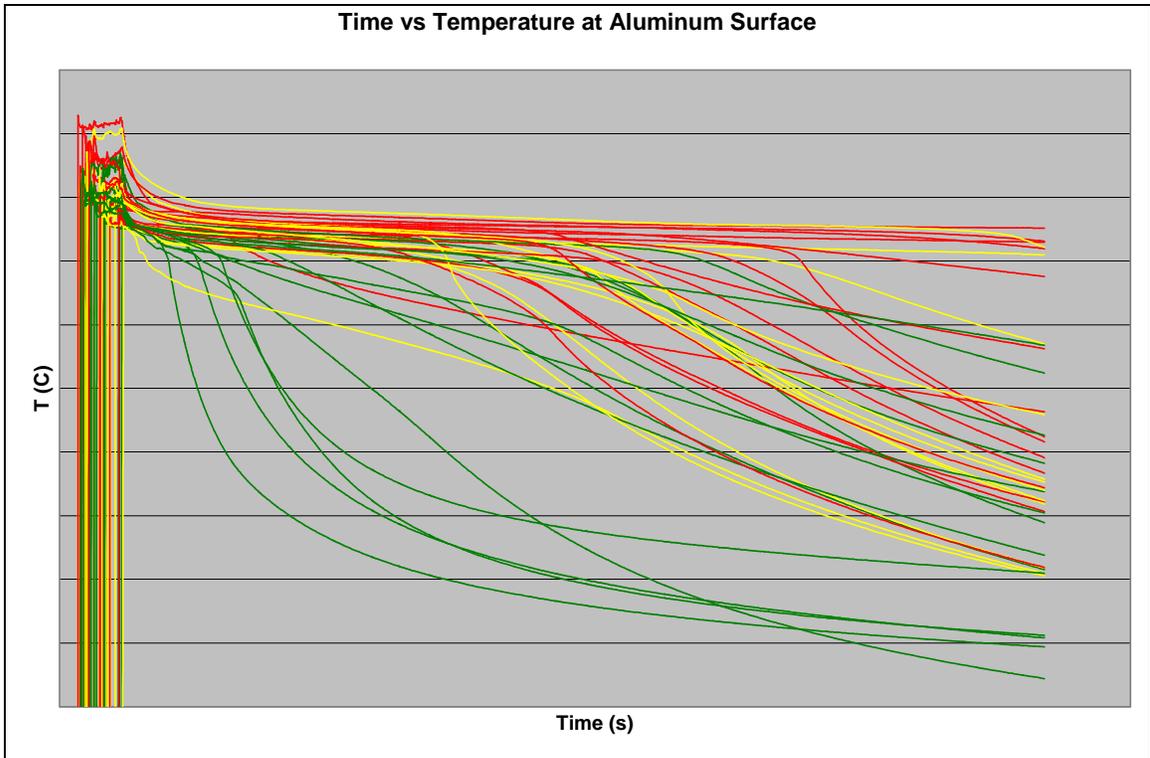
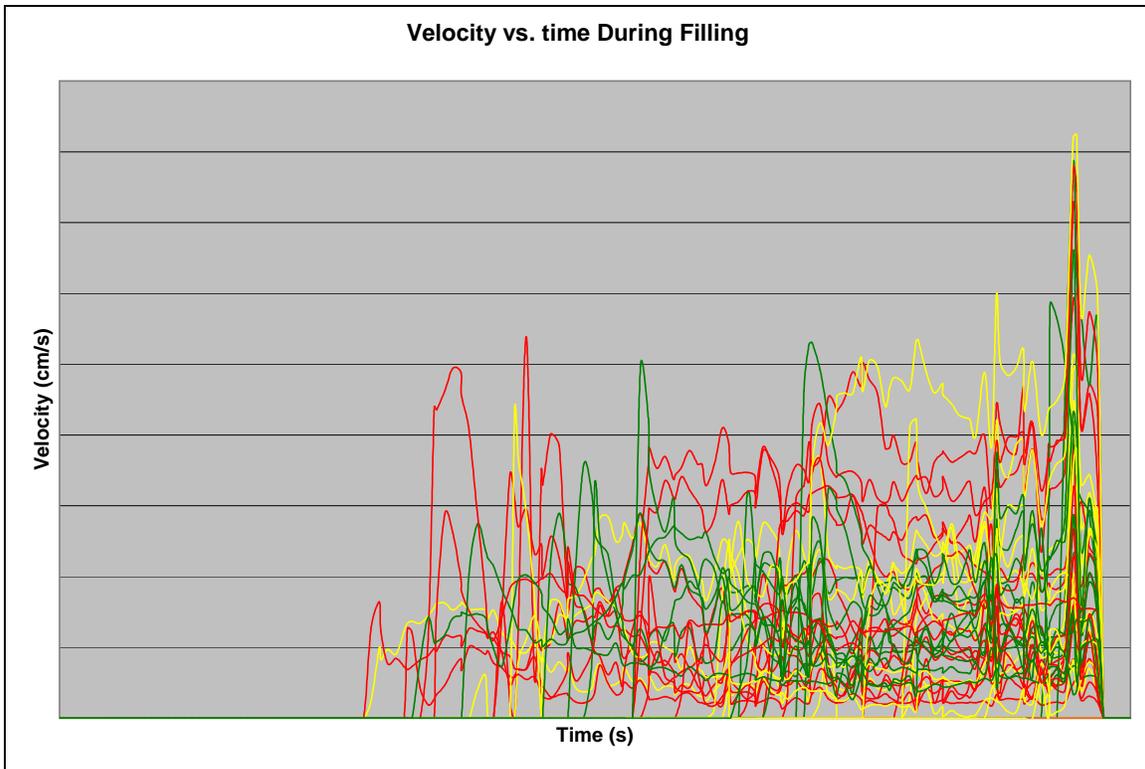


Figure 41: Time vs. temperature curves for each of the 41 locations in the surface of the aluminum. Green curves indicate no soldering; yellow moderate soldering; red severe soldering.



**Figure 42: Velocity vs. time curves for each of the 41 locations in the surface of the aluminum. Green curves indicate no soldering; yellow moderate soldering; red severe soldering.**

It is difficult to draw any firm conclusions from these plots because of the large amount of data plotted in each. However, a general trend of hotter temperatures in the case of the locations with more severe soldering can be seen. There does not seem to be such a clear separation in the case of velocity.

Figure 43 and Figure 44 are plots of the average temperature for each group are plotted. In these plots, the trend of hotter temperatures leading to more severe soldering is more clearly seen.

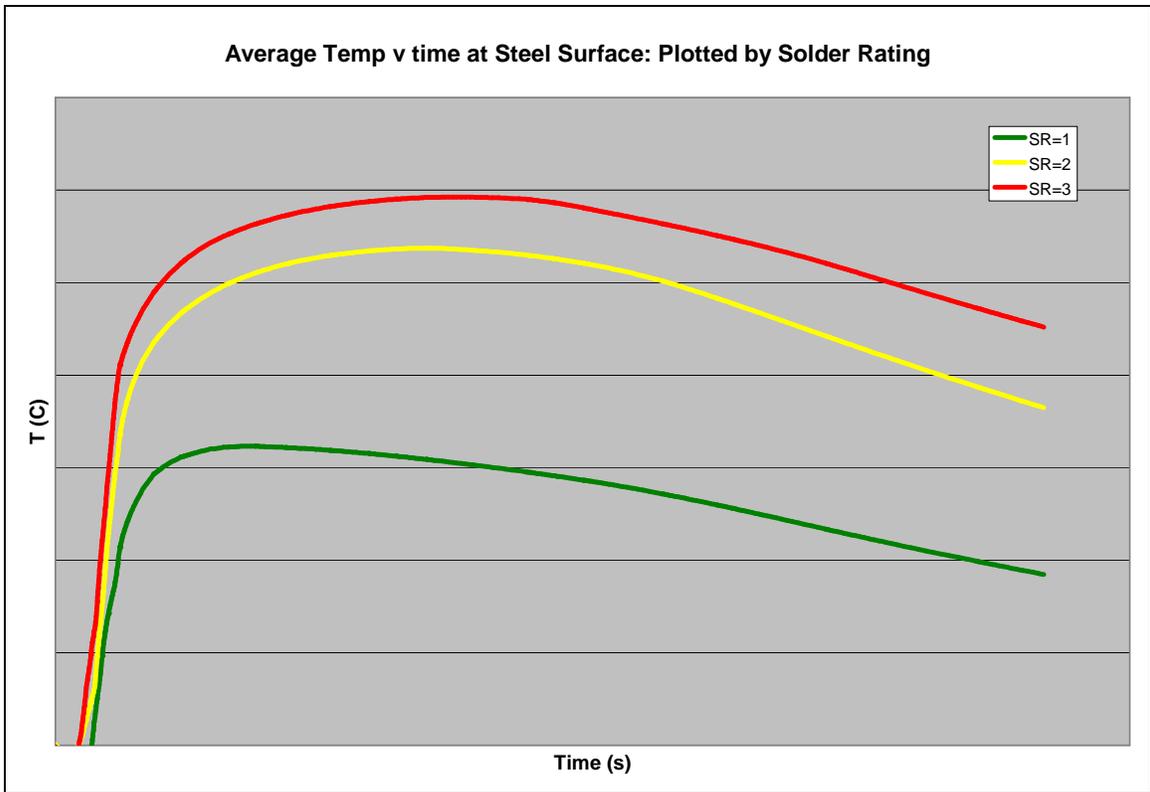


Figure 43: Average temperature for locations with solder rating 1 (green), 2 (yellow) and 3 (red) at the steel surface.

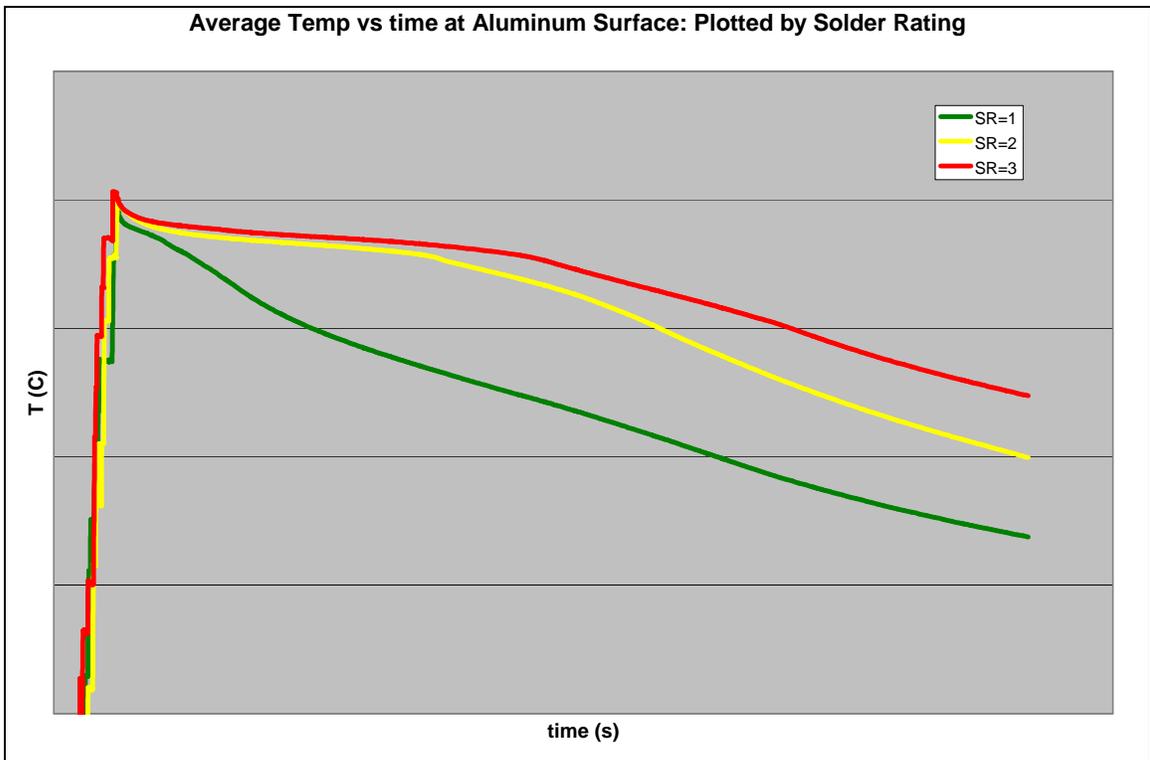


Figure 44: Average temperature for locations with solder rating 1 (green), 2 (yellow) and 3 (red) at the aluminum surface.

Figure 45 plots the average velocity by solder rating. This graph reinforces the theory that the velocity of metal during filling is not a strong predictor of soldering, but again it is difficult to draw firm conclusions from the plots.

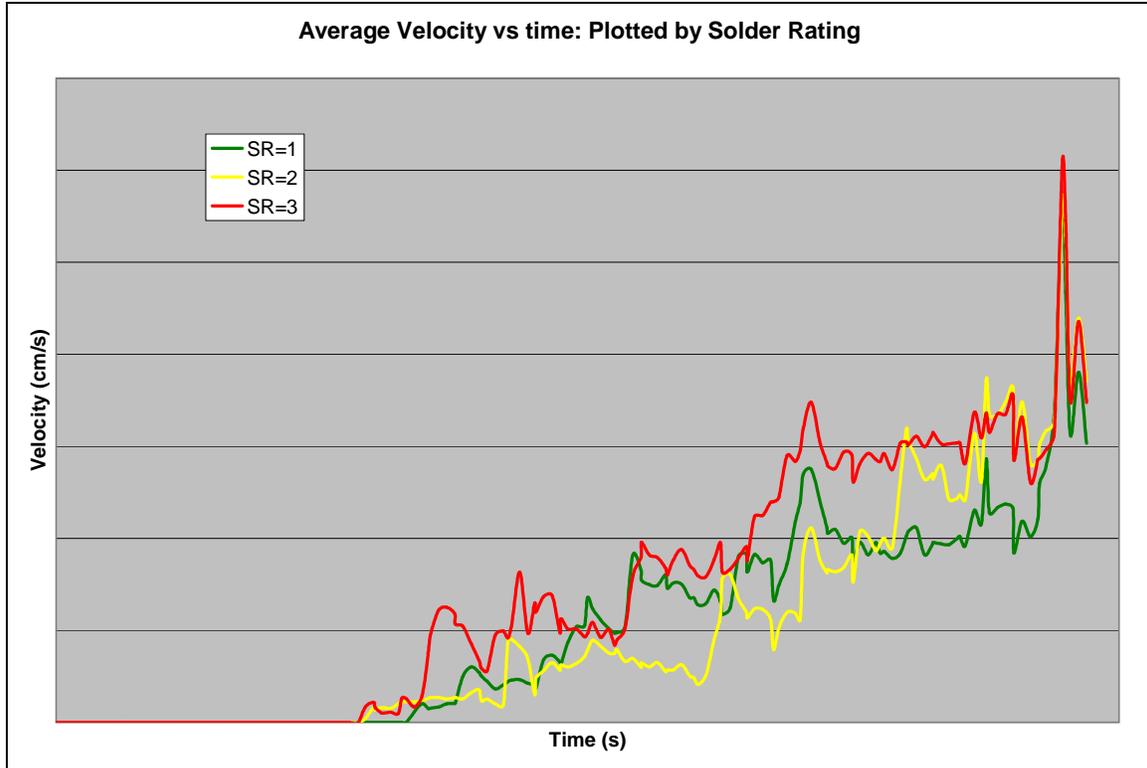


Figure 45: Average velocity during filling for locations with solder rating 1 (green), 2 (yellow) and 3 (red) at the aluminum surface.

