



Heat Treatment Optimization for Cold-Sprayed Aluminum 6061

Major Qualifying Project
submitted to the faculty of
Worcester Polytechnic Institute
in partial fulfillment of the
requirements for the Degree of Bachelor of Science

Submitted: *April 26, 2012*

Sponsoring Agency: United States Army Research Lab (ARL)

Submitted to:

Dr. Richard D. Sisson, Jr., Project Advisor
Danielle Belsito, Ph.D. Candidate

Respectfully Submitted by:

Caitlin Kelley
Lauren Ketschke
Baillie McNally

Abstract

Cold-sprayed aluminum 6061 alloy test samples were heat treated at several solutionizing temperatures, aging temperatures, and aging times to determine the effect of heat treatment on microstructure and hardness. The cold-spray process sprays 10-100 micrometers particles of the aluminum alloy, at velocities of 400-1200 m/s onto a substrate to form a coating of the highly cold worked aluminum alloy. The as- cold sprayed samples were solutionized at 530 °C, and aged at 200 °C for 30 minutes to 4 hours. In addition, as- cold sprayed test samples were heat treated at temperatures from 200 to 400 °C for times ranging from 15 minutes to 4 hours. Vickers and Knoop microhardness values as well as scanning electron microscope images were compared to determine the effects of heat treatments on the hardness and microstructure. Overall the heat treatments caused a decrease in hardness values in all of the samples, with the largest decrease being 47% from the as-sprayed control sample. The highest hardness value and largest growth of precipitates was recorded after 1 hour of heat treatment at 200°C with a growth in precipitates of approximately 30%.

Table of Contents

Abstract	2
Table of Contents	3
Table of Figures	5
Table of Tables	6
1. Introduction.....	7
2. Background.....	8
2.1 Cold Spray.....	8
2.1.1 Characteristics	8
2.1.2 Powder.....	10
2.1.3 Advantages	10
2.2 Heat Treatment	10
2.2.1 Annealing	11
2.2.2 Solution Heat Treatment.....	11
2.2.3 Quenching.....	11
3. Methodology	13
3.1 Material Selection	13
3.2 Sample Preparation	13
3.3 Heat Treating.....	15
3.3.1 The 100 Series	15
3.3.2 The 200 Series	16
3.3.3 The 300 Series	16
3.3.4 The 400 Series	17
3.4 Hardness Testing	17
3.5 Statistical Analysis	18
3.6 Microstructure Analysis	19
4. Results.....	20
4.1 100 Series	20
4.2 200 Series	21
4.3 300 Series	21
4.4 400 Series	22
4.5 Microstructure Analysis	23

5. Conclusions.....	25
5.1 Future Work	25
References.....	27
Appendix A – 100 Series Data.....	28
Appendix B – 200 Series Data.....	30
Appendix C – 300 Series Data.....	32
Appendix D.....	34

Table of Figures

Figure 1: Cold Spray Process.....	8
Figure 2: Particle Deforming on Impact	9
Figure 3: Atomization	10
Figure 4: Original Cold Sprayed Material	14
Figure 5: Sample Preparation.....	14
Figure 6: Faces of Interest.....	18
Figure 7: 100 Series, Face A - Knoop Hardness.....	20
Figure 8: 200 Series, Face A - Knoop Hardness.....	21
Figure 9: 300 Series, Face A - Knoop Hardness.....	22
Figure 10: 400 Series, Face A - Knoop Hardness.....	22
Figure 11: As-sprayed control sample (left) and Sample 100-4 (right).....	23
Figure 12: As-sprayed control sample (left) and Sample 200-3 (right).....	23
Figure 13: Sample 300-1 (left) and Sample 300-5 (right)	24
Figure 14: Sample 400-1 (left) and Sample 400-5 (right)	24

Table of Tables

Table 1: Aluminum 6061 Specifications	13
Table 2: Sample Series Descriptions	15
Table 3: 100 Series Times.....	16
Table 4: 200 Series Times.....	16
Table 5: 300 Series Times.....	17
Table 6: 400 Series Times.....	17
Table 7: Samples Selected for Further Examination	19

1. Introduction

The Army Research Lab (ARL) is always looking to improve the survivability of war fighters through refining and developing technologies that can make equipment and vehicles more efficient and safe. Military vehicles are designed to help protect soldiers by being mobile, fuel efficient, and protective. Accordingly, the materials they are built from must have low density, high strength, and high toughness. However, these vehicles occasionally need reinforcement or repair. ARL has been investigating the process of cold spraying aluminum alloys to serve this purpose. ARL is exploring various uses for aluminum alloy cold-spray technology including corrosion resistance, dimensional restoration and repair, field repair, and electromagnetic interference shielding of components, which keeps electric waves from entering or leaving a location (Champagne, Helfrich, Trexler, & Gabriel, 2012) (Army, 2012). However, cold spray is still a relatively new technology and little research has been done to observe the results of different treatments and processes performed to a cold sprayed material. This project looks to determine the effects of heat treatment on the hardness and microstructure of cold spray aluminum 6061 in order to expand the Army Research Lab's understanding of the properties and effects of cold spray.

2. Background

2.1 Cold Spray

The cold spray process forcefully combines powder particles to create a coating or a piece of bulk material. This is accomplished by injecting the powder particles into a high velocity stream of inert gas, such as air, helium, or nitrogen. It is forced through a de Laval nozzle, accelerating to supersonic velocities where it is ejected onto a substrate causing plastic deformation on impact, ultimately forming bonds between the solid particles, creating a solid layer of material. These layers can be made as thick or thin as the application desires. A diagram of the process can be seen below in Figure 1 (Army, 2012).

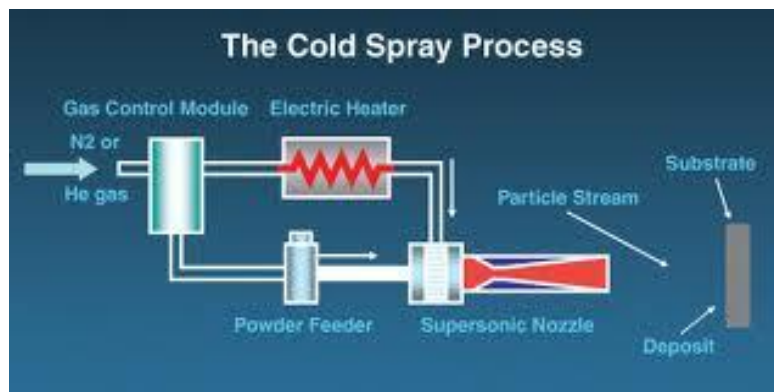


Figure 1: Cold Spray Process

The speeds upon contact can be greater than 1000m/s but the temperature of the stream is always below the recrystallization temperature of the metal. This preserves the integrity of the material and also gives the process the name “cold spray” (Champagne et. al, 2010).

2.1.1 Characteristics

Cold spray results in high levels of work hardening because of the great amount of force created when the particles bond together. During impact and bonding, the particles experience

severe plastic deformation, flattening them as you can see in Figure 2 below (Champagne et. al, 2010).

