Surface modification of SiC to improve joint strength via a Corona plasma treatment

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Abstract:

The high mechanical and chemical properties of SiC make it difficult to texture and modify its surface using such conventional methods as mechanical machining and wet etching. Among possible alternative strategies, Atmospheric Pressure Plasmas (APPs) could be used, cutting cost and time, but much still has to be understood about their feasibility for the surface treatment of ceramic materials.

In this work, the effectiveness of a commercial corona discharge system in modifying the surface of SiC has been evaluated, focusing on its positive effect on the joint strength of adhesively bonded plasma-treated SiC.

The objective of the study has been to observe the surface changes, in terms of chemical composition and texture, that take place as a result of exposure to corona plasma and to compare the obtained results with previous studies on laser and low-pressure plasma textured SiC samples. These very first results, derived from characterization and mechanical testing, suggest that this approach could be a promising alternative.

Keywords: (D) SiC; (B) Surfaces; (A) Joining.

Contents

1. Introduction

Silicon carbide (SiC) is one of the most well-known technical ceramics because of its particular properties, which make it interesting, as a bulk or as a composite material, for a wide variety of applications, in several fields, ranging from aerospace to electronics [1–6]. SiC has been classified as a semiconductor material with a high degree of hardness, a high elastic modulus, high chemical resistance, high thermal stability, high thermal conductivity, and high resistance to thermal loadings, but a low coefficient of thermal expansion (CTE). It is often integrated with other materials or joined to itself to manufacture final components; however, this is not a trivial operation and many different techniques have been developed [7,8] according to the targeted purpose and service conditions. Among the possible solutions that can be proposed to improve the performances of joints for technical ceramics, one consists in promoting interlocking between the joining material and SiC, through the formation of anchoring points on the surface.

Several approaches can be used to obtain the desired interlocked structure. One promising approach relies on the use of a laser to provide a texturing technique [9], that overcomes the typical issues concerning surface mechanical machining – such as tool wear and crack formation. Laser texturing can also be used to replace the chemical etching of SiC, which is a challenging process because of its chemical inertness. Ultra-short-pulsed lasers are particularly effective since they enable the formation of very narrow and precise patterns on the ceramic surface. However, this is a costly approach, and other techniques might therefore be of interest.

Plasma-based etching techniques are known to be effective for the modification of ceramic surfaces, as can be seen from their use in the manufacturing of electronic devices [10,11], where fluorine-based plasmas are typically used. These strategies provide accurate control on the process but impose strict limitations on the maximum size and number of the components that can be treated because of the dimensions of the vacuum chamber. Furthermore, the equipment is usually complex and a high-level of maintenance is required [12]. Therefore, they are not suitable for continuous operations, and are limited to the treatment of single batches. However, by selecting the appropriate conditions, it is possible to work on a large area, that is, about 1 square meter, thereby improving adhesion onto advanced ceramic materials, such as SiC and Si3N4, as already demonstrated by Casalegno et al [13].

According to the treatment-material pairing, both laser and plasma surface techniques can provide a physical modification, a chemical modification or a combination of both. The latter was observed for the laser treatment studied by Suess et al. [14], where a cauliflower-structured silica layer was formed, replacing the original SiC surface. In that work, the new surface provided different chemical and tribological properties than the original.

As fore-mentioned other surface modification strategies are wet etching [15], which it is not particularly effective because of the inertness of SiC, and mechanical machining [16]. The latter is less reliable for ceramic materials because of the risk of inducing detrimental cracks that can propagate during the life of the component.

Atmospheric-pressure plasmas (APPs)[17] are of interest when large samples or large batches need to be produced, since they provide several advantages over low-pressure plasmas. For example, they enable continuous operations to be made, since they do not involve any restrictions due to the need of a vacuum system and they can be used to treat large surface areas. Corona discharge [18] systems are part of the family of APPs. They are used as a manufacturing technique that is employed in the plastic industry for surface activation and the removal of contaminants [19,20], e.g. as a pre-treatment prior to the printing or bonding operations of polymeric components. Several corona plasma reactor geometries are available, and custom-made systems designed for specific requirements are quite common. They can be found in the product portfolios of manufacturers that are available online.

Corona discharge occurs when a gas is ionized by an external electric field, and it results in the appearance of filamentary plasma that stretch from the electrode. The induced plasma is not in thermal equilibrium, since electrons have a higher temperature than ions, which remain close to room temperature.

The utilization of corona plasma as a surface modifier to improve the bond strength of feldspar porcelain and to generate a high proton concentration on soda-lime glass was reported by Komagata et al. [21] and Ikeda et al. [22], respectively. To the best of our knowledge, no work has been published concerning the use of corona plasma as a surface modification treatment for advanced ceramic materials.