### 'Solder Rate' Results

Using the constants calculated for the soldering rate Arrhenius equation above, the temperature data vs. time can be converted to soldering rate vs. time. The results of this conversion are plotted for each location in Figure 46. Comparing this plot to the plot of time vs. temperature in the aluminum at the part surface, it can be seen that the differences in temperature are more pronounced, especially at higher temperatures. Additionally, as temperature falls, the soldering rate falls more quickly; this should more closely model the situation. Indeed, the locations where soldering is not occurring have very low soldering potentials throughout the cycle.

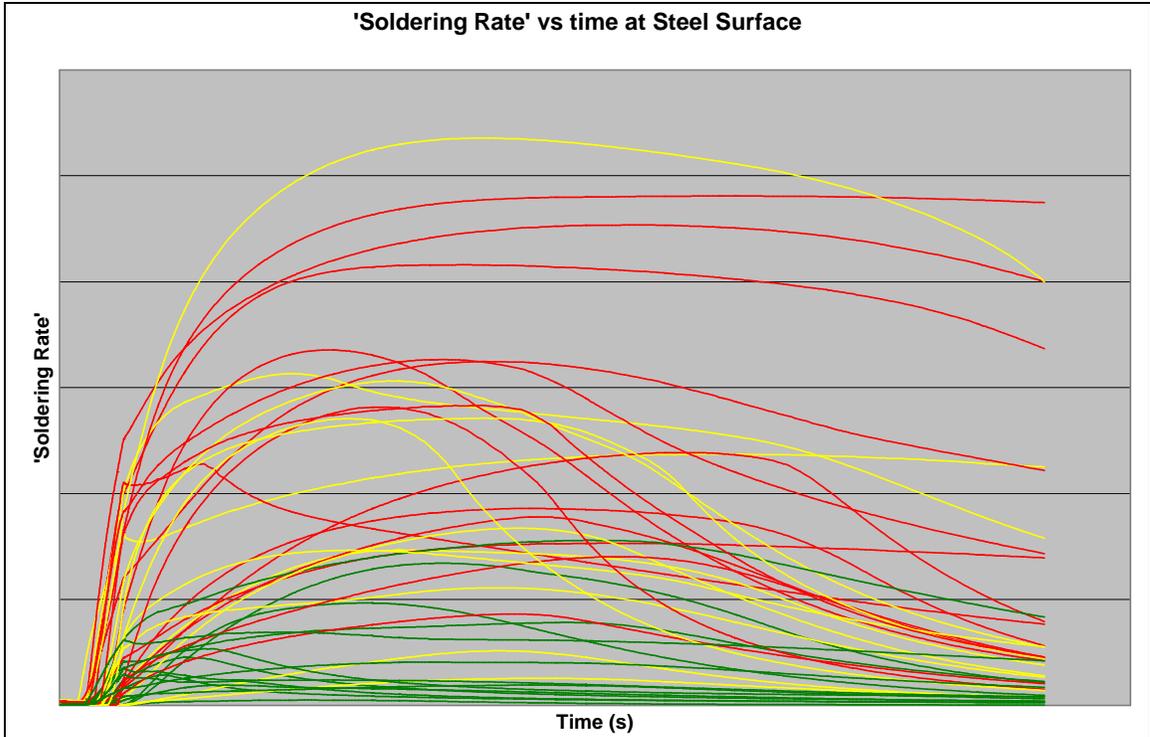


Figure 46: Time vs. 'Soldering Potential' curves for each of the 41 locations in the surface of the die steel. Green curves indicate no soldering; yellow moderate soldering; red severe soldering.

Integrating the area under these curves should combine the temperature and time effects into one variable. The results of this integration are plotted by solder rating in



**Figure 47: Results of integration of the solder rate vs. temperature curves, plotted by solder rating.**

As the goal is to identify a variable which can predict soldering, the results can be tested with ANOVA and t-tests. If the results are different as grouped by solder rating, then we know that the integrated area is a predictor of soldering.

One Way Analysis of Variance						Thursday, April 24, 2008, 11:56:46 AM
<b>Data source:</b> Data 1 in Notebook 1						
Dependent Variable: Col 2						
<b>Normality Test:</b> Failed (P < 0.050)						
Test execution ended by user request, ANOVA on Ranks begun						
Kruskal-Wallis One Way Analysis of Variance on Ranks						Thursday, April 24, 2008, 11:56:46 AM
<b>Data source:</b> Data 1 in Notebook 1						
Dependent Variable: Col 2						
Group	N	Missing	Median	25%	75%	
2.000	12	0	25.213	19.622	45.325	
3.000	15	0	36.547	29.122	56.872	
1.000	14	0	4.239	2.322	12.410	
H = 21.323 with 2 degrees of freedom. (P = <0.001)						

The differences in the median values among the treatment groups are greater than would be expected by chance; there is a statistically significant difference ( $P = <0.001$ )

To isolate the group or groups that differ from the others use a multiple comparison procedure.

All Pairwise Multiple Comparison Procedures (Dunn's Method) :

Comparison	Diff of Ranks	Q	P<0.05
3 vs 1	19.976	4.487	Yes
3 vs 2	5.167	1.114	No
2 vs 1	14.810	3.143	Yes

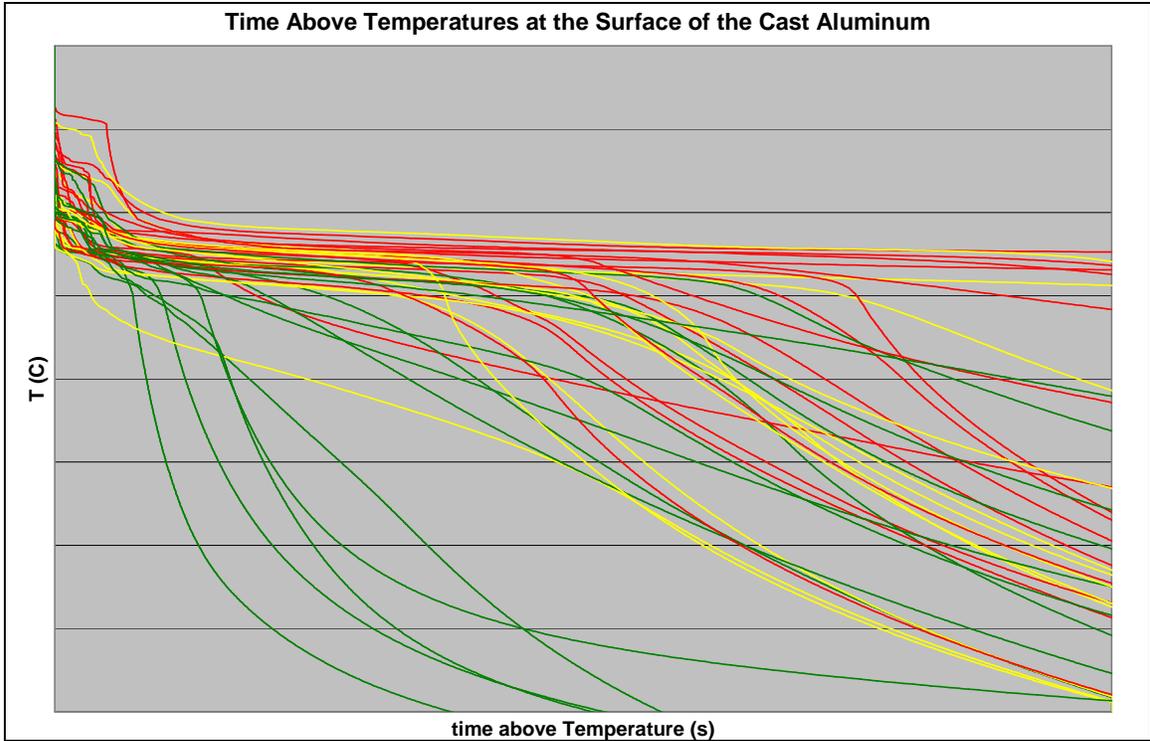
Note: The multiple comparisons on ranks do not include an adjustment for ties.

From these results, it can be seen that the area is a good predictor of soldering, as indicated by the p value of  $<0.001$  ( $>99.9\%$  confidence in the difference). However, the groups 2 and 3 are not statistically significant, so it is difficult to tell the severity of soldering by this method.

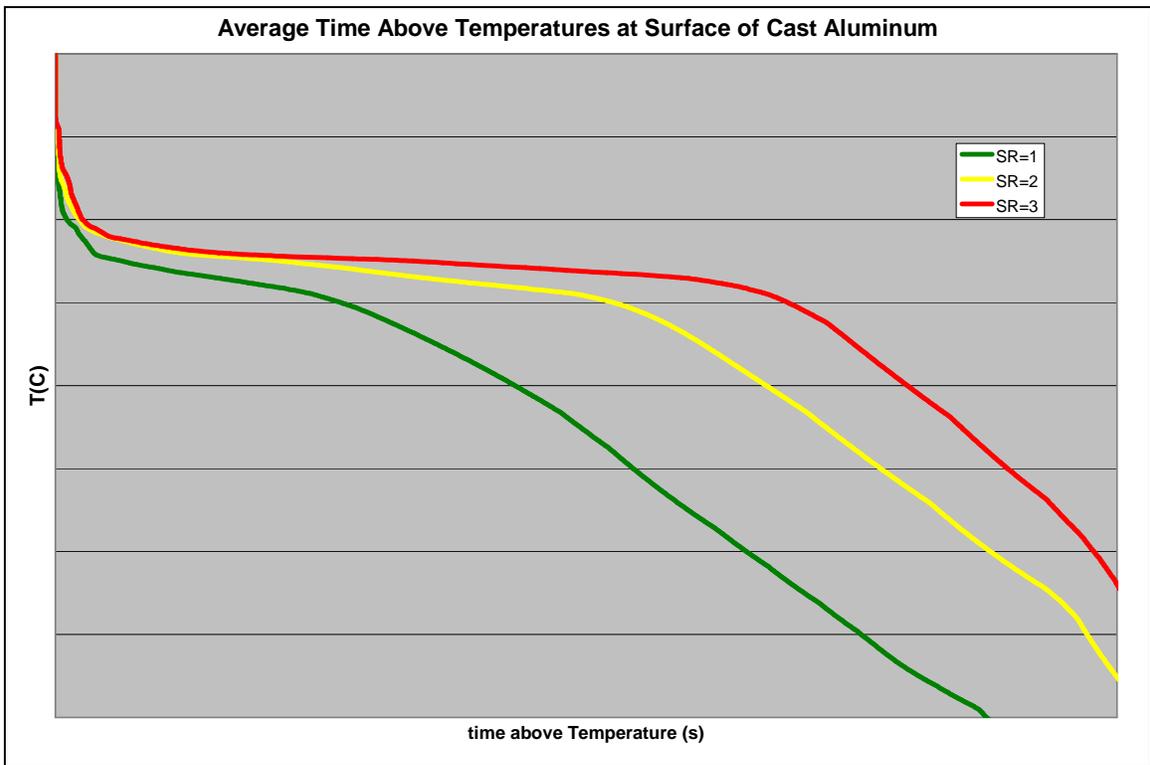
#### **'Bearing Curve' Analysis Results**

Figure 48 through Figure 51 show plots generated by the bearing curve method. For example, in Figure 15, as temperature increases up the y-axis, the amount of time spent above that temperature, plotted on the x-axis, decreases.

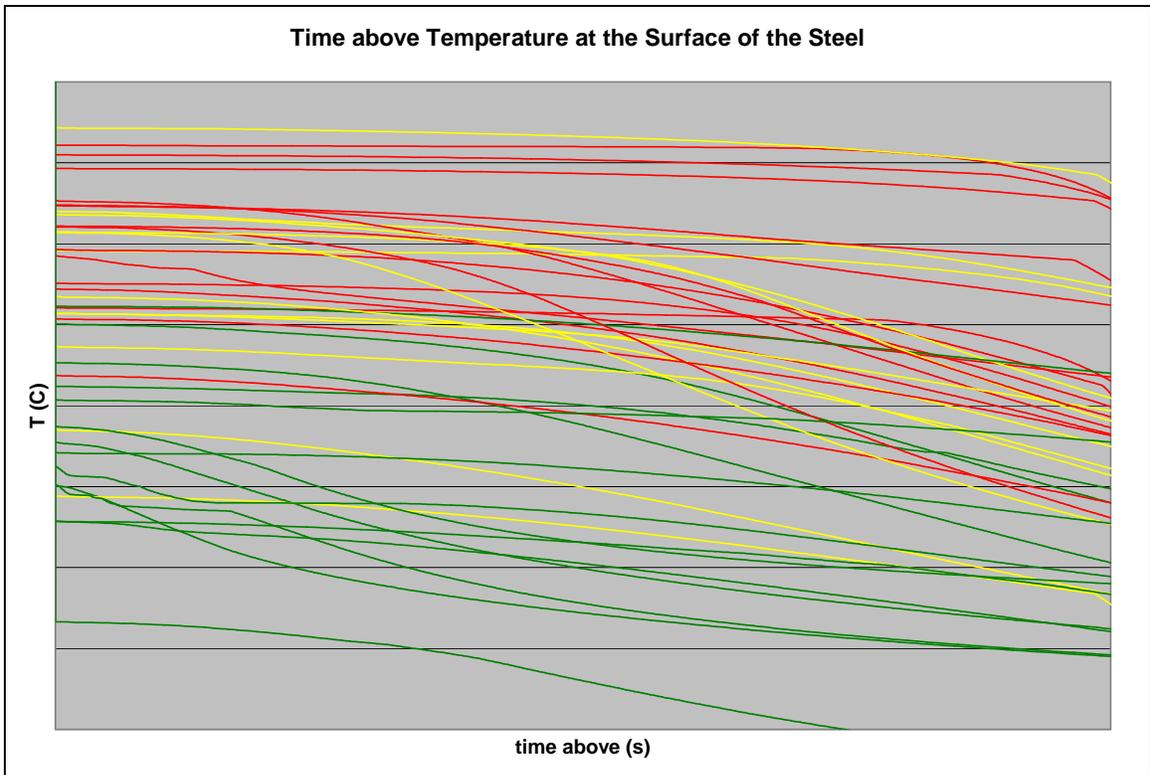
As with the time vs. temperature curves plotted above, there is a general trend where more time above hotter temperatures seems to relate to soldering.



