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PT401 Paint Process Development and Infrared Reflectance Measurements

Sariah Cassidy
Utah State University

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PT401 Paint Process Development and Infrared Reflectance Measurements

By: Sariah Cassidy

Mentored by: Dr. Jim Dyer, SDL scientist and adjunct professor of Physics

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Abstract

Z302 became less obtainable when the manufacturer required expensive massive drums of the black specular paint as the minimum order. As they no longer accommodate customers with pint-sized needs, companies, such as the National Institute of Science and Technology (NIST), are searching for a replacement. PT401 has been used on previous projects with satisfactory results. In accordance to requirements from NIST, a process for painting with PT401 8-10mils thick will be developed, and adherence, durability and specular reflectance and diffuse properties in the infrared will be documented.

Background

The process of painting was to be documented at AMFI with Dennis Morely. However, the warehouse environment in which the painting was done introduced many contaminating variables and a reproducible process could not be documented. Painting at SDL commenced in a clean lab with a hood and a highly controlled environment. Painting supplies were purchased for this purpose.

Two important characteristics are required with specular black coating for spacecraft and other equipment sent into space: durability and predictable specular reflectance.

The coating must be able to maintain color, adhesion, and specular properties in a vacuum heated environment. The NAPCO oven will be used to bring coated parts up to 200 C in vacuum. Also, 8-10 mils paint thickness is required to eliminate possible thin film interference in the infrared spectrum.

Reflectance will be measured with a Varian 680 FT-IR spectrometer, and a Seagull angular reflectance accessory. Infrared data collection in the mid IR (2-20 µm) is of particular interest to NIST for specular behavior.

Paint Procedure

This procedure was developed through trial and error. Part of the process required by NIST was that the paint be able to endure vacuum bake-out at 200 C. Often, the paint would peel up off the substrates during bake-out. A full work instruction is documented at Space Dynamics Lab.

Substrates

NIST substrates are copper are as follows:
• 1” circles
• 3” donut with matching 1” center

Figure 1

NIST sample parts
The 1” circles were machined to fit tightly into the 1” center of the donut.

Cleaning
PT401 paint was designed for up to 5 mils thickness coating. In order to eliminate the possibility of thin film interference, it was required that a process be developed to be able to build up the thickness to 8-10 mils. Adhesion was a major problem in building up thicknesses greater than 5 mils. Substrates with smooth and scotch brighted surfaces failed to maintain bonding with the paint. It was found that parts must be bead blasted for paint to have grip on the copper. After bead blasting, the substrates were precision cleaned. The steps for cleaning are as follows:

- Acetone rinse
- Sonicated in soapy water 5 min
- Sonicated in DI water 3 min
- Sonicated in acetone 2 min
- DI rinse
- Copper bright dip 9:1 diluted sonicated 3 min
- DI rinse
- Sonicate in acetone 2 min
- Sonicate in IPA 2 min
- IPA rinse
- GN2 blow dry
- Masked around their edges using blue masking tape.

Mixing
PT402 is prepared according to the instructions from the manufacturer. It may change as catalyst concentrations may vary between volumes of paint ordered. For the pint sized order mix by volume:

<table>
<thead>
<tr>
<th>PT402 component A</th>
<th>4 parts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Component B</td>
<td>1 part</td>
</tr>
<tr>
<td>PT1045 Thinner</td>
<td>10 parts</td>
</tr>
</tbody>
</table>

This makes a very thinned solution, which allows a thicker spray on the substrate without adding too much PT402. Only a very thin layer of PT402 is needed (~0.2 mils).

The epoxy primer, PT500, is similarly mixed by volume:

<table>
<thead>
<tr>
<th>PT500 Component A</th>
<th>3 parts</th>
</tr>
</thead>
</table>
Component B | 1 part
------------|------
PT1003 Thinner | 8% or less

One hour of induction time is required for best results.

