Advanced Engineered Solutions

A Global Leader in

Specialty Chemicals
Surface Finishing Equipment
Engineered Powders
Analytical Controls
Customer Requirements for Nickel Plating

Chelsea Edmonds
Lynne Michaelson
Introduction

Why Nickel?

- Corrosion Resistance
- Barrier Deposit
- Mechanical & Physical Properties
- Leveling Deposit
- Strength & Elongation
- Brightness
Nickel Deposition

Electroplating = Current Source

- Overall Faster Deposition
- Highest Throughput

Electroless = Chemical Rxns

- Can plate electrically isolated and non-conductive parts
- Can achieve greater uniformity

![Diagram of nickel deposition processes](image-url)
Nickel Electrodeposition

With Current Flow:
- Ni metal anode dissociates into Ni²⁺
- \( \text{Ni}^{++} + 2 \text{e}^- = \text{Ni} \) metal at cathode
- Ni²⁺ replenished by anode

\[ \text{Cathode Current Efficiency} = \left( \frac{\text{Exp't wt. gain}}{\text{theor. wt.}} \right) \times 100 \]
\[ \text{Anode Current Efficiency} = \left( \frac{\text{Anode wt. loss}}{\text{theor. loss}} \right) \times 100 \]

Cathode Eff. < Anode Eff.
- Increasing Ni²⁺
- Increasing pH

\( \uparrow \text{Ni}^{++} \text{ offset by drag out} \)
\( \text{pH requires constant adjustment} \)
Anodes

Function:
  o Conduct & Distribute Current Uniformly
  o Soluble anode replaces Ni ions

Types: Inert vs. Soluble
  o Soluble = Pure Grade & Activated Sulfur
    Activated sulfur required in chloride & bromide free solutions
  o Inert = mixed metal oxide, platinum

Position:
  o Anodes should be a little shorter and positioned away from the edge of the plating rack.
  o Soluble anode bars dissolve bottom up, giving poor thickness distribution from top to bottom.
Typical bath components for Ni Electroplating

- **Nickel ion source**: Nickel Sulfate, Nickel Sulfamate
- **Sol’n Conductivity & anode dissolution**: NiCl, NiBr
- **Buffer / pH control**: Boric Acid*
- **Grain Refiner / stress reducer**: Carriers (aromatic organic sulfur compounds)
- **Ductility & Leveling**: Brighteners (low conc. Consumed by electrolysis)
- **Anti-Pitting Agents**: Wetting Agents that lower surface tension

*Boric acid is on the candidate list for substance of very high concern (SVHC) so new formulations are being developed to eliminate boric acid.
Types of Ni Electroplating

Barrel Plating
- Components tumble freely without nesting or locking together
- Barrel loading should be <50% of barrel volume
- Carrier = high, Brightener/Leveler = low
- Mesh size of barrel as large as possible

Rack Plating
- Parts are loaded into racks and held in place
- Rack makes electrical contact with part – design of contact points is critical
- Quality of plating impacted by arrangement of rack in plating tank; i.e. anode to cathode spacing, solution flow, etc.
# Application of Different Ni Baths

<table>
<thead>
<tr>
<th>Decorative</th>
<th>Functional</th>
<th>Electroforming</th>
<th>Activation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Watts Nickel</strong></td>
<td><strong>Electroless Nickel</strong></td>
<td><strong>Sulfamate Nickel</strong></td>
<td><strong>Wood’s Nickel Strike</strong></td>
</tr>
<tr>
<td>Nickel Sulfate, Nickel Chloride, Boric Acid</td>
<td>Nickel Sulfate, Hypophosphosphate</td>
<td>Nickel Sulfamate, Nickel Bromide, Boric Acid</td>
<td>NiCl, HCl</td>
</tr>
</tbody>
</table>

https://www.shimiferz.com/services/electro-forming/

Watts Nickel Bath Formulation

*By Professor Oliver P. Watts in 1916*

<table>
<thead>
<tr>
<th>Operating Parameters</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfate</td>
<td>35.0 to 45.0 oz./gal</td>
</tr>
<tr>
<td>Nickel Chloride</td>
<td>6.0 to 12.0 oz./gal</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>4.0 to 6.0 oz./gal</td>
</tr>
<tr>
<td>pH</td>
<td>3.5 to 4.5</td>
</tr>
<tr>
<td>Temp.</td>
<td>40° - 60° C</td>
</tr>
<tr>
<td>Current Density</td>
<td>20 to 70 ASF</td>
</tr>
</tbody>
</table>

