Plasma Spray Coatings as Treatments for Aluminum, Titanium and Steel Adherends

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Plasma spray coatings have been evaluated as surface treatments for aluminum, titanium and steel substrates prior to adhesive bonding. These treatments are environmentally benign in that they involve no chromates and emit no liquid or gaseous wastes. The coatings can be engineered for specific applications and are better suited for localized repair than chemical processes. For aluminum adherends, a 60Al-Si/40polyester coating gives a performance equivalent to that of the best chemical treatment (phosphoric acid anodization) for some epoxy adhesives. With stronger, tougher adhesives, a Ti-6Al-4V coating provides improved performance to match that of phosphoric acid anodization. A Ti-6Al-4V coating on titanium substrates exhibits identical initial strength and durability to the best chemical controls under moderate temperature conditions. At high temperatures, the plasma spray coating continues to exhibit excellent durability while oxide-based treatments readily fail due to oxygen dissolution into the metal. For steel adherends, an Ni-Cr-Zn coating provides enhanced corrosion resistance and bondability even after exposure to aggressive environments or ambient conditions over long periods of time. Additionally, rubber bonds with the plasma spray coating are more tolerant to surface contamination than those with grit-blasted surfaces. These investigations indicate that the plasma spray process is more robust than conventional processes and can give equivalent or (in some cases) superior performance. © 1997 by John Wiley & Sons, Ltd.

INTRODUCTION

Joining of metals is a key process in the fabrication of components and structures in the transportation and building/construction industries. Adhesive bonding has several potential advantages over other joining processes, such as welding or mechanical fastening,[1-3] especially in conjunction with lightweight structures such as aircraft, as shown in Table 1. For some structures, such as metal–polymer composite laminates for superior fatigue resistance, adhesive bonding is essential in manufacture.

Modification of the metal surface prior to adhesive bonding is critical in order to achieve high performance, especially durability, under humid conditions—the limiting factor governing state-of-the-art joint performance.[4-6] These treatments are designed to:
(1) remove organic contamination;
(2) remove mill scale and other oxides formed during fabrication and storage;
(3) modify the surface to provide a suitable microrough morphology that is wetted by the primer/adhesive and is stable under expected service and test conditions.

A convoluted microrough morphology is necessary to mechanically interlock or physically bond with the adhesive. This interlocking allows stresses to be transferred from adherend to adhesive without relying on interfacial chemical bonds, which are generally disrupted in the presence of moisture.

Surface treatments for metallic adherends prior to adhesive bonding commonly involve chromates: e.g. Forest Products Laboratory (FPL) or chromic acid anodization (CAA) and/or strong acids or bases such as CAA and phosphoric acid anodization (PAA). These processes generate wastes that are harmful to the

Table 1. Advantages of adhesive bonding over other joining processes

- Ability to join dissimilar materials
- Minimization of stress concentrations
- Minimization of adherend damage during preparation, e.g. heating or mechanical damage
- Superior fatigue and damage tolerance
- Superior corrosion resistance
- Increased design flexibility
- Minimization of weight
- Superior damping/noise reduction
- Superior appearance
- More cost effective

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environment and require expensive handling and disposal. Plasma spray treatments exhibit several advantages over conventional chemical treatments:

(1) Environmentally benign (no liquid or vapor wastes).
(2) Flexibility to design coatings/treatments for specific applications.
(3) Insensitive to surface contamination or ambient degradation.
(4) Indefinite shelf-life prior to bonding.
(5) Independent of substrate chemistry/material.
(6) Suitable for repair and localized re-work.
(7) Suitable for remote (field) use.
(8) Established thermal spray infrastructure.
(9) Low total processing costs.

The plasma spray process involves rapidly heating the powder material to the molten or semi-molten state and then propelling it against the substrate at high velocities. The high impact velocity and molten state provide good bond strength to the substrate. Very little heat is transferred to the substrate, with typical substrate temperatures ranging from 100° to 300 °C. Thus, metallurgical change, distortion and oxidation are avoided.

Plasma spraying has the important advantage of versatility. A wide range of coatings (metals, ceramics and polymers) can be deposited onto an equally wide range of substrates, and the coating properties can be optimized for a given application, independent of the substrate. Metals, ceramics and polymers can all be deposited onto a similar range of substrates. Because of this versatility, in addition to adhesion the plasma spray coatings have been used for thermal barriers, wear resistance, EMI/RF shielding, corrosion resistance, slip/slide resistance and biocompatibility. If desired, the composition and, hence, the properties of a coating can be graded from the substrate to the surface.

