EVALUATION OF SOLDERING AND WASHOUT IN HEAT RESISTANT MATERIALS FOR DIE INSERTS

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Cleveland, June 11, 2002
OBJECTIVES

• Evaluate thermal fatigue resistance of nickel and cobalt based superalloys (718, 706, 625 and Incoloy 909).

• Develop a soldering/washout test with a molten aluminum jet impinging on the test piece. H13 serves as control/baseline material.

• Evaluate soldering/washout of copper, nickel base and refractory alloys with good thermal fatigue properties.

• Evaluate soldering/washout of nitro-carburized H13 pins.

• Evaluate soldering/washout of PVD coated H13 pins.
Evaluation of nickel-based alloys for use as inserts in die casting applications.

JUSTIFICATION

Nickel-based superalloys have high strength at high temperatures and superior thermal fatigue resistance. They are widely used in turbine blade applications and commercially available.
Dissolution at the Corners of Nickel Alloy Specimens
CONCLUSIONS

• While they possess good thermal fatigue resistance, nickel-containing superalloys are prone to dissolve in molten aluminum when overheated.

• In order to be utilized in die casting, these alloys require a protective coating yet to be identified or developed. Conversely, better cooling with internal cooling lines closer to the surface would be required.
UBE VSC 315 Ton Squeeze Cast Machine at CWRU
Schematic Diagram of the Accelerated Soldering Test

- Molten Aluminum
- Shot sleeve
- Gate
- Test pin
- Die cavity
- Plunger
Schematic of the Washout/Soldering Testing Set-up

- Cavity
- Test Pins
- Plunger
- Shot Sleeve
- Al jet from slit ca. 300 in/sec
Sub-Insert for Soldering&Washout Experiments

The hard H-13 pin is coated with a silvery soldered layer of Al.
Test Pin Design and Position

- Pin location
- Biscuit
- Runner
- Thin gate creating jet of molten metal
- Test pin
- Casting

Dimensions:
- 5
- 19
- 30
- 5
- 10
Results of Soldering & Washout Experiment

- 50 Shots
- 100 Shots

Images show samples subjected to different numbers of shots and materials, highlighting the effects on various alloys.
### Chemical Composition of Copper Base Pin (wt%)

<table>
<thead>
<tr>
<th>Alloy Type</th>
<th>Be</th>
<th>Co</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu Base</td>
<td>1.7</td>
<td>0.2</td>
<td>98.1</td>
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</table>

### Chemical Composition of Pins (wt%)

<table>
<thead>
<tr>
<th>Alloy Type</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>Fe</th>
<th>Ni</th>
<th>Nb</th>
<th>V</th>
<th>W</th>
<th>Hf</th>
<th>Zr</th>
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<tbody>
<tr>
<td>Ni-718</td>
<td>0.02</td>
<td>0.05</td>
<td>0.05</td>
<td>17.6</td>
<td>2.86</td>
<td>1.01</td>
<td>0.48</td>
<td>18.7</td>
<td>53.6</td>
<td>5.09</td>
<td></td>
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<tr>
<td>Ni-625</td>
<td>0.052</td>
<td>0.06</td>
<td>0.2</td>
<td>20.9</td>
<td>8.45</td>
<td>0.32</td>
<td>0.23</td>
<td>4.38</td>
<td>61.1</td>
<td>3.36</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H13</td>
<td>0.4</td>
<td>0.35</td>
<td>1</td>
<td>5.25</td>
<td>1.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mo-785</td>
<td></td>
<td></td>
<td></td>
<td>97.5</td>
<td>0.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.18</td>
<td></td>
<td>0.13</td>
<td></td>
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<tr>
<td>Ti-6Al-4V</td>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td>6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Anv. 1150</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td></td>
<td></td>
<td>2</td>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td></td>
</tr>
</tbody>
</table>
TOTAL CRACK AREA AFTER 15,000 THERMAL FATIGUE CYCLES (1"x1"x7")
AVERAGE MAXIMUM CRACK LENGTH AFTER 15,000 THERMAL FATIGUE CYCLES (1"x1"x7")

