

# Cold Spray of metallic coatings on polymer based composites for the lightning strike protection of airplane structures

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## Abstract

Unlike their metal counterparts, composite structures do not readily conduct away the electrical currents generated by lightning strikes. Cost reduction and expected production growth of the next middle range airplanes require automated manufacturing process of polymer components. The development of an automated technology to metallize polymer based composite for lightning strike protection is the aim of the CO3 project (EU Grant agreement: ID831979). In this study, thermal and electrical conductivities of composites were achieved by cold spray deposition of Cu or Al coatings. Critical points to be addressed were substrate erosion during cold spray, lack of polymer-metal adhesion and poor deposition efficiency. Several strategies were tested: i) a thin polymer film was cured at the substrate surface before cold spraying, to enable implantation of metallic particles in the film, helping coating build-up and protecting the fibers of the composite. ii) Cold spraying a mix of metal and polymer powders to improve coating adhesion and prevent fiber damage. iii) Supercritical Nitrogen Deposition technology, prior to cold spray, to mechanically anchor metallic particles into the polymer. Subsequent cold spraying of purely metallic coatings was more efficient and showed better adhesion. All coatings were tested in terms of adhesion strength and electrical conductivity.

*Table 1. List of acronyms*

CFRC	Carbon Fiber Reinforced Composites
HPCS	High Pressure Cold Spray
LPCS	Low Pressure Cold Spray
PEEK	PolyEther – Ether - Ketone
PEI	PolyEtherImide
PPF	Protective Polymer Film
SCND	SuperCritical Nitrogen Deposition
SEM	Scanning Electron Microscopy

## Introduction

Polymers and Carbon Fiber Reinforced Composites (CFRCs) have a great potential application in aircraft structures for light weighting purposes and its resistance to corrosion. As a drawback, these materials exhibit vulnerabilities to lightning strikes due to their limited electrical conductivity, which can result in serious physical damage effects such as erosion, ablation or entire explosion of the structure [1, 2]. Airplane skin is divided in several zones, classified depending on the potential strength of the lightning strike received, which is a function of the geometry and of the position of the part within the structure. Parts located near the extremities and having high curvature are exposed to the most intense strikes (zones 1A). The intensity decreases as the code increase (1A, 1B, 1C, 2A, etc.) [3, 4]. To get flying certification, parts have to withstand specific lightning strike tests in laboratory, where the effect of a true strike is experimentally simulated by dedicated systems. The severity of parameters of the test (tension/current waves applied to the part) depends on the abovementioned code for the zone of exposure. Actually, the most widespread solution for lightning strike protection is the application of metallic meshes, available in different materials (typically Al, Cu or bronze) and with different mass per unit surface to adapt to the different zones. In many parts of the airplane, metallic meshes are still manually laid up. Since composite manufacturing is following the road to full automation, especially for cost reduction, the metallization should follow as well. The development of an automated technology to metallize polymer based composite for lightning strike protection is the aim of the CO3 project, funded by Clean Sky II (EU Grant agreement: ID831979). Metallized CFRCs will withstand standard lightning tests corresponding to the most severe case of zone 1A. At the end of the project, a real-scale demonstrator of a wing tip will be produced and coated. CO3 stands for Cold sprayed COating on COmposites, so given the name, the process chosen is quite obvious. Warmer spraying technologies,

such as plasma, flame, electric arc spray for example, although highly efficient, do not work properly with temperature sensitive powders and substrate materials [5, 6]. Cold Spray is proposed as an alternative technique, mainly because of the lower process temperatures and its ability to obtain dense and non-oxidised metallic coatings, two important factors for electrical conductivity. The challenge of applying a metallic coating onto CFRCs by cold spray relies in finding suitable powders and spraying conditions to prevent fibre damage and erosion of the substrate material, as already shown by numerous researches. To give a quick literature overview of these studies, in 2006 Sturgeon et al. [7] cold sprayed aluminum powder on a short carbon fiber reinforced PEEK. Adherent and dense coatings could be achieved using helium as a spraying gas. However, it must be stressed that the composite made with short carbon fibers differs significantly with respect to continuous fiber materials which are mostly used in airplane manufacturing. In a similar study Zhou et al. [8], still using short carbon fibers/PEEK composite, a high pressure cold spray system was used to spray aluminum and copper. For the successful bonding of copper particles, an aluminum bond coat was used. In the same context, Malachowska et al. in 2018 [9] achieved the metallization of PA6 and PC with copper particles, via a tin interlayer. Another way to produce cold spray metal coatings on polymers and composites was based on deposition of few polymer particle layers that fused with the substrate [10, 11]. In parallel, several works [6, 12] recommended to cold spray mixed Al-Sn powders with a lower melting point material, at 300°C for various pressures (0.4 to 1 MPa). The work showed the difficulty to achieve dense and adherent coatings onto both carbon fibers and epoxy based materials. The use of mixture powders of metal and polymer powders was shown to be an interesting way to limit fiber failure and to produce adherent and electrically conductive coatings [13].