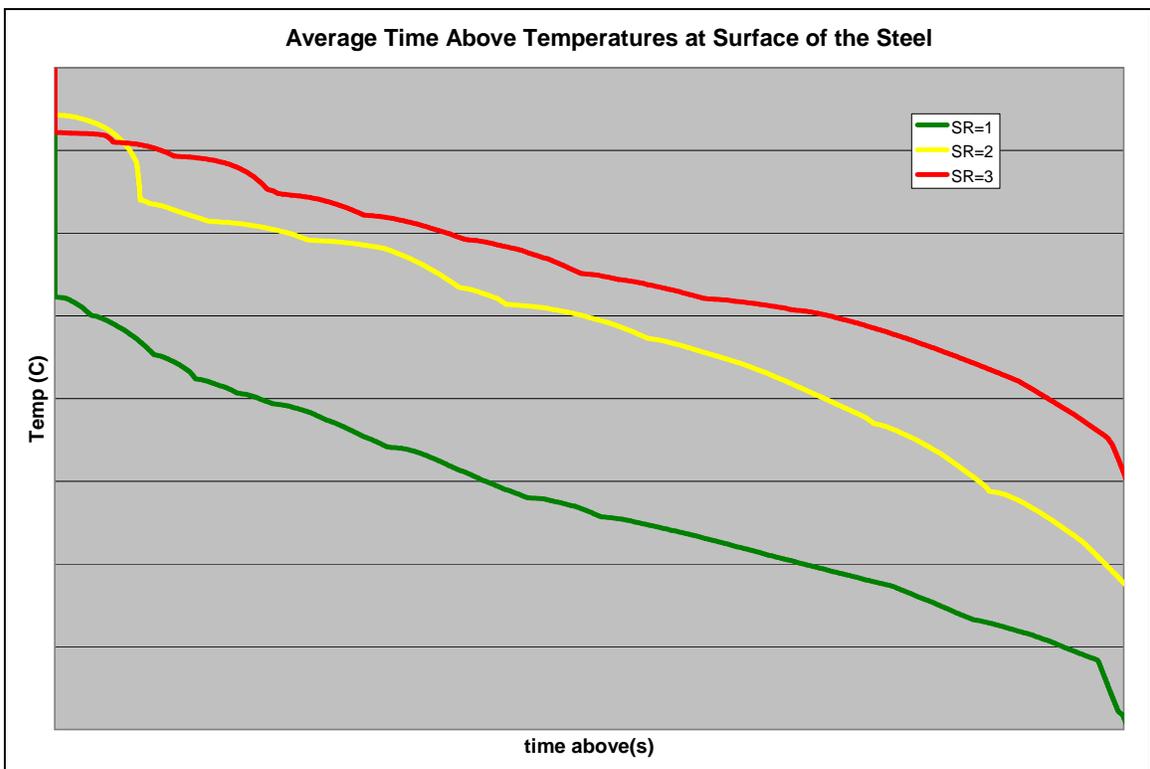
**Figure 48: Bearing Curve analysis of the time vs. temperature data at the surface of the cast aluminum.**



**Figure 49: Average time above temperatures at the surface of the cast aluminum.**



**Figure 50: Bearing Curve analysis of the time vs. temperature data at the surface of the die steel.**



**Figure 51: Average time above temperatures at the surface of the die steel.**

## Statistical Test Results

Table 13: One-Way ANOVA Results between solder rating groups (1,2,3)

One-Way ANOVA	
Variable	Significance (p)
H13: Max T	<0.001
H13: Avg T	<0.001
H13: Avg Rate	<0.001*
Al: Max T	0.077
Al: Avg T	0.006*
Max Vel	0.517
Avg Vel	0.379*
Max Al $t>T @ T_{crit, Al}^{1,2,3}$	<0.001*
Max Fe $t>T @ T_{crit, Fe}^{1,2,3}$	<0.001*
* <i>Kruskal-Wallis test</i>	

Table 13 shows the results of a one-way ANOVA, testing for significant differences between groups sorted by solder rating. The groups are significantly different at a wide range of temperature variables, with the exception of the maximum aluminum temperature. This is intuitive, since at some point during filling each location in the casting reaches a temperature close to the casting temperature, subtracting any heat losses during the metal flow to that point. The velocity variables do not show any statistical significance between groups.

The notation  $T_{crit, Al}^{1,2,3}$  refers to the critical (highest significance between group means) aluminum temperature for groups 1, 2 and 3 (no soldering, moderate soldering, severe soldering, respectively). The notation will be used throughout the results section.

Figure 52 shows how the aforementioned critical temperatures were determined throughout the bearing curve analyses. Temperature is plotted on the x-axis, while the significance between groups (using the non-parametric Kruskal-Wallis test) is plotted on the y-axis for each temperature. The plot (Figure 52) shows that the temperature of the steel has a wider range where the significance is large between groups.

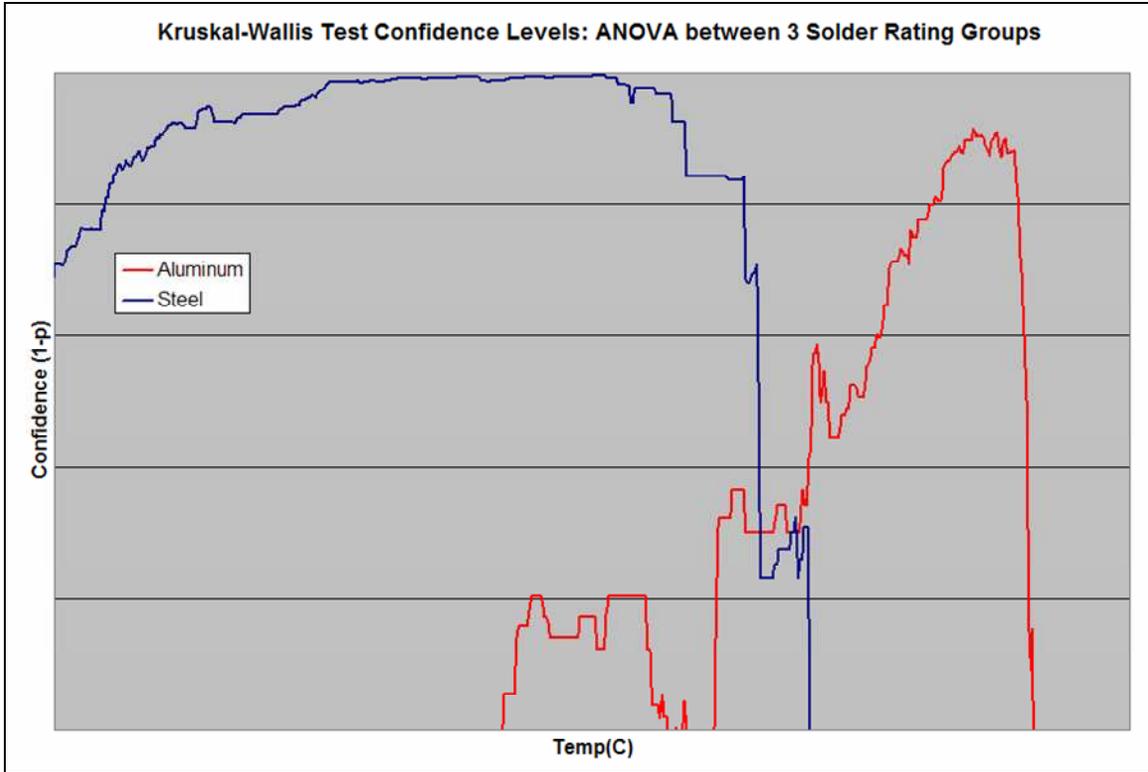


Figure 52: Results of the Kruskal-Wallis test at each temperature for the bearing curve analysis.

In order to predict soldering with any precision, however, each of the three groups need to be able to be differentiated from the others. In order to do that, t-tests between each of the groups are performed.

Table 14: t-test results between groups 1 and 2

1v2	
Variable	Significance (p)
H13: Max T	<0.001
H13: Avg T	<0.001
H13: Avg Rate	0.001*
Al: Max T	0.571
Al: Avg T	0.029
Max Vel	0.460
Avg Vel	0.949
Max Al t>T @ T <sub>crit,Al</sub> <sup>1,2</sup>	0.016*
Max Fe t>T @ T <sub>crit,Fe</sub> <sup>1,2</sup>	<0.001*
*Mann-Whitney Test	

Table 15: t-test results between groups 1 and 3.

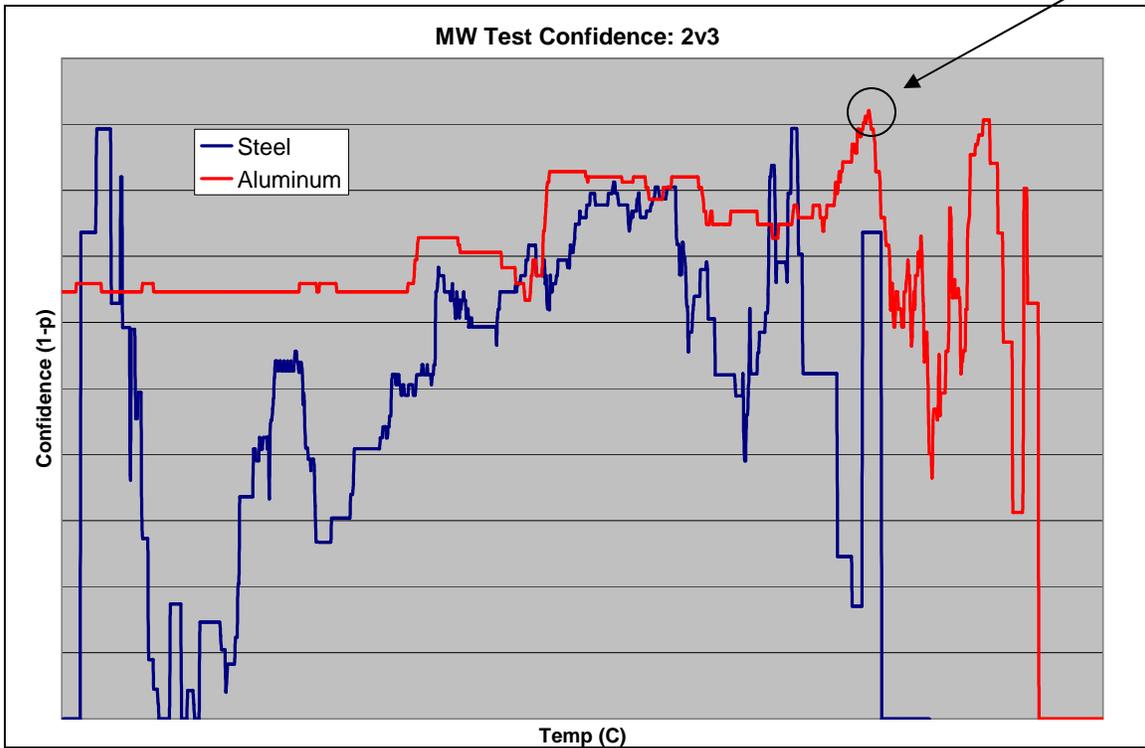
1v3	
Variable	Significance (p)
H13: Max T	<0.001
H13: Avg T	<0.001
H13: Avg Rate	<0.001*

Al: Max T	0.077*
Al: Avg T	0.002*
Max Vel	0.230*
Avg Vel	0.166
Max Al t>T @ T <sub>crit,Al</sub> <sup>1,3</sup>	<0.001*
Max Fe t>T @ T <sub>crit,Fe</sub> <sup>1,3</sup>	<0.001*
<i>*Mann-Whitney Test</i>	

**Table 16: t-test results between groups 2 and 3.**

<b>2v3</b>	
<b>Variable</b>	<b>Significance (p)</b>
H13: Max T	0.149
H13: Avg T	0.174
H13: Avg Rate	0.213*
Al: Max T	0.143
Al: Avg T	0.162
Max Vel	0.799
Avg Vel	0.196*
Max Al t>T @ T <sub>crit,Al</sub> <sup>2,3</sup>	0.079*
Max Fe t>T @ T <sub>crit,Fe</sub> <sup>2,3</sup>	0.106*
<i>*Mann-Whitney Test</i>	

As Table 16 illustrates, the true difficulty in the problem is not telling when soldering will occur, but rather in telling how severe it will be when it occurs. Only one variable, the amount of time that the aluminum spends above T<sub>crit,Al</sub><sup>2,3</sup> degrees C, is different between groups with >90% confidence. Looking at the plot of the confidence with temperature however, this result is somewhat suspect since appears to be simply a quick spike in the data rather than a general trend, since the entire curve is rather noisy.



**Figure 53: Results of the Mann-Whitney test between groups 2 and 3 at each temperature for the bearing curve analysis. Maximum confidence occurs at 565.5C in the Al curve, as indicated.**

To confirm the fact that the true difficulty in predicting soldering behavior is the ability to differentiate between different levels of soldering severity, tests were run to test the differences between areas where soldering is not occurring (solder rating 1, group NS – no solder) and areas where soldering is occurring (solder ratings 2 and 3, group S - solder). The results of these tests are shown in Table 17.

**Table 17: t-test results between groups solder (S, ratings 2 and 3) and no solder ( rating 1).**

SvNS	
Variable	Significance (p)
H13: Max T	<0.001
H13: Avg T	<0.001
H13: Avg Rate	<0.001*
Al: Max T	0.107
Al: Avg T	0.003*
Max Vel	0.226*
Avg Vel	0.721*
Max Al t>T @ $T_{crit,Al}^{S,NS}$	<0.001*
Max Al t>T @ $T_{crit,Fe}^{S,NS}$	<0.001*

\*Mann-Whitney Test

All variables, except for the two velocity variables and the maximum aluminum temperature, are different at a confidence level of >99%. This confirms the fact that the

group means are significantly different when comparing locations where soldering is occurring to those where soldering is not occurring. The Mann-Whitney plot for SvNS is shown at the end of the report.

A similar analysis was performed on both the U222 Carrier geometries available: the older long runner version and the more up-to-date short runner geometry. The results of the two were not appreciably different.

### ***IV.III Review of MAGMA Die Soldering Module***

MAGMA offers a simple die soldering prediction module. After the previous results were arrived at, this module was reviewed, with the results in mind, in order to attempt to determine how useful the module might be. If it is accurate, this module would be the most cost effective way of implementing a solder prediction effort, since the coding and implementation will be nearly already complete.

The MAGMA die solder prediction module is adapted from the sand casting burn-on calculation module. The module calculates the length of time during the solidification of the part that the temperature of each cell at the surface of the mold is above a certain criterion temperature. The criterion temperature can be changed by the user in a configuration file. The results can then be plotted on either the mold or casting surface.

The underlying theory is that a length of time  $t$  above a certain temperature  $T$  will cause the die lubricant to break down and expose the die surface to the cast metal, leading to soldering. MAGMA suggests 450 degrees Celsius and 15 seconds for the  $T$  and  $t$ , respectively, but these values obviously depend on many variables specific to any casting system (lubricant chemistry and concentration, alloy, etc.).

#### **IV.III.I Evaluation Procedure**

In order to analyze the U222 Carrier system, the module was activated and several cycles were run on the latest U222 model with the short runner, each cycle with a different criterion temperature. Five temperatures were considered for analysis, suggested by the results of some of the previous work. Using these five temperatures should give a good idea of where the best target temperature is for the module for the U222 Carrier, and hopefully for future models.

Images of the die solder rating system (yellow: moderate soldering; red: severe soldering) are compared to screenshots of the casting with the results of the soldering

module shown on the casting surface. Optimally, the red severe soldering regions will correspond to the longest times in the solder module results.

### IV.III.II Results

One of the significant issues with the way that the module is set up is the time during which the calculation is performed. The problem is that the MAGMA module considers the entire solidification time of the cycle, both with the die closed and with it open and the part removed, but not the filling cycle, as the relevant time period for soldering (call this time  $t_2$ ). In fact, the casting is only in contact with the mold for the mold filling time and the part of solidification before the die opens (call this time  $t_1$ ), and this is the time during which soldering can occur, while the casting is in contact with the mold. Due to this, there is some disagreement between the results. Table 18 shows the difference in these two time periods. It should be noted that  $t_2$  is significantly larger than  $t_1$ .

**Table 18: Time period T1, where the casting is in contact with the mold and T2, where MAGMA is calculating the die soldering criterion.**

filling	solidification	
	die closed	die open
$t_1$		
	$t_2$	

The reason for this is probably due to the fact that the module is adapted from the sand casting burn-on module. An open die is never really a consideration as part of the cycle in a sand casting, but it is a large portion of the cycle in permanent mold casting, after one shot is complete and preparations are being made for the next shot, so the module does not directly translate to permanent mold casting in that regard.

The question is, how damaging is this to the results of the module? Is the extra time added proportional or a constant so that it can be removed easily; or does it add more time to some locations than others? To test this, the time above  $t_1$  (x-axis on the following plots) was compared to the time above  $t_2$  (y-axis on the following plots).

