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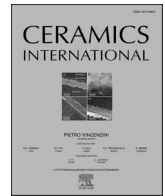
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# Plasma etching as a surface engineering technique for SiC/SiC composites to improve joint strength

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## ABSTRACT

Silicon carbide fiber reinforced silicon carbide (SiC/SiC) composites have unique properties that make them suitable for demanding applications. These materials have to be joined with other materials or with the same type of material to produce the final component. This work investigates the effectiveness of a fluorine-based plasma process on SiC/SiC composites as a surface engineering technique to manufacture a brush-like textured surface. The studied pre-treatment induced an interlocking effect at the composite/joining material interface, thus improving the joint strength of the joined components.

Several plasma conditions were considered and the most promising one (CF<sub>4</sub>, 20 sccm, 200 W, 30 min) was selected to assess the effect on the mechanical performance of SiC/SiC brazed with a commercial brazing alloy (Cusil-ABA®). The selected plasma pre-treatment led to a 55% increase in the apparent shear strength of the joined SiC/SiC composites.

## 1. Introduction

Silicon carbide fiber-reinforced silicon carbide (SiC/SiC) composites can withstand harsh environments and can also provide specific mechanical properties, such as high elastic modulus and strength, coupled with low density. Furthermore, their composite architecture overcomes the limits imposed by their intrinsic brittleness, thereby extending the possibility of their use. These properties make them interesting for challenging applications, where harsh conditions (e.g. high temperatures, aggressive chemicals, etc.) have to be taken into consideration, for example, in the aerospace [1] and energy fields [2,3].

It is usually necessary to join certain parts to manufacture the desired product. This is not a trivial operation, especially when the final component has to be used in an aggressive environment.

It is particularly difficult to bond SiC/SiC composites via direct joining techniques. SiC melts incongruently at around 2700 °C [4], a temperature at which it transforms from a monophasic solid to a biphasic system of liquid and carbon. In such a case, welding is not possible. The alternative solutions are direct diffusion-based joining techniques, such as Spark Plasma Sintering (SPS) [5], which has proved to be effective in bonding ceramic composites [6], but imposes the

necessity of strict limitations on the shape and size of the components.

Mechanical joints may be difficult to manufacture because of the risk of introducing defects, such as cracks, into the composite during drilling and/or shaping operations [7]. Furthermore, it is necessary to evaluate the possibility of local stress concentrations, which may arise due to the presence of the manufactured holes.

Therefore, indirect joining is considered a more likely choice for assembling the final part. Several types of joining material can be used, according to the temperature range of the application and other requirements: polymeric adhesives, brazing alloys, and glass-ceramics [8]. Regardless of the nature of the adhesive, it is possible to improve the bonding strength by introducing a texturing effect onto the bonding surface, which is expected to be beneficial for mechanical interlocking. Indeed, the presence of valleys and asperities on the surface can promote infiltration of the joining material, thereby creating anchoring points. Additionally, the contact area between the two materials increases.

The fibers and the matrix composite architecture in SiC/SiC composites enables a brush-like texturing effect to be manufactured as a result of the different responses of each phase to such stimuli as chemical and thermal etching (Fig. 1) [9]. Valenza et al. [10] manufactured a structure for SiC/SiC using a selective thermal removal (STR) treatment.

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This pre-treatment was effective in preferentially removing fibers from the surface, and in providing anchoring points for the joining material, because of the brush-like profile of the surface. Unfortunately, since the entire volume of the material underwent the thermal process, the mechanical properties of the SiC/SiC composite were compromised [11]. This result highlighted the need of a texturing treatment, confined to the surface, to preserve the performances of the components.

A major barrier to etching-based texturing is the chemical inertness of SiC, which, however, is also one of its most interesting properties as a material for applications in harsh environments. Indeed, few etchants are available for the wet etching of SiC. As a result of the interest in SiC as a semiconductor, plasma-based dry etching strategies that are able to guarantee a high level of accuracy have been developed [12,13]. In this case, the removal of material is possible thanks to reactions with highly reactive free radicals and energetic ion bombardment. Some common plasma-forming gases for this application are: tetrafluoromethane (CF<sub>4</sub>), and other fluorine-based gases that may be used alone or mixed with hydrogen or oxygen, in order to have better control of the process [14, 15].

