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Microstructure and Properties of Thermally Sprayed Functionally Graded Coatings for Polymeric Substrates

M. Ivosevic, R. Knight, S.R. Kalidindi, and G.R. Palmese
Drexel University, Philadelphia, Pennsylvania

J.K. Sutter
Glenn Research Center, Cleveland, Ohio

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M. Ivosevic, R. Knight, S.R. Kalidindi, and G.R. Palmese
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Drexel University
Philadelphia, Pennsylvania 19104

J.K. Sutter
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Abstract

The use of polymer matrix composites (PMC's) in the gas flow path of advanced turbine engines offers significant benefits for aircraft engine performance but their useful lifetime is limited by their poor erosion resistance. High velocity oxy-fuel (HVOF) sprayed polymer/cermet functionally graded (FGM) coatings are being investigated as a method to address this technology gap by providing erosion and oxidation protection to polymer matrix composites. The FGM coating structures are based on a polyimide matrix filled with varying volume fractions of WC-Co. The graded coating architecture was produced using a combination of internal and external feedstock injection, *via* two computer-controlled powder feeders and controlled substrate preheating. Porosity, coating thickness and volume fraction of the WC-Co filler retained in the coatings were determined using standard metallographic techniques and computer image analysis.

The pull-off strength (often refered to as the adhesive strength) of the coatings was evaluated according to the ASTM D 4541 standard test method, which measured the greatest normal tensile force that the coating could withstand. Adhesive/cohesive strengths were determined for three different types of coating structures and compared based on the maximum indicated load and the surface area loaded. The nature and locus of the fractures were characterized according to the percent of adhesive and/or cohesive failure, and the tested interfaces and layers involved were analyzed by Scanning Electron Microscopy.

Introduction

For the successful integration of high temperature polymer matrix composites (PMC's) into jet engines, key long-term performance characteristics such as erosion resistance and thermo-mechanical fatigue (TMF) behavior must be addressed. Even with significant benefits such as weight savings, improved strength, reduced part counts and reduced manufacturing costs, the successful application of PMC's into the gas flow path of advanced turbine engines is still limited by their poor erosion resistance. Yet, there is little published information describing solutions for this erosion problem on polymer composites [1]. Erosion-resistant coatings are needed to protect the composite materials, at least through the first overhaul interval, and preferably for the full life of the component.

In addition, components used in engine gas flow paths must exhibit an acceptable surface finish to ensure good aerodynamic performance. The goal of this work was to develop thermally sprayed erosion/oxidation resistant functionally graded (FGM) coatings for PMC substrates. The goal was to grade the coating composition from pure polyimide, similar to the matrix of the PMC substrate on one side, to 100% WC-Co on the other [2]. Both step-wise and continuous gradations of the WC-Co loading in these coatings are being investigated. A 100% WC-Co outer layer should provide improved wear and oxidation protection to the substrate. The thermosetting polyimide coating matrix should help manage the differences in coefficient of thermal expansion (CTE) between pure WC-Co and the PMC substrate material, and improve the TMF properties of the coating/substrate system. Secondary goals included improved surface finish relative to coatings developed during a previous study [3] and evaluation of the thermo-mechanical fatigue (TMF) properties of the coating/substrate system. Results reported here focus on coating microstructure and aspects of the adhesive and/or cohesive properties of the FGM coatings.

Background

The limited work reported on the thermal spray deposition of erosion-resistant coatings on fiber-reinforced polymer composites [4–6] almost exclusively used metallic (zinc, nickel or aluminum) bondcoats or polymeric (polyamide, polyimide or PEEK) layers to enhance the adhesion of the wear-resistant topcoats (typically carbides, borides or nitrides) to PMC substrates. However, for applications subject to thermal fatigue or thermo-mechanical fatigue this may be a limitation owing to the large differences in CTE within the overall coating/substrate system. Work on the plasma spraying of epoxy-based materials [7, 8] reported some difficulty in obtaining satisfactory build-up and properties of thermosetting polymeric coatings. It was reported that external substrate preheating might enhance the deposition behavior and curing reaction during the thermal spraying of thermosetting polyimide materials. Preliminary proof-of-concept evaluations resulting in the successful deposition and build-up of pure polyimide and polyimide/WC-Co powders onto electrically preheated PMR-15 substrates were previously reported [2, 7]. Improved substrate preheating set-ups were subsequently developed during this work.