The results of this comparison can be seen in Figure 54. Unfortunately, it appears that this problem can significantly affect the results. The locations circled in red in the figure would seem to suffer from 15-20% more soldering if  $t_2$  is used. However, these points are actually in contact with the cast metal above the critical temperature for

roughly the same amount of time as those below them (same value for  $t_1$ ), and should have nearly the same soldering behavior.

The correlation between the times is actually fairly good, at  $R^2=0.9651$ , but this is probably somewhat misleading, since most of the error appears in only a few locations, so these few points would be seriously inaccurate if the results were to be used; and it is these points that we are most interested in distinguishing, since the degree of soldering is very important. Therefore, some way is needed of correcting this error.

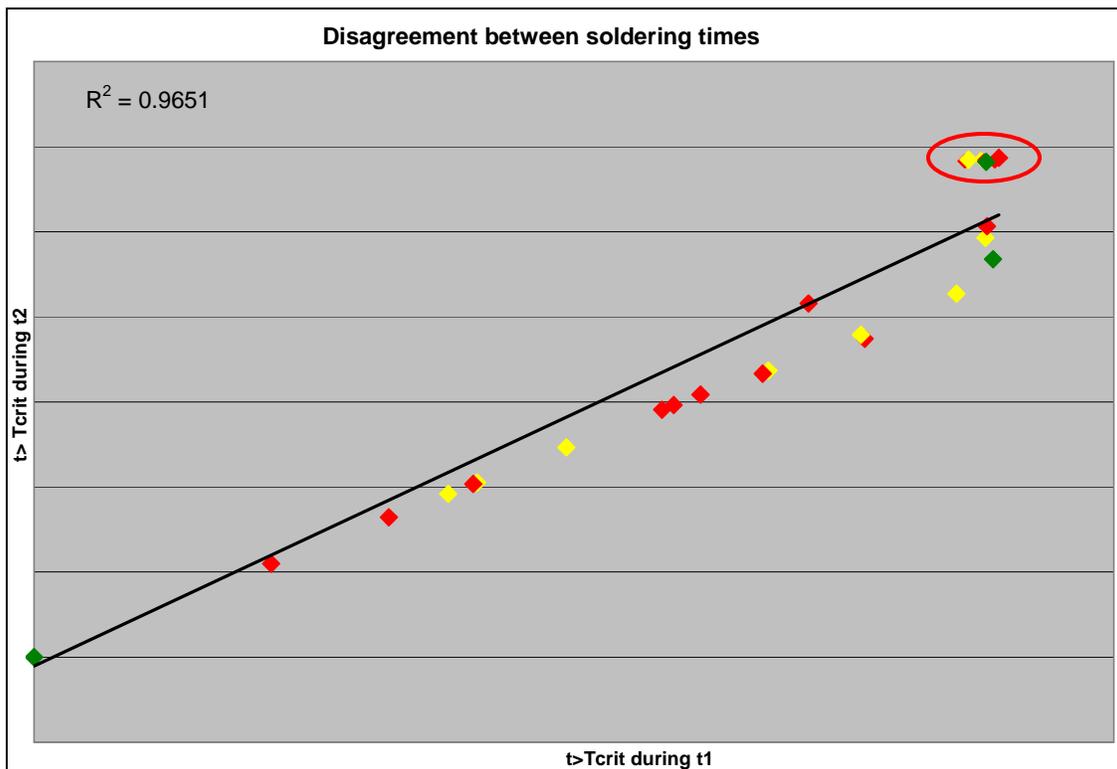


Figure 54: Plot comparing the time the mold is above 453.5C during the period MAGMA calculates the die solder module results and the time the casting is in contact with the mold.

One potential solution to this is to cap off the results, with the maximum time possible equal to  $t_1$ , since it is only possible to be in contact with the casting for this long above the critical temperature, and any additional time above this will solely be due to the unrealistic calculation period. In Figure 55, the results of this 'capping' are shown (on the y-axis). The correlation improves to  $R^2=0.9969$ .



**Figure 55: Plot comparing the time the mold is above 453.5C during the period MAGMA calculates the die solder module results (3-91s), capped at the dwell time; and the time the casting is in contact with the mold (filling + dwell time).**

There is only 1 location where the capped time above  $T_{crit}$  during  $t_2$  differs significantly from time above  $T_{crit}$  during  $t_1$ . These results indicate that it is best to use this capping technique in order to reduce error. The results shown below in the images will be of the solder module results capped at  $t_1$ , rather than the raw module results, since the capping seems to improve the accuracy of the predictions.

The capping was performed by changing the scale of the presented results, with a range from 0- $t_1$  seconds.

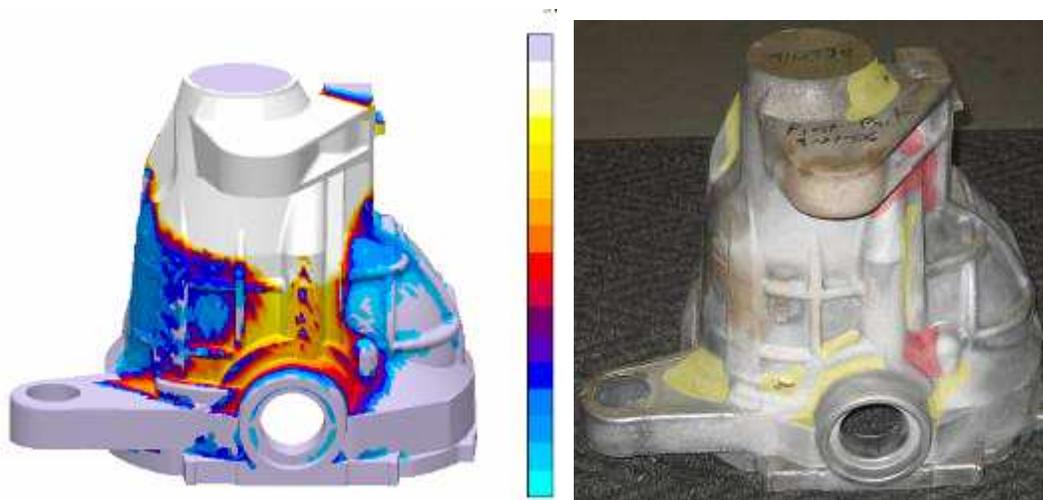
The module was run at five criterion temperatures. The five temperatures were selected based upon some of the results of the original study. These were selected as follows:

1. T1: selected as a low temperature
2. T2: critical temperature for comparisons between groups 1,2,3; groups 1,3 and groups S,NS
3. T3: selected as an intermediate temperature
4. T4: critical temperature for comparisons between groups 2,3
5. T5: selected as a high temperature

Using five temperatures hopefully allows for the determination of an optimal  $T_{crit}$  to be used in future projects. The findings from this investigation are summarized as follow.

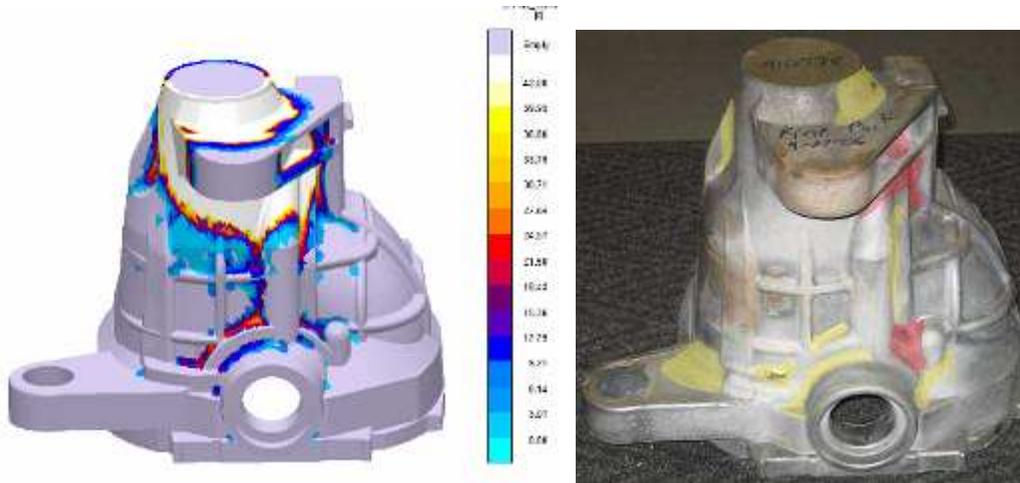
At T2, suggested by the data mining work, the module predicts significant soldering in the areas near the gate. The actual parts in production do not experience such significant soldering behavior over this entire region; although there are definitely some locations in this area that do solder significantly. The areas in this location that do seem to solder severely are those locations where a piece of steel is surrounded by a large amount of cast aluminum during the cycle. These regions should be hotter than the others in the area, but the middle temperature found by the data mining work must not be a criterion temperature capable of making this distinction. Away from the large hot area by the gate, the module seems to correlate reasonably well to the rating images, with the critical time being somewhere around 30-35 seconds (nearly the entire time the cast part touches the mold).

The two images in Figure 56 represent typical results from this criterion temperature.



**Figure 56: Representative image showing results at T2.**

At the higher critical temperature found from data mining, T4, soldering is predicted well in the areas near the gate. However, there are some locations further from the gate that suffer from soldering that are not predicted by the module. Figure 57 shows a representative example of these modeling results.



**Figure 57: Representative image showing results at T4.**

The results from T1, T3 and T5 are as expected, following the trend of over prediction of soldering near the gate compared to the prediction in areas further from the gate. No optimal  $T_{crit}$  was determined from this analysis.

#### ***IV.IV Conclusions***

- The temperature of the die steel is the most important factor to control (assuming the alloy and part geometry are set). This is proven by statistical tests, which show that the temperature of the steel (maximum, average) is a much better indicator of soldering than the temperature of the cast metal at the surface of the die.
- The time above  $T_{crit}$  predictor is the most promising for a simple solution to prediction of soldering, as MAGMASoft already markets a die solder prediction module which measures this variable. An optimal critical temperature and the means to translate from a given time above that temperature to a severity of soldering are determined through the data mining approach. These results have been supplied to Contech.
- Cast metal velocity does not appear to strongly predict soldering. However, the velocities during squeeze casting are much lower than in high pressure die casting, where velocity may play a larger role.
- The results above are specific to an ADC12 casting under squeeze casting conditions. Care should be taken in generalizing the results above to other, different systems under different casting pressure, using different alloys and any other significant process differences.

## **V. Reduction of Die Soldering: Strontium Modification**

As is discussed in the literature review, a small addition of strontium (0.02-0.03 wt%) to an aluminum alloy changes the surface energy of the alloy significantly. This change is responsible for a reduction in die soldering. With this potential for solder mitigation in mind, experiments were carried out on the U222 Carrier casting at the Pierceton, IN casting plant to document the entire process effects of using strontium modified metal.

Specifically, reductions in die soldering and increases in porosity scrap were monitored. The target strontium level for the study was .020-.030 wt% Sr, consistent with other modification programs at Contech and in the literature.

The initial investigation revealed that there are significant benefits in terms of downtime reduction when using strontium modified metal. However, the scrap rate also increased significantly, to a point where the benefits of using strontium were outweighed. In order to realize the benefits of strontium, process changes were proven necessary. A follow-up investigation to determine these optimal parameters was carried out subsequently. These investigations are discussed below.

### ***V.1 Initial Investigation: Method***

The frequency and severity of soldering was measured through a die-face cleaning log filled out by the machine operators. The log detailed the length of time and locations of all die face cleanings. The amount of soldering was quantified by determining the length of cleaning time necessary per shot.

Additionally, a series of photos was taken of the several high-solder areas of the die face for the cases of unmodified and modified ADC12 in order to visually document the development of soldering on the die face. These photographs were taken at set intervals of 40 castings over the course of one run from cleaning to cleaning.

Die Cast and Machining EPS (Employee Production Sheet) reports were collected for each shift during the trial. Scrap rates were calculated by dividing the number of scrap parts (found on the Machining EPS, identified by date code and cavity number) by the total number of good parts cast on those dates (found on die cast EPS).

Metal chemistry samples were collected over the course of the trial to monitor the composition of the metal, specifically the Sr level.

A three-day preliminary study of the process status using unmodified ADC12 was conducted as a way to test data collection methods and to use as a baseline for comparison with the data from the run using Sr-modified ADC12. Due to data collection methods at the plant, the three days used for die-cleaning monitoring did not coincide completely with the three days used for scrap rate monitoring. The dates for the baseline study are shown in the chart.

**Table 19: Baseline Study Dates**

Date	Shift	Test	
		Cleaning	Scrap
25-Jul	3		
25-Jul	1		
25-Jul	2		
26-Jul	3		
26-Jul	1		
26-Jul	2		
27-Jul	3		
27-Jul	1		
27-Jul	2		
28-Jul	3		

Starting first shift on July 30 (7:00 AM) strontium modified ADC12 began to be added to the furnace at DCM 16. At 2:30 PM, the chemistry was tested, and it was found that the Sr level had only risen to 0.00875 wt%. At this point, the decision was made to charge the furnace with Sr tubes in order to achieve the target Sr level. This was done, and the strontium level rose to the target level by the start of third shift, where it was at 0.02385.

However, since no PCM (Process Change Memo) had been created for the change in metal chemistry, the modified ADC12 stopped being added some time during 3<sup>rd</sup> shift on the morning of July 31. When the chemistry was tested at the start of 1<sup>st</sup> shift on July 31, the Sr level had fallen to 0.00678. At this point, a formal PCM was created, and the furnace was charged with Sr tubes again at approximately 9:30 AM. By 11:30, the Sr level had risen to above 0.020, and was maintained within the target range by using Sr-modified ADC12 ingots in the furnace and by adding 1 Sr tube every two hours.

However, during 2<sup>nd</sup> shift on July 31 and 1<sup>st</sup> shift on August 1, the Sr-modified parts went through the machining line, with very high scrap rates. Since the U222

Carriers were at that point under heavy demand by the customer, the decision was made to end the trial to ensure that the demand could be met.

Due to the decision to prematurely end the trial, only four shifts of die cleaning data were collected while the Sr level was within the target range (start of 1<sup>st</sup> shift on July 31-end of 1<sup>st</sup> shift on August 1). Scrap rates were calculated for July 30-31 and August 1.