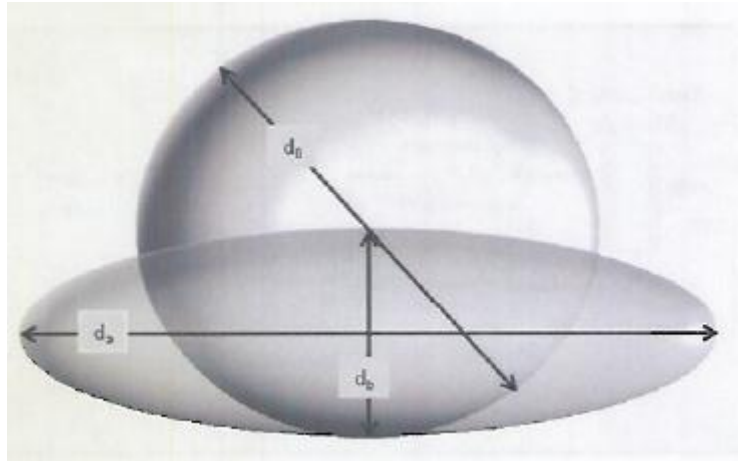


Figure 2: Particle Deforming on Impact

The great amount of strain deformation the particle undergoes can be represented by the equation below.

$$\sigma = [A + B\varepsilon^n][1 + C \ln(\frac{\dot{\varepsilon}}{\dot{\varepsilon}_0})][1 - (T^*)^m]$$

σ	Stress
ε	Strain
$\dot{\varepsilon}$	Strain rate
$\dot{\varepsilon}_0$	Normalization factor (-1 s^{-1})
A, B, C, n, m	Empirical constants
T^*	$(T - T_{\text{room}}) / (T_{\text{melt}} - T_{\text{room}})$ Where T is impinging temperature

With the powder being flat and condensed, cold spray has a low porosity and high density, also preventing oxidation. Work hardening makes the cold spray have many dislocations and residual stresses, causing high hardness and low ductility. The overall hardness level from aluminum can increase by about 48% because of the increase in dislocation densities that are associated with work hardening (Champagne et. al, 2010). The increased hardness level is one of the main appeals of cold spray and can be used for applications such as repairing cracks in tanks or providing support to the panels of an Apache helicopter.

2.1.2 Powder

One of the most popular ways to create the metallic powder used for cold spray is through a process called atomization. The desired metal is melted down and poured into a chamber. As it pours through, a stream of high speed water or gas dissolves the stream into tiny droplets. When the droplets fall, they solidify into particles. The powder is then collected from the bottom of the chamber and dried. Figure 3 below shows the atomization process (Powder Preparation, 2009).

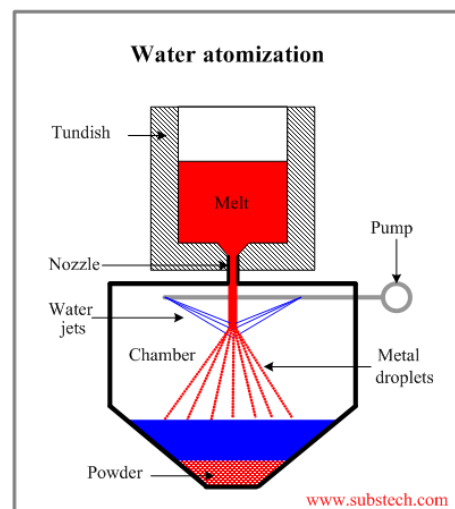


Figure 3: Atomization

2.1.3 Advantages

Cold spray has many advantages over other coating systems that are of use to the ARL. Operating at low temperatures means there is no requirement for high temperature and combustible gasses. It also has the ability of being a portable system, meaning it can be brought directly into the field and repairs can be made on site (Army, 2012) (International, 1998).

2.2 Heat Treatment

Heat treatment is defined as “any of the heating and cooling operations that are performed for the purpose of changing the mechanical properties, the metallurgical structure, or

the residual stress state of the metal product.” For aluminum, this refers mostly to increasing strength and hardness (International, 1998)

2.2.1 Annealing

Annealing occurs when the aluminum alloy is heated to a certain temperature in order to reverse the effects of work hardening. The treatment temperature is set above the recrystallization temperature which means that the grains will begin to grow again. This temperature is a direct result of the amount of strain and residual stress in the material prior to annealing (International, 1998).

2.2.2 Solution Heat Treatment

Solutionizing aluminum allows for the maximum amount of solute to form in the solid metal, greatly increasing the amount of precipitates. This is accomplished by heating the aluminum alloy to just below the eutectic melting temperature and leaving it for a predetermined amount of time. Keeping the temperature in check is vital to the outcome of the solutionizing process. Overheating will raise the temperature above the eutectic point causing localized melting and a reduction in material property values. Under heating may not let all of the solutes grow into the solid solution, leaving fewer precipitates for future treatments. Overall, time is not as important as temperature (Totten & Mackenzie, 2003). There are no exact time and temperature combinations for cold spray aluminum 6061 but various sources suggest that regular aluminum 6061 be solutionized for one hour at 530°C (International, 1998) (Totten & Mackenzie, 2003).

2.2.3 Quenching

Quenching is an extremely important part of the heat treatment process. The process involves cooling the metal after heat treatment in various mediums and at various speeds. There

are both rapid and slow quenches, and different quenching fluids include water, oils, and air. The goal of quenching is to stop precipitation growth and preserve the metal at its current state immediately after heat treatment. By cooling off the material, the high temperatures cannot continue to have an effect on the material properties past the designated heating time (Totten & Mackenzie, 2003).

3. Methodology

3.1 Material Selection

The material used in this project was bulk cold sprayed Aluminum 6061, which was chosen because it is the aluminum alloy of interest to the sponsor, the Army Research Lab (ARL). ARL obtains the Aluminum 6061 powder from Valimet who create the powder through atomization. The specifications of the cold spray 6061 were obtained from ARL and can be seen below in Table 1.

Table 1: Aluminum 6061 Specifications

Element	Percent Composition (%)
Silicon	0.40-0.80
Iron	0.70
Copper	0.15-0.40
Manganese	0.15
Magnesium	0.8-1.2
Chromium	0.04-0.35
Zinc	0.25
Titanium	0.15
Other	0.15
Aluminum	remainder

3.2 Sample Preparation

The sample received from the Army Research Lab was originally one bulk piece, as shown in Figure 4 below. The cold spray layer was applied to the aluminum alloy plate substrate in the x-y direction, and was sprayed to form a bulk sample.

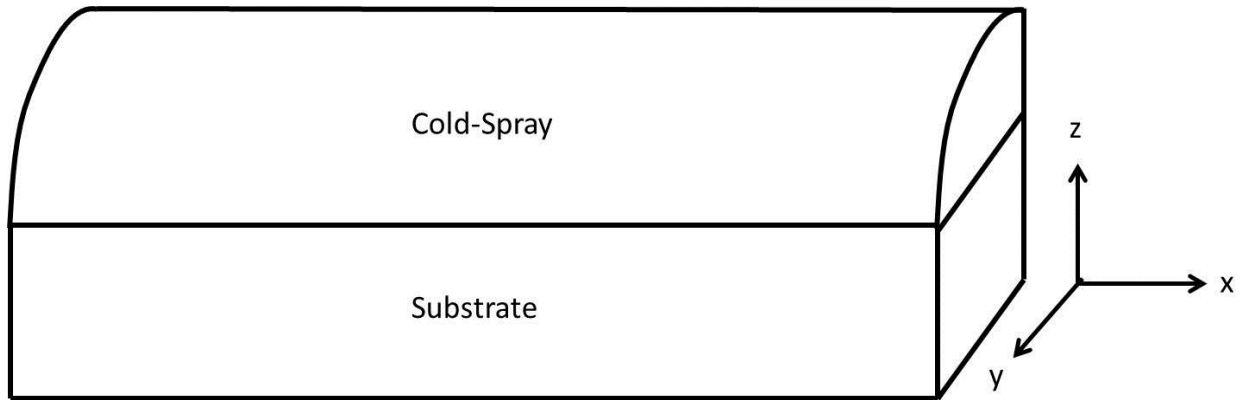


Figure 4: Original Cold Sprayed Material

ARL cut the bulk sample into six equal pieces, and one of these was reserved as a backup. On each sample, the substrate was cut off, and the remaining bulk cold spray was cut into five equal pieces as seen in Figure 5 where the dotted lines indicate where cuts were made. Cutting was done on a Mark V Series 600 cutter with a 605 blade for medium hardness, non-ferrous materials. Each set of five pieces formed a series that was each subjected to a different heat treatment process.

