EFFECTS OF CURRENT DENSITY ON SIZE AND SURFACE MORPHOLOGY OF HIGH SPEED DIRECT NANO-CRYSTALLINE NICKEL PLATING ON TITANIUM SURFACE

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ABSTRACT
Electroplating known as “electrodeposition” is a method of producing a coating, usually metallic, on a surface by applying electric current. In this research work, electrolyte Ni was deposited on Ti by high speed electroplating directly without any pre-cleaning/pre-treatment with varied applied current density. This study reports the effects of deposition of Ni on Ti and it was found that the plating thickness increased with higher current densities, also fine grained structures obtained as the current density increased. Hardness of the deposits decreased as the current density is increased. These showed that the current density affects the coating thickness of Ni-Ti alloy coatings. High speed electroplating is electroplating on metal without any pretreatment process and eliminating oxide layer at high current density which result in high deposition rate of cation on cathode surface.

Keywords: electroplating, nanocrystalline, current density, SEM, XRD.

INTRODUCTION
Titanium and titanium alloys have been widely used due to the low density, sensitive to corrosion, high mechanical strength and ease of fabrication. Due to its high strength and low weight titanium, is beneficial for use in many applications in the aerospace, industrial, and medical fields [1-5]. It is a very active metal and hence it is very difficult to deposit nickel (Ni) coatings with good level of adhesion on their surface because they are rapidly oxidized by oxygen to form an adherent oxide layer. The effective bonding of coating to the titanium (Ti) substrate is the biggest problem due to the presence of oxide. One of the common processes for coating metallic objects is by electroplating. It is a low cost technique for preparing metallic films with variable thickness. In order to enable Ti surface to be electroplated with adherent coating, it is necessary to do the pretreatment sequences. This need is associated with the presence of a very thin oxide layer which forms due to chemical reactivity of Ti with oxygen. An innovative process for depositing nano-crystalline nickel directly on aluminum without any pretreatment required [6-10]. In previous work, Ni-Co alloy was deposited on Ti surfaces at high speed electrolyte movement [6]. It has been demonstrated that deposited nickel has nano-crystalline structure with the particle of nickel is less than 100 nm [11]. Whereas, other extensive researchers deposited nano-crystalline nickel directly on Al, Ti and stainless steel without any pre-treatment [12-15]. Thus, in this investigation, Ni was directly plated on Ti surfaces without any pretreatment process and with good level of adhesion. High speed electroplating is a technique where high speed movement of the plating solution is 2.7 m/s and the rate of plating is higher than 600 \textmu m/h [11].

EXPERIMENTAL PROCEDURES
High speed electroplating technique of nano-crystalline nickel was applied directly on Ti surface. Ti rod samples have dimensions of 10 mm width and 40 mm length were prepared as substrate. Effect of current density were investigated during the electroplating process at 0.1, 0.3, 0.5, 0.9, 1.1, 1.5A and Watts bath solution were used. The samples were cut at the cross section, mounted it before ground using 220-1000 grit size paper and polished it. Specimen was placed at anode and cathode where the anode was Ni and the cathode was Ti. The Ni solution was placed inside the solution bath. The temperature was set up and controlled by using temperature control unit. After plating the samples, the deposits of Ni on Ti and the surface morphology was characterized by scanning electron microscope (SEM). Then, the graded composition was analyzed by energy dispersive X-ray dispersion (EDX) and X-ray diffraction (XRD) along the thickness direction of the deposit. Vickers indentations were made on the surface of polished samples with load of 49N along the cross surface as a function of distance along depth and Vickers hardness, HV and a loading time 15 s with an average of five measurements.

RESULTS AND DISCUSSIONS
X-Ray Diffraction pattern
The XRD patterns of nickel plating are shown in Figure-1 where it shows the crystallites nickel with orientation have strong (111) plane and second and third peaks of [1], (220) planes respectively. The preferred orientation was presented by the X-ray peak of (111) plane with other planes. The average crystallites size has been estimated by using Scherrer formula. Based on the XRD result, the peak of sample that plated at higher current density (1.1.45 A/cm\textsuperscript{2}) shows higher peak intensity than...
the sample that plate at lower current density (0.127 A/cm²). According to the Figure-1, the XRD peaks of nickel coating at current density 0.382 A/cm² were wider than current 0.127 A/cm². The peaks width increase with increasing the current density. The average crystallites size for all three samples is shown in Table-1. The table proves that, all the coatings are nanocrystalline and the crystallites size obtained by Scherrer formula decreased with increasing plating current density. XRD result shows particle size decreased as the applied current was increased to 0.9A (1.145 A/cm²). The diffraction peaks from both parameters revealed that the as deposited nickel coatings are highly crystalline with the preferential orientation along the (111) plane. The other prominent peaks in the pattern are [1] and (220) whereby their intensities were increase gradually with current density.