The potential benefits of using polymer matrix coatings filled with commercial or nano-sized ceramic (e.g. silica or alumina) reinforcements or fillers has been reported by a number of authors [9, 10]. The high velocities and non-uniform velocity distributions of thermal spray jets, in combination with variations in particle size, density and morphology can, however, result in significant segregation when dissimilar materials are co-sprayed. This represents a serious challenge when the desired feedstock materials have significant differences in density, as was the case for the two materials used in this work, polyimide and WC-Co, which have densities of 1.39 and 12.5 gm/cm^3, respectively. Consequently, spraying techniques that minimized material segregation were a key requirement. One approach considered for minimizing material segregation was dry ball-milling to produce a "composite" feedstock, with the ceramic phase embedded into the polymer component. Ball-milling of the polyimide and WC-Co powders was investigated here, however, as reported previously [2], little or no embedding of the harder WC-Co particles into the softer, yet somewhat brittle, thermosetting polyimide particles was obtained.

An alternative approach investigated to minimize material segregation was a powder feeding configuration that allowed simultaneous internal and external feeding of the two materials (Fig. 1). External feeding of the much denser WC-Co component and internal feeding of the polyimide enabled a balance between the differences in momentum and kinetic energy of the two feedstock materials to be established. Moreover, this configuration also helped to minimize the differences in heating of the polymer and cermet feedstocks. Internal feeding of the polyimide material, with a much lower thermal conductivity (~0.2 W/mK), afforded longer residence times within the HVOF jet than those experienced by the externally fed, higher thermal conductivity (~100 W/mK) WC-Co.

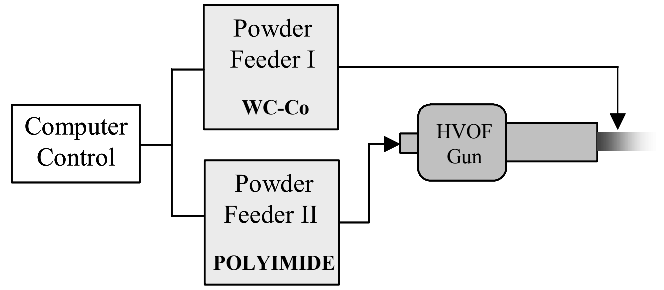

Figure 1: Simultaneous internal/external powder feeding of the polyimide and WC-Co feedstocks.

Experimental Approach

Thermosetting polyimide (end-capped) of an oligomeric form was selected as the matrix material for the proposed FGM coatings. Molding polyimide material produced by Maverick Corp. was mechanically crushed and cryo-ground (Shamrock) to produce a powder that flowed well, with a particle size distribution in the range (–100, +20 µm), suitable for HVOF spraying. A previous study [3] utilized a conventional 88/12 WC-Co powder (Sulzer Metco 71VF-NS) for the functional surface. A similar WC-Co powder - Amperit 515.0 (H.C. Stark, Inc.), with a particle size range of (–22.5, + 5.6 µm) was selected as the reinforcing/filler material. Substrates were 25 x 75 x 3 mm (1 x 3 x 1/8 in.) coupons of carbon-fiber reinforced polymer matrix (PMR-15) composite material. Prior to spraying, the PMR-15 substrates were grit blasted using 250 µm (60 mesh) Al_2O_3 grit.

Pure thermosetting polyimide coatings and composite polyimide/WC-Co composite coatings were sprayed using a Stellite Coatings, Inc. Jet Kote® II HVOF combustion spray system, using hydrogen as fuel gas. All the coatings were sprayed using the spray parameters summarized in Table 1.

Table 1: HVOF spray parameters for depositing pure polyimide and various ratios of polyimide/WC-Co coatings on graphite fiber reinforced PMR-15 composite substrates.

HVOF Spray Parameter	Value
Spray distance (m)	0.15
Polyimide feed rate (gm/min)	2 - 9
WC-Co feed rate (gm/min)	20 - 50
Carrier gas	Ar
Carrier gas flow rate (m^3/s)	0.5 x 10^{-4}
H_2:O_2 ratio	0.4 - 0.5
H_2 and O_2 flow rates (m^3/s)	3 x 10^{-3} / 6-7 x 10^{-3}
Surface speed (m/s)	0.11
Substrate temperature (°C)	230 - 340

Coating thicknesses obtained were in the range 300–600 µm (12–24 mils). A 100% WC-Co outer layer, or topcoat, was sprayed using a UTP UNI-Spray-Jet™ flame-powder spray system, again using hydrogen as the fuel gas. The flame spray parameters are summarized in Table 2.

