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Metallic Coating for Carbon Fiber Reinforced Polymer Matrix Composite Substrate

Amine Rezzoug, Said Abdi, Nadjet Bouhelal, Ismail Daoud

***Abstract*—**This paper investigates the application of metallic coatings on high fiber volume fraction carbon/epoxy polymer matrix composites. For the grip of the metallic layer, a method of modifying the surface of the composite by introducing a mixture of copper and steel powder (filler powders) which can reduce the impact of thermal spray particles. The powder was introduced to the surface at the time of the forming. Arc spray was used to project the zinc coating layer. The substrate was grit blasted to avoid poor adherence.

The porosity, microstructure, and morphology of layers are characterized by optical microscopy, SEM and image analysis. The samples were studied also in terms of hardness and erosion resistance. This investigation did not reveal any visible evidence damage to the substrates. The hardness of zinc layer was about 25.94 MPa and the porosity was around (~6.70%). The erosion test showed that the zinc coating improves the resistance to erosion. Based on the results obtained, we can conclude that thermal spraying allows the production of protective coating on PMC. Zinc coating has been identified as a compatible material with the substrate. The filler powders layer protects the substrate from the impact of hot particles and allows avoiding the rupture of brittle carbon fibers.

***Keywords*—**Arc spray, coating, composite, erosion.

I. Introduction

POLYMER MATRIX COMPOSITES (PMCs) are very competitive materials for aerospace applications where high reliability is a fundamental requirement, due to their high strength to weight ratio compared to metals [1]. To increase the propulsion efficiency of the turbine engine, more and more metal parts are being replaced by PMCs all over the world. Initial applications were focused on structural and semi-structural airframe components [1]. Global advantage for the integration of PMCs into aircraft engines include: reduced weight and part counts, improved strength and reduced manufacturing cost; but PMCs are limited by their operational temperature and poor erosion resistance [2] and this makes it somewhat difficult for making more use of PMCs as a substitution of the metallic structures in aero-propulsion applications [3].

Turbine engines suffer from erosion by particles in the air. The erosion is serious because PMCs are generally softer than metals, and the speed of the particles is high [2], [3]. Interest has been expressed in using PMCs in engine parts for which erosion and wear from both hot gases and particles is a limiting factor [4].

As a surface modification technology, thermal spray has gained great success in the protection of metal parts. Several attempts have been made to use these technologies to produce protective coatings for PMCs [2]-[4]. A promising coating solution to alleviate wear and erosion problems with limited additional weight was investigated in this work.

Knowing that polymers have in common a low melting temperature, low surface tension, a slight roughness and a high chemical inertness (non-reactive) [5], provide an intimate and enduring contact between a coating and a substrate that have far different physic-chemical and mechanical characteristics is extremely difficult. Numerous parameters must be optimized. Once we get this condition checked, an appropriate level of adhesion between the substrate polymer and inorganic coating is established, assuring a high mechanical functionality for this multimaterial [5], [6].

The difference in properties between the coating and the substrate-based polymer can create stresses at the interface, leading to delamination and crack formation. In fact, when depositing a metal compound on a polymer substrate, two main problems must be solved: (a) the coating shall be deposited with minimal impact of heat and speed, such that the substrate is not degraded. (b) Ensuring that the adhesion of the final coating is reliable in service [7].

Limited work has been done in the surface protection of polymer composites using the thermal spray technology. Nevertheless, recently some investigations have described solutions to this erosion problem by the deposition of metal coatings. Erosion-resistant coatings are needed to protect the composite materials, at least through the first overhaul interval, and preferably over the full lifetime of the component [8].