Test Materials

- P.G. H13/Oil/49Rc
- Bohler W303/Oil/45Rc
- Brush Cast Nybril EX/44Rc
- Brush Wrought Nybril EX/44Rc
- Brush Cast Nybril 360/35Rc
- Brush Cast Nybril 360-1/34Rc
- Brush Cast Nybril 360-2/35Rc
- Bohler W100/44Rc
- Kind RPU1/48Rc
- Kind TQ1/48Rc
- Brush QMAX/24Rc
- CSM PM Mo/20Rc
- CMW Anviloy 1150/37Rc
- Kulite Kuldie/33Rc
- Allvac IN718/46Rc (Pitting Depth)
Broken Pins

- Cu-Alloy: 3 Shots
- Grey Cast Iron: 3 Shots
- Ti-6Al-4V+10% TiC: 59 Shots
Appearance of Pins after 50 Shots
(with Soldered Al Dissolved in NaOH)

Hard-H13

Ti-6Al-4V

Mo-785

Anvilloy 1150
Washout in the Hard H13 Pin Impinged Directly by the Al (50 shots)
Effects of Pin Material on Soldering

Weight of Soldered Al on Pin Surface (g)

Hard-H13 Base
Soft-H13
Ni-625
Ni-718
Mo-Base
Ti-Base
Anv.1150
Intermetallic and Soldered Layers on a H13 Pin[1]

Interfacial Morphology and Microhardness Profile of the Reaction Zone between H21 Steel and Molten A380 Alloy\(^2\)

*(Rotating at 1292°F with a Speed of 300 rpm for 9 Hours)*

![Image of interfacial morphology and microhardness profile]

Comparison of Microhardness of Intermetallic compounds [3]

<table>
<thead>
<tr>
<th>Layer</th>
<th>Microhardness (HV, GPa) [30]</th>
<th>Layer</th>
<th>Microhardness (HV, kg/mm²) [5]</th>
<th>Layer</th>
<th>Microhardness (HV, kg/mm²) [27]</th>
</tr>
</thead>
<tbody>
<tr>
<td>18Cr-10Ni stainless steel (in pure Al)</td>
<td>1.8 ± 0.2</td>
<td>H12 tool steel (in A380)</td>
<td>239</td>
<td>1121 tool steel (in A380)</td>
<td>271</td>
</tr>
<tr>
<td>Fe₂Al₅-type</td>
<td>8.9 ± 0.9</td>
<td>Fe₃Al₂-type</td>
<td>985</td>
<td>Fe₃Al₂-type</td>
<td>974</td>
</tr>
<tr>
<td>FeAl₃-type</td>
<td>4±3 to 3.06</td>
<td>Fe₃Al₂-type</td>
<td>1096</td>
<td>Fe₃Al₂-type</td>
<td>403</td>
</tr>
<tr>
<td>pure Al</td>
<td>0.6 ± 0.1</td>
<td>A380 alloy</td>
<td>100</td>
<td>A380 alloy</td>
<td>94</td>
</tr>
</tbody>
</table>

*1 GPa ≈ 100 kg/mm².


Relationship between Surface Roughness and Percentage of Area Solder/Build up after 50 Shots for PVD TiN, CrN, TiCN, Nitrided and Uncoated core Pins [4]

Effects of Pin Material on Washout

Determined by weighing the pins before and after shots w/o soldered layer (cleaned with sodium hydroxide)
Washout Induced Weight Loss

Determined by weighing the pins before and after shots w/o soldered layer (cleaned with sodium hydroxide)

- H-H13: 50 Shots
- Mo-Base: 100 Shots
- Ti-Base: 100 Shots
- W-Base: 100 Shots
Determined by weighing the pins before and after shots w/o the soldered layer (cleaned with sodium hydroxide)
Soldering-Washout & Thermal Fatigue Resistance Ranking

10-Best    1-Worst

- Material 1
- Material 2
- Material 3
- Material 4
- Material 5
- Material 6
- Material 7
- Material 8
- Material 9
- Material 10

Soldering and Washout Resistance Level
Thermal Fatigue Resistance Level
Effect of Pin Material on Soldering

![Graph showing the effect of pin material on soldering percentage over shot numbers. The graph compares H13-Base, Anviloy 1150, Ti-6Al-4V, and Mo-785. Each material shows a different trend in the percentage of pin surface with soldering.]
Effects of Pin Material & Number of Shots on Percentage Area Covered with Soldering

![Graph showing the effects of pin material and number of shots on percentage area covered with soldering. The graph compares different materials such as Anviloy 1150, Ti-6Al-4V, Mo-785, Hard-H13, Soft H13, Ni-Base Alloy, and Cu-Base Alloy.](image-url)
CONCLUSIONS

• The Washout/Soldering set-up allows exposure of die materials to a jet of molten aluminum (ca. 300 in/sec). Each shot is about six pounds of molten metal.