The use of plasma etching is not only limited to semiconductor applications, it can in fact be proposed as a surface preparation technique. Casalegno et al. [16] proved that a Reactive Ion Etching (RIE) plasma treatment, using CF<sub>4</sub> as a plasma gas, was effective in modifying the surface of SiC to improve the strength of joined SiC. To the best of the authors' knowledge, the research on the dry etching of SiC/SiC is still somewhat unexplored. Zimmer et al. [17] proposed laser-induced plasma etching for the precise machining of SiC/SiC composites. In their work, the reactive plasma was generated by exciting a CF<sub>4</sub>/O<sub>2</sub> mixture through a femtosecond-pulsed laser. However, this method can be considered more like laser texturing [18] than traditional dry etching.

This work investigates the use of a low-pressure plasma treatment to obtain a brush-like surface on SiC/SiC composites, thus effectively improving the mechanical strength of joints manufactured by brazing through the promotion of interlocking structure at the composite/braze interface.

## 2. Materials and methods

2D Keraman SiC/SiC composites, manufactured by BJS composites (Germany), were used for the experimental activity. This material consists of plies of Tyranno S fibers, a pyrolytic carbon interphase, and a chemical vapor infiltrated (CVI) SiC matrix. Plies of Tyranno S fibers are arranged along the thickness of the composite in a 0°/90° piling, therefore showing perpendicular-to-the-surface oriented fibers, suitable for providing a brush-like structure. The material was supplied in the

form of 100 x 100 x 4,5 mm specimens and was then cut into smaller pieces (approximately 10 x 10 x 4,5 mm) using a precision cutting machine (*ATM Brilliant 220, Germany*). After cutting, the specimens were mirror-polished using SiC grinding paper, with a grit of up to P2400. They were then ultrasonically cleaned in an ethanol bath for 20 min at room temperature.

A plasma treatment was carried out, under several different conditions, on one of the 10 x 4.5 mm surfaces, according to the thickness of the sample. The samples were placed on the powered electrode of an RF (13.56 MHz) plasma system. The reactive atmosphere was composed of both CF<sub>4</sub> alone and in a mixture with H<sub>2</sub> (10%) [19]. The power and working pressure were fixed at 200 W and  $9 \times 10^{-2}$  mbar, respectively, while the treatment time was varied from 5 to 30 min (Table 1).

Top view micrographs of the samples were collected by means of an SEM-EDS scanning electron microscope (*JEOL Benchtop Scanning electron microscope equipped with an EDS Analyzer and SUPRA ZEISS FE-SEM*), before and after the treatments to observe the evolution of the surface and to evaluate the effects for each etching condition. EDS analysis was carried out in order to assess the presence of fluorine on the surface after the treatment and prior to the joining step.

Cusil-ABA® [20], a silver-copper-titanium system (63 wt% Ag, 35.25 wt% Cu, 1,75 wt% Ti), was selected for the brazing process since Ag-Cu-Ti alloys are known to be effective active brazes for joining SiC based materials [21–24]. The Cusil-ABA® braze is known to be an effective joining material for SiC substrate and several works on the evaluation of its properties are available [24–26]. Since the aim of this work was the assessment of the effectiveness of the plasma treatment, this brazing system was considered a good option because of its reliability. Three foils (80 μm thick) were used to manufacture each joint in order to mitigate the depletion of the brazing alloy from the joining area due to the creation of voids during the brazing process. The joining process was carried out at 850 °C for 15 min under a vacuum ( $10^{-5}$  mbar) to prevent oxidation. This brazing temperature was chosen from the Cusil-ABA® data sheet [20]. No pressure was applied.

Cross-sections of the CuSil-ABA® joined SiC/SiC composite were analyzed by means of SEM-EDS to investigate the quality of the composite/brazing alloy interface as well as for the presence of anchoring points between the SiC/SiC surface and the Cusil-ABA® alloy. EDS analysis was carried out to check the alloy composition in the joined area. A more detailed analysis of braze-SiC/SiC interactions was out of the scope of this work.