Plasma spraying has been shown to provide excellent high-temperature bond performance with titanium (unlike conventional oxidation treatments)[7] and a durability approaching that of PAA for aluminum for some adhesives.[8,9] Success has also been reported using alumina and other coatings on aluminum,[8-12] titanium and other coatings on titanium,[8,10-12] and passive metal coatings on steel.[13-15]

EXPERIMENTAL

Substrates were 2024-T3 or 7075-T6 aluminum, Ti-6Al-4V, D6AC or 4130 steel. The panels were degreased and grit-blasted within 4 h of plasma spraying either with a mixture of alumina, fused silica, silicon carbide and crystalline silica, or with pure alumina (80 grit size). Grit blasting was performed at near-normal incidence at a pressure of 1.5 kPa. Both metallic and metallic/polymer composite coatings were evaluated. No primer was used prior to bonding in order to fully test the inherent stability of the coating and the interface and to evaluate the feasibility of eliminating primers using bonding.

Plasma spraying was achieved using a Metco plasma gun mounted on a robot articulated-arm for controlled, reproducible coatings. To remove any moisture, the substrate was heated to ~100 °C by the plasma torch by rastering the gun over the specimen prior to injecting powder into the gun. During plasma spraying, the speci-

mens were air-cooled from the back and sides. The coatings were formed with several passes of the spray to build up the desired thickness. Unless indicated otherwise, the nominal coating thickness was 50 ± 5 μm (2 mils) as measured by a micrometer over several spots on the panels.

Both chemical and mechanical control treatments were performed for comparison. The chemical treatments included optimized FPL and PAA as conventional aerospace processes for aluminum. Titanium chemical controls included CAA, Turco 5578 and Pasa-Jell 107. Grit blasting was the only control for steel substrates.

The most discriminating test for the different surface treatments was the double-cantilever-beam wedge test (ASTM D-3762). It provides a rapid screening of surface treatments and has been correlated with in-service bond performance.[16,17] The five 25×150 mm specimens were exposed to >95% relative humidity at 60 °C for ~8 days. Crack length was measured as a function of exposure time by periodically removing the specimens from the humidity chamber and inspecting both sides under a microscope. The initial crack length indicates the initial strength, while the final crack length reflects the bond durability. In both measurements, improved performance (greater withstood stress) is reflected by smaller crack lengths. Other tests included the pneumatic adhesion tensile testing instrument (PATTI) for expoxy tensile measurements, and peel and tensile measurements for rubber.

Three epoxy adhesives were used in this evaluation for wedge tests: Cytec FM-123 and FM-73 (121 °C cure) and Cytec FM-300 (177 °C cure). To test the high temperature performance of the titanium treatment, one wedge test was performed using LaRC TPI polyimide adhesive cured at 400 °C. Tensile button (PATTI) tests were performed using either 3M 1838 or 3M 2216 two-part room-temperature-curing epoxy to bond aluminum stubs. Acrylonitrile butadiene rubber (NBR) and ethylene propylene diene monomer (EPDM) rubber were bonded to steel using Lord Chemlok 205/233 and Chemlok 205/(236 or 252) primer/adhesive systems, respectively.

In some of the steel experiments, the steel surface was coated with Conoco HD-2 grease prior to bonding in order to determine the sensitivity of the bondline to contamination. A uniform grease layer was applied by spraying a solution of grease using a raster pattern. Grease levels were determined by witness specimens of aluminum foil.[18-20]

Selected specimens, both as-sprayed and matching failed surfaces, were characterized with x-ray photoelectron spectroscopy (XPS). The XPS measurements were obtained using a Surface Science Instruments Model SSX 100-03 spectrometer with a monochromatized Al Kα x-ray source and a hemispherical electron energy analyzer with multichannel detection.

RESULTS AND DISCUSSION

Aluminum

Wedge test results for a 60%Al-Si/40%polyester plasma spray coating using Cytec FM-300M epoxy aerospace

adhesive are given in Fig. 1. For this adhesive, the plasma spray coating gives a performance very similar to that of PAA—the current state-of-the-art in aluminum adherend surface treatments—as demonstrated by both the initial crack length (strength) and final crack length (durability). The locus of crack propagation under both dry and wet conditions was predominately within the coating for the plasma spray treatment. For PAA, the locus of failure was within the adhesive when dry and interfacial when wet. The similarity in performance suggests that the strength/toughness of the coating is very similar to that of this adhesive.