This manuscript presents the results obtained when applying a corona plasma to SiC plates in order to induce a surface modification. The original surface underwent a change of texture and composition as a result of the treatment, which led to an increase in the strength of adhesive bonded SiC. The result are discussed and compared with those reported in [14] and [13].

2. Materials and methods

The substrates selected for this activity were 25 mm x 25 mm x 4 mm *Boostec SiC* [23] tiles. Samples were surface treated using a corona-plasma generator (*Tantec SpotTEC* model [24]), equipped with one treatment head containing two electrodes. Air was fluxed between the electrodes to convey the plasma filaments onto the surface that had to be treated. Settings of the corona generator are reported in Table 1.

The preliminary part of the activity was carried out for 10 and 2 minutes, keeping the specimen fixed under the corona plasma head, without any motion. The gap between the electrodes and the surface of the samples was approximately 5 mm. Another processing set-up was then used, this time continuously moving the treatment head over the surface at a distance of around 5 mm for 5 minutes in order to improve the uniformity of the surface treatment. Samples were prepared for joint shear strength tests adopting the latter conditions (5 mm distance, 5-minute time process)

A Scanning Electron Microscope was used to analyze the microstructure of the surface before and after the treatment. EDS (Energy-dispersive X-ray Spectroscopy) analysis was performed to investigate the composition of the surface. The equipment used for this aim was a *Benchtop Scanning electron microscopy (JEOL*) and a *MERLIN ZEISS FE-SEM* equipped with an EDS analyzer.

An investigation on the effect induced by the treatment on surface roughness was conducted using a confocal microscope (*ZEISS LSM 900*, *ZEN 3.1 software*, Germany) with a 20x magnification. The average values for roughness parameters were then calculated according to ISO 25178 for three different samples for each type (treated and untreated). After the surface treatment, the parts were joined together using *Hysol EA 9321 glue* [25] as the bonding material. This is a two-component epoxy resin reinforced with dispersed aluminum particles. The adhesive was chosen in order to be able to compare the obtained results with those of previous works [13,14].

The adhesive was manually deposited onto each SiC surface and then a sandwich-like sample was manufactured. After this step, a curing treatment was carried out at 85°C for 1 h in a drying oven (*Heraeus*). During the curing step, samples were kept aligned using steel plates. A 6 KPa pressure was applied on the joining area while curing.

At least 3 joined treated and untreated samples were tested by means of the single lap offset (SLO) test under compression in order to measure the apparent shear strength. A sketch of the SLO configuration is provided in [26]. The mechanical tests were carried out at room temperature by means of a *universal testing machine SINTEC D/10*, equipped with a 50 kN load cell. The crosshead speed was set at 1 mm/min.

3. Results and discussion

3.1. Surface treatment – Preliminary tests

Remarkable changes in the appearance of the SiC surface were noticed after the 10-min exposure to corona plasma. Large, pitted areas, in contrast with the surrounding glossy SiC, appeared on the surface as a result of the discharge on the surface of the tile, above all near the edges. These changes were visible to the naked eye.

One of the edges of the SiC tile is shown in Fig.1 before (a) and after (b) the Corona plasma treatment, as observed by means of an electron microscope. A locally well-distributed white coating appeared on the surface of the sample after 10 minutes of treatment, with the electrodes approximately 5 mm from the surface. The brighter appearance of the surface after corona exposure may be attributed to an accumulation of the charge during the SEM analysis (the SEM images were acquired using the same brightness and contrast parameters). This change in the electrical properties suggested the formation of an oxide. The result of the EDS analysis carried out near the edge is shown in Fig. 2; only Si, O and C were found in the analyzed regions (Fig.2.b). The amount of each element was coherent with the presence of a silica (SiO_2) layer on the SiC surface and was within the expected accuracy of a few percent for EDS quantification. The presence of large amounts of oxygen and silicon, together with the higher tendency of charge accumulation observed by means of electron microcopy, indicate the formation of an oxide layer on the surface. However, carbon is still present, except in site 2, probably due to the contribution of the underlying and surrounding unreacted silicon carbide and/or the contamination of the surface during handling.

During the treatment, it was observed that the plasma filaments were not distributed homogeneously over the surface and were discharged in preferential locations. Indeed, they showed a tendency to accumulate at the edges of the tile. A possible explanation for this result is the natural tendency of the charge to accumulate around sharper regions. Because of this behavior of the discharge, the surface modification was quite inhomogeneous and there were large regions, in particular the central one, that remained untreated.