In the present study, different strategies were tested, alone and in combination between them, to achieve the goal of CFRC metallization: Protective Polymer Film (PPF), SuperCritical Nitrogen Deposition (SCND), Low Pressure Cold Spray (LPCS) and High Pressure Cold Spray (HPCS). The different solutions are on the way of optimization and are being tested in terms of adhesion and electrical conductivity.

## Materials and methods

Aluminum and copper powders were used as feedstock materials for the surface metallization of CFRCs by cold spray and SCND. In some LPCS tests they were mixed with PEEK powders to produce metal-polymer composite coatings. The powders used are listed in Table 2. SEM images of powders used in the present study are shown in Fig. 1 a, b and c.

Two types of CFRCs were used as substrate materials: thermoset based and thermoplastic based. The latter were commercial PEEK based panels with continuous carbon fibres. The literature review presented above already showed that

cold spraying is easier on thermoplastic based materials than on thermosets. Thermoset based CFRCs were produced by LIST by injection moulding, using the bi-component epoxy based HexFlow® RTM6-2 as resin and a carbon fabric from Hexcel as reinforcement (Type of yarns: Warp, HexTow AS4C GP 3K; Weft : HexTow AS4C GP 3K; nominal weight 200 g/m<sup>2</sup>, weave style TWILL 2/2, 6 plies [0°]). During substrate manufacturing, a PPF could be added on top of the carbon fabric, as shown in Fig. 1d. Temperature cycling and curing monitoring curves are shown in Fig. 1e.

The SCND technique consists in the addition of copper powder particles to a Super Critical Nitrogen jet at very high pressure, flowing out of a specific nozzle. Particles are then accelerated at high velocities and sprayed to the substrate surface at low temperatures. This process can be used as a new and colder spraying technique or as an innovative surface preparation, before cold spraying the conductive coating. The process parameters chosen were the following. Nitrogen pressure: 300 MPa; standoff distance: 300 mm; transverse speeds in the range from 5 to 10 m.min<sup>-1</sup>; step (distance between spraying lines) in the range from 5 to 10 mm.

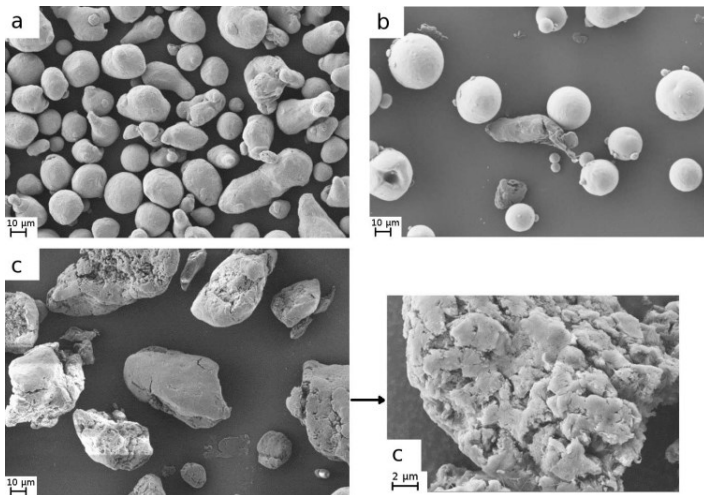
In cold spray, the heated and pressurized gas accelerates powder particles to be sprayed through a converging/diverging “de Laval” nozzle, reaching supersonic speeds during its expansion. When particles impact on the substrate, they can adhere, forming a coating by a building-up process. The kinetic energy of the particles, rather than high temperature, allow particles to plastically deform and bond together to produce coatings. Due to the relatively low temperatures, cold spray avoids or minimizes many deleterious shortcomings of traditional thermal spray methods such as high-temperature oxidation, evaporation, melting, crystallization, residual stresses [14]. Cold spray is a solid-state coating deposition technology which is nowadays industrially in use for the application of functional coatings, in the repair/refurbishment of components and, more recently, as an additive manufacturing process to fabricate individual components. Two types of equipment can be found today on the market: low pressure systems (limited to 0.8-1.5 MPa, depending on the model) and more performing high pressure ones (reaching up to 6 MPa and 1100°C). First HPCS experiments were carried out by using a CGT Kinetiks 4000/47 system (with pressure up to 4 MPa and temperature up to 850°C) with nitrogen gas. A PBI (Polymer) nozzle was used for spraying aluminum powder, while a 24TC (Tungsten Carbide) nozzle for spraying copper. The gun was mounted on a Reis robotic system RVL 26. Later, for copper large panel coatings, an Impact Innovations (Rattenkirchen, Germany) 5/11 system, reaching up to 6 MPa and 1100°C mounted on a Kuka robot, was used. The LPCS system used was a DYCOMET model 523 (Akkrum, The Netherlands) operating with compressed air, with pressure up to 0.6 MPa and temperature up to 600°C. LPCS system differs from the high pressure one for a different powder injection point, located downstream to the nozzle throat, as well as for the range of pressure-temperature couples that can be used.

Table 2: Feedstock powders.