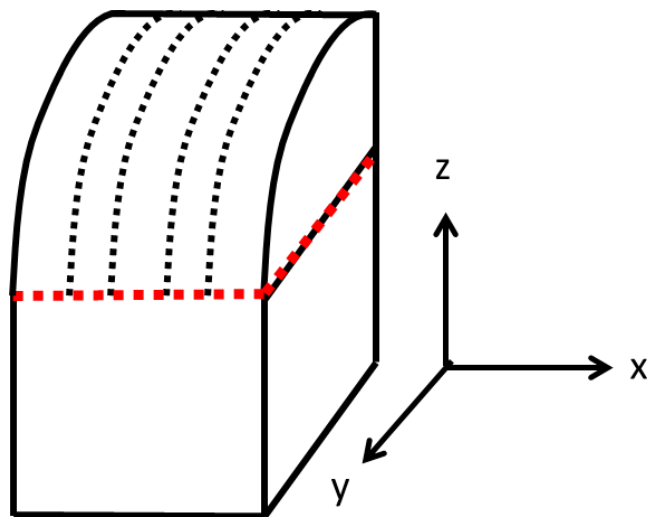


Figure 5: Sample Preparation

3.3 Heat Treating

Each series of bulk cold sprayed aluminum 6061 was put through a different heat treatment process at varying times and temperatures. The series and their associated processes are outlined in Table 2 below, and the details of each process follow.

Table 2: Sample Series Descriptions

Series	Description
Control	No heat treatment
100	Solutionized at 530°C, quenched in ice water, and aged at 200°C for varying times
200	Heat treated at 200°C for varying times, and quenched in ice water
300	Heat treated at 300°C for varying times, and quenched in ice water
400	Heat treated at 400°C for varying times, and quenched in ice water

3.3.1 The 100 Series

Each of the five samples in the 100-series was solutionized for one hour at 530°C in a Thermo Scientific Thermolyne furnace. After solutionizing, the samples were quenched in ice water for five minutes in order to prevent any further change in microstructure. Each sample was then placed back in the furnace at 200°C for aging. One sample was taken out of the furnace at 15 minutes, 30 minutes, 1 hour, 2 hours, and 4 hours during the aging process in order to observe the effect that aging time had on hardness. When removed from the furnace after aging, the sample was quenched in ice water for five minutes. Each time a sample was removed from the furnace, the door was opened for the smallest duration possible to prevent a drop in temperature.

Table 3: 100 Series Times

Sample Name	Aging Time
100-1	15 minutes
100-2	30 minutes
100-3	1 hour
100-4	2 hours
100-5	4 hours

3.3.2 The 200 Series

The five samples of the 200-series were placed in a 200°C furnace, removed at the intervals below in Table 4, and quenched in ice water for five minutes. Each time a sample was removed from the furnace, the door was opened for the smallest duration possible to prevent a drop in temperature.

Table 4: 200 Series Times

Sample Name	Heat Treating Time
200-1	15 minutes
200-2	30 minutes
200-3	1 hour
200-4	2 hours
200-5	4 hours

3.3.3 The 300 Series

The five samples of the 300-series were placed in a 300°C furnace, removed at the intervals below in Table 5, and quenched in ice water for five minutes. Each time a sample was removed from the furnace, the door was opened for the smallest duration possible to prevent a drop in temperature.

Table 5: 300 Series Times

Sample Name	Heat Treating Time
300-1	15 minutes
300-2	30 minutes
300-3	1 hour
300-4	2 hours
300-5	4 hours

3.3.4 The 400 Series

The five samples of the 400-series were placed in a 400°C furnace, removed at the intervals below in Table 6, and quenched in ice water for five minutes. Each time a sample was removed from the furnace, the door was opened for the smallest duration possible to prevent a drop in temperature.

Table 6: 400 Series Times

Sample Name	Heat Treating Time
400-1	15 minutes
400-2	30 minutes
400-3	1 hour
400-4	2 hours
400-5	4 hours

3.4 Hardness Testing

Immediately following the heat treatment of each series, the samples were cut so that various faces could be analyzed. The faces of interest were the x-y direction (the direction of spray) and the y-z direction. The x-y direction was named the A face, and the y-z direction was named the B face. Figure 6 below shows where the sample was cut and the faces of interest.

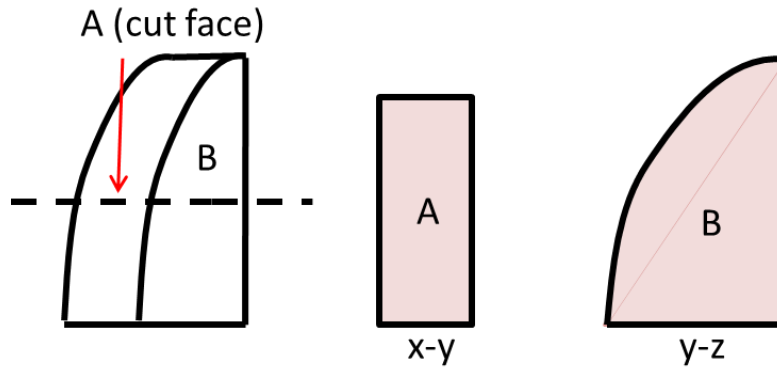


Figure 6: Faces of Interest

Both faces were mounted using a BEUHLER SimpliMet 3000. Once mounted, each face was tested for Knoop and Vickers microhardness values. Knoop microhardness was taken on a BUEHLER 1600-3000 using 200 grams of force, whereas Vickers microhardness was obtained by using a Shimadzu HMV-2000 using 100 grams of force for ten seconds. On each machine, six data points were recorded for each face, and the average of the six values was calculated. The hardness values obtained were plotted against the heat treating time for each sample series.

3.5 Statistical Analysis

A Dixon Q-test statistical analysis was performed on the data to determine if any outliers were present within a 95% confidence level. The six data points for each hardness test were ordered from smallest to largest and denoted by $x_1, x_2, \dots, x_{n-1}, x_n$ respectively. The upper and lower bounds for the Q value were obtained using the following equations. Any Q values greater than 0.625 indicated that the data set contained an outlier and the outlier was removed from the microhardness values.

$$Q_{lower} = r_{10} = \frac{x_2 - x_1}{x_n - x_1}$$

$$Q_{upper} = r_{10} = \frac{x_n - x_{n-1}}{x_n - x_1}$$

3.6 Microstructure Analysis

In order to understand the change in the microstructure that occurred during the various heat treating processes, the team selected samples from each series that displayed a significant change in hardness values. Table 7 below summarizes the samples that were selected from each series.

Table 7: Samples Selected for Further Examination

Series	Samples Selected
Control	Control A, Control B
100	100-4A
200	200-1A, 200-5A
300	300-1A, 300-5A
400	400-4A

Each of the selected samples was polished using 1 μ m, 0.5 μ m, and 0.3 μ m aluminum silicate solution. Using the Nikon Epiphot fitted with a camera; images of each sample were recorded at 100x magnification. Scanning electron microscope images were taken of selected samples to view the microstructure and growth of precipitates of the samples. A JEOL JSM-7000F scanning electron microscope was used to obtain images of the samples at a magnification of 1,000X with an Oxford Instruments INCAx-act camera.

4. Results

Knoop and Vickers microhardness tests results revealed that the heat treatments significantly decreased the hardness values of the samples. There was no discernible difference between the hardness values of the different faces of the samples, or a difference between Knoop and Vickers microhardness values. Knoop microhardness values on face A of the samples will be discussed because there were no outlying data points, while Vickers microhardness results had four outlying data points. The remaining hardness results can be found in Appendix A-D.

