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Hybrid Bond Layers for Cold Spray Metallization of CFRP Surfaces

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Abstract To metallize non-metallic surfaces by cold spray, bond layers (BLs) are often used to achieve a degree of compatibility between the substrate and the deposit and generally to mitigate the disparate properties of substrate and deposit. The objective of the present work is to design hybrid (Al-epoxy) BLs for metallization of epoxy-based carbon fiber-reinforced polymers (CFRPs) and to determine parametric effects on adhesion and deposition yield. The few studies that have focused on metal deposition onto thermoset composites have highlighted risks of erosion of the thermoset matrix and of fiber fracture. To address these problems, a BL is introduced to reduce erosion of CFRP and to provide surfaces for impinging powder to cold-weld onto and interlock. Effort is devoted to engineer a BL compatible with cold spray and to develop understanding of the quantitative effects of BL microstructure on adhesion. The BL microstructure strongly affected the adhesion of the deposit, which relied on BL-CFRP, powder-BL, and powder-powder bonding. Analysis of initial stages of deposition revealed matrix erosion and

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subsequent compaction, both contributing to mechanical interlocking. The findings indicate a pathway for CS deposition onto CFRP through judicious design of BLs.

Keywords adhesive strength · cold spray · deposition behaviors · hybrid bond layer · interfacial microstructure · metallization of polymer composites · thermoset composites

Introduction

The objective of this work is to determine the effects of microstructural features of the bond layer on the CS metallization of carbon fiber-reinforced polymers (CFRPs). In particular, a hybrid metal-polymer bond layer is designed and demonstrated for cold spray deposition of metal onto CFRP. Thermosets and CFRPs undergo erosion by impinging powder and do not readily bond with metal (Ref 1-3). In such cases, the use of a bond layer (BL) can mitigate substrate erosion and make the substrate compatible with CS metallization.

Polymers and composites often fail to meet design requirements because of poor resistance to impact and erosion in service, as well as poor electrical and thermal conductivity (Ref 4). Surface metallization can impart metallike durability while retaining the high specific strength (strength-density ratio) intrinsic to composites and polymers (Ref 4, 5). In particular, metallization of polymer composites can impart resistance to erosion and abrasion (Ref 6). Potential applications include leading edges of turbine engine fan blades and wind turbine blades (Ref 7, 8), thermal management to dissipate heat loads, and in-field repairs.

In the past decade, cold spray studies have reported that thermoplastic metallization was feasible because of thermal softening (Ref 3, 6, 9-15), whereas work on thermoset composites has underscored the risk of substrate erosion (Ref 3, 6, 10, 15, 16). Cold spray of copper and aluminum powders erodes the thermoset matrix and fractures the reinforcing fibers upon powder impact, which hinders the formation of a continuous, densified, and defect-free deposit (Ref 16). On the other hand, soft and ductile powder such as tin tends to adhere only weakly to thermoset substrates (Ref 17-23). Few studies (Ref 10, 16, 23) have reported the adhesive strength between cold-sprayed metal deposits on thermoset substrates because of the difficulty in obtaining a continuous coating and weak deposit adhesion (spontaneous delamination). Mechanical interlocking of cold-sprayed tin with carbon fiber and surface epoxy cracks generated by impinging powder (Ref 16, 23) yielded an adhesive strength of 13 MPa. To address these problems, a hybrid, metal-polymer bond layer was designed and demonstrated reduced erosion and increased cold-welding of impinging powder.

Approaches to make thermoset composite surfaces amenable to spray metallization have been proposed in the literature. Electroless nickel plating followed by nickel and copper electroplating on CFRP substrates required multiple steps before copper could be deposited by cold spray (Ref 24, 25). Metal filler-reinforced surface layers have been metallized by detonation gun spray and arc spray (Ref 26, 27). These studies showed that mechanical interlocking and bonding to the filler particles were essential to adhesion. Metal mesh reinforced surface layers have also been metallized by arc spray, air plasma spray and yielded adhesive strength of 7.6, 22.6 MPa, respectively (Ref 27, 28). These studies highlighted the importance of grit blasting of the surface layer to provide regions for mechanical interlocking. Cold spray can eliminate the need for prior surface roughening while still achieving coating-substrate interlocking.

The present work provides insights into the design requirements for hybrid metal-polymer bond layers and the powder adhesion mechanisms. A relationship between the exposed metal surface area and adhesive strength is demonstrated. The deposition mechanisms onto isolated (filler particle) and continuous (wire mesh) metal reinforced bond layers are identified, as well as the dependence of these mechanisms on microstructural parameters of the BL. The findings provide a basis for design of hybrid bond layers for cold spray onto CFRP substrates.