Metals	Size
Al (99.9% high purity) from Toyal	20-50 $\mu$ m
Cu (99.95% high purity) from Safina	15-38 $\mu$ m
Polymer	Size
PEEK Vicote702 from Vitrex	26-88 $\mu$ m

Table 3: List of PPF applied to substrates.

PPF material	Thickness	Functionalization process
PEEK amorphous APTIV® 2000 Victrex®	300 $\mu$ m	Film co-curing with/without plasma treatment
PEEK amorphous APTIV® 2000 Victrex®	50 $\mu$ m	Film co-curing with/without plasma treatment
PEEK amorphous APTIV® 2000 Victrex®	25 $\mu$ m	Film co-curing with plasma treatment
PEEK semi-crystalline APTIV® 2000 Victrex®	25 $\mu$ m	Film co-curing with plasma treatment
PEI	25 $\mu$ m	Film co-curing with plasma treatment
PEI	12 $\mu$ m	Spray coating



d

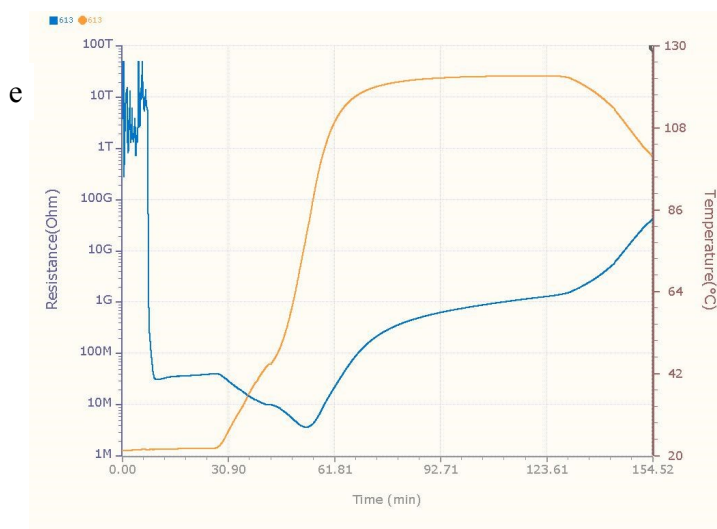


Figure 1: top left, SEM top view images of powders: (a) Al, (b) Cu, (c) Cu mixed with 20%vol. PEEK, (c) PEEK Vicote702. (d): image of PPF application on carbon fabrics before resin injection. (e) PPF temperature/cure monitoring.

Three main physical coating properties were identified as the most relevant for the final application: superficial mass, adhesion and electrical conductivity. All these are finally related to coating microstructures that were characterized by scanning electron microscopy (ZEISS Sigma 300). Cross-section images were used for quantitative phase analysis by image analysis open source routines implemented in Python (Simple Morphological Image Library, <http://smil.cmm.mines-paristech.fr>). The tensile bond strength of the coatings was tested according to ASTM C633-1 norm “Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings”, using an Instron tensile testing machine. This test consists in quantitatively measuring the resistance of the coating-substrate assemblies applying a growing pull-off force until rupture. Cylindrical standard samples with a diameter of 25 mm were manufactured, consisting in steel cylinders to which the coated CFRC was glued, using the DUOPOX®AD895 glue by DELO (Germany). The tensile stress, applied perpendicularly to the plane of the coating, was uniformly increased at a rate of 0.5 MPa.s<sup>-1</sup>. The measured force at rupture, together with the known sample area, allowed estimating the bond strength of the coatings.

The Van der Pauw method [15] was used for the measurement of electrical surface conductivity. To assure a good quality of the measure, the coating must be continuous, with uniform thickness (largely smaller than surface size). Conductivity measurement, then, is easier if the sample is symmetrical. The best shape is a clover-like one, as shown in Fig. 2, because it forces the current to flow through the center of the sample, assuring that the length of the path travelled by electrons is

approximately the same between all the four points. The contact points used for these measurements should lie on the edge of the sample and must be very small compared to the sample size. The measurement method is illustrated in Fig. 2, on the left. A certain current  $i$  is applied between two points on the same edge and the potential  $V$  is measured between the opposite points. Then, the same procedure is applied three times, after turning the sample by 90° each time. Four values are obtained. The resistivity  $\rho$  of the coating was computed using the following formula:

$$\rho = \frac{\pi d R_{eq}}{\ln(2)} f\left(\frac{R_{12,34}}{R_{23,41}}\right)$$

where:  $d$  is coating thickness,  $R_{eq}$  the mean resistance measured between the 4 couples of points,  $f\left(\frac{R_{12,34}}{R_{23,41}}\right)$  a shape factor, directly linked to the sample geometry. The shape factor can be computed by the following relation:

$$\cosh\left(\frac{R_{12,34} - R_{13,24}}{R_{12,34} + R_{13,24}}\right) \cdot \frac{\ln(2)}{f} = \frac{1}{2} \exp\left(\frac{\ln(2)}{f}\right)$$

If  $R_{12,34} = R_{23,41}$  then  $f$  is equal to 1. Measurements were realized with a dedicated set up, made by a measuring chamber “HFS600E-PB4” from Linkam, with four measuring tips (Fig. 2, on the right), coupled to a Keithley unit allowing current generation and voltage measurement. The maximal current was 1 A.

