FY17 IRTD Executive Summary

One Step Plasma Passivation & Precision Cleaning

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Over the past several years, Kennedy Space Center (KSC) researchers developed two solvent-free techniques for precision cleaning of ground support equipment (GSE) that possess low commodity requirements and produce small or no waste streams. During testing of low-pressure air plasma as one of these cleaning techniques, physical changes in the surface of stainless steel test pieces were observed; cursory testing revealed that the test part had developed a thick oxide layer on the surface compared to untreated parts. This result indicated that the plasma process might not only serve as a green technology for precision cleaning of parts, but it may *also* passivate the surface at the same time. Current methodology in use at KSC's Components Refurbishment & Chemical Analysis (CRCA) Facility for passivation of corrosion-resistant steels for aerospace applications follows an SAE International Standard, AMS2700, where parts are submerged in either a nitric acid or nitric acid/sodium dichromate bath at predetermined temperatures and contact times. These baths require constant testing to ensure effective treatment, use corrosive and carcinogenic chemicals, and produce hazardous waste. The passivation bath is one of the multiple steps employed at the facility, while cleaning with multiple environmentally-unfriendly solvents comprise several more steps before parts are recertified to a specific cleanliness level in their clean room. Based on the behavior of the stainless steel surface during cleaning optimization studies, it was believed that the plasma technology could be extended to passivation, combining what is currently many steps into a single, solvent- and waste-free system. The three objectives of this project were:

- Determine if low pressure air plasma is capable of passivating various alloys
- Optimize the plasma system's operating parameters to achieve sufficient levels of passivation
- Combine this process with previously developed plasma-based precision cleaning processes into a one-stop, solvent-free preparation technique

Materials Engineers at KSC and the Refurbishment Supervisor at CRCA were consulted for advice on selecting specific test alloys that represented a range of chemically diverse materials in addition to exemplifying a large volume of the material types passivated at CRCA. Based these discussions, the seven alloys chosen for investigation were as follows: 303 SS, 304 SS, 316 SS, 410 SS, Inconel 718, A286, and Grade 9 (Titanium 3Al-2.5V). All seven alloys were characterized in as received form and after passivation at CRCA using x-ray photoelectron spectroscopy (XPS). This data can be used for comparison with alternative passivation methods carried out in this project or in future projects. 304SS, 316SS, and A286 steels were subjected to plasma treatment in this project.

Initially, ASTM B-117 salt fog chamber exposure was chosen as the method to evaluate the effectiveness of passivation. However, initial tests showed that samples in their as received, CRCA passivated, or plasma passivated states all performed similarly in this test, so salt fog exposure was not used in future testing. Passivation of these alloys is done to mitigate the effects of defects in these materials that occur during manufacture or machining. The defects occur infrequently, so it would take a large sample size

(>100) to get statistically significant results from salt fog testing. Attempts were made to create defects in the samples, but this did not work.

XPS was chosen as the method to determine if plasma passivation was a suitable replacement for traditional passivation methods. XPS is a powerful technique that measures the chemistry of a surface. Depth profiles of as received, CRCA passivated, and plasma passivated were compared to determine if the plasma passivation could produce the same chemistry as passivation by CRCA. The assumption was that if the chemistry was the same, the performance would also be the same. Surface roughness was measured and compared between the different treatments to see if the passivation method imparted any physical changes.

Finally, samples were contaminated with common aerospace greases and plasma cleaned and passivated in the same process.

The key observations in this study were:

- Solvent-based passivation performed at CRCA decreased surface carbon contamination and increased the relative amounts of oxygen, iron, and chromium in the first several layers of the material as compared to as received material.
- Solvent-based passivation had little effect on the chemistry of the Inconel 718 and Grade 9 samples.
- Plasma passivated samples show a significant increase in oxygen over several layers of etching which is mostly due to the formation of a silicon oxide layer. The silicon oxide is likely coming from the walls of the plasma chamber, which are glass.
- Plasma passivation does not selectively enrich chromium near the surface of a treated part as was seen in solvent based passivation.
- Plasma treatment is capable of removing carbon based contamination common to aerospace components and creating an oxide layer in a single treatment.
- Although plasma passivation affected the surface roughness, it is likely too small an effect to make a difference.
- Samples that were contaminated could be plasma cleaned and passivated at the same time, yielding the same results as samples that were clean prior to plasma passivation.

Plasma passivation increased oxygen content on the surface, as did solvent passivation, but the surface chemistry is still different. Higher chromium or nickel contents in the surface of stainless steels are known to improve corrosion resistance, and it is unclear if the changes in surface chemistry caused by plasma passivation would impart the same effect on corrosion resistance. Based solely on surface chemistry, plasma passivation is not equivalent to the traditional passivation methods done at CRCA. Since salt fog testing of these alloys was inconclusive, it was impossible to differentiate between the corrosion resistance of as received parts and passivated parts. If future testing is to be performed, alloys more susceptible to corrosion should be chosen so the changes in corrosion resistance can be evaluated in addition to the surface chemistry.