Experimental Methods

Bond Layer Preparation and Cold Spray Deposition

Commercial purity Al powder $(38.9 \pm 16.2 \,\mu\text{m}, D50 = 31 \,\mu\text{m})$ was cold sprayed onto CFRP laminates with a hybrid metal-polymer bond layer (BL) on the surface.

The CFRP consisted of ten plies (0/90) of prepreg (Toray 2510, Toray Industries, Inc., Tokyo, Japan). The reinforcements of the hybrid bond layer included Al filler particles and woven Al 5056 120×120 wire mesh (McMaster-Carr, Elmhurst, IL, USA) embedded in an epoxy film adhesive with Al filler (LOCTITE® EA 9658 AERO, Henkel, Düsseldorf, Germany). Two types of hybrid bond layers were investigated, as described in Table 1. The components of the hybrid layers included an epoxy film adhesive filled with Al particles, and an Al wire mesh embedded in the film. The contents of Al filler (g/ cm^2) were 0.16 and 0.24 g/cm². Figure 1 shows the three types of wire mesh-original with round wires (BL-Round), wires flattened once (BL-Flat1) and twice (BL-Flat2). The bond layer and CFRP were compacted and cocured in an out-of-autoclave process following manufacturer's guidelines. The substrate was then cold sprayed with a VRC Gen III CS system (VRC Metal Systems,

Table 1 Al filler BL with 0.16, 0.24 g/cm² loading content and Al mesh BL flattened 0-2 times

Al Filler BL (g/cm ²)	BL-0.16 (0.16)	BL-0.24 (0.24)	
Al Mesh BL	BL-Round	BL-Flat1	BL-Flat2



Fig. 1 Al 120×120 mesh (a) original—Round, (b) flattened once—Flat1, and (c) flattened twice—Flat2





Table 2 Material properties and Johnson–Cook parameters

Material	Pure Al (Ref 37, 38)	Al 5056 (Ref 39)	Epoxy (Ref 40, 41)	Film adhesive
Density (kg/m ³)	2710	2640	1400	1884.7
Young's modulus (GPa)	68.9	71	2.8	5.52
Poisson's ratio	0.33	0.33	0.3	0.31
Thermal conductivity (W/m·K)	210	117	0.2	0.55
Specific heat, (J/kg·K)	904	904	1000	948.93
Inelastic heat fraction	0.9	0.9	0.9	0.9
Yield strength, A (MPa)	148.4	140	20.66	67.92
Hardening coefficient, B (MPa)	345.5	426	110.4	197.39
Strain-hardening exponent, n	0.183	0.34	0.5497	0.41
Strain rate constant, C	0.001	0.015	0.0441	0.03
Softening exponent, m	0.895	1		
Melting temperature, T _m (K)	916	911		
Reference temperature, $T_r(K)$	293	300		
Reference strain rate, $\dot{\epsilon_0}$ (1/s)	1	1	1	1



Fig. 3 Cross section of (a) BL-0.24 and (b) BL-Round before cold spray



Fig. 4 Thickness evolution of (a) Al filler BL and (b) CS deposit on Al mesh BL

Rapid City, SD, USA) using He at 400 °C, 2 MPa, a standoff distance of 25.4 mm, and a raster velocity of 400 mm/s. The mean powder velocity of 203.5 m/s was measured using a laser velocimetry system (HiWatch HR1 CS, Oseir, Tampere, Finland).

Microstructural Characterization and Lug Shear Test

The hybrid metal-polymer bond layer and the deposits were sectioned and polished. The thickness of the Al filler BL and the deposit on Al mesh BL were measured using light microscopy (VHX-5000, Keyence, Osaka, Japan). The samples were also ion polished (JEOL SM-09010, Tokyo, Japan) for SEM (Nova NanoSEM 450, Thermo Fisher Scientific, Waltham, MA, USA) imaging to investigate the microstructural evolution as a function of the number of CS passes, using both plan and cross sections. The cross sections were used to calculate the root-meansquare (RMS) roughness of the interface between the deposit and BL along mesh wires. Lug shear strength of the deposit was measured according to MIL-J-24445A which consisted of shearing off the deposit at the bond line without the use of adhesives. The resulting fracture surfaces were examined by SEM to determine the adhesion mechanism.