Also shown in Fig. 1 is the performance of grit-blasted adherends—a sometimes used non-chemical treatment for bonding. The durability of grit-blasted joints is highly dependent on blasting procedures. The poor performance shown is representative of typical grit blasting operations. Under carefully controlled conditions, performance can be improved to rival FPL performance, but further improvements are not feasible. The grit blasting treatment is suitable for applications requiring only moderate bondline strength or minimal exposure to moist conditions. Wedge tests results using all three Cytec adhesives investigated are given in Fig. 2 for the 60%Al-Si/40%polyester plasma spray coating. As before, for FM-300, the plasma spray coating gives performance very similar to that of PAA, as demonstrated by both the initial and final crack lengths. For

FM-123 and FM-73, the tougher adhesives exhibit a smaller initial crack length for the chemical controls for which propagation was cohesive within the adhesive. Although crack growth for the plasma spray adherends is relatively small, similar to that for PAA, the initial crack length is greater than that of the controls so that the final crack length and hence the stress that the joint can withstand is intermediate between those of FPL and PAA.

Surface analysis of the surfaces formed during crack growth under dry conditions revealed that the crack propagated predominantly through the polyester phase of the composite coating. As demonstrated in the surface behavior diagram (SBD)[21,22] of Fig. 3, the crack propagated through a mixed interphase of the adhesive and the coating, but the failure surface was polyester rich—that is, the composition falls to the left (polyester side) of the dashed line of stoichiometric composition. The extent of the polyester phase suggested that the coating had larger regions of the two phases than expected, i.e. the coating was heterogeneous on a larger scale than predicted from random mixing of the two components.

Three parameters were evaluated to improve performance: Al/polyester ratio, thickness and powder mixing. Tests using a range of coating composition, from 100% Al to 100% polyester, showed that while initial crack length was optimized by a 80Al-Si/
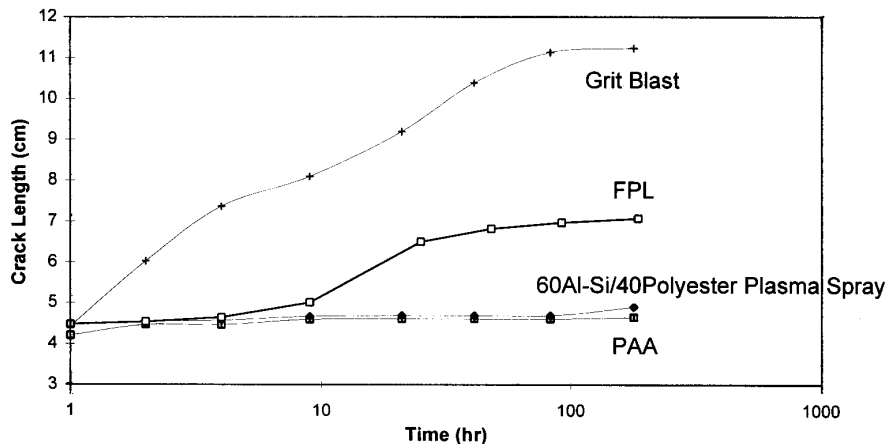

Figure 1. Wedge test results for FPL, PAA, grit blast and plasma spray 60Al-Si/40polyester treatments for Cytec FM-300M epoxy adhesive.
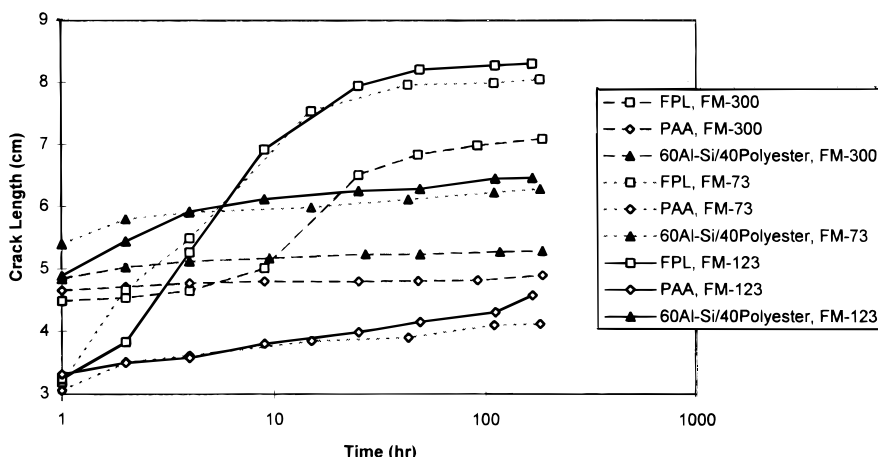

