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Atmospheric pressure plasma jet for surface texturing of C/SiC

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ABSTRACT

C/SiC composites are materials to be used in harsh environments overcoming the limits imposed by the intrinsic brittleness of their ceramic constituents while providing both high mechanical performances at high-temperature temperatures and low weight. In order to manufacture the final component, joining C/SiC, to itself or to other materials, is often necessary, and it is critical to maximize the strength of the joints (similar or dissimilar) in order to meet reliability criteria.

In the present work, a pre-joining treatment based on an atmospheric pressure plasma jet (APPJ) was proposed to introduce a brush-like texture on the surface via the selective removal of carbon fibers. The investigation of treated surfaces via electron microscopy and confocal 3D-profilometry confirmed that the treatment was effective in introducing a brush-like texture and in increasing the available contact area. Wettability test and inspection of cross-section of CB4 wetted samples were then carried out. The latter confirmed the formation of anchoring points given by the brush-like texture. Finally, the effectiveness of the treatment in improving the joint strength was assessed by comparing the apparent shear strength of CB4 brazed composites, with and without the APPJ pre-treatment. The joints with plasma pre-treated C/SiC showed a shear strength of about 66 MPa, 44% more than the strength of joints produced with untreated C/SiC.

1. Introduction

Carbon-fiber-reinforced ceramics are employed for aerospace applications because of their resistance to high temperatures, their ability to retain mechanical properties even at high temperatures, and their low density [1]. Carbon-fiber reinforced carbon (C/C) composites provide exceptional performances up to 2500 °C, but they are very sensitive to oxygen when the temperature exceeds 400 °C [2]. This strong limitation encouraged the development of carbon-fiber-reinforced silicon carbide (C/SiC) composites. SiC matrix and coating prevent the oxidation of the carbon fibers, extending the possible application for these composites in oxidizing environments [3].

C/SiC have to be integrated with other materials, of the same type or different (usually metals), in order to manufacture the final component. However, such integration is rather complicated because of the service conditions that are targeted by the part and it is achieved during joining operations [4]. Most solutions rely on indirect joining techniques. Common joining materials are polymeric adhesives, when low temperatures are targeted [5], brazing alloys [6], and glass ceramics [7].

The joining material and the joining processes have a critical role in the effectiveness of the joint, but the surface of the adherend plays an equally important role. Indeed, the interface between the adherend and the joining material plays a key role for the good quality of the joint. Surface preparation is therefore an important preliminary step prior to joining and it may include cleaning out contaminants, surface activation, functionalization and texturing. According to the type of material and the desired effects on the surface, several surface techniques can be considered.

Among all the effects that may be promoted on the surface, one is the interlocking mechanism between the materials to be joined and the joining material. This phenomenon raises when the joining material infiltrates valleys formed on the surface forming anchoring points at the interface that provide a reinforcement effect, beneficial for the mechanical strength of the joint.

In order to provide the interlocking, it is necessary to tune both surface roughness and texture. With a focus on ceramic materials, texturing can be achieved by mechanical machining, but this may result in fast tool wear due to the high hardness and to the risk of originating

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Table 1
Technical specifications for the PlasmaTEC-X (adapted from Ref. [19]).

	Generator	Nozzle
Main Voltage	220V	
Main Frequency	50 Hz	
Output voltage	550 VA	425 W
Ramp-up time	10 ms	
Compressed air supply	5–6 bar	

defects who may trigger in-service failures. Other techniques can take over these issues such as plasma etching and laser texturing [8]. The latter has the great advantage of being versatile and very accurate in engineering high-precision superficial pattern when the laser-material coupling is optimized. Instead, plasma etching requires the use of masks in order to provide textures [9].

Ceramic Matrix Composites (CMCs) are composed by several constituents, such as fibers, matrix and interphase. The presence of multiple constituents introduces new opportunities for texturing via plasma etching. Indeed, the different set of properties, provided by each constituent, results in a different response when the material undergoes a treatment enabling selective removal of the less resistant constituent and therefore providing texturing without masking.

In particular, the selective removal of fibers or matrix can provide a brush-like texture rich in distributed cavities that can be infiltrated by the joining material. Such structure was first manufactured on SiC/SiC using a thermal treatment [10], but the exposure of the entire volume of material to high temperatures resulted in the reduction of the mechanical properties of the composite [11]. A similar thermal process was delivered by Niu et al. on C/C [12].