Finite Element Analysis

Finite element analysis calculations (ABAQUS/Explicit) simulated the Al powder impact at a velocity of 203.5 m/s on hybrid metal-polymer bond layers. The substrate models

depicted the BL geometries and dimensions, and the powder particles were modeled as solid spheres. The impact simulations were conducted in 3D (mesh type C3D4T, four-node thermally coupled tetrahedron, linear displacement and temperature). The mesh resolution was 1/32 of the powder diameter. A fixed boundary condition was applied to the bottom of the substrate, and a free boundary condition was applied to the sides. Contacts between the powder and the substrates were defined as surface-to-surface hard contact with a 0.3 coefficient of friction. Powder impact was assumed to be adiabatic with the inelastic heat fraction set to 0.9. Figure 2(a) shows the two locations of single powder impact on Al filler BL:

- 1. Al filler
- 2. Epoxy

Figure 2(b) shows the three locations of single powder impact on Al mesh BL:

- 1. Metal mesh
- 2. Interface of metal mesh and film adhesive
- 3. Film adhesive

The Johnson–Cook (JC) strain rate dependence plasticity model (Ref 29-33) was used to calculate yield stress and is expressed as

$$\sigma_f = \left[A + B arepsilon_p^n
ight] \left[1 + C {
m ln} \left(rac{\dot{arepsilon}_p}{\dot{arepsilon}_0}
ight)
ight] \left[1 - \left(rac{T - T_r}{T_m - T_r}
ight)^m
ight]$$

The film adhesive was treated as a homogeneous particulate polymeric composite comprised of an epoxy matrix and aluminum filler (volume fraction of 37%). Kerner's equation (Ref 34, 35), the inverse rule of mixtures, and the Maxwell model (Ref 36) were used to obtain the Young's modulus, Poisson's ratio, and thermal conductivity of the film adhesive, respectively. The rule of mixtures was used to calculate the density, specific heat, and the Johnson-Cook parameters. In conjunction with the Johnson-Cook plasticity model, a tensile failure model (ABAQUS/ Explicit) was used to determine the fracture onset of film adhesive. Fracture occurs when the pressure stress exceeds the hydrostatic cutoff stress of 120 MPa. Material properties and Johnson-Cook parameters for pure Al, Al 5056, epoxy, and film adhesive used in the simulation are listed in Table 2.

Results and Discussion

Figure 3 shows typical cross sections of Al Filler BL and Al Mesh BL. The Al filler particles of BL-0.24 were evenly dispersed within the BL with no apparent agglomeration (Fig. 3a). On the other hand, Fig. 3(b) shows a less concentrated Al filler content compared to Fig. 3(a) because no







Fig. 6 FEA showing CS powder erosion of Al filler BL when CS powder was in (a) direct and (b) indirect contact with Al filler particle

additional Al filler particles were added. The Al mesh was placed near the surface in BL-Round to interact and interlock with impinging cold-sprayed powder. In both cases, the substrate surfaces were flat and were cold sprayed without prior surface treatment.

BL and deposit thickness as a function of number of passes revealed the dependence of deposition yield on BL design, as well as a competition between powder deposition and substrate erosion. Figure 4 shows that a BL comprised of epoxy reinforced by continuous metal wire mesh, rather than dispersed filler particles of Al, was required for CS metallization of CFRP. Regardless of Al filler content, the BL was consistently eroded, manifested by the reduction in BL thickness with increasing number of CS passes (Fig. 4). The eroded thickness ranged from 100 to 300 μ m with no clear dependence on BL thickness or number of passes.

On the other hand, Fig. 4(b) shows increasing deposit thickness for all three wire meshes, indicating that incorporation of continuous metal wires promoted deposition. The woven wires absorbed and dissipated the impact energy of CS powder, preventing degradation or fracture of the epoxy matrix. The metal wires also served as an effective substrate for metal-metal deposition. The first pass of BL-Round shows a coating thickness $\sim 100 \ \mu m$ less than the first pass coating thicknesses of BL-Flat1 and BL-Flat2. The difference indicated that the round metal wires resulted in greater erosion of the BL compared to flat metal wires. For subsequent passes, the thicknesses obtained were comparable.

The decrease in BL thickness for bond layers with Al filler particles was caused by erosion, represented by BL-0.24 after three passes (Fig. 5). Cracks consistently formed in the BL and propagated upon impingement of powder, causing detachment of filler and spallation of epoxy. Figure 5(a) shows the roughened surface and the presence of near-surface cracks. Figure 5(b) shows that cracks





generally extend vertically around Al filler particles and laterally into the epoxy. The top surface shows roughness, partially enclosed Al powder in epoxy, and craters formed either by impinging Al powder or by detached Al filler. The observations support the contention that dispersed filler particles in epoxy did not provide a surface suitable for metallic bonding or mechanical interlocking.