Figure 2. Wedge test results for FPL, PAA and plasma spray 60Al-Si/40polyester treatments using three different adhesives.
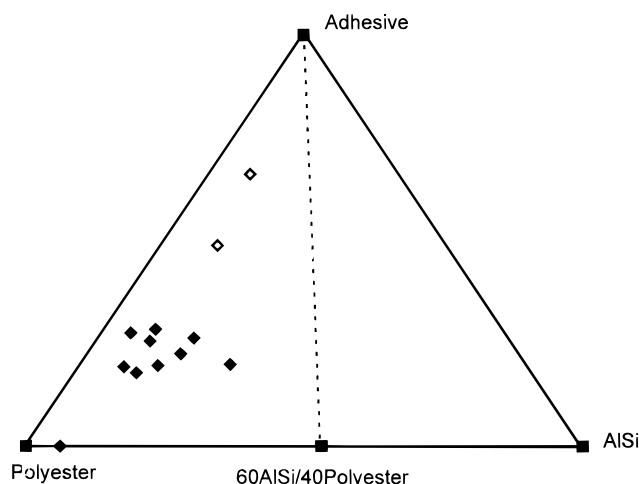
Figure 3. Surface behavior diagram showing the locus of dry crack propagation of 60Al-Si/40polyester wedge test specimens. The solid diamonds are the standard powder mix coatings; the open diamonds are the improved powder mix coatings (see below). The dashed line represents the 60/40 stoichiometric ratio.

20polyester ratio, the best durability was achieved by the 60Al-Si/40polyester blend.[8,9] Thus, most of the effort concentrated on this composition.

A compilation of results over a range of Al-Si/ polyester coating thicknesses revealed that optimum final crack length is obtained with a thickness of 2–3 mils, although initial crack length was improved at thinner coatings (Fig. 4). The considerable scatter indicates that other factors besides coating thickness are also important in determining bond performance.

To reduce electrostatic charging and clumping of the powder during mixing and handling, it was blended, sieved and chopped several times. The equipment was carefully grounded, as was the spray hopper. A wire was inserted in the feed line to the spray gun to further reduce electrostatic charging. This procedure resulted in a more homogeneous coating showing an improved initial crack length that was closer to that of the controls but still slightly longer (Fig. 5). The final crack length was also improved, indicating that the joint could withstand greater stress even after humidity expo-

sure. X-ray photoelectron spectroscopy showed the crack propagated within the interphase region where the coating and the adhesive are mixed together with ~50% propagation within the adhesive. However, within the coating, the crack still predominantly travelled through the polyester phase (Fig. 3).

Further improvements in wedge test performance were achieved by eliminating the polymeric phase and depositing a stable metal, such as titanium. The results (Fig. 6) showed that performance of the plasma spray treatment was equivalent to that of PAA, even with the stronger FM-73 adhesive. Dry failure was cohesive in the adhesive and, in fact, resulted in a lower crack length (greater withstood stress) than the chemical controls. We speculate that the mixed interphase between the adhesive and the coating serves to toughen the bondline and increase the stress required to separate the adherends. The stress was so great that there was noticeable plastic deformation of the adherends following driving of the wedges. Final crack length was nearly identical to that of PAA specimens.
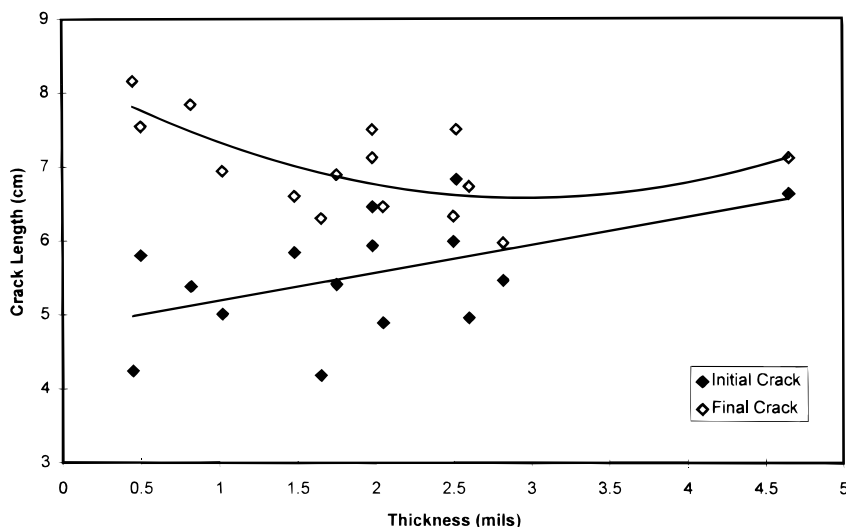

