The Evaluation of On-line Performance Test Methods for Aqueous Cleaner for Metal Surface Finishing Processes

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Due to environmental regulations, the use of Ozone Depleting Chemicals (ODC’s) is being phased out. To meet this new regulation, Lockheed Missiles & Space Company is currently using an aqueous cleaner to replace 1,1,1 trichloroethane, an ODC, to prepare aluminum surface prior to adhesive bonding, conversion coating, or anodizing.

Currently a vendor recommended titration method is being used to monitor and control the aqueous cleaner. This titration method only measures the alkalinity of the bath. To better control the process, a method of measuring the performance of the bath is desired. This test should be able to indicate the performance of the bath on a numeric scale, and be quick, simple and inexpensive.

This presentation consists of two parts. The first part evaluates three different methods of measuring contaminant adhering to the test panels. The methods are:

- Weight Gain
- Optically Stimulated Electron Emission (OSEE)
- Nonvolatile Residue (NVR)

The second part of the study of the presentation evaluates the use of these methods to control the aqueous cleaner.
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Two Part Study

- Dynamic range of the 3 methods
- Application of the test methods on an Aqueous cleaner
Test Methods

• Objective: Evaluate performance test methods
  - Weight Change
  - Non-Volatile Residue (NVR)
  - Optically Stimulated Electron Emission (OSEE)
How OSEE Works

UV

Contaminant or dielectric surface

Metallic Substrate

e-

UV

e-
Preparation of Panels for Wt. Gain, OSEE, & NVR Analysis

1. Aq. Cln.
2. Alk. Cln
3. Deoxidize
4. Scotch Brite
5. OSEE >900
6. TCA/EtOH + contam.
7. OSEE
8. Wt. Panels
9. NVR
## Composition of the Contaminant Mixture

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Make Up (% wt/wt)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rust Lick</td>
<td>40</td>
</tr>
<tr>
<td>Acculube</td>
<td>40</td>
</tr>
<tr>
<td>Tap Magic</td>
<td>10</td>
</tr>
<tr>
<td>Aluma Cut</td>
<td>10</td>
</tr>
</tbody>
</table>
Weight Gain vs. Weight of Dissolved Contaminant
OSEE Signal vs. Weight Gain

Response vs. Wt Gain mg/sq ft

OSEE

NVR
### NVR Concerns

<table>
<thead>
<tr>
<th>Wt. Gain</th>
<th>NVR</th>
<th>Wt. After NVR</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (0.2)</td>
<td>0.18 (0.24)</td>
<td>0.1 (+0.2)</td>
</tr>
<tr>
<td>1.8 (0.5)</td>
<td>0.12 (0.16)</td>
<td>0.5 (0.4)</td>
</tr>
<tr>
<td>3.3 (0.3)</td>
<td>1.38 (0.33)</td>
<td>0.37 (0.07)</td>
</tr>
<tr>
<td>5.3 (0.8)</td>
<td>2.06 (0.63)</td>
<td>1.0 (0.3)</td>
</tr>
<tr>
<td>7 (1)</td>
<td>4.3 (1.2)</td>
<td>0.2 (0.2)</td>
</tr>
<tr>
<td>9.8 (0.8)</td>
<td>5.22 (0.8)</td>
<td>1.1 (0.4)</td>
</tr>
</tbody>
</table>

All Units are in mg/sq ft
Conclusion
Dynamic Ranges of the 3 Test Methods

NVR

OSEE

Wt. Gain

Wt. of Contaminate (mg/sq ft)
Application of 3 Test Methods on an Aq. Cleaner
## ‘Clean’ vs. ‘Dirty’ Panel

Wt. of Conta. Deposit
- ‘Clean’ = 0 mg/sq ft
- ‘Dirty’ = 14.2 mg/sq ft

<table>
<thead>
<tr>
<th>[Contaminant] g/l</th>
<th>Wt. Gain (mg/sq ft)</th>
<th>OSEE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Clean</td>
<td>Dirty</td>
</tr>
<tr>
<td>0</td>
<td>0.6</td>
<td>0.8</td>
</tr>
<tr>
<td>5</td>
<td>1.1</td>
<td>0.8</td>
</tr>
<tr>
<td>10</td>
<td>0.6</td>
<td>0.5</td>
</tr>
<tr>
<td>20</td>
<td>1.4</td>
<td>1.3</td>
</tr>
<tr>
<td>30</td>
<td>1.1</td>
<td>1.3</td>
</tr>
</tbody>
</table>
Panel Weight Change vs. Contaminant Loading in Cleaner

Wt Gain mg/sq ft

[Contaminant] g/L
OSEE Response vs. Contaminant Loading in Cleaner
# Effect of Agitation

## Process vs. Rinse H2O

<table>
<thead>
<tr>
<th>Flow rate (a)</th>
<th>‘Clean’ Panel Wt1 (std. dev.)</th>
<th>‘Clean’ Panel OSEE (std. dev.)</th>
<th>‘Dirty’ Panel Wt2 (std. dev.)</th>
<th>‘Dirty’ Panel OSEE (std. dev.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.4 (0.3)</td>
<td>837 (96)</td>
<td>1.2 (0.2)</td>
<td>806 (119)</td>
</tr>
<tr>
<td>1</td>
<td>0.6 (0.2)</td>
<td>593 (71)</td>
<td>0.60 (0.2)</td>
<td>552 (74)</td>
</tr>
<tr>
<td>3.8</td>
<td>1.0 (0.4)</td>
<td>665 (210)</td>
<td>0.8 (0.3)</td>
<td>804 (121)</td>
</tr>
<tr>
<td>Average</td>
<td>1</td>
<td>700</td>
<td>0.9</td>
<td>720</td>
</tr>
</tbody>
</table>

### Agitated Rinse

<table>
<thead>
<tr>
<th>Flow rate (a)</th>
<th>‘Clean’ Panel Wt1 (std. dev.)</th>
<th>‘Clean’ Panel OSEE (std. dev.)</th>
<th>‘Dirty’ Panel Wt2 (std. dev.)</th>
<th>‘Dirty’ Panel OSEE (std. dev.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>3 (1)</td>
<td>231 (44)</td>
<td>4.4 (0.9)</td>
<td>233 (67)</td>
</tr>
<tr>
<td>1</td>
<td>1.3 (0.2)</td>
<td>397 (118)</td>
<td>1.1 (0.3)</td>
<td>145 (53)</td>
</tr>
<tr>
<td>3.8</td>
<td>2.0 (0.3)</td>
<td>205 (46)</td>
<td>2.3 (0.4)</td>
<td>212 (61)</td>
</tr>
<tr>
<td>Average</td>
<td>2</td>
<td>278</td>
<td>2.6</td>
<td>197</td>
</tr>
</tbody>
</table>

### Stagnate Rinse

a) Flow rate of cleaner L/min sq ft

b) Weight of contaminant (mg/sq ft) left on panels after being processed by cleaner
Conclusion

- Clean to equilibrium
- Agitation of Process Solution is not important
- Agitation of Rinse is critical
- Wt. Gain: suitable, > 2 mg/sq. ft
- OSEE: most sensitive, 1 – 10 mg/ft²
- NVR: least desirable