Electroless nickel deposition onto silicon surfaces for micro and nanoelectronics applications and microtechnology processes

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We studied and characterized the processes of electroless nickel deposition in order to use it in microelectronics and microtechnology. We perform electroless nickel deposition on silicon surfaces with acid solution on palladium preactivated surfaces as well as alkaline solution without palladium preactivation. Electroless deposition was also performed on aluminum surfaces, deposited by evaporation onto silicon oxide. MOS capacitors were also fabricated to perform electrical characterizations of the structure (nickel-aluminum-oxide-silicon) using capacitance-voltage plots (C-V curves). In addition, we also presented the use of electroless nickel deposition for chemical sensors which were fabricated using conventional microelectronics techniques.

1 Introduction

Nowadays semiconductor materials are very important for many areas such as electronic components, integrated circuits, optical devices, sensors and others. However, the construction of the devices and circuits depend on many factors: quality of environment, purity of materials etc. Metallization processes are the final step on device fabrication and seriously affect its performance [1, 2]. Electroless Ni (EN) is a very known technique and has been used for selective deposition and, in many cases, is an alternative for metallization for back-end processes because it is a quick, low cost and low energy process. Therefore, nickel electroless plating can be an important step in the fabrication of microdevices and micromachines.

EN is a chemical reduction process which depends upon the catalytic reduction process of nickel ions in an aqueous solution (containing a chemical reducing agent) and the subsequent deposition of nickel metal without the use of electrical energy. The EN deposition is described by the following electrochemical classical reaction [3]:

\[
\text{Ni}^{2+} + \text{H}_2\text{PO}_2^- + \text{H}_2\text{O} \rightarrow \text{H}_3\text{PO}_4^- + 2\text{H}^+ + \text{Ni}^0. \tag{1}
\]

This reaction can occur in both alkaline or acid environment. In order to perform depositions by using acid solutions on silicon, the surface was pre-activated by pre-deposition of palladium. To obtain Ni ions, we used nickel chloride, and on the other hand, to reduce Ni ions, we used sodium hipophosphite. However, in this procedure it is very important to control the PH, therefore ammonium hydroxide (NH₄OH) and hydrofluoric acid (HF) were used to regulate alkaline or acid PH, respectively.

For deposition on aluminium surfaces (which is quickly corroded in alkaline solution) it was used an initial deposition by using acid solution and, after that, a deposition by using alkaline solution. In order to analyse the growth rates, different immersion times were used: 30 s, 60 s, 180 s and 300 s.

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2 Experimental procedures  For all experiments we used n-type silicon substrates, with resistivity in the range of 1 to 10 $\Omega\cdot$cm. Before deposition, the substrates were cleaned by conventional chemical processes [4]. Table 1 presents the solutions used for the electroless platings [5–7]:

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Surface</th>
<th>Solution</th>
<th>Pre-activation</th>
</tr>
</thead>
<tbody>
<tr>
<td>silicon</td>
<td>silicon</td>
<td>0.1 M NiCl$_2$ and 0.25 M Na$_2$PO$_2$, HF (PH ≅ 4), 60 °C</td>
<td>Palladium</td>
</tr>
<tr>
<td>silicon</td>
<td>silicon</td>
<td>0.1 M NiCl$_2$, 0.25 M Na$_2$PO$_2$, NH$_4$OH (PH ≅ 9), 85 °C</td>
<td>No preactivation</td>
</tr>
<tr>
<td>silicon</td>
<td>aluminium (deposited onto silicon oxide)</td>
<td>0.1 M NiCl$_2$ and 0.25 M Na$_2$PO$_2$, HF (PH ≅ 4), 70 °C</td>
<td>No preactivation</td>
</tr>
</tbody>
</table>

3 Experimental results  Figure 1 shows typical results of SEM, 5000x, sample inclination 60°, for each one of the three cases. Figure 1a shows nickel film on silicon surface, preactivated with palladium, by using acid solution. Figure 1b shows nickel film on silicon surface, without preactivation film, by using alkaline solution. Figure 1c shows the nickel film on aluminium surface, by using acid and alkaline solutions, as described in the previous section.

![Fig. 1 SEM images of nickel depositions](image)

In the two first cases, a good recovering of surface with regular film can be observed. In the latter case, referred to the deposition on aluminium, we clearly observe holes, probably due to aluminium corrosion process occurring during the depositions. In all cases rounded grains with diameters in the range of 0.5 $\mu$m to 0.8 $\mu$m were formed. We can observe no significant difference for both alkaline and acid solutions. Deposition on aluminium surface presented a higher roughness probably due to corrosion attack in the first deposition step.

4 Characterization of electrical measurement  In order to carry out CV measurements we used a CV tracer HP-4285 equipment with a set point frequency at 1 MHz. The CV characteristics of MOS capacitors were obtained before and after electroless nickel deposition. After the measurements, the parameters

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from the CV curves, maximum capacitance ($C_{MAX}$), oxide thickness ($t_{OX}$) and threshold voltage ($V_t$), were then extracted to analyse the influence of the electroless nickel deposition on the MOS structure. Figure 2 presents a typical capacitor measured before and after electroless deposition. For most capacitor measurements, including ours, a decreasing in maximum capacitance was observed while the other parameters remain constant. Therefore we can infer that the decreasing $C_{MAX}$ is caused by a lateral corrosion of aluminium during first deposition.

5 Conclusions

In this work, we investigated the EN deposition on silicon and aluminum surfaces under different conditions by SEM, RBS, and AFM techniques. The results show that the EN deposition results in sample films with low roughness and good adhesion. Moreover, we emphasize that the EN deposition is an easy procedure and of low cost. Various methods were tested. The results on Si surfaces show similar results for electroless deposition in both acid and alkaline solutions, with and without preactivation. For structures formed by nickel/aluminium/silicon RBS, AFM and SEM the analysis showed a good alternative for metallization process depositions on aluminum surfaces two steps, first in acid solution followed by immersion in alkaline solution, with good control to protect aluminium films with a nickel layer.

References