To avoid any detrimental effect on the composite properties and make the process more effective in terms of cost and time, the selective removal treatment should be confined at the surface like any etching process. According to the type of material that has to be textured, the viable process can be selected. For instance, a brush-like texture was originated on a SiC/SiC composite by means of a low-pressure reactive plasma taking advantage on the slight differences in composition and structure between the fibers and the matrix [13]. Low pressure plasmas are a standard for etching operations as demonstrated by their decades-long use in the semiconductor industry [14], however they impose some limitations given by the presence of vacuum equipment that introduces complexity and expensive maintenance in the process and makes extremely difficult to work on large parts and continuous operations. On contrary, atmospheric pressure plasmas (APPs) have the potential to overcome these limitations and therefore they may be interesting for some industrial fields [15].

To date, APPs are largely employed, in particular in the plastic industry, but their use for texturing CMCs has never been reported before. Among APPs, the Atmospheric Pressure Plasma Jet (APPJ) can provide a stable plasma plume directly on the surface, mitigating inhomogeneity issues [16]. Furthermore, APPJ can be used in scanned mode to treat large surfaces and integrated in the production lines. Commercial systems commonly work using air as plasma gas, but for more specific application other gas mixtures are used [17,18].

In C/SiC, the difference in oxidation resistance between the C fibers and the SiC matrix can be leveraged in order to produce a selective removal at the surface and therefore introducing a brush-like texture available for being infiltrated by the braze. Oxidation treatment in furnaces results in excessive damage to the composite, while the use of an air-fed APPJ can limit the effect at the surface.

This study proposes the utilization of a commercially available atmospheric pressure plasma jet (APPJ) as a surface preparation tool for creating a brush-like texture. The primary objective is to assess the effectiveness of this texture in enhancing joint strength. The research investigates the evolution of the surface resulting from the APPJ treatment. Subsequently, specific conditions are identified that facilitate the formation of the desired brush-like texture while minimizing material removal and allowing for shorter treatment duration. These conditions are of particular interest for potential industrial application of the technique. Lastly, the study evaluates changes in the wettability of the braze and examines the impact of the pre-treatment on joint strength.

2. Materials and methods

Uncoated 2D Keraman C/SiC (MT Aerospace, Germany) were selected as materials to be joined. They were supplied as 100 mm × 100 mm plates with a thickness of 4 mm and they were cut in smaller samples with a size of 10 mm × 10 mm by means of a high-precision cutting machine (ATM Brillant, Verder Group, Netherlands), equipped with a diamond wheel.

The thickness of the composite was chosen as surface to be plasma-treated because of the 2D structure of the composites. Indeed, the 0°/90° piling exposed fibers perpendicular to the plasma jet viable for providing the brush-like modification. The C/SiC surface was polished using SiC grinding papers up to 2400 grit before the plasma treatment.

The plasma system used for the activity is a PlasmaTec-X generator equipped with one nozzle (Tantec, Denmark). In Table 1, the technical specification given by the manufacturer are reported.

The plasma treatments were carried out by selecting a distance of the surface from the nozzle at 5 mm and the air flux at 1500 l/h. Such parameters were kept fixed. Several treatments were tested with different time lengths. Samples were weighted before and after the treatment to estimate any variation of their mass.

Three samples were analyzed for each condition: 30 s, 1 min, 5 min and 10 min. Temperature was controlled via a thermal image camera (E6, FLIR, USA). Surfaces were then inspected via a Benchtop SEM (JEOL, Japan). A slice of approximately 400 μm was cut parallel to the surface in order to inspect the quality of the composite after the plasma treatment.

After these preliminary tests, the operating parameters used for further investigations were chosen, which are 30 s, 1500 l/h, 5 mm. This setting, indeed, minimized the weight loss of the composite. Furthermore, a short treatment it is particularly interesting for being implemented in industrial pre-bonding operations.

Surface features of the untreated and treated samples were quantitatively analyzed by the confocal technique using a 3D non-contact profilometer (Sensofar S-neox) placed on a vibration isolation system (Halcyonics Micro 40). Quantitative measurements have been performed according to the ISO 25178 standard using the software embedded in the system (SensoSCAN).

Ag-Cu-Ti alloy, indicated as Brazetec CB4 (Saxonia, Germany), was selected as brazing material and used in form of foils with a thickness of around 100 μm. Its nominal composition, according with the datasheet [20] is: 70.5 wt% Ag; 26.5 wt% Cu; 3 wt% Ti; the melting range is 780–805 °C and the suggested working temperature is in the range 850–950 °C. Such braze is known to be effective for ceramics [21,22] and to promote their wettability thanks to Ti migration at the interface [23,24].