M. Ivosevic et al. [2], [8], [9] investigated high velocity oxy-fuel (HVOF) sprayed functionally graded coatings for polymeric substrates and studied the microstructure and properties of these coatings. A 100% WC-Co outer layer should provide improved erosion and oxidation protection to the substrate. The key advantage of the coating is that the properties of each component can be utilized effectively and the need for compromise is reduced. For that, they designed a functionally graded coating structure based on a polyimide matrix filled with varying volume fractions of WC –Co. in order to solve the problem of thermal expansion coefficient mismatch between the substrate and the protection coating. They found that the commonly used pretreatment would cause substrate damage, and result in low bond strength. The addition of a zinc bond coat

Amine Rezzoug, Selsabil Rokia Laraba, Ismail Daoud and Said Abdi, are with the Laboratory of Materials Science and Engineering, Faculty of Mechanical Engineering and Process Engineering, Univ of Sci & Tech. (USTHB), Algiers, Algeria (e-mail: Rezzoug16@gmail.com).

was found to eliminate ablation of the graded part of the coating, which arose during the deposition of pure WC–Co. Furthermore, it was shown that the zinc bond coat played a role in enhancing the erosion resistance.

A. Liu et al. [3] cited concerns with polyimide degradation due to impinging hot particles in plasma spray. Carbon/polyimide samples were grit-blasted or sanded prior to coating. Damage to the carbon fibers resulting from grit-blasting was observed but the shear bond strengths reported were greater for these samples than for the sanded ones. Moreover, the mass loss of the coated PMC in erosion testing was half of that of the uncoated one. The top layer was deposited by arc spray, which proved to be a suitable technique to produce erosion-resistant coatings on PMCs. furthermore they concluded that, materials with high melting point were not suitable for direct coating on PMCs. In another study, R. Wang et all [10] investigated various bond coat materials and spray processes. It was observed that deposition of high melting point materials such as copper or nickel as bond coats using plasma spray led to low shear bond strengths as a result of polyimide degradation induced by the plasma jet. Although low melting metals such as zinc and aluminum appear as candidate materials [11].

F. Robitaille et al. [4] presented a new method for applying metallic coating on high fiber volume fraction continuous fiber carbon/epoxy PMCs using the pulsed gas dynamic spraying process. Copper particles were co-cured with the PMC laminate in a single operation. No surface preparation was required before spraying therefore no damage was induced in fibers. Low sprayed powder temperatures (zinc) were used to avoid degrading the metal powder or the epoxy. External substrate heating was not required. Cold spray is also used by R. Lupoi et al [12] for Deposition of metallic coatings on polymer surfaces, Results have demonstrated that materials travelling at deposition velocities, can leads to severe contact stresses, therefore the predominant effect is erosion of the polymer. Aluminum, due to its low specific weight, has better ability to be a coating on PMCs. Based on these results, we can conclude that thermal spray processes allow the production of metallic coatings on PMCs, zinc was identified as a suitable bond coat or coating for PMCs, filler particles layer protects the material from the impact of hot particles and the damage caused by surface preparation prior to spraying.

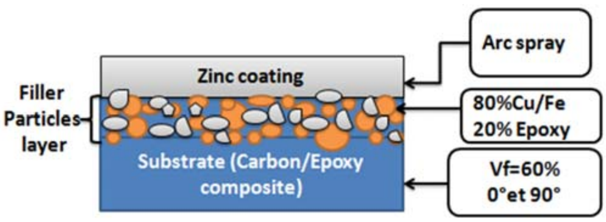

Fig. 1 Coating concepts studied in this work

The overall goal of this work was to demonstrate a method of modifying the surface of a composite to allow a metallic coating process onto PMCs (Fig. 1). Introduction of a mixture copper/ steel powder might reduce particle impact throughout thermal spraying. The substrate was a carbon/epoxy composite. The Cartelization of microstructure and mechanical properties of Zn coating deposited on PMCs was investigated.

II. Materials and Methods

A. Realization of the Substrate

The 2.6 mm thick PMC substrate consisted of an epoxy laminating system (EPOCAST® 50-A1 Resin) reinforced with a carbon-fiber plain weave TAFTAS (REV T TYPE D CLASSE 7). Laminate was produced with six layers of fabrics oriented at 0° and 90°. Fiber volume fraction (v_f) was about 60% and PMC substrate dimension was 250 × 300 × 2.6 mm. The density of Epoxy and Carbon fiber was 1.21 g/cm^3 and 1.8 g/cm^3 respectively. The PMC substrate was produced by vacuum molding and cured via a heating system at 60°C. It was supplied at the Algerian airline maintenance Center, Algiers. In order to realize the composite plate, we have implemented a number of equipment required (Fig. 2).

