Grain Structure and Roughness Characterization of Electrolytic Nickel Phosphorous, Electrolytic Nickel and Electroless Nickel Phosphorous on Copper by Focus Ion Beam Voltage Contrast

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Abstract
Impact of high temperature at back end assembly with temperature up to 400°C to electroless Nickel Phosphorous (NiP) and electrolytic Nickel (Ni) and Nickel Phosphorous (NiNiP) grain structure and surface roughness is normally ignored.

Focus Ion Beam Voltage Contrast, FIB, showed that the amorphous structure of electroless NiP did not change after the heating at 400°C. For electrolytic Ni, two layers of nickel were observed and for electrolytic NiNiP, the amorphous NiP layer did not change after the heating but the Ni layer changes to bigger grain structure.

Electroless NiP samples showed no significant differences on its surface roughness before and after heat treatment. However, significant difference observed on electrolytic Ni samples in all parameters tested. Increased surface roughness is generally observed at electrolytic Ni. For electrolytic NiNiP samples there was a slight difference on its surface roughness before and after heat treatment due to the structure change for Ni layer influencing the overall surface roughness.

It is concluded that heating at high temperature will change the grain structure of Ni but not the NiP layer and due to this, it changes the surface morphology and roughness of electrolytic Ni and electrolytic NiNiP but not to electroless NiP. Thus temperature in back end assembly process is critical to determine the grain structure, surface morphology and surface roughness.

1. Introduction
Understanding how the grain structure, surface morphology and surface roughness towards back end assembly process heat treatment for electroless NiP, electrolytic Ni and electrolytic NiNiP is limited. Nickel Phosphorous, NiP, coating is known to act as an excellent corrosion resistance layer\(^1\).

Grain structure will affect the leadframe surface structure. Without knowing the surface structure, it is quite impossible to understand the cleaning effectiveness. Generally, the rougher the surface is the harder for it to be cleaned. Improper cleaning will lead to quality issue\(^2\) as shown in Figure 1.

![Figure 1: (a) Tin peeling (b) Soldering failure (c) Dewet.](image)

In this study, FIB was used to understand the grain structure of nickel, Ni and nickel phosphorous NiP. Besides that FESEM was used to understand the surface morphology when the sample subjected to heat treatment.

2. Experimental
Sets of copper leadframe plated with electroless nickel phosphorous (NiP), electrolytic nickel (Ni) and nickel nickel phosphorous (NiNiP) taken from the leadframe store were used in this study. These leadframes were similar to the leadframe used in production mode. Each of the leadframe was exposed to heat at 400°C for 1 hour to simulate the average highest temperature exposure at backend assembly processes. Analysis were done on samples before and after heat treatment for comparison and better understanding on the changes of leadframe surface. Focus ion beam (FIB) was used to reveal the grain structure of the leadframes before and after heat treatment using Field of View (FOV) of 8 and 80 microns. Besides that, FESEM was deployed to understand the surface morphology when the sample subjected to heat treatment.

3. Result and Discussion
As shown in Figure 2 and Figure 3, for electroless NiP the amorphous structure and the thickness did not change after been subjected to heat treatment of 400°C\(^3\).

![Figure 2: Grain structure of electroless NiP before heat treatment (a) at FOV 80µm (b) at FOV 8µm with thickness of 4.023 microns.](image)
However, for electrolytic Ni, two layer of grain structures were observed. The top layer showed bigger grain structure with the thickness of 1.711 microns while the second layer maintain its original grain structure as before heat treatment with thickness of 0.609 microns. The thickness of electrolytic Ni before and after the heat treatment did not showed significant different where the thickness was 2.414 microns and 2.320 microns respectively. Refer to Figure 4 and Figure 5.

For electrolytic NiNiP, the NiP layer with amorphous structure did not change when subjected to heat treatment. The NiP thickness also did not showed significant different in the thickness where the thickness before and after heat treatment are 0.219 microns and 0.195 microns respectively. However, the Ni layer grain structure changed from small to big grain structure after been subjected to heat treatment but the thickness did not change dramatically that are 1.504 microns before the heat treatment and 1.391 microns after the heat treatment. Refer to Figure 6 and Figure 7.

Table I: Summary of grain structure condition before and after heat treatment.

<table>
<thead>
<tr>
<th>Leadframe Type</th>
<th>Heat Treatment At 400°C For 1 Hour</th>
<th>Before</th>
<th>After</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electroless NiP</td>
<td>4.023µm amorphous NiP observed</td>
<td></td>
<td>Not much changes of grain structure observed at 4.117µm amorphous NiP layer</td>
</tr>
<tr>
<td>Electrolytic Ni</td>
<td>2.414µm Ni layer with small grain structure observed</td>
<td>Two layers of Ni observed. 1.711µm of 1st Ni layer observed with bigger grain structure than before heat</td>
<td></td>
</tr>
</tbody>
</table>
Electrolytic NiNiP

<table>
<thead>
<tr>
<th>Layer Type</th>
<th>Deposited Material</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrolytic NiNiP</td>
<td>0.219µm NiP</td>
<td>Deposited on top of Ni layer with 1.504µm.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat</td>
<td>0.609µm of 2nd Ni layer with the similar grain structures as seen in before heat treatment.</td>
</tr>
<tr>
<td></td>
<td>0.195µm NiP remain the similar structure as before heat treatment. The 2nd layer of 1.391µm Ni layer observed bigger grain structure after heat treatment.</td>
</tr>
</tbody>
</table>

Investigation on the surface morphology using FESEM on electroless NiP showed no significant morphology changes observed before and after heat treatment. For electrolytic Ni, the surface showed a significant surface morphology changed after the heat treatment while for electrolytic NiNiP oxide growth was detected at the uneven curve opening. Refer to Figure 8, Figure 9 and Figure 10.

Figure 8: Electroless NiP (a) before heat treatment (b) after heat treatment.

Figure 9: Electrolytic Ni (a) before heat treatment (b) after heat treatment.

The same phenomenon also observed for the surface roughness where no surface roughness changed observed for electroless NiP. However, for electrolytic Ni and electrolytic NiNiP, there was a significant difference observed on the surface roughness before and after heat treatment, in all parameters tested. Box plot as shown in Figure 11 to Figure 15 confirmed these findings.

Figure 11: Quadratic roughness.

Figure 12: Rmax Maximum surface roughness.
4. Conclusions

In this study, FIB showed that heat treatment at 400°C for one hour did not change the amorphous structure of electroless NiP. No significant difference on its surface roughness and surface morphology as shown by FESEM.

Two layer of nickel grain structures developed for electrolytic Ni after the heat treatment. Bigger grain structure located at the top layer while smaller grain structure similar to before heat treatment was at the bottom layer. No significant different in the overall thickness between before and after the heat treatment. FESEM showed a significant surface morphology changed and significant difference observed on the surface roughness.

For electrolytic NiNiP, the amorphous layer of NiP did not change after heating but the Ni layer changed to bigger grain structure. FESEM showed oxide growth detected at the uneven curve opening. There is a significant different on its surface roughness between before and after heat treatment. No significant different in the overall thickness between before and after the heat treatment.

Thus, heat treatment affect the grain structure, surface morphology and surface roughness for electrolytic Ni and electrolytic NiNiP but not to electroless NiP. Any changes to be made to the temperature at the back end assembly process may have impact to the performance of the cleaning treatment prior to plating process.

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