The brazing temperature has been chosen at 950 °C, according to previous experimental activity [23] and in order to avoid the miscibility gap between two different liquids; moreover, the increased activity of Ti at higher temperature promotes a better wetting behavior.

AgCuTi alloys has been previously used for joining CMCs [11,25] and therefore it was considered suitable for assessing the effectiveness of the treatment in improving the joint strength.

Wettability of the CB4 braze on the C/SiC surfaces was studied by the sessile drop method starting from arc-melted alloys having the same composition of the brazing alloys used for joining trials. For each test, a piece of the alloy in the form of a small ball with mass of about 0.1 g was placed on the top of the surface and then they were inserted in a vacuum furnace [26] at the temperature of the joining treatment (950 °C) to evaluate the evolution of the contact angle during 30 min of liquid-solid

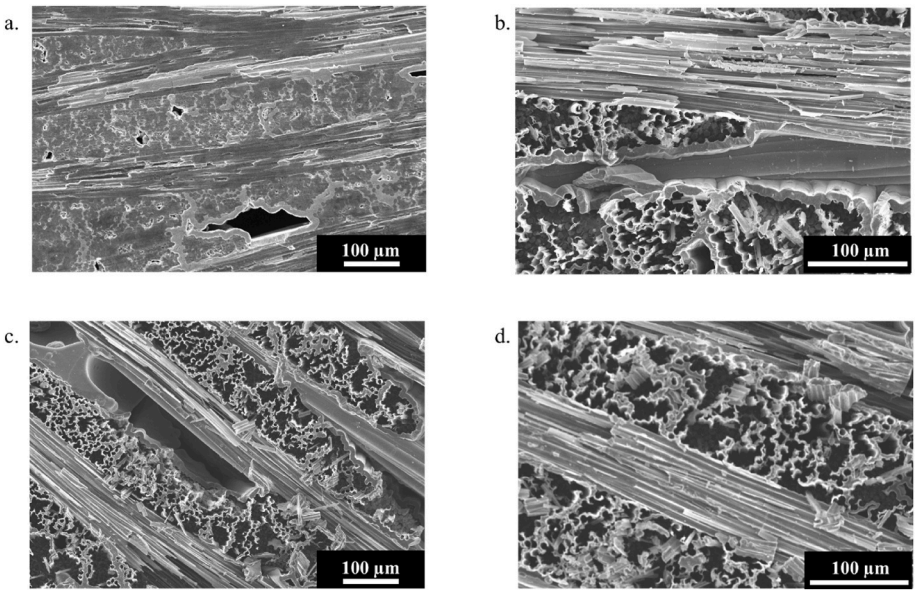


Fig. 1. SEM pictures Comparison of the top views of C/SiC before (a) and after APPJ: 1 min (b), 5 min (c) and 10 min (d), at higher magnification. Images taken in Secondary Electrons (SE) mode.

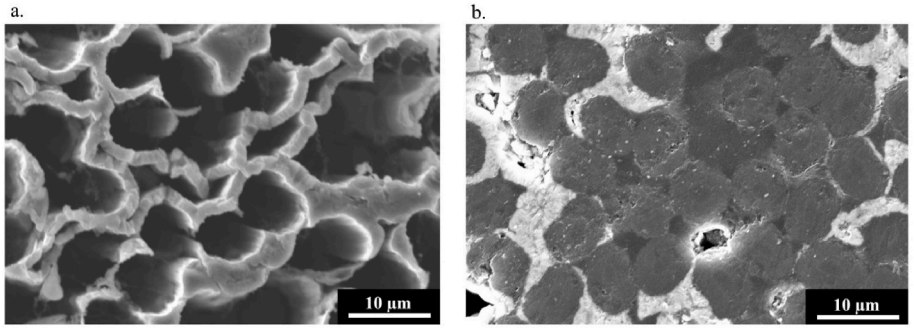


Fig. 2. 5 min APPJ-treated C/SiC: exposed surface (a) and an internal surface taken 400 µm below (b).

