COMPOSITE ROBUST BONDING – AUTOMATION OF SURFACE PREPARATION PROCESS FOR STRUCTURAL BONDING APPLICATIONS

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Abstract
Adhesive bonding is the preferred method for joining composite parts. The key step of this process is the surface treatment. Current state of the art in Aerospace Industry is based on the use of preimpregnated peel-plies or manual sanding/grit blasting operations. Both are manual methods, and thus, very operator dependent. A step forward to avoid manual operations and save recurring cost, is the development of automatic surface pretreatments. Today, two of the most promising alternatives for industrial purposes are: Vacuum Grit Blasting (VBG) and Atmospheric Pressure Plasma (APP). APP technology is mainly based on chemical modification of the surface by introduction of new polar functionalities. Vacuum Grit Blasting technology is based on mechanical modification of the surface using accelerated abrasive particles. The most sensitive parameters of every of these two technologies were evaluated to find the optimum process parameters for the surface treatment of the Carbon Fiber Reinforced Prepreg (CFRP) substrate studied in this work. Bonding performances were evaluated by means of Mode I Fracture toughness energy (G_{IC}). Surface characterization of the substrate, by means of SEM and XPS, has been used to, demonstrate no damage of the laminate, to observe creation of nano- or micro-roughness on surface topography, characterize changes in the chemical nature of the surface and to confirm elimination of contaminants. After a complete analysis, the main process parameters for the substrate analysed were identified. In the case of APP technology, nozzle speed of 100mm/s and distance to the substrate between 13-15 mm, resulted in an increase of polar groups (O, N) and elimination of contaminant (F). In the case of VGB technology, 100mm/s nozzle speed with a mass flow between 150-180g/min were the most suitable windows process parameters found. Although the main advance of Vacuum Grit Blasting technology, compared with the well-known Pressure Grit Blasting, is the used of vacuum system to clean the surface during the process, the study performed has demonstrated that a cleaning operation is mandatory after the treatment in order to remove completely the remains of abrasive particle on the surface.

1. Introduction

Aircraft bonded structures include multiple interfaces (e.g., composite-to-composite, composite-to-metal, or metal-to-metal), and these interfaces requires additional surface preparation prior to bond. Composite bonding structures required a qualified surface preparation of all previously cured substrates to activate the surface.
Current state of the art on Aerospace Industry is the extended used of preimpregnated (wet) peel-plies for co-bonding or secondary bonding processes. There are two general forms of peel ply: Dry and wet.

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Wet peel ply, based on a dry peel ply (dry fabric) impregnated with an epoxy polymer resin, is commonly the most used. This wet peel-ply is placed on the last layer of the CFRP laminate to be prepared for a subsequent bonding process. After curing, the peel ply is removed from the surface immediately before bonding.

Peel ply surface preparation is an attractive option from manufacturing standpoint because it reduces the human factors present in other surface preparation techniques, such as manual sanding or grit blasting. However, experience has demonstrated that this is also a sensitive process that depends on multiple variables (resins compatibility, fracture path between peel-ply and substrate, manufacturing process, storage, moisture…) indicating that there is not a fundamental understanding of all these variables affecting this process and which occasionally cause quality issues and “weak bonding” in the CFRP parts ([1]).

This investigation was aimed at finding alternative automated surface treatments for CFRP composite bonding in an industrial environment. Thus, the technology is required to prove robustness, reliability, homogeneity and reproducibility of the process in all cases. After a State of the Art study, two technologies were preselected based on different important criteria, such as maturity of the technology, industrial feasibility, applicability, homogeneity, possibility of re-treatments, health and safety concerns, and etcetera. The two preselected technologies were Atmospheric Pressure Plasma (APP) and Vacuum Grit Blasting (VGB).

Bonding performances were evaluated by means of Mode I Fracture toughness energy (G_{IC}). Surface characterization of the substrate, by means of SEM and XPS, has been used to demonstrate no damage of the laminate, to observe creation of nano- or micro-roughness on surface topography, characterize changes in the chemical nature of the surface and to confirm elimination of contaminants. A common test campaign was launched for both technologies to find the correct process parameters for the substrate analysed. Different ranges of the most critical parameters of each technology were studied (APP: Nozzle speed and nozzle distance; VGB: Nozzle speed and mass flow).