Table 2: Typical flame spray parameters used for spraying pure WC-Co topcoats onto polyimide/WC-Co coatings.

Flame Spray Parameter	Value
Spray distance (m)	0.08
WC-Co feed rate (gm/min)	70
Carrier gas	Ar
Gas pressure - H_2 (MPa)	2900
Gas pressure - O_2 (MPa)	5075
Gas pressure - Cooling air (MPa)	1450
Surface speed (m/s)	< 0.11
Substrate temperature (°C)	15

Spraying of the graded coating structures required reliable, repeatable simultaneous feeding of two materials, WC-Co and polyimide. A LabVIEW® -based computer-controlled system was developed and used for simultaneously controlling two identical Praxair Model 1207 volumetric powder feeders (Fig. 1). Electric strip heaters (Omega type PT 502/120) were used as external heat sources in contact with the rear of the substrate, together with a hot air gun directed at the front face [11]. Preheating was only used while spraying the pure polyimide and polyimide/WC-Co layers, not during the flame spraying of the WC-Co topcoat.

Porosity, coating thickness and volume fraction of the WC-Co filler retained in the coatings were determined using standard metallographic techniques - sectioning, mounting and polishing - and computer image analysis using Scion Image software. The pull-off strength, often referred to as adhesion, of the coatings was determined according to the ASTM D-4541 standard test method by measuring the greatest normal tensile force that the coatings could withstand. A schematic of the pull-off test method used is shown in Figure 2.

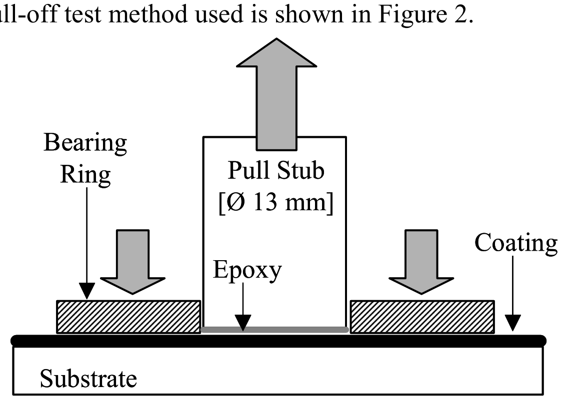

Figure 2: Schematic of the ASTM D 4541 pull-off test method.

The test conditions used were as follows:

- Self-aligning sample fixtures adapted for laboratory use on an Instron 5800R mechanical testing machine (Model 58R1127 in conjunction with Instron Merlin software).
- Temperature and relative humidity of 20 °C and 65%, respectively.
- 12.7 mm (0.5 in.) aluminum pull stubs (type PS-25, M. E. Taylor Engineering, Inc.) prepared in accordance with the ASTM D 2651 guide for the preparation of metal surfaces for adhesive bonding.
- 3M type DP-460 off-white adhesive, with a curing time of 60 minutes at a temperature of 60 °C.

The bearing ring was located concentrically around the loading fixture on the coating surface (Fig. 2).The adhesive/cohesive strengths of three different types [Table 3] of coating were measured and compared based on the maximum indicated load and the original surface area stressed. Six samples of each coating type were tested and the mean value of the results reported.

Table 3: Types of coating evaluated by tensile adhesion testing.

Coating Designation	Composition
Pure Polyimide Coating	*A + B*
2-Layer Coating	*A + B + C*
3-Layer Coating	*A + B + C + D*

Where:

A – Carbon-fiber reinforced PMR-15 substrate.
B – Pure polyimide coating layer.
C – WC-Co/polyimide composite coating layer.
D – Pure WC-Co topcoat.

The nature and locus of the failures were characterized according to the percent of adhesive and cohesive failures. The surfaces of the as-tested samples were subsequently analyzed by scanning electron microscopy using an Amray Model 1830 SEM.

Results and Discussion

An important first step of this work was the development of HVOF spray parameters for depositing the pure polyimide matrix material onto PMC substrates, owing to the reported difficulty in obtaining satisfactory initial coating build-up. An adherent pure polyimide coating (region B, Fig. 3) was obtained after extensive parameter development and understanding of the substrate preheating requirements. These coatings exhibited good adhesion and clean coating/substrate interfaces with little or no apparent debonding.

