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Texture and Young's Modulus of Nickel/Gamma-Alumina Composites

Abdulaziz Alamr

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TEXTURE AND YOUNG'S MODULUS OF NICKEL / GAMMA-ALUMINA COMPOSITES

By

Abdulaziz Alamr

A Thesis
Submitted to the
Faculty of the Graduate College
In partial fulfillment of the
Requirement for the
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Department of Construction Engineering, Materials Engineering, and Industrial Design

Western Michigan University
Kalamazoo, Michigan
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Abdulaziz Alamr
TEXTURE AND YOUNG’S MODULUS OF NICKEL/GAMMA-ALUMINA COMPOSITES

Abdulaziz Alamr, Master of Science
Western Michigan University, 2000

To improve Young’s modulus of 2024-T3 rolled aluminum specimens, it was electroplated with nickel reinforced with nano-particles of γAl₂O₃. Taguchi method was used to investigate the effect of coating variables, which were current density, coating time, and mixing rate, on the coating thickness. The coatings were tested for crystallographic texture, using pole figures of (200) and (111) for nickel and (400) for γAl₂O₃. Surface topography was measured using WYKO Vertical Interference Microscope. The crystallographic texture of nickel in the coating depended on the coating conditions. A universal testing machine was used to measure the elastic modulus. The values of Young’s modulus demonstrated strong anisotropy as a result of the crystallographic texture. The effect of coating parameters on the surface topography was studied.
TABLE OF CONTENTS

ACKNOWLEDGMENTS ........................................................................................................ ii
LIST OF TABLES ........................................................................................................ vi
LIST OF FIGURES ......................................................................................................... viii

CHAPTER

I. INTRODUCTION ........................................................................................................... 1
  Modulus of Elasticity .............................................................................................. 1
  Crystallographic Texture .................................................................................. 4
  Aluminum and Aluminum Alloy ........................................................................ 7
  Nickel .................................................................................................................. 9
  Gamma-Aluminum Oxide .................................................................................. 10
  Fabrication .......................................................................................................... 10
  Electroplating ...................................................................................................... 11
  Taguchi Method .................................................................................................. 13

II. EXPERIMENTAL APPARATUS AND SOLUTIONS .................................................. 16
  Apparatus ............................................................................................................ 16
    Holding Tanks .................................................................................................. 16
    Agitator ............................................................................................................ 16
    Immersion Heater ............................................................................................ 18
    Electrolytic Solution Heater ............................................................................ 18
    Anode ............................................................................................................... 19
    Aluminum Substrate ....................................................................................... 20
# Table of Contents—continued

Power Supply ................................................................. 20  
Experiment Solutions ....................................................... 21  
Nickel Sulfamate .............................................................. 21  
Alkaline Degreaser ........................................................... 23  
Alkaline Etching .............................................................. 23  
Acid Etching ................................................................. 24  
Zincate ................................................................. 24  

## III. EXPERIMENTAL DETAILS ..................................................... 27  
Experimental Procedure ..................................................... 27  
Surface-Preparation Processes ........................................... 31  
Electroplating-Stage One .................................................... 35  
Results and Discussion ...................................................... 37  
Electroplating-Stage Two .................................................... 44  
Results and Discussion ...................................................... 46  
Electrodeposition ............................................................. 51  
Preferred Orientations ...................................................... 55  
Elastic Modulus .............................................................. 55  
Surface Topography ........................................................ 55

## IV. RESULTS AND DISCUSSION .................................................. 57  
Young’s Modulus ............................................................ 57  
Pole Figures ................................................................. 61  
Surface Topography ........................................................ 70
Table of Contents—continued

Particle Concentrations ................................................................. 73
Discussion ..................................................................................... 73

V. CONCLUSION .............................................................................. 77

APPENDICES

A. Item Suppliers ............................................................................ 79
B. Analyzing of Coating Data by Minitab Program ......................... 81
C. Determination the Slope of Elastic Zone and Coefficient of
   Determination of the Coated Specimens by Least Square Method .... 96
D. Standard 2-Theta XRD of Coated Specimens ............................. 109
E. Surface Topography of Coated Specimens ................................ 114

REFERENCES ................................................................................... 124
# LIST OF TABLES

1. The Designation of Aluminum Alloys ................................................................. 8
2. The Mechanical Properties of Al 2024-T3 ............................................................. 9
3. Mechanical Properties of Nickel ..................................................................... 9
4. Properties of Nanosize $\gamma$-Aluminum Oxide ...................................................... 10
5. L9 Orthogonal Array .................................................................................... 14
6. Compositions and Operation Conditions of Sulfamate Bath .................................. 21
7. Composition of Modified Zincate ........................................................................ 26
8. L9 Fractional Orthogonal Array .......................................................................... 28
9. Factor Levels .................................................................................................... 28
10. Electroplating Procedure for the First Stage ....................................................... 34
11. Electrolytic Solution Operation Conditions ......................................................... 36
12. Coating Thickness Measurements ..................................................................... 38
13. Mean Thickness and Standard Deviation of the Electroplating-Stage One Specimens ............................................................ 39
14. Contribution of the Factors to the Coating Thickness ......................................... 42
15. Contribution of the Factors to Standard Deviation .............................................. 43
16. Coating Conditions of Electroplating-Stage Two ............................................... 45
17. Pretreatment Process Time of Electroplating-Stage Two ..................................... 45
18. Coating Thickness, $\mu$m, of Electroplating-Stage Two ....................................... 46
19. Mean Thickness and Standard Deviation of Electroplating-Stage Two Specimens ............................................................................................................. 48
20. Coating Thickness Measurement (µm) ................................................................ 49
<table>
<thead>
<tr>
<th>No.</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>21.</td>
<td>Mean Thickness and Standard Deviation</td>
<td>49</td>
</tr>
<tr>
<td>22.</td>
<td>Definition of Low Conditions of Coating Process</td>
<td>51</td>
</tr>
<tr>
<td>23.</td>
<td>Definition of High Conditions of Coating Process</td>
<td>52</td>
</tr>
<tr>
<td>24.</td>
<td>Electrodeposition Conditions</td>
<td>53</td>
</tr>
<tr>
<td>25.</td>
<td>The Substrate Thickness, Coating Thickness, Standard Deviation of Coated Tensile Test Specimens</td>
<td>54</td>
</tr>
<tr>
<td>26.</td>
<td>Slope and Coefficient of Determination of the Coated Specimens</td>
<td>58</td>
</tr>
<tr>
<td>27.</td>
<td>Young’s Modulus of Coated Specimens</td>
<td>59</td>
</tr>
<tr>
<td>28.</td>
<td>Young’s Modulus of the Coating Layer</td>
<td>60</td>
</tr>
<tr>
<td>29.</td>
<td>The Average and Standard Deviation of Young’s Modulus of Coating Layer</td>
<td>60</td>
</tr>
<tr>
<td>30.</td>
<td>Average Roughness, ( R_s ), and Root Mean Square Roughness, ( R_q ), of Coated Specimens</td>
<td>71</td>
</tr>
<tr>
<td>31.</td>
<td>Calculated Values of Particle Concentrations in the Coating</td>
<td>73</td>
</tr>
<tr>
<td>32.</td>
<td>Calculated Particle Concentrations Using SEM</td>
<td>75</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

1. Schematic Representation of the Elastic Modulus as Function of the Volume Fraction of the Reinforcement ................................................................. 2
2. Schematic Stress-Strain Diagram Showing Linear Elastic Deformation .................................................... 3
3. PVS Fabrication Method ............................................................................................................... 11
4. Schematic of Electroplating Layout ......................................................................................... 12
5. Bubblator Setup ....................................................................................................................... 17
6. Bubblator Air Controller Setup ............................................................................................. 17
7. Immersion Heater .................................................................................................................. 18
8. Plating Solution Heater .......................................................................................................... 19
9. Nickel Oxide Depolarized Anode ......................................................................................... 20
10. Flat Specimen Dimensions .................................................................................................. 29
11. Electroplating and Electrodeposition Process ........................................................................ 30
12. Alkaline Degreaser with an Immersion Heater ..................................................................... 31
13. Pretreatment Equipments ...................................................................................................... 35
14. Electroplating Bath Setup ..................................................................................................... 36
15. Division of the Coated Specimen .......................................................................................... 37
16. Section Division of Coated Specimen ................................................................................... 37
17. Factor Effects Dot-Plot for Coating Thickness ...................................................................... 40
18. Factor Effects Dot-Plot for Standard Deviation .................................................................... 40
19. Factor Effects Plot for Mean Thickness ................................................................................ 40
20. Factor Effects Plot for Standard Deviation ........................................................................... 41
List of Figures—continued

21. Half-Normal Plot for Mean Thickness .................................................. 41
22. Half-Normal Plot for Standard Deviation ............................................. 42
23. The Setup of Electroplating-Stage Two ............................................... 44
24. Mean Thickness Plot for the Electroplating-Stage Two Specimens .......... 47
25. Standard Deviation Plot of Electroplating-Stage Two Specimens .......... 47
26. Mean Thickness Plot of Electroplating-Stage Two Specimens ............... 50
27. Standard Deviation Plot of Electroplating-Stage Two Specimens .......... 50
28. Tensile Specimen Dimensions ......................................................... 52
29. Slope and Coefficient of Determination of Sample Number 27 ............. 57
30. The Average of Young’s Modulus for Coating Layer ............................ 61
31. (200) Pole Figure of Specimen Number 25 ....................................... 62
32. (111) Pole Figure of Specimen Number 25 ....................................... 62
33. (200) Pole Figure of Specimen Number 33 ....................................... 63
34. (111) Pole Figure of Specimen Number 33 ....................................... 63
35. (200) Pole Figure of Specimen Number 41 ....................................... 64
36. (111) Pole Figure of Specimen Number 41 ....................................... 64
37. (200) Pole Figure of Specimen Number 49 ....................................... 65
38. (111) Pole Figure of Specimen Number 49 ....................................... 65
39. (200) Pole Figure of Specimen Number 26 ....................................... 66
40. (111) Pole Figure of Specimen Number 26 ....................................... 66
41. (200) Pole Figure of Specimen Number 37 ....................................... 67
42. (111) Pole Figure of Specimen Number 37 ....................................... 67
List of Figures—continued

43. (200) Pole Figure of Specimen Number 45 ................................................. 68
44. (111) Pole Figure of Specimen Number 45 ................................................. 68
45. (200) Pole Figure of Specimen Number 53 ................................................. 69
46. (111) Pole Figure of Specimen Number 53 ................................................. 69
47. (400) Pole Figure of Gamma-Alumina ...................................................... 70
48. Surface Topography of a Coated Specimen .............................................. 71
49. Average Roughness, \( R_a \), of Coated Specimens ...................................... 72
50. Root Mean Square Roughness, \( R_q \), of Coated Specimens ....................... 72
Modulus of Elasticity

Young’s modulus of materials is one the most important mechanical properties and its knowledge is of prime importance in applied and/or fundamental fields. For examples, the Young’s modulus is the most important parameter one must have in design of bridges, especially in making bridge safe against vibrations. The second example would be the crankshaft of engine. The crankshaft needs to be supported between bearings based on its Young’s modulus. The number of bearings depends on how stiff the material of crankshaft is. Since Young’s modulus is proportional to the atomic bond of the material, heat treatment processes cannot change Young’s modulus. Young’s modulus cannot change much by alloying, but texture can have significant influence. Coating the material or using the material as a matrix and another material whose Young’s modulus is higher than that of the matrix as a reinforcement to make a composite increases its Young’s modulus. Young’s modulus of the composite should fall between an upper region represented by

\[ E_c = E_r V_r + E_m V_m \] (1.1)

and a lower region

\[ E_c = \frac{E_m E_r}{V_m E_r + V_r E_m} \] (1.2)
where $E_c$, $E_r$, and $E_m$ are the Young's modulus of the composite, the reinforcement, and the matrix, respectively, $V_r$ and $V_m$ are the volume fraction of the reinforcement and the matrix respectively. Figure 1 plots upper-and lower-region $E_c$-versus-$V_r$.

![Graph showing the relationship between Young's modulus of composite and volume fraction of reinforcement](image)

Figure 1. Schematic Representation of the Elastic Modulus as Function of the Volume Fraction of the Reinforcement.

When continuous aligned fibers reinforce a matrix and the stress is applied along the direction of alignment, Young’s modulus of the composite in the longitudinal direction can be obtained using the upper region equation. When the composite is loaded in the transverse direction, the lower region equation is used to obtain Young’s modulus in the transverse direction. When short discontinuous and randomly orientated fibers reinforce a composite, the Young’s modulus can be determined by the following equation \(^{(4)}\)

$$E_c = KE_r V_r + E_m V_m$$

(1.3)
where $K$ is a fiber efficiency parameter that depends on the volume fraction and the $E_r/E_m$ ratio. Its magnitude will be in the range 0.1 to 0.6.\(^{(4)}\)

The elastic modulus can be measured in different ways. The elastic modulus can be obtained from the stress-strain curves of tension tests by calculating the stresses and strains of two points on the straight line (the slope) as shown in figure 1.\(^{(5)}\)

![Figure 2. Schematic Stress-Strain Diagram Showing Linear Elastic Deformation.](image)

To reduce the error, which might be obtained by plotting the numerical form data of load-versus-extension, of determining the Young's modulus, least square method can be used. Young's modulus can be obtained using Specification E111\(^{(6)}\) in the American Society for Testing and Material by applying the following equations:

\[
E = \frac{(\Sigma XY) - K\overline{XY}}{(\Sigma X^2 - K\overline{X}^2)} \quad (1.4)
\]

\[
\overline{X} = \frac{\Sigma X}{K} \quad (1.5)
\]

\[
X = \frac{\Sigma X}{K} \quad (1.6)
\]

where
\[ Y = \text{applied axial stress} \]
\[ X = \text{corresponding strain} \]
\[ Y = \text{Average of } Y \text{ values} \]
\[ X = \text{Average of } X \text{ values, and} \]
\[ K = \text{the number of } X, \text{ and } Y \text{ data pairs.} \]

Equation (1.7) is used to achieve the coefficient of determination, \( r^2 \), which is ranged in value from 0 to 1. If it is 1, there is a perfect correlation in the sample. \(^6\)

\[
r^2 = \left( \frac{\sum XY - \frac{\sum X \sum Y}{K}}{\sqrt{\left( \frac{\sum X^2}{K} - \frac{(\sum X)^2}{K} \right) \left( \frac{\sum X^2}{K} - \frac{(\sum Y)^2}{K} \right)}} \right)^2
\]

\[(1.7)\]