The apparent shear stress strength of the CuSil-ABA® joined SiC/SiC composite was calculated on 4 samples for each type of joint (pre-treated and untreated SiC/SiC samples) by means of a compressive single lap offset (SLO) test. The SLO test was carried out at room temperature using a universal testing machine (SINTEC D/10). A 50 kN load cell was

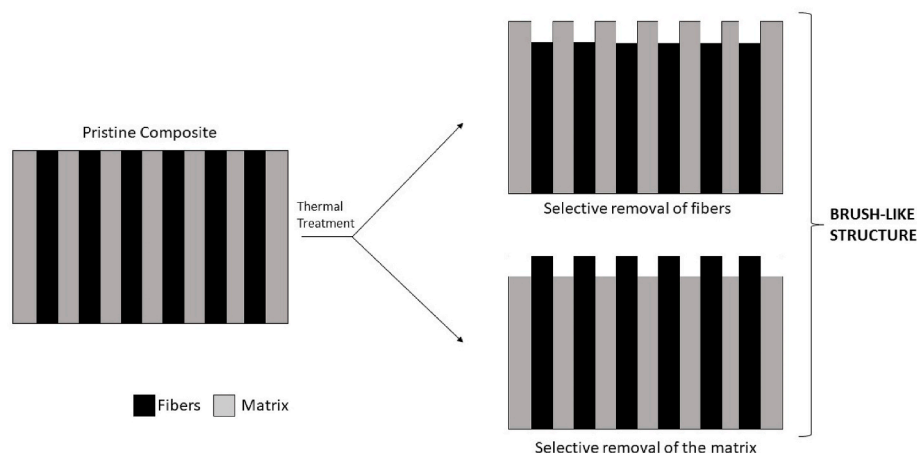


Fig. 1. Graphical sketch of the brush-like structures of composites formed by means of the selective removal of fibers or of the matrix, as reported in Ref. [10].

utilized and the crosshead speed was set at 1 mm/min. Further information on the SLO set-up is available in Ref. [27].

### 3. Results and discussion

Fig. 2a and Fig. 2b show comparisons between the SiC/SiC surfaces after each tested pre-treatment at different magnifications.

It is evident that the plasma treatment induced a preferential recession of the fibers on the flat polished surface that resulted in the formation of the desired brush-like structure, which has been hypothesized to be beneficial for enhancing the SiC/SiC joint strength (Fig. 1). The matrix also underwent etching, but the effect was less evident than for the fibers. As previously mentioned, even though both constituents of the composite were made of SiC, their slightly different properties, due to the manufacturing processes [28], provided a different response to etching, which was similar to what had already been observed for the STR treatment [10]. The matrix consists of CVD-deposited SiC, which is crystalline and highly stoichiometric. Instead, Tyranno S fibers (first generation type) are made of SiC nanocrystallites and residual carbon particles embedded in an amorphous SiOC matrix [29]. The overall composition of Tyranno S fibers is far from a stoichiometric Si/C ratio. The diversity in crystallinity and composition between fibers and matrix can justify the different etching resistance.

The difference in the duration and in the gas mixture of the plasma pre-treatments resulted in observable variations in the outcomes (Fig. 2b). After only 5 min (pre-treatment E, CF<sub>4</sub>, 5 min), it was possible to observe the first effects on the fibers and matrix. The fiber/matrix interface was no longer detectable and the fibers looked slightly corrugated. After 15 min (pre-treatments A and B), the appearance of the SiC/

SiC surface was similar to the one observed after the shorter pre-treatment (E). No remarkable differences were observed between treatments A and B, despite the difference in the gas mixture composition (Fig. 2b), while the formation of some pores in the matrix surrounding the fibers was observed for treatments B and E (see Fig. 2b and e).

After 30 min of exposure, the effects of the dry etching were more noticeable. The fibers were sharpened slightly by the deeper recession that occurred close to the fiber/matrix interface, while the matrix layer surrounding the fibers became thinner, as can be seen for pre-treatments C and D in Fig. 2a. Furthermore, the CF<sub>4</sub>-only pre-treatment (C) looked slightly more aggressive than the CF<sub>4</sub>/H<sub>2</sub> one (D). This observation was coherent with what has been reported over the years for the dry etching of SiC. Indeed, the etch rate of SiC strongly depends on the plasma conditions and especially on the composition of the etch gas. For SiC, the expected etch products are of the SiF<sub>x</sub> type. In general, the latter is produced by the interaction between fluorine radicals (also ions in minority form) and silicon atoms of the materials being etched. An explanation of why the treatment by pure CF<sub>4</sub> was found more aggressive than the treatment by the mixture of CF<sub>4</sub>/H<sub>2</sub> could rely on the role of H<sub>2</sub> gas. H<sub>2</sub> injection in plasma discharge produces hydrogen atoms (radicals), which can scavenge from discharge the fluorine radicals (generated by the fragmentation of the CF<sub>4</sub> molecules in the plasma phase) producing hydrofluoric acid, and effectively lowering the fluorine in the plasma, and consequently the number of interacting fluorine radicals with the SiC surface [30].