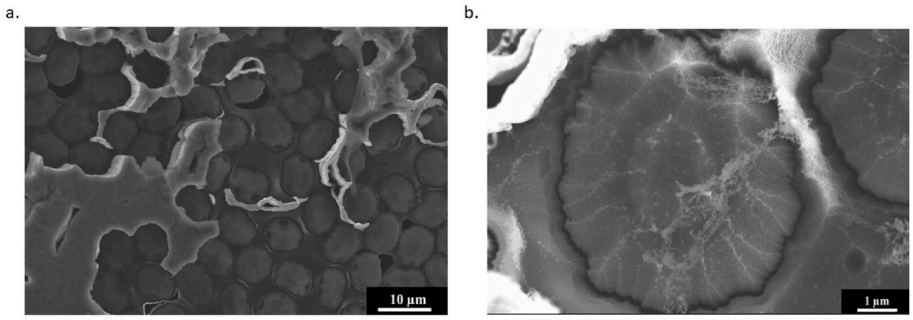


Fig. 3. Top-view of 30 s APPJ treated C/SiC surface (a) with a detailed of the carbon etched fiber (b) (SE mode).

contact. After the wetting tests, samples were cross-sectioned and polished for microstructural examination by SEM-EDS (Scanning Electron Microscope – Energy Dispersive Spectroscopy).

Finally, joined samples were manufactured for mechanical tests. One foil of CB4 was inserted between the two C/SiC surfaces and then a thermal treatment was carried out in a tubular furnace (Nabertherm, Germany) equipped with a vacuum system (950 °C, 10 min, 10 °C/min, vacuum atmosphere). The joined samples were analyzed by means of SEM-EDS.

At least three samples for each type of joints (prepared with untreated and APPJ treated C/SiC) were tested in Single Lap Offset (SLO) compressive shear stress test at room temperature. Additional information on SLO configuration are available in Ref. [27]. The equipment used for the test was a universal testing machine Sintec D10, fitted with a 50 kN load cell that worked at a crosshead speed of 1 mm/min. Post mortem analyses of the fracture surfaces were carried out by means of SEM and EDS.

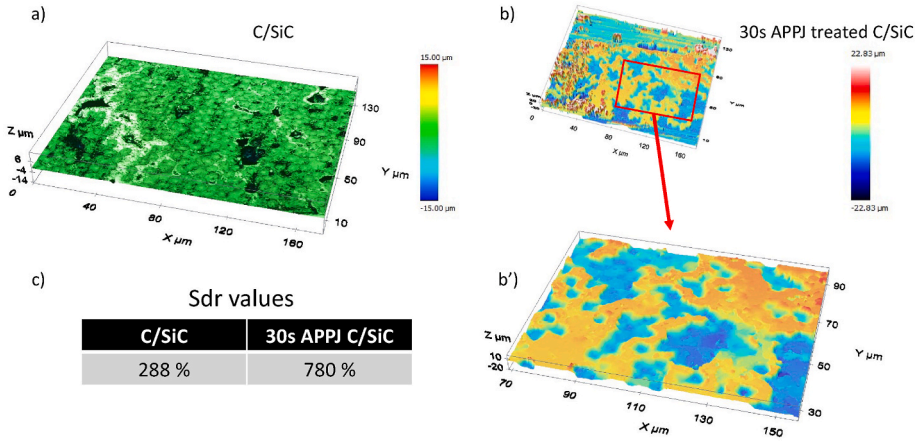


Fig. 4. 3D reconstruction of the surfaces of a polished C/SiC (a) and a 30-s APPJ etched C/SiC (b); (b'): zoomed view of a region of b with fibers perpendicular to the surface; (c): Sdr values.