**Crystallographic Texture**

Most processes of deformation a polycrystalline metal such as rolling, forging, and drawing change the randomly oriented grains to be parallel to the direction of deformation. Consequently, a strong preferred orientation or texture develops in the metal. \(^3,7\) Preferred orientation has received much attention because of the important effect they have on the properties of the commercial products. A fine-grain metal specimen in which the grains have random lattice orientation will possess identical properties in all directions (isotropic), but a specimen with a preferred orientation will have directional, or anisotropic properties which may be wanted or unwanted, depending upon the intended use of the material. \(^8\)

An example, for anisotropy of mechanical properties is the deep drawing of cups. Ears grow symmetrically around the edge with associated difference in thickness
of the cup as a result of the anisotropy of the mechanical properties. In contrast, a sheet, which has appropriate texture, can be stronger in useful direction than material in which a texture has not been developed. \(^{(8,9)}\)

Fiber texture and rolling texture are the most common textures produced by cold work. \(^{(7)}\) Uniaxial deformation processes such as wire-drawing and extrusion produce fiber texture. Fiber texture resembles the arrangement of the oriented grains in fibrous materials such as stretched rubber. Fiber texture consists of orientations that have a particular crystallographic direction parallel to the axis of the wire and other directions spread with equivalent possibility around the wire axis giving cylindrical symmetry to the whole. \(^{(10)}\) The arcing of rings in X-ray diffractions reveals fiber texture. \(^{(7)}\) Wires of body-center cubic metals deformed by drawing have fiber texture with \(<110>\) direction parallel to the wire axis. On the other hand, face-center cubic metal can have double fiber texture with both \(<111>\) and \(<100>\). \(^{(7)}\) The apparent mechanical properties of a metal will be changed as a result of crystallographic texture. For example, Young's modulus of \([111]\) and \([100]\) for nickel can be calculated as follow: for nickel, \(S_{11} = 0.733 \text{ E-11 m}^2/\text{N}, S_{12} = -0.274\text{E-11 m}^2/\text{N},\) and \(S_{44} = 0.80\text{E-11 m}^2/\text{N},\) where \(S\) indicates elastic compliance. \(^{(11)}\) Young's modulus for any direction can be calculated by using the following equation \(^{(12)}\)

\[
\frac{1}{E_{[hk\ell]}} = S_{11} - 2\left(S_{11} - S_{12} - \frac{1}{2}S_{44}\right)(\alpha^2 \beta^2 + \alpha^2 \gamma^2 + \beta^2 \gamma^2) \tag{1.8}
\]

where \(\alpha, \beta,\) and \(\gamma\) are the direction cosines of the \([hk\ell]\) direction and the \([100], [010],\) and \([001]\) directions, respectively. \(^{(12)}\) Thus,

For \([100]\): \(\alpha = 1, \beta = \gamma = 0\) and \(\alpha^2 \beta^2 + \alpha^2 \gamma^2 + \beta^2 \gamma^2 = 0\)

For \([111]\): \(\alpha = \beta = \gamma = 0.57\) and \(\alpha^2 \beta^2 + \alpha^2 \gamma^2 + \beta^2 \gamma^2 = 0.33\)
Now evaluate equation (1.8) using the values of $S_{ij}$ and the just obtained geometrical relationship to obtain $E_{[100]}$ and $E_{[111]}$ for nickel.

$$\frac{1}{E_{[hkl]}} = S_{11} - 2\left(S_{11} - S_{12} - \frac{1}{2}S_{44}\right)\left(\alpha^2 \beta^2 + \alpha^2 \gamma^2 + \beta^2 \gamma^2\right)$$

For $E_{[100]}$

$$\frac{1}{E_{[100]}} = 0.733 \times 10^{-11} \text{ m}^2/\text{N}$$

$$E_{[100]} = 136 \text{ GPa}$$

For $E_{[111]}$

$$\frac{1}{E_{[111]}} = 10^{-11} \frac{m^2}{N} \left(0.733 - 2(0.73 + 0.27 - 0.4)\right)0.33$$

$$E_{[111]} = 298 \text{ GPa}$$

For most cubic materials $E_{<111>}$, which is the modulus measured along the $<111>$ direction, is larger than $E_{[100]}$. The modulus along any arbitrary $[hkl]$ direction is obtained from $E_{<111>}$ and $E_{<100>}$ as

$$\frac{1}{E_{[hkl]}} = \frac{1}{E_{<100>}} - 3\left(\frac{1}{E_{<100>}} - \frac{1}{E_{<111>}}\right)\left(\alpha^2 \beta^2 + \alpha^2 \gamma^2 + \beta^2 \gamma^2\right)$$

Rolling texture can be described in an approximate way by choosing one or two ideal orientations involving the plane $(hkl)$ that lies parallel to the plane of the sheet and the direction $[uvw]$ that lies in the rolling direction. Rolling texture has both a specific crystallographic direction parallel to the direction of rolling and a plane of low indices in the rolling plane.\(^{(7,8)}\)
Orientations in electrodeposits are of considerable technical importance. The deposit is affected by the nature of the electrolyte, its hydrogen-ion concentration, the nature and condition of the substrate material, the temperature, and current density. Under some conditions the orientation of the base material is copied by the deposit.\(^{(13)}\) It is investigated that the grain in the deposit material are often continuations of the grain in the substrate material. Clean, freshly etched surface, small current density, and similarity in structure between the substrate metal and deposit are the suitable conditions to continuations of substrate-metal grains into the electrodeposit.\(^{(8,13)}\) Plating conditions can alter both the degree of orientation and the nature of the texture.\(^{(10)}\)

Directional properties of material can be mapped on a stereographic projection. Examples are the modulus of elasticity, the yield point, and the electrical conductivity.\(^{(8,14,15)}\)

### Aluminum and Aluminum Alloys

The unique combinations of properties provided by aluminum and its alloys make aluminum one of the most versatile, economical, and attractive metallic materials for a broad range of uses. The density of aluminum is 2.7 g/cm\(^3\) which is approximately one-third as much as steel (7.83 g/cm\(^3\)). Aluminum displays excellent electrical and thermal conductivity. Aluminum is nonferromagnetic, a property of importance in the electrical and electronic industries. It is nonpyrophoric. This is important in applications where flammable or explosive-materials handing or exposure is involved. The ease with which aluminum may be fabricated into any form is one of its important features.\(^{(16)}\)

The mechanical, physical, and chemical properties of aluminum alloys depend
upon composition and microstructure. The addition of selected elements to pure aluminum enhances its properties and usefulness.\(^{(16,17)}\) Because of this, most application of aluminum utilize alloys having one or more elemental additions. The major alloying additions used with aluminum are copper, silicon, manganese, and zinc. The total percentage of these elements can equal up to 10 percent of the alloy composition. There are also impurity elements present. However, their total percentage is usually under 0.15 percent in aluminum alloys.\(^{(16,17)}\)

Aluminum alloys can be divided into two categories: cast composition and wrought composition. Composition for both types is designated by a four-digit number which indicates the principal impurities. A hyphen and the basic temper designation—a letter and a possible one-to three digit number. This indicates the mechanical and/or heat treatment to which the alloy has been subjected, follow the four-digit number.\(^{(17)}\) An example of the design of this system is the 2024-T3. The numbers 2024 indicates the elements which make up this alloy. The first digit of the four-digit designation indicates the group as shown in the table 1.\(^{(17)}\)

### Table 1

<table>
<thead>
<tr>
<th>Element</th>
<th>Digit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum, = 99.00%</td>
<td>1xxx</td>
</tr>
<tr>
<td>Copper</td>
<td>2xxx</td>
</tr>
<tr>
<td>Manganese</td>
<td>3xxx</td>
</tr>
<tr>
<td>Silicon</td>
<td>4xxx</td>
</tr>
</tbody>
</table>

The second digit in the designation indicates alloy modification. In this example, zero indicates the original alloy. Integers 1 through 9 demonstrate
modifications of the original alloy. The last two of the four digits in the 2xxx have no special significance, but serve only to indicate the different aluminum alloys in the group. T3 means that the alloy was solution heat treated, cold work, and then naturally aged. \(^{(16)}\) Table 2 \(^{(17)}\) demonstrates the mechanical properties of 2024-T3.

Table 2

<table>
<thead>
<tr>
<th>Tensile Yield (MPa)</th>
<th>Hardness HB</th>
<th>Elongation %</th>
<th>Elastic Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>345</td>
<td>120</td>
<td>18</td>
<td>71</td>
</tr>
</tbody>
</table>

Nickel

Nickel is important to modern industry due to its ability to withstand a wide variety of severe operating conditions involving corrosive environments, high temperatures, high stresses, and combination of these factors. There are several reasons for this capability. Pure nickel is ductile and tough because it has a Face-Centered Cubic up to its melting temperature. Nickel is made by conventional techniques such as wrought, cast, and powder metallurgy. Table 3 \(^{(16)}\) presents the mechanical make up of nickel.

Table 3

<table>
<thead>
<tr>
<th>Young’s Modulus (GPa)</th>
<th>Shear Modulus (GPa)</th>
<th>Yield Strength (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation %</th>
</tr>
</thead>
<tbody>
<tr>
<td>207</td>
<td>70</td>
<td>59</td>
<td>317</td>
<td>30</td>
</tr>
</tbody>
</table>
Gamma-Aluminum Oxide

Gamma-aluminum oxide, \((\gamma-\text{Al}_2\text{O}_3)\) is a well-known transition alumina with a cubic spinel structure with lattice parameters of 0.7924 nm. \(^{(18)}\) The nanosize \(\gamma\)-alumina used in this experiment was commercially available (Nanophase Technologies Corporation). The average Particle Size was 37nm, Specific Surface Area (SSA), which measures the surface area in square meters per gram of material, was 30-60 \(\text{m}^2/\text{g}\). The material was 99.5+\% pure. The nanosize \(\gamma\)-alumina color was white to off white. Table 4 \(^{(19)}\) represents properties of nanosize \(\gamma\)-aluminum oxide \(^{(18, 19, 20)}\)

Table 4

| Properties of Nanosize \(\gamma\)-Aluminum Oxide \(^{(19)}\) |
|-----------------|-----------------|-----------------|-----------------|
| **Young’s Modulus** | **Poisson’s Ratio** | **True Density** | **Bulk Density** |
| (GPa) | | \(\text{g/cm}^3\) | \(\text{g/cm}^3\) |
| 253\(\pm\)22 | 0.24\(\pm\)0.2 | 0.10 | 3.6 |

Fabrication

Physical Vapor Synthesis (PVS) process is used to fabricate Gamma-aluminum oxide. In this particular process, plasma is used to heat the precursor of metal. As a result, the metal atoms boil off to create a vapor. Then, a gas is introduced to cool the vapor. The result of this process is the vapor condensing into liquid molecular clusters. The molecular clusters are then frozen into solid nanoparticles as the cooling process continues. The metal atoms are mixed with oxygen atoms, which forms aluminum oxide. Figure 3 illustrates PVS fabrication method. \(^{(19)}\)
The process of depositing a coating having a desirable form by means of electrolysis is known as electroplating. Generally, its purpose is to alter the characteristics of surface to provide improve appearance, ability to withstand corrosive agents, resistance to abrasion, or other desired properties.\textsuperscript{(21)}

In electroplating, the part or semifinished products (sheets, strip, wire) is immersed into aqueous electrolytes whose principal components are salts or other soluble compounds of the plated metal. The items to be coated are connected to the negative pole of a d.c. source; they form cathodes. The anodes usually consist of plate or rods of metal to be coated.\textsuperscript{(22)} They are connected to the positive pole of the d.c. source a shown in figure 4.
Faraday's laws govern this process. They are stated as follows:

1. The amount of chemical change produced by an electric current is proportional to the quantity of electricity that passes.

2. The amounts of different substances liberated by a given quantity of electricity are proportional to their chemical equivalent weights. \(^{(21)}\)

The deposition of metals on the cathode is a crystallization process and occurs in two stages: 1- formation of crystal nuclei, and 2- growth of crystallization centers. Both of these processes proceed at a certain rate which is dependent upon the type of electrolyte, on the electrolysis conditions (temperature, current density, agitation), on the kind of metal being deposited, and on the solvent. \(^{(23, 24, 25)}\) Hunt \(^{(23)}\) explains the crystallization mechanism as follow: the metal ions reaching the surface of the cathode tend to enter the crystal lattice quickly; they must, therefore, free themselves from the ionic atmosphere and water of hydration. It is assumed that metal ions capable of
entering the crystal lattice partly retain the ionic atmosphere and water of hydration. The more rapid the growth of the metal layer and the more quickly an ion becomes covered, the smaller are the chances of these foreign substances returning into the solution. The term electrocrystallization denotes the mechanism by which this deposit is formed. Frederick (25), on the other hand, explains this process as follow: the total energy required for the total process, changing metal ion in solution to metal atom in the lattice, is made up for several components. A metal ion probably reacts first with the cathode surface at some point on the flat area of a growing crystal; here it becomes absorbed in a state intermediate between the ionic state in solution and the state existing in the metal lattice. This state is called an adion. This adion moves over the surface by diffusion to a growth site, where it is integrated into the growing lattice. The initial growth is thus sideward, producing a monolayer (a layer one atom thick); but at some point in the process this sideward growth stops and a new layer is formed over the last one. Continuous of the process creates a crystallite or grain.(25)

Taguchi Method

The factorial design of experiments is the method that uses the technique of laying out the condition of experiments involving multiple factors. A full-factorial design will identify all possible combinations for a given set of factors. Since most industrial experiments usually involve a significant numbers of factors, a full-factorial design results in a large number of experiments. To reduce the number of experiments to a practical level, only a small number from all the possibilities is selected. The selection method of limiting the number of experiments that produces the most information is known as a partial fraction experiment. Even though this method well knows, there are no general guidelines for its application or the analysis of the results.
gained from performing the experiments. Taguchi constructed a special set of general design guidelines for factorial experiments that cover many applications. Taguchi method uses a special set of array known as orthogonal array. These standard arrays stipulate the way of conducting the minimal number of experiments, which could give an informative look at all the factors that affect the performance parameter. The crux of the orthogonal array method lies in choosing the level combinations of the input design variables for each experiment.\(^{26,27}\)

While there are numerous customary orthogonal arrays available, each of arrays is meant for a definite number of independent design variables and levels. For instance, if one wants to perform an experiment to understand the influence of four various independent variables with each variable having 3 levels, then an L9 orthogonal array could be the best choice as demonstrated in table 5.\(^{26}\) The L9 orthogonal array is

Table 5

<table>
<thead>
<tr>
<th>RUN</th>
<th>VARIABLES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
</tr>
<tr>
<td>1</td>
<td>+</td>
</tr>
<tr>
<td>2</td>
<td>+</td>
</tr>
<tr>
<td>3</td>
<td>+</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>-</td>
</tr>
</tbody>
</table>
Purposed for understand the effect of 4 independent factors each having 3 factor level values. (28) This array assumes that there is no interaction between any two factors. (29) While in many cases, no interaction model assumption is valid, there are some cases where there is a clear evidence of interaction.