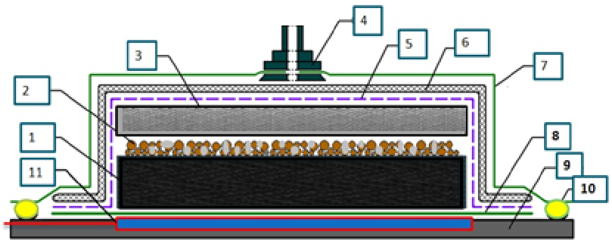

Fig. 2 Schematic of vacuum molding device for manufacturing PMC, 1: impregnated fabric, 2: filler powder, 3: homogenization thickness plate, 4 air pumping pipe 5: Perforated Film, 6: Bleeder, 7: vacuum bag, 8: separator film, 9: molding plate, 10 sealing tape, 11: heating System

B. Layer of Filler Powder

Even for the most thermally resistant polymer matrix composites, 372.15°C is the upper limit of their working temperature for long time service, and 530.8°C for short time service [5]. The sprayed particles of higher temperature can cause degradation of the mechanical properties of PMC, or even damage it. Therefore, from this point of view, we used a mixture of copper and stainless steel powder as filer layer for co-curing in PMC (Fig. 3). The copper powder Metco 55 (Sulzer) electrolytic type; is a copper powder (99% of purity) with an average particle sizes of 47 µm and a density of 8.92g/cm3. For the stainless steel alloy powder Metco 42C (Sulzer) the density was 8.02g/cm3, it was manufactured by gas atomization and the average particle size was also 47 µm. SEM images of the powder showed that the particles of stainless steel have an irregular shape with a sub-angular structure whereas the particles of copper have spherical morphology (Fig. 3).

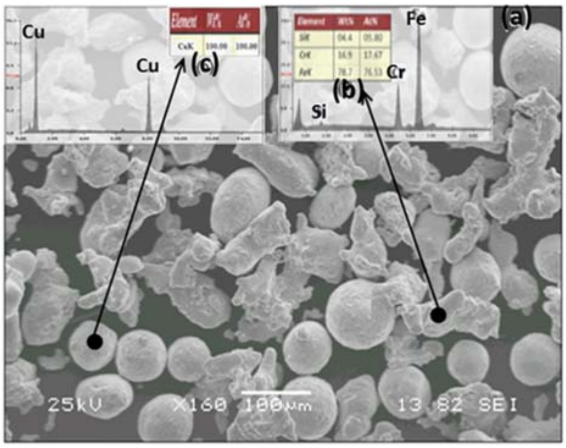

Fig. 3 SEM micrograph of the mixture (copper/ stainless steel) powder (a), chemical analysis by EDS of the stainless steel powder (b) and of copper powder (c)

TABLE I
ARC SPRAY PARAMETERS FOR ZINC COATING

Arc current (A)	80A
Arc voltage (V)	38V
Torch to substrate distance (mm)	130-140 mm
Pressure of compressed air (MPa)	0.37 MPa
Projection angle	90°

TABLE II
THE TEST CONDITIONS BASED ON ASTM G76−13

Nozzle	Ceramic Conical tube [2.4 mm ID*50 mm L]
Erodent	Angular Al2O3 [nominal particle size 250 µm]
Compressed air pressure	0.13MPa
Impingement angle	90°
Spray distance	10 mm
Test time	20 s
Test temperature	25 °C

Before curing, the laminates were manually covered with a layer of powder on the upper face. The excess was removed by tilting the plate of the molding progressively and then by a vacuum. After that, composites were bagged, particles were lightly pressed, and the excess resin bled out of the fabric and through the particles to be encapsulated on the top face. These particles may promote coating adhesion and avoid brittle PMC failure [4].