The EDS analysis of the plasma pre-treated surfaces measured a concentration of F that was very close to the detection limit of the technique (<0,1 %wt) [31], while no significant chemical changes

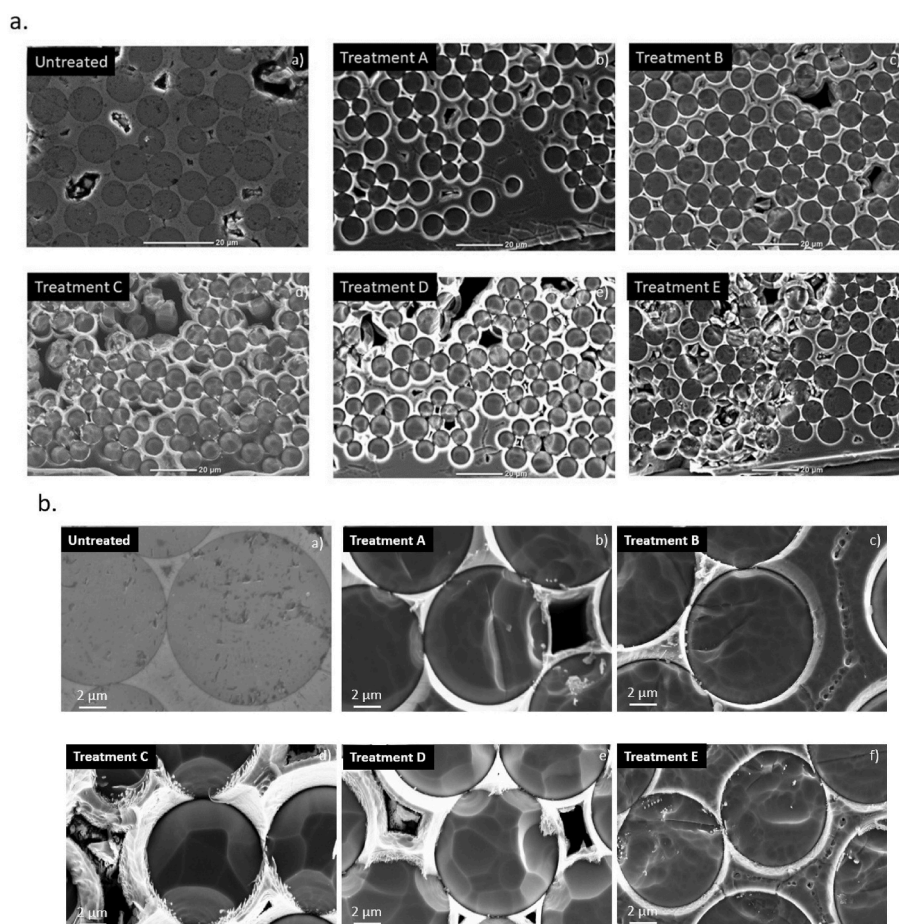


Fig. 2. SEM top view of the untreated and the pre-treated SiC/SiC: (a) low magnification; (b) high magnification.

occurred on the SiC/SiC surface. Therefore, the surface composition may be considered to have been unaffected by the treatment and changes in the joint properties should be attributed exclusively to the evolution of the surface structure.

According to these observations, pre-treatment C provided the most marked effect on the composite surface and was therefore chosen to study the effect of the plasma pre-treatment of SiC/SiC joints (Fig. 2). As a consequence, C-treated samples were joined using brazing alloy to compare them with joints manufactured with non-treated SiC/SiC.

Fig. 3 shows cross-sections of the Cusil-ABA® joined samples obtained with pre-treated and untreated SiC/SiC. The interface between Cusil-ABA® and SiC/SiC was sound and homogeneous along the whole length of the joint for both the treated and untreated samples, as expected for a brazing alloy belonging to the Ag–Cu–Ti system. The brazing alloy showed excellent wettability on both the fibers and matrix, and no porosity was detectable within the joint. EDS analysis confirmed that the composition of the alloy in the joint was in accordance with data from the literature [24–26]: a distribution of phases rich in Cu and in Ag, and Ti at the interface with SiC because of the formation of a reaction layer. The pores and voids close to the alloy/composite interface were completely filled by the braze, as a result of the SiC/SiC manufacturing process, as indicated by the black arrows in Fig. 3a and 3b. It is worth recalling that the joining process was carried out without the use of external pressure.