2. Technology Principles

2.1 Atmospheric Pressure Plasma (APP)

Plasma is an extremely reactive gas generated by applying energy to a gas or mixture of gases in order to activate the electronic structure of the species and produce neutrons, excited free electrons, ions, radicals… which impact the surface with energies sufficient to break the molecular bonds on the surface of most polymeric substrates. The energy source can be of thermal nature, electric current or electromagnetic radiations.

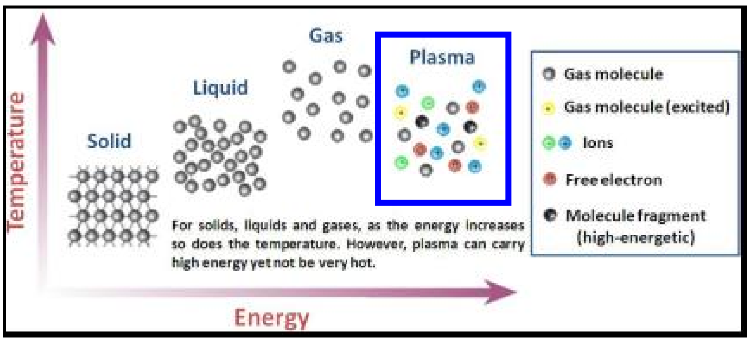

Figure 1. Plasma generation. Relationship between temperature and energy [5].

Atmospheric Pressure Plasma (APP) is environmentally friendly, in contrast with other surface treatment technologies like low-pressure or high-pressure plasmas, APP has can be directly applied in productions lines using only electricity and air. APP does not require the use of any vacuum chamber or any specific gas which simplifies the automation of this technology and mitigate the impact on Health & Safety for the operators.

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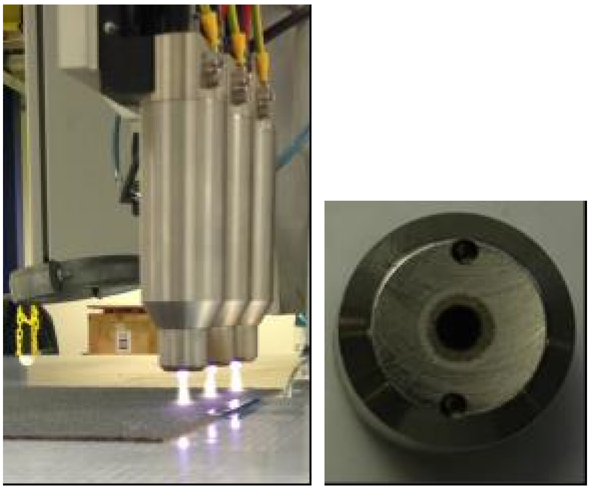
The effects of air-plasmas in the treatment of polymers are dominated by the presence of oxygen because it is extremely reactive to hydrocarbons. Surface reactions during and after plasma treatment lead to the predominant formation of different oxygen-containing functional groups like hydroxyls (-OH), carbonyls (=C=O) or carboxyl (-C(=O)O). APP treatment produce different effects on the substrate: surface cleaning and surface chemical modification (cross-links). This modification of the substrate will improve the surface wettability creating new polar functionalities that will create cross-links with the adhesive in the subsequent bonding process.
The equipment used for this study is based on atmospheric pressure plasma jets. The APP equipment used on this study is based on a power generator, a high voltage transformer box and three non-rotating plasma jets (PFW10) as seen in Figure 2. Different types of nozzle are available on the market. The selection of the type of nozzle was based on previous trials where this static nozzle was identified as the most intensive in terms of plasma generation.