Furthermore, the corona treatment carried out for 2 minutes did not lead to any significant or perceivable differences, thus suggesting that the exposure time might not be a critical variable for the formation of oxide and that the oxide might form in a short time because of the energy released by the discharge. This phenomenon may be attributed to the energy transferred to the SiC surface by the corona discharge filaments when they reach the surface. Indeed, areas were identified where dark pits of oxide were visible to the naked eye and they seemed to coincide with the impact sites of the corona streamers. Furthermore, it is worth noticing that the corona filament path was not confined to between the two electrodes, but diverged and formed two different plasma streams that originated at the electrodes and ended up on the SiC surface (Fig. 3), thus suggesting an electrical conductance.

As a result of the previous observation, the electrodes were moved randomly over the surface for 5 minutes to improve the homogeneity of the treatment. Introducing random motion of the electrode head over the sample mitigated the accumulation tendency at the edges and resulted in a homogenous distribution of the silica-grown region over the entire surface. The better homogeneity was first observed by naked eye, paying attention to the color variation on the surface and then confirmed by investigation at SEM.

The silica structures grown on silicon carbide showed a cauliflower structure (Fig. 4) that suggested a possible improvement in the joint strength, because of a better anchoring of the joining material through infiltration of the glue. A similar structure was observed for a silica layer obtained on SiC by means of a laser treatment, as reported in [14].

Moreover, an adhesive, and in general any joining material with low viscosity, can penetrate this structure and create a mechanical anchoring system that can be expected to increase the mechanical performance of a joint.

The oxidizing effect of corona is well-known and, apart from being exploited for cleaning and polymer surface activation, it has also been reported for a silicon wafer [27]. However, it is difficult to find a reliable explanation for the reaction of SiC to oxygen, which has only been observed for much higher energy beams (e.g. laser) or higher temperatures. At this stage, the SiC corona-induced oxidation may be explained by considering a punctual huge increase in temperature provided by the plasma filaments, but a rigorous and focused investigation is needed to fully understand this mechanism. The growth of silicon oxide has been reported in dry air in a temperature range between 800°C and 1400°C, but oxygen plasma already proved to promote oxidation at room temperature [28]. Indeed, it should be taken into consideration that the species generated by the ionization of air induced by the corona discharge, such as oxygen ions, are highly reactive[29]. These reactive ions may promote oxidation at temperatures much lower than 800°C. Therefore, the combination of plasma filaments that are high in energy and the presence of highly reactive ions might therefore explain the oxidation of the SiC.

An open porosity of the silica structure can clearly be observed in Figure 5; the pores are homogenously distributed in both orientations, that is, parallel and perpendicular to the surface. Such a structure leads to an extensive increase in the surface area.

Measurements obtained from the confocal microscopy provided information on the surface roughness. The arithmetical mean height (Sa) increased after the treatment, from 0.045 ± 0.001 µm to 0.120 ± 0.008 µm, together with maximum height (Sz), (from 1.31 ± 0.185 µm to 3.82 ± 0.56 µm). These results confirmed the roughening effect of the corona treatment.

Furthermore, the Sdr value, the ratio between the calculated area and the projected area, was observed to be around 1% for the untreated surface and approximately 6% after the treatment, confirming the increase in surface area provided by the new texture.

The cauliflower-structured silica layer formed by the corona discharge system was found to be stable, since no significant changes were observed under the electron microscope or EDS, even one full year after the treatment. The considered modified non-joined samples had been stored in sealed laboratory bags.

Single-lap offset shear strength tests were carried out to evaluate whether the formation of the corona-induced SiO_2 was effective in increasing the joint strength.

3.2. Joint strength tests

The Hysol adhesive bonded SiC (treated and untreated) samples were tested and the fractured surfaces are reported in Fig.6.

The average joint strength recorded for the corona-treated joined components was approximately 68.8 ±2.3 MPa, while the values registered for the untreated samples was about 61.5 ± 5 MPa. Therefore, a more than 10% increase in the joint strength was observed after the corona plasma treatment of the SiC surface, compared to the untreated samples. However, the most interesting results concerned the joint fracture. In this specific case, it was observed that all the corona-treated samples underwent cohesive failure, while the untreated samples underwent adhesive failure. A comparison between two samples, one treated and one untreated, is shown in Fig.6. The difference in the failure mode can clearly be seen by the naked eye.

One of the goals of adopting adhesive joining strategies is to maximize the probability of the fracture occurring within the adhesive. Cohesive fracturing is usually a sign of a good quality of the joining process, because it suggests that the mechanical strength at the joining interface is higher than that of the joining material itself [13].