The penetration of the alloy enabled by the presence of voids on the as-received SiC/SiC provided some local anchoring points for the brazing alloy, where it infiltrated for tens of microns in the composite. This occurred also for treated SiC/SiC. Both treated and as-received SiC/SiC benefited from this effect due to their randomly distributed porosities. The plasma treatment provided an additional contribution because, as can be observed in Fig. 3b, the plasma-induced brush-like structure (indicated by the dotted blue boxes) is formed where fibers are perpendicular to the surface. This new surface texture increased the availability of anchoring points originated by erosion of fibers, regularly distributed along the joints, that were penetrated by the braze at the composite surface. The interlocking effect for plasma-etched composites was caused by the combination of two contributions: the random cavities (depth of tens microns) and the brush-like texture induced by the plasma treatment, regularly distributed along the surface (depths of a few microns). The enhanced infiltration in the brush-like textured regions is clearly visible in Fig. 4b for the high magnification.

The SEM observation revealed that the alloy was able to perfectly wet the SiC/SiC surfaces and fill all the holes and gaps introduced during the plasma treatment. The brush-like interface (perpendicular to the joining seam) was several microns long; it even reached 50  $\mu\text{m}$  in some points, but it was difficult to distinguish the effect of the plasma etching from the intrinsic porosity of the composite.

Moreover, the good spreading and the same morphology shown by the brazing alloys on both the untreated and treated SiC/SiC surfaces demonstrated that the plasma modification of the surface topography did not affect the good interfacial behavior of the brazing alloy.

Since the microstructural analysis of the joined specimens corroborated the increase in the interlocking points between the alloy and the composite after the plasma pre-treatment, mechanical tests were carried out to evaluate the effect of the dry etching process on the apparent shear strength of the SiC/SiC joints.

All the joined samples, obtained with both the untreated and the pre-treated SiC/SiC, showed mixed cohesive failure, thus confirming the good quality of the joints produced with the chosen brazing alloy (Fig. 5a–b). SEM inspection of the surface of the cross-section of the fracture surfaces (Fig. 5c–d) provided additional confirmation of the enhanced interlocking effect due to the additional brush-like structure obtained with the plasma pre-treatment. Fig. 5e–f shows a much larger amount of the brazing alloy on the fracture surface of plasma-treated samples; the higher penetration of the brazing alloy into the etched composites led to higher adhesion at the braze/composite interface and the failure propagation mainly within the alloy. At higher magnification (Fig. 6), it is possible to observe that the untreated fibers were completely clean after failure (Fig. 6a), thus indicating an adhesive failure mode at the braze/fiber interface. Besides, the plasma-treated fibers are covered by the brazing alloy (Fig. 6b and related EDS analysis), thus suggesting a cohesive failure at the fiber/alloy interface for plasma-treated samples. Furthermore, it is worth noting that the joint strength measured for the untreated joined SiC/SiC was  $76 \pm 27$  MPa, while that of the brush-like structured samples was  $118 \pm 33$  MPa. No remarkable differences were observed for the load-displacement curves, except for the peak load. The coefficient of variance (standard deviation/mean value) was approximately 0.25 for the plasma-etched samples and 0.31 for the untreated samples. Thus, it was similar for both cases, which could be related to the joining process, influenced by the operator. Bonding the two parts along their thickness (only 4.5 mm) is a rather complicated operation and this may slightly affect the distribution of values for the recorded joint strengths, but not the trend.

In short, the additional interlocking effect due to the brush-like SiC/SiC surface effectively obtained with the selected plasma pre-treatment on SiC/SiC (pre-treatment C in Table 1) led to a 55% increase in the apparent shear strength of the joined samples. The interlocking mechanism promoted by the plasma-modified surface complemented the adhesion properties of the Ag–Cu–Ti brazing alloy, thus providing a significant increase in the mechanical performances of the joined SiC/SiC composites.