Figure 4. Initial and final crack lengths for wedge test specimens with 60%Al-Si/40%polyester coatings as a function of coating thickness. The adhesive is Cytec FM-123. The lines represent a linear (initial crack) or second-order polynomial (final crack) best fit.
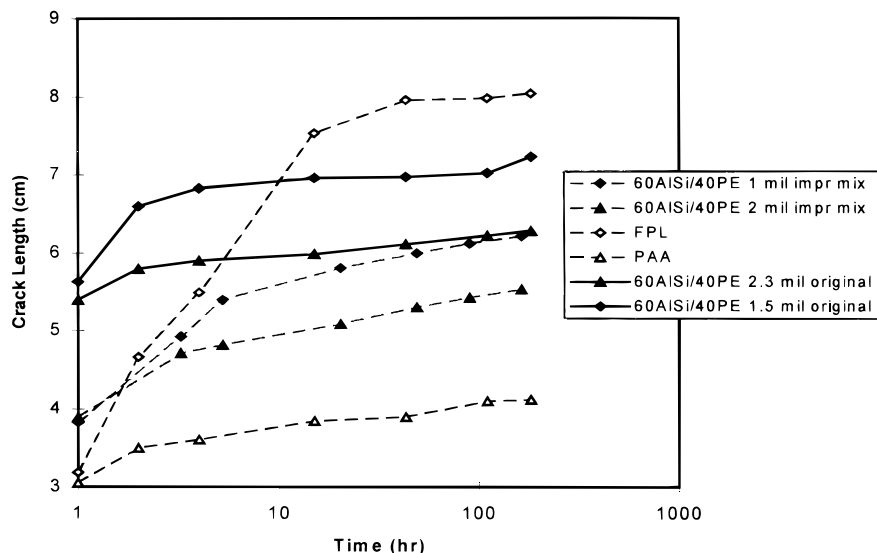
Figure 5. Wedge test results comparing original and electrostatic reduced mixing of 60%Al-Si/40%polyester. Also shown are the FPL and PAA controls. The adhesive was Cytec FM-73.

Titanium

Under moderate service or test conditions, several treatments for titanium give excellent performance, including chromic acid anodization (CAA), Turco 5578, sodium hydroxide anodization (SHA) and Ti-6Al-4V plasma spray, with failure generally cohesive within the adhesive.[7,8,23–28] These have recently been reviewed in Ref. 29. Most of the studies have involved testing at moderate temperatures, such as the wedge test illustrated in Fig. 7. Each of the best performing surfaces has a sufficiently microscopically rough morphology to provide opportunities for mechanical interlocking (physical bonding) with the primer or adhesive. However, under exposure to high temperatures, joints prepared with oxide-based treatments exhibit poor performance and begin to fail within the oxide or interfacially between the oxide and polymer or between the oxide and metal.[7,23,28,30] Plasma spray Ti-6Al-4V coatings on Ti-6Al-4V still gave failure within the adhesive—the ultimate test of a surface preparation. This performance

is illustrated in Fig. 8, which shows high-temperature wedge tests using LaRC TPI polyimide adhesive. The CAA joints failed between the oxide and the metal. The initial crack length was much longer than that of the plasma-sprayed joint, whose crack propagated within the adhesive. Some CAA specimens did not even survive driving of the wedge.