Finite element analysis of a single CS powder particle impinging on Al filler embedded in the BL also demonstrated the erosion mechanism. Two cases of impingement were investigated (Fig. 6) to describe and predict possible interactions between CS powder and filler BL. When the powder directly impinged on filler embedded in epoxy, the filler served as a stress concentration site, which could initiate cracks. When powder impinged on epoxy with filler in proximity, the stresses were concentrated similarly. With successive impacts, there was low probability for powder to accumulate on the filler BL. Isolated Al filler particles in the epoxy matrix did not effectively absorb CS powder impacts, preventing deposition.

Figure 7 shows the interactions between CS powder and the BL-Round during the first and second passes. During the first pass, powder deposited onto Al wires and eroded

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epoxy exposed in mesh openings, revealing a grid-like pattern analogous to a projection of the wire mesh (Fig. 7a, b). In certain regions, deposition was also observed between wires that bridged the wire openings filled with epoxy. In both cases of deposition, powder-powder bond lines were apparent because of inadequate peening. CS powder completely covered and bonded loosely to the wires.

After a single pass, CS-deposited powder conformed to the round wire surface with a clear interface outline between (Fig. 7b, sites indicated by arrows). The curved wire substrate presented inherent difficulties for impinging powder because of the gradual change in impact angle associated with wire curvature. Nevertheless, during the first pass, metallic bonding was achieved, although it was limited and infrequent, as shown in Fig. 7(c). CS powder particles were flattened and displayed fine grains, both caused by severe plastic deformation upon impact. The fine grains were also present at the top and bottom of the wire cross section, a consequence of deposition at a 90° angle.

The second pass filled the eroded regions with CS powder and formed a continuous deposit on the BL. The deposit extended into previously eroded regions,



Fig. 8 Cross section and plan view (epoxy only) of simulated stress distribution of CS powder impinging (a) Al wire, (b) interface of Al wire and epoxy, and (c) epoxy matrix



Fig. 9 (a) SE and (b) BSE (outlined in (a)) micrographs of the interface microstructure of BL-Round-6-Pass. The numbers label three regions of CS powder-BL interaction

effectively anchoring the coating in the bond layer via a tortuous coating-substrate interface, shown in Fig. 7(d). However, discrete interfacial cracks formed because of relatively weak bonding of powder to epoxy and the inadequate metallic bonding between powder and wires because of low impact velocity. Cracks were observed between Al wires and the epoxy matrix. These cracks were attributed to debonding induced by stresses that arose

during cold spray, and mismatch in coefficient of thermal expansion between Al and epoxy. Peening of powder at mesh openings was not sufficient to fully eliminate porosity. These findings signal the need for further refinements to material, design, and process.

Partial deposition and erosion of the BL-Round by CS were also predicted in simulations of CS powder impingement on Al wire embedded in epoxy.



Fig. 10 BSE micrograph of BL-Round-6-Pass (left) vs BL-Flat2-1-Pass (right) in (a, b) regions 1, (c, d) 2, and (e, f) 3

Figure 8(a) shows that loads were transferred along the wire direction and into the epoxy that absorbed the powder impact, leading to cracks at the wire-epoxy interface. Figure 8(a) also shows that the peak stress in epoxy (22 MPa) was an order of magnitude less than the stress in the Al wire (399 MPa), allowing the Al wire to provide a substrate for metal–metal deposition. Erosion occurred when the powder directly contacted epoxy, as shown by white arrows in Fig. 8(b-c). These results were consistent with experimental findings presented in Fig. 7(b) in terms of the inadequate metallic bonding between Al powder and Al wire mesh and the erosion of epoxy after a single pass.

Figure 9 shows the microstructure of a consolidated CS coating on BL-Round after six passes. The coating was dense and exhibited no apparent powder-powder interfaces or evidence of entrapped epoxy debris (from erosion) or

porosity, as shown in Fig. 9(a). The microstructure of the coating-BL interface resembled that of Fig. 7(d), indicating that with the CS parameters selected, two passes were sufficient to fully transition from metal-polymer deposition to metal-metal deposition.