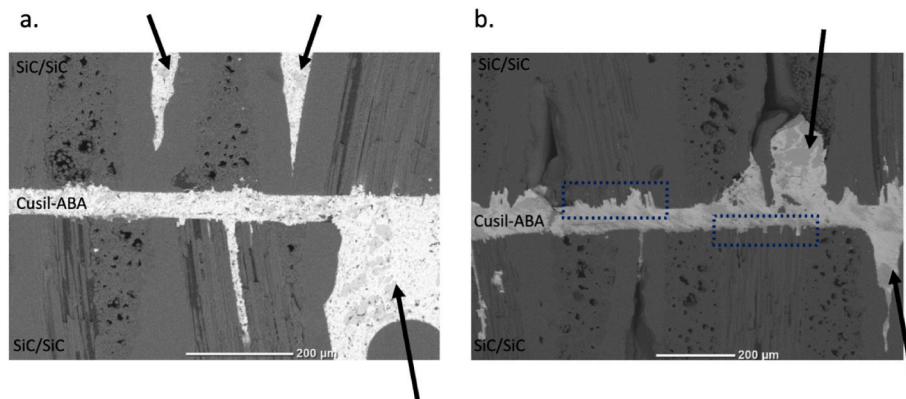


Fig. 3. SEM micrographs of the cross-section of the untreated (a) and plasma pre-treated SiC/SiC (b) joined with Cusil-ABA®. The dashed blue boxes indicate the induced brush-like structures, while the black arrows denote the intrinsic cavities of the as-received composites.

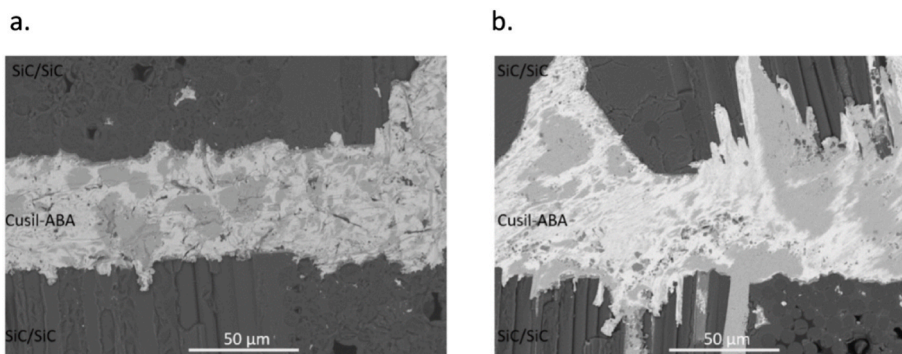


Fig. 4. SEM micrographs of the cross-section of the untreated (a) and plasma pre-treated SiC/SiC (b) joined with Cusil-ABA® at a high magnification.