Since the adhesive failed cohesively over the entire joining area of the treated samples, the surface was modified quite uniformly by the corona treatment. The enhanced mechanical behavior of joints after the corona treatment can be the result of the induced texture together with the change in composition. Harris et al.[30] observed that the formation of a laser-induced silica layer on SiC provided a better chemical bonding with the epoxy adhesive due to the presence of hydroxyl groups. Therefore, the corona-induced cauliflower silica layer obtained in this work can play a significant role in providing a stronger mechanical and chemical bonding.

Comparing the obtained values with those collected in [14] (34.8 ± 3.4 MPa) and in [13] (44.3 ± 2.4 MPa), for laser and low-pressure plasma treated SiC joined with the same adhesive, it is possible to draw two conclusions.

First, the joint strength values in the present study are higher than those found in the previous studies, for both the untreated and corona-treated samples. This difference may be attributed to the different curing treatments: 85 °C for 1h in the present work compared to 7 days at room temperature in the other two studies. This is a remarkable result because, besides providing higher joint strength, this curing treatment is much shorter (1 h compared to 7 day).

Second, the corona plasma treatment here resulted in cohesive failure, just like the low-pressure plasma treatment proposed in [13], and performed better than pulsed laser irradiated SiC [14], which led to adhesive failure. This result is promising since it reinforces the hypothesis of the validity of a corona plasma treatment as an effective and low-cost treatment that can be used prior to joining.

A comparison of the different results obtained for the Hysol-joined SiC samples after the corona discharge plasma treatment, the laser treatment, and low-pressure plasma treatment, together with the recorded untreated SiC joint values, is provided in Table 2.

4. Conclusions

This work has been aimed at evaluating the feasibility of a commercial corona plasma system as an effective joint strength enhancer for adhesive bonded SiC. The corona treatment was found to be effective in modifying the SiC surface, where it formed a silica cauliflower structure that can promote infiltration of the joining material and the formation of a stronger bonding with the adhesive. Accordingly. the surface modification was both chemical and physical.

Mechanical tests conducted on adhesive bonded SiC samples confirmed the improvement resulting from the surface treatment. Indeed, they showed a difference, in terms of fracture type, between the corona-treated and untreated SiC samples that were cohesive and adhesive, respectively. Furthermore, the treated samples failed for higher stress values and the cohesive failure occurred over the entire surface, thus suggesting the treatment produced a uniform effect. Therefore, these results would seem to confirm that the corona discharge treatment is effective in improving the mechanical performances of joints.

It is noteworthy that the curing treatment proposed in this paper (1h, 85°C) for the epoxy resin resulted in a remarkable increase in the bonding strength compared with previous works. There is still a need to perform more research on the use of corona discharging for the surface preparation of silicon carbide prior to joining since no articles on the topic have been published so far. Future work will be devoted to better understanding the mechanism behind the surface modification induced by corona and to investigate the effects of corona discharging on other advanced ceramics in order to further explore its potential for industrial applications.

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Figure captions:

Fig. 1: One of the edges of a Boostec SiC tile before (a) and after 10 minutes of Corona plasma exposure (b).

Fig. 2: EDS analysis on the near-edge region of SiC before (a) and after the corona treatment (b).

Fig. 3: Plasma filaments discharging from electrodes to the SiC surface.

Fig. 4: Lateral view of the silica-grown layer on the SiC surface.

Fig. 5: Top view of the corona-induced silica layer visible after SiC exposure to the corona discharge system.

Fig. 6: Comparison of the fracture surfaces. Adhesive failure for the untreated Hysol-joined SiC sample (left) and cohesive failure for the corona-treated Hysol-joined SiC sample (right). The adhesive is gray in color.

Table captions:

Table 1: Settings of corona plasma generator.

Table 2: Joint strength and failure modes for different surface treatments.

Fig.1: One of the edges of a Boostec SiC tile before (a) and after 10 minutes of Corona plasma exposure (b).