Lastly, PT401 was mixed. Issues arose while mixing small portions of paint in contrast with its catalyst. The small amount of paint mixed induced a larger relative error. Therefore, it was decided to mix by weight to eliminate much of this error. The density was determined by weighing 25 ml of each component. With that information the volumes were converted to weight for mixing:

<table>
<thead>
<tr>
<th>Component</th>
<th>Weighed first</th>
</tr>
</thead>
<tbody>
<tr>
<td>PT401 Component A</td>
<td>Weighed first</td>
</tr>
<tr>
<td>No Peel</td>
<td>Comp. A weight x 0.0435</td>
</tr>
<tr>
<td>No Pop</td>
<td>Comp. A weight x 0.0882</td>
</tr>
<tr>
<td>Component B catalyst</td>
<td>Comp. A weight x 0.135</td>
</tr>
<tr>
<td>PT1002 Thinner</td>
<td></td>
</tr>
</tbody>
</table>

The No Peel and No Pop additives help the final texture to be smooth. Also, if the ratio of Comp A/B was too low the paint would discolor and sweat. Too little catalyst caused the paint curing time to increase. Adding 4:1 thinner to admix solution by volume allows for the final product to produce the desired finish.

**Application**

Application of primer and paints were by airbrush. A cylinder of GN₂ was used as the air pressure. A gauge was installed on the cylinder to maintain desired gas pressures of 18-20 psi and 8-10 psi.

PT402 acid wash was applied to the copper substrates first as it prepares the copper for adhesion. The acid wash is applied in a very thin layer. Using the airbrush with pressure set to 18-20 psi, the acid wash is best sprayed just enough to get the substrate wet as shown in figure 2. Allow 15 minutes for the PT402 layer to dry before further applications.

PT500 acid primer was applied over the dry PT402 coat. Two coats of PT500 are applied by passing the airbrush over the substrate either horizontally or vertically. Desired thickness is 6-9 mils. These coats should be identical in application and thickness.
The yellow or green primer color should be easily seen but the copper color on the substrate still visible beneath the coat. After the second coat the copper should still be visible but the color of the primer bright as shown in figure 3. Too much primer will obstruct good adhesion. Too little will not be enough to promote adhesion. At least an hour and no more than 24 hours should be allowed for the PT500 coats to dry before applying PT401.
PT401 application was more complicated. The first several layers of paint were laid thin. No more than three passes were made with the airbrush per layer of PT401. After each layer time was allowed for the paint to become tack-free. This means that when a handless glove finger is tapped lightly on the paint, no paint comes off on the glove. When the paint is tack-free and new layer was laid. After 5-8 layers the paint was left for a 2-3 days to cure. It can take up to 7 days to fully cure at ambient temperature. After the thin layers had a few days to cure, the last two steps of a fast cure method was implemented. When paint was not allowed a few days to set and the bake out it would peel up off the substrate during the final painting stage.

<table>
<thead>
<tr>
<th>Fast Cure Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient Temperature</td>
</tr>
<tr>
<td>Oven bake at 65 C</td>
</tr>
<tr>
<td>Oven bake at 121 C</td>
</tr>
</tbody>
</table>

After paint had been cured, the thickness was measured to be 5-8 mils thick using an Elcometer to determine the thickness. 320-grit sandpaper is then used to sand down the paint thickness to 3-5 mils. The larger the substrate the smaller the final sanded thickness should be.

The sanded PT401 paint was then prepared for the final coat. The total amount of PT1002 thinner to admixed PT301 should be 4:1. The super thinned paint allowed for a thick amount of paint to be laid down with a smooth finish. A puddle method was used to put down the thick layer. This helped eliminates the problem of edge build up. The substrates need to be leveled for this method. After laying the substrates flat and level, a puddle of paint is airbrushed or poured onto the substrate until the entire surface is covered in a puddle. If the surface tension is too low, as the paint dries it will form a dip in the middle of the paint and a rise on the edges. Maximizing the surface tension forms the best uniform coat. Paint was allowed to dry 1-7 days before final curing in an oven following the fast cure procedure.