<table>
<thead>
<tr>
<th>Current (Amps)</th>
<th>Current density (A/cm²)</th>
<th>Crystallite size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>0.127</td>
<td>40.4</td>
</tr>
<tr>
<td>0.3</td>
<td>0.382</td>
<td>39.4</td>
</tr>
<tr>
<td>0.9</td>
<td>1.145</td>
<td>27.9</td>
</tr>
</tbody>
</table>

Surface Morphology of Nickel Plating on Titanium

Figure-2 shows SEM micrographs of surface morphology of sample nickel plating on titanium at different current densities. The surface morphologies shows decreasing grain size when current density increased up to 1.5A. Increase in current density resulted in higher over-potential that increase the nucleation rate. Based on the figure, increasing the current density would produce finer particles. The size of crystallites decreased with increasing current density and become smooth round shape structure. The particle at lower over potential (low current density) produce higher surface irregularities and roughed compare to the particle size at a higher over potential (current density) which produce a smooth surface and finer particle.

Cross-Sectional Microstructure Analysis

The thickness of nickel layers effects on current densities were measured from morphology analysis. Table-2 shows the measured thickness of nickel plated layers by using Watt’s solution. Three titanium samples were plated with nickel using Watt’s solution. Figure-3 shows the scanning electron micrograph of cross section of nickel plating on titanium at different current densities. As indicated in the figure, thickness of coating has a relation with current density which is increasing with increasing current density. Upon the increasing current density, the deposition rate of nickel on titanium increased, therefore the rate of plating also increased, lead to thickening of nickel layer. By increasing the current density, the thickness of nickel layer increased.
density, voltage (potential difference) decreased according to power equation, \( P = ivt \), where \( i \) is the current flow, \( v \) is the voltage and \( t \) is time. From Faraday’s first law of electrolysis, “The amount of any substance deposited, evolved, or dissolved at an electrode is directly proportional to the amount of electrical charge passing through the circuit”. The dissolution of anodes increased gradually with the amount of current density applied and longer electroplating time was utilized [16]. From the Faraday’s law equation it can be stated that for a certain time interval, when the current applied to the electroplating cell was increased, the amount of metal that was coated increased. Thus, thickness also increased with increased current density.

**Figure-2.** SEM images showing the effect of current densities on the grain size of (a) 0.3 A (b) 0.9 A (c) 1.1 A (d) 1.5 A at 4000 X magnification.
Figure-3. SEM image of cross section nickel plating on titanium using different current density of (a) 0.1 A (b) 0.3 A (c) 0.5 A.

Figure-4 shows a plot of coating thickness against current density. Thickness increase almost linearly as the current density is increased. Micro-hardness testing is done on the cross view of the samples which are measured along the cross-section perpendicular to substrate and coating deposition direction. Kendrick studied the properties of deposits from a Watt’s and sulphamate bath as a function of the current density and found that the hardness increased as the current density was increased by using Watt’s bath but the hardness decreased as the current density was increased by using sulphamate bath [17]. Figure-5 shows the graph of micro-hardness at cross section at with different current density.

<table>
<thead>
<tr>
<th>Speed (rpm)</th>
<th>Rate of solution movement (m/s)</th>
<th>Temperature (°C)</th>
<th>Time (minute)</th>
<th>Current (Amps)</th>
<th>Current density (A/cm²)</th>
<th>Average thickness (µm)</th>
<th>Rate of deposition (µm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>500 rpm</td>
<td>5.5 m/s</td>
<td>75 °C</td>
<td>3 minutes</td>
<td>0.1</td>
<td>0.127</td>
<td>19.4</td>
<td>6.47</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.3</td>
<td>0.382</td>
<td>23.9</td>
<td>7.97</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.5</td>
<td>0.637</td>
<td>27.8</td>
<td>9.27</td>
</tr>
</tbody>
</table>

Table-2. Effect of current density.
CONCLUSIONS

Plating thickness increased with higher current densities, also fine grained structures were obtained as the current density was increased. Hardness of the deposits decreased as the current density is increased. Nickel deposit at current densities below 1.5 A/cm$^2$ showed the better level of adhesion compared to the sample produced at current densities above 2.0 A/cm$^2$. The pitting appeared on the surface of nickel plating at 1.5 A/cm$^2$.

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REFERENCES