4.1 100 Series

The 100 series, which was solutionized and aged at various times, showed a decrease in hardness from the as-sprayed control sample. However, it was the only series to show a typical hardness curve where the hardness peaked after 2 hours of aging at 96.25 H_K, shown in Figure 7.

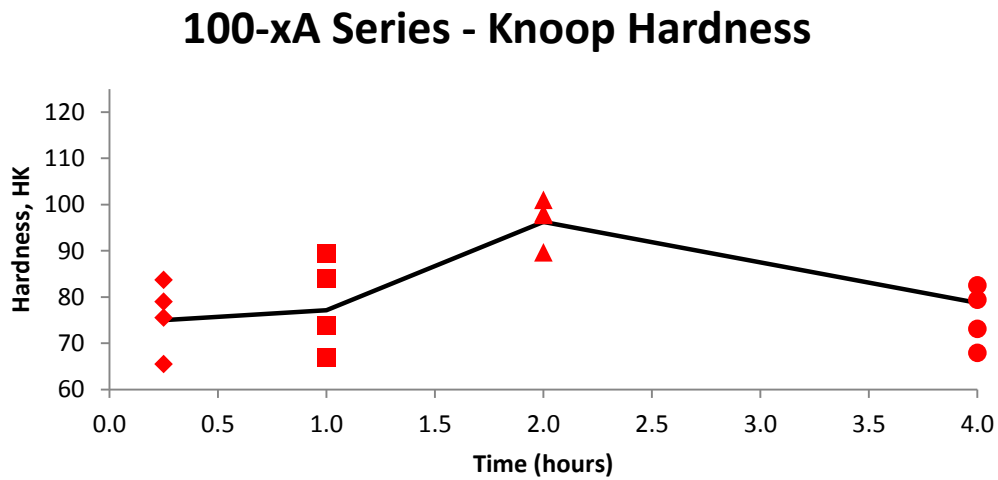


Figure 7: 100 Series, Face A - Knoop Hardness

4.2 200 Series

The 200 series, which was aged at 200°C for various times, showed the highest hardness values of all the samples. The curve does not resemble a typical hardness curve, however it does peak at a hardness value of 117 H_K after 1 hour of heat treatment, shown in Figure 8.

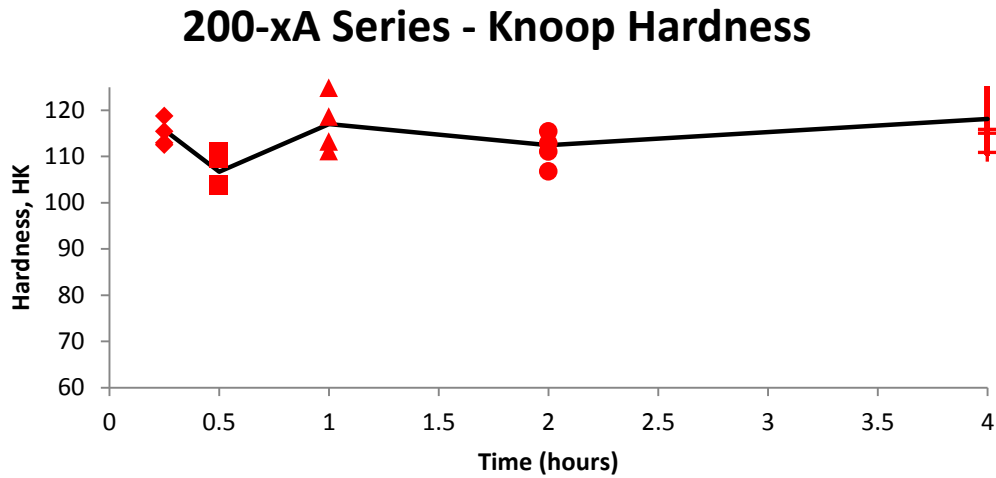


Figure 8: 200 Series, Face A - Knoop Hardness

4.3 300 Series

The hardness values of the 300 series, which was heat-treated at 300°C for various times, decreased throughout the heat treatment. After 4 hours of heat treatment, the lowest hardness value was recorded at 80 H_K, shown in Figure 9.

300-xA Series - Knoop Hardness

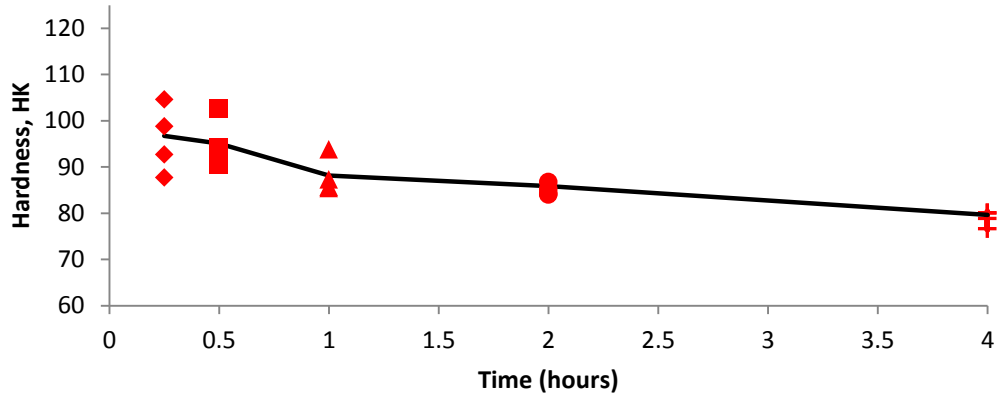


Figure 9: 300 Series, Face A - Knoop Hardness

4.4 400 Series

The 400 series, which was heat-treated at 400°C for various times, also showed the same decrease in hardness values throughout the heat treatment as the 300 series did. The hardness values, however, were slightly lower than the 300 series values. After 4 hours of heat treatment, the hardness lowered to 60 H_K, which is 47% lower than the as-sprayed control sample, which is shown in Figure 10.

400-xA Series - Knoop Hardness

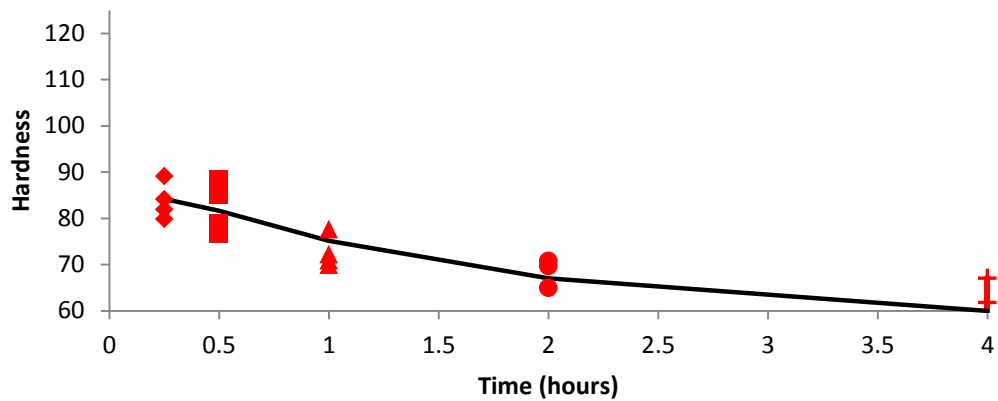


Figure 10: 400 Series, Face A - Knoop Hardness

4.5 Microstructure Analysis

Scanning electron microscope (SEM) images were taken of selected samples to compare the growth of the precipitates. The precipitates, appearing as small white areas, affect the hardness of the sample. They were measured and compared to the size of the as-sprayed sample precipitates. The SEM image comparisons were obtained at a magnification of 1,000X.

