CHARACTERISATION OF ALUMINIUM AND ITS ALLOYS AFTER SURFACE PRE-TREATMENT PRIOR TO ELECTRODEPOSITION

ROBERT WILLIAMS-OYENIGBA
and
C.F. ODUOZA
OUTLINE OF PRESENTATION

- Introduction
- The zinc immersion process
- Aims and objectives
- Methodology
- Results
- Conclusion
Introduction

Problem Definition - Aluminium, a versatile engineering material, reacts readily in the presence of oxygen to form an oxide film which makes it difficult to plate.

Types of surface pre-treatment are:

1. Mechanical Preparation (Dennis and Such, 1993).
2. Chemical Etching (Work, 1933 (in Wernick et.al., 1987)).
3. Stannate immersion process (Bryan, 1953 (in Wernick et.al., 1987)).
5. Zinc immersion process (Wernick, et.al., 1987).
The Zinc Immersion Process

This is a surface pre-treatment method whereby a zincate solution is used to deposit zinc on the surface of aluminium and its alloys to improve its quantitative adhesion.

The basic net reactions of the Zinc immersion process are:

1. The dissolution of aluminium oxide:
\[ \text{Al}_2\text{O}_3 + 2\text{NaOH} + 3\text{H}_2\text{O} \rightarrow 2\text{NaAl(OH)}_4 \]

2. The deposition of zinc:
\[ 3\text{Na}_2\text{Zn(OH)}_4 + 2\text{Al}_{\text{substrate}} \rightarrow 2\text{NaAl(OH)}_4 + 4\text{NaOH} + 3\text{Zn} \text{ (deposited)} \]

Types of the zinc immersion process:
- The single zinc immersion process (Hewitson, 1927 (in Wernick et.al., 1987)).
- The double zinc immersion process (Korpuin, 1939 (in Wernick et.al., 1987)).
Aim and Objectives

**Aim** - To study and compare the surface characterisation of three types of aluminium alloys after a double zinc immersion process.

**Objectives:**

- To study the kinetics of the double zincating process by weight characterisation.
- To determine how variations in the immersion times of the zincating process affects the surface characteristics.
- To study and compare the double zinc immersion process for three different types of aluminium alloy.
- To verify the effect of the double zinc immersion process on the adhesive properties of aluminium/aluminium alloys prior to electrodeposition.
Methodology

A laboratory-scale experiment carried out on samples of three types of aluminium alloys. The stages of the experiment are:

1. Alkaline cleaning at 60°C for 3 minutes
2. Acid dip at room temperature for 3 minutes
3. First zinc immersion at room temp. for 90 seconds
4. Acid dip at room temperature for 45 seconds
5. Second zinc immersion at room temp. at varied immersion times
6. Water rinse
7. Water rinse
8. Water rinse
9. Water rinse

Fig1: The stages of the experiment
Methodology (continued)

Resources

The aluminium samples and their compositions are given in the table below:

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Main Alloying Element Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Si</td>
</tr>
<tr>
<td>Al 1050</td>
<td>0.25</td>
</tr>
<tr>
<td>Al 6061</td>
<td>0.4-</td>
</tr>
<tr>
<td></td>
<td>0.8</td>
</tr>
<tr>
<td>Al 6082</td>
<td>0.7-</td>
</tr>
<tr>
<td></td>
<td>1.3</td>
</tr>
</tbody>
</table>

Other resources used are:

- Minco cleaner – An alkaline compound (MacDermid plc).
- 66 – Microetch – An acidic compound (MacDermid plc).
- Bondal dip – A Modified Alloy Zincate solution (MacDermid plc).
Methodology (continued)

Surface Analytical Techniques
The surface analytic techniques used are listed below:

- X-Ray Fluorescence Spectroscopy (A Spectro Xepos Spectrometer).
## Results

### The weight characterisation

#### Weight characterisation of Al 1050

<table>
<thead>
<tr>
<th>First Experiment</th>
<th>Before zincating (g)</th>
<th>After zincating (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion time of 25 seconds</td>
<td>2.06</td>
<td>2.051</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>2.14</td>
<td>2.093</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.10</td>
<td>2.0399</td>
</tr>
<tr>
<td><strong>Second experiment</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Immersion time of 25 seconds</td>
<td>1.955</td>
<td>1.89</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>2.074</td>
<td>2.008</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.0133</td>
<td>2.002</td>
</tr>
</tbody>
</table>

#### Weight Characterisation of Al 6061

<table>
<thead>
<tr>
<th>First experiment</th>
<th>Before zincating (g)</th>
<th>After zincating (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion time of 25 seconds</td>
<td>2.3018</td>
<td>2.2655</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>2.4166</td>
<td>2.4288</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.89</td>
<td>2.9075</td>
</tr>
<tr>
<td><strong>Second experiment</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Immersion time of 25 seconds</td>
<td>2.5872</td>
<td>2.4205</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>2.3925</td>
<td>2.2804</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.2895</td>
<td>2.297</td>
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</table>
## Results (continued)