Similar poor performance for CAA and other oxide treatments was observed when the adherends were kept at high temperature for various times prior to bonding tensile pull buttons. The plasma-sprayed adherends were unaffected even after 165 h at 450 °C, while the CAA adherends showed 90% strength loss after only 3 h (Table 2).[7,28] In similar experiments, both SHA and PasaJell showed dramatic losses in tensile strength and change of the locus of failure following aging at 200–400 °C.[7] Long-term lap shear tests at 232 °C also showed loss of strength of CAA, SHA and PasaJell bondments.[31]

The cause of the poor performance of the oxide-treated bondments is the diffusion or dissolution of
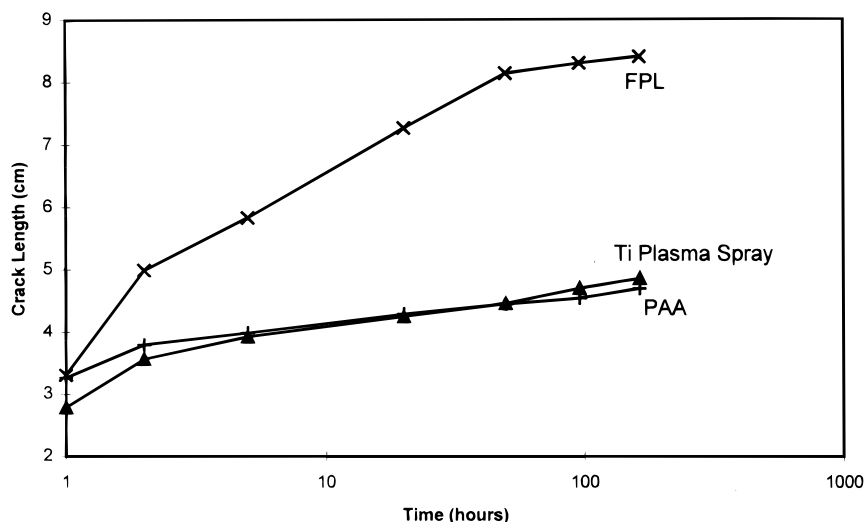

Figure 6. Wedge test results for a 2 mil Ti-6Al-4V coating on aluminum. Also shown are the chemical controls. The adhesive is Cytec FM-73.
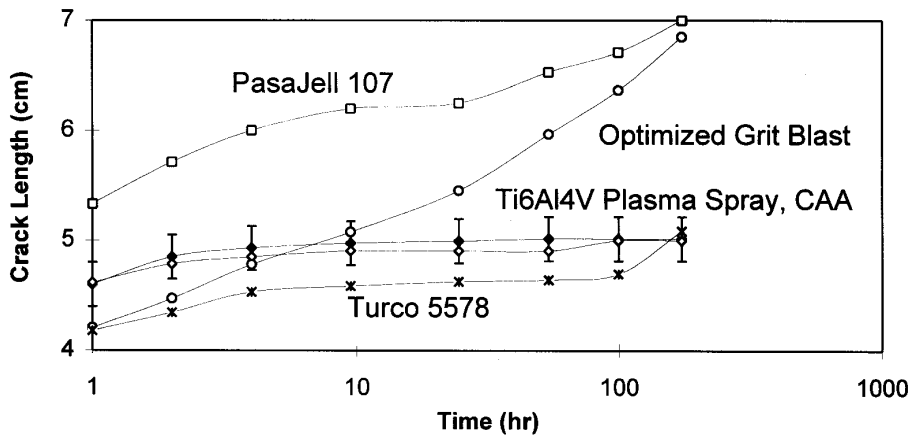
Figure 7. Wedge test results for different titanium surface treatments using Cytec FM-300M adhesive. Test conditions were 50 °C and >95% relative humidity.

oxygen into the titanium at high temperatures. This oxygen dissolution has two deleterious effects on bond performance (Fig. 9). The oxide itself would be non-stoichiometric and most likely contain voids, vacancies

Table 2. Adhesion tests of titanium adherends exposed to high temperatures

Treatment	Time at 450 °C (h)	Tensile strength (MPa)	Failure mode
CAA	0	22.8	Cohesive
CAA	3	2.7	Oxide
CAA	24	<0.7	Oxide
Plasma spray	0	22.8	Cohesive
Plasma spray	24	20.4	Cohesive
Plasma spray	165	20.8	Cohesive

and other chemical/physical defects that would weaken it. Additionally, oxygen dissolved in the alloy at concentrations as low as 0.1–1.0 at.% results in a highly embrittled zone just under the oxide/metal interface. Either effect would allow the joint to fail at or near the oxide/metal interface.

Because the plasma spray coatings contain minimal oxygen (only that resulting from the ambient oxide of the powders and the coatings), these failure mechanisms involving oxygen diffusion are not active. No thermally induced failures have been observed for plasma spray Ti-6Al-4V coatings on Ti-6Al-4V.