## V.II Initial Investigation: Results & Discussion

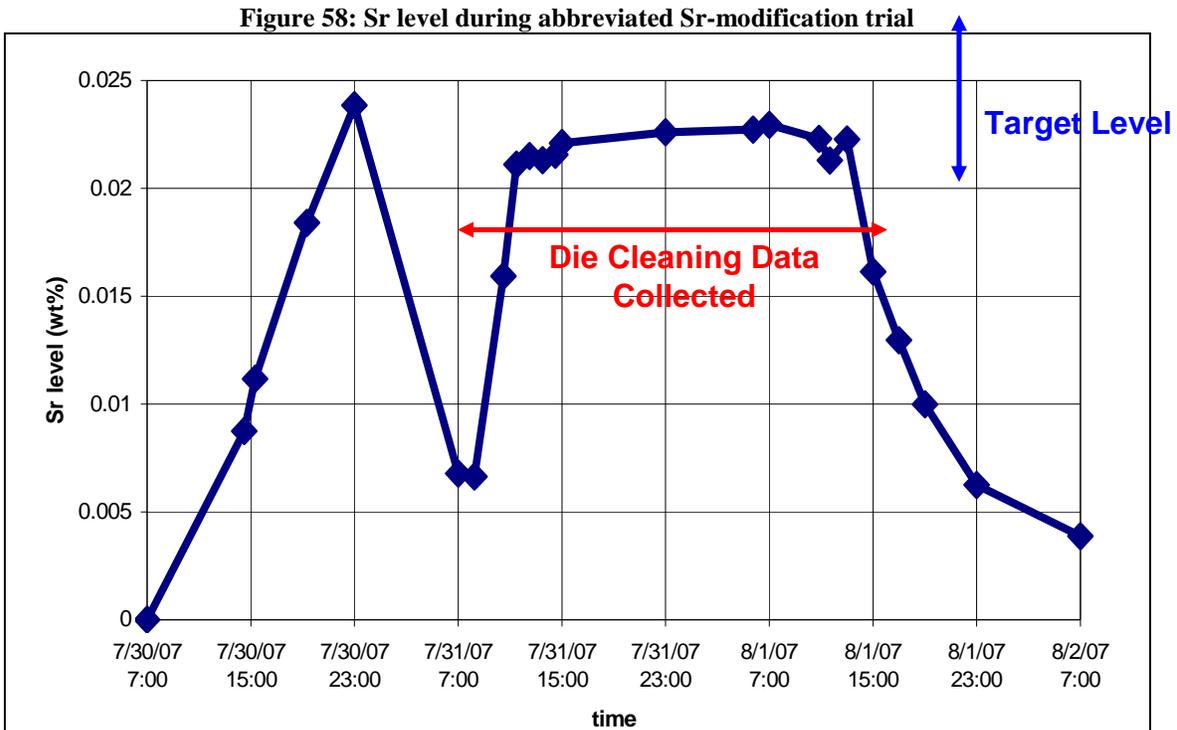


Figure 58 shows how the Strontium level varied over the length Sr-modification trial. This is not the optimal case, but it was the most that could be done at the time. The experience gained in carrying out this investigation was invaluable for success in the follow-up studies. The results of the process monitoring are discussed below.

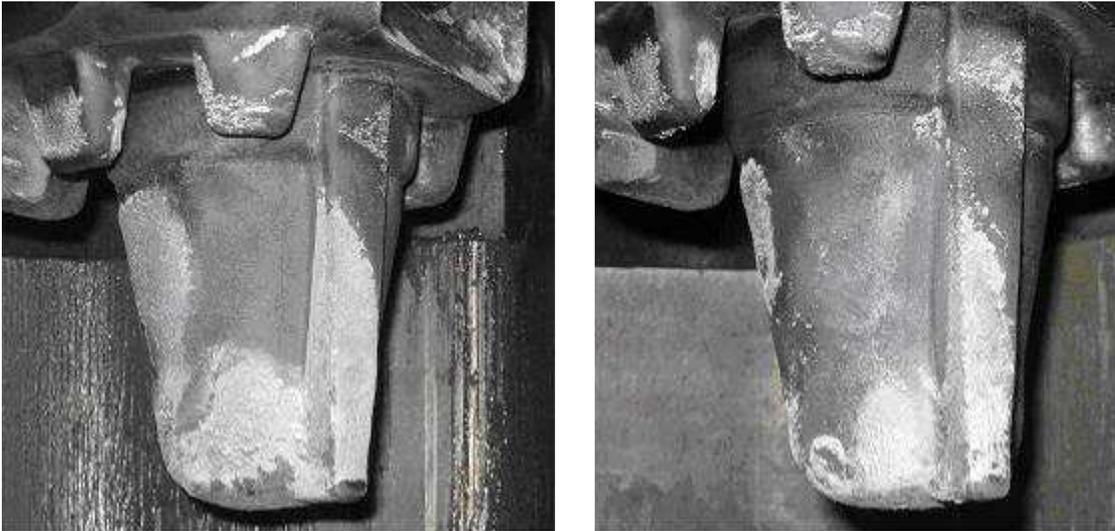
### V.II.I Effect of Strontium Modification on Die Soldering

During the nine shifts while die-cleaning data was collected for the preliminary study with unmodified ADC12, 2043 shots were made. During that time, the operators spent 305 minutes cleaning the die. This is a total of 0.149 minutes of cleaning per shot.

Over the four shifts where die-cleaning data was collected during the strontium modified trial, 1142 shots were made. In that time, the operators took 125 minutes to

clean the die. This is a total of 0.109 minutes of cleaning per shot, a 26.8% reduction in die-cleaning time from when the unmodified alloy was being used.

Visually, a difference in solder build-up is apparent when considering pictures of the die face during the trials. As seen in **Error! Reference source not found.** below, taken 40 shots after cleaning, significantly more stuck aluminum has built up on the core tip when unmodified ADC12 is being used. The tip is much cleaner in the image on the right where Strontium modified aluminum is being used, although there is still some aluminum stuck to the die.



**Figure 59: Core Tip, 40 shots after cleaning. Unmodified ADC12 at left, Sr-modified ADC12 at right.**

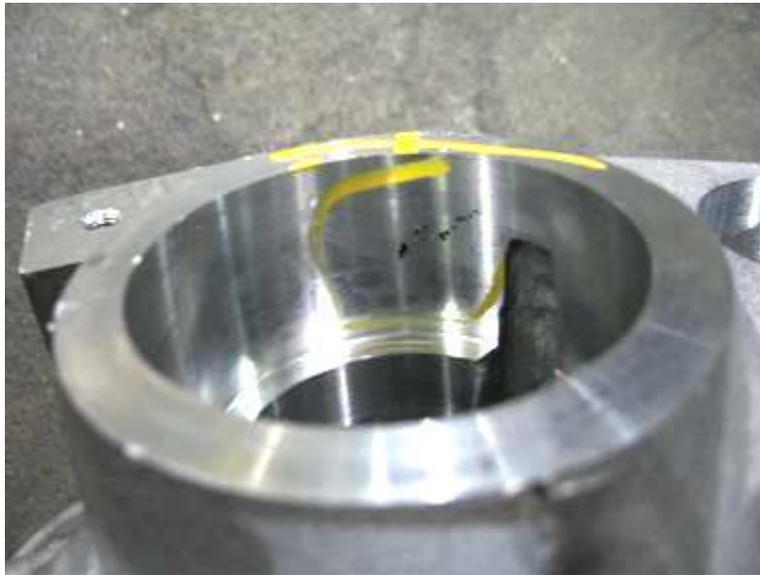
Similarly, 120 shots after cleaning, it can be seen in the axle bore area of the die that the solder has spread over a wider area in the case of unmodified alloy than when strontium modified metal is being used.



**Figure 60: Axle Bore, 120 shots after cleaning. Left: Unmodified ADC12, Right: Sr-modified ADC12**

### **V.II.II Effect of Strontium Modification on Scrap Rate**

One concern with Strontium modification is porosity, discussed in the literature review section. There is typically some porosity in the part, but it is in a location where it machined away during subsequent operations. As long as there is no visible porosity after machining, the part passes quality inspection. However, strontium has the effect of changing the location and perhaps the amount of porosity in the part, sometimes into an area where it is visible to inspectors after machining. In this case the part must be scrapped. It is this porosity scrap that increased to the point where the trial needed to be stopped.



**Figure 61: Example of visible porosity after machining, resulting in increased scrap.**

Table 2 shows the number of parts cast and machining scrap rate by day. The increase in scrap rate when Strontium modification is occurring is very significant. It should also be noted that these three days do not represent a time period where Strontium modification was being used constantly, since the Strontium level was changing, as was discussed above. This explains the lower scrap rate on July 30, where the Sr level was not at the target for the entire day.

**Table 20: Scrap Rates by Day. 7/25-7/27: unmodified; 7/30-8/1: modified**

Date	Cast Parts	Por. Scrap	Leak Scrap	Blist. Scrap	Scrap Rate
25-Jul	664	2	1	0	0.5%
26-Jul	550	10	1	1	2.2%
27-Jul	644	19	2	1	3.4%
<b>Unmodified</b>	<b>1858</b>	<b>31</b>	<b>4</b>	<b>2</b>	<b>2.0%</b>
30-Jul	574	73	3	0	13.2%
31-Jul	825	214	1	1	26.2%
1-Aug	775	146	0	0	18.8%
<b>Modified</b>	<b>2174</b>	<b>433</b>	<b>4</b>	<b>1</b>	<b>20.1%</b>

The drop in scrap rate from July 31-August 1 can potentially be explained by a process change made at 5 AM on that day in response to the very high scrap rates being seen in the parts cast the previous day. The dwell time on the die was increased at this point by 3 seconds. This change allows for pressure applied to the critical region of the die for longer, so feeding from the gate below can continue and hopefully prevent some

shrinkage porosity from forming. The lower rate may also be attributed to the reduced strontium level in production later in the day.

The method for calculating scrap rate is somewhat unreliable due to the fact that the operators sometimes neglect to write the date code and cavity number of the scrap parts in their EPS. Due to this, it was sometimes necessary to make educated guesses when assigning scrap parts to a specific date. However, with the magnitude of the change, this error is not significant enough to cast doubt onto the findings of the study.

### **V.II.III Effect of Strontium Modification on Process Output**

If it is accepted that the most detrimental aspect of die soldering is the loss of production during process downtime, and that erosion and other effects can be neglected, a process output model can be designed to compare the beneficial effects of strontium modification on reducing cleaning time with the detrimental effects of increased scrap rate, and used to determine whether or not strontium modification is justified.

The theoretical output for one day can be determined by considering cycle time, solder cleaning time and scrap rate, and the effect of strontium on the overall U222 Carrier output of the plant can be determined.

Die-cleaning time can be looked at as an increase in cycle time, since there is an associated time required to clean the die for each shot that is made. A formula for the total potential for parts made in a given production time where no downtime is required for anything other than solder is given:

$$\text{Total Parts} = (1 - \text{scrap rate}) * (\text{production time in seconds}) / (\text{Cycle time} + \text{Cleaning time/shot})$$

By entering the results of data collected during the trial, we can see that a strontium modification program would result in an 18.85% reduction in production rate.

From these results, using strontium modified metal in the U222 process is not justified at this point under these process conditions.

### **V.III Follow-up Investigation: Method**

The major objective to the follow-up trial was to address the issues raised by the initial trial, namely the high scrap rate that was caused by the strontium addition. To address this issue, a DOE was designed to test several process parameters for their

effect on the scrap rate in order to determine how the benefits of the Strontium modification (reduced die soldering time) might be realized.

It is suspected that the strontium modification changes the location of the shrinkage pores in the part and/or disperses the porosity, which generally results a large pore, into smaller pores. This change in location of the resulting porosity results in visible pores after machining; leading to scrapped parts and the higher scrap rate. Three variables were selected to study how the porosity location is effected:

- Strontium content: Past studies have shown that the effects of strontium are different depending on the content.
- Dwell time: The problem is suspected to be a feeding problem, since the pores which cause the scrap are shrinkage pores. An increase in dwell time should allow more time for these pores to be filled by the applied pressure, thus reducing the overall porosity in the cast part and lowering the chances that a pore may end up in a bad location.
- Metal temperature: The metal temperature may affect the effects of strontium or alter the solidification path of the part in a way that might counteract or alter any porosity movement due to strontium. It should be noted that the metal temperature is the temperature in the dip-well; some heat is lost on the transfer from the dip-well to the shot sleeve.

An L8 orthogonal array was used to study the variables. An L4 would have been sufficient, but would not have allowed for any study of interaction effects. The array is shown in Table 21. After discussions with process engineers at the plant, no fourth variable was selected, and the trial stayed with three variables in order to simplify the practical aspect of carrying out the trial.

**Table 21: Planned experimental array for follow-up investigation.**

Run No.	Sr	Metal T	Sr X MT	Dwell t	Sr X Dw t	MT X Dw t	-----
1	0.015	T <sub>high</sub>	1	t <sub>low</sub>	1	1	-
2	0.015	T <sub>high</sub>	1	t <sub>high</sub>	2	2	-
3	0.015	T <sub>low</sub>	2	t <sub>low</sub>	1	2	-
4	0.015	T <sub>low</sub>	2	t <sub>high</sub>	2	1	-
5	0.025	T <sub>low</sub>	1	t <sub>low</sub>	2	2	-
6	0.025	T <sub>low</sub>	1	t <sub>high</sub>	1	1	-
7	0.025	T <sub>high</sub>	2	t <sub>low</sub>	2	1	-
8	0.025	T <sub>high</sub>	2	t <sub>high</sub>	1	2	-

20 castings were made for each run. The strontium level was reached with a combination of charging with Al-Sr tubes in both the dip-well and holding furnace at each step in Strontium level, and also by feeding the furnace with Sr-modified ADC12

alloy throughout the trial. Once stabilized, the strontium content was checked once per trial. Any unusual events or machine errors were noted. The parts were each engraved with the run and part number in order to identify them later on.

The parts were heat treated, shot blasted, machined and leak tested and then given a final inspection to determine an effective scrap rate for each run. The severity of the porosity that led to scrap was ranked on a 0-4 scale (example images for each rating are shown in Appendix 3) for each part. Additionally, one part per run was collected with the biscuit still attached and sectioned to get a macro view of the location of the porosity in the part.

Slightly too much strontium was added when originally charging the furnace for the beginning of the trial. In order to accommodate this, the planned Sr levels of 0.015 and 0.025 wt% were adjusted upward to 0.020 and 0.030 wt%, resulting in the modified array in Table 22.

**Table 22: Final experimental array for follow-up investigation.**

Run No.	Sr	Metal T	Sr X MT	Dwell t	Sr X Dw t	MT X Dw t	-----
1	0.020	T <sub>high</sub>	1	t <sub>low</sub>	1	1	-
2	0.020	T <sub>high</sub>	1	t <sub>high</sub>	2	2	-
3	0.020	T <sub>low</sub>	2	t <sub>low</sub>	1	2	-
4	0.020	T <sub>low</sub>	2	t <sub>high</sub>	2	1	-
5	0.030	T <sub>low</sub>	1	t <sub>low</sub>	2	2	-
6	0.030	T <sub>low</sub>	1	t <sub>high</sub>	1	1	-
7	0.030	T <sub>high</sub>	2	t <sub>low</sub>	2	1	-
8	0.030	T <sub>high</sub>	2	t <sub>high</sub>	1	2	-

#### ***V.IV Follow-up Investigation: Results & Discussion***

Table 23 shows the Strontium levels for each run. The concentration on trial 1 is low, but this was taken before any warm-up shots were made with the machine while the Strontium level was rising. The sample from level 2 was taken just 30 shots later, so it is likely that the Strontium level for the majority of the run was at or very close to the target.

**Table 23: Sr levels for each experimental run.**

Run #	Sr
1	0.01318
2	0.02118

3	0.01818
4	0.01736
5	0.02674
6	0.0283
7	0.03415
8	0.033967

The pass rates for each run are shown in Table 24. The porosity rating results were shown in Table 25. Some clerical errors were made, resulting in some parts being missed and not assigned a rating.

**Table 24: DOE experimental run pass rates.**

Run #	Pass Rate
1	17.65%
2	5.00%
3	10.00%
4	16.67%
5	0.00%
6	10.53%
7	25.00%
8	15.00%

**Table 25: DOE Experimental run Porosity Ratings.**

Run Results		Porosity Rating						
Run No.	Pass Rate	0	1	2	3	4	Average	Missing
1	17.65%	0	3	9	5	0	2.12	3
2	5.00%	1	0	14	5	0	2.15	0
3	10.00%	0	2	7	8	3	2.60	0
4	16.67%	1	2	4	9	2	2.50	2
5	0.00%	0	0	8	9	3	2.75	0
6	10.53%	1	1	8	7	3	2.50	0
7	25.00%	3	2	12	2	1	1.80	0
8	15.00%	0	3	9	6	2	2.35	0

Clearly the scrap rates were much higher than what was hoped for. The Strontium modification moves the porosity around into an unacceptable location which leads to scrap. Unfortunately, time did not permit a large number of castings from the sample to be x-rayed to more specifically look at the location of the porosity, which is not entirely visible at the surface after machining. However, two parts each from runs 4 and 7 were sectioned and x-rayed, and it appeared as if the porosity was somewhat randomly located. There did not seem to be much consistency between the parts cast

from each of the runs, which suggests that the porosity may not be controllable, although these conclusions are not firm, since more parts were not x-rayed.