(a) **(b)**

Figure 2. (a) APP system used on this study. (b) Detail of the static nozzle (PFW10)

2.2 Vacuum Grit Blasting (VGB)

Grit blasting technology is a surface treatment process widely used in a variety of different industries. The general mechanism of this treatment is based on accelerated abrasive particles that impact to the substrate (Figure 3). As most of the abrasive technologies the principle is to abrade the surface removing the contaminant and creating roughness to create mechanical interlocking between the substrate and the adhesive.

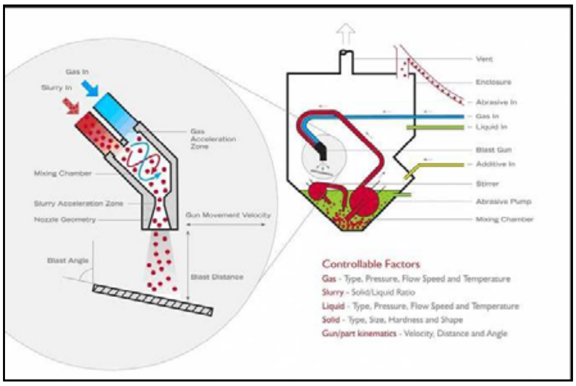


Figure 3. Scheme of Grit Blasting machine

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There are different types of abrasive media (metallic, mineral, synthetic...) with different hardness, shape and density, and for each type a wide range of particle size can be supplied. The selection of the correct abrasive media is a crucial and will depend on the requirements of the process and the substrate to be treated.
Most of blasting medias have two basic shapes: spherical or angular. Spherical media produce smoother surface finish whereas angular medias produce a rougher surface finish.
For this study, two types of abrasive media were considered: Glass beads and Urea Plastic (Jet Plast II –MIL-P-85891). Glass beads particles are spherical whereas Jet Plast particles are angular.
There are different systems to propel the abrasive media towards the substrate (air blasting, mechanical wheel, hydro blasting).
The main advantage of this technology compared with the traditional sand blasting processes, where the abrasive particle is propel toward the surface with the use of compressed air, is the use of a vacuum system to accelerate the abrasive particle and at the same time clean the surface during treatment by aspiration (See Figure 4). This technology minimizes waste and provides worker protection by continuously recycling of the blasting material and containing the dust during the treatment process [7]. The emission of particles can be also minimised, since the working zone is sealed off from the environment by a blasting hood. Type of nozzle is also essential selection on this technology. There are different types of nozzles. For this study a circular nozzle of 17mm with a rectandular blasting hoog of 20x10 mm was selected.

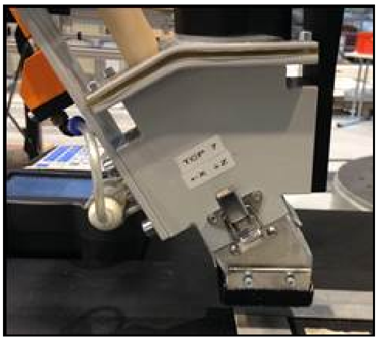

Figure 4. Vacuum Grit Blasting equipment

Blast media type, media acceleration and volume, combined with blasting distance from the substrate, angle of impact and number of cycles, are important factors in the blast process capabilities.

4. Test matrix

The materials selected on this study were typical epoxy resin systems 180ºC cure. Epoxy carbon fibre reinforced unidirectional tape prepreg (UD) was used to manufacture precured and cobonded semipanels conforming GIC specimens. For bonding, a typical epoxy resin adhesive film was also used (Carrier Knit).
To evaluate bonding performance, mode I fracture toughness energy (GIC) test at -55ºC/AR(As received) was selected as the most reliable method used today. Airbus Test Method AITM1.0053 was used to perform the test and manufacture the specimens. Failure modes were also identified following the categorization shown in Figure 5.

