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**CRYOGENIC TESTING OF FLUOROPOLYMER-COATED
STAINLESS STEEL TUBING**

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SUMMARY

Stainless steel tubing coated internally with two different types of fluorinated polymers were subjected to microscopic examination after a welding operation had been performed on the tubing. The welded assemblies were photographed and subjected to repeated cycles between liquid helium and room temperature. The green tetrafluoroethylene (TFE) coating peeled back in the area subjected to welding heat and displayed cracking all over its surface without regard to proximity to the weld area. The dark fluorinated ethylene propylene (FEP) coating showed a tendency to char or burn away progressively in the weld area. The dark (FEP) coating did not crack as extensively as the green TFE coating, but did show a few areas of "crazing" or cracking of the topmost surface after cryogenic exposure.

INTRODUCTION

To reduce the heat flow through fuel line walls carrying liquid hydrogen, the thermal resistance of the wall must be raised. One way to do this is to add a thin layer of plastic inside a metallic line. To evaluate the potential of such coatings to remain attached to the metal wall of the tubing, the Space Propulsion Branch of the Propulsion Laboratory at Marshall Space Flight Center (NASA-George C. Marshall Space Flight Center) procured the services of a commercial coatings supplier. Two stainless steel tubes (2-in.-nominal diam, 0.020-in.-thick wall) were each coated with a different fluorinated organic polymer coating. These were provided to Oak Ridge National Laboratory (ORNL) for testing and examination in accordance with a test plan provided by Space Propulsion Branch.

TEST SEQUENCE DESCRIPTION

SPECIMEN PREPARATION

The sample tubes were taken to the Metallurgy Department at the Oak Ridge Gaseous Diffusion Plant. There, 2-in. lengths were cut from the end of each tube. These lengths were sectioned and examined in detail.

The tubes were then sent to the Development Maintenance Department with instructions to weld a flange onto the dark FEP-coated tube and to execute a tube-to-tube weld on the green TFE-coated tube. (This represented a change to the original test plan, which was coordinated with the Space Propulsion Branch representative. The dark FEP coating would be a more difficult target for photographs and inspection in the tube-to-tube weld.) The welds were to be made with minimum heating consistent with mechanical integrity. Practice welds showed that a 1/16-in. tungsten wire electrode with argon gas blanket and argon gas purge through tube interior would produce a satisfactory weld.

Following the welding, the tubes were photographed to show coating conditions. The tubes were then forwarded to the ORNL Fusion Energy Division for liquid helium exposure.

CRYOGENIC EXPOSURE

The tubes were cycled between ambient temperature and 1 atm of liquid helium by dipping into a Dewar containing the liquid helium. The tubes were allowed to reach equilibrium, as indicated by a cessation of excessive boil-off. The tubes were then warmed back to ambient temperature using laboratory heat guns set to a low temperature. (This change in procedure was coordinated with the Space Propulsion Branch representative who was on-site for the testing. Extra care not to overheat the tubing was exercised.) This cycle was repeated five times for each tube.

When no gross damage was evident after the cyclic exposure, the tubes were placed in the liquid helium and left for a long-term soak. This soak lasted until the helium supply in the Dewar had boiled away, ~ 12 to 16 h, and there was a gradual rise back to ambient temperature.

EXAMINATION AFTER EXPOSURE

The tubes were cut into sections to permit examination of the condition of the interior coating. The welded areas were sectioned to permit examination of the coating in the region of the weld. The areas near the weld, along with representative areas of the tubing away from these areas, were photographed.

RESULTS

EXAMINATION BEFORE EXPOSURE

The two coatings displayed a marked difference in appearance, both to the unaided eye and under low-magnification examination. The green TFE coating was quite rough and had dropletlike protrusions all over the surface (see Fig. 1). The coating was variable as to the size and density of these bumps, as may be seen in Figs. 2 and 3, which show different areas of the tube.

The green coating was, because of these bumps, quite variable in thickness. Figure 4 shows an 800X magnification. This variation extended from almost uncoated areas to areas nearly 0.030 in. thick. The extreme etching of the stainless steel is seen at the bottom, with the coating varying in thickness. Between the coating and the metal, a thin primer layer can be seen.

The tube-to-tube weld was made on the tubing containing the green coating. The area around the weld is shown in Fig. 5. The coating completely decomposed in the area subjected to the heat of welding. The coating peeled back, forming a flap along the edge of the area discolored by the heat on the tube exterior.

Fig. 1. Green coating at low magnification (20X).

Fig. 2. Green coating in different area (40X).

Fig. 3. Green coating showing variability (40X).