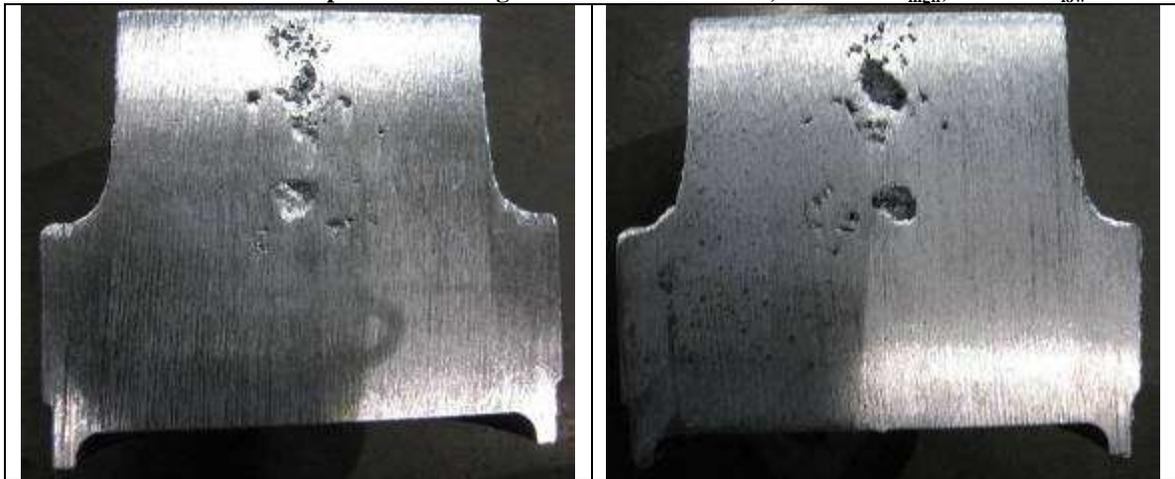
The main effects were also calculated for both the pass rate and the average porosity rating, shown in Table 26. Unfortunately, it does not seem that there is much effect of any of the variables; the castings were very bad across the board. No further analysis was done to the data, since it does not look like using Sr-modified ADC12 in the U222 Carrier process is feasible without a significant amount of additional work.

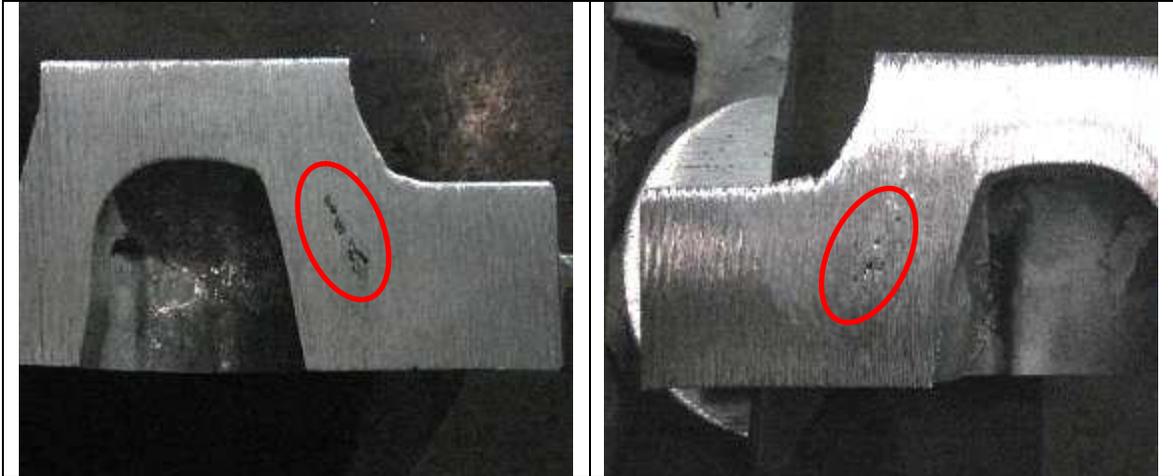
**Table 26: Main Effects Table**

Variable Level	Pass Rate	Avg. Por. Rating
Sr: 0.020 wt%	12.33%	2.34
Sr: 0.030 wt%	12.63%	2.35
Metal T: T <sub>high</sub>	15.66%	2.10
Metal T: T <sub>low</sub>	9.30%	2.59
Dwell t: t <sub>low</sub>	13.16%	2.32
Dwell t: t <sub>high</sub>	11.80%	2.38

One casting along with the biscuit was sectioned and photographed from each run. These images can show how the porosity is distributed in the part and can show the effects of different process parameters. In Table 27, images of a sectioned biscuit region of the casting are shown. The areas circled in the bottom two images shows the porosity which would lead to scrapped parts.

**Table 27: Sectioned pieces of casting from run 2. Sr: ~0.021; Metal T: T<sub>high</sub>; Dwell t: t<sub>low</sub>**



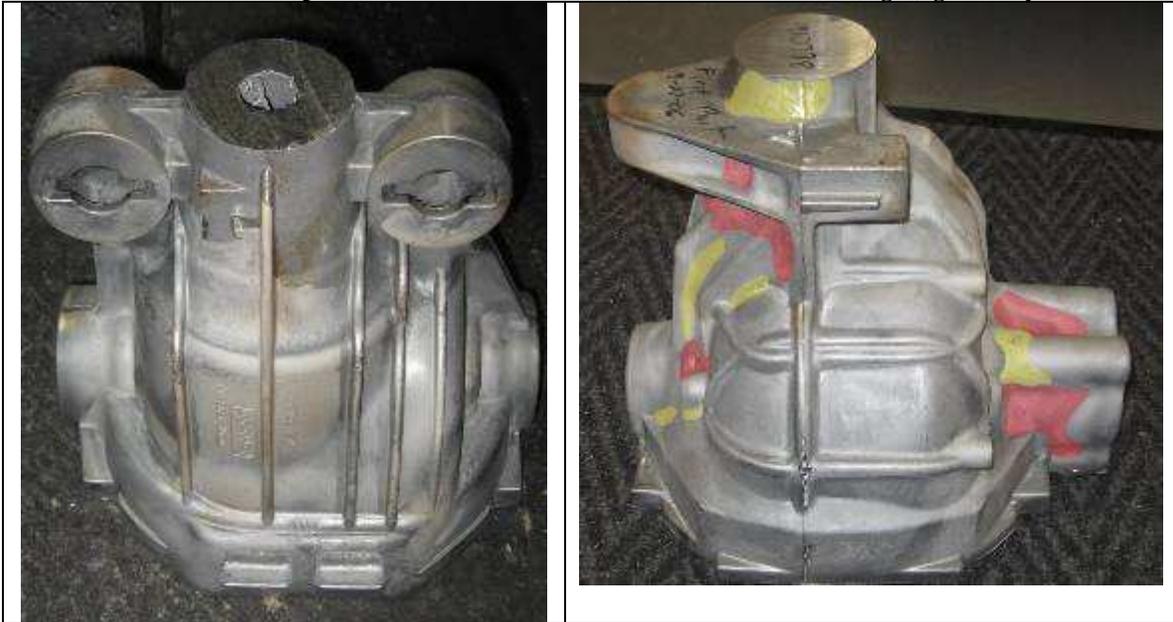


### ***V.V U251 Carrier: Strontium Modification Investigation***

Another part at the Pierceton plant that suffers from severe soldering is the U251 Carrier. Although no detailed data was collected due to confusion over what trials were going to be performed, using Sr-modified ADC12 to cast the U251 Carrier was tested. The U251 is a similar part to the U222 Carrier, but with some minor differences (see Table 28).

One of the most important of the difference in geometry is the lack of a large thick section (relatively) at the base of the casting. These thick sections promote a hot spot in the U222 Carrier and lead to the eventual formation of shrinkage porosity in those locations. The fact that the U251 does not have sections as thick as the U222 makes it less vulnerable to the problems that can occur with a Sr addition, and also make it slightly less susceptible to soldering, although soldering is a significant issue.

**Table 28: Comparison of U251 Carrier (left) and U222 Carrier (right) geometry.**



Two quads (168 pieces) were cast with a Strontium level between 0.015 and 0.025 wt% and machined (unfortunately, the specific Strontium levels were discarded when the trial was originally cancelled and it did not appear that any data was going to be useful from the trials). This is not a long enough trial to draw any firm conclusions on how much reduction the Sr-level led to, but anecdotally it was successful, similar to the U222 results.

Of these 168 pieces, none failed post-machining inspection for any reason. Some pieces were x-rayed and revealed that the use of Strontium may even reduce the amount of porosity in the part, possibly by shifting it to a location in the casting which is trimmed or machined away.

It seems that the use of Strontium to modify the ADC12 for casting the U251 Carrier is, at the least, not harmful to the process, and most likely is beneficial at reducing the downtime associated with soldering. In order to justify the use of Strontium in the process, however, the cost of the Strontium addition must be justified. No data has been collected on this part on how much downtime Strontium prevents, but it should be very close to the case of the U222 Carrier, where downtime was reduced by 26.8%. If this assumption is acceptable, a cost-benefit analysis next needs to be performed to justify the use of Strontium.

## ***V.VI Conclusions***

- Using Strontium to modify ADC12 is an effective way of reducing die soldering, with the results in this work showing that die soldering is reduced by >25% with an addition of Strontium. In the case of the U222 Carrier, soldering was reduced by 26.8%.
- Due to Strontium's effects on the nucleation of the final liquid to freeze, the location of shrinkage porosity in a part can be moved and dispersed. This potentially can result in an increase in scrap rate. In the case of the U222 Carrier, scrap rate was increased by 18.1%. Parts which have a significant amount of shrinkage porosity should be carefully studied when deciding when a Strontium addition may be used.
- Using Strontium modification on the U222 Carrier program is not justified, due to the large scrap increases, unless a significant process study is conducted to determine the necessary process parameters to successfully cast the part with a Strontium addition. The same scrap increase is not seen in the U251 Carrier program. If the additional cost of the Strontium for modification does not outweigh the benefits of reduced soldering, then Sr-modified ADC12 should be used in the U251 program.

## **VI. Conclusions & Future Work Suggestions**

The thesis has resulted in interesting results in three somewhat divergent areas: surface metrology as a means for quantifying die soldering, prediction of die soldering using MAGMASoft and the effects of strontium modification. In each of the sections above, the results have been presented along with the important conclusions that should be noted from each section.

It is now important to note how the results of the work above can complement each other in order to increase the ability to reduce the effects of die soldering further than the results from each section can be used independently. Some exciting potential future research has also been suggested by the work, and these ideas will be discussed as well.

The surface metrology work is interesting as a means to expedite future die soldering tests. The studies used in the strontium modification trials required over 2000 shots to be made in order to collect enough data for conclusions to be drawn. If further studies are pursued that can validate the reproducibility and accuracy of the surface metrology studies, this number of shots can be greatly reduced. For example, perhaps 3 runs of 200 shots from cleaning to cleaning could be made with both unmodified and modified alloy. The surfaces of the parts before and after the runs could be evaluated and used as a way to measure soldering. This could cut down the time required for the trials, a very tight commodity, significantly.

The ability to predict soldering beforehand could have enabled the benefits of strontium modification in the U222 Carrier case to be realized. It is thought that the process has been optimized so that the porosity in the part, using unmodified ADC12 to not be an issue. However, since the strontium modification affects the last liquid solidifying, the location of these pores changes and these small process changes are no longer relevant. Had the program initially begun using modified metal, the optimal process may have been able to be determined during an initial period where the process was optimized using modified metal.

It is also important to note the seemingly random distribution of the porosity in some of the parts x-rayed and sectioned. It may be that any parts with large pores will behave unpredictably when Strontium is added, and parts like these should be avoided when identifying candidates for Strontium modification.

## ***VI.I Future Work Suggestions***

- In order to verify the results of the prediction work, the results should be reviewed extensively in other casting systems at Contech, especially those that utilize ADC12 initially, and then progressing to other alloys and/or processes to see how well the results translate. Not until this work is completed can the results found here be verified and confidently used in new programs.
- An economic study of the U251 Carrier should be carried out to evaluate whether or not a Strontium modification is justified. The U251 Carrier is a good candidate for Strontium modification because it experiences a good deal of soldering and initial studies showed that no problems with porosity as seen in the case of the U222 Carrier.
- If future studies are planned to test the response of the die soldering problem to process changes, the surface metrology work can be extended in order to refine the method, due to its potential to quickly generate results.
- With the ability to predict soldering potentially gained, the question now becomes what to do in the case that it is known that soldering will occur. Strontium modification can be first evaluated, with the potential porosity problems now better understood. Another potential solution is the increased use of inserts which can be more quickly replaced in the case of soldering damage. Additionally, the results of the aluminizing trials which will be completed beyond the conclusion of my work should be analyzed closely, as the process has the potential to mitigate soldering significantly (other die coatings may be explored as well). Finally, alternate die materials can be explored, either materials which do not react with aluminum (e.g.: ceramic materials, Tungsten) or tool steels with high heat conduction coefficients.