The 100 series' precipitates did not increase in size, but the sample did form more inclusions, black regions, as can be seen in the comparison in Figure 11.

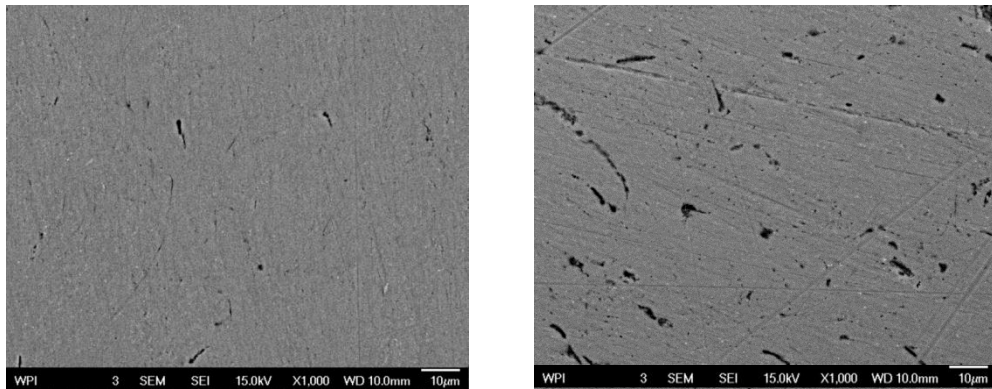


Figure 11: SEM image of unetched as-sprayed control sample (left) and sample 100-4 (right)

The precipitates of the 200 series, sample 3, which was heat-treated at 200°C for 1 hour, showed the largest growth after heat treatment, as shown in Figure 12. The approximate calculated growth of the precipitates from the as-sprayed control sample was 30%.

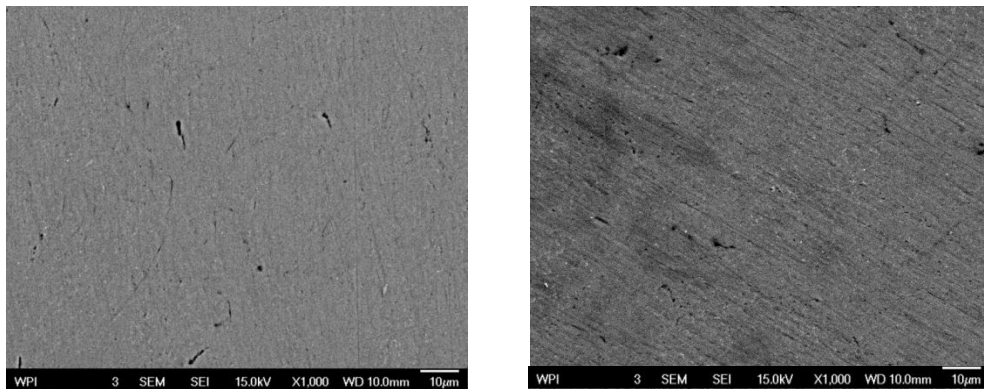


Figure 12: SEM image of unetched as-sprayed control sample (left) and sample 200-3 (right)

The 300 series' precipitates did not increase in size. Sample 1 which was heat-treated for 15 minutes, and Sample 5 which was heat-treated for 4 hours are shown in Figure 13.

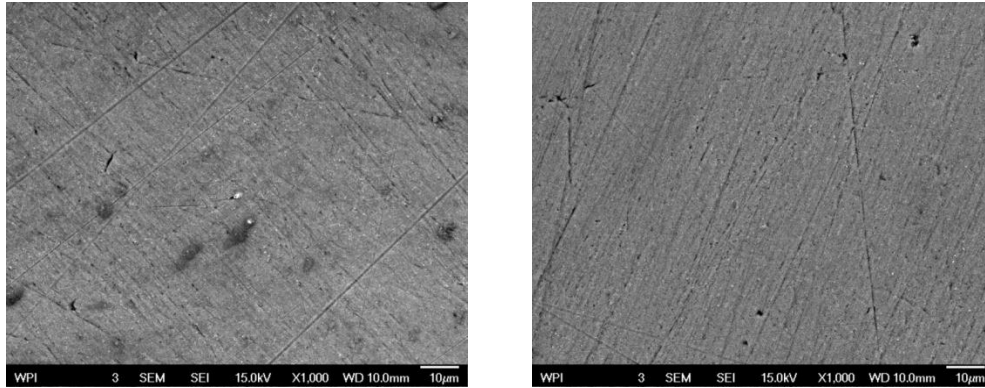


Figure 13: SEM image of unetched Sample 300-1 (left) and Sample 300-5 (right)

The 400 series' precipitates also did not increase in size. Sample 1 which was heat-treated for 15 minutes, and Sample 5 which was heat-treated for 4 hours are shown in Figure 14.

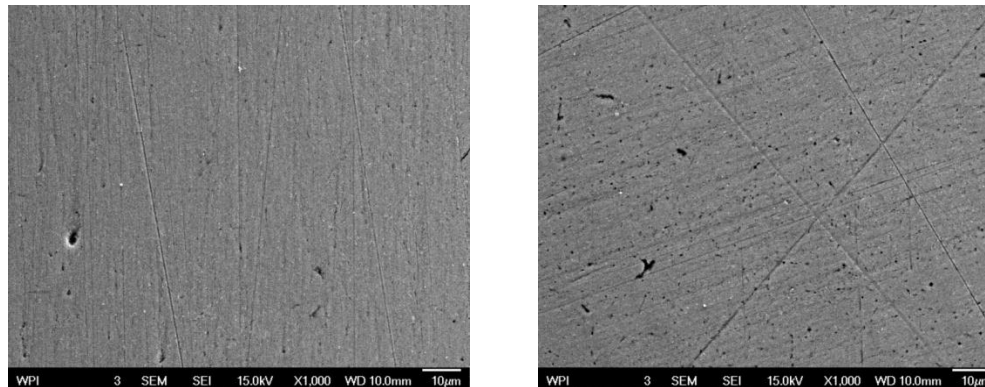


Figure 14: SEM image of unetched sample 400-1 (left) and sample 400-5 (right)

5. Conclusions

The following conclusions were made based on the hardness data and microstructural images.

- Heat treatment of cold sprayed Al 6061 lowers hardness by as much as 47%
- Precipitates grew by a maximum of 30% when material was heat treated at 200°C for 1 hour
- Optimal heat treating condition to yield maximum hardness was 200°C for 1 hour

After each sample was subjected their respective heat treatments, the hardness of all of the samples decreased. The 200 series samples had the highest hardness values, and the hardness decreased with increasing heat-treatment temperature. The lowest hardness value recorded was from sample 400-5, which was heat-treated at 400°C for 4 hours. This hardness value of 60 H_K was a decrease of 47% from the as-sprayed control sample.

The only significant growth of the precipitates was in sample 200-3, which was heat-treated at 200°C for 1 hour. The approximate calculation of the growth from the as-sprayed control sample was 30%. When the precipitates grow throughout the heat treatment, the hardness is increased. This conclusion was proven by the fact that the highest hardness value was recorded from sample 200-3 at 117. This shows that the optimal heat treatment condition in this experiment was at 200°C for 1 hour.

5.1 Future Work

In order to supplement the findings of this project, the team has a variety of recommendations for further research in order to learn more about the effects of heat treating on cold sprayed Aluminum 6061.

First, the precipitates formed during different heat treating temperatures and times should be examined to determine which phases are present in the cold spray after heat treatment. This could be done through the use of a transmission electron microscope (TEM) and energy dispersive spectroscopy (EDS). The phases observed can be compared to those predicted through the aluminum and magnesium phase diagram for 6061.

