A Comparison between the Effect of Heat Treatment and Plasma on the Wear Activities and Corrosion Resistance of Ni-B Electroless Coating with AISI 4340 Steel


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Abstract
The study the hardness, wear corrosion resistance of as-coating, heat treatment and plasma-nitriding of Ni-B electroless coatings was deposit on 4340 steel. After the process of coating, samples were plasma-nitriding in percentage hydrogen/nitrogen ratio (50%), in the temperature 400°C, at 4 hours were compared with heat treatment. Characterization by of FESEM, XRD, microhardness, Corrosion Resistance and surface roughness measurements. Microhardness was show highest hardness of 1050 HV was obtained for Ni-B plasma-nitriding formula whereas the greatest hardness of 800 HV was become for sample of Ni-B heat treated. Ni-B coating is used in the enhancement of properties of the corrosion of AISI 4340 steel is due to the thickness of electroless deposition, which is uniform, and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, it should be noted that the value of Ecorr is less negative and the rate of corrosion and Icorr is lower on the plated sample than on the uncoated sample. The rate of the wear for samples is decrement by plasma-nitriding with deposition time at (4h) and the temperature 400°C. Rate of wear for the materials depends not just into condition of wear test, but also with several properties of material and features as topography, hardness and friction coefficient.

1. Introduction
Plating technique by using of aqueous solutions obtain a great consideration due to the aims like easy of the coating technique, large rate of deposition, lower cost, uniform layer of plating [1-4]. The technique has feature like larger hardness, thickness, resistance wear, good solderability, good resistance of corrosion, deposit of amorphous or microcrystalline, lower coefficient of friction, lower resistivity and excellent magnetic properties [5-10]. Technique of Ni-B the electroless coating is obtaining to good the surface properties for larger types of substrates [11-12]. Commonly, technique of Ni–B electroless coating is thought to be better when compared with Ni-P coating also it more attractive in many industries [13-16]. Improved of mechanical properties, wear resistance and resistance of corrosion, and coatings of nanocomposite have great deal of attention. Nanocomposite coating has another appropriate identified solid lubricant like MoS₂ [17], PTFE [18], Cu-CNT [19], and Ni-P-CNT [20].

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The tensile strength for carbon nanotubes with elastic modulus, result it used in ceramic and metallic composites, In plasma nitriding, the nitrogen is presented to the surface of the substrate when being diffused with the metal. The energy of the large electrical voltage is formed on the plasma when atoms of nitrogen are accelerated to impinge on the substrate and Figure (1) Show diagram planner of plasma nitriding system [21-27]. Studying effects behaviour for Ni-B electroless coatings with Plasma nitriding and heat treatment coating on microhardness and roughness, resistance of corrosion and microstructure of 4340 steel.

**Figure (1).** Show diagram planner for the plasma nitriding system

**2. Experimental Procedure**

**2.1. Preparation of Substrate**

The substrate metal used is 4340 steel. Samples (diameter (20 mm) × height (10 mm)) were used as the base metal. The analysis of chemical for 4340 steel is shown Table (1).

<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Si</th>
<th>S</th>
<th>Mn</th>
<th>P</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>W%</td>
<td>0.36</td>
<td>0.29</td>
<td>0.01</td>
<td>0.67</td>
<td>0.01</td>
<td>0.81</td>
<td>1.3</td>
<td>0.15</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

All specimens were polished and grinded. Specimens were immersion in solution include (30g/L Na OH, 60g/L NaCO₃ and 60 g/L NaPbO₄ materials for one minute's period at 70°C temperature with moving the electrolyte with magnetic stirring by supply power (5 volt) caused remove any oil from surface of metal, the samples were dried and then, it directed immersion in solution of coating

**2.2. Electroless Bath Preparation**

After finishing fromsurfaces preparing for plating, the coating bath of electroless were prepared with concentrations explain in Table (2). Conducted for Ni-B electroless coatings in (1 hour, at 95 ± 1 °C). During coating, solution of the bath was mixed by a magnetic stirrer with decrease of the fluctuations of ionic concentrations. The sample was a monastery in two-way direction every 10 min to produce thickness of uniform coating. Figure (2) experimental part to electroless deposition process.
Table (2). Electroless bath conditions.

<table>
<thead>
<tr>
<th>Bath composition</th>
<th>g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>(NiCl₂·6H₂O -Nickel chloride)</td>
<td>24</td>
</tr>
<tr>
<td>(NaBH₄- Sodium borohydride)</td>
<td>0.8</td>
</tr>
<tr>
<td>(EDA -Ethylenediamine (98%))</td>
<td>60 ml/l</td>
</tr>
<tr>
<td>lead Nitrate</td>
<td>0.02</td>
</tr>
<tr>
<td>(NaOH- Sodium hydroxide)</td>
<td>90</td>
</tr>
<tr>
<td>SDS -Sodium Dodecyl Sulfate</td>
<td>2</td>
</tr>
</tbody>
</table>

**Condition**

| pH           | (12-14) OR 13 |
| Temp.        | 95 °C        |
| Time         | 1 HR         |

Figure (2). Experimental setup to electroless deposition process.