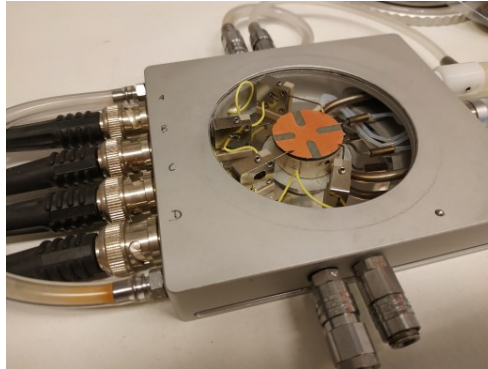
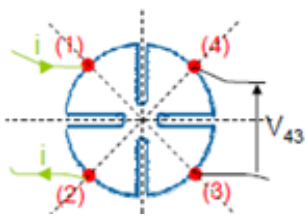


Figure 2: on the left, schematic view of the Van der Pauw method; at the center, picture of one of the tested samples; on the right, picture of the measuring set up.

## Results and discussion

Results of the different strategies will be presented in the following order: SCND (with and without PPF), HPCS (with PPF), LPCS (with and without PPF). Other strategy combinations are still ongoing, such as PPF+SCND+HPCS or PPF+SCND+LPCS.

The first trials of SCND onto thermoset based CFRCs were done with Cu powder and varying process parameters. No Cu deposition could be obtained, as can be seen in SEM top-view observations in Fig. 3A. Only few particles could stick to the

composite, while in many areas the surface of the material was damaged. Broken fibers and damaged polymer matrix are visible. Copper particles impacted at high speed and, due to their low temperatures, they were rigid enough to erode the substrate. In fact, epoxy based CFRCs are known to have fragile behavior and, thus, are less resistant to impacting particles than thermoplastic based materials. These first trials evidenced the detrimental effects of particles with high kinetic energy and low deformability, the latter due to the low temperatures of the process.

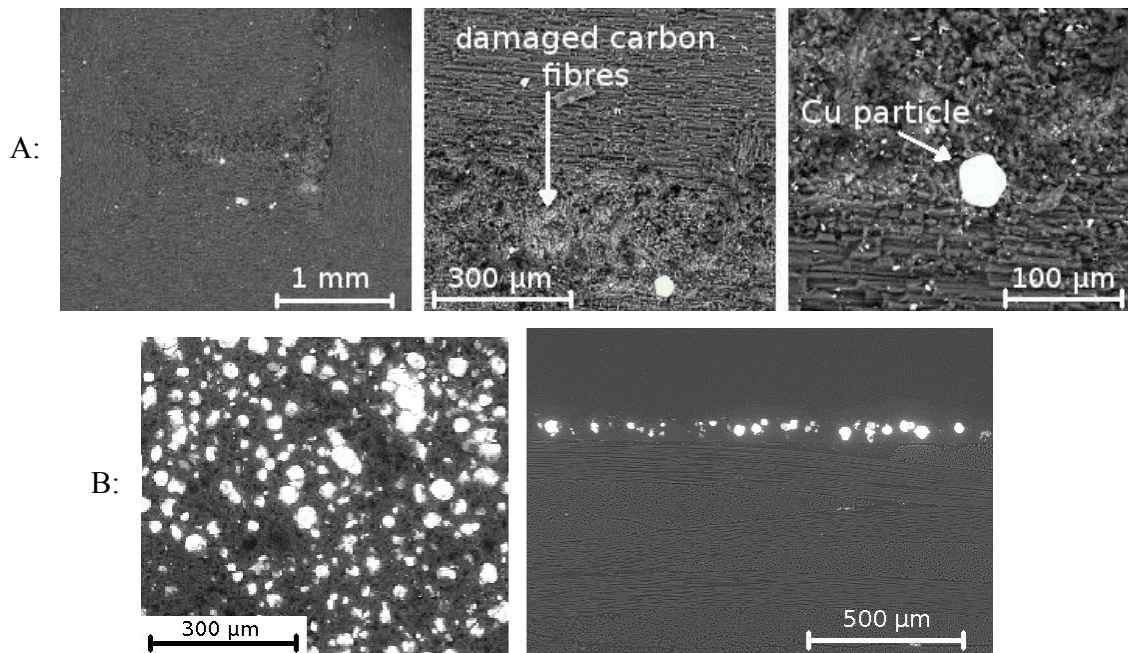


Figure 3: A: SEM top views of thermoset based CFRC after SCND (magnification increases from left to right). B: SEM images of CFRC sample with PPF after SCND of copper particles (1 pass, 10 m.min<sup>-1</sup>); on the left, top view; on the right, cross-section.