*Decorative & Functional Products*
Wood’s Nickel Strike Formulation

<table>
<thead>
<tr>
<th>Operating Parameters</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Chloride</td>
<td>10.0 to 20.0 oz./gal</td>
</tr>
<tr>
<td>Hydrochloric Acid</td>
<td>5.0% to 15.0% by volume</td>
</tr>
<tr>
<td>Temp.</td>
<td>RT</td>
</tr>
<tr>
<td>Current Density</td>
<td>voltage enough to cause gassing</td>
</tr>
</tbody>
</table>

*Activation of nickel and nickel alloys such as Inconel and stainless steel*
Sulfamate Nickel Formulation

**Operating Parameters**

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfamate</td>
<td>8.0 to 12.0 oz./gal</td>
</tr>
<tr>
<td>Nickel Bromide</td>
<td>1.0 to 1.5 oz./gal</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>3.0 to 5.0 oz./gal</td>
</tr>
<tr>
<td>pH</td>
<td>3.0 to 4.5</td>
</tr>
</tbody>
</table>

*Functional and electroforming due to low stress*
Stress

Develops from electro crystallization and/or the co-deposition of impurities

Tensile Stress
Cracking: Deposit tries to contract

Compressive Stress
Blisters: Deposit expands

Watts Nickel Solution w/o additives = 125 to 185 MPa Tensile
(Sulfamate Ni Solutions can have lower stress)

Sulfur-containing organic additives (saccharin), carriers, & secondary’s help form compressively stressed Ni deposits

(2) Y. Oda et al, IPC 2009, p.4
Impurities Introduced into Plating Baths

- Insoluble: Dust abrasives and anode fines that cause roughness
- Metallic: Parts dropped into the tanks, drag in from chemistries upstream, leaching agents from resist and rack coatings
  
  *Can be observed on hull cell panels as a dark cloudy haze starting from the low current density areas*

- Organic: Oil and grease dragged in from the cleaner or not cleaned off in the first place
  
  *Can be observed on hull cell panels as a cloudy light colored haze starting from the low current density areas.*
Purification Procedures

- Continuous filtration to minimize roughness.
- Low current density electrolysis.
- High pH treatments to help precipitate iron, aluminum and silicon at a pH of 5.0 to 5.5.
- Removal of organics by using activated carbon.
- Hydrogen peroxide or potassium permanganate can be used to help with the carbon treatment.
Leveling & Brightness

Leveling:
What? plated metal preferentially fills in defects & scratches on the surface

How? Organic additives in plating sol’n adsorb on micropeaks limiting current flow while increasing current density in microgrooves

Brightness:
Combination of leveling, grain refining, and crystal growth.
Engineering & Functional Testing

Thickness testing:
- Microscopic examination of cross sections.
- Kocour de-plate
- Beta Backscatter
- XRF – X-ray Fluorescence
- Weight gain per the measured surface area

Adhesion testing:
- Bending, twisting, and tape testing.
- Thermal shock, for steel 300° C and zinc alloys 150° C and quench.
Engineering Properties

Ductility: *Ability of a plated deposit to undergo deformation without cracking*

Test: 1 mil Ni deposit on Cu foil. Bend 180° over a 12 um mandrel and look for cracks down to base material

- Additive free deposits have elongation ~30%
- Semi bright deposit have elongation ~8%

Corrosion: *Corrosion resistance may depend on deposit thickness*

Test: Salt Spray box, Fuming Nitric Test in Desiccator

- > 5 um for use under gold & other coatings
- ~125 um for severe applications; i.e. bumpers & auto wheels
Thank you