C. Zinc Layer

Pure zinc was used as the coating material. It was chosen based on its low melting point (420°C) which should not cause degradation of the substrate and good wettability on nonmetal surface. A less intense spray method, arc spray, seems more suitable for spraying coatings on composites. It is a low cost coating method with high efficiency, compared with plasma spray and HVOF, the temperature and the velocity of the sprayed particles are lower too [5]. The process uses metal in wire form. molten metallic droplets is created by the impingement of a fast moving gas upon the continuously melting tips of consumable wires fed into an arc formed between the wires. The molten metal on the wire tips is atomized and propelled onto a prepared substrate by a stream of compressed air or other gas [13]. In order to have a good adherence between the sprayed layer and substrate, we have respected the projection parameters summarized in Table I. The metal coating was carried out at Metallization Algeria Company. Moreover, the PMC substrate was cleaned and then grit blasted prior to spraying. The substrates was cleaned with acetone to remove grease or moisture and vacuum dried, after that they were lightly grit blasted using a metallic abrasive powders based on cast iron. This preparation is in order to create an anchor tooth profile at the surface.

D.Analysis Techniques

Porosity percentage, coating thickness were determined by optical microscopy (Nikon) at 400× magnification using standard metallographic techniques. The specimens were sectioned and polished to a 0.1 µm surface finish. The computer image analysis was performed using ImageJ software. Ten measurements were taken per sample at various positions in the coating. Their roughness was evaluated with a piezo-electric transducer roughnessmeter (TR100). Average surface roughness (*Ra*) values were averaged out of five measurements.

Microstructural characterization was performed at cross-sections of the sample obtained by resin cold mounting. Morphological characteristics were examined by a JEOL JSM 6360 and LEICA S430 scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS).

The Vicker's microhardness (Hv0.1) of the different layers was measured according to the ASTM standard E92-82 [14] using a SHIMADZU Vicker's microhardness instrument (manufactured by Shimadzu Co). The test was performed with a pyramidal diamond indenter with an edge angle of 115°, and a load of 100 g for 10 s on polished sections, several tests were performed and the mean value was given. It is known that the depth of the impression of the indenter must be less than 1/5±1/10 of the thickness of the layer in the indentation method in order to avoid the effect of the substrate [15].

Solid particle erosion testing of coated, co-cured and uncoated polymer matrix was carried out using a grit blasting gun at room temperature. Testing was performed by a PRESSURE SAND BLASTER (JONNESWAY, model TA2A0257), the nozzle diameter was 2.4 mm. The angle of impingement was about 90°. The solid particle for erosion was a highly abrasive erodent Corundum based on pure Al_2O_3 (COBRA) of 250 µm (Fig. 4). The pressure of the compressed air was 0.13 MPa and the Spray distance was about 10 mm. The erosion weight loss was determined using an analytical balance KERN ABS-N. The samples were cleaned and weighed after the erosion testing. Weighing of the mass of the specimen before and after erosion to provide the erosion mass loss. The test conditions (Table II) were based on ASTM G76−13 ''Standard Test Method for Conducting Erosion Test by Solid Particle Impingement Using Gas Jets" [16]**.** Three replicate test specimens of 25x25 mm were taken from the laminates using a cutting water jet.

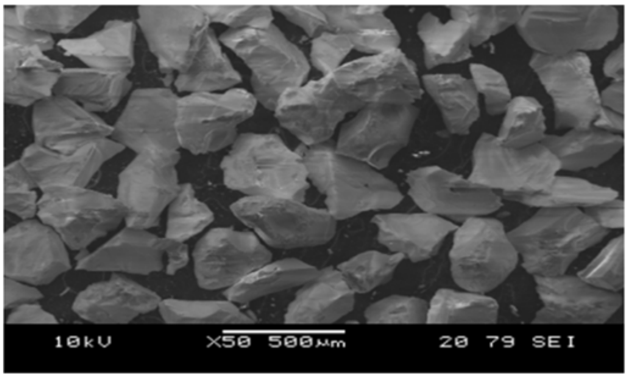

Fig. 4 SEM micrograph of Al_2O_3 impacting particles

III. Results and Discussion

The quality of all laminates was assessed by ultrasonic scanning prior to the test; there was no internal crack which means that the substrate had no major defect. We note that all attempts to spray zinc coatings directly onto (carbon/epoxy) PMC under arc spray conditions failed because of the high temperature flux involved in the spraying, leading to substrate degradation. In addition, the speed of sanding particle causes the rupture of brittle carbon fiber.