Better results were obtained thanks to a PPF. A PEEK film was applied on CFRC surface before copper SCND. In this configuration, it was possible to deposit a larger number of particles per unit surface and to preserve carbon fibers, as shown in Fig. 3B. On the left, a SEM top view illustrates the density of Cu particles while, on the right, a SEM cross section shows that Cu particles penetrated into PEEK PPF without reaching underlying carbon fibers. PEEK ductility, in fact, allowed Cu particle penetration. Its plastic deformation absorbed the high kinetic energy of impinging Cu particles and avoided their rebounding. At the same time, it effectively protected the fragile thermoset based material. Nevertheless, despite a considerable density of copper particles could be achieved, these coatings were not good electrical conductors. In fact, the measured electrical resistivity was in the order of  $10^8 \Omega \cdot \text{cm}$ , to be compared with a reference value on copper tape of  $2.9 \cdot 10^{-6} \Omega \cdot \text{cm}$ . This is certainly due to the fact that SCND coatings were not continuous and homogeneous. During the process, impinging particles could hardly form a sufficient bonding to already deposited metallic particles and, thus, rebounded away. Electrical conductivity suffered from the lack of a continuous path into the metallic phase for electric conduction.

HPCS tests were conducted using copper and aluminum feed-stock powders, on substrates with PPF. In fact, preliminary tests on bare substrates resulted in erosion and carbon fiber damage. Copper coatings were cold sprayed at high pressure onto thermoset based CFRCs with 25, 50 and 300  $\mu\text{m}$  PEEK PPF. A large range of spraying parameters was tested (temperature between 400 and 500°C, pressure between 0.8 and 1.4 MPa, standoff distance within 30-110 mm, transverse speed in the range 200-400 mm.s<sup>-1</sup>). Fig 4 shows the cosmetics

of some coatings performed on different PEEK PPF thicknesses under different HPCS spraying conditions.



Figure 4: HPCS copper coatings obtained with different spraying conditions. Substrates, made of thermoset CFRC, had, in each column, the same PEEK PPF thickness (300  $\mu\text{m}$  on the left, 50  $\mu\text{m}$  on the middle and 25  $\mu\text{m}$  on the right).

Cu particles possessed high kinetic energy and could penetrate deeply into the PEEK PPF, whose final thickness resulted largely decreased. The extended interface developed suggests a good anchoring of the coating to the substrate, to be verified by future tensile strength measurements. Coated samples showed different colors with the variation of spraying parameters, from pale pink to red-brown, passing by orange (Fig. 5).



Figure 5: photographic image of Cu HPCS onto thermoset CFRC with PPF, with different spraying parameters (temperature, pressure and stand-off distance).

This can be due to variations of coating oxidation state due to the exposure at different temperatures, depending on spraying parameters. Higher gas temperatures and shorter stand-off distances resulted in higher surface temperatures and, thus, in higher coating oxidation (darker brown). By using a simple multi-meter/ohm-meter, darker coatings resulted electrically non-conductive. Fig. 6A shows the effect of PPF thickness on coating thickness. The process window is very sensitive to PPF thickness in its lower range, between 25 and 50  $\mu\text{m}$ , while beyond 50  $\mu\text{m}$  no noticeable improvement could be observed. A thicker PPF facilitates coating deposition and homogeneity. Fig 6B shows pull-off test results for different PPF thicknesses. Again, 50  $\mu\text{m}$  PPF layers offer bonding strengths close to those of 300  $\mu\text{m}$  PPF. Fig. 6C shows electrical conductivity measurements obtained by the Van der Pauw method. Coating mean electrical conductivity was 40% of annealed copper ( $6.1 \cdot 10^5 \Omega^{-1}\text{cm}^{-1}$ ). This is probably due to the high density of defects induced by work hardening during particle deformation at impact. Increasing PPF layer thickness beyond 50 micrometers had a detrimental effect on coating electrical conductivity. This can be due to a lower mechanical resistance of the skin, resulting in a bumping effect at particle impact and in a less cohesive coating.