The orthogonal array has the subsequent properties that diminish the number of experiments to be performed.

1. The vertical column under each independent variables of the table above has a special combination of level setting. All the level settings appear an equal number of times. For L9 array under variable 4, level +, level 0, and level - appear three times. This is called the balancing property of orthogonal arrays.

2. All the level variables of independent variables are used for experiment performance.

3. The level value sequences for conducting the experiments will not change. This means one cannot perform experiment 1 with variable 1, level 0 setup and experiment 4 with variable 1, level + setup. The reason for that the array of each factor columns is equally orthogonal to any other column of level values. (26,29)
CHAPTER II

EXPERIMENTAL APPARATUSES AND SOLUTIONS

Apparatus

An apparatus was designed to achieve the maximum quality of the coating. The apparatus was modified and designed by the people involved in the project. The apparatuses, which were used in this project, were ordered from an assortment of vendors, appendix A.

Holding tanks

A polypropylene cubic tank of 304.8x304.8x304.8x12.7 mm (LC Fabricators, Inc) was used in order to guarantee that the specimens would be completely sunken in the electrolytic solution. The volume capacity of the tank was 18 liters. Due to its properties, high melting temperature, 176°C and excellent chemical resistance \(^{(30)}\), polypropylene was used in the coating process since the coating bath temperature was 55±3°C.

Agitator

To ensure uniform particle distribution in the coating bath and to prevent agglomeration, a special setup of an agitator, figure 5, was designed and used to agitate the electrolytic solution. \(^{(31,32)}\) Chlorinated Polyvinyl Chloride CPVC tubes were used as a Bubblator. CPVC was used due to its properties of high melting temperature, and high transition glassy temperature, 115-135°C. \(^{(33)}\)
In order to achieve maximum efficiency of performance, bubbles were provided by pressurized air through 2.5 mm diameter holes, which were drilled in four sides of the bubblator. Each side had nine holes with 26.5 mm length between any two adjacent holes. To ensure cleanliness of the air, it was filtered through air filter via air tube (True Value). Using airflow regulator (Grainger) controlled the air pressure. Figure 6 illustrates the setup of this process.
**Immersion Heaters**

To approach the accurate condition of precoating solution temperatures, heat was provided to alkaline degreaser and alkaline etch by VYCOR immersion heater whose power is 250 watt (VWR Company) with 139.7 mm heated length, 355.6 mm overall length, and 9 mm overall diameter. Immersion heater, figure 7, is ideal for heating acid and plating solutions. Immersion heater is enclosed in a red VYCOR sheath that completely shields the heating section. Corrosion resistance of the jacket ensures freedom from chemical and ion contamination. In order to obtain the maximum efficiency of heating, only glass portion should be immersed.

![Immersion Heater](image)

**Electrolytic Solution Heater**

It is well known that the structure of electrodeposited materials is related to the plating variables such as current density and temperature. To ensure a constant electroplating temperature, Teflon heater (LC Fabricator, Inc) with max power of 1 KW 120 VCR equipped with over temperature protection and a digital temperature controller provided and adjusted electrolytic solution heat. Figure 8 illustrates the Teflon heater and
Figure 8. Plating Solution Heater.

**Anode**

A Nickel Oxide Depolarized anode having an oval section with two diameters, 31.75x82.55mm, and 177mm long (MacDermid) attached to a copper titanium anode, figure 9, was used as an anode in this experiment. Depolarized anode whose coefficient is 100% is made from nickel of very high purity with the addition of less than 1 per cent of green nickel oxide. Therefore, the microstructure of the anode is countless of fine crystal, each separated at its boundary walls by a grain of green nickel oxide. Consequently, solubility of the nickel is assured without introduction of any wicked ingredients. Due to having warm and agitated solution, anode bags (Plating Products Inc) were used to cover the anode to prevent any particles from the surface of anode being transferred to the surface of substrate and therefore producing roughness in the deposit. \(^{(36, 37)}\)
Aluminum Substrate

Rolled sheet of Al 2024-T3 (Schupan Aluminum) with thickness of 0.635 mm was used in this experiment as a substrate for the electroplating and electrodeposition processes. A rectangular specimen and a tensile specimen, see figures 10 and 27 in chapter III, whose area was 2903.22 mm² for each to guarantee alike coating conditions were used in this study.

Power Supply

A DC power supply (L.C Fabricator, Inc) whose model is 1TSL-1C012-111 was used in this study. The power supply converts the alternating current 110-volt, 15amp grounded outlet into direct current that is essential to the plating process in this study since it is a steady-state current that permits for coating with a 12 volt, 100amp maximum. The power supply is equipped with automatic voltage stabilization with current limiting ±1% control and automatic constant current ±1% with voltage limiting and automatic crossover.
Experiment Solutions

Nickel Sulfamate

Due to low internal stress of deposits, high current density, excellent for electroforming, superior throwing power that is the relationship between the current density and uniformity of coating thickness, and simplicity, the sulfamate nickel solution is the most common nickel plating solution used for functional industrial applications. The formula of the sulfamate nickel solution is Ni(SO₃NH₂)₂. Among the nickel plating solution, the sulfamate bath is a general-purpose bath that yields deposits of low internal stress which is a result of a low current density treatment. Due to the very high solubility of nickel sulfamate, maintaining a higher nickel metal concentration in this solution than in other nickel baths is possible, permitting the use of lower operating temperature and higher plating rate. To minimize anode passivity, a small amount of nickel chloride is usually added to the bath. Nickel sulfamate is the nickel salt of sulfamic acid. The nickel sulfamate, which was used in this study, was purchased dissolved from MacDermid. Compositions and operation conditions of sulfamate bath are shown in table 6.

Table 6
Compositions and Operation Conditions of Sulfamate Bath (37, 40)

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel chloride, NiCl₂6H₂O</td>
<td>0-30 g/l</td>
</tr>
<tr>
<td>Nickel sulfamate, Ni (SO₃NH₂)₂</td>
<td>263-450 g/l</td>
</tr>
<tr>
<td>Total nickel as metal</td>
<td>62-113 g/l</td>
</tr>
<tr>
<td>Boric acid, H₃BO₃</td>
<td>30-45 g/l</td>
</tr>
<tr>
<td>pH</td>
<td>3-5</td>
</tr>
<tr>
<td>Temperature</td>
<td>38-60 °C</td>
</tr>
<tr>
<td>Current density</td>
<td>2.5-30 A/dm²</td>
</tr>
</tbody>
</table>
A principle function of the chloride ion is to improve anode dissolution by reducing polarization. It also increases the conductivity of the bath and has marked effects at the cathode. It increases the throwing power as a result of increasing cathode efficiency. Boric acid serves as a weak buffer in a nickel solution. Its principal effect is that of controlling the pH in the cathode film. In the absence of a buffer, nickel deposits at ordinary temperatures tend to be hard, cracked, and pitted.\(^{(40)}\)

Aluminum alloys have special problems with regard to electroplating; aluminum is a very active metal because its negative electrode potential causes a natural, impervious oxide film which must be removed before plating. This film forms rapidly, and it is not sufficient merely to remove it; it must be replaced by a more receptive film for satisfactory plating. Pure aluminum is rarely used, and the various aluminum alloys have various microstructures that cannot all be treated in a similar way. Common alloying elements include silicon, copper, manganese, and magnesium, and many others may be encountered. Since the various alloys and products of aluminum behave differently electrochemically due to their different composition and metallurgical structures, it is complex to find a preplating procedure that is similarly acceptable for all types and tempers of aluminum alloys. To obtain consistent results for electroplating on aluminum in this study, it is essential that various cleaning and conditioning treatments provide a surface of uniform activity for the deposition of the initial metallic layer.\(^{(41)}\) The next solutions were used as preplating treatment solutions. Their composition and operation conditions are explained in the next chapter.
Alkaline Degreaser

Disodium trioxosilicate and de-ionized water constituted the alkaline degreaser solution (MacDermid). Its process was to remove the organic contaminants from the surface of the aluminum substrate. Alkaline degreaser, solvent cleaning, is one of the most commonly used techniques due to its simplicity, ease of operation, and ability to clean a variety of organic impurities such as oil, grease, metal chips, and hydrocarbons.\(^\text{[36, 42, 43]}\) Alkaline degreaser is in a bath at 55±3°C. The efficiency of an alkaline degreaser depends on the nature of the contaminations. Alkaline degreaser can remove only those impurities which are soluble in this solution. Alkaline degreaser causes several hazards due to pollution caused by usage and discharge of the chemicals. A proper exhaust ventilation and respiratory protection system were essential when alkaline degreaser was used.\(^\text{[43]}\)

Alkaline Etching

Sodium hydroxide (NaOH) (Chemistry Department, Western Michigan University) and de-ionized water constituted the alkaline etch. Alkaline etch reduces or eliminates surface scratches, nicks, extrusion-die lines, and other imperfections. Sodium hydroxide (NaOH) is the alkaline most commonly used. The surface must also be chemically clean; this is accomplished by using an alkaline etching that does not remove a significant amount of metal. Its reaction with aluminum is exothermic, produces hydrogen gas and sodium aluminate, and cause a rise in the temperature of the bath, depending on the relationship between rate of metal removal and tank volume.\(^\text{[36, 42, 43]}\) Alkaline etching is done in a bath at 55±3°C. The technique adopted for alkaline etching was immersion the substrate and rotated it slowly.
Acid Etching

De-ionized water, nitric acid HNO₃, and hydrochloric HCl constituted the acid etching solution (Chemistry Department, Western Michigan University). Acid etching is a process used to remove thin oxide films with a minimal amount of metal loss, oil, grease, and to roughen the substrate surface to increase the adhesion by dissolving them in a solution of mineral acid and organic acid with a detergent or surfactant. This process can leave the aluminum surface covered with undissolved alloying elements. Acid etching is mainly employed as a final or near final preparation of the substrate surface before coating. This process is used in combination with other cleaning process to achieve the desirable quality of the surface. The technique adopted for acid etching was immersing the substrate and rotating it slowly. The hazards in acid etching are exposure to spray/mist or acid gas; therefore, a proper exhaust ventilation, goggles, face shields, impervious gloves, and respiratory protection system were used whenever acid etching was used.

Zincate

It is difficult to plate other metal on aluminum since it is covered by a strongly adherent film of oxide which is highly resistive and which prevents an electrodeposition from sticking to the surface. Nickel plating of aluminum has involved an intermediate deposition step prior to electroplating. The purpose of intermediate step is to remove the surface oxide and replace it with a thin metallic film that is either resistant to oxidation or has an oxide that is easily reducible in subsequent steps. The intermediate step consists of immersion into an alkaline zincate. The basic solution contains dissolved zinc in an excess of sodium hydroxide. The standard immersion baths contain between 400 to 550 gram/liter sodium hydroxide and 40 to 100 gram/liter zinc oxide with 1 gram/liter ferric chloride and 10 gram/liter Rochelle salt (Potassium hydrogen tartrate). The goal of
The zincate process is to produce a thin zinc coating on the aluminum. Zinc exists in the +2 valence state, while aluminum exists in the +3 valence state. Theoretically, if only zinc and aluminum were participating in the electrochemical reaction, three zinc atoms would deposit for every two aluminum atoms dissolved. The molar ratio of zinc deposited to aluminum dissolved would therefore be 3:2. The exchange process occurring during the zincate treatment of aluminum, can be represented by the following general equation.

\[
3Na_2Zn(OH)_4 + 2Al = 2NaAlO_4 + 3Zn + 4Na(OH) + 4H_2O
\]

The above equation summarizes the partial reactions, which are proceeding simultaneously on the anodic and cathodic regions of the aluminum surface.

\[
Al + 3OH^- = Al(OH)_3 + 3e^-
\]

\[
Al(OH)_3 = AlO_2 + H_2O + H^+
\]

\[
Zn(OH)_4^{2-} = Zn^{2+} + 4OH^-
\]

\[
Zn^{2+} + 2e^- = Zn
\]

\[
H^+ (H_2O) + e^- = \frac{1}{2} H_2
\]

Double zincating technique is used to improve the performance of the pretreatment process. The first zincate dip removes the oxide layer and replaces it with a zinc film. This zinc film is then dissolved in acid etching; the surface is then in good condition to receiving the second zinc-immersion film. Deposition of the final zinc-immersion film is carried out in a highly alkaline solution of sodium zincate, prepared by dissolving zinc oxide in sodium hydroxide (MacDermid). For best performance the zinc
film should be as thin as possible consistent with satisfactory subsequent electroplating. Rochelle salt and ferric chloride are added to the zinc-immersion bath to give more uniform coverage in the ensuing electroplating operations. Zincate process is carried out in Pyrex bath at room temperature. As mentioned, aluminum is more active than any metal, which can be plated on it; for this reason, once a break develops in the coating, corrosion may be very rapid. In addition, the zinc film is anodic to aluminum and therefore tends to protect both the substrate and the electrodeposited.

Table 7 illustrates the composition of modified zincate.

Table 7
Composition of Modified Zincate

| Constituent                                      | Concentration, g/|l |
|------------------------------------------------|------------------|
| Sodium hydroxide (NaOH)                        | 525              |
| Zinc oxide (ZnO)                               | 100              |
| Sodium potassium tartrate, Rochelle salt (KNaC₄H₄O₆·4H₂O) | 10               |
| Ferric chloride (FeCl₃·6H₂O)                   | 1                |

Four polypropylene dip tanks (VWR) with 457 mm length, 457 mm depth, and 100 mm width were used as waste disposal tanks to contain the consumed solutions.
CHAPTER III

EXPERIMENTAL DETAILS

Experimental Procedure

Electroplating and electro-codeposition were used in this experiment. Best levels of the significant factors were obtained by electroplating nickel on a substrate of Al 2024-T3. Electrolytic co-deposition process was used to form the layer of nickel and with second phase γ-alumina particles on the substrate. The independent factors, which were used to obtain the desired layer thickness, were current density, time, and agitation. (32, 36, 49, 50)

Two stages of electroplating process took place to ensure the maximum quality of coating. Eighteen specimens were coated in the first stage, and the coating thickness measurements were analyzed by using Taguchi method. The electroplating-stage one results of the coating thickness measurements were verified by applying them in the electroplating-stage two process.