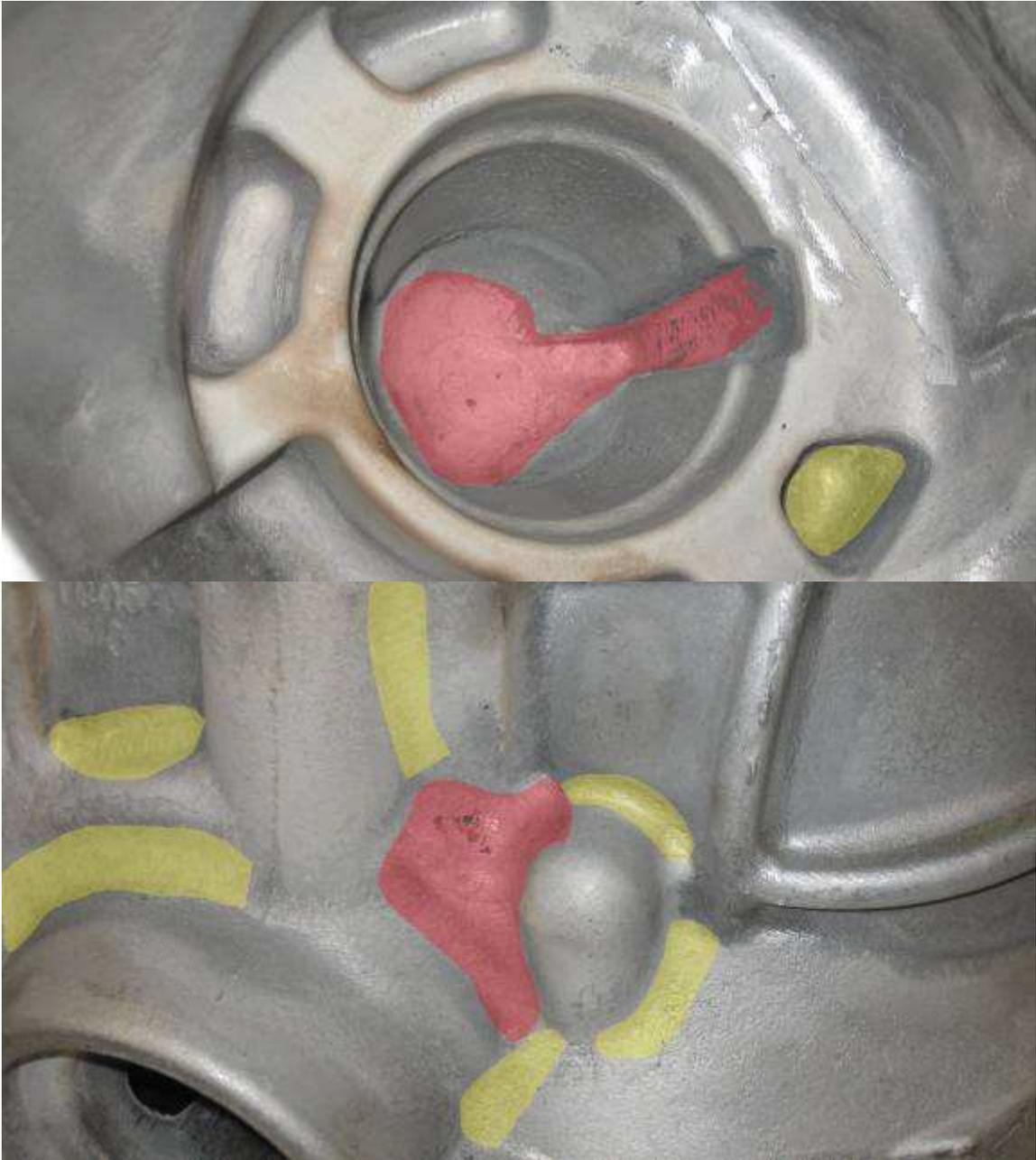
## Appendix 1: Qualitative Solder Rating Images





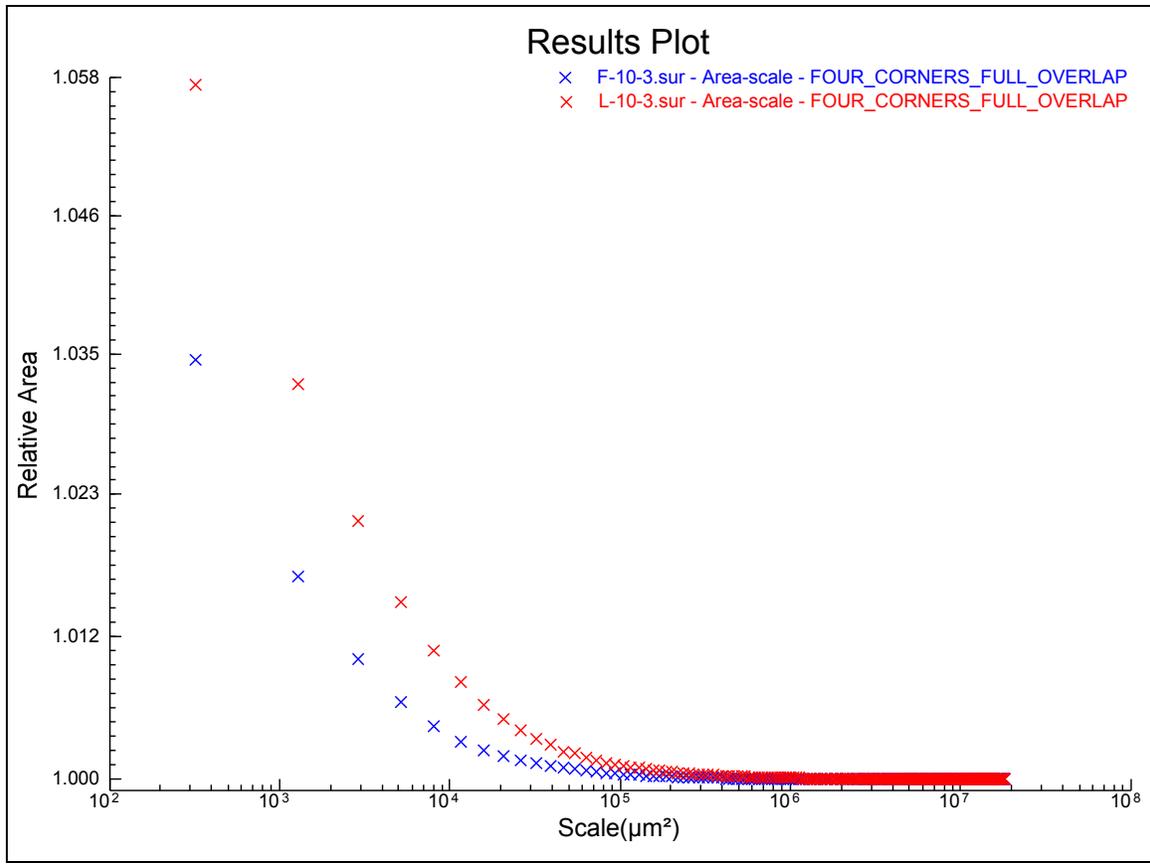






## **Appendix 2: Relative Area Scale Analysis**

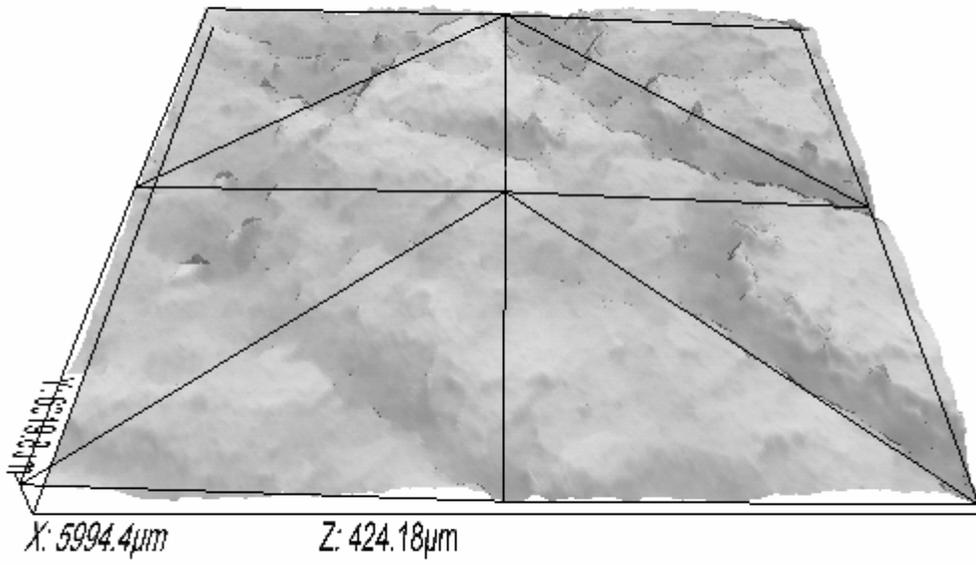
Relative Area scale analysis is a surface roughness analytical technique, where the surface is represented by tiling with triangles, the size of the triangles representing the scale of observation. The ratio of the measured area to the nominal area of the measurement at each scale is the relative area. The result is a plot similar to the following:



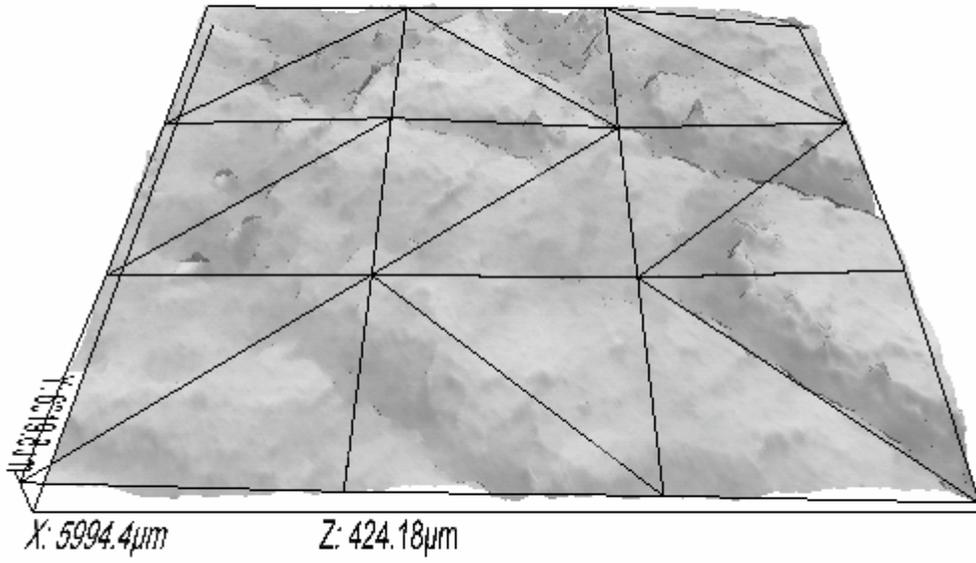
As is apparent, as the size of the triangles grows smaller, the relative area becomes larger, as the triangles can fit and represent an increasing number of the smaller features of the surface.

As it relates to this project in particular, it is seen that at location 10, the relative area increases over the series of shots as die soldering progresses. This indicates that the surface is becoming rougher as soldering increases.

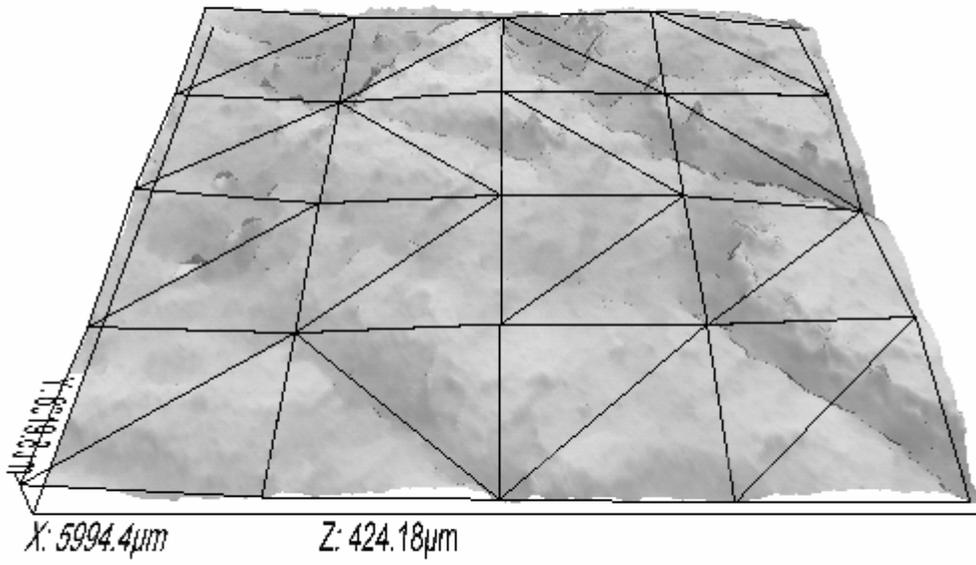
The following images are a series of triangular tilings of the surface, as the size of the triangles decreases and the relative area increases.



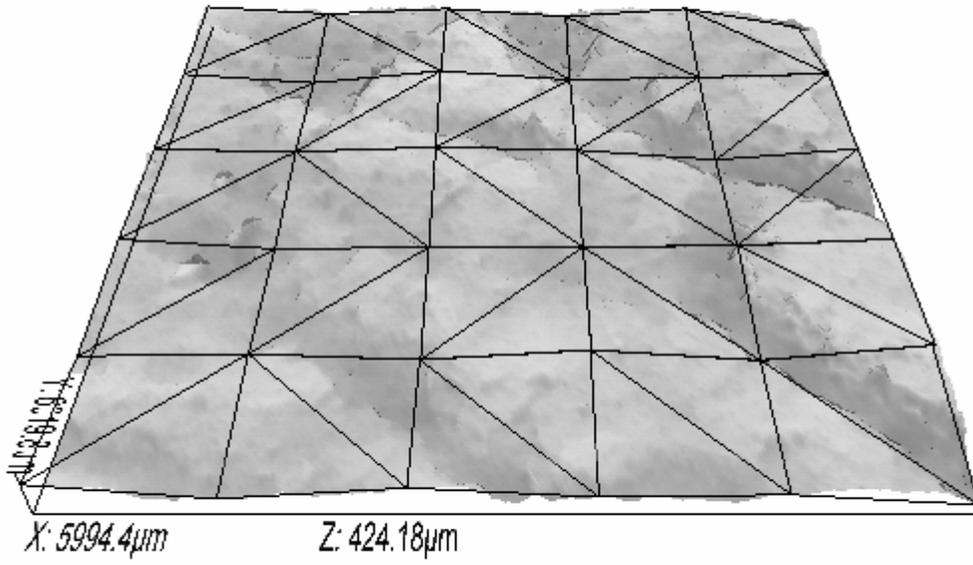
Scale= 4415797.4 µm<sup>2</sup>  
Tiles=8  
Relative Area=1.00049



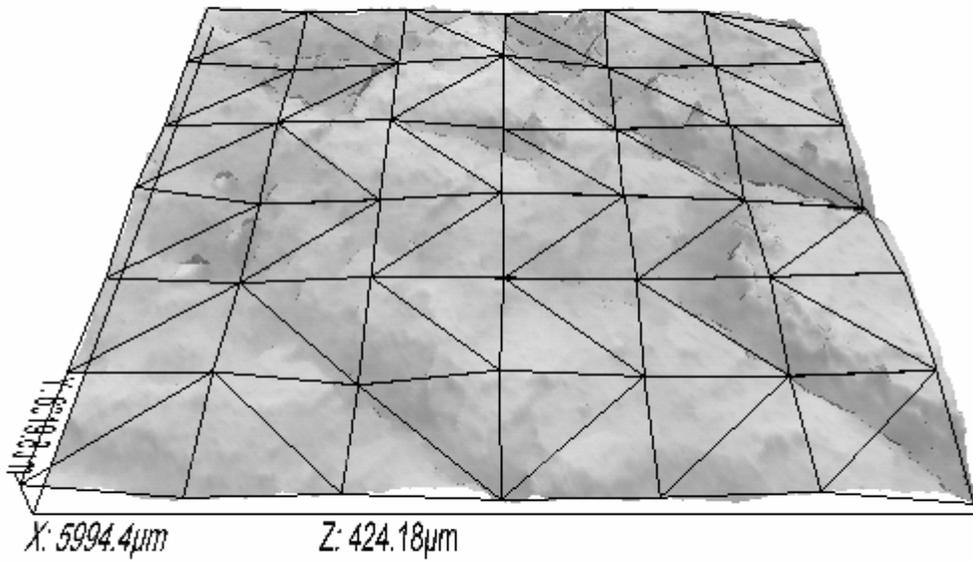
Scale=1962576.6 µm<sup>2</sup>  
Tiles=18  
Relative Area=1.001033