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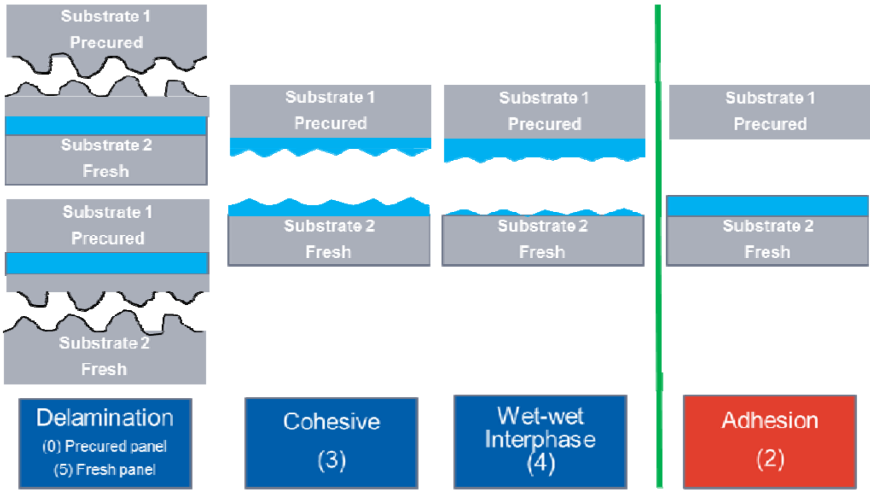

Figure 5. Identification of failure modes

Typical contamination on the parts is caused by the release materials used during the curing cycle.
For this study, the precured panels were manufactured following the manufacturing process of a representative part were this new technology can be employed. The vacuum bag configuration was selected using Frekote700NC (**source of silicon contamination by transfer)** as liquid release agent on the tool side and Wrightlon-5200 (ETFE) (**source of fluorine contamination by transfer)** as release film on the bag side. Based on the selected part configuration, precured panels were treated directly by bag side.
Two different test matrices were performed to find out the optimal process parameters of each technology for the CFRP substrate. Tables 1 and 2 show the summary of the selected parameters studied.
Cleaning operations were also considered during this study. For APP technology the panels were cleaned with isopropyl alcohol (IPA) before the treatment to remove excess of ancillary materials. For VGB, relaying on the cleaning provided by the vacuum system, extra cleaning operation were not included in the trials.

Table 1. APP test matrix

Specimen Type	*Nozzle Speed (mm/s)*	*Nozzle Distance (mm)*
APP 1		10
APP 2		13
APP 3	100	14
APP 4		15
APP 5		17
APP 6		10
APP 7		13
APP 8	150	14
APP 9		15
APP 10		17

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Table 2. VGB test matrix

Specimen Type	*Abrasive particle*	*Overlap (mm)*	*Incidence angle (º)*	*Nozzle Speed (mm/s)*	*Mass Flow (g/min)*
VGB 1	Glass Bead	8	60	100	120
VGB 2					150
VGB 3					180
VGB 4					210
VGB 5	Jet Plast Type II				380
VGB 6					410
VGB 7					450
VGB 8					480

3. Results

3.1 Atmospheric Pressure Plasma results (APP)

The surface of some of the samples APP pre-treated was analyzed by X-Ray Photoelectron Spectroscopy (XPS). Figure 6 shows the increased amount of polar groups (oxygen and nitrogen content) Additionally, no traces of fluorine were detected, indicating that APP treatment eliminates the fluorine content from the release film used to manufacture the panels.