Steel

Plasma-sprayed Ni-Cr-Zn surfaces have been evaluated as corrosion-resistant coatings for steel adherends. The
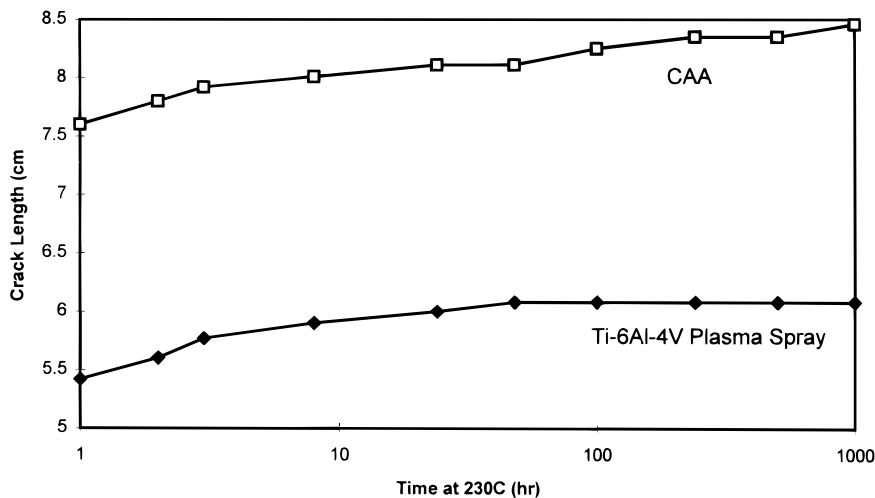

Figure 8. Wedge test performed at 230 °C with LaRC TPI polyimide adhesive and titanium adherends prepared by CAA or plasma spray.
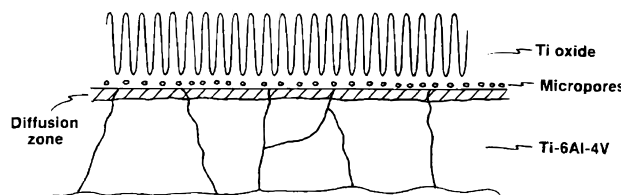

Figure 9. Schematic diagram showing proposed failure mechanism for CAA titanium bonds exposed to dry, high-temperature environments.

bondable stainless surface (BOSS) coatings are very rough on a visible to near-microscopic scale, with a mean surface roughness of ~7.6 μm (300 μin.) on the coating. For comparison, the roughness of a grit-blasted steel case surface is typically 1.8–3.8 μm (70–150 μin.). Both surfaces demonstrate a large-scale convoluted and irregular roughness. In each case, the convoluted morphology provides adequate opportunity for physical bonding or mechanical interlocking with the rubber.

Peel and tensile tests using acrylonitrile butadiene rubber (NBR) and ethylene propylene diene monomer (EPDM) rubber gave identical results for plasma-sprayed and grit-blasted steel adherends—cohesive failure within the rubber. A more severe test of the strength of the BOSS/steel and polymer/BOSS interfaces was obtained with tensile tests using epoxy adhesives (Table 3). Tensile strengths as high as 63 MPa (9160 psi) were measured. This value represents the cohesive strength of the coating and the bond between the coating and the substrate.

The bondability of the coatings after various environmental exposures was identical to those obtained shortly after spraying, indicating that exposure to accelerated conditions or to long-term ambient storage, did not affect the bondability of the coating. During the most severe exposures, the small amount of Zn acts as a sacrificial anode and protects the steel from corrosion. At the same time, the Ni-Cr acts as a structural framework so that bonds are not disrupted by corrosion of the Zn.

The greater roughness of the BOSS coating, compared to normal grit-blasted steel, suggests that the BOSS surface would be more tolerant of surface contamination. Tests of surfaces contaminated with HD-2 grease demonstrate this increased tolerance. The BOSS-coated and grit-blasted steel panels were contaminated with various levels of grease and bonded with EPDM insulation.

Figure 10 shows the peel strengths as a function of contamination level for both substrates. The grit-blasted specimens exhibited a sharp decrease in peel strength at ~100 μg cm^{-2} of grease, with the locus of failure shifting from cohesive in the rubber to the primer/steel interface. In contrast, the BOSS-coated specimens showed no decrease in peel strengths up to 200 μm cm^{-2} contamination and no change in the locus of failure.