**Weight characterisation of Al 6082**

<table>
<thead>
<tr>
<th>First Experiment</th>
<th>Before zinctating (g)</th>
<th>After zinctating (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion time of 25 seconds</td>
<td>1.9326</td>
<td>1.9302</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>1.9623</td>
<td>2.0254</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.0209</td>
<td>2.0074</td>
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<tr>
<td><strong>Second experiment</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Immersion time of 25 seconds</td>
<td>2.0181</td>
<td>1.9825</td>
</tr>
<tr>
<td>Immersion time of 30 seconds</td>
<td>1.9018</td>
<td>1.8417</td>
</tr>
<tr>
<td>Immersion time of 35 seconds</td>
<td>2.0701</td>
<td>2.0843</td>
</tr>
</tbody>
</table>

**SEM Analysis of Al 6061**

Second immersion time of 25 seconds

Second immersion time of 30 seconds

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Results (continued)

SEM Analysis of Al 6061

Second immersion time of 35 seconds

Second immersion time of 30 seconds

SEM Analysis of Al 6082

Second immersion time of 25 seconds

Second immersion time of 35 seconds
Results (continued)

SEM Analysis of Al 1050

Second immersion time of 25 seconds

SEM Analysis of Al 1050

Second immersion time of 30 seconds

SEM Analysis of Al 1050

Second immersion time of 35 seconds

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# Results (continued)

## XRF Spectroscopy of the aluminium samples

### Al 1050

<table>
<thead>
<tr>
<th>Immersion times</th>
<th>Aluminium %</th>
<th>Zinc %</th>
<th>Iron %</th>
<th>Copper %</th>
<th>Nickel %</th>
</tr>
</thead>
<tbody>
<tr>
<td>25-sec</td>
<td>97.06</td>
<td>0.09288</td>
<td>0.181</td>
<td>0.02625</td>
<td>0.01089</td>
</tr>
<tr>
<td>30-sec</td>
<td>95.07</td>
<td>0.1473</td>
<td>0.1881</td>
<td>0.03826</td>
<td>0.01705</td>
</tr>
<tr>
<td>35-sec</td>
<td>95.94</td>
<td>0.1676</td>
<td>0.1891</td>
<td>0.04244</td>
<td>0.01878</td>
</tr>
</tbody>
</table>

### Al 6061

<table>
<thead>
<tr>
<th>Immersion times</th>
<th>Aluminium %</th>
<th>Zinc %</th>
<th>Iron %</th>
<th>Copper %</th>
<th>Nickel %</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 seconds</td>
<td>96.07</td>
<td>0.1087</td>
<td>0.1643</td>
<td>0.03483</td>
<td>0.01549</td>
</tr>
<tr>
<td>30 seconds</td>
<td>96.13</td>
<td>0.1296</td>
<td>0.18</td>
<td>0.03405</td>
<td>0.1296</td>
</tr>
<tr>
<td>35 seconds</td>
<td>95.74</td>
<td>0.1375</td>
<td>0.1803</td>
<td>0.03839</td>
<td>0.01826</td>
</tr>
</tbody>
</table>

### Al 6082

<table>
<thead>
<tr>
<th>Immersion times</th>
<th>Aluminium %</th>
<th>Zinc %</th>
<th>Iron %</th>
<th>Copper %</th>
<th>Nickel %</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 seconds</td>
<td>92.86</td>
<td>0.1445</td>
<td>0.4123</td>
<td>0.0916</td>
<td>0.0916</td>
</tr>
<tr>
<td>30 seconds</td>
<td>91.59</td>
<td>0.198</td>
<td>0.4423</td>
<td>0.1067</td>
<td>0.02706</td>
</tr>
<tr>
<td>35 seconds</td>
<td>91.06</td>
<td>0.2016</td>
<td>0.4462</td>
<td>0.1071</td>
<td>0.02636</td>
</tr>
</tbody>
</table>

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Results (continued)

XRD analysis of the aluminium samples

A diffraction profile for Al 1050 at 25 seconds

A diffraction profile for Al 1050 at 30 seconds
Results (continued)

A diffraction profile for Al 1050 at 35 seconds

A diffraction profile for Al 6061 at 25 seconds
Results (continued)

A diffraction profile for Al 6061 at 30 seconds

A diffraction profile for Al 6061 at 35 seconds
Results (continued)

A diffraction profile for Al 6082 at 25 seconds

A diffraction profile for Al 6082 at 30 seconds
Results (continued)

A diffraction profile for Al 6082 at 35 seconds

Calculation of grain size
The Scherrer method was used to calculate the grain size of the samples. The equation is given below:

\[ D_{vol} = \frac{K \lambda}{FWHM \cdot \cos \theta} \]
Conclusion

- Al 1050 exhibits better quantitative adhesion property than Al 6061 and Al 6082.
- The zinc deposit on Al 1050 is thinner and more compact than Al 6061 and Al 6082.
- For optimum adhesion property, the sequence and parameters of the double zinc immersion process is dependent on the composition of the aluminium alloying elements such as Magnesium (Mg) etc.
- The immersion times has a profound effect on the degree of adhesion.