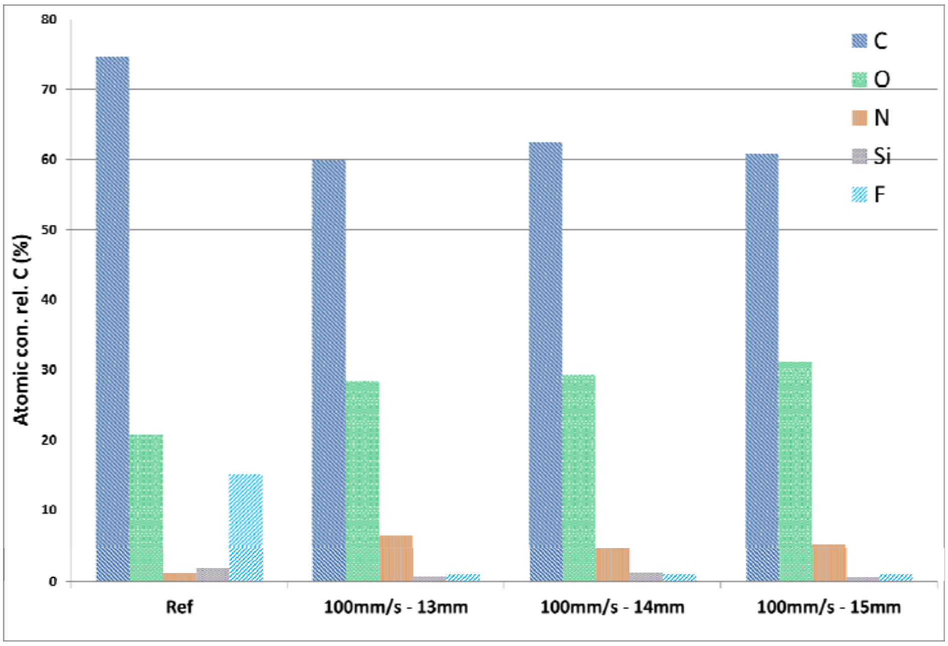

Figure 6. XPS analysis on APP samples

Figure 7 summarises the mechanical results obtained from the GIC tests performed at -55ºC/AR (As Received) used to analyse the bonding performances of CFRP coupons surface treated by APP.