Due to having just three factors, L9 standard orthogonal array was chanced to fractional orthogonal array in order to investigate the effect of the three independent factors on the coating thickness. Table 8 illustrates the fractional orthogonal array design.
Table 8

L9 Fractional Orthogonal Array

<table>
<thead>
<tr>
<th>Run</th>
<th>Current Density (A/dm²)</th>
<th>Time (Min)</th>
<th>Agitation (m³/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-1</td>
<td>-1</td>
<td>-1</td>
</tr>
<tr>
<td>2</td>
<td>-1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>-1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
<td>-1</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>1</td>
<td>-1</td>
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<td>7</td>
<td>1</td>
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<tr>
<td>9</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 9 presents the three levels of each factor.

Table 9

Factor Levels

<table>
<thead>
<tr>
<th>Factor</th>
<th>Level</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-1</td>
<td>0</td>
<td>+1</td>
</tr>
<tr>
<td>Agitation (m³/h)</td>
<td>1.70</td>
<td>2.26</td>
<td>2.80</td>
</tr>
<tr>
<td>Current Density (A/dm²)</td>
<td>13.77</td>
<td>20.66</td>
<td>27.55</td>
</tr>
<tr>
<td>Time (min)</td>
<td>10</td>
<td>15</td>
<td>20</td>
</tr>
</tbody>
</table>
The rectangular specimens, which were 0.635mm thick, were sheared using standard metal shop shearing machine at the engineering workshop at Western Michigan University. The specimens were cut parallel to the rolling direction in a rectangular shape with dimensions 25.40mm x 152.40mm as shown in figure 10. The cutting process was done without scratching the surface of specimens, which would influence of the coating adhesions.

![Figure 10. Rectangular Specimen Dimensions.](image)

The procedure by which all the specimens were electroplated and electrodeposited was illustrated in figure 11. Using the variables that are shown in table 9 eighteen rectangular specimens (1-18) were electroplated in the first stage of electroplating process. Based on the results of the first stage of electroplating, six rectangular specimens (19-24) were electroplated in the second stage of electroplating. Then using figure 11 electrodeposited thirty-two specimens (25-65), which have two different geometry shapes.
Figure 11. Electroplating and Electrocodeposition Process.
Surface-Preparation Processes

Metal surface preparation is important for any coating process. The surface preparation is the removal of contamination or unwanted impurities such as soil from the surface.\(^{(42)}\) Surface cleaning is required for a strong interface between the coating layer and the substrate material.\(^{(36,42,51)}\)

Chemical etching technique was used to prepare a substrate surface in this experiment.\(^{(43)}\) Surface preparation processes, which were used, could be described as follow:

1. Alkaline Degreaser. Disodium trioxosilicate and de-ionized water constituted the alkaline degreaser which a 4000 ml Pyrex beaker was used to contain. The solution was prepared by adding 60 gram of disodium trioxosilicate to one liter of de-ionized water. Then the solution, figure 12, was heated to 55±3°C by using

![Figure 12. Alkaline Degreaser with an Immersion Heater.](image-url)
immersion heater. 300mm long glass stirring rod was used to stirring the solution. The total concentration of this solution was 240g of disodium trioxosilicate, and 4000-ml de-ionized water. A substrate was immersed and rotated very slowly for 8 minutes.

2. Alkaline Etch. Sodium hydroxide (NaOH) and de-ionized water constituted the alkaline etch. A 4000 ml Pyrex beaker was used to contain this solution. The solution was prepared by adding 60 gram sodium hydroxide of to one liter of de-ionized water. The solution, then, was heated to 55±3°C by using immersion heater as shown in figure 12. And the solution was stirred simultaneously. The purpose of alkaline degreaser is to reduce or eliminate surface scratches, nicks, and other imperfections. The substrate was immersed and rotated very slowly to ensure that the surface etching would be uniformly done. The substrate was immersed in alkaline solution for three minutes.

3. Acid Etch. De-ionized water, nitric acid HNO₃, and hydrochloric HCl constituted the acid etching solution. A 4000ml a Pyrex beaker was used to contain the solution. It was a concentration of 25% HNO₃, 25%HCl, and 50% de-ionized water. The total concentration was 1000-ml HNO₃, 1000-ml HCl, and 2000-ml de-ionized water. Adding the HNO₃ to de-ionized water, and adding the HCl to the solution prepared the acid solution. The solution was used at room temperature. The aim of this process was to remove oxide layer. The substrate was immersed and rotated very slowly to ensure that the acid etching process cleaned all the surfaces of the substrate for three and half minutes.

4. Zincate. Caustic soda (Metex 6811) and de-ionized water constituted the
zincate. 4000ml a Pyrex beaker was used to contain the solution. It was a concentration of 720g of caustic soda (Metax 6811) and 3000-ml of de-ionized water. The solution was used at room temperature. The aim of this process was to form thin adherent film of zinc. The substrate was immersed and rotated very slowly in order to have a uniform. The immersion and rotation time was one minute and half.

Double zincating technique was used to improve the performance of the pretreatment process.\(^{44, 47}\) Therefore, the acid etching and zincate processes were repeated as shown in figure 11. The duration of the repeating processes was one minute and a half for acid-etch process and twenty seconds for the zincate process.

Aluminum is reactive, and oxides form very rapidly on its surface during exposure to air. Oxide films that develop prevent metallic bonds from forming between the coating and the substrate. To avoid this problem, the aluminum substrate was flushed directly with de-ionized water after each previous process and before moving on to the next step of the pretreatment processes.\(^{44, 52}\) The aluminum substrate was dried by using hair drier in order to diminish the duration of the pretreatment processes and to avoid the presence of the water on the substrate surface, which would effect on the efficiency of the next process.

Table 10 summarizes the pretreatment processes and presents the composition and the time of each step for the first stage of electroplating.
### Table 10

Electroplating Procedure for the First Stage

<table>
<thead>
<tr>
<th>Bath (Pretreatment steps)</th>
<th>Time (sec)</th>
<th>Composition</th>
<th>Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkaline Degreaser</td>
<td>480</td>
<td>Disodium trioxosilicate, 60g/l</td>
<td>55±3</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>Alkaline Etch</td>
<td>180</td>
<td>Sodium hydroxide (NaOH) 60g/l</td>
<td>55±3</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>Acid Etch</td>
<td>210</td>
<td>50% H₂O, 25%HNO₃, 25%HCl</td>
<td>27</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>Zincate</td>
<td>90</td>
<td>Metex 6811, 240g/l</td>
<td>27</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>Acid Etch</td>
<td>90</td>
<td>50% H₂O, 25%HNO₃, 25%HCl</td>
<td>27</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>Zincate</td>
<td>20</td>
<td>Metex 6811, 240g/l</td>
<td>27</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
<td></td>
<td>27</td>
</tr>
</tbody>
</table>

Figure 13 illustrates the procedure setup of pretreatment processes.
Figure 13. Pretreatment Equipments. 1, 2, 3, and 4 indicate the alkaline degreaser, alkaline etch, acid etch, and zincate respectively.

**Electroplating-Stage One**

Barret nickel sulfamate \([\text{Ni}(\text{SO}_3\text{NH}_2)_2.4\text{H}_2\text{O}]\) solution (MacDermid, Inc.) was used as the electrolytic medium. Eighteen liters of Barret nickel sulfamate solution was used in 304.8mm length x 304.8mm width x 304.8mm height polypropylene tank (VWR). Table 8 presented the electroplating factors and their levels that were used in this experiment. To diminish the percentage of error of the experiment, the sequence of electroplating condition runs, which is shown in table 8, was randomly done.\(^{(26, 28)}\)

A one-substrate surface was numbered to identify the sample and then taped before the electroplating process. Eighteen samples were electroplated in the first stage. Using a run in table 8 electroplated every two samples. Operation conditions of the electrolytic solution are shown in table 11.
Table 11
Electrolytic Solution Operation Conditions

<table>
<thead>
<tr>
<th>Factor</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>55 ± 3 °C</td>
</tr>
<tr>
<td>PH</td>
<td>3.6-4.4</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>29.0-31.0</td>
</tr>
<tr>
<td>Cathode Efficiency</td>
<td>95-100%</td>
</tr>
<tr>
<td>Anode Efficiency</td>
<td>100%</td>
</tr>
</tbody>
</table>

Electroplating bath setup can be shown in figure 14. The specimen was held
by a jumper clamp and then immersed in the electroplating bath; the power was turned on simultaneously. After a specimen was coated, the power was turned off. The sample was removed and immediately flushed with de-ionized water.

Results and Discussion

The coated specimens were divided into three sections: A, B, and C as shown in figure 15. The front, mid, and rear of each section was measured after the sample was mounted as shown in figure 16. An adjacent steel strip was used in order to protect the coating thickness layer from rounding during the polishing, which would have effected on the accuracy of coating thickness. Coating thickness was measured by using a graded optical microscope in material science laboratory at Western Michigan University.

Figure 15. Division of the Coated Specimen.

Figure 16. Section Division of Coated Specimen.
Table 12 presents the coating thickness measurement of the first stage of the electroplating processes. The dimension of the coating thickness values is micrometer.

Table 12

Coating Thickness Measurements, µm

<table>
<thead>
<tr>
<th>Run</th>
<th>Sample</th>
<th>Section A</th>
<th>Section B</th>
<th>Section C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Front</td>
<td>Mid</td>
<td>Rear</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>9.20</td>
<td>10.35</td>
<td>10.73</td>
</tr>
<tr>
<td>3</td>
<td>16.48</td>
<td>16.10</td>
<td>15.53</td>
<td>17.63</td>
</tr>
<tr>
<td>5</td>
<td>15.53</td>
<td>14.57</td>
<td>16.48</td>
<td>16.87</td>
</tr>
<tr>
<td>7</td>
<td>15.33</td>
<td>13.03</td>
<td>14.18</td>
<td>15.14</td>
</tr>
<tr>
<td>8</td>
<td>17.25</td>
<td>15.53</td>
<td>15.91</td>
<td>15.33</td>
</tr>
<tr>
<td>9</td>
<td>28.94</td>
<td>27.60</td>
<td>26.45</td>
<td>29.52</td>
</tr>
<tr>
<td>10</td>
<td>32.20</td>
<td>30.48</td>
<td>27.79</td>
<td>27.60</td>
</tr>
<tr>
<td>11</td>
<td>41.21</td>
<td>38.34</td>
<td>37.95</td>
<td>40.25</td>
</tr>
<tr>
<td>12</td>
<td>44.09</td>
<td>46.00</td>
<td>38.34</td>
<td>47.92</td>
</tr>
<tr>
<td>14</td>
<td>23.77</td>
<td>22.04</td>
<td>24.53</td>
<td>24.15</td>
</tr>
<tr>
<td>15</td>
<td>30.28</td>
<td>33.74</td>
<td>37.57</td>
<td>34.50</td>
</tr>
<tr>
<td>16</td>
<td>27.60</td>
<td>26.64</td>
<td>26.45</td>
<td>27.03</td>
</tr>
<tr>
<td>17</td>
<td>40.44</td>
<td>37.57</td>
<td>46.49</td>
<td>46.00</td>
</tr>
<tr>
<td>18</td>
<td>48.88</td>
<td>26.45</td>
<td>35.65</td>
<td>45.04</td>
</tr>
</tbody>
</table>

Table 13 presents the mean thickness and the standard deviation values of the coated samples in the stage one. The measurement unit is micrometer.
Coating thickness measurement was analyzed by using Minitab™ program, appendix B. The aim of this process was to determine which factor has the most significant effect on the coating thickness and at which level the factor should be set. Figures 17 and 18 illustrate the factor effects dot-plot for the coating thickness and standard deviation, respectively. Figures 19 and 20 present the factor effects plot for the mean thickness and standard deviation, respectively. Figures 21 and 22 illustrate

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Mean (µm)</th>
<th>Standard Deviation (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.60</td>
<td>0.78</td>
</tr>
<tr>
<td>2</td>
<td>10.40</td>
<td>1.20</td>
</tr>
<tr>
<td>3</td>
<td>15.60</td>
<td>2.30</td>
</tr>
<tr>
<td>4</td>
<td>14.10</td>
<td>2.90</td>
</tr>
<tr>
<td>5</td>
<td>15.40</td>
<td>1.20</td>
</tr>
<tr>
<td>6</td>
<td>18.55</td>
<td>1.20</td>
</tr>
<tr>
<td>7</td>
<td>15.65</td>
<td>1.80</td>
</tr>
<tr>
<td>8</td>
<td>15.4</td>
<td>0.90</td>
</tr>
<tr>
<td>9</td>
<td>29.85</td>
<td>2.00</td>
</tr>
<tr>
<td>10</td>
<td>27.50</td>
<td>2.50</td>
</tr>
<tr>
<td>11</td>
<td>42.60</td>
<td>4.20</td>
</tr>
<tr>
<td>12</td>
<td>40.70</td>
<td>5.80</td>
</tr>
<tr>
<td>13</td>
<td>21.45</td>
<td>1.50</td>
</tr>
<tr>
<td>14</td>
<td>23.15</td>
<td>1.50</td>
</tr>
<tr>
<td>15</td>
<td>36.10</td>
<td>4.15</td>
</tr>
<tr>
<td>16</td>
<td>27.75</td>
<td>1.50</td>
</tr>
<tr>
<td>17</td>
<td>48.95</td>
<td>7.80</td>
</tr>
<tr>
<td>18</td>
<td>43.70</td>
<td>8.00</td>
</tr>
</tbody>
</table>
the half-normal plot for mean thickness and standard deviation, respectively.

Figure 17. Factor Effects Dot-plot for Coating Thickness.

Figure 18. Factor Effects Dot-plot for Standard Deviation.

Figure 19. Factor Effects Plot for Mean Thickness.
Figure 20. Factor Effect Plot for Standard Deviation.

Figure 21. Half-Normal Plot for Mean Thickness.
Figure 22. Half-Normal Plot for Standard Deviation.

Tables 14 and 15 illustrate the contribution of the factors in the coating thickness and standard deviation, respectively.

Table 14

<table>
<thead>
<tr>
<th>Factor</th>
<th>Contribution %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current Density</td>
<td>51.54</td>
</tr>
<tr>
<td>Time</td>
<td>45.07</td>
</tr>
<tr>
<td>Agitation</td>
<td>3.39</td>
</tr>
</tbody>
</table>
Based on figure 17, factor effects dot-plot, figure 19, factor effects plot for mean thickness, figure 21, half-normal plot for coating thickness, and table 14, contribution of the factors in the coating thickness, current density and time were the most significant factors in coating thickness. The best levels of the two factors, figure 19, were +1 and +1 for current density and time, respectively. Even though agitation factor was not significant comparing to current density and time, figure 19, agitation factor had to be set at -1 level since it was subsequently used in electrodeposition process.