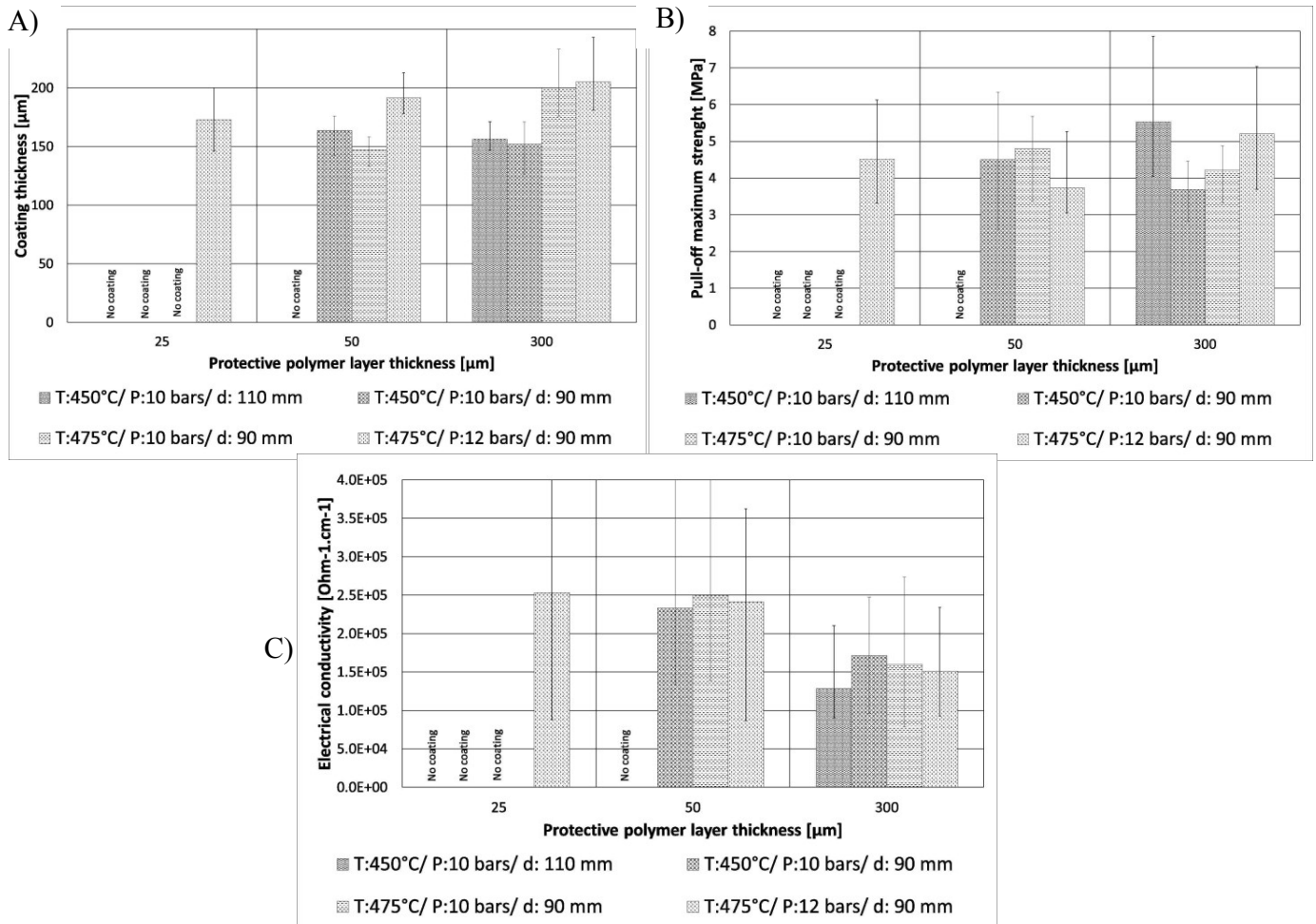


Figure 6: effect of PPF thickness on HPCS copper coating properties: A) on mean coating thicknesses; B) on mean pull-off strength; C) on electrical conductivity.

HPCS of aluminum powders was performed on thermoset based CFRC substrates with a 50  $\mu\text{m}$  PEEK PPF. Spraying temperature varied between 350 and 380°C and the pressure between 1 and 1.5 MPa. The standoff distance was fixed at 30 mm and the transverse speed in the range 100-250  $\text{mm}\cdot\text{s}^{-1}$ . The most promising process parameter combinations resulted in a 100  $\mu\text{m}$  thick and homogeneous coating, as showed in Fig. 7, on the left. A notable difference with the copper coating was that Al particles did not penetrate in the PPF. This was probably due to Al lower mass density, resulting in lower kinetic

energies of impacting particles, and to its lower yield stress, concentrating more plastic deformation in particles, when compared with Cu. Further tests focused on different PPFs, made by PEI polymer with a thickness between 8 and 12  $\mu\text{m}$ . Observation of PEI PPF revealed the presence of film cracks in some areas. Coating adhesion seemed much poorer in cracked areas, whereas a more homogenous coating could be produced on intact zones.

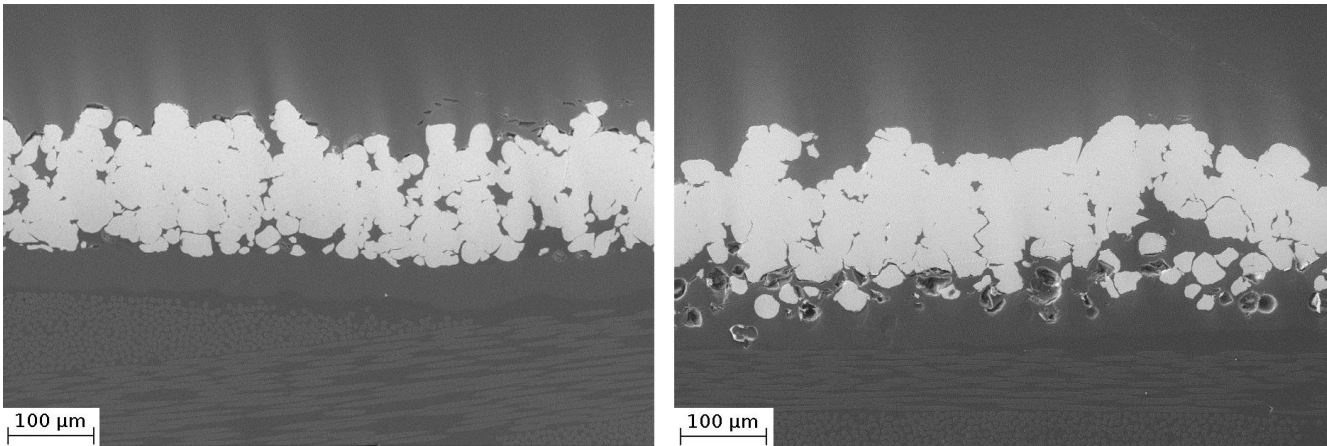


Figure 7: SEM cross sections of HPCS coatings on thermoset based CFRC substrates with PPF; on the left aluminum coating, on the right copper.