• The two pins arrangement allows for comparing the evaluated material to a control H13.

• Evidence of severe washout has been observed in copper based alloys after three shots. Alloys with mutual solubility in aluminum are prone to dissolve when impinged by a hot jet of molten aluminum.

• Nickel base alloys form intermetallic compounds with aluminum. These compounds promote soldering, and bonding of the casting to the pins. As a result, the pins elongate during ejection.
CONCLUSIONS (cont.)

• Hard H13 has better soldering and washout resistance than soft H13.

• The best soldering and washout resistance in this study is exhibited by Anviloy, followed by Ti-6Al-4V and Mo alloys. This behavior is consistent with the low chemical affinity to Al and high melting temperature of these alloys.
Hardness Distribution in Nitro-Carburized Diffusion Layers
Hardness Distribution in Nitro-Carburized Diffusion Layers
Hardness Distribution in Nitro-Carburized Diffusion Layer

![D 5-A2](image1)

![D8-H2](image2)

Distance from Surface (um) vs. Hardness (HRC) for D 5-A2 and D8-H2.
Effect of the Nitro-Carburizing Treatment on Soldering

The diagram illustrates the percentage of pin surface with soldering (%) against shot number for different materials: H13-Base, B (thin), U (thick), D5A-1 (thin), and D8H-1 (thick). The graph shows how the percentage of soldering increases with the shot number for each material.
Effect of Nitro-carburizing on Soldering After 50 Shots

Nitro-carburizing improves soldering resistance of H13

The difference between coatings is small.
Effect of Nitro-Carburizing on Washout

Thick nitro-carburized layers are better for washout than thin layers.
Appearance of H13 and Nitro-Carburized Pins after 30 Shots

Hard-H13

D 5A-1  D 5A-2

B  D 8H-1

D 8H-2  U
Appearance of H13 and Nitro-Carburized Pins after the Test (Soldered Al Dissolved in NaOH)
Impingement Surface of Thin Nitro-Carburized layer in “B” Pin
(after 50 Shots - Soldered Al Dissolved in NaOH)
Impingement Surface of **Thin** Nitro-Carburized layer in “B” Pin (after 150 Shots-Soldered Al Dissolved in NaOH)
Impingement Surface of **Thick** Nitro-Carburized Layer in “U” Pin (after 150 Shots and Soldered Al Dissolved in NaOH)

![Cracked Coating](image.png)
Average Max Crack Length of Nitrocarburized H13

- **B - Thin**
- **D8H - Medium**
- **D5A - Thin**
- **H13 51 HRC**
- **H13 44-46 HRC**

2"X2"X7", WC7
Total Crack Area of Nitrocarburized H13

<table>
<thead>
<tr>
<th>Material</th>
<th>Total Crack Area ($\times 10^6 , \mu m^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B - Thin</td>
<td>475</td>
</tr>
<tr>
<td>D8H - Medium</td>
<td>200</td>
</tr>
<tr>
<td>D5A - Thin</td>
<td>150</td>
</tr>
<tr>
<td>H13 51 HRC</td>
<td>110</td>
</tr>
<tr>
<td>H13 44-46HRC</td>
<td>60</td>
</tr>
</tbody>
</table>

2" X 2" X 7", WC7
CONCLUSIONS

1. Nitro-carburizing improves somewhat the resistance of H13 pins to soldering (sticking of Al) but does not prevent it.

2. Nitro-carburizing improves significantly the resistance of H13 pins to washout (loss of base metal).

3. Thick nitro-carburized layers provide better resistance to washout. However, they tend to crack more readily than thin coatings.