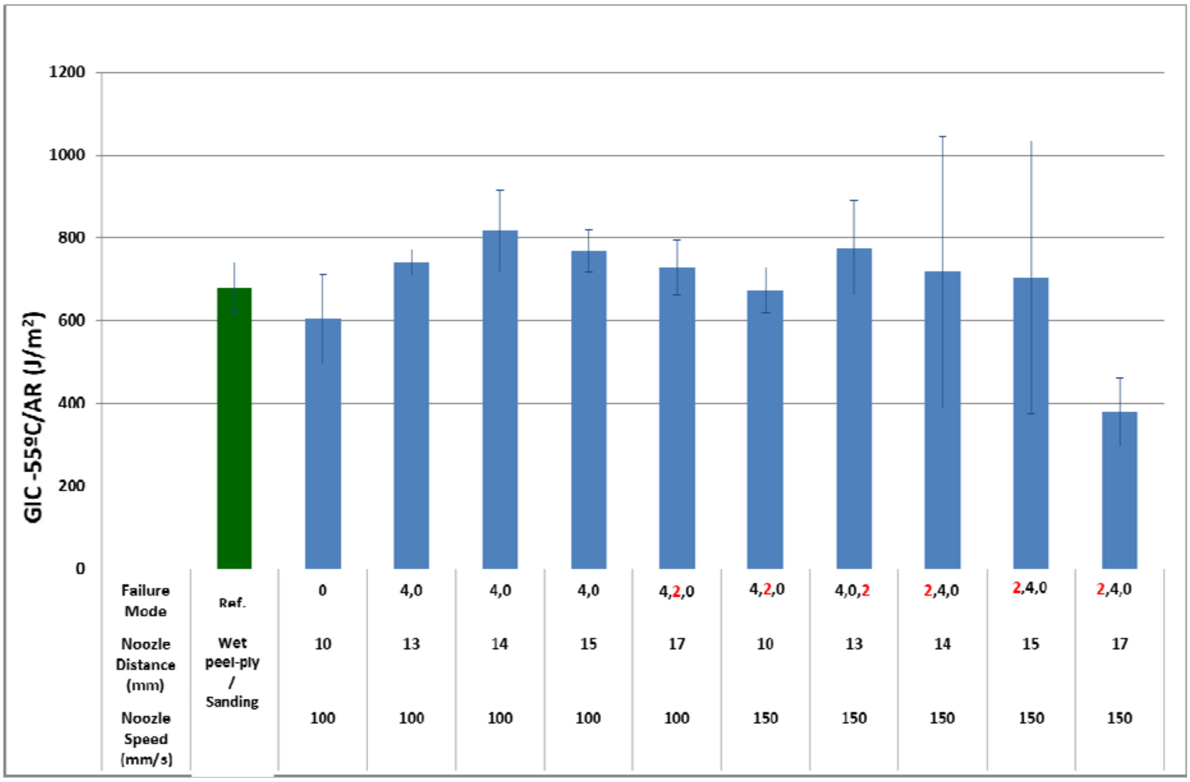

Figure 7. APP test results

As it can be seen, all panels treated with nozzle speed of 150mm/s present certain percentage of adhesion failure mode (2) in all the distances evaluated. The high scatter observed on the results is due to the high variance on failure modes observed on the specimens. Due to this lack of treatment homogeneity, 150mm/s speed was excluded from the process parameter window.
Panels treated at 100mm/s shows, in general, better results than the panels treated at 150mm/s. For 10mm of distance, the results obtained show damage on the first CFRP ply as seen on the delamination failure mode (0) observed in these specimens. Between distances to the substrate of 13-15mm, all the panels showed acceptable GIC values and failure modes (4,wet-wet interphase) and with the same standard values of fracture toughness energies than those observed in this material combination when the reference surface treatment (wet peel-ply or manual sanding) is employed. For 17mm of distance APP treatment is not effective since adhesion failure (2) appears in the GIC specimens.
Based on this observations, nozzle speed of 100 mm/s in the distance nozzle-substrate of 13-15 mm are considered as the APP process window parameters for the material combination (CFRP substrate + film adhesive) evaluated.

3.2 Vacuum Grit Blasting (VGB)

Figure 8 summarises the mechanical results obtained from the test matrix carried out for VGB technology. As mentioned, GIC test at -55ºC/AR were used to analyse bonding performances.

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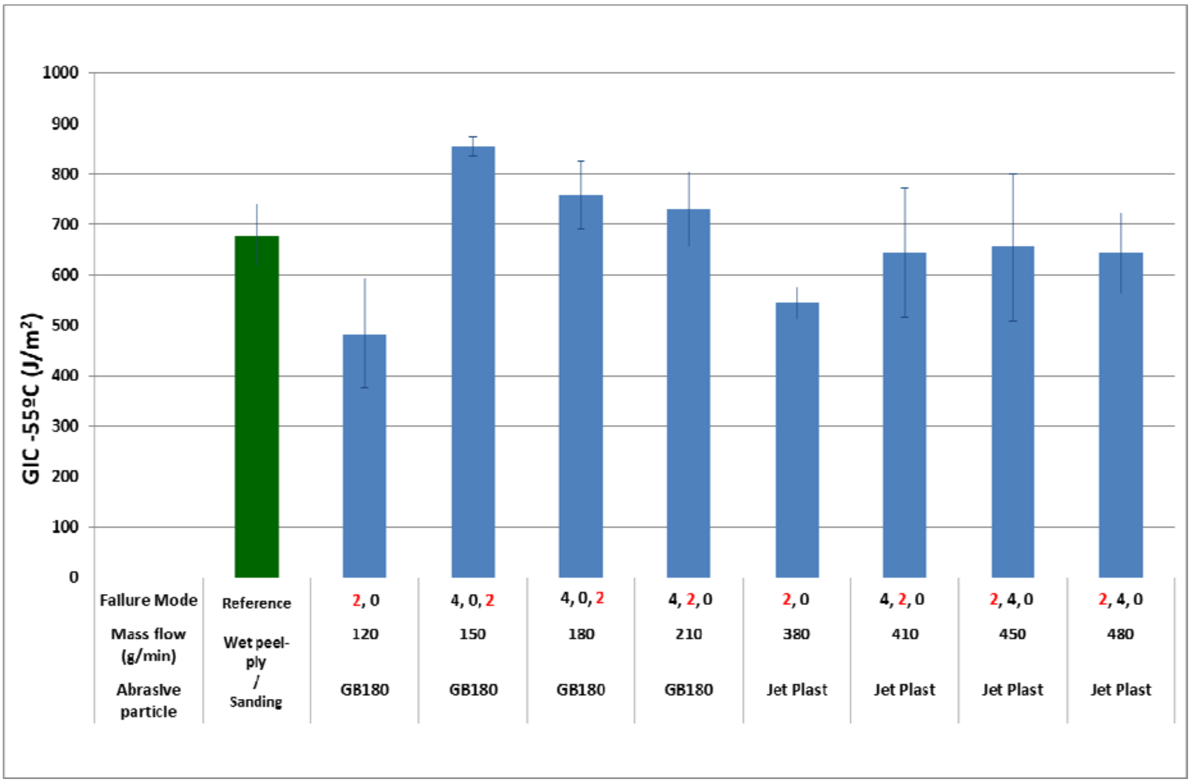