A significant level of porosity – the large black areas within region B of Fig. 3 - was observed in the polyimide coating. The high void level (~26%) may have been due to gas evolution during the crosslinking reaction (the onset of polymerization of the oligomer end-groups) that began above ~280 °C. Out-gassing of the PMC substrate material may also have contributed to the excessive porosity observed, even though the substrates were vacuum dried at ~120 °C for ~2 days and stored in a desiccator prior to spraying. Since gas evolution during crosslinking is a function of the polyimide chemistry and imidization kinetics, this problem may be addressed in the future by further studying these relations.

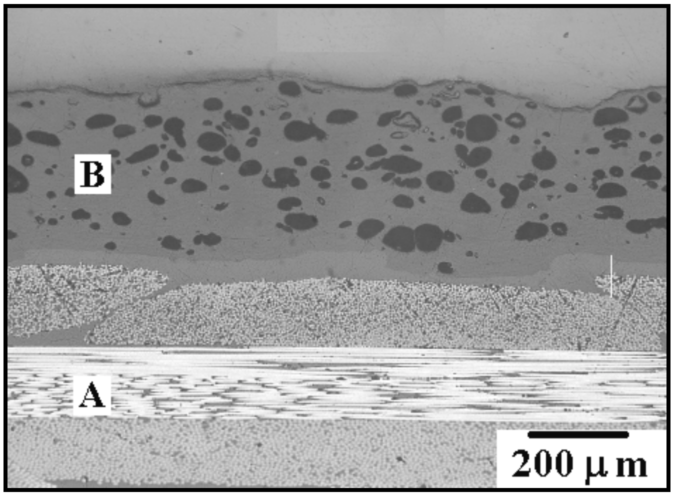

Figure 3: Pure polyimide coating (B) HVOF sprayed onto a carbon-fiber reinforced PMC substrate (A).

Two- and three-layer polyimide/WC-Co coating micro-structures are shown in Figs. 4 and 5.

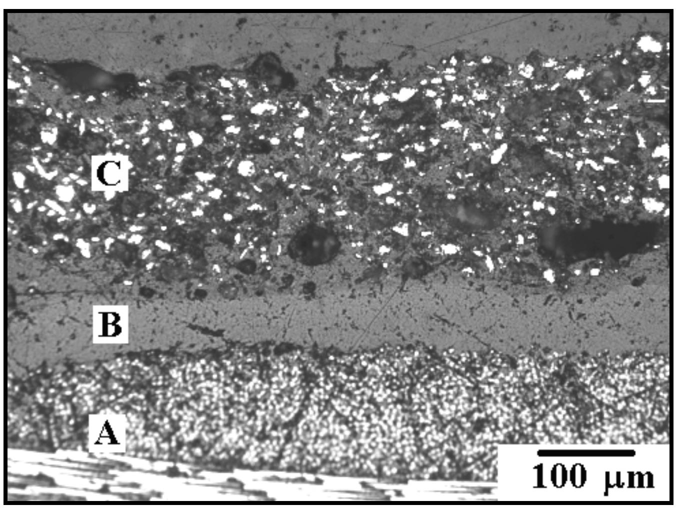

Figure 4: Two-layer HVOF sprayed FGM coating comprising a WC-Co/polyimide outer layer (C) and a pure polyimide layer (B) on a PMC substrate (A).

Image analysis of the WC-Co/polyimide layers (region C) shown in Figs. 4, 5, 8 and 13, indicated that the composite layer had the following typical composition:

- 46% polyimide matrix.
- 26% WC-Co filler.
- 28% voids/porosity.

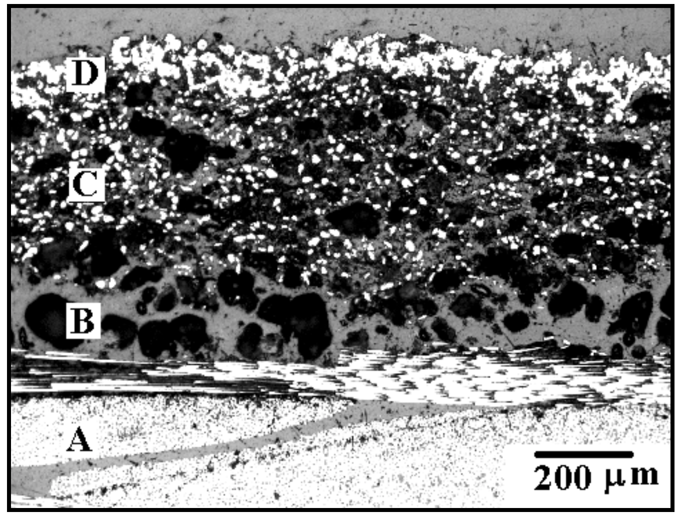

Figure 5: Three-layer FGM coating comprised of a pure WC-Co topcoat (D), a WC-Co/polyimide layer (C) and a pure polyimide layer (B), on a PMC substrate (A).