2.3. Heat Treatment and Plasma Nitriding for Plating Samples

Samples (Ni-B) were placed in a (5kW) direct current of plasma to increase PECVD chamber. Heat treatment process was conducted in vacuum furnace for electroless coating samples at 400 °C for 4 hours. Conditions for the plasma-nitrided treatment of electroless bath, was shown at Table (3). In the ending of the process, the specimens were gradually cool in the chamber to arrival the room temperature.

Table (3). conditions of plasma nitriding treatment.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Pulse Dc plasma nitriding (PPN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>400 °C</td>
</tr>
<tr>
<td>Pressure</td>
<td>10⁻³ torr</td>
</tr>
<tr>
<td>discharge voltage</td>
<td>400-500 V</td>
</tr>
</tbody>
</table>
### 2.4. Characterization

Microhardness of layers for the coating were calculated with vickers hardness tester, a load (25 g, 15 sec). Then, the average value was taken. Figure (3) show device of vickers hardness tester. Wear test was conducted by Pin-on-disc technique (ASTM G 99 standard). Calculate of specific wear rate was with: \( W_s = \frac{w}{lL} \), which \( L \) is the normal load, \( w \) is the mass loss and \( l \) is the sliding distance. The surface roughness value was calculated coated before and after treatment of heat using parameter \( Ra \) in \( \mu m \). In this work, Surface roughness was used to Calculated \( Ra \) with accuracy 0.05 \( \mu m \). The coatings were examined for the identification for the crystalline phase. Behavior of corrosion for the coatings was calculated by Tafel extrapolation technique.

<table>
<thead>
<tr>
<th>Frequency</th>
<th>8.9 kHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duty cycle</td>
<td>70%</td>
</tr>
<tr>
<td>Current(A)</td>
<td>2-3</td>
</tr>
<tr>
<td>Gas composition</td>
<td>(50%N₂:50%H₂)</td>
</tr>
<tr>
<td>Duration (h)</td>
<td>4</td>
</tr>
</tbody>
</table>

**Figure (3).** Vickers hardness tester.

### 3. Result and Discussion

#### 3.1. X-Ray Diffraction

XRD of plating Ni-B explain in Figure (4). XRD for deposited electroless plasma-nitrided Ni-B coating was shown in Figure (5) that an amorphous structure. Amorphous element as boron prevent the nucleation phase of nickel and formation of intermetallic compounds Ni₂B and Ni₃B, also boron nitride (BN) [28-30].

**Figure (4).** Patterns XRD for plated samples (Ni-B).
3.2. Surface Morphology

Figure (6) shows the microstructure of Ni-B coating sample and it is noted that microstructure was a cauliflower type [31, 32].

Figure (6). FESEM for electroless coated Ni-B.

Figure (7) FESEM of heat treatment the of Ni-B coating samples show in appearance are matte, also coarseness of microstructure [33].

Figure (7). FESEM for heat treated Ni-B.

Figure (8) FESEM of plasma-nitrided of Ni-B coating samples show are change of surface Ni-B to cauliflower microstructure with very clear [31, 34].
3.3. Surface Roughness
Data regarding the roughness of the base metal, to plated, annealed and plasma-nitriding specimens has been illustrated in Table (4). At noted, a plasma-nitried lead to a good increase in roughness and more than the annealing this fact is agreed with [35, 36].

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ra(µ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate</td>
<td>0.02</td>
</tr>
<tr>
<td>a plated- Ni-B</td>
<td>0.1</td>
</tr>
<tr>
<td>annealed- Ni-B</td>
<td>0.26</td>
</tr>
<tr>
<td>a plasma-nitrided- Ni-B</td>
<td>0.34</td>
</tr>
</tbody>
</table>

3.4 Microhardness
Figure (9) shows microhardness of the samples that underwent the plasma-nitrided and heat treatment. The affects the plasma-nitrided and heat treatment for coating its properties significantly. Hardness are enhanced by heat treatment and the plasma-nitrided. As a noted generally, because of changes of the thermally induced microstructural, and an effect on value of the hardness compared with the value as-deposited state this fact is agree with [37]. The increase of hardness is releated with Ni3B, Ni2B and BN phases [40].

![Figure 8](image.png)
**Figure (8).** FESEM for plasma-nitrided Ni-B.