Figure 8. VGB test results

As is seen in Fig.8, in general, panels treated with glass beads abrasive particles (GB180) presented better results than the observed for Jet Plast.

Adhesion failure mode (2) is observed in all specimens treated with Jet Plast abrasive particles for all different process parameters studied. The scatter observed on the results is also high due to the non-homogeneity of the treatment. Further trials would be necessary to find effective treatment parameters for this work conditions (CFRP substrate / abrasive particle).Considering the high mass flow already studied here, the use of Jet Plast particle for industrial purposes is not recommended.

In general, Glass Beads particles (GB180) presented better results than Jet Plast. For low mass flow (120g/min), the main failure mode observed was adhesion (2) confirming the low mass flow is not enough to achieve an effective surface activation.

For the higher mass flow studied in this work, 210 g/min, GIC values increased, however, significant areas with adhesion failure mode were also observed.

For medium mass flow ranges, 150 and 180 g/min, the obtained GIC values were higher and in the same range of standard values of fracture toughness energy than this material combination when treated with wet peel-ply or manual sanding. Nevertheless, local areas with adhesion failure can be observed. Based on these results, surface analysis was performed directly on GIC specimens to find the explanation of the local adhesion failure modes found.

Microscopic investigation and surface analysis were performed by SEM and XPS respectively, on specimens after GIC tests to try to identify the source of the adhesion failure. As it can be observed in Figures 9 and 10, there are traces of abrasive particles on the precured semipanel surface. After detailed analysis by EDX it could be confirmed that these traces correspond to metals (Cu, Ti, Al, and Fe) present in the composition of abrasive particles (Al_2O_3, TiO_2, SiO_2…). This observation led us to believe that a cleaning operation after the grit blasting process will be mandatory to remove completely the particles remaining. Due to the small size of abrasive particles, the power of vacuum system is not enough to clean completely the surface and the particules are trapped on the cavities of the rough surface.