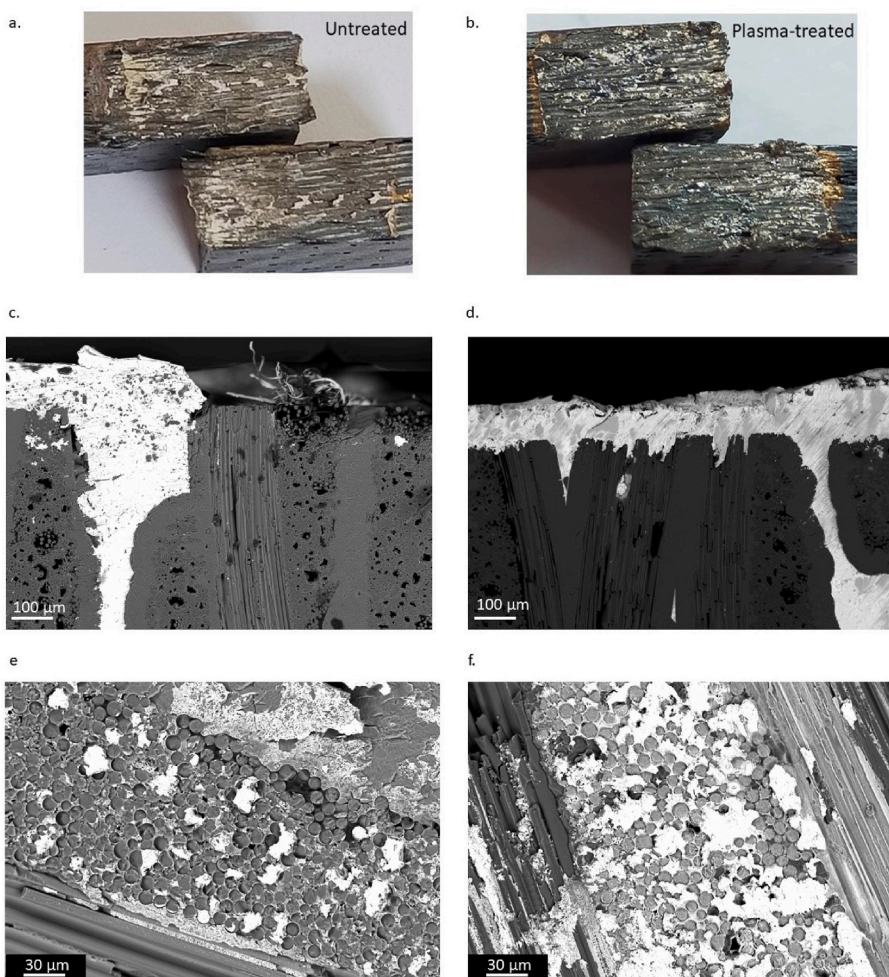


Fig. 5. Fracture surfaces of the joined SiC/SiC samples: untreated (a) and plasma-treated (b). Cross-sectional and top views taken by SEM of the fracture surface for untreated (c,e) and plasma-treated (d,f).

#### 4. Conclusions and future works

The work presented here has investigated and proved the effectiveness of a fluorine-based low-pressure plasma in engineering the surface of SiC/SiC composites by providing a brush-like texture through the selective removal of fibers. The most promising results were obtained using CF<sub>4</sub>, 20 sscm, with 200 W for 30 min. SiC/SiC joints were manufactured using an Ag–Cu–Ti alloy and it has been demonstrated that the braze infiltration was enhanced by the plasma-induced brush-like structure, and the expected positive effect was confirmed by the

mechanical testing results. Indeed, 55% higher joint strengths were recorded for the plasma-treated SiC/SiC than for the untreated composites.

Future works will be focused on optimizing the process parameters, on evaluating the opportunity for industrialization of the treatment and on the study of the effectiveness of the plasma pre-treatments on different ceramic matrix composites, such as C/SiC and C/C.

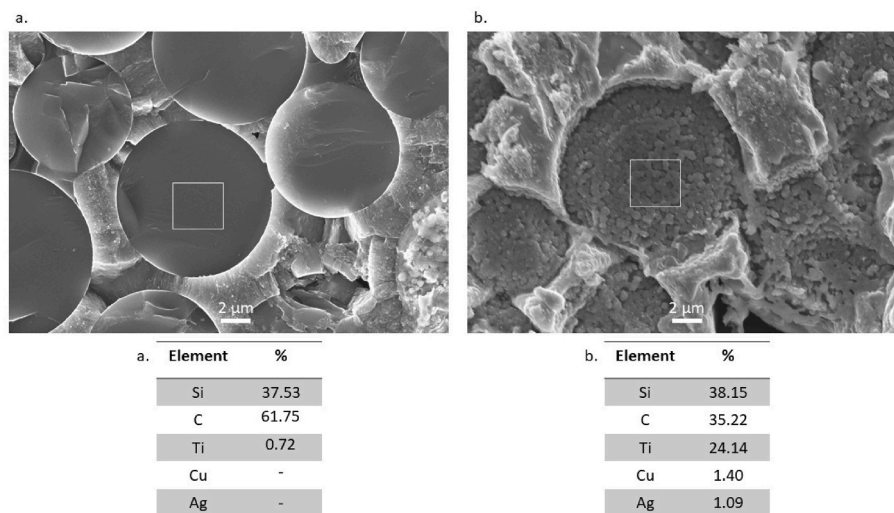


Fig. 6. EDS analysis of the untreated (a) and plasma-treated (b) SiC fibers on the fracture surface. Analyzed regions are indicated by white boxes.

Table 1

Etching conditions tested for SiC/SiC.

Treatment	Gas	Flow	Power	Exposure Time
A	CF <sub>4</sub>	20 sscm	200 W	15 min
B	CF <sub>4</sub> /H <sub>2</sub>	18/2 sscm	200 W	15 min
C	CF <sub>4</sub>	20 sscm	200 W	30 min
D	CF <sub>4</sub> /H <sub>2</sub>	18/2 sscm	200 W	30 min
E	CF <sub>4</sub>	20 sscm	200 W	5 min

## 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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