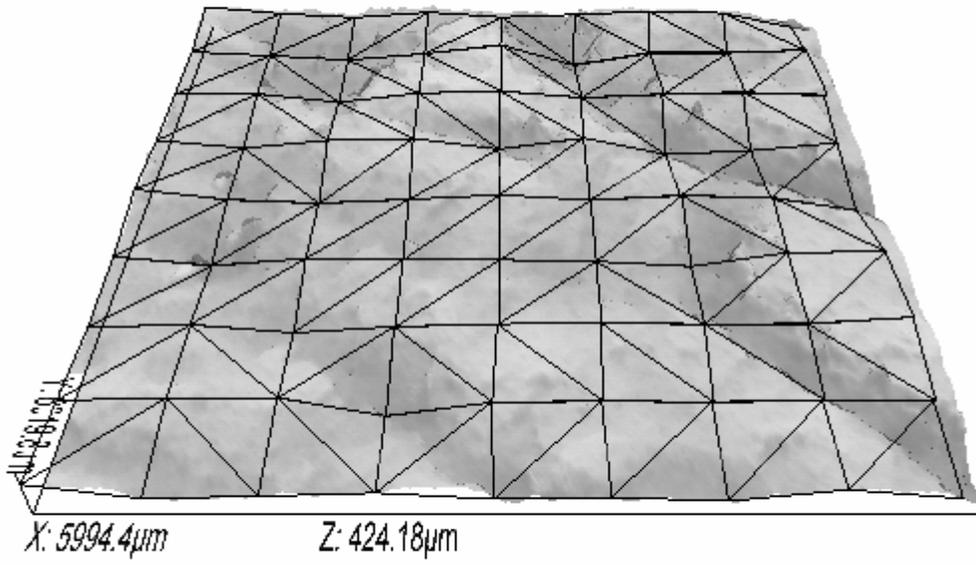
Scale=1085159.0  $\mu\text{m}^2$   
Tiles=32  
Relative Area=1.002586



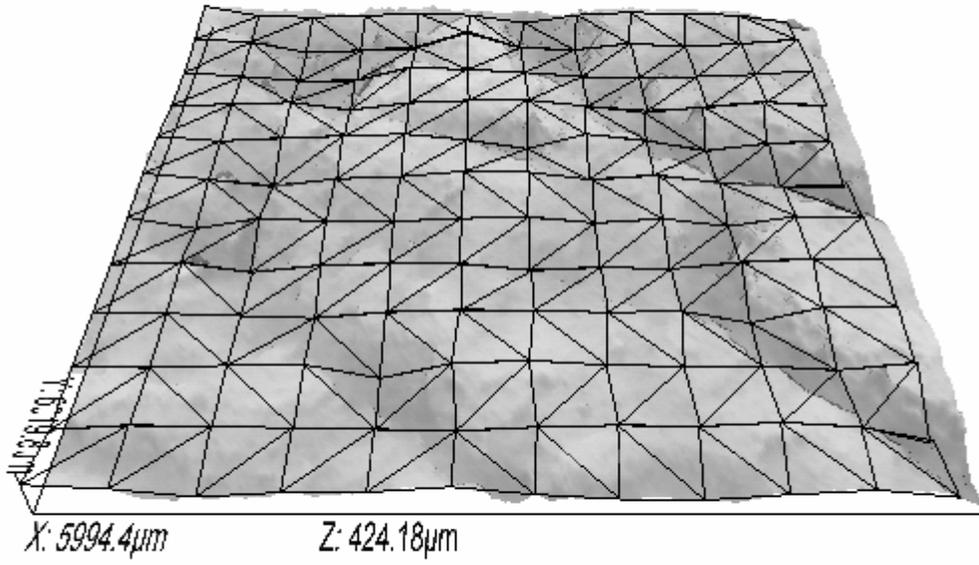
Scale=712579.9  $\mu\text{m}^2$   
Tile=50  
Relative Area=1.004085



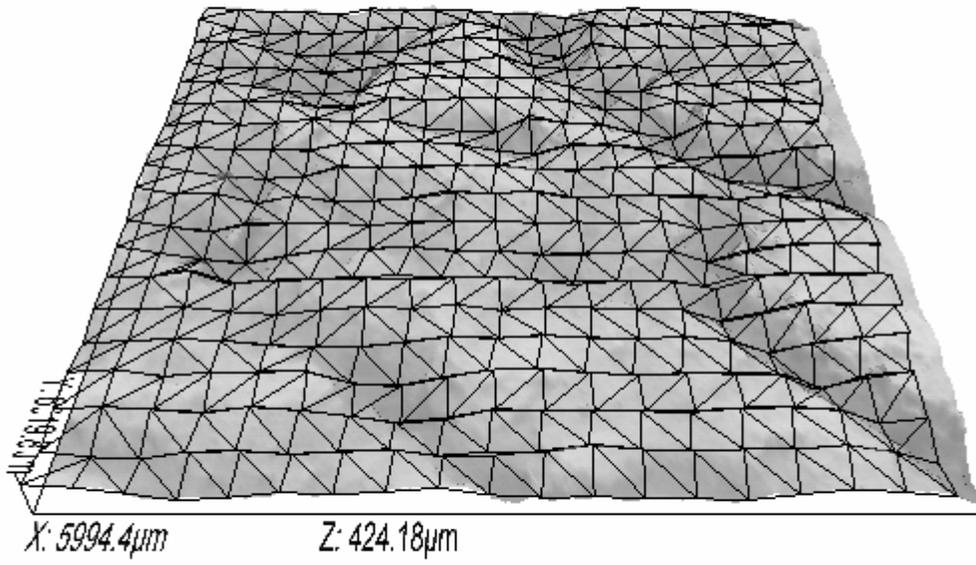
Scale=490644.16  $\mu\text{m}^2$   
Tiles=72  
Relative Area=1.006279



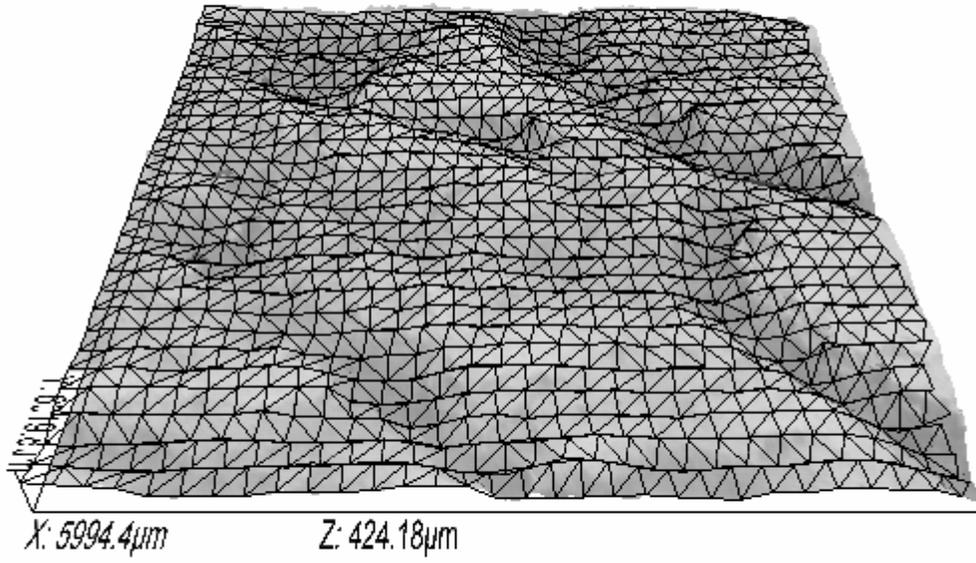
Scale=271289.77  $\mu\text{m}^2$   
Tiles=128  
Relative Area=1.010911



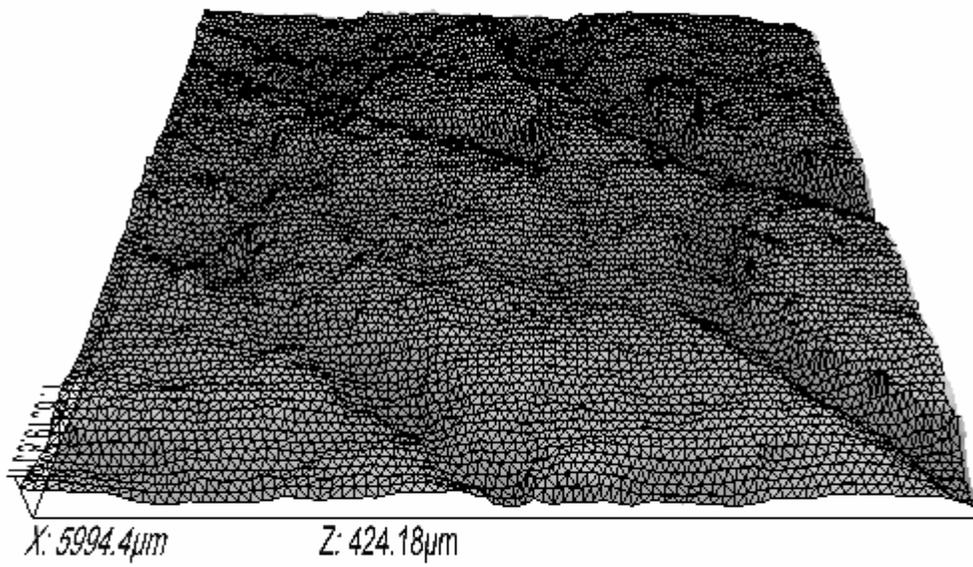
Scale= 142257.77  $\mu\text{m}^2$   
Tiles=242  
Relative Area= 1.016193



Scale=54516.048  $\mu\text{m}^2$   
Tiles=648  
Relative Area=1.028712



Scale=20645.119  $\mu\text{m}^2$   
Tiles=1747  
Relative Area=1.043388



Scale=2903.2199  $\mu\text{m}^2$   
Tiles=13199  
Relative Area=1.084763

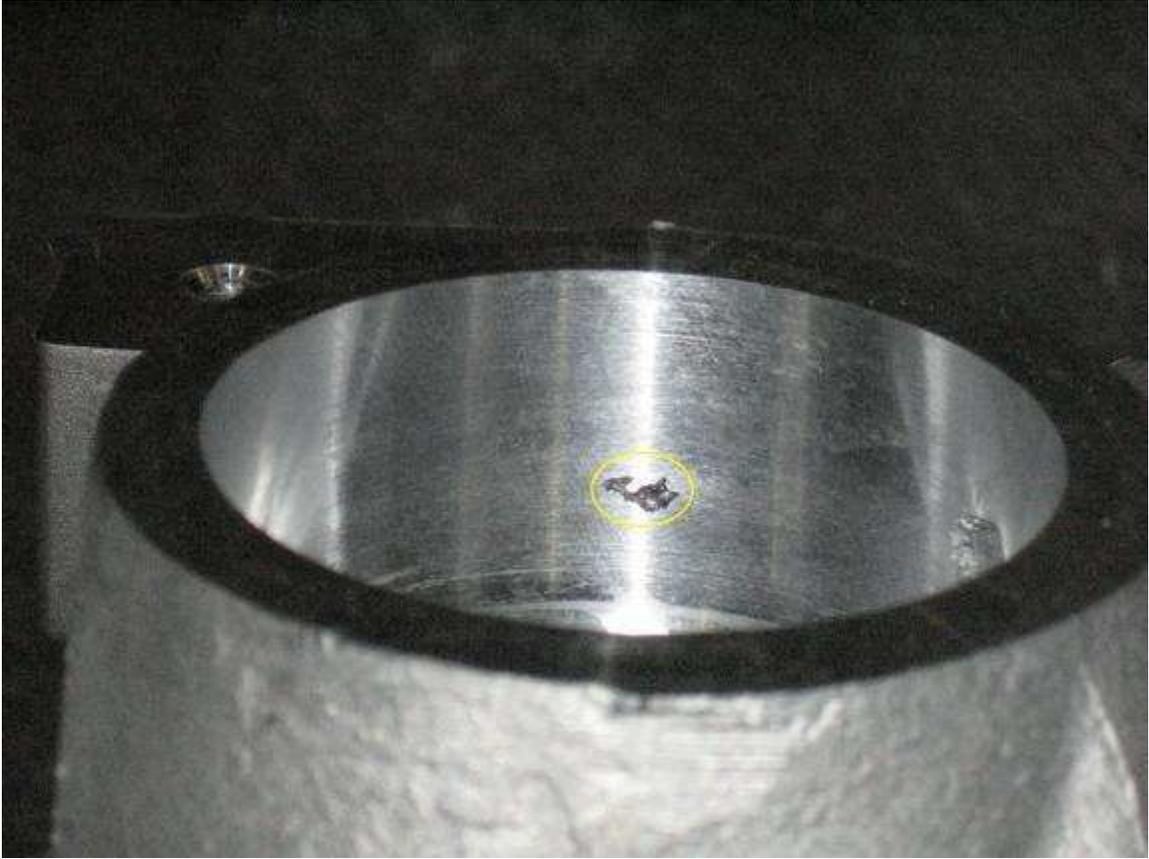
### Appendix 3: Porosity Severity Rating Scale Examples



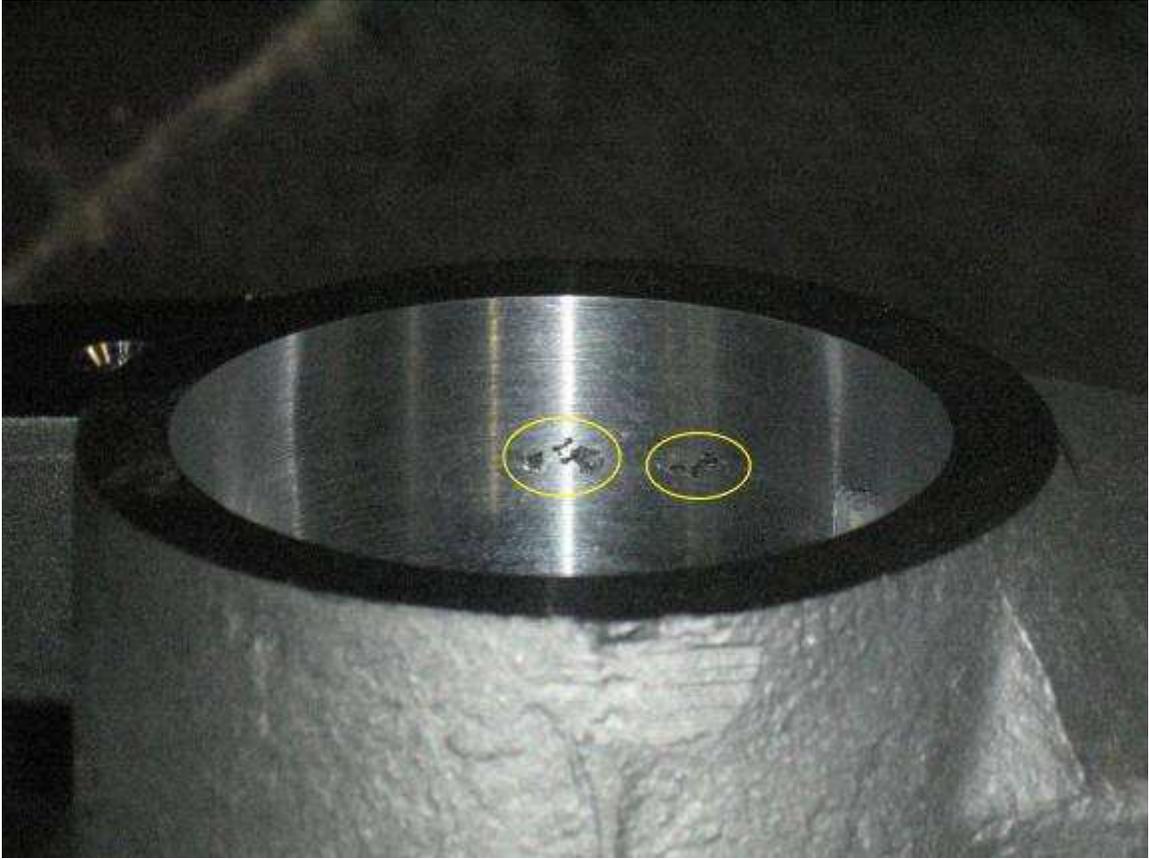
Porosity Rating =1



**Porosity Rating=2**



**Porosity Rating=3**



**Porosity Rating=4**