4. Based on these observations, thick nitro-carburized coatings are desirable in “soldering intensive” applications; thin nitro-carburized coatings may be desirable in “thermal fatigue intensive” applications that can induce cracking.
## Characteristics of PVD Coatings

<table>
<thead>
<tr>
<th>Coating</th>
<th>Coating Process</th>
<th>Thickness* (µm)</th>
<th>Hardness** (Hv -kg/mm²)</th>
<th>Oxidation Temperature (F)**</th>
</tr>
</thead>
<tbody>
<tr>
<td>CrN+W (I)</td>
<td>PVD</td>
<td>3.5</td>
<td>2500±400</td>
<td>2,278</td>
</tr>
<tr>
<td>CrN (P)</td>
<td>PVD</td>
<td>5.0</td>
<td>2500±400</td>
<td>2,278</td>
</tr>
<tr>
<td>(TiAl)N (B)</td>
<td>PVD</td>
<td>2.0</td>
<td>2600±400</td>
<td>2,278-2,368</td>
</tr>
<tr>
<td>CrC (B)</td>
<td>PVD</td>
<td>6.25</td>
<td>1850±200</td>
<td>2,278</td>
</tr>
</tbody>
</table>

* Measured  
** Literature
Cross-Section of PVD Coatings and Pin Substrates

- CrN+W
- (TiAl)N
- CrN
- CrC
Appearance of PVD Coated Pins before Testing & after 30 Shots

CrN+W

Before

30 Shots

CrN

Before

30 Shots

(TiAl)N

Before

30 Shots

CrC

Before

30 Shots
Soldering Appearance of H13 Pin with CrN+W PVD Coating
Soldering Appearance of H13 Pin with CrN PVD Coating
Soldering Appearance of H13 Pin with (TiAl)N PVD Coating
Soldering Appearance of H13 Pin with CrC PVD Coating
Effect of PVD Coating on Soldering
Effect of PVD Coating Materials on Soldering (after 30 shots)

Weight (g)

1. Weight of Soldered Al on Pin Surface Is Determined by Weighting Pins after 30 Shots with and without the Soldered Aluminum

2. Soldered Aluminum Cleaned with Sodium Hydroxide
Effect of PVD Coatings on Washout Resistance

- H13-Base
- CrN+W PVD Coating
- CrN PVD Coating
- (TiAl)N PVD Coating
- CrC PVD Coating
Washout Comparison of PVD Coatings and Nitrocarburizing

- H13-Base
- (TiAl)N PVD Coating
- CrC PVD Coating
- Nitrocarburizing D5A-Thin
- Nitrocarburizing D8H-Thick
Degradation of H13 Pin with CrN+W PVD Coating
Degradation of H13 Pin with CrN+W PVD Coating (magnified)
Degradation of H13 Pin with CrN PVD Coating
Degradation of H13 Pin with CrN PVD Coating (magnified)
Degradation of H13 Pin with (TiAl)N PVD Coating
Degradation of H13 Pin with (TiAl)N PVD Coating (magnified)
Degradation of H13 Pin with CrC PVD Coating
Degradation of H13 Pin with CrC PVD Coating (magnified)
Degradation of H13 Pin with CrN+W PVD Coating (Pin Holes and Worn off)
Degradation of H13 Pin with CrN+W PVD Coating after 210 Shots (Pin Holes and Worn off)
Degradation of H13 Pin with (TiAl)N PVD Coating (Pin Holes and Worn off)
Degradation of H13 Pin with (TiAl)N PVD Coating after 210 Shots (Pin Holes and Worn off)
Degradation of H13 Pin with CrC PVD Coating (Only Pin Holes)
Degradation of H13 Pin with CrC PVD Coating after 240 Shots
(Only Pin Holes)
Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots

- H13 Base
- Soldered Aluminum Alloy
- CrN+W PVD Coating
- Molten Aluminum Jet Impinging Surface
- Mounting Material

PECVD 15.0 kV x100 300 \mu m
Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots
Cross Section View & Failure Mode of CrN+W PVD Coated H13 Pin after 120 Shots
CONCLUSIONS

• The “thick” CrC PVD coating was the best performer among the PVD coatings evaluated so far.

• However, this coating is primarily recommended for small cores; It is more susceptible to thermal fatigue cracking than the thin coatings.

• The thin PVD coatings fail at surface imperfections in the substrate and at defect sites in the coating (pin-holes). Good coatings practices are essential in ensuring a high performance coating.