Fig. 4. Magnified cross section of tubing with green coating (800X). Etched metal surface is at bottom.

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Fig. 5. Green coating in weld area before exposure.

The dark FEP coating was much smoother than the green coating (see Fig. 6). The reason for this was evident when the cross section (see Fig. 7) is examined. The dark coating is attached to the etched stainless steel and then is covered with a transparent overcoating. This overcoating seals the darker plastic. There is, however, considerable variability in the dark coating. Figure 8 shows an area where the clear overcoating has run under the dark coating. It is not clear from this examination whether the dark plastic was not fully bonded to the metal or whether the clear overcoating ran under the dark coating and lifted it. The clear overcoating has evidently bonded the loosened dark material and does not show a break on the surface.

The tube containing the dark coating was welded to a flange. The extra heat required to weld to the more massive flange created a larger zone affected by the heating. The response of the dark coating was markedly different from the green coating. Figure 9 shows that the coating is completely missing in the weld area. A short distance away, a region of char gradually blends into undisturbed dark material. This region evidently has lost the clear overcoating which has pyrolyzed or evaporated. This area blends into an area where the clear overcoating is apparently present but discontinuous. The clear overcoating gets denser until it appears to be undisturbed. The boundary of the undisturbed area again corresponds roughly to the edge of the area on the exterior tube surface that is discolored by the heat.

EXAMINATION AFTER EXPOSURE

After thermal exposure, the welded areas were cut from the tubing and examined. The balance of the tubing was cut open and surveyed. Representative areas are shown in Figs. 10, 11, and 12.

The green TFE coating did not seem to detach in bulk beyond the initial area affected by the welding (see Fig. 10). The extensive cracking of the coating can be seen. Similar cracking was induced on another sample by a single immersion in liquid nitrogen. Cracking of this degree is disturbing since it will tend to entrap liquid hydrogen which will, upon depressurization, expand and attempt to pry the coating from the substrate. This cracking was present all over the exposed material and was not restricted to the heated zone.

The dark FEP coating responded much better to the thermal cycling. It did not debond, and no damage was evident to the unaided eye. Microscopic examination of the surface (see Fig. 11) showed the coating to be largely intact.

A few small, randomly located areas of local "crazing" (cracking) of the clear coating were found on the dark coating. Figure 12 shows that these were not as extensive as the cracking of the green coating. It cannot be stated conclusively that these areas were not present before cryogenic exposure because the examination is, of necessity, destructive. They were not present in the 2-in. section removed before testing, but that short section represents only a small fraction of the tubing.

Fig. 6. Dark coating at low magnification (20X).

Fig. 7. Dark coating showing layers composing coating (800X).

Fig. 8. Dark coating showing clear layer running under dark layer (800X).

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Fig. 9. Dark coating in weld area before exposure (coated area below weld).

Fig. 10. Green coating after exposure (20X). Weld area is at top.

Fig. 11. Dark coating after exposure (20X). Weld area is at top.

Fig. 12. "Crazed" area of dark coating (40X).

CONCLUSIONS

Based on this testing, the following conclusions emerge.

1. The green TFE coating is very irregular and has major variations in thickness and coverage.
2. The green TFE coating cracks readily when exposed to liquid helium. Laboratory tests show cracking develops on the first exposure and is serious even at liquid nitrogen temperatures.
3. Welding causes the green TFE coating to peel away from the heated area. The peeled areas remain attached and do not seem to be sites for initiation of gross separation of the coating.
4. The dark FEP coating is quite smooth and fairly regular in thickness. There is variability due to the two component coatings.
5. The dark FEP coating did not crack extensively or separate from the tubing. "Crazing" of the coating was noted in a few small random areas, but this could not be conclusively related to liquid helium exposure.
6. The dark FEP coating responded to welding with a progression from decomposition to melting to undisturbed coating. All of these components were still adhering to the steel tubing. The heated area did not seem to serve as an initiation point for further debonding.

ASSESSMENT

The green TFE coating is probably not desirable for use as a fuel line coating. Flowing fluid and pressure variations probably will cause the green TFE coating to detach from the wall.

The dark FEP coating survived exposure to both welding and thermal cycling in much better condition. It presents a better surface to flowing fluid. The "crazing" should be investigated to determine if it is thermally induced or if it is a manufacturing defect.

Note that all of these conclusions are based on static exposure (no flow) to liquid helium. No external loads or vibration were applied to the coatings while they were chilled. Since organic materials are known to become brittle when exposed to liquid helium or other cryogens, these conditions should be examined before committing to the use of these materials.