Several studies had cited this limit of PMC [3], [11]. However, filler particles layers into the laminate of carbon/epoxy PMC has resulted in successful coating of metal by arc spraying without causing any obvious damage to the fibers or the matrix (Fig. 5).

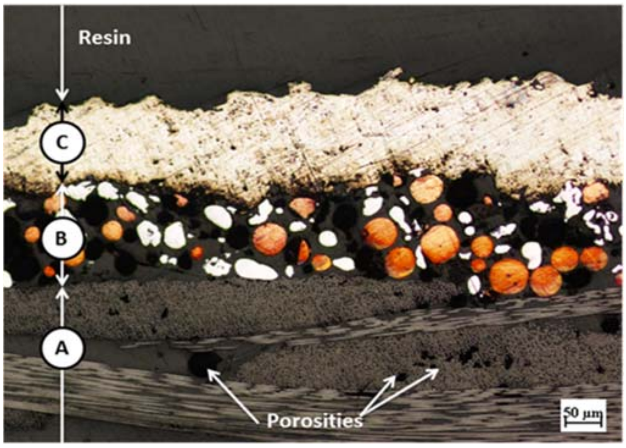

Fig. 5 Microstructure of cross-section of carbon-epoxy CMP with powder layer and zinc coating layer, (A): carbon-epoxy composite, (B): powder layer and (C): zinc coating layer

Fig. 5 shows a cross-section of the coating system; the powder is encapsulated by epoxy which bleeds out of PMC. The excess epoxy resin forms a matrix for the powder layer. The adhesion provided by the epoxy prevents powder from moving under the composites. Based on image analysis the layer encapsulated powder has a thickness ranging between 100 µm and 250 µm. The thickness is affected by the Tafta forms of carbon fabric which is wavy, also by the powder morphology and size. Dark spheres appear in the powder layer; these zones are result of particles powder pull out during sample polishing. Furthermore, the powder pull out can be explained by a weak powder-epoxy adhesion, even the powder size was close to that of abrasive paper. We note that metallographic preparation of samples for examination is rather difficult. This is due to coating system made of different materials, also because the powder layer.

Fig. 6 shows an overall view of the top side of powder layer on carbon-epoxy PMC. We notice a homogeneous distribution of powder; although there are few rich areas with stainless steel powder and poor of copper powder and vice versa.

Fig. 6 Photomicrograph of the top side of the Carbon/epoxy PMC Substrate- with the powder layer

Coating thicknesses obtained were in the range 80-150 µm. From Fig. 5, we can conclude that particles provided coating adhesion with good coherence between layer-to-layer and coating to substrate.

The porosity percentage of the coating was determined by image analysis, according to ASTM E2109 Standard [17], [18]. A relatively low porosity was observed in the coatings according to metallographic picture Figs. 5 and 7. Image analysis of the Zinc coating shown in (Fig. 8) indicates that the porosity was about 6.70 %; this is due to insufficient kinetic energy of the arc spraying (Fig. 7). Many parameters of arc spray can be improved in order to decrease porosity, or choose more kinetic energy spraying. However, the porosity percentage was acceptable for this kind of coating [19].

Fig. 7 shows that coating exhibits a very irregular surface. Consolidation of particles inside the coating is affected by spray parameters and processes. Coatings are generally characterized by shape of drops flattened and porosity nears the interface between the droplets.

