Characterization of TiO₂ Reinforced Electroless Ni-P-B-W-TiO₂ Multi Alloy Coatings: The Effect of TiO₂ Concentration

Seda Ulu¹, Hasan Algül¹, Abdulkadir Akyol¹, Oğuzhan Bilaç¹, Harun Gül², Mehmet Uysal¹, Ahmet Alp¹
¹Sakarya University, Engineering Faculty, Metallurgical and Materials Engineering Department, 54187, Sakarya, Turkey
²Düzce University, Gumusova Vocational School, 81850 Düzce, Turkey

Abstract

In this study, TiO₂ particle is added to the electroless Ni-P-B-W acidic bath for coating on aluminum substrate. To investigation of TiO₂ content on the properties of nickel based multi alloys system, TiO₂ particles is added to the coating bath at different concentrations. The characterization and mechanical properties of the particulate added composite coatings is investigated.

Keywords: Electroless coating, Nickel, Boron, Tungsten, DMAB, Hypophosphite, TiO₂ Concentration, Composites coating.

1. Introduction

Electroless plating is involved electrochemical activities, called reduction and oxidation reactions, take place among the chemicals entering the reaction[1]. Electroless nickel plating, are an autocatalytic process aimed to deposition nickel from an aqueous nickel solution onto a substrate without electric current [2].

Electroless nickel coatings can be grouped according to reducing agent, hypophosphites for nickel phosphorus coatings, amino borates and boron hydrides for nickel boron coatings, and hydrazine for pure nickel coatings are determines the coating content [3].

Electroless nickel coatings are enable to the production of materials that can serve many purposes with high hardness, corrosion resistance, abrasion resistance, lubricity etc.. Electroless nickel, nickel alloy or nickel composite coatings are used in all areas of the industry due to their mechanical, tribological and electrical properties. electroless coatings are gaining importance because of their complex geometry and can be applied to any materials. [4-7].

Composite coatings can be formed by adding soft or hard particles to the coatings in cases where superior properties than nickel and alloys made by the electroless coating method are desired. [4]

In this study, in order to utilize the lubricating and abrasion resistance of electroless Ni-B coatings and high corrosion resistance of electroless Ni-P coatings two reductive and to improve the thermal stability W added bath system was installed and TiO₂ particles were added to this bath composition. Characterization studies were carried out on the resulting coatings.

2. Experimental Procedure

In this study, 6xxx series aluminum alloy was chosen as the substrate. The sample was prepared in desired sizes to be coated on the plate in dimensions of 3x5x1. The surfaces of these samples were sanded with 1200 grits of sandpaper. An acid solution consisting of 1% H₂SO₄, 1,25% HF and 2,5% HNO₃ was used for surface cleaning and activation. Zincate plating is applied as preliminary treatment to prevent adhesion problem caused by surface oxides on aluminum surfaces.

Ni-P-B-W-TiO₂ coating bath composition and operating conditions is given Table 1. NiSO₄ was used as nickel source, NaPO₂H₂ used as a phosphorus source and reducing agent, DMAB used as boron source and reducing agent. Sodium tungstate was used as a tungsten source. Sodium acetate and lactic acid were used as the complexing agent and thiourea was used as stabilizer. PH adjustment was made with sodium hydroxide. TiO₂ (10-15-20 g / L) was used at different concentrations for the composite coating. SDS was used as the surface active material to increase the wettability of TiO₂ particles. The TiO₂ particles added with SDS were mechanically stirred for 30 min. The prepared TiO₂ particles were added to a electroless Ni-P-B-W coating bath. Coating was carried out at 85°C for 1 hour at a stirring speed of...
In order to reveal the intermetallic phases such as Ni₂P, Ni₃P, Ni₃B, Ni₁B in the after-coating layer and to improve their abrasion properties, they were heat-treated at 400 °C for 2 hours in Ar-H₂ gas atmosphere and then cooled to room temperature.

Table 1. Electroless bath chemical composition and working parameters.