![Figure 9](image.png)
**Figure (9).** Microhardness of coating, the plasma-nitrided and heat treatment.
3.5. Wear Behaviour

Figure (10a) indicates the relationship between distance the friction coefficient for sample of substrate. Figure(10b) indicate the relationship between distance the friction coefficient for samples of as-plated. Those behavior, and amorphous crystallization of a plated specimen during wear test because of generation of the heat which produce at occurrence of stresses of tensile at the interface of amorphous andcrystalline phases [26,32,41,42]. Figures (10) indicate the relationship between distance the friction coefficient of plasma nitriding and heat treatment specimen, show was decrease of the friction coefficient. Because, large microhardness value and Ni:B, Ni:B and BN phases.

![Figure 10](image)

Table (5) show of rate of specific wear, friction coefficient and mass loss of as-coated, annealed and plasma-nitrided samples. It can be noted in the plasma-nitriding Ni-B has the lesser rate of wear specifiedis related by the big mutual solubility of iron and nickel [43, 44].

**Table (5). Results of rate of specific wear, friction coefficient of samples.**

<table>
<thead>
<tr>
<th>(Sample)</th>
<th>(Coefficient of friction)</th>
<th>(Mass loss (mg))</th>
<th>(Specific wear rate) (g/N.M)x 10^-4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate</td>
<td>0.68</td>
<td>1.7</td>
<td>3.4E-4</td>
</tr>
<tr>
<td>a plated- Ni-B</td>
<td>0.61</td>
<td>0.5</td>
<td>1E-4</td>
</tr>
<tr>
<td>a annealed- Ni-B</td>
<td>0.55</td>
<td>0.45</td>
<td>0.9E-4</td>
</tr>
<tr>
<td>a plasma-nitried- Ni-B</td>
<td>0.45</td>
<td>0.2</td>
<td>0.4E-4</td>
</tr>
</tbody>
</table>

Figure (11) Analysis EDS of worn surface for samples, show to oxygen amount addition in plasma-nitriding samples [45].
3.6. Corrosion Behaviour

Table (6) include the results of test of electrochemical, $E_{corr.}$, $I_{corr.}$ and corrosion rate coatings, evaluated by Tafel extrapolation technique in solution of 3.5% NaCl.

**Table (6).** The results of test of electrochemical.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$E_{corr.}$ (mV)</th>
<th>$I_{corr.}$ (μA)</th>
<th>Corrosion Rate (mpy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Substrate</td>
<td>-571</td>
<td>49.49</td>
<td>6.9</td>
</tr>
<tr>
<td>(Nickel-Boron)a plated</td>
<td>-551</td>
<td>27.83</td>
<td>4</td>
</tr>
<tr>
<td>(Nickel-Boron) a annealed</td>
<td>-606</td>
<td>13</td>
<td>1.4</td>
</tr>
<tr>
<td>(Nickel-Boron) a plasma nitriding</td>
<td>-391</td>
<td>9</td>
<td>1</td>
</tr>
</tbody>
</table>

Potentiodynamic polarization curves measured on uncoated and NiB coated 4340 steel are shown on Figure (12). It is clear that the use of a Ni-B coating in enhancement of properties of the corrosion of AISI 4340 steel is due to thickness of electroless deposition is so uniform and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, the value of $E_{corr}$ is less negative, rate of the corrosion and $I_{corr}$ is lesser on the plated sample than uncoated 4340 steel are shown in Table(6) [46]. Graphic observation of the curves after various treatments are shown in Figure (12), which indicates less negative ($E_{corr}$) values caused by nitrogen diffusion [39].
Figure (12). polarization of Tafel curves of the (a) as-substrate; (b) Ni-B electroless coated samples. (c) Ni-B, annealed samples. (d) Ni-B, plasma nitriding samples.

3.7. Adhesion Test
Figure (13) shows SEM image of Rockwell C on the coating sample. long and deep cracks are noted around the indentation point in the coating sample. These results show that the coating sample has an acceptable adhesion strength, as shown in Figure (12 b), however the substrate has poor adhesion, show in Figure (12 a) [47].

Figure (13). Images SEM of Rockwell C effect an indentation on coating sample, (a) substrate and (b) coating.

4. Conclusions
the formation of BN and Ni2B, Ni3B phases by plasma-nitrided of electroless(Ni-B) coating, which increased microhardness. the surface roughness increased, but decreased mass loss with plasma nitride of coatings because the bigger hardness. plasma-nitriding process may be a good replacement for heat treatment. also plasma-nitrided os electroless Ni-B coating, produce diffusion of nitrogen atoms in matrix of Ni and creation of Ni2B, Ni3B, and BN phases, lead to increase the hardness. The big resistance of wear related with the sample of (Ni-B). These can be associated with the smaller size of grain. Ni-B coating is used in the enhancement of properties of the corrosion of AISI 4340 steel is due to the thickness of electroless deposition, which is uniform, and the cold of Ni-B coating to act as an active barrier between corrosion media and metal, it should be noted that the value of Ecorr is less negative and the rate of corrosion and Icorr is lower on the plated sample than on the uncoated sample.
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References