Neglecting voids, this indicated that the relative proportions of the polyimide matrix and WC-Co filler were 58% and 42%, respectively.

Results of the tensile adhesion tests are shown in Fig. 6. Coatings similar to those shown in Figs. 4 and 5 both exhibited lower pull-off strengths (5–6.2 MPa) than the pure polyimide coatings (~8.4 MPa).The tensile strength of the PMC substrate was determined using the same pull-off test to establish a reference value for the substrate adhesive/cohesive characteristics. Uncoated substrates failed due to delamination in the direction perpendicular to thickness at a tensile strength ~17.6 MPa. The locus of failure of the samples tested was characterized according to the location and percent of adhesive and cohesive failures, together with SEM characterization of the interfaces and coating layers.

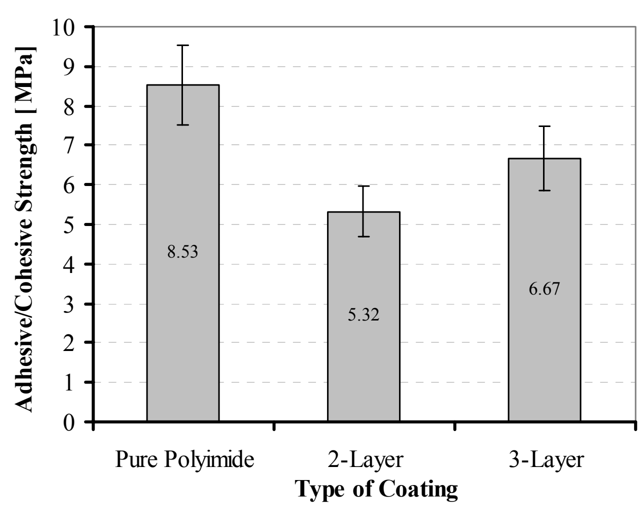

Figure 6: Results of the pull-off tensile adhesion tests for three types of coating (ASTM D-4541).

Most of the samples failed due to delamination within the substrate (Fig. 7). These results were surprising given that the tensile strength of the reference (uncoated) substrate (~17.6 MPa) was much higher than the tensile strength of any of the coatings tested. This may have been due to the damage caused by the grit blasting used to roughen the substrate surfaces prior to spraying. The uncoated substrates tested were not grit blasted prior to testing.

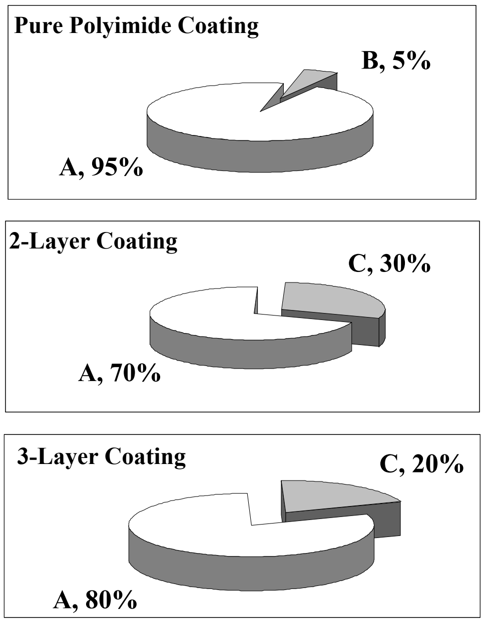

Figure 7: Estimated percentages and locations of adhesive and cohesive failures. Pure polyimide coating – failure mainly within the substrate (A); two- and three-layer coatings – failure within both the substrate (A) and composite layers (C).

Grit blasting damaged the surface of the substrate, likely reducing its tensile strength, by breaking carbon fibers, as shown in Fig. 8.