The consolidation may result of particle impact of subsequent spray passes (Fig. 7). Considering the adhesion performance, the porosity and surface roughness of the coating may have some advantages if an upper coating is needed and the layer of Zinc will be bond-coat layer between substrate and a top layer [8].

Surface roughness was estimate by the arithmetic average roughness; so Ra was chosen as the roughness parameter. The Ra consists of 5 measurements for each surface. Ra of the substrate before being grit blasted was 2.10±1.54 μm. After grit blasting Ra was improved to reach an average of 5.08±0.99 μm; creating a rougher interfacial area topographically increases the real contact surface coating / substrate (develops greater area). Grit blasting creates a surface that allows the anchoring of the coating. The average roughness was about 10.94±3.50 μm for the arc sprayed coating surface. A relatively high level of rough surface finish is confirmed by the SEM morphology (Fig. 7). The surfaces are rougher than the original grit-blasted surface which is especially the case for low speed sprayed coatings. Indeed, the microhardness $HV_{0.1}$ shows that the surface hardness is greatly affected by the powder. The surface hardness increases from 3.53±0.03 $HV_{0.1}$ (Carbon/epoxy) to 69.85 ± 3.88 $HV_{0.1}$ (copper powder) and 397.33 ± 33.70 $HV_{0.1}$ (stainless steel powder). Microhardness of the surface of the zinc coating is about 25.94 ± 2.72 $HV_{0.1}$. Microhardness of coating was higher than that of carbon/epoxy composite but less than the both powders which were harder. Powders layer gives a good compromise between hardness and ductility, allowing the deformation of the projected droplets; also protecting the substrate at the time of grit blasting and the projection coating. It is noted that our zinc coating was softer than zinc coating sprayed by pulsed gas dynamic spraying process [4]. In this case the velocity of the sprayed particles has an important role to play in improving hardness.

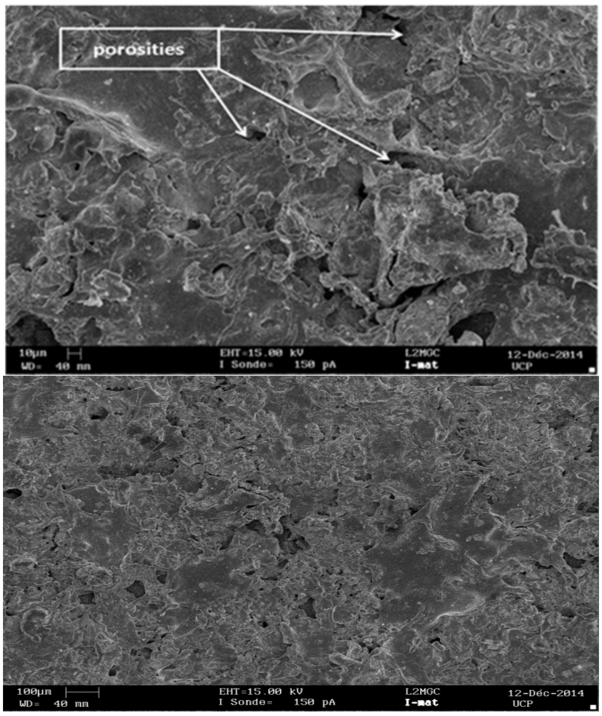

Fig. 7 SEM micrograph of surface morphology of the arc sprayed zinc at different magnifications of the same sample

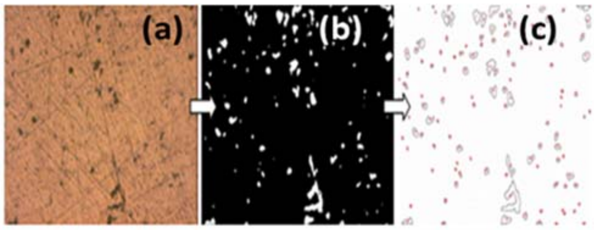

Fig. 8 steps of the image analysis to determine the porosity by Image-J (a: microstructures of Zinc layer, b: binary image and c: outline image X100)