Based on figure 18, factor effects dot-plot, figure 20, factor effects plot for standard deviation, figure 22, half-normal plot for standard deviation, and table 15, contribution of the factors in the standard deviation, time and current density were the most significant factors in the standard deviation. The best levels of the two factors, figure 20, were -1 and -1 for time and current density, respectively. Due to using in electrocodeposition process, agitation factor levels were taken into account although it was not a significant factor in coating thickness standard deviation. The best level of agitation factor was +1, figure 20.

In order to increase the coating thickness, as it can be seen in figure 19, the three factors, current density, time, and agitation, had to be set at +1, +1, and -1,
respectively. In order to increase the uniformity of the coating, the three factors, time, current density, and agitation, had to be set at -1, -1, and +1, respectively.

**Electroplating-Stage Two**

From previous results it was obvious that there was a conflict between best level setting of coating thickness and best level setting of coating thickness standard deviation. The aim of this process was to avoid this conflict and to verify the previous results. Therefore, the previous electroplating setup, figure 14, was changed as shown in figure 23.

Figure 23. The Setup of Electroplating-Stage Two.

Using the new setup and using the conditions, which are shown in table 16, three samples (19-21) were electroplated. The procedure of pretreatment processes, which was followed in this stage, was the same procedure, which is shown in table 10. But the time of each process was changed as shown in table 17.
Table 16

Coating Conditions of Electroplating-Stage Two

<table>
<thead>
<tr>
<th>Sample</th>
<th>Current Density</th>
<th>Time</th>
<th>Agitation</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>+1</td>
<td>+1</td>
<td>+1</td>
</tr>
<tr>
<td>20</td>
<td>0</td>
<td>+1</td>
<td>-1</td>
</tr>
<tr>
<td>21</td>
<td>+1</td>
<td>-1</td>
<td>+1</td>
</tr>
</tbody>
</table>

Table 17

Pretreatment Process Time of Electroplating-Stage Two

<table>
<thead>
<tr>
<th>Bath</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkaline Degreaser</td>
<td>300</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
<tr>
<td>Alkaline Etch</td>
<td>120</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
<tr>
<td>Acid Etch</td>
<td>150</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
<tr>
<td>Zincate</td>
<td>60</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
<tr>
<td>Acid Etch</td>
<td>60</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
<tr>
<td>Zincate</td>
<td>20</td>
</tr>
<tr>
<td>De-ionized H₂O rinse</td>
<td>30</td>
</tr>
</tbody>
</table>

Even though the best levels of current density for coating thickness and coating thickness standard deviation were +1 and -1, figures 19 and 20, respectively, 0 level was chosen in stage two instead of -1 level in order to increase the uniformity of the coating layer.
The electroplating-stage one setup contained two noticeable disadvantages: 1) There was a non-reproducible distance between the anode and the cathode, which resulted in a variable coating parameter for each trial. Variability in initial conditions ultimately resulted in unstable data, and no consistent pattern could be found. The variability in anode to cathode distance was attributed to the repeated removal and placement of samples. 2) The weight of the jumper cable used was also suspected of contributing to the slight misalignment of the sample, which resulted in non-uniform coating thickness, figure 14.

To correct the setup problems from stage one, chlorinated polyvinyl chloride (CPVC) tube was attached to the electrolytic solution bath, and used as the specimen holder. This corrected the problem of variable anode to cathode distance, by keeping a fixed distance. The use of a lighter, more flexible cable (smaller wire diameter), helped to control the position of the specimen within the electrolytic solution tank, figure 23.

Results and Discussion

Table 18 summarizes the coating thickness measurement of the specimens and the thickness unit is micrometer.

Table 18

<table>
<thead>
<tr>
<th>Sample</th>
<th>Section A</th>
<th>Section B</th>
<th>Section C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Front</td>
<td>Mid</td>
<td>Rear</td>
</tr>
<tr>
<td>19</td>
<td>59.39</td>
<td>59.9</td>
<td>60.9</td>
</tr>
<tr>
<td>20</td>
<td>48.8</td>
<td>48.2</td>
<td>48.26</td>
</tr>
<tr>
<td>21</td>
<td>36.9</td>
<td>36.08</td>
<td>37.54</td>
</tr>
</tbody>
</table>
Figures 24 and 25 illustrate the mean thickness and standard deviation of electroplated-stage two specimens.

Figure 24. Mean Thickness Plot for the Electroplated-Stage Two Specimens.

Figure 25. Standard Deviation Plot of Electroplated-Stage Two Specimens.
Table 19 presents the values of mean thickness and standard deviation of electroplated-stage two specimens.

Table 19

Mean Thickness and Standard Deviation of Electroplated-Stage Two Specimens

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Thickness (µm)</th>
<th>Standard Deviation (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>60.10</td>
<td>0.60</td>
</tr>
<tr>
<td>20</td>
<td>48.40</td>
<td>0.35</td>
</tr>
<tr>
<td>21</td>
<td>36.65</td>
<td>0.75</td>
</tr>
</tbody>
</table>

From table 19, and figure 24, the sample, which was electroplated by using high level of current density, 27.55A/dm², high level of time, 20 minutes, and high level of agitation, 2.8 m³/h, number 19, had the highest thickness. From table 19, and figure 25. The sample, which electroplated by using the mid level of current density, 20.66 A/dm², the high level of time, 20 minutes, and lowest level of agitation, 1.7 m³/h, number 20, had the highest coating uniformity.

Comparison the results of standard deviation which are shown in table 19 with the results of standard deviation which are shown in table 13 proved the effect of electroplating setup on the coating uniformity. For example, even though more time was used to electroplate sample number 20, its coating uniformity was higher than that of sample number 9.
To verify the results that were obtained in electroplating-stage two, three more specimen, 22-24, were electroplated.

Table 20 presents the coating thickness measurement of the specimens.

Table 20

Coating Thickness Measurement (µm)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Section A</th>
<th></th>
<th>Section B</th>
<th></th>
<th>Section C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Front</td>
<td>Mid</td>
<td>Rear</td>
<td>Front</td>
<td>Mid</td>
</tr>
<tr>
<td>22</td>
<td>58.65</td>
<td>59.34</td>
<td>59.8</td>
<td>58.84</td>
<td>58.46</td>
</tr>
<tr>
<td>23</td>
<td>48.07</td>
<td>48.96</td>
<td>48.92</td>
<td>48.15</td>
<td>48.92</td>
</tr>
<tr>
<td>24</td>
<td>37.46</td>
<td>36.28</td>
<td>37.5</td>
<td>36.46</td>
<td>38.34</td>
</tr>
</tbody>
</table>

Table 21 presents the mean thickness and standard deviation.

Table 21

Mean Thickness and Standard Deviation

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Thickness (µm)</th>
<th>Standard Deviation (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>59.30</td>
<td>0.60</td>
</tr>
<tr>
<td>23</td>
<td>48.70</td>
<td>0.35</td>
</tr>
<tr>
<td>24</td>
<td>37.40</td>
<td>0.70</td>
</tr>
</tbody>
</table>

Figures 26, and 27 present the plot of mean thickness and standard deviation, respectively.
Figure 26. Mean Thickness Plot of Electroplating-Stage Two Specimen.

Figure 27. Standard Deviation Plot of Electroplating-Stage Two Specimens.

From table 21, figures 26 and 27 specimens number 22 and 23 had highest mean thickness and highest coating uniformity, respectively. These results matched the results of the previous electroplating process.
Electrodeposition

Nanocrystalline Ni/γAl₂O₃ composites were formed by electrodeposition. The deposition was achieved by adding different amounts of γAl₂O₃ nanopowder having a medium particle diameter of 37 nm (Nanophase Technology Co) in a nickel sulfamate bath maintained at 55±3°C and pH values ranging from 3.6-4.0 followed by DC plating with current densities between 20.66 and 27.58 A/dm². The co-deposition was carried out on aluminum substrate. The bath was agitated by using compressed air passed through CPVC tubes, figure 5, in order to avoid agglomeration. The flow of the air, which was used to agitate the bath, was controlled by a flow meter and a filter to ensure the cleanness of the bath from the soil, figure 6.

For the sake of simplicity, the conditions of the coating in this study were named: low conditions and high conditions. Tables 22, and 23 present the definition of the low conditions and high conditions of the coating process respectively.

Table 22

<table>
<thead>
<tr>
<th>Current Density</th>
<th>20.66 A/dm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>20 min</td>
</tr>
<tr>
<td>Agitation</td>
<td>1.7 m³/h</td>
</tr>
</tbody>
</table>
Two types of specimens were prepared: a rectangular specimen and a tensile specimen. Tensile test dimensions were prepared as defined in American Society for Testing and Material Specification ASTM E8 \(^{(53)}\), figure 28. Rectangular specimen dimensions were calculated to match the area of the tensile specimen. Therefore, the area of the two specimens was 0.29 dm\(^2\) in order to ensure similar coating conditions.

**Table 23**

<table>
<thead>
<tr>
<th>Definition of High Conditions of Coating Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current Density</td>
</tr>
<tr>
<td>Time</td>
</tr>
<tr>
<td>Agitation</td>
</tr>
</tbody>
</table>

G- Gage length             50.0±0.1 mm  
W- Width                   12.5±0.2 mm  
T- Thickness               Thickness of material  
R- Radius of fillet         12.5 mm  
L- Over length              200 mm  
A- Length of reduced section 57 mm  
B- Length of grip section   50 mm  
C- Width of grip section    20 mm

Figure 28. Tensile Specimen Dimensions.

Source: ASTM E8 (Metal Test Methods and Analytical Procedure)
Table 24 summarizes the electrodeposition conditions, which were used to electrodeposit the specimens. The pretreatment procedure, which was used in electroplating-stage two, was used in this process. Due to increasing the coating uniformity in electroplating-stage two, a level of the time factor was changed from 10 minutes to 15 minutes to obtain the desired coating thickness.

Table 24

Electrodeposition Conditions

<table>
<thead>
<tr>
<th>Sample</th>
<th>Geometry</th>
<th>Current Density</th>
<th>Time</th>
<th>Agitation</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>A/dm²</td>
<td>Min</td>
<td>m³/h</td>
<td>g/l</td>
</tr>
<tr>
<td>25</td>
<td>Rectangular</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>26</td>
<td>Rectangular</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td></td>
</tr>
<tr>
<td>27-29</td>
<td>Tensile Test</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>30-32</td>
<td>Tensile Test</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>Rectangular</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>8</td>
</tr>
<tr>
<td>34-36</td>
<td>Tensile Test</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>8</td>
</tr>
<tr>
<td>37</td>
<td>Rectangular</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>8</td>
</tr>
<tr>
<td>38-40</td>
<td>Tensile Test</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>8</td>
</tr>
<tr>
<td>41</td>
<td>Rectangular</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>14</td>
</tr>
<tr>
<td>42-44</td>
<td>Tensile Test</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>14</td>
</tr>
<tr>
<td>45</td>
<td>Rectangular</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>14</td>
</tr>
<tr>
<td>46-48</td>
<td>Tensile Test</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>14</td>
</tr>
<tr>
<td>49</td>
<td>Rectangular</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>22</td>
</tr>
<tr>
<td>50-52</td>
<td>Tensile Test</td>
<td>27.55</td>
<td>15</td>
<td>2.8</td>
<td>22</td>
</tr>
<tr>
<td>53</td>
<td>Rectangular</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>22</td>
</tr>
<tr>
<td>54-56</td>
<td>Tensile Test</td>
<td>20.66</td>
<td>20</td>
<td>1.7</td>
<td>22</td>
</tr>
</tbody>
</table>
The same procedure, which was used in the previous process to measure the coating thickness, was also followed in this process. Table 25 presents the thickness of the substrate, coating thickness, and the standard deviation of coated tensile specimens.

Table 25

The Substrate Thickness, Coating Thickness, Standard Deviation of Coated Tensile Test Specimens

<table>
<thead>
<tr>
<th>No</th>
<th>S. T(mm)</th>
<th>C. T(mm)</th>
<th>St.D</th>
<th>No</th>
<th>S. T(mm)</th>
<th>C. T(mm)</th>
<th>St.D</th>
</tr>
</thead>
<tbody>
<tr>
<td>27</td>
<td>0.630</td>
<td>0.045</td>
<td>0.34</td>
<td>30</td>
<td>0.630</td>
<td>0.034</td>
<td>0.20</td>
</tr>
<tr>
<td>28</td>
<td>0.632</td>
<td>0.046</td>
<td>0.07</td>
<td>31</td>
<td>0.632</td>
<td>0.036</td>
<td>0.20</td>
</tr>
<tr>
<td>29</td>
<td>0.630</td>
<td>0.042</td>
<td>0.39</td>
<td>32</td>
<td>0.625</td>
<td>0.038</td>
<td>0.41</td>
</tr>
<tr>
<td>34</td>
<td>0.610</td>
<td>0.048</td>
<td>0.45</td>
<td>38</td>
<td>0.600</td>
<td>0.041</td>
<td>0.64</td>
</tr>
<tr>
<td>35</td>
<td>0.600</td>
<td>0.047</td>
<td>0.56</td>
<td>39</td>
<td>0.600</td>
<td>0.041</td>
<td>0.98</td>
</tr>
<tr>
<td>36</td>
<td>0.620</td>
<td>0.048</td>
<td>0.46</td>
<td>40</td>
<td>0.618</td>
<td>0.042</td>
<td>0.52</td>
</tr>
<tr>
<td>42</td>
<td>0.600</td>
<td>0.038</td>
<td>0.37</td>
<td>46</td>
<td>0.613</td>
<td>0.036</td>
<td>0.37</td>
</tr>
<tr>
<td>43</td>
<td>0.620</td>
<td>0.042</td>
<td>0.37</td>
<td>47</td>
<td>0.617</td>
<td>0.036</td>
<td>0.13</td>
</tr>
<tr>
<td>44</td>
<td>0.620</td>
<td>0.041</td>
<td>0.15</td>
<td>48</td>
<td>0.605</td>
<td>0.034</td>
<td>0.37</td>
</tr>
<tr>
<td>50</td>
<td>0.607</td>
<td>0.036</td>
<td>0.64</td>
<td>54</td>
<td>0.600</td>
<td>0.034</td>
<td>0.37</td>
</tr>
<tr>
<td>51</td>
<td>0.600</td>
<td>0.036</td>
<td>0.32</td>
<td>55</td>
<td>0.615</td>
<td>0.043</td>
<td>0.64</td>
</tr>
<tr>
<td>52</td>
<td>0.603</td>
<td>0.034</td>
<td>0.71</td>
<td>56</td>
<td>0.600</td>
<td>0.036</td>
<td>0.64</td>
</tr>
</tbody>
</table>

Where “No”, “S.T”, “C. T”, and “St.D” refer to specimen number, the
substrate thickness in mm, the coating thickness in mm, and the standard deviation respectively.

