Electrolyte Maintenance Technology Platform: Applying Learning Across Electrochemical Machining and Stripping Processes

“...to be known as the company that changed the focus of electrochemical engineering from the art of complex chemistries to the science of pulse/pulse reverse electric fields...”

~50+ Patent Portfolio

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Starting from scratch for every development activity is time- and resource-consuming

Effective R&D applies lessons learned from one project to another

Expands commercialization potential

Technology platform of pulse-reverse waveforms combined with unique cell designs
Industrial Electrolyte Management

- Electrolyte management is **critical** to avoid excess costs associated with replacement and waste disposal.

- Faraday is developing electrolyte management technologies:
  1. **Recycling Electrochemical Machining ((R)ECM)** to enable a zero-discharge process,
  2. **Stripping/Recycling** of the components of a **High Velocity Oxy-Fuel (HVOF)** coating, and
  3. **Chrome Stripping** that does not form hexavalent chromium.

- Lessons learned in each project are applied to the other projects to accelerate and enhance the chance of success.
Basis of Technologies: Pulse Reverse Waveforms

“Tuned” to:
- Machine/Strip coating
- Control speciation
- Enhance mass transfer
- Control current distribution

Provides fundamental guidance
NOT Predictive theoretical model


Cathodic Pulse “Tuned” to:
- Reduce oxide/depassivate surface
- Control metal ion speciation
Prior Work – PRC Electrochemical Machining

- Machining
- Electropolishing
- Deburring
- Radiusing

Ni alloys, Ti alloys, Al Alloys
Stainless Steels
Steel
Cu,
Mo, Nb, Ta alloys
Co-Cr
Prior Work – Pulsed Electrowinning

• Evaluation of pulsed fields on silver recovery for Swagelok
• **Direct-current winning:** Lower plating efficiency, poor plate adhesion
• **Pulsed-current winning:** Improve plating efficiency / adhesion

**Ag:** ~200 ppm → < 1 ppm  
Ni: 322 ppm → 171 ppm  
Fe: 14.2 ppm → 3.8 ppm  
Cu: 2.2 ppm → 0.05 ppm  
Cd: 2 ppm → 0.02 ppm  
Cr: 4 ppm → 0.3 ppm  
Pb: 29 ppm → 0.13 ppm  
Zn: 5 ppm → 0.3 ppm
Combined Prior Work to Create (R)ECM

- **DC ECM:**
  - Large volume of sometimes hazardous waste (300x)
  - Metal ion buildup adversely affects performance

- **Recycling ECM (R)ECM:**
  - Combined PRC ECM and PC EW
  - Metals are recovered
  - Waste is avoided
  - Water usage is minimized

(R)ECM: Lesson Learned

1. Screen electrolytes for electrowinning performance
2. Develop PC/PRC parameters for ECM from EW electrolyte
3. Develop PC parameters for EW
4. Integrated (R)ECM system testing
(R)ECM $\alpha$-Scale System

Cell design gave flexibility to investigate various anode to cathode gaps / # / material
Machining SAE 4150: Maintain [Fe] “target” 2000 mg/L by adjusting EW unit operation
- # Cathodes: 3 to 2
- EW current density: 11 to 18 A/dm²
Maintain \([\text{Ni}], [\text{Fe}], [\text{Mo}]\) by sequential operation of ECM and EW unit processes.
LESSONS APPLIED:

• Pulsed Current to:
  – Increasing machining rate and improve surface finish

• Pulsed ElectroWinning to:
  – Reclaim Fe, Ni, Mo, Cu metal
  – Extend machining electrolyte lifetime to decrease operating/disposal costs

LESSON LEARNED:

• Select ElectroWinning electrolyte first
  – Use EW electrolyte for machining

• Primary current distribution important for efficient removal of metal ions
Transition from Machining to Stripping/Recovery

• Why remove metallic coatings?
  1. Reclaim parts with defective or damaged coating
  2. Overhaul parts damaged during operation
  3. Remove undesired metal deposits from plating fixtures

• Based on (R)ECM results, Faraday identified an opportunity:
  – Strip High Velocity Oxy-Fuel (HVOF) WC-Co coatings
  – Recover components for recycling

• Efficient HVOF coating removal requires:
  – Increase stripping rate & decrease part tank time (72 hrs)
  – Increase stripping solution lifetime
  – Reclamation of stripped metals (currently not done)
HVOF Stripping/Recycling / Lesson Applied from (R)ECM

1. Screen electrolytes for electrowinning performance
2. Develop PC/PRC parameters for Stripping from EW electrolyte
3. Develop PC parameters for EW
4. Integrated Stripping/Recycling system testing

BUT: Limited by Bureaucratic Constraints
Not Allowed to Change the Mil-Spec Stripping Electrolyte