LPCS represents an economically interesting alternative to HPCS, when it comes to spraying materials with moderate mechanical properties, as Al in the case of this study. Moreover, in the case of fragile and thermosensitive substrates LPCS presents another advantage, as shown in other works [16]. When compared to HPCS, new gas process parameter ranges are accessible, in particular in the low pressure domain, with relatively high temperatures. In this conditions, particles can impact at lower velocities with higher temperatures, when compared to HPCS. The combination of “softer” and “slower” particle impacts can help limiting substrate erosion phenomena. The first LPCS tests onto PEEK based CFRCs, with aluminum and copper as feedstock powders, were not fully successful. In fact, exposed carbon fibers were damaged by impacting particles. The application of a PEEK PPF showed to be a viable solution to protect the fibers and made possible the LPCS of both thermoplastic and thermoset based CFRCs. Nevertheless, those coatings were more porous and with a seemingly reduced anchoring in the PPF than those obtained by HPCS, resulting in poorer electrical conductivity and adhesion. In successive LPCS experiments, an irregularly shaped PEEK powder (Vicote702) was mixed with Cu or with Al powders to produce metal-polymer composite coatings onto different thermoset and thermoplastic based CFRCs, with or without PPF. The influence of cold spray parameters (gas pressure, temperature and standoff distance) was investigated. Fig. 8, on the right, shows a SEM cross-sectional image of Cu-20%vol. PEEK onto a PEEK based CFRC with PPF. Copper

particles seem to be embedded into a polymer matrix (co-sprayed PEEK powder), which resulted to be an effective coating binder, promoting the adhesion and cohesion of the coating. Only low values of copper content in the coating could be achieved, estimated by image analysis. The highest was 50% vol. of Cu, in the coating shown in Fig. 8 on the right. Electrical conductivity is thus expected to be low. Fig. 8, on the left, shows a SEM cross-sectional image of Al-20%vol. PEEK composite coating. In this case, the metal content is higher and the electrical conductivity was measured to be  $1.3 \cdot 10^3 \Omega^{-1}\text{cm}^{-1}$ . Some other results with Al are shown in Table 4, on which further characterizations are still ongoing.

LPCS of composites metal-polymer coatings seemed more promising with Al powders than with Cu powders. This solution would be even more interesting for the final application if it would be possible to spray the composite on bare substrates, without the need of a PPF. Thus, some tests were done on thermoplastic based CFRCs with and without PEI PPF. In both cases, a thick Al-20% vol. PEEK composite coating was achieved by optimizing the process parameters, as shown in Fig. 9. The first characterizations are shown in Table 5, showing a remarkable value for electrical conductivity. The thin PEI PPF seemed to remain perfectly intact after the spraying. In the same way, the coating applied on the bare substrate did not erode the polymer matrix and did not damage any carbon fiber, confirming that LPCS of Al-PEEK mixed powder is well adapted to this kind of material.