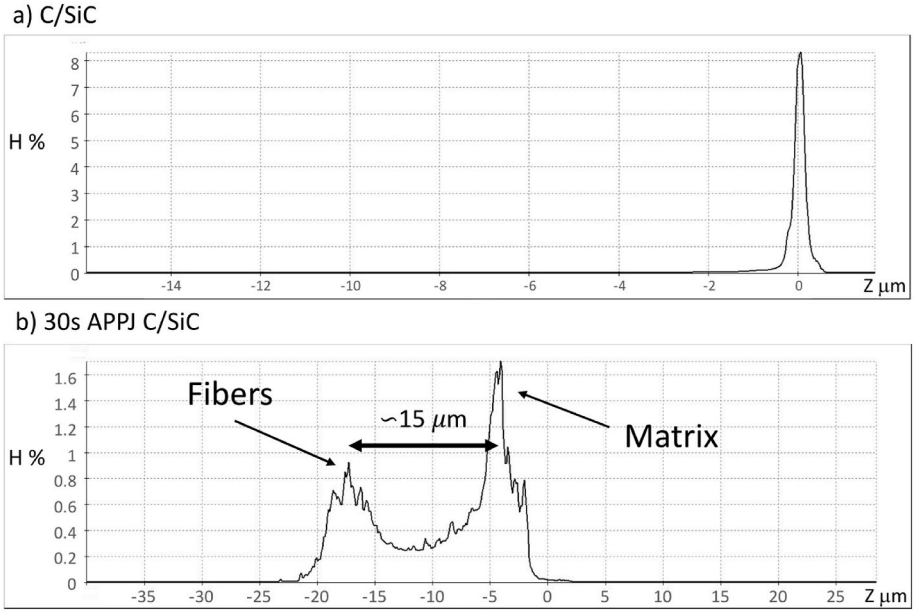


Fig. 5. Height distribution for the surfaces of untreated C/SiC (a) and 30 s APPJ treated C/SiC (b).

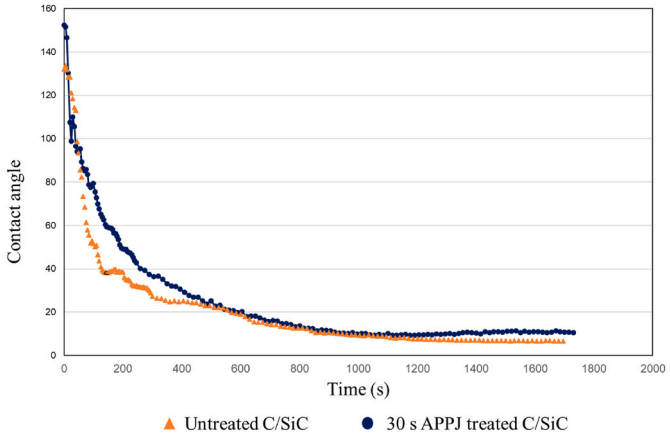


Fig. 6. Evolution of the contact angle at 950 °C of CB4 braze on the surface of untreated C/SiC (orange triangle) and 30 s APPJ treated C/SiC (blue circle).

3. Results and discussion

Preliminary APPJ treatments on C/SiC were carried out for 30 s, 1 min, 5 min and 10 min. The weight loss increased with time: after 30 s of APPJ treatment, the average weight loss was about 0.18%, 0.42% after 1 min, 0.55% after 5 min and 0.82% after 10 min.

Fig. 1 compared the C/SiC surface before and after the plasma treatment: polished (1.a), 1 min APPJ (1.b), 5 min APPJ (1.c) and 10 min APPJ (1.d). The most evident effect was the remarked removal of the carbon fibers, which left cavities surrounded by the SiC matrix on the surface. This can be observed for all time lengths and it can be deducted that the main contribution to weight loss of samples was given by progressive removal of fibers. This appeared to be intense even after 1 min of treatment, while the matrix globally seemed to be almost unaffected by the treatment, but collapsed in some regions where it had no contact anymore with the bunches of fibers.

Air-fed APPJ are known to promote activation of polymeric surface by oxidation of the surface [28]. Furthermore, the formation of reactive oxidizing (ROS) species by APPJ system has been extensively reported in literature, in particular for biomedical applications [29–31]. This occurs