Fig. 9 shows the erosion rate of coated, filler and uncoated samples after 20s of erosion. The wear rate is calculated as

$$\text{wear rate} = \frac{(M_b - M_a)}{M_b} \quad (1)$$

where the M_b was the mass of the sample before erosion testing and M_a was the mass of the sample after erosion testing. The filler layer does not protect against erosion; the epoxy resin facilitates the removing of powder which is explained by a significant mass loss (erosion rate was 649.31x10^{-4}g/g). Poor cohesion between the powder and the resin accelerates the erosion phenomenon. Actually, the powder layer is a key step for metallic coating but it is not recommended for erosion resistance. Indeed, the PMC without filler of powder layer shows better erosion resistance (erosion rate was 229.33x10^{-4} g/g). In other hand, zinc coatings had clearly enhanced erosion resistance of the PMC (erosion rate was 6.55x10^{-4} g/g).

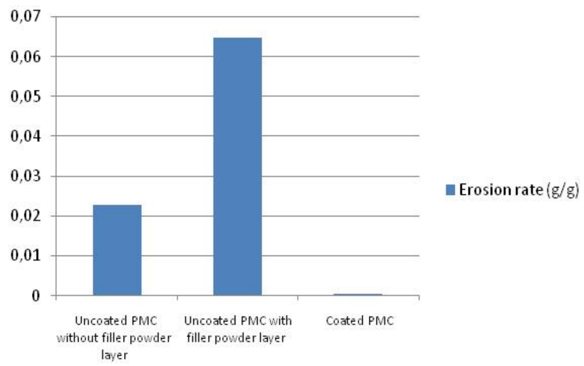

Fig. 9 Erosion rate comparison between coated, filler and uncoated samples after 20s of erosion

The morphology appearances of an uncoated substrate and the coated composite, after erosion testing for 20 s at 90° impingement angle are shown in Fig. 10. The eroded area of composite substrate with filler layer is more damaged than the coated one. It was found that metallic coating can provide protection for polymer matrix composite. However, the density of the PMC is different from filler powder layer and zinc top layer. From this point of view, calculating the erosion rate by volume loss is more suitable.

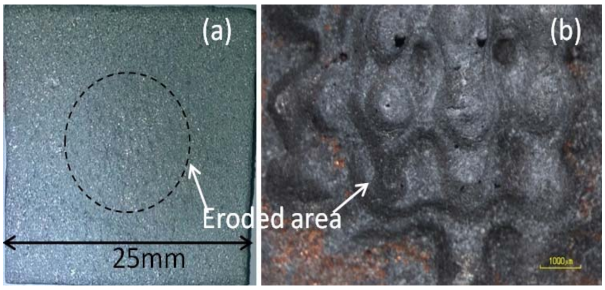

Fig. 10 The surface macrographs of zinc coating (a) and carbon/ epoxy PMC Substrate with the powder layer (b) after erosion testing for 20s at 90°

IV. Conclusion

This paper presents an experimental process to protect polymer matrix composite (PMC) by metallic coating. The metallic layer was obtained by arc spray. To promote the feasibility of coating, a layer of filler powder was added to the (PMC). According to the results of this study, we can conclude that the PMC can be coated by thermal spraying without degrading the material. Thermal spray coating is a viable technology for the polymer composite surface protection. Coating can be achieved by introducing barrier such as filler powder layer. Thanks to intrinsic property, Zinc was identified as a suitable metallic coating for (PMC). The porosity of the coating is in the average of similar coating achieved by arc spray. Microhardness of coating on an average has been increased as well as the erosion resistance.

For an eventual future work, some points might be taken into consideration; we can start by understanding the spray-substrate interactions, a range of parameters need to be developed to study their effect on the coating. Also, a detailed study of powder size distribution could be conducted to achieve more uniform layer. Moreover, post heat treatment methods could be developed for coatings. Furthermore, the effect of the thickness of the different layer must be studied. Filler powder layer on PMCs also open the possibility of spraying other types of materials for other applications such as heat and electrical conductivity or tribological properties.

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