Introduction

Electroplating baths are made of many components, such as metals, salts, and additives. To obtain a proper plating the concentration of these components should be monitored. For example, the components and methods used to analyse acid copper bath are the following:

- Suppressor (S)
- Brightener (B)
- Leveller (L)

The Hull Cell requires very experienced operators and many tests, moreover it produces non-trivial results that are sometimes difficult to interpret.

For this reason, the Cyclic Voltammetry Stripping (CVS) method was developed in the past. This method is valid for simple baths but is not reliable for decorative applications baths. In addition, the analysis must be repeated several times to determine all additives. Gabrielli showed that with Electrochemical Impedance Spectroscopy (EIS) it is possible to obtain many information about the system with only a single measurement.

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>0.5</td>
</tr>
<tr>
<td>B</td>
<td>1.0</td>
</tr>
<tr>
<td>L</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Organic additives are mixtures that can be divided in three macro categories:

- Suppressors (S)
- Brighteners (B)
- Levellers (L)

Results

In the collected data we have identified differences both in the chronopotentiometries, which will be the subject of further investigations, and in the EIS spectra.

In particular, in the Nyquist diagram we found two capacitive loops, two inductive, and a third less evident capacitive loop. The first two are not substantially influenced by the variation of additives. The variations of S and L are evident, while those of B are less obvious.

Discussion

The obtained spectrum is not trivial because of the complex matrix and the simultaneous deposition of the metal. Starting from the Gabrielli studies, we were able to fit the experimental data with a equivalent electrical circuit.

In order to find a relationship between the complex formulation of the samples and the results obtained, a multivariate analysis was used.

Conclusions

- We found a correlation between r and the concentration of the additives.
- S and L produce large variation in the EIS, the effect of B is milder.
- Our goal has been achieved! Now we will carry out new experiments to increase the number of points and build a calibration surface for the determination of additives.

References


Acknowledgements

Regione Toscana and the Project ‘Intelligent and green Surface Coating’ funded by the POR Toscana 2014-2020. The authors gratefully acknowledge the laboratory at University of Firenze for the EIS measurements.