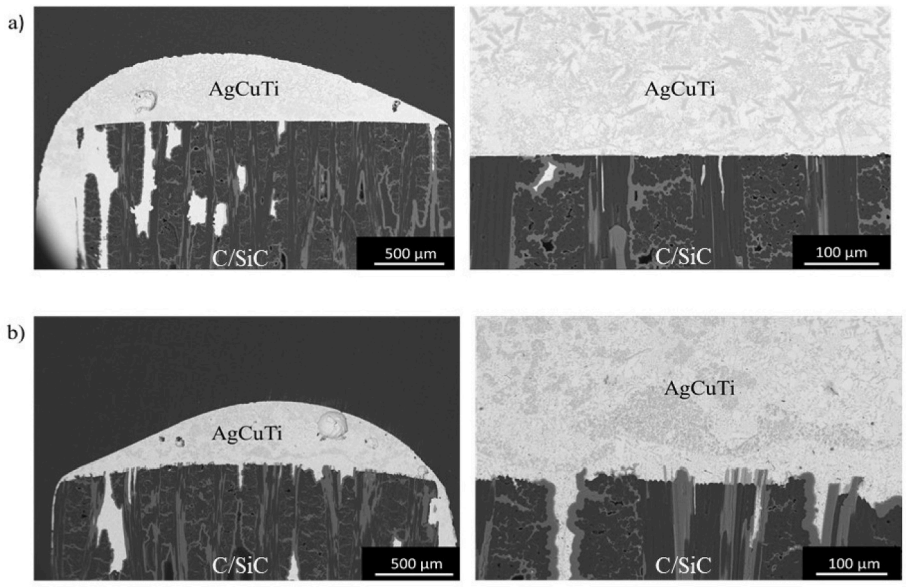


Fig. 7. Cross-sections of CB4 wetted C/SiC after wetting tests: untreated (a) and 30s APPJ treated (b) C/SiC. Micrograph was taken in BSE mode.

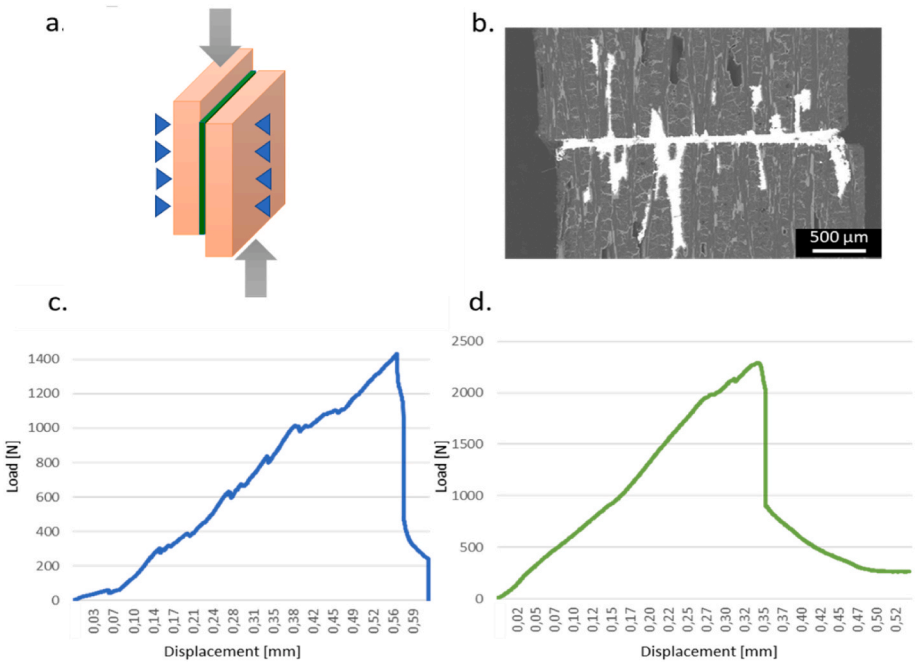


Fig. 8. Sketch of the SLO test configuration (8.a.), micrograph of a cross-section of a 30s APPJ joined C/SiC taken using BSE mode (8.b), stress-strain curve of an untreated joint (8.c) and stress-strain curve of a 30s APPJ treated joint (8.d).

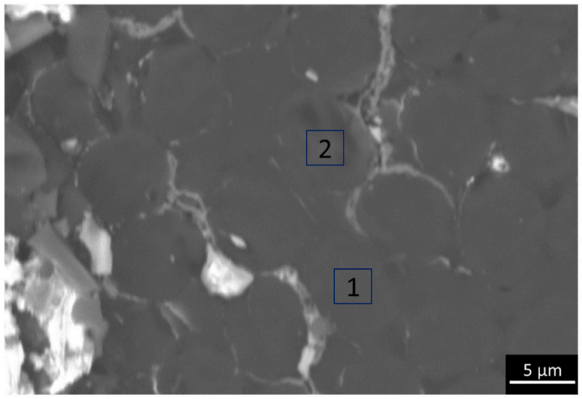
even when an inert plasma-forming gas like helium is used because of the secondary excitation of the surrounding air that forms oxygen-containing species. When using an air-fed the primary plasma itself, generated by excitation of air in the nozzle, is expected to be rich of oxidizing species. Therefore, even if in this work it was not possible to identify and quantify the ions and radicals generated by the plasma, it is reasonable to speculate that the surface interacted with a strong oxidizing environment.