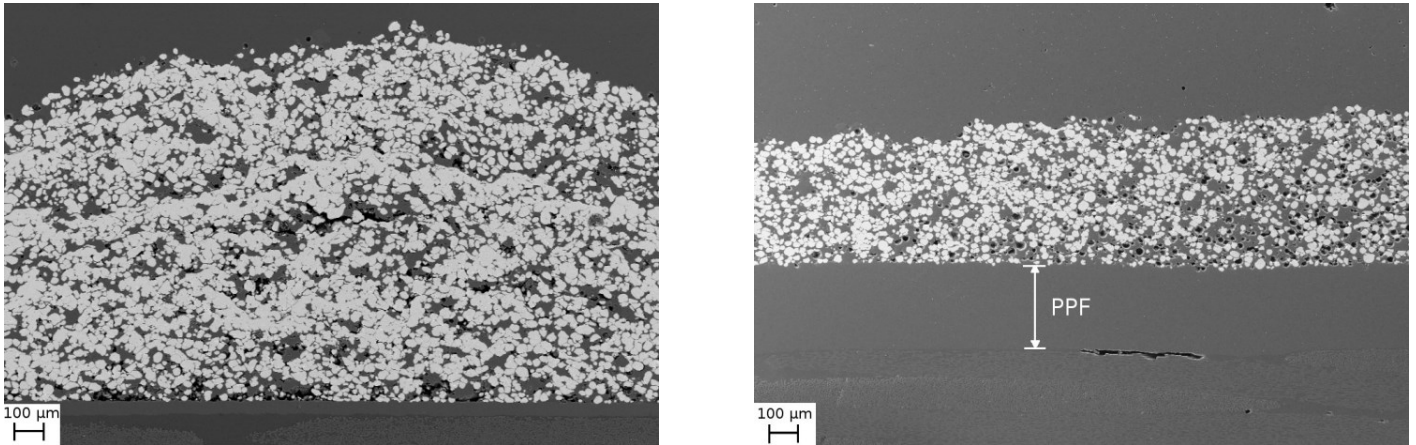


Figure 8: SEM cross sections of LPCS polymer-metal composite coatings on thermoplastic CFRC substrates with PEEK PPF. On the left, Al-PEEK 20%vol, on the right Cu-PEEK 20%vol.

Table 4: Properties of Al-20%vol.PEEK composite coatings on thermoplastic based CFRCs.

Conditions		Thickness [ $\mu\text{m}$ ]	%vol. of Al	Electrical conductivity [ $\Omega^{-1}\text{cm}^{-1}$ ]	Tensile bond strength [MPa]
SoD [mm]	T [ $^{\circ}\text{C}$ ]				
20	300	$1115 \pm 7$	77.6	-	-
	350	$1330 \pm 200$	71.6	$1.3 \cdot 10^4$	1
30	250	$327 \pm 19$	59.1	-	-
	300	$655 \pm 35$	56.5	-	-
40	250	$165 \pm 11$	40.4	-	-
	300	$404 \pm 19$	59.8	-	-

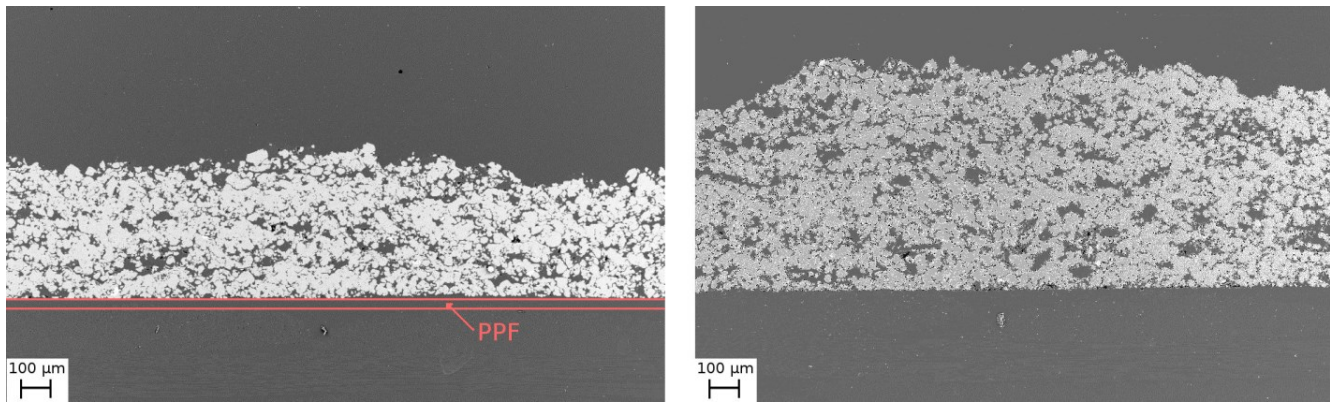


Figure 9: SEM cross sections of LPCS Al-PEEK 20%vol. coatings, on the left with PPF PEI, on the right without PPF.



Table 5. Properties of Al-20%vol.PEEK composite coatings on thermoplastic based CFRCs with and without PPF.

Conditions		Thickness [μm]	%vol. of Al	Electrical conductivity (Ω.cm)
SoD [mm], Temp [°C]	PPF			
20, 350	Yes	400±20	82.5	-
	No	750±50	78.2	1.3 10 <sup>5</sup>

## Conclusions

Various efforts have been made to metallize polymer-based composites via different strategies, with the purpose of obtaining an electrical conductive coating with good adhesion. Four technologies and some of their combinations were tested to metallize CFRC substrates: i) application of a Protective Polymer Film (PPF), ii) SuperCritical Nitrogen Deposition (SCND), iii) Low Pressure Cold Spray (LPCS), iv) High Pressure Cold Spray (HPCS). The PPF alone is non-conductive, so it must be employed in combination with the other technologies. A quantitative characterization of the film adhesion has yet to be performed. SCND, although not capable in these first experiments of producing a conductive metallic layer, showed a potential interest as an innovative technology for surface preparation before cold spraying, in particular after the application of a PPF. Cold spray technology viability for the metallization of thermoset and thermoplastic based CFRCs was demonstrated for both HPCS and LPCS. Copper coatings by HPCS after the application of PPF seemed the most promising solution for thermoset based CFRCs. In the case of thermoplastic based materials, LPCS of Al-PEEK mixed powders without any PPF was retained as the most interesting solution explored up to now. Other combinations of the used technologies will be tried in the next phases of the project, as well as a complete optimization of spraying materials and parameters.

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