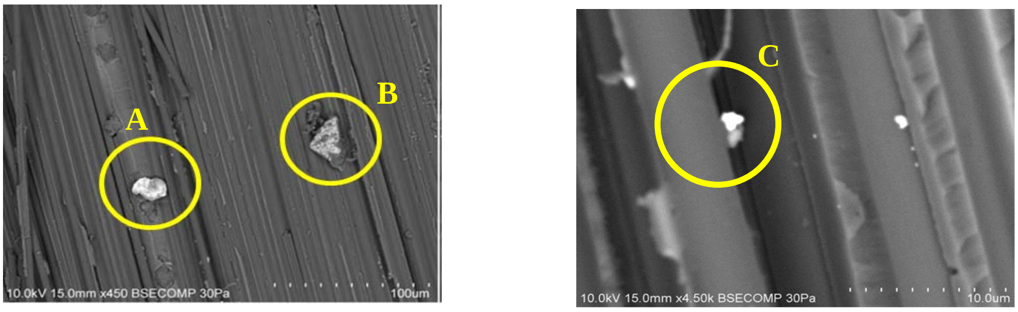

Figure 9. SEM imagines on GIC specimens

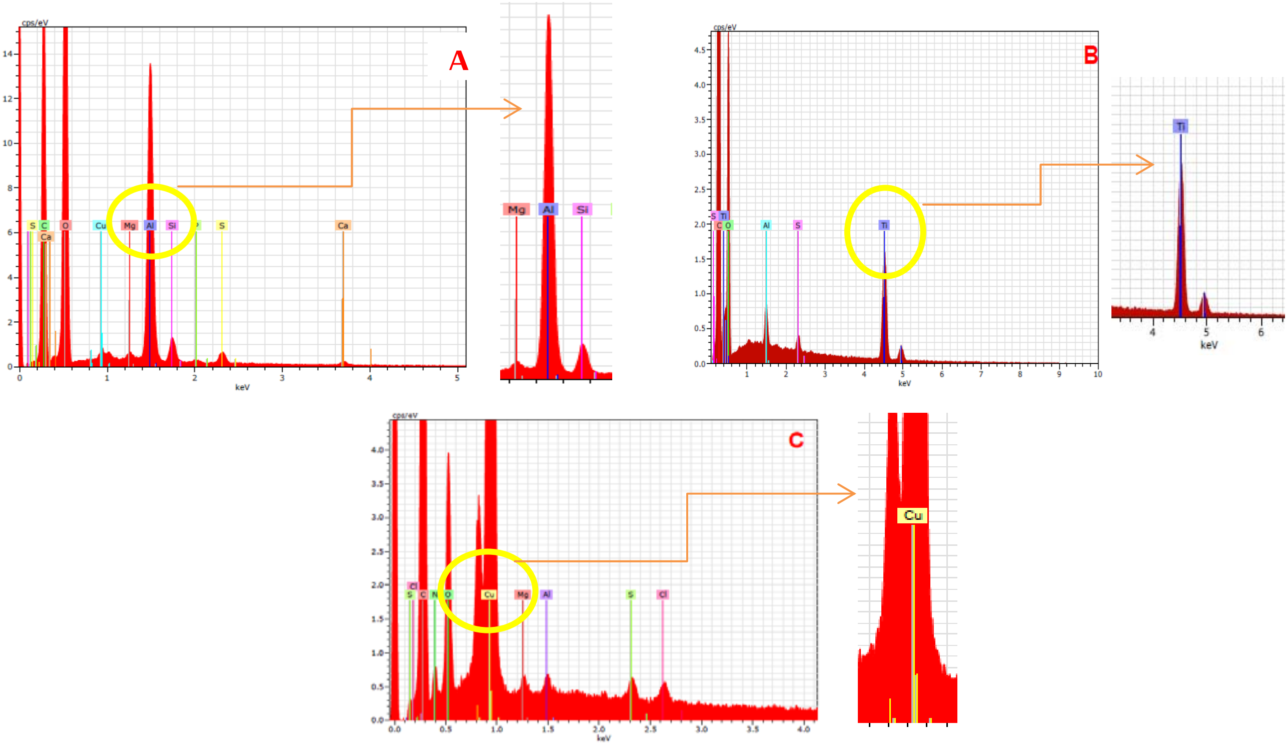

Figure 10. SEM-EDX spectrum on A, B and C details of Figure 11.

4. Conclusions

In the case of Atmospheric Pressure Plasma, its efficiency as automatic surface treatment prior to bonding operations has been proved. With a 3 static nozzle equipment, 100mm/s nozzle speed and a distance range to the surface between 13-15 mm, were found to be efficient process parameters for the epoxy CFRP substrate and the adhesive film used in this work. A wettability increase through the incorporation of new polar functionalities and the complete elimination of fluorine surface contamination (confirmed by XPS) resulted in a successful bond line, as confirmed by results from GIC (-55ºC/AR), with fracture toughness energies meeting the requirement and wet-wet failure modes.

With VGB technology, 100mm/s nozzle speed with a mass flow range between 150-180g/min seems to be appropriate process parameters for this substrate. However, due to the lack of homogeneity and/or reproducibility, local percentages of adhesion failure on GIC specimens and the detection of abrasive particles remaining on the surface after the surface treatment, a new test campaign, including cleaning operations after treatment is recommendable .

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