Preferred Orientations

Rotating anode X-ray diffractometer, Rigaku, was used to do the pole figures and standard 2-theta diffraction of the coated specimens at the University of Michigan. The continuous scan method was used to do pole figure XRD. The start tilt angle was 15° while the tilt stop angle was 90°, and the tilt angle step was 5°. Beta axis, the azimuth, or the rotation of the sample plane had been set to rotate, the azimuth speed, at 360°/min. The data were being taken, the azimuth step, every five degrees. The background angle was 48°.

Elastic Modulus

MTS 810 Servo-Hydraulic Test Frame machine, which is capable of 88960 N loads, was used to carry out the tensile test for the analysis of the Young’s modulus. MTS Basic Software Package was used to analyze the test data. The data were recorded on a computer disk, which is attached with this thesis. The continuous strain-control test was used in this experiment. An extensometer was used to measure the strain of the specimens. Strain was increased in increments 0.15% over 200 seconds until failure. As the strain was increased, the load acting on a specimen also increased.

Surface Topography

The WYKO Vertical Interference Microscope was used to determine the surface topography of the coated specimens at Western Michigan University. The
surface topography is the three-dimensional representation of the geometric surface irregularities. The size of the field of view was 1.9 x 2.5mm. Vertical Scanning Interferometry (VSI) mode was used to determine surface topography. Average roughness, $R_a$, which is the mean height as calculated over the entire measured array, and root mean square roughness, $R_q$, which is rms average of the measured height deviation taken within the evaluation length or area and measured from the mean linear surface, were determined by using:

$$R_a = \frac{1}{N} \sum_{i=1}^{N} |Z_i|$$

(3.1)

where $|Z|$ indicates the individual height values without regard to sign, and $N$ indicates the sum of the individual heights values. $R_a$ is used to describe the roughness of machined surfaces while $R_q$ is used to describe the finish of optical surface.

$$R_q = \sqrt{\frac{Z_1^2 + Z_2^2 + Z_3^2 + \cdots + Z_N^2}{N}}$$

(3.2)
CHAPTER IV

RESULTS AND DISCUSSION

Young’s Modulus

The slope of stress-strain curve which was obtained by least square method, equation 1-4, and coefficient of determination, $R^2$, equation 1-7, were calculated using Microsoft Excel® program as shown in figure 29, see appendix C for the rest of the specimens. Table 26 presents the slope of the stress-strain curve and the coefficient of determination of the coated specimens.

Figure 29. Slope and Coefficient of Determination of Sample Number 27.

Sample 27

$y = 636037x + 199.44$

$R^2 = 0.9993$
Table 26

Slope and Coefficient of Determination of the Coated Specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>Line Equation</th>
<th>$R^2$</th>
<th>Sample</th>
<th>Line Equation</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>27</td>
<td>$Y = 636037x + 199.44$</td>
<td>0.9993</td>
<td>30</td>
<td>$Y = 615559x + 171.58$</td>
<td>0.9995</td>
</tr>
<tr>
<td>28</td>
<td>$Y = 64667x + 183.34$</td>
<td>0.9991</td>
<td>31</td>
<td>$Y = 624565x + 180.98$</td>
<td>0.9994</td>
</tr>
<tr>
<td>29</td>
<td>$Y = 626625x + 137.95$</td>
<td>0.9994</td>
<td>32</td>
<td>$Y = 619529x + 263.4$</td>
<td>0.9995</td>
</tr>
<tr>
<td>34</td>
<td>$Y = 636631x + 226.78$</td>
<td>0.9991</td>
<td>38</td>
<td>$Y = 615161x + 214.84$</td>
<td>0.9993</td>
</tr>
<tr>
<td>35</td>
<td>$Y = 625721x + 204.23$</td>
<td>0.9994</td>
<td>39</td>
<td>$Y = 609545x + 241.11$</td>
<td>0.9993</td>
</tr>
<tr>
<td>36</td>
<td>$Y = 640959x + 264.7$</td>
<td>0.9994</td>
<td>40</td>
<td>$Y = 637679x + 187.07$</td>
<td>0.9991</td>
</tr>
<tr>
<td>42</td>
<td>$Y = 611156x + 185.32$</td>
<td>0.9993</td>
<td>46</td>
<td>$Y = 622017x + 182.84$</td>
<td>0.9995</td>
</tr>
<tr>
<td>43</td>
<td>$Y = 640113x + 151.64$</td>
<td>0.9994</td>
<td>47</td>
<td>$Y = 631959x + 242.03$</td>
<td>0.9990</td>
</tr>
<tr>
<td>44</td>
<td>$Y = 634221x + 176.79$</td>
<td>0.9995</td>
<td>48</td>
<td>$Y = 604898x + 179.76$</td>
<td>0.9996</td>
</tr>
<tr>
<td>50</td>
<td>$Y = 624578x + 244.73$</td>
<td>0.9991</td>
<td>54</td>
<td>$Y = 619562x + 226.23$</td>
<td>0.9994</td>
</tr>
<tr>
<td>51</td>
<td>$Y = 621511x + 256.77$</td>
<td>0.9991</td>
<td>55</td>
<td>$Y = 660308x + 193.67$</td>
<td>0.9992</td>
</tr>
<tr>
<td>52</td>
<td>$Y = 614849x + 198.29$</td>
<td>0.9995</td>
<td>56</td>
<td>$Y = 592658x + 177.39$</td>
<td>0.9996</td>
</tr>
</tbody>
</table>

The stresses of coated specimens were calculated by using the next equation.

$$
\sigma = \frac{F}{A}
$$

(4.1)

where the force, $\Delta F$, was obtained by applying two values of the strain, whose range was from 1.00E-3 to 4.00E-3 as shown in figure 29, in the slope equation for each
specimen. The area, $A$, was calculated by adding the substrate thickness and coating thickness from table 25 above and multiplying the sum by the standard width of tensile test specimen, figure 22, 12.5 ±0.2.

Young's modulus of the coated specimens, $E_{Al+coating}$, was calculated, as shown in table 27, by using Hooke's law, equation 1.1.

**Table 27**

Young's Modulus of Coated Specimens

<table>
<thead>
<tr>
<th>Sample #</th>
<th>High Conditions</th>
<th>Low Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$E$ (MPa)</td>
<td>$E$ (MPa)</td>
</tr>
<tr>
<td>27</td>
<td>74240</td>
<td>75365</td>
</tr>
<tr>
<td>28</td>
<td>75150</td>
<td>76140</td>
</tr>
<tr>
<td>29</td>
<td>73450</td>
<td>75575</td>
</tr>
<tr>
<td>34</td>
<td>76160</td>
<td>76485</td>
</tr>
<tr>
<td>35</td>
<td>76140</td>
<td>76950</td>
</tr>
<tr>
<td>36</td>
<td>75530</td>
<td>76100</td>
</tr>
</tbody>
</table>

In order to calculate the Young's modulus of the coating layer, the following equation was used.\(^{(5)}\)

$$E_{coating} = \frac{E_{Al+coating} \cdot t_{Al+coating} - E_{Al} \cdot t_{Al}}{t_{coating}}$$  \hspace{1cm} (4.2)

$E_{Al}$, which was measured by tensile test of three samples of Al 2024-T3, see attached disk, was measured to be 68 GPa.

Table 28 illustrates the Young's modulus of the coating layer.
Table 28

Young’s Modulus of the Coating Layer

<table>
<thead>
<tr>
<th>Sample #</th>
<th>E (MPa)</th>
<th>Sample #</th>
<th>E (MPa)</th>
<th>Sample #</th>
<th>E (MPa)</th>
<th>Sample #</th>
<th>E (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>High Conditions</td>
<td></td>
<td>Low Conditions</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>160200</td>
<td>42</td>
<td>188400</td>
<td>30</td>
<td>161300</td>
<td>46</td>
<td>201100</td>
</tr>
<tr>
<td>28</td>
<td>172300</td>
<td>43</td>
<td>194300</td>
<td>31</td>
<td>168600</td>
<td>47</td>
<td>213100</td>
</tr>
<tr>
<td>29</td>
<td>153600</td>
<td>44</td>
<td>188600</td>
<td>32</td>
<td>164300</td>
<td>48</td>
<td>190800</td>
</tr>
<tr>
<td>34</td>
<td>177800</td>
<td>50</td>
<td>217300</td>
<td>38</td>
<td>183300</td>
<td>54</td>
<td>235300</td>
</tr>
<tr>
<td>35</td>
<td>178100</td>
<td>51</td>
<td>223800</td>
<td>39</td>
<td>173900</td>
<td>55</td>
<td>236900</td>
</tr>
<tr>
<td>36</td>
<td>170500</td>
<td>52</td>
<td>218900</td>
<td>40</td>
<td>191600</td>
<td>56</td>
<td>160700</td>
</tr>
</tbody>
</table>

Since every three specimens were coated at the same conditions, the average and the standard deviation of Young’s modulus of nickel coating for coating layer was calculated as shown in table 29.

Table 29

The Average and Standard Deviation of Young’s Modulus for the Coating Layer

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Coating Condition</th>
<th>Particles Concentration</th>
<th>E coating MPa</th>
<th>St.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>27-29</td>
<td>High</td>
<td>-</td>
<td>162000</td>
<td>9500</td>
</tr>
<tr>
<td>30-32</td>
<td>Low</td>
<td>-</td>
<td>164700</td>
<td>3700</td>
</tr>
<tr>
<td>34-36</td>
<td>High</td>
<td>8 g/litter</td>
<td>175500</td>
<td>4280</td>
</tr>
<tr>
<td>38-40</td>
<td>Low</td>
<td>8 g/litter</td>
<td>182900</td>
<td>8800</td>
</tr>
<tr>
<td>42-44</td>
<td>High</td>
<td>14 g/litter</td>
<td>190400</td>
<td>3300</td>
</tr>
<tr>
<td>46-48</td>
<td>Low</td>
<td>14 g/litter</td>
<td>201600</td>
<td>1200</td>
</tr>
<tr>
<td>50-52</td>
<td>High</td>
<td>22 g/litter</td>
<td>220000</td>
<td>3400</td>
</tr>
<tr>
<td>54-56</td>
<td>Low</td>
<td>22 g/litter</td>
<td>236000</td>
<td>1150</td>
</tr>
</tbody>
</table>
These results can be illustrated graphically by figure 30.

![Young's Modulus Graph](image)

Figure 30. The Average of Young’s Modulus for Coating Layer.

**Pole Figures**

Pole figures for the coated specimens are presented as follow:

1. High conditions. The next eight figures, figures 31 to 38, present the (111) and (200) pole figures for the four coated specimens which were coated by using the high conditions. The 2-theta used for each plane was determined by running a standard 2-theta XRD first, appendix D.
Figure 31. (200) Pole Figure of Specimen Number 25.

Sample: 25-200  
21-Aug-00  12:26  
\( h = 2 \quad k = 0 \quad l = 0 \)  

R.D  
2-theta = 52°  

Plot Levels:

Figure 32. (111) Pole Figure of Specimen Number 25.

Sample: 25-111  
21-Aug-00  11:46  
\( h = 1 \quad k = 1 \quad l = 1 \)  

R.D  
2-theta = 38.54°  

Plot Levels:
Figure 33. (200) Pole Figure of Specimen Number 33.

Sample: 33-200
21-Aug-00  13:51
h = 2    k = 0    l = 0
2-theta = 52.00°

Plot Levels:

<table>
<thead>
<tr>
<th>Level</th>
<th>Plot</th>
</tr>
</thead>
<tbody>
<tr>
<td>25.0%</td>
<td>3960.75</td>
</tr>
<tr>
<td>50.0%</td>
<td>3842.85</td>
</tr>
<tr>
<td>75.0%</td>
<td>1100.25</td>
</tr>
<tr>
<td>91.7%</td>
<td>14522.75</td>
</tr>
</tbody>
</table>

Figure 34. (111) Pole Figure of Specimen Number 33.

Sample: 33-111
21-Aug-00  13:13
h = 1    k = 1    l = 1
2-theta = 44.75°

Plot Levels:

<table>
<thead>
<tr>
<th>Level</th>
<th>Plot</th>
</tr>
</thead>
<tbody>
<tr>
<td>25.0%</td>
<td>21550</td>
</tr>
<tr>
<td>50.0%</td>
<td>1944.17</td>
</tr>
<tr>
<td>75.0%</td>
<td>846.50</td>
</tr>
<tr>
<td>91.7%</td>
<td>790.47</td>
</tr>
</tbody>
</table>
Figure 35. (200) Pole Figure of Specimen Number 41.

Sample: 41-200
21-Aug-00 15:15
h = 2  k = 0  l = 0

2-theta = 52.00°

Plot Levels:

Figure 36. (111) Pole Figure of Specimen Number 41.

Sample: 41-111
21-Aug-00 14:38
h = 1  k = 1  l = 1

2-theta = 44.70°

Plot Levels:
Figure 37. (200) Pole Figure of Specimen Number 49.

Sample: 49-200
21-Aug-00 16:37
h = 2  k = 0  l = 0
2-theta = 52.10°
Plot Levels:

Figure 38. (111) Pole Figure of Specimen Number 49.

Sample: 49-111
21-Aug-00 16:01
h = 1  k = 1  l = 1
2-theta = 44.80°
Plot Levels:
2. Low conditions. The next eight figures, figures 39 to 46, present the (111) and (200) pole figures for the four coated specimens which were coated by using the low conditions.

Figure 39. (200) Pole Figure of Specimen Number 26.

Sample: 26-200  
24-Aug-00 18:37  
h = 2  k = 0  l = 0  
2-theta = 51.85°  
Plot Levels:

Figure 40. (111) Pole Figure of Specimen Number 26.

Sample: 26-111  
24-Aug-00 18:01  
h = 1  k = 1  l = 1  
2-theta = 44.45°  
Plot Levels:
Figure 41. (200) Pole Figure of Specimen Number 37.

Sample: 37-200
24-Aug-00  17:17
h = 2  k = 0  l = 0

R.D
2-theta = 51.86°

Plot Levels:

Figure 42. (111) Pole Figure of Specimen Number 37.

Sample: 37-111
24-Aug-00  16:40
h = 1  k = 1  l = 1

R.D
2-theta = 44.50°

Plot Levels:
Figure 43. (200) Pole Figure of Specimen Number 45.

Sample: 45-200
24-Aug-00  15:55
h = 2  k = 0  l = 0

2-theta = 52.00°

Plot Levels:

Figure 44. (111) Pole Figure of Specimen Number 45.