Table 3. Bond strength measurements for plasma-sprayed steel

| Coating	Steel	Polymer	Exposure	Test	Strength	Failure mode
Ni-Cr	D6AC	NBR	Fresh	180° peel	25.9 kN m^{-1}	Rubber
Ni-Cr	D6AC	NBR	Fresh	Tensile button	4.33 MPa	Rubber
Ni-Cr	4130	EPDM	Fresh	90° peel	4.6 ± 0.2 kN m^{-1}	Rubber
Ni-Cr	4130	EPDM	7 days, humidity	90° peel	4.9 ± 0.5 kN m^{-1}	Rubber
Ni-Cr	4130	EPDM	30 days, humidity	90° peel	4.9 ± 0.4 kN m^{-1}	Rubber
Ni-Cr-Al	4130	1838 epoxy	Fresh	Tensile button	>27 MPa	Epoxy
Ni-Cr-Al	4130	1838 epoxy	18 months, ambient	Tensile button	>27 MPa	Epoxy
Ni-Cr-Al	4130	1838 epoxy	5 years, ambient	Tensile button	26 MPa	Epoxy
Ni-Cr-20Zn	D6AC	2216 epoxy	8 days alternate immersion	Tensile button	18 ± 0.5 MPa	Epoxy
Ni-Cr	D6AC	1838 epoxy	Fresh	Tensile button	63 MPa	Coating or coating/steel
Ni-Cr-Zn	D6AC	1838 epoxy	6 months beach	Tensile button	47 MPa	Epoxy/coating
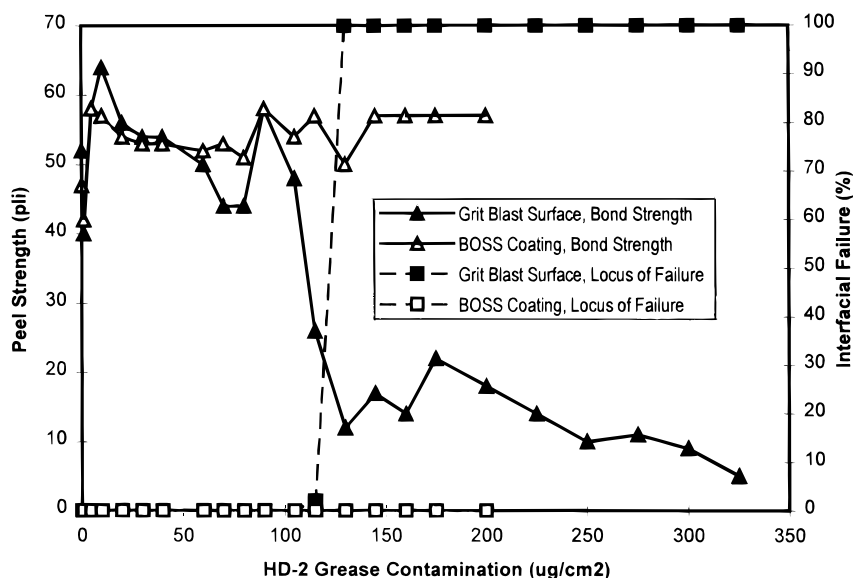
Figure 10. Peel strengths and percentage of interface failure for RDL 5066 EPDM insulation bonded to grit-blasted and BOSS-coated steel contaminated with various levels of HD-2 grease.

SUMMARY AND CONCLUSIONS

Plasma spray coatings provide bond performance equivalent to that of the best conventional treatments for aluminum and titanium adherends under moderate conditions. For titanium joints exposed to high temperatures, the plasma spray coatings exhibit superior performance. Plasma-sprayed steel substrates demonstrate excellent corrosion protection. Rubber and epoxy bonds to the coated steel exhibit failure within the polymer even after the substrate had been exposed to aggressive environments or long-term ambient exposure. Additionally the rubber bonds demonstrated greater tolerance to surface contamination than equivalent bonds prepared with grit-blasted steel.

The plasma spray process is environmentally benign compared to most conventional processes, with no gaseous or liquid wastes being emitted into the atmosphere or hydrosphere. The process is suitable for both original manufacture and subsequent repair. Furthermore, the ability to deposit a wide range of materials onto an equally wide range of substrates allows coatings to be engineered to specific applications and requirements.

Acknowledgements

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