In air at atmospheric pressure, carbon fibers are affected by oxidation starting from 400 °C [32]. The temperature of the plasma plume, according to the measurements carried out with a thermal camera, never exceeded 250 °C. The rapid removal of fibers at such temperature can be explained by the introduction in the environment of highly energetic and reactive oxygen-rich species formed by the APPJ treatment. The SiC matrix has a higher oxidation resistance – oxidation begins when temperature is higher than 1000 °C - and therefore was not affected by the treatment, as confirmed by EDS analysis (not reported here).

The underlying (around 400 µm) layers looked unaffected by the treatment. Fig. 2 compares the surface treated for 5 min and the underlying surface cut at a depth of approximately 400 µm for the same sample. It can be noticed that the inner surface was completely unaffected by the treatment, carbon fibers included. This confirmed that the effect of the plasma was limited to the surface.

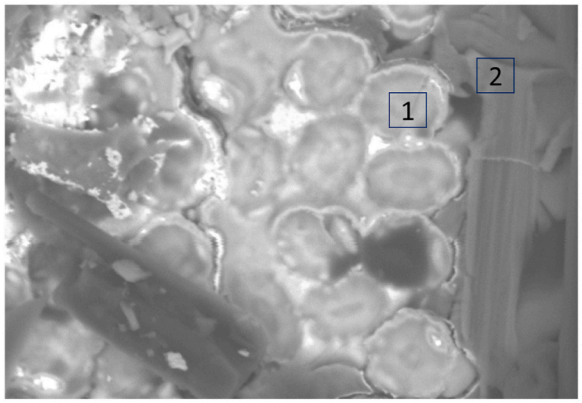
The APPJ treatment provided a strong oxidation of the carbon fibers. Longer exposure did not damage the composite. However, to make this treatment viable for industrial applications, shorter treatment durations

a.



		C	O	Si	Ti	Cu	Ag
1	At %	100	-	-	-	-	-
2	At%	100	-	-	-	-	-

b.



		C	O	Si	Ti	Cu	Ag
1	At %	13.2	2.12	2.55	62.53	14,55	5.13
2	At%	53.12	2.52	43.52	0.41	0.27	0.16

Fig. 9. EDS analysis on the fracture surface after mechanical tests of untreated C/SiC (a) and APPJ C/SiC (b). Images taken using BSE mode.

are particularly advantageous. For this reason, the shortest plasma treatment (30 s) was selected for the assessment.

After the 30 s plasma treatment, indeed, the fiber removal on the C/SiC surface appeared less intense compared with the other plasma treatments (Fig. 3). Channels were shorter in depth and fibers at their bottom are easier to be detected, on contrary to the other conditions of removal.

The eroded fiber, visible at the end of the channel (Fig. 3), looked slightly blunt at its center and detached at the interface with the matrix. Fibers are rather flat, suggesting that the treatment may induce a homogeneous removal.

For this reason, the 30 s APPJ treatment was used to prepared the samples before joining. The surface of polished and 30 s plasma treated C/SiC were analyzed with profilometry (Fig. 4). The surface area is crucial for providing interlocking: the higher the surface area, the higher is the contact between the adherend and the joining material, and the superficial texture is responsible for the anchoring points. The values calculated for the Developed Interfacial Area Ratio (Sdr) [33] are given in the table of Fig. 4. Sdr provided an estimation on how large is the deviation of the surface from an ideal plan (Sdr = 0). The higher the Sdr, the less planar is the surface.

For both samples the Sdr values are high and this can be explained by the irregular distribution of fibers, matrix and cavities given by the manufacturing process. The Sdr for the polished C/SiC (Fig. 4a) was 288%, but its surface structure looked pretty homogeneous, since slight color variations are visible in the profilometer reconstruction. Whereas, the 30-s APPJ treated C/SiC (Fig. 4b) exhibited a wider color distribution with two regions that can be identified. One constituted by higher points that are close to yellow and, according to the SEM observation, fit with the matrix and another which was blue-colored and corresponds to the etched fibers. The Sdr after treatment raised to 780%, an exceptionally high value. The Sdr values have to be intended as suitable for indicating the surface evolution given by the APPJ treatment and it is evident the remarkable increase of the exposed area.

The presence of two regions at different heights was pointed out by the height distribution graphs which are shown for a polished C/SiC and a 30 s APPJ treated C/SiC in Fig. 5a and 5b, respectively. The untreated samples exhibited a single peaked distribution, indicating that most of the surface lies on the same height. On the other hand, for the APPJ treated samples two peaks were detected: the left-side one corresponds to lower height surface, i.e. to the eroded fibers while the right-side one is associated with the matrix.