<table>
<thead>
<tr>
<th>Bath chemical composition</th>
<th>Working parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiSO₄·6H₂O</td>
<td>33 g/L</td>
</tr>
<tr>
<td>NaPO₃·H₂O</td>
<td>25 g/L</td>
</tr>
<tr>
<td>C₂H₃NaO₂</td>
<td>16 g/L</td>
</tr>
<tr>
<td>C₄H₁₂BBrN₂</td>
<td>2.5 g/L</td>
</tr>
<tr>
<td>C₁₂H₁₅O₄S·Na</td>
<td>0.1 g/L</td>
</tr>
<tr>
<td>CH₄N₂S</td>
<td>0.001 g/L</td>
</tr>
<tr>
<td>C₃H₆O₃</td>
<td>28 ml/L</td>
</tr>
<tr>
<td>Na₂WO₄</td>
<td>50 g/L</td>
</tr>
<tr>
<td>TiO₂ Particles</td>
<td>10-20 g/L</td>
</tr>
</tbody>
</table>

3. Result and Discussion

Fig 1. Shows the surface and cross-sectional images of coatings obtained from titanium oxide powder added electroless Ni-B-P-W-TiO₂ composite coating baths at different concentrations.

Fig 1. seen that when the electroless Ni-B-P-W-TiO₂ composite coating surface images are examined the amount of titania on the surface increases depending on the TiO₂ concentration added to the coating bath. Similarly, when the TiO₂ concentration in the coating bath is increased, the amount of dust particles included in the cross section increases. Particle addition increases the coating thickness up to a concentration of 15 g / L TiO₂, whereas when the TiO₂ concentration is 20 g / L, the coating thickness decreases. Table 2. shows the effect of titania amount on thickness.
Fig 1. Surface and cross-sectional FESEM images of coatings obtained from titanium oxide powder added electroless Ni-B-P-W-TiO₂ composite coating baths at different concentrations, (a),(b) 10 g/L TiO₂, (c),(d) 15 g/L TiO₂, (e),(f) 20 g/L TiO₂

Table 2. Deposition thickness depending on the amount of TiO₂

<table>
<thead>
<tr>
<th>Amount of TiO₂</th>
<th>Deposition Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 g/L</td>
<td>22.93μm</td>
</tr>
<tr>
<td>15 g/L</td>
<td>35.61 μm</td>
</tr>
<tr>
<td>20 g/L</td>
<td>26.71 μm</td>
</tr>
</tbody>
</table>

Fig 2. shows XRD patterns before and after thermal treatment of the coatings obtained from titanium oxide dust added electroless Ni-B-P-W-TiO₂ composite coatings different concentrations.

When the XRD patterns are examined, it is seen that the coatings are amorphous before heat treatment. Intermetallic phases such as Ni₂B, Ni₃B, Ni₂P, and Ni₃P appeared after heat treatment applied at 400°C for 2 hours in Ar-H₂ gas atmosphere.

Fig 3. Effect of different Titania ratios on the hardness of Ni-P-B-W-TiO₂ coatings

When Figure 3 examined, it was observed that the increase in titania rate positively affects hardness. At the same time, the intermetallic phases formed after heat treatment were seen to have the effects of increasing the hardness.

4. Conclusion

The electroless Ni-P-B-W-TiO₂ coatings are successfully coated on the aluminum surface 10, 15, 20 g / L. In composite coatings made with Titanium reinforcing, the maximum coating thickness was 35.61 μm and it was obtained with addition of 15 g / L. titanium.

XRD analyzes revealed that intermetallic phases such as Ni₃B, Ni₅B, Ni₃P and Ni₅P were formed after heat treatment.

When the hardnesses of composite coatings were compared, hardness enhancing effects of intermetallics formed after heat treatment were observed. And also the increase in the rate of titania has led to an increase in hardness.
Acknowledgment

This work is supported by the Scientific and Technological Research Council of Turkey (TUBITAK) under the contract number 116M998. The authors thank the TUBITAK MAG workers for their financial support.

References