Sample: 45-111
24-Aug-00  15:18
h = 1  k = 1  l = 1

2-theta = 44.65°

Plot Levels:
Figure 45. (200) Pole Figure of Specimen Number 53.

Sample: 53-200
24-Aug-00  13:58
h = 2  k = 0  l = 0

R.D

2-theta = 51.4

Plot Levels:

<table>
<thead>
<tr>
<th>%</th>
<th>Plot Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.3%</td>
<td>0.00</td>
</tr>
<tr>
<td>25.0%</td>
<td>13.50</td>
</tr>
<tr>
<td>41.7%</td>
<td>21.50</td>
</tr>
<tr>
<td>66.4%</td>
<td>28.50</td>
</tr>
<tr>
<td>75.0%</td>
<td>34.50</td>
</tr>
<tr>
<td>91.7%</td>
<td>39.50</td>
</tr>
</tbody>
</table>

Figure 46. (111) Pole Figure of Specimen Number 53.

Sample: 53-111
24-Aug-00  12:21
h = 1  k = 1  l = 1

R.D

2-theta = 44.60°

Plot Levels:

<table>
<thead>
<tr>
<th>%</th>
<th>Plot Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.3%</td>
<td>0.00</td>
</tr>
<tr>
<td>25.0%</td>
<td>3.25</td>
</tr>
<tr>
<td>41.7%</td>
<td>6.25</td>
</tr>
<tr>
<td>66.4%</td>
<td>9.25</td>
</tr>
<tr>
<td>75.0%</td>
<td>12.25</td>
</tr>
<tr>
<td>91.7%</td>
<td>15.25</td>
</tr>
</tbody>
</table>
Figure 47 illustrates (400) pole figure of gamma-alumina of coated specimen number 45.

Figure 47. (400) Pole Figure of Gamma-Alumina

Surface Topography

Figure 48 illustrates the surface topography of a coated specimen. The average roughness, \( R_a \), and the root mean square roughness, \( R_{\text{rms}} \), were measured. See appendix E for all the specimens.
Figure 48. Surface Topography of a Coated Specimen.

Table 30 presents the values average roughness, $R_a$, and root mean square roughness, $R_q$, of the coated specimens. Average roughness and root mean square roughness of uncoated Al 2040-T3 were 570, and 734 nm respectively.

Table 30

Average Roughness, $R_a$, and Root Mean Square Roughness, $R_q$, of Coated Specimens

<table>
<thead>
<tr>
<th>Particles Con</th>
<th>$R_a$ (nm)</th>
<th>$R_q$ (nm)</th>
<th>Particles Con</th>
<th>$R_a$ (nm)</th>
<th>$R_q$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>507</td>
<td>653</td>
<td>Ni</td>
<td>578</td>
<td>751</td>
</tr>
<tr>
<td>8 g/litter</td>
<td>540</td>
<td>698</td>
<td>8 g/litter</td>
<td>561</td>
<td>721</td>
</tr>
<tr>
<td>14 g/litter</td>
<td>566</td>
<td>731</td>
<td>14 g/litter</td>
<td>618</td>
<td>797</td>
</tr>
<tr>
<td>22 g/litter</td>
<td>559</td>
<td>730</td>
<td>22 g/litter</td>
<td>657</td>
<td>861</td>
</tr>
</tbody>
</table>
Figures 49 and 50 illustrate the values of the average roughness and the root mean square roughness, which is shown in table 30, of low and high condition coated specimens graphically respectively.

**Figure 49.** Average Roughness, $R_a$, of Coated Specimens.

**Figure 50.** Root Mean Square Roughness, $R_q$, of Coated Specimens.
Particle Concentrations

Particle concentrations can be calculated using equation (1.1) \((4)\), and Young’s modulus, taking standard deviations into account, table 29.

\[ E_c = E_{\gamma-Al_2O_3} V_{\gamma-Al_2O_3} + E_{Ni} V_{Ni} \]

where \( E_c \), \( E_{\gamma-Al_2O_3} \), and \( E_{Ni} \) are the modulus of the composite, gamma-aluminum oxide, and nickel respectively. \( V_{\gamma-Al_2O_3} \) and \( V_{Ni} \) are the volume fraction of nickel and gamma-aluminum oxide.

Table 31 summarizes calculated values of particle concentrations in the coating.

Table 31
Calculated Values of Particle Concentrations in the Coating

<table>
<thead>
<tr>
<th>Coating Conditions</th>
<th>Particle Concentrations ( g/l )</th>
<th>Particle Concentration ( % \text{vol} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>High</td>
<td>8</td>
<td>15</td>
</tr>
<tr>
<td>Low</td>
<td>8</td>
<td>20</td>
</tr>
<tr>
<td>High</td>
<td>14</td>
<td>30</td>
</tr>
<tr>
<td>Low</td>
<td>14</td>
<td>41</td>
</tr>
<tr>
<td>High</td>
<td>22</td>
<td>62</td>
</tr>
<tr>
<td>Low</td>
<td>22</td>
<td>79</td>
</tr>
</tbody>
</table>

Discussion

It was obvious from table 29, and figure 30 that increasing the particle concentrations increased the Young’s modulus. The Young’s modulus averages of the
specimens, which were coated by nickel only, were 162 and 164.7 GPa for high and low conditions respectively. These values, which equaled the same results that obtained by Ari-Gur \(^{(55)}\), were less than the Young’s modulus value of nickel, 204GPa. \(^{(17)}\) Although \(E_{\{111\}}\) (298 GPa), is higher than \(E_{\{100\}}\), (136 GPa), as it can be seen in chapter I, Young’s modulus is decreased. The lower value of the Young’s modulus of those specimens is attributed to that the intensity of preferred orientation of (200) is much higher than that of (111) as it can be seen in figures 31, 32, 39, and 40.

Increasing \(\gamma\)-aluminum oxide concentration increased Young’s modulus of the coating. Increasing the particle concentrations from 8, 14, to 22 g/litter increased Young’s modulus 7.8%, 14.9%, and 26.3%, respectively, for high condition coated specimens while Young’s modulus increased 9.9%, 18.3%, and 30%, respectively, for low condition coated specimen.

The coating conditions had effect on the Young’s modulus of the coating layer as shown in figure 30. Low condition coated specimens had higher Young’s modulus than high condition coated specimens at the same particle concentration. At particle concentrations of 8, 14, and 22 g/litter Young’s modulus of low condition coated specimens were 4%, 5.6%, and 6.8% higher, respectively, than Young’s modulus of high condition coated specimens. Taking the standard deviation of Young’s modulus into account as it can be seen in table 29 discloses that the increasing of Young’s modulus is unnoticeable.

Very strong (200) preferred orientations were observed in the coated specimens parallel to the surface as shown in \(\{200\}\) pole figures, which matched the results that were obtained by Chan \(^{(13)}\), Barrett \(^{(8)}\), and Marc. \(^{(3)}\) Consequently, the \(<100>\) direction was parallel to the rolling direction.
Close investigation of (111) pole figures disclosed that low condition coated specimens had much higher <111> fiber texture intensity than high condition coated specimens which may explain the difference in the Young’s modulus between the low and high condition coated specimens. Very strong preferred orientation of the gamma-alumina particles was observed as it could be seen in (400) pole figure of gamma-alumina. Calculated values of particle concentrations in the coating did not match the particle concentrations that were calculated using scanning electron microscopy (SEM). Table 32 summarizes calculated particle concentrations using SEM.

Table 32

<table>
<thead>
<tr>
<th>Coating Conditions</th>
<th>Particle Concentrations</th>
<th>Particle Concentrations</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/l</td>
<td>% Vol</td>
</tr>
<tr>
<td>High</td>
<td>8</td>
<td>16</td>
</tr>
<tr>
<td>Low</td>
<td>8</td>
<td>31</td>
</tr>
<tr>
<td>High</td>
<td>14</td>
<td>24</td>
</tr>
<tr>
<td>Low</td>
<td>14</td>
<td>68</td>
</tr>
<tr>
<td>High</td>
<td>22</td>
<td>76</td>
</tr>
<tr>
<td>Low</td>
<td>22</td>
<td>53</td>
</tr>
</tbody>
</table>

Table 30, which summarized the results of surface topography of the specimens, and figure 49 have shown that the average roughness decreased by coating the substrate by nickel only and then increased by adding gamma-aluminum oxide particles that indicated increasing the particle concentrations on the coating layer. The average roughness and the root mean square of low condition coated specimens were higher than that of high conditions coated specimens which indicated
that the low condition coated specimens had higher particle concentrations than high condition coated specimens as it can be seen in table 31. Surface topography figures indicates that the ridges parallel to the rolling direction still appear though the substrate is coated.

Strength of the (111) pole figures, particle concentrations in the coating, and surface topography of low condition coated specimens explained the increasing of Young's modulus of low condition coated specimens due to having higher preferred orientations and higher particle concentration.
CHAPTER V

CONCLUSION

Unexplored electrodeposition of nickel / nano-γAl₂O₃ composites was done in this study. The effects of coating variables, namely coating time, current density, and mixing rate (agitation), on the coating thickness was analyzed using Taguchi method. As expected, current density and coating time were the two most significant variables that affect the coating thickness. The agitation factor, that was not previously investigated, affected the uniformity of the coating as well as the mean thickness. Following the changes made in the electroplating bath setup, uniform coating was achieved. The coating conditions had a significant effect on the modulus of elasticity, the crystallographic texture, and surface topography.

Increasing the concentration of the particles in the electroplating bath increased the elastic modulus of the composite nickel/γAl₂O₃. Young’s modulus of “low conditions” coated specimens was higher than that of “high conditions” coated ones. That is due to the higher particle concentrations in the coating of “low conditions” coated specimens.

Unexplored textures in nickel/nano-γAl₂O₃ composites as well as the texture of the nano-γAl₂O₃ particles were investigated. Strong crystallographic textures of both nickel and γAl₂O₃ were present in the coating by using (200) and (111) pole figures for nickel and a (400) pole figure for γAl₂O₃. The (200) pole figures of nickel disclosed fiber texture, did not exceed 30° tilt angle, while the texture of (111) was spread on a much larger angular range. Nickel texture intensity of (111) of “low conditions” coated specimens was higher than that of “high conditions”. The values
of Young’s modulus demonstrated strong anisotropy as a result of the crystallographic texture.

The effect of the coating conditions on the surface topography of the coated specimens was apparent. The “low conditions” coated specimens had higher average roughness and RMS roughness than the “high conditions” coated ones as a result of increased particle concentrations in the coating.
Appendix A

Item Suppliers
### Table A-1

<table>
<thead>
<tr>
<th>ITEM</th>
<th>INFORMATION</th>
<th>SUPPLIER</th>
<th>PHONE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anode Bag</td>
<td>Plain Weave Polypro</td>
<td>Plating products Inc</td>
<td>1-765-457-1194</td>
</tr>
<tr>
<td>Immersion Heater</td>
<td>Vycor quartz w/cord and plug 120 V</td>
<td>VWR</td>
<td>1-800-932-4158</td>
</tr>
<tr>
<td>Al₂O₃ Powder</td>
<td>1 Kg</td>
<td>Nanophase Cor</td>
<td>1-630-323-1200</td>
</tr>
<tr>
<td>Barrett-SN</td>
<td>Nickel Sulfamate</td>
<td>MacDermid</td>
<td>1-800-325-4158</td>
</tr>
<tr>
<td>Latex glove med</td>
<td></td>
<td>Chemistry department</td>
<td></td>
</tr>
<tr>
<td>HNO₃ &amp; HCl</td>
<td></td>
<td>Chemistry department</td>
<td></td>
</tr>
<tr>
<td>Flow meter</td>
<td>100 SCFH Rotameter</td>
<td>GRAINGER</td>
<td>1-616-381-8500</td>
</tr>
<tr>
<td>Filter / REG&amp;LUB</td>
<td>¼ &quot; MINI filter/reg&amp;lub</td>
<td>GRAINGER</td>
<td>1-616-381-8500</td>
</tr>
<tr>
<td>Zincate</td>
<td></td>
<td>MacDermind</td>
<td>1-800-987-3435</td>
</tr>
<tr>
<td>Alkaline Degreaser</td>
<td></td>
<td>MacDermid</td>
<td>1-800-987-3435</td>
</tr>
<tr>
<td>NaOH</td>
<td></td>
<td>Chemistry department</td>
<td></td>
</tr>
<tr>
<td>Al 2024-T3</td>
<td>0.635 mm sheet</td>
<td>Schupan Aluminum</td>
<td>616-382-3434</td>
</tr>
<tr>
<td>Anode</td>
<td>Nickel oxide depolarized nickel</td>
<td>King Supply Allied</td>
<td>1-847-698-4564</td>
</tr>
<tr>
<td>Coating Tank</td>
<td>Polypropylene Tank (305x305x304x12.7)</td>
<td>L. C Fabricators,</td>
<td>1-908-241-4252</td>
</tr>
<tr>
<td>Coating Bath Heater</td>
<td>Teflon Heater, 1Kw 120V</td>
<td>L. C Fabricators,</td>
<td>1-908-241-4252</td>
</tr>
<tr>
<td>D. C Power Supply</td>
<td>100 amp at 12 Volts out put</td>
<td>L. C Fabricators,</td>
<td>1-908-241-4252</td>
</tr>
</tbody>
</table>
Appendix B

Analyzing of Coating Data by Minitab Program
MTB> name c1 ‘current density’
MTB> set c1
DATA> -1 -1 -1 0 0 0 1 1 1
DATA> end
MTB> name c2 ‘time’
MTB> set c2
DATA> -1 0 1 1 0 1 -1 0 1
DATA> end
MTB> name c3 ‘agitation’
MTB> set c3
DATA> -1 0 1 0 1 -1 1 -1 0
DATA> end
MTB> name c4 ‘front’
MTB> set c4
DATA> 0.48 0.86 0.81 0.8 1.51 2.15 1.12 1.58 2.11
DATA> end
MTB> name c5 ‘mid’
DATA> 0.54 0.84 0.76 0.68 1.44 2 1.07 1.76 1.96
DATA> end
MTB> name c6 ‘rear’
MTB> set c6
DATA> 0.56 0.81 0.86 0.74 1.38 1.98 1.21 1.96 2.53
DATA> end
MTB> name c7 ‘front’
MTB> set c7
DATA> 0.43 0.92 0.88 0.79 1.54 2.1 1.17 1.8 2.4
DATA> end
MTB> name c8 ‘mid’
MTB> set c8
DATA> 0.49 0.89 0.81 0.79 1.66 2.19 1.07 1.61 2.27
DATA> end
MTB> name c9 'rear'
MTB> set c9
DATA> 0.48 0.95 0.8 0.85 1.62 2.09 1.23 2.12 3.11
DATA> end
MTB> name c10 'front'
MTB> set c10
DATA> 0.47 0.61 0.67 0.8 1.53 2.5 1.08 1.98 2.68
DATA> end
MTB> name c11 'mid'
MTB> set c11
DATA> 0.52 0.63 0.81 0.9 1.62 2.5 0.99 1.93 2.93
DATA> end
MTB> name c12 'rear'
MTB> set c12
DATA> 0.53 0.83 0.83 1 1.71 2.5 1.13 2.22 3
DATA> end
MTB> name c13 'front'
MTB> set c13
DATA> 0.52 0.9 1.05 0.9 1.68 2.3 1.24 1.44 2.55
DATA> end
MTB> name c14 'mid'
MTB> set c14
DATA> 0.49 0.96 1 0.81 1.59 2.4 1.15 1.39 1.38
DATA> end