Most importantly, the effects of the various heat treating processes on ductility should be examined. Although cold spray has significant benefits to the hardness of the surface of a part, it has a negative effect on the ductility. The annealing of a cold sprayed part could potentially recover the ductility without compromising all of the hardness. Testing should be done to investigate how heat treating cold spray affects the ductility. Furthermore, the effects of ductility and hardness after heat treating cold spray could be coupled and optimized in order to create the best possible coating for military vehicles.

References

Army, U. (2012, October 13). *ARL Center for Cold Spray-Cold Spray Process*. Retrieved April 2012, from United States Army Research Lab:

<http://www.arl.army.mil/www/default.cfm?page=370>

Champagne, V., Helfrich, D., Trexler, M., & Gabriel, B. (2012). The effects of cold spray impact velocity on deposit hardness. *Modeling and Simulations Materials Science and Engineering* .

International, A. (1998). *Aluminum and Aluminum Alloys*. Davis and Associates.

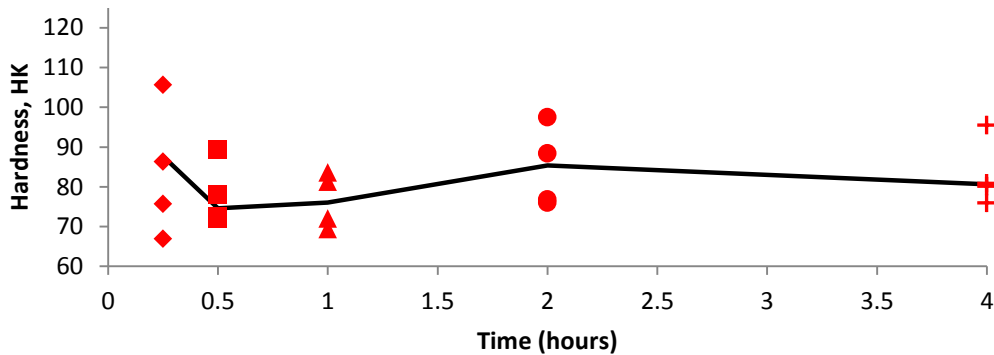
Powder Preperation. (2009, February 8). Retrieved April 2012, from SubsTech: Substances and Technologies: http://www.substech.com/dokuwiki/doku.php?id=powder_preparation

Totten, G., & Mackenzie, S. (2003). *The Handbook of Aluminum*. Marcel Dekker Inc.

Appendix A – 100 Series Data

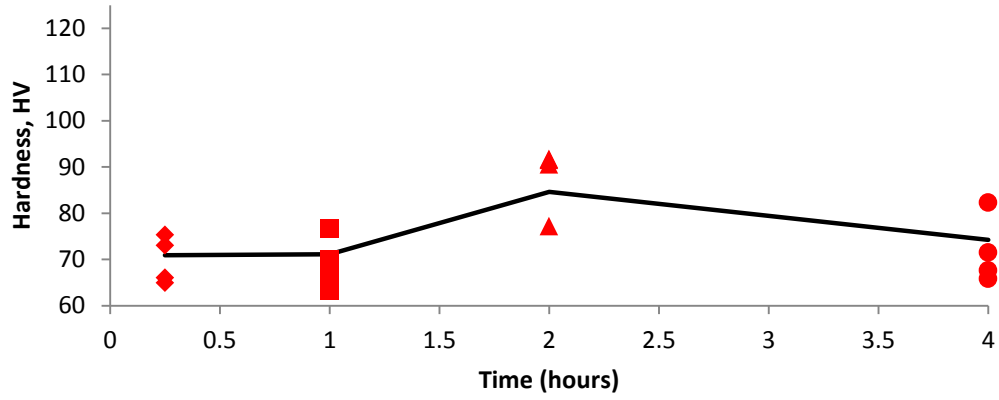
100-x Knoop Hardness							
Sample	1	2	3	4	5	6	Average
100-1A	65.50	79.00	83.70	75.50	76.80	69.60	75.02
100-1B	66.90	75.70	86.30	105.60	79.60	111.50	87.60
100-2A							
100-2B	89.40	78.00	72.50	71.90	65.00	70.60	74.57
100-3A	89.30	84.00	73.90	66.90	68.90	79.80	77.13
100-3B	72.00	69.30	83.60	81.20	71.90	78.20	76.03
100-4A	101.00	97.70	89.60	98.10	99.40	91.70	96.25
100-4B	97.50	76.00	76.80	88.40	85.70	87.90	85.38
100-5A	67.90	82.50	79.40	73.10	79.90	89.70	78.75
100-5B	95.50	80.80	75.90	80.10	82.20	69.30	80.63

100-xB Series - Knoop Hardness

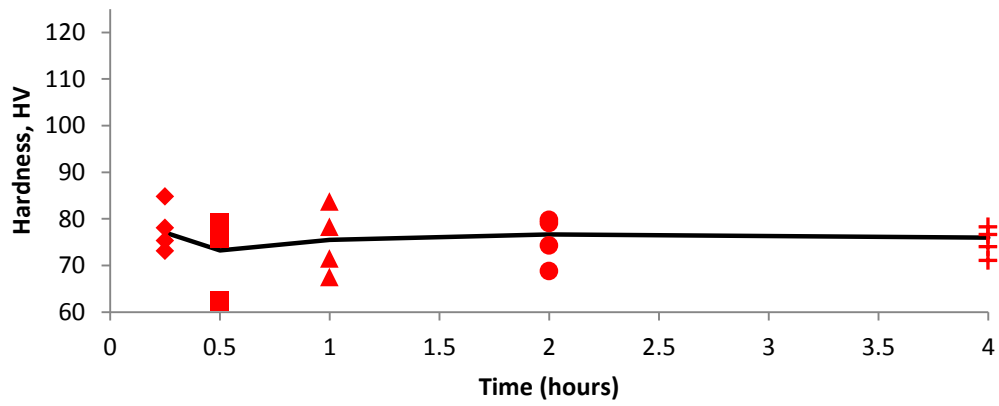


100-x Vickers Hardness							
Sample	1	2	3	4	5	6	Average
100-1A	66.00	75.30	64.90	73.00	81.50	64.90	70.93
100-1B	78.10	75.30	73.10	84.80	62.60	88.50	77.07
100-2A							
100-2B	79.10	62.50	62.20	75.90	73.00	86.60	73.22
100-3A	70.00	76.60	67.40	63.30	79.90	69.30	71.08
100-3B	78.30	83.70	67.50	71.50	70.30	81.80	75.52
100-4A	77.20	91.70	91.50	90.50	71.20	85.50	84.60
100-4B	68.80	79.80	74.30	79.10	83.40	74.70	76.68
100-5A	82.30	67.60	65.80	71.50	79.60	78.50	74.22
100-5B	71.10	76.60	74.00	78.30	68.00	87.80	75.97