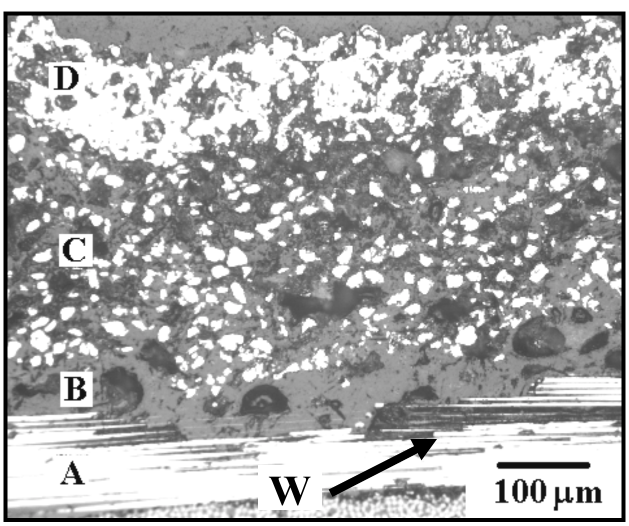

Figure 8: Three-layer composite coating showing the substrate surface fiber damage [W] caused by grit blasting.

SEM micrographs showing the failed surface of a pure polyimide coating are shown in Figs. 9 and 10. The mode of fracture in the thermosetting polyimide was brittle, associated with the formation of cracks in regions between the gas voids where localized stress concentrations likely occurred. At a higher magnification of a region between gas voids (Fig. 11), parallel flat plateaus were observed, indicating brittle crack propagation along multiple parallel planes.

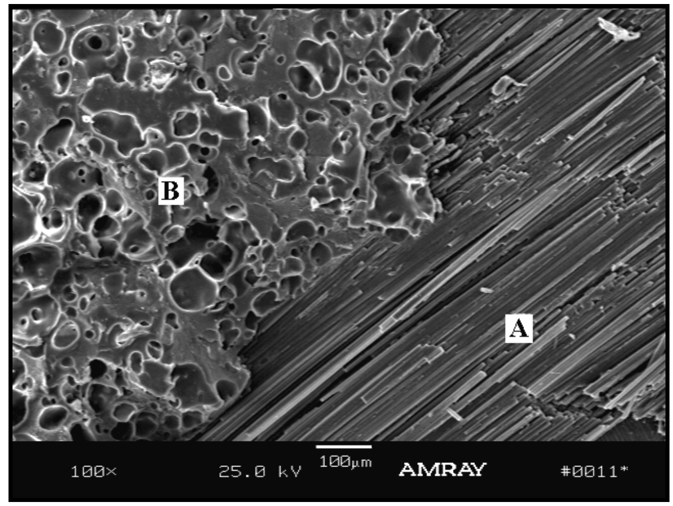

Figure 9: Fracture surface of a failed pure polyimide coating. Cohesive failure within the polyimide coating (B) combined with cohesive failure in the substrate (A).

The two- and three-layer coatings both failed by the same mechanism, a combination of the cohesive failure within the composite layer (region C) and cohesive failure within the substrate (region A), as shown in Fig. 7.

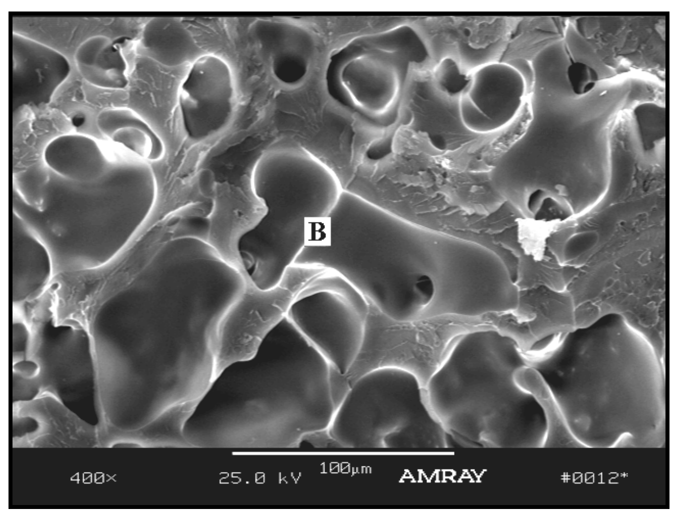

Figure 10: Detail of the fracture surface of a pure polyimide coating (B).

SEM micrographs showing the failed surfaces of tested two- and three-layer coatings are shown in Figs. 12, 13 and 14.

Cracks appeared to have propagated partially along the interfaces between the WC-Co filler and the polyimide matrix, as shown at the left side of Fig. 14. This may have contributed to the lower pull-off strengths measured for the two- and three-layer coatings relative to the pure polyimide coating.