MTB> name c15 'rear'
MTB> set c15
DATA> 0.49 0.93 0.94 0.83 1.45 2 1.28 1.38 1.86
DATA> end
MTB> name c16 'front'
MTB> set c16
DATA> 0.61 0.63 0.99 0.8 1.44 2.5 1.26 1.41 2.35
DATA> end
MTB> name c17 'mid'
MTB> set c17
DATA> 0.6 0.67 1.02 0.76 1.35 2.3 1.08 1.4 2.5
DATA> end
MTB> name c18 'rear'
MTB> set c18
DATA> 0.65 0.69 1 0.82 1.39 1.94 1.26 1.56 2.17
DATA> end
MTB> name c19 'front'
MTB> set c19
DATA> 0.54 0.65 0.86 0.8 1.38 2 1.19 1.54 2.53
DATA> end
MTB> name c20 'mid'
MTB> set c20
DATA> 0.5 0.61 0.89 0.74 1.38 2.17 1.12 1.55 2.56
DATA> end
MTB> name c21 'rear'
MTB> set c21
DATA> 0.47 0.58 0.96 0.77 1.25 1.5 1.29 1.36 2.62
DATA> end
MTB> rmean c4-c21 c22
MTB > let c23 = (c4-c22)**2+(c5-c22)**2+(c6-c22)**2+(c7-c22)**2+(c8-c22)**2
MTB > let c24 = (c9-c22)**2+(c10-c22)**2+(c11-c22)**2+(c12-c22)**2+(c13-c22)**2+(c14-c22)**2
MTB > let c25 = (c15-c22)**2+(c16-c22)**2+(c17-c22)**2+(c18-c22)**2+(c19-c22)**2+(c20-c22)**2+(c21-c22)**2
MTB > let c26 = c23+c24+c25
MTB > let c27 = (1/(17))*c26
MTB > let c28 = c27**.5
MTB > table c1;
SUBC> means c22.
MTB > let c23 = (c4-c22)**2+(c5-c22)**2+(c6-c22)**2+(c7-c22)**2+(c8-c22)**2
MTB > let c24 = (c9-c22)**2+(c10-c22)**2+(c11-c22)**2+(c12-c22)**2+(c13-c22)**2+(c14-c22)**2
MTB > let c25 = (c15-c22)**2+(c16-c22)**2+(c17-c22)**2+(c18-c22)**2+(c19-c22)**2+(c20-c22)**2+(c21-c22)**2
MTB > let c26 = c23+c24+c25
MTB > let c27 = 1/17*c26
MTB > let c27 = (1/17)*c26
MTB > let c28 = c27**.5
MTB > table c1;
SUBC> means c22.

Tabulated Statistics

Rows: Current Density
<table>
<thead>
<tr>
<th>Thickness</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>-1</td>
<td>0.7272</td>
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<tr>
<td>0</td>
<td>1.4930</td>
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<tr>
<td>1</td>
<td>1.7489</td>
</tr>
<tr>
<td>All</td>
<td>1.3230</td>
</tr>
</tbody>
</table>

MTB > table c2;
SUBC> means c22.

**Tabulated Statistics**

Rows: time

<table>
<thead>
<tr>
<th>Thickness</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>-1</td>
<td>0.8313</td>
</tr>
<tr>
<td>0</td>
<td>1.3124</td>
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<tr>
<td>1</td>
<td>1.8254</td>
</tr>
<tr>
<td>All</td>
<td>1.3230</td>
</tr>
</tbody>
</table>

MTB > table c3;
SUBC> means c22.
Tabulated Statistics

Rows: agitation

Thickness
Mean

-1 1.4533
0 1.3343
1 1.1815
All 1.3230

MTB > regr c22 6 c29-c34;
SUBC> coeff c35.

Regression Analysis

The regression equation is
Thickness = 1.32 + 0.511 CL + 0.0850 CQ + 0.497 tL - 0.0053 tQ - 0.136 AL
+ 0.0056 AQ

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coef</th>
<th>StDev</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>1.3230</td>
<td>0.1058</td>
<td>12.50</td>
<td>0.006</td>
</tr>
<tr>
<td>CL</td>
<td>0.5108</td>
<td>0.1296</td>
<td>3.94</td>
<td>0.059</td>
</tr>
<tr>
<td>CQ</td>
<td>0.08497</td>
<td>0.07484</td>
<td>1.14</td>
<td>0.374</td>
</tr>
<tr>
<td>tL</td>
<td>0.4970</td>
<td>0.1296</td>
<td>3.83</td>
<td>0.062</td>
</tr>
<tr>
<td>tQ</td>
<td>-0.00531</td>
<td>0.07484</td>
<td>-0.07</td>
<td>0.950</td>
</tr>
</tbody>
</table>
AL  -0.1359  0.1296  -1.05  0.404
AQ   0.00562  0.07484  0.08  0.947

S = 0.3175  R-Sq = 94.2%  R-Sq(adj) = 76.9%

Analysis of Variance

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P</th>
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</thead>
<tbody>
<tr>
<td>Regression</td>
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<td>3.2899</td>
<td>0.5483</td>
<td>5.44</td>
<td>0.163</td>
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<td>Residual Error</td>
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<td>0.2017</td>
<td>0.1008</td>
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<td>Total</td>
<td>8</td>
<td>3.4915</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CL</td>
<td>1</td>
<td>1.5657</td>
</tr>
<tr>
<td>CQ</td>
<td>1</td>
<td>0.1300</td>
</tr>
<tr>
<td>tL</td>
<td>1</td>
<td>1.4823</td>
</tr>
<tr>
<td>tQ</td>
<td>1</td>
<td>0.0005</td>
</tr>
<tr>
<td>AL</td>
<td>1</td>
<td>0.1109</td>
</tr>
<tr>
<td>AQ</td>
<td>1</td>
<td>0.0006</td>
</tr>
</tbody>
</table>

MTB > let c37=c35*c36
MTB > let c38=c37**2
MTB > let c39=100*c38/sum(c38)
MTB > print c40 c35-c39

Data Display
Row  IAB  coff  C36  C37  C38  C39

1  CL  0.51083  2.44949  1.25128  1.56570  47.5917
2  CQ  0.08497  4.24264  0.36049  0.12996  3.9502
3  tL  0.49704  2.44949  1.21749  1.48227  45.0558
4  tQ -0.00531  4.24264 -0.02252  0.00051  0.0154
5  AL -0.13593  2.44949 -0.33295  0.11086  3.3696
6  AQ  0.00562  4.24264  0.02383  0.00057  0.0173

MTB > let c41= abso(c37)
MTB > dotplot c37

Dot-plot

---+---------+---------+---------+---------+---------+---C37
  -0.30  0.00  0.30  0.60  0.90  1.20

MTB > let k1=37
MTB > exec'c:\half.min.txt'
Executing from file: c:\half.min.txt
MTB >
MTB >
MTB > # half normal plot
MTB > #######
MTB > end
MTB > let c42=log(c28)
MTB > table c1;
SUBC> means c42.

**Tabulated Statistics**

Rows: Current Density

<table>
<thead>
<tr>
<th>logS</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>-1</td>
<td>-2.3778</td>
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<tr>
<td>0</td>
<td>-2.0058</td>
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<tr>
<td>1</td>
<td>-1.5322</td>
</tr>
<tr>
<td>All</td>
<td>-1.9719</td>
</tr>
</tbody>
</table>

MTB > table c2;
SUBC> means c42.

**Tabulated Statistics**
Rows: time

logS
Mean

-1  -2.6560
0   -1.7689
1   -1.4908
All -1.9719

MTB > table c3;
SUBC> means c42.

Tabulated Statistics

Rows: agitation

logS
Mean

-1  -1.8390
0   -1.8278
1   -2.2489
All -1.9719
MTB > regr c42 6 c29-c34;
SUBC> coeff c43.

Regression Analysis

The regression equation is
\[ \log S = -1.97 + 0.423 \, CL - 0.0169 \, CQ + 0.583 \, tL + 0.102 \, tQ - 0.205 \, AL + 0.0720 \, AQ \]

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coef</th>
<th>StDev</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-1.97190</td>
<td>0.04760</td>
<td>-41.43</td>
<td>0.001</td>
</tr>
<tr>
<td>CL</td>
<td>0.42282</td>
<td>0.05830</td>
<td>7.25</td>
<td>0.015</td>
</tr>
<tr>
<td>CQ</td>
<td>-0.01693</td>
<td>0.03366</td>
<td>-0.50</td>
<td>0.665</td>
</tr>
<tr>
<td>tL</td>
<td>0.58260</td>
<td>0.05830</td>
<td>9.99</td>
<td>0.000</td>
</tr>
<tr>
<td>tQ</td>
<td>0.10151</td>
<td>0.03366</td>
<td>3.02</td>
<td>0.095</td>
</tr>
<tr>
<td>AL</td>
<td>-0.20499</td>
<td>0.05830</td>
<td>-3.52</td>
<td>0.072</td>
</tr>
<tr>
<td>AQ</td>
<td>0.07203</td>
<td>0.03366</td>
<td>2.14</td>
<td>0.166</td>
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</tbody>
</table>

\[ S = 0.1428 \quad R-Sq = 98.9\% \quad R-Sq(adj) = 95.6\% \]

Analysis of Variance

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
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<td>3.64532</td>
<td>0.60755</td>
<td>29.80</td>
<td>0.033</td>
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<tr>
<td>Residual Error</td>
<td>2</td>
<td>0.04078</td>
<td>0.02039</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>8</td>
<td>3.68610</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Source  DF  Seq SS
CL  1  1.07266
CQ  1  0.00516
tL  1  2.03652
tQ  1  0.18547
AL  1  0.25212
AQ  1  0.09340

MTB > copy c43 c43;
SUBC> omit 1.
MTB > let c44 = c43*c36
MTB > let c45 = c44**2
MTB > let c46 = c45*100/sum(c45)
MTB > print c40 c43-c46

Data Display

<table>
<thead>
<tr>
<th>Row</th>
<th>IAB</th>
<th>LogS</th>
<th>coeff</th>
<th>C44</th>
<th>logS</th>
<th>SS</th>
<th>con%</th>
</tr>
</thead>
<tbody>
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<td>1</td>
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<td>1.07266</td>
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<tr>
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<td>-0.07184</td>
<td>0.00516</td>
<td>0.1416</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
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MTB > let c47 = abso(c44)
MTB > dotplot c44
MTB > let k1=44
MTB > exec 'c:\half.min.txt'
Executing from file: c:\half.min.txt
MTB >
MTB >
MTB > # half normal plot
MTB > #FFFFFFFFFFFFFFFFFFFFF#
MTB > end
MTB > %Main 'CL' 'CQ' 'tL' 'tQ' 'AL' 'AQ';
SUBC>  Response 'Thickness'.
Executing from file: C:\PROGRAM FILES\MTBWIN\MACROS\Main.MAC

Macro is running ... please wait
MTB > %Main 'CL' 'CQ' 'tL' 'tQ' 'AL' 'AQ';
SUBC>  Response 'logS'.
Executing from file: C:\PROGRAM FILES\MTBWIN\MACROS\Main.MAC

Macro is running ... please wait
MTB > Save "C:\My Documents\thesis\MINITAB-1.MPJ";
SUBC>  Project;
SUBC> Replace.

Worksheet size: 100000 cells
Retrieving project from file: C:\WINDOWS\DESKTOP\MINITA~1.MPJ
MTB >
Appendix C

Determination the Slopes of Elastic Zone and Coefficients of Determination of the Coated Specimens by Least Square Method
Figure C-1. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 28.

Figure C-2. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 29.
Figure C-3. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 30.

Figure C-4. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 31.
Figure C-5. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 32.

Figure C-6. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 34.
Figure C-7. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 35.

Figure C-8. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 36.
Figure C-9. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 38.

Figure C-10. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 39.
Figure C-11. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 40.

Figure C-12. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 42.
Figure C-13. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 43.

Figure C-14. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 44.
Figure C-15. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 46.

Figure C-16. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 47.
Figure C-17. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 48.

Figure C-18. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 50.
Figure C-19. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 51.

Figure C-10. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 52.
Figure C-21. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 54

Figure C-22. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 55.
Figure C-23. The Slope of Elastic Zone and Coefficient of Determination of Coated Specimen Number 56.
Appendix D

Standard 2-Theta XRD of Coated Specimens
1-Low Condition Coated Specimens

Figure D-1. Standard 2-Theta XRD of Coated Specimen Number 26

Figure D-2. Standard 2-Theta XRD of Coated Specimen Number 37
Figure D-3. Standard 2-Theta XRD of Coated Specimen Number 45

Figure D-4. Standard 2-Theta XRD of Coated Specimen Number 53
2- High condition coated specimens

Figure D-5. Standard 2-Theta XRD of Coated Specimen Number 25

Figure D-6. Standard 2-Theta XRD of Coated Specimen Number 33
Figure D-7. Standard 2-Theta XRD of Coated Specimen Number 41

Figure D-8. Standard 2-Theta XRD of Coated Specimen Number 49
Appendix E

Surface Topography of Coated Specimens
Figure E-1. Surface Topography of Al 2024-T3 Coated Specimen.
1- Low Condition Coated Specimens

Figure E-2. Surface Topography of Coated Specimen Number 26.
Figure E-3. Surface Topography of Coated Specimen Number 37.
Figure E-4. Surface Topography of Coated Specimen Number 45.
Figure E-5. Surface Topography of Coated Specimen Number 53.
2- High Condition Coated Specimens

Figure E-6. Surface Topography of Coated Specimen Number 25.
Figure E-7. Surface Topography of Coated Specimen Number 33.
Figure E-8. Surface Topography of Coated Specimen Number 41.
Figure E-9. Surface Topography of Coated Specimen Number 49.
REFERENCES