100-xA Series - Vickers Hardness



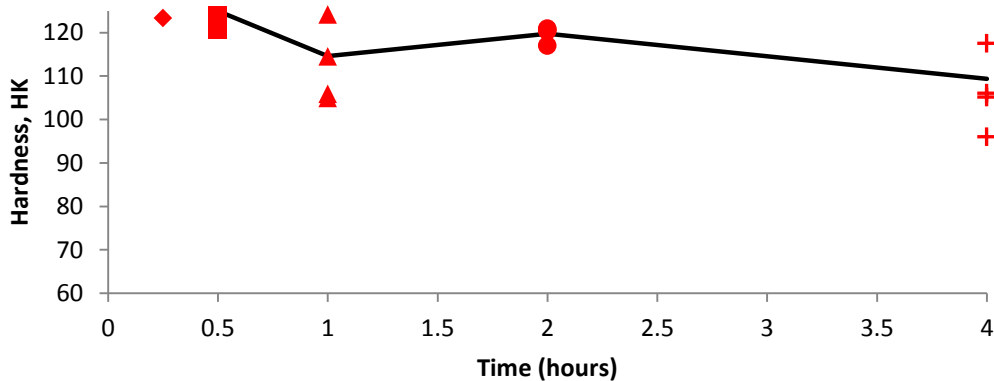
100-xB Series - Vickers Hardness



Appendix B – 200 Series Data

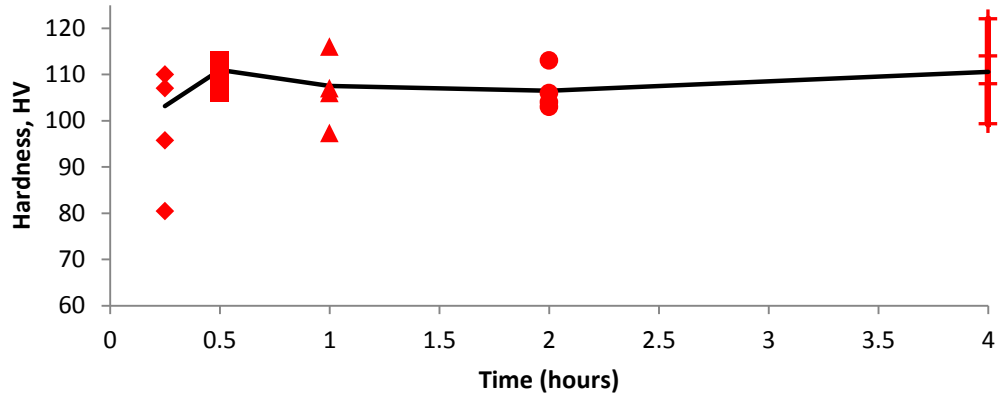
200-x Knoop Hardness							
Sample	1	2	3	4	5	6	Average
200-1A	118.70	112.50	112.90	115.40	113.40	121.80	115.78
200-1B	123.30	127.60	127.10	138.50	132.90	138.30	131.28
200-2A	103.80	111.00	103.70	109.60	106.30	105.80	106.70
200-2B	120.70	130.40	122.60	123.80	129.30	123.00	124.97
200-3A	111.10	113.20	124.80	118.60	121.70	112.80	117.03
200-3B	114.50	105.90	104.90	124.10	113.90	124.40	114.62
200-4A	106.70	115.40	112.80	111.00	117.50	111.30	112.45
200-4B	120.90	120.70	120.40	117.00	116.10	123.30	119.73
200-5A	115.00	110.80	115.80	125.90	115.10	126.10	118.12
200-5B	105.10	96.00	117.50	106.00	107.70	123.80	109.35

200-xB Series - Knoop Hardness

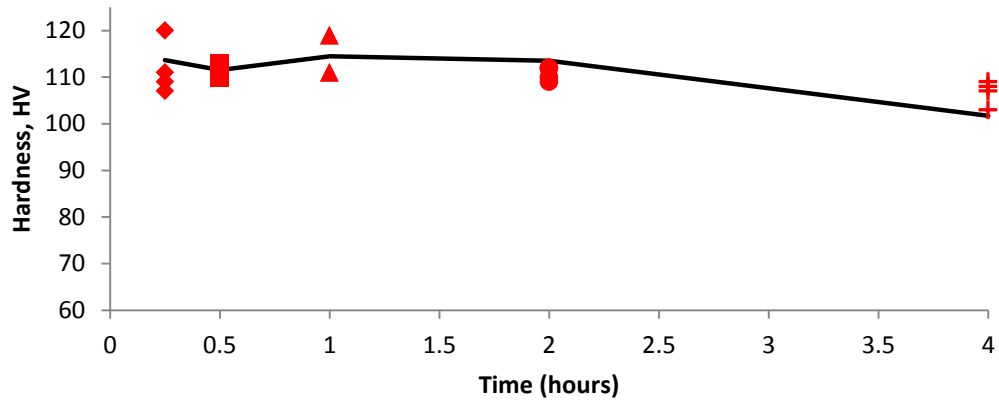


200-x Vickers Hardness							
Sample	1	2	3	4	5	6	Average
200-1A	80.40	107.00	110.00	95.70	112.00	114.00	103.18
200-1B	109.00	107.00	111.00	120.00	118.00	117.00	113.67
200-2A	113.00	111.00	107.00	106.00	108.00	121.00	111.00
200-2B	112.00	110.00	113.00	112.00	109.00	113.00	111.50
200-3A	107.00	97.30	106.00	116.00	111.00	108.00	107.55
200-3B	111.00	111.00	119.00	119.00	112.00	115.00	114.50
200-4A	113.00	106.00	103.00	104.00	109.00	104.00	106.50
200-4B	109.00	112.00	110.00	112.00	123.00	115.00	113.50
200-5A	108.00	99.30	114.00	122.00	113.00	107.00	110.55
200-5B	107.00	103.00	108.00	109.00	108.00	75.30	101.72

200-xA Series - Vickers Hardness



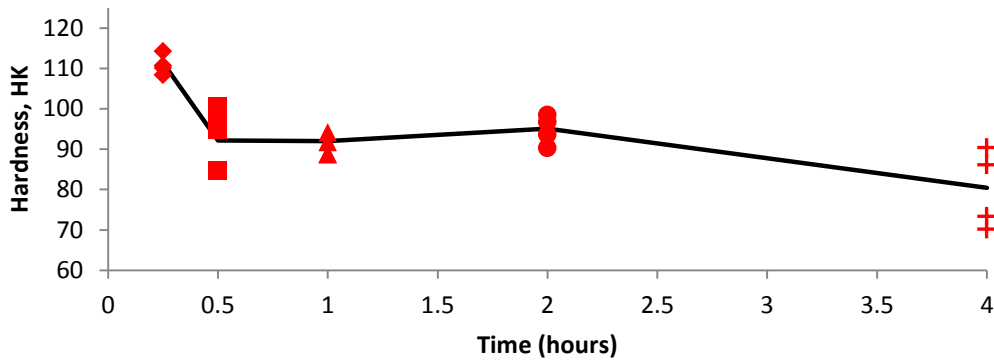
200-xB Series - Vickers Hardness



Appendix C – 300 Series Data

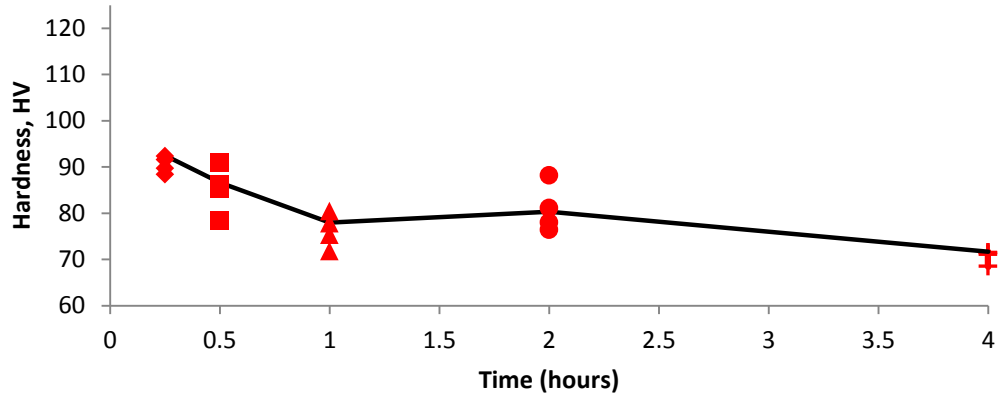
300-x Knoop Hardness							
Sample	1	2	3	4	5	6	Average
300-1A	87.70	98.80	92.70	104.60	94.30	102.40	96.75
300-1B	110.70	114.20	108.40	110.10	113.40		111.36
300-2A	90.60	94.20	91.70	102.60	99.50	91.70	95.05
300-2B	97.70	94.70	100.50	84.70	89.30	86.10	92.17
300-3A	87.30	85.70	93.80	85.40	90.30	86.20	88.12
300-3B	88.70	91.70	88.80	94.10	92.50	96.30	92.02
300-4A	85.20	84.20	84.00	86.70	92.20	82.70	85.83
300-4B	90.30	93.60	96.70	98.50	100.40	90.90	95.07
300-5A	76.60	78.80	80.10	80.00	80.70	81.70	79.65
300-5B	73.30	70.20	90.40	86.10	83.80	78.60	80.40