The presence of the two peaks in the distribution is a further indication of the successful selective removal at the surface. The difference between the maximums of each peak was approximately 15 µm. Similar height distributions and differences in peaks altitude were reported for all the analyzed samples and therefore 15 µm may be considered as the average etching depth given by the treatment.

Several profilometer analysis were carried out on multiple samples to investigate the evolution of the surface. They returned results confirming the same surface evolution given by the treatment, suggesting a good reproducibility. However, it must be remembered that the distribution of pores, fibers and matrix of each specimen has a strong influence in the APPJ treatment.

The selective removal caused at the surface by the treatment was, therefore, confirmed by both electron microscopy inspection and subsequent profilometry.

Once evaluated how the surface changed as a result of the treatment, its effect on the wetting of the brazing alloy selected for the test was investigated. The evolution of the contact angles of the CB4 braze on the surface of untreated C/SiC and 30 s APPJ treated C/SiC at 950 °C is reported comparatively in Fig. 6. The wettability was very good for both the types of samples being the final contact angle 7° and 10° for the C/SiC and the APPJ treated C/SiC respectively; also, no evident differences were noticed in the evolution of contact angles vs. time before and after the treatment (Fig. 6). This indicates that the treated surface does not present differences in chemistry compared to the untreated ones. Both the kinetics of wetting, guided by the interfacial interactions of active Ti with the composite, and the final contact angles agree with the literature reported for Ag-Cu-Ti alloys in contact to ceramic [10,21–24,34,35].

After wetting tests, the cross-sections of samples were investigated in order to evaluate the interface between the braze and C/SiC surface (Fig. 7). The infiltration of the braze in the composite was remarkable in both polished and APPJ treated C/SiC. The open cavities left by the manufacturing process of the composites were completely filled by the alloy. This has to be taken in account because when the joining process can result in a detrimental depletion of material from the joining area, that may result in a weak joint.

CB4 alloy formed a sound interface with untreated C/SiC (Fig. 7a). Looking at the cross-section, the surface of untreated C/SiC was flat, with fibers and matrix at the same height. No anchoring points for interlocking were detectable, with exception for the regions with infiltrated cavities. The surface of 30 s treated C/SiC showed matrix and fibers at different height (Fig. 7b). This can be observed, in particular, where fibers are perpendicular to the surface. There, the matrix (light grey) was higher than fibers (dark grey), as expected from profilometry analysis. The CB4-C/SiC interface was continuous also for the treated samples, but additional infiltration arose because of the selective removal of fibers.

The joined samples in SLO configuration (Fig. 8a) were then tested to

evaluate the effectiveness of the APPJ-induced brush-like texture in improving the joint strength. The joints produced with pre-treated C/SiC (Fig. 8b) showed a shear strength of 65.8 ± 2.5 MPa, 44% more than the strength of joints produced with untreated C/SiC (45.5 ± 0.6 MPa). In both cases the joined samples failed with a mixed mode (cohesive/adhesive). Typical stress-strain curves of two joints are shown in Fig. 8c-d.

Regions with the brush-like texture looked to retain more braze on fibers compared with untreated samples (Fig. 9). The presence of the CB4 was confirmed by EDS analysis, confirming an enhanced adhesion on such regions manufactured by the APPJ treatment.

4. Conclusions

An air-fed commercially available APPJ was used to apply a preliminary surface treatment, before joining, on C/SiC composites using different operating parameters. A promising treating procedure was identified and the related obtained surface used for wetting and joining test considering CB4 alloy as brazing material.

C/SiC treated surface showed a brush-like texture, given by the plasma-induced preferential oxidation of C fibers perpendicular to the surface. Operating parameters, such as 30 s, 1500 l/h and 5 mm, appeared to be the most effective in providing the selective removal while keeping the treatment short.

The wetting behaviour of a liquid brazing alloy, namely CB4, was tested at 950 °C and it was similar for both the types of substrates with an equilibrium contact angle of 7° and 10° for the C/SiC and the APPJ treated C/SiC respectively.

The APPJ treatment provided interlocking between the braze and the C/SiC because of the formation of anchoring points that were proved to be effective when joined samples were mechanically tested and provided an increase of joint strength higher than 44% after treatment.

In conclusion, APPJ proved to be a promising technique for preparing C/SiC prior to joining by introducing a texture able to promote the manufacturing of more reliable joints. This, coupled with the flexibility of the technique, is promising for extending the treatment to industry by working on large size parts and in continuous mode.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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