**Vacuum Bake-out**

Painted and cured parts were put in the vacuum oven and brought up to 100 C with the vacuum on. After parts reached the targeted temperature, a visual inspection through the oven window was performed to check that the paint had not peeled off the substrates. If paint adherence was unhindered the
temperature was brought up 50 C. Inspections ensued until the final temperature of 200-210 C. Once the parts reached this final target the oven was turned off and the cycle was complete. At this point the substrates were conditioned according to the customer’s requirements and ready for specular analysis.

**Spectrometer and Accessories**

A Varian 680 FT IR spectrometer was used to take spectra of painted parts. It is a Michelson interferometer, which means there is a beam splitter in the light path. As can be seen in figure 5, half the beam is sent to a stationary mirror and the other half to a moving mirror. The movement of the mirror creates a set of constructive and destructive interference waves. These waves are sent on to bounce off the sample where absorption bands form in the signal. Then the beam is directed to the detector where it is translated into a readable graph by Resolution Pro software.

The Seagull variable angle accessory was used in the sample compartment of the spectrometer. It allowed for individual control over the angles of incidence and reflection. The beam traveled in to the accessory and hit a series of mirrors until it was positioned at the desired angles of incidence and reflection. The mirrors seen at the top of the accessory in figure 6 were adjusted for angles 0-90 degrees from the normal of the sample.

The Pike integrating sphere, in figure 7, was used to gather total reflectance from the sample. The integrating sphere is a small 2” hollow gold sphere into which a beam from the spectrometer is directed. The beam then hits a mirror angled to bounce the beam off the sample laid on the sample port. All the light reflection is caught by the sphere and eventually is sent to the detector portal. The integrating sphere has its own high sensitive cooled Mercury cadmium telluride (MCT) detector.

**Infrared Spectral Reflectance**

Samples were characterized in the infrared
wavelengths of 2-20 μm for both reflectance and diffuse reflectance. The reflectance measurements were made with the Varian FT-IR and diffuse measurements were made with the integrating sphere.

The following are samples of spectra gathered from this experiment. The first graph in figure 7 is a background collected using a certified diffuse gold sample. Diffuse gold was a good comparison PT401 because the paint is shiny and reflective but is very black.

Sample Analysis

![Graph](image)

**Figure 8**

In figure 9, a sample, 26D, of MWCNT (Multi-Walled Carbon Nanotube) coating was taken against the background in figure 8. MWCNT paint is diffuse, absorptive and very black. It is a good comparison for PT401 on the other end of the reflective spectrum.
Figure 9

Figure 10 is spectra gathered from sample 30D with PT401 10 mils thick. Paint was laid with thin layers and final finish was not smooth.
On the Pike integrating sphere, the background was scanned with diffuse gold as can be seen in figure 11.

![Figure 11](image)

In figure 12, the total reflectance spectrum was taken with the integrating sphere for the MWCNT sample 26D. Reflectance is not very high for MWCNT coating.

![Figure 12](image)

The total reflectance spectra of the PT401 at 10 mils are about 0.01 point higher in the IR wavelengths than the MWCNT coating. This can be seen by comparing figure 12 and 13.
Conclusion and Future Work

The most challenging development was the process for paint. The finish on the final product had to be glossy without polishing and adhere to copper. Copper is notorious for non-adherence especially with paint. Any step omitted in the instructions above resulted in the paint not adhering to the substrates. The process is reproducible and viable samples were successfully painted with the thickness NIST requested.

Methods were developed for diffuse and reflectance measurements using the instruments at Space Dynamics Lab.

The database of the characteristics of diffuse and specular reflectance is incomplete. Further testing will continue on PT401 paint. Samples have been prepared using the puddle method with smooth surfaces and thicknesses of 8-10 mils. Space Dynamics Lab has the capability to scan painted parts up to 100 µm for analysis and documentation.
Works Cited