The as-sprayed coating spontaneously detached from BL-Flat1 and BL-Flat2 after cold spray deposition for lug shear tests. The delamination was related to the lower interface roughness, which decreased from RMS 26.1 (with BL-Round) to RMS 11.2 (with BL-Flat1), and 7.9 μ m (BL-Flat2), reducing the degree of interlocking. BL-Round provided surfaces for interlocking and metallic bonding between CS powder and Al mesh in regions 1 and 2, and mainly metallic bonding in region 3 (Fig. 9a). Within the deposit, powder deformed evenly and exhibited no correlation between grain size and distance from the wire,

Fig. 11 Substrate side of the fracture surface of (a) BL-Round after shear test, (b) BL-Flat1 and (c) BL-Flat2 before shear test (delaminated after CS)



indicating that peening was only effective to the immediate underlayer (Fig. 9b). This observation underscored that deposit bonding to BL relied primarily on the first two passes.

The morphology of the coating-BL interface depended primarily on mesh shape (round vs flat), as shown by comparison of BL-Round and BL-Flat2 (Fig. 10). The sections in Fig. 10 reveal three distinct regions (also shown in Fig. 9a). Region 1 shows deformed CS powder conformed around the Al wires (Fig. 10a). The area surrounded by three wires outlined in white (two parallel wires on top, one perpendicular beneath) in BL-Round was only slightly eroded. In BL-Flat2, the bottom wire was nearer the surface (Fig. 10b). However, the thin surface layer of epoxy and the leveled surface caused crack formation at the interface, preventing interlocking.

Region 2 showed similar anchoring mechanisms, with powder filling the valley (outlined in white) where two perpendicular wires met (Fig. 10c-d). However, the penetration depth of BL-Round was 91 μ m, compared to only 17 μ m of BL-Flat2. Region 3 showed that in both BL-Round and BL-Flat2, discrete cracks and intermittent metallic bonding were present. The cracks in BL-Flat2 were straight, while those in BL-Round were not. These cracks can be reduced/eliminated by adjustment of CS parameters. The significant finding is that the high aspect ratio (3:1) and leveled surface of the *flattened* wires provided a relatively flat interface and an unobstructed/linear path for crack propagation.

The microstructural differences described above affected the values of strength measured. The shear strength of BL-Round was 14.5 ± 1.5 MPa, whereas the shear strength of BL-Flat1 and BL-Flat2 was much less and could not be measured because the coatings delaminated from the BL after CS. The delamination was attributed to crack propagation through the coating-BL interface, which readily occurred because of the absence of mechanical interlocking with the wire mesh and the flat interface. Using a simple rule of mixtures, interlocking (deposit extensions into the mesh opening) in the 27% open area of the Al mesh yields a maximum shear strength of 16.2 MPa with 60 MPa as the bulk pure Al shear strength (Ref 42). The 1.7 MPa difference was attributed to the incomplete bonding of powder in the mesh openings and can be reduced by adjusting CS parameters.

The plan view of the fracture surfaces in Fig. 11 shows the interlocking features of the CS deposit. The CS powder was anchored in epoxy at the wire mesh openings of BL-Round, which is consistent with Fig. 7 and 10. Figure 11(a) also shows voids (white arrows) formed by the detachment of coating after testing. These interlocking features were absent in BL-Flat1 and BL-Flat2, as shown in Fig. 11(b-c). The ratio of bonded powder to craters on wire surfaces decreased for flatter wires. This finding was attributed to the change in impact angle on a curved surface (compared to a flattened wire surface) and will be the subject of future work.

Conclusions

Design and implementation of a hybrid metal-polymer bond layer enabled cold spray deposition of fully dense CP Al onto CFRP laminates and promoted adhesion. The bond layer consisted of a metal wire mesh embedded in an adhesive film, and the effects of metal content, form, and shape were determined. The first two passes of powder that impinged on the bond layer effectively governed deposition and adhesion mechanisms, as shown by experimental findings and finite element analysis.

This work outlines a viable approach for cold spray of metal onto CFRPs, a problem that is largely unsolved today. A proposed solution-a conformable hybrid bond layer-can be applied to both polymer and composite substrates through co-curing or secondary bonding, both of which are compatible with conventional manufacturing processes for polymers and composites. Surface metallization by CS can be used to extend the service life of polymer and composite components, and CS can be repeated for repair and restoration of the surface layer. Refinements of the BL microstructure have potential to expand the use of cold spray to impart metal-like durability to polymers and composites with increased resistance to erosion and impact, and increased conductivity. Custom bond layers can be tailored to the intended applications and engineered to join different classes of materials for components that face variable service conditions. Interface engineering of the bond layer microstructure can enable hybrid process routes, opening new possibilities for lightweighting of metallic components.

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