**CONSTITUENTS**

<table>
<thead>
<tr>
<th>Component</th>
<th>Specification</th>
<th>Concentration (Optimum)</th>
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</thead>
<tbody>
<tr>
<td>Sodium Citrate (aka trisodium citrate dihydrate)</td>
<td>N/A (Na₃C₆H₅O₇ • 2H₂O)</td>
<td>(0.8 lb/gal makeup)**</td>
</tr>
<tr>
<td>Sodium Percarbonate (aka sodium carbonate peroxhydrate)</td>
<td>N/A (2Na₂CO₃ • 3H₂O₂)</td>
<td>(0.4 lb/gal makeup)**</td>
</tr>
<tr>
<td>pH</td>
<td></td>
<td>9.0-11.0 (10.0-10.6)**</td>
</tr>
</tbody>
</table>

REFOCUS: Maintain Electrolyte by pH Adjustment and Eliminating Peroxide
Lesson Applied: Pulse Current Improved Rate

<table>
<thead>
<tr>
<th>Elapsed Time (min)</th>
<th>Average Stripping Current (A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before</td>
<td>0.0</td>
</tr>
<tr>
<td>16 min</td>
<td>0.5</td>
</tr>
<tr>
<td>60 min</td>
<td>1.0</td>
</tr>
<tr>
<td>150 min</td>
<td>1.5</td>
</tr>
<tr>
<td>200 min</td>
<td>2.0</td>
</tr>
<tr>
<td>210 min</td>
<td>0.0</td>
</tr>
</tbody>
</table>

- DC: Solid line
- PC: Dashed line
HVOF Beaker Tests – Summary

- Complete stripping in as low as 3 to 4 hours (24 h in all cases)
  - Pulsed current showed an increase in stripping rate over DC
  - Peroxide slightly increased stripping rate and only within first few hrs
  - pH could be maintained with NaOH

- Recovery of Cobalt on cathode
  - W recovery to be demonstrated

- Coatings strip edges → center
  - Primary current distribution important
  - Need to scale up to larger cells
Lesson Applied: Used (R)ECM EW cell design to accelerate program
HVOF Stripping

- Current distribution significant for stripping performance
  - Parts ‘screened’ from cathode strip much more slowly
- Parts photographed after 4 h processing
- All parts stripped completely within 24 h
(R)ECM Design – Lesson Applied

**Current Process**
- Direct Current Power Supply
- Stripping Electrolyte with Hydrogen Peroxide

**Enhanced Process**
- Pulse Current Power Supply
- Stripping Electrolyte **without** Hydrogen Peroxide
**LESSONS APPLIED:**

- Pulsed Current:
  - Decrease stripping time from 72 hours to as little as 3 to 4 hours

- Pulsed ElectroWinning to:
  - Reclaim Cobalt metal for recovery
  - Could return stripping bath to stripping process to decrease operating/disposal costs

- (R)ECM cell design
  - Better current distribution for more efficient stripping

**LESSON LEARNED:**

- Understand bureaucratic constraints (electrolyte) early
Chrome Stripping

- Electrolytic stripping of Cr:
  - NaOH (60 g/L) + Na₂CO₃ (75 g/L) at 4-6 V
  - Operating conditions favor Cr⁶⁺

- Phase I:
  - PRC increased conversion of Cr⁶⁺ to Cr³⁺
  - *Stripping Process worked with oxalic acid – no Cr⁶⁺ in solution*

- Phase II:
  - *Oxalic acid incompatible with client waste treatment system*
  - Need to find another electrolyte
  - Alternative approaches
    - Electrowinning of Cr
**Cr Stripping/Recycle – Lessons Applied/Learned**

**LESSON LEARNED:**
- Understand bureaucratic constraints (waste treatment) early

**LESSON BEING APPLIED:**
- Use Electrowinning to recover Cr to maintain stripping electrolyte

**RECENT LESSON LEARNED:**
- May have identified an electrolyte that can plate chrome
- Feed back to (R)ECM
Summary of Lessons Learned/Applied

- **Pulse reverse ECM**: Use PRC to improve rate and surface finish. Pulse improves efficiency.

- **(R)ECM Ni, Mo, Fe, Cu**: Use EW to recover value. May have electrolyte that can plate chrome.

- **Efficient cell design**: Select EW electrolyte first.

- **HVOF Stripping-Recycling**: Use PRC to improve rate.

- **Select EW electrolyte first**: Understand Bureaucratic Constraints.

- **Oxalic Acid Incompatible WT**: Phase I Cr Stripping. Use EW to recover value. Select EW electrolyte first.

- **Phase II Cr Stripping-Recycling**: Use EW to recover value.
Common Themes/Lessons Learned

- Racking and fixturing design critical to effective process
  - (R)ECM tank design used for Cr and HVOF stripping/recovery
  - Don’t assume industrial client is cognizant of primary current distribution constraints

- Create added value by recovery of metals
  - Cr stripping did not require this, but should add value

- Design stripping electrolyte to facilitate electrowinning
  - (R)ECM strategy
  - Being applied to Cr Stripping

- Transition process from lab to depot
  - Learn bureaucratic lessons in dealing with large organizations
  - Patience is required
Acknowledgements

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