300-xB Series - Knoop Hardness

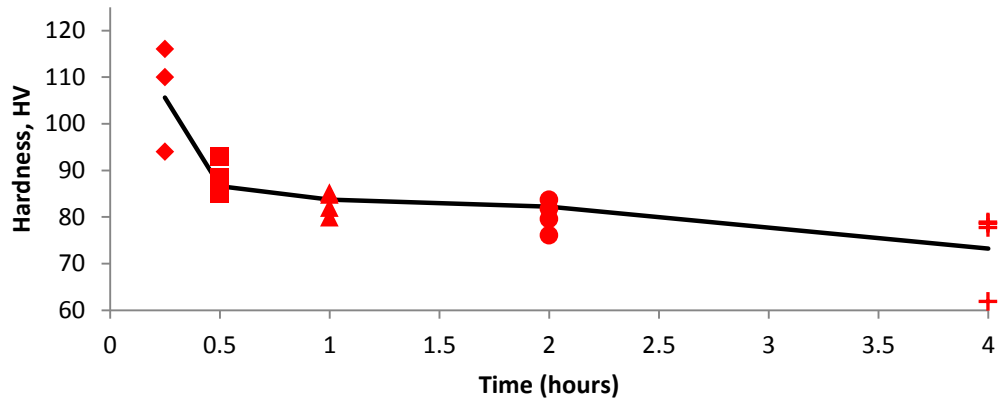


300-x Vickers Hardness							
Sample	1	2	3	4	5	6	Average
300-1A	88.40	92.30	91.60	89.70	99.30	93.80	92.52
300-1B	116.00		94.00	110.00	110.00	98.20	105.64
300-2A	78.50	86.10	90.90	85.30	90.30	88.50	86.60
300-2B	85.00	88.50	88.20	93.00	76.70	88.50	86.65
300-3A	80.40	71.80	77.80	75.30	74.00	88.70	78.00
300-3B	82.00	79.90	85.20	84.80	87.80	82.50	83.70
300-4A	81.10	78.00	88.20	76.40	74.90	83.20	80.30
300-4B	79.60	76.10	81.80	83.70	85.90	86.10	82.20
300-5A	71.50	71.10	71.40	68.50	70.30	77.20	71.67
300-5B	78.60	78.90	61.90	77.70	77.00	65.50	73.27

300-xA Series - Vickers Hardness



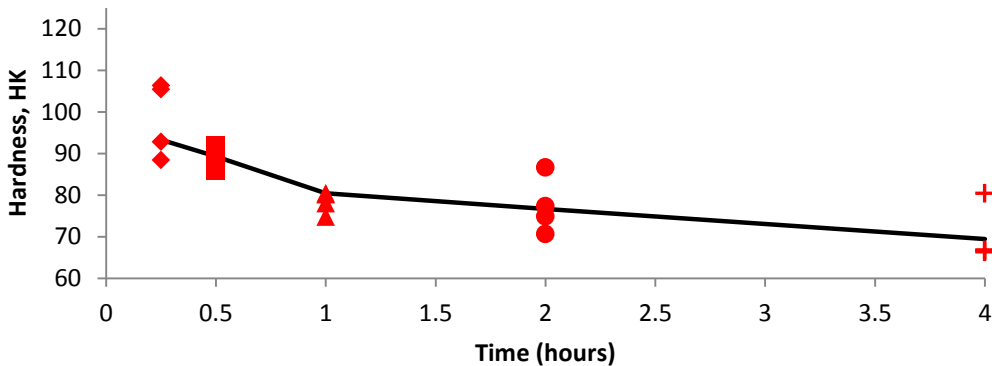
300-xB Series - Vickers Hardness



Appendix D

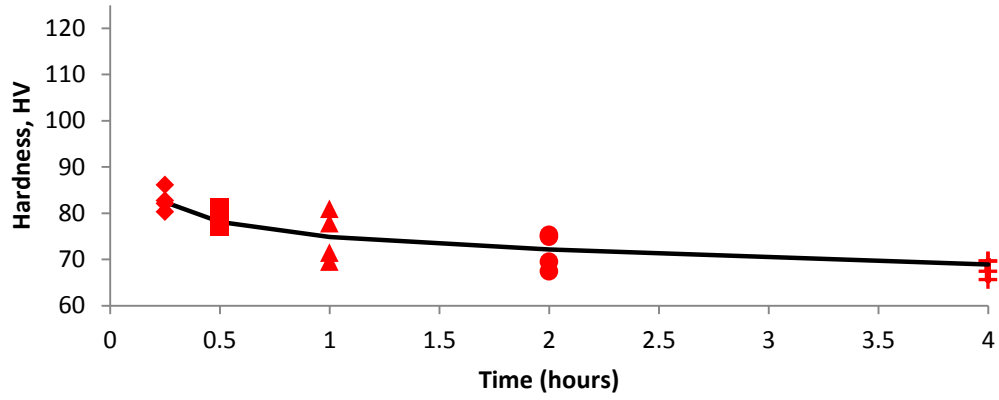
400-x Knoop Hardness							
Sample	1	2	3	4	5	6	Average
400-1A	79.80	89.10	81.90	84.10	82.60	87.80	84.22
400-1B	106.30	105.40	88.40	92.80	88.10	78.50	93.25
400-2A	78.90	76.70	88.40	85.10	85.10	75.80	81.67
400-2B	85.80	89.40	88.00	91.80	92.30	88.60	89.32
400-3A	69.90	77.60	70.90	72.20	75.80	84.20	75.10
400-3B	80.40	74.80	78.00	80.20	84.30	84.80	80.42
400-4A	70.70	64.90	59.20	69.60	68.00	69.80	67.03
400-4B	74.90	77.30	70.60	86.60	74.40	76.30	76.68
400-5A	61.70	51.50	58.10	67.00	59.70	61.90	59.98
400-5B	66.60	66.30	66.80	80.40	64.20	72.50	69.47

400-xB Series - Knoop Hardness



400-x Vickers Hardness							
Sample	1	2	3	4	5	6	Average
400-1A	80.30	86.10	82.70	82.10	80.10	83.20	82.42
400-1B	82.50	82.50	78.50	81.30	84.00	83.40	82.03
400-2A	77.00	78.60	81.10	79.90	75.90	76.30	78.13
400-2B	78.30	84.60	82.00	76.40	78.00	80.40	79.95
400-3A	71.40	69.50	77.80	80.90	73.50	75.90	74.83
400-3B	69.30	79.60	80.60	77.70	74.40	80.30	76.98
400-4A	67.40	69.50	74.90	75.30	75.20	70.60	72.15
400-4B	69.60	77.70	64.40	68.10	68.00	77.80	70.93
400-5A	69.60	67.40	69.60	65.60	71.50	69.70	68.90
400-5B	57.30		81.80	70.10	72.20	77.00	71.68

400-xA Series - Vickers Hardness



400-xB Series - Vickers Hardness