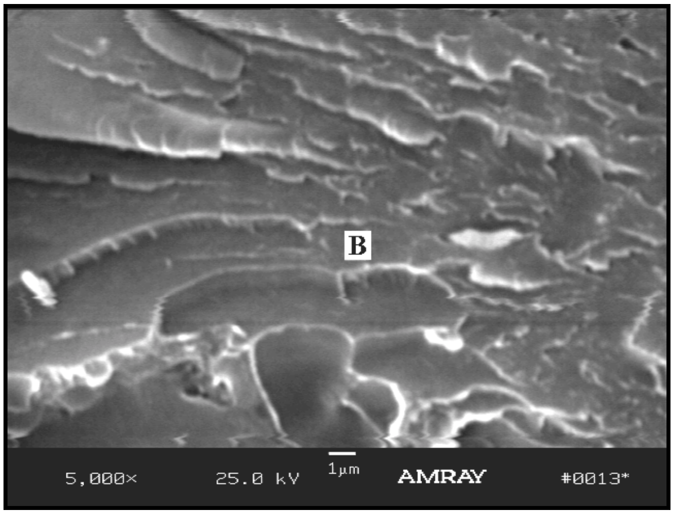

Figure 11: High magnification (500X) image of the inter-pore region of a tested pure polyimide coating. The parallel plateau and "river" patterns indicated brittle crack propagation along multiple parallel planes in the polyimide coating (B).

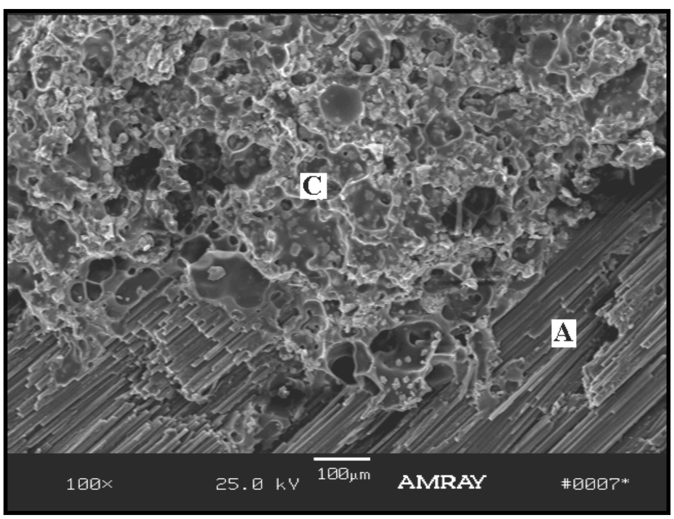

Figure 12: Typical image of the fractured surface of two- and three-layer coatings. Cohesive failure within the composite layer (C) was combined with delamination within the substrate (A).

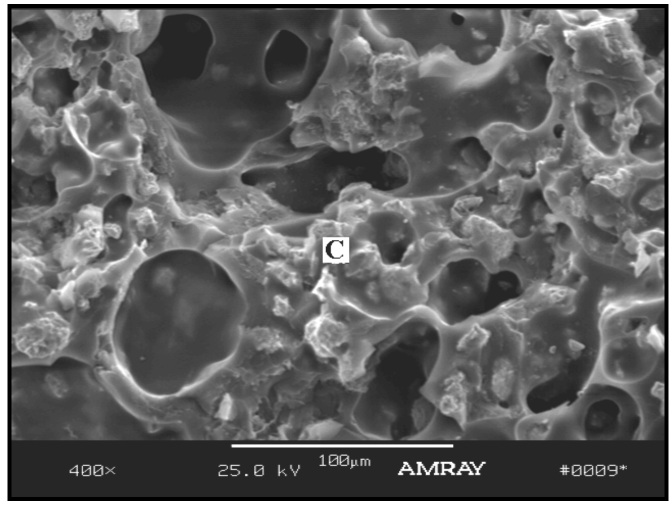

Figure 13: Fracture surface of a two-layer coating within the WC-Co/polyimide composite layer (C).

Most of the tensile failures within the WC-Co/polyimide layer (region C) occurred due to crack propagation between WC-Co particles (Fig. 14) which was believed to be due to low elongation prior to yielding and the high modulus of rigidity of the thermosetting polyimide matrix.

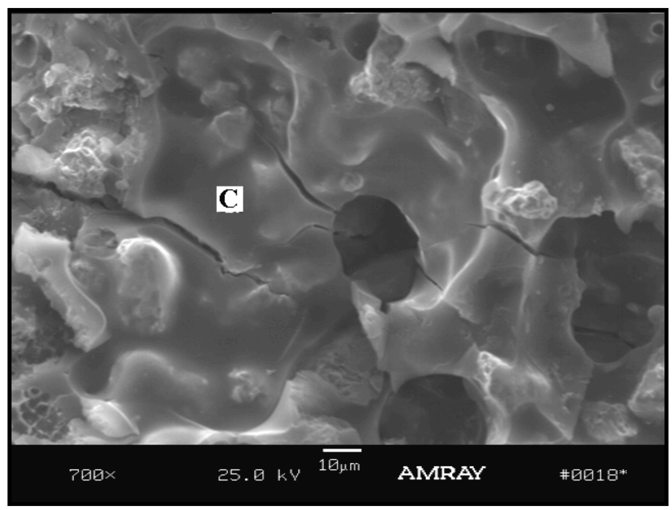

Figure 14:Brittle cracks propagating from a pore within the composite layer (C) of a three-layer coating.

Summary and Conclusions

The feasibility of depositing FGM coatings consisting of layers of pure polyimide, polyimide + WC-Co and pure WC-Co has been demonstrated using a combination of internal and external feeding of the two feedstock materials. Pure polyimide and polyimide + WC-Co were sprayed by the HVOF process and the WC-Co topcoat was flame sprayed.

The porosity and volume fraction of the WC-Co filler retained in the sprayed coatings were determined by standard metallographic techniques and image analysis. The relative proportions (on a pore-free basis) of polyimide matrix and WC-Co filler were 58% and 42%, respectively. The porosity of the pure polyimide coating was determined to be ~26%, likely due to gas evolution during crosslinking above 280 °C.

The tensile adhesion behavior of three types of coatings sprayed onto carbon-fiber reinforced PMC substrates has been evaluated using a standard pull-off tensile adhesion test (ASTM D 4541). The adhesive/cohesive strengths of the different coating types were measured and compared to that of an uncoated substrate. The two- and three-layer composite coatings both exibited lower pull-off strengths (5–6.2 MPa) than pure polyimide coatings (~8.4 MPa), and in all cases these values were lower than the tensile strength (~17.6 MPa), of the uncoated PMC substrate used as a "reference" for assessing coating adhesion/cohesion. The nature and locus of the failed surfaces following tensile testing were characterized according to the percent adhesive and cohesive failures. The majority of the failures were due to delamination within the substrate, which occurred at lower strengths than those exhibited by uncoated substrates. The difference was likely due to substrate damage caused during grit blasting prior to coating.

The mode of failure in the thermosetting polyimide was brittle fracture associated with the formation of cracks at inter-pore regions where localized stress concentrations would occur. The two- and three-layer FGM coatings both failed by the same mechanism - a combination of cohesive failure in the composite layer and delamination within the substrate.

Additional work is being carried out, including continued spray parameter development and optimization, assessment of the repeatability of the results and detailed characterization of the coatings, including evaluation of the thermo-mechanical fatigue properties of the coating/substrate system, and their erosion resistance. Polyimide chemistry optimization will also be investigated in order to reduce gas evolution during crosslinking reactions.

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M. Ivosevic, R. Knight, S.R. Kalidindi, G.R. Palmese, and J.K. Sutter

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National Aeronautics and Space Administration
John H. Glenn Research Center at Lewis Field
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13. ABSTRACT ***(Maximum 200 words)***

The use of polymer matrix composites (PMC's) in the gas flow path of advanced turbine engines offers significant benefits for aircraft engine performance but their useful lifetime is limited by their poor erosion resistance. High velocity oxy-fuel (HVOF) sprayed polymer/cermet functionally graded (FGM) coatings are being investigated as a method to address this technology gap by providing erosion and oxidation protection to polymer matrix composites. The FGM coating structures are based on a polyimide matrix filled with varying volume fractions of WC-Co. The graded coating architecture was produced using a combination of internal and external feedstock injection, via two computer-controlled powder feeders and controlled substrate preheating. Porosity, coating thickness and volume fraction of the WC-Co filler retained in the coatings were determined using standard metallographic techniques and computer image analysis. The pull-off strength (often referred to as the adhesive strength) of the coatings was evaluated according to the ASTM D 4541 standard test method, which measured the greatest normal tensile force that the coating could withstand. Adhesive/cohesive strengths were determined for three different types of coating structures and compared based on the maximum indicated load and the surface area loaded. The nature and locus of the fractures were characterized according to the percent of adhesive and/or cohesive failure, and the tested interfaces and layers involved were analyzed by Scanning Electron Microscopy.

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