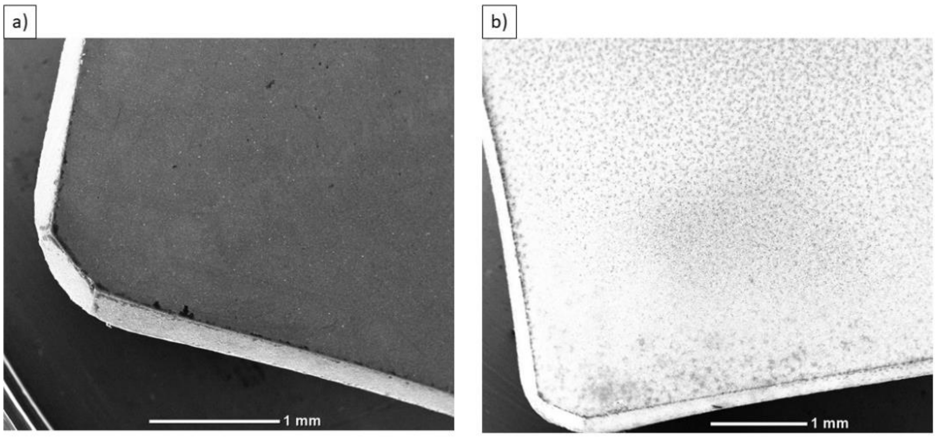
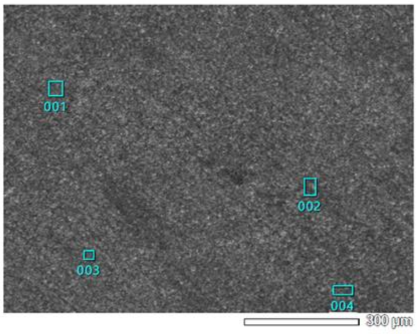


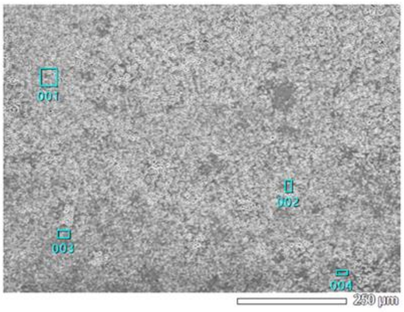
Fig. 2(color): EDS analysis on the near-edge region of SiC before (a) and after the corona treatment (b).

a.



Site 1			Site 2		
Si (%at)	C (%at)	O (%at)	Si (%at)	C (%at)	O (%at)
61.66	35.93	2.41	48.44	48.02	3.55
Site 3			Site 4		
Si (%at)	C (%at)	O (%at)	Si (%at)	C (%at)	O (%at)
55.25	40.50	4.25	48.63	47.63	3.74

b.



Site 1			Site 2		
Si (%at)	C (%at)	O (%at)	Si (%at)	C (%at)	O (%at)
34.32	14.46	51.22	43	-	57
Site 3			Site 4		
Si (%at)	C (%at)	O (%at)	Si (%at)	C (%at)	O (%at)
38.01	19.77	42.22	37.90	17.34	44.76

Fig. 3(color): Plasma filaments discharging from electrodes to the SiC surface.

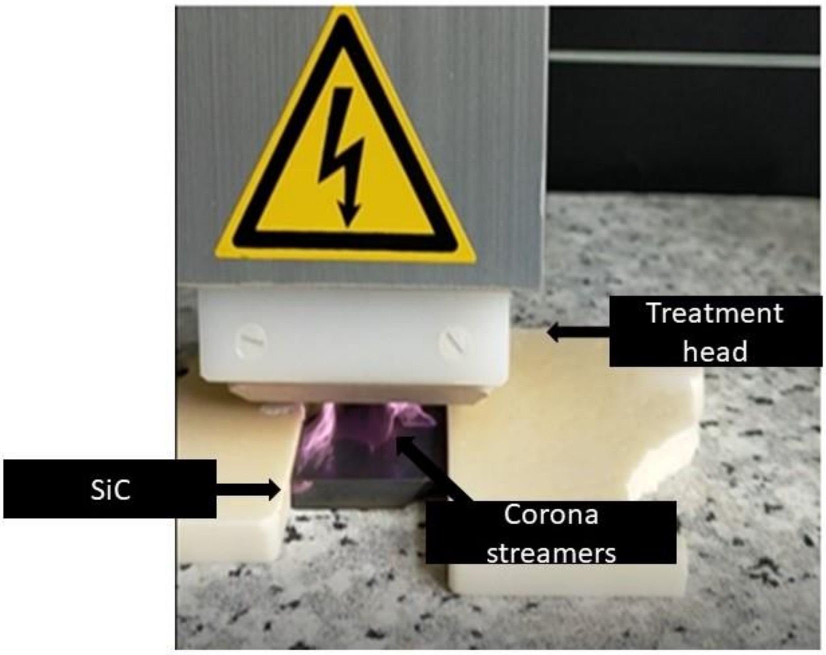


Fig. 4: Lateral view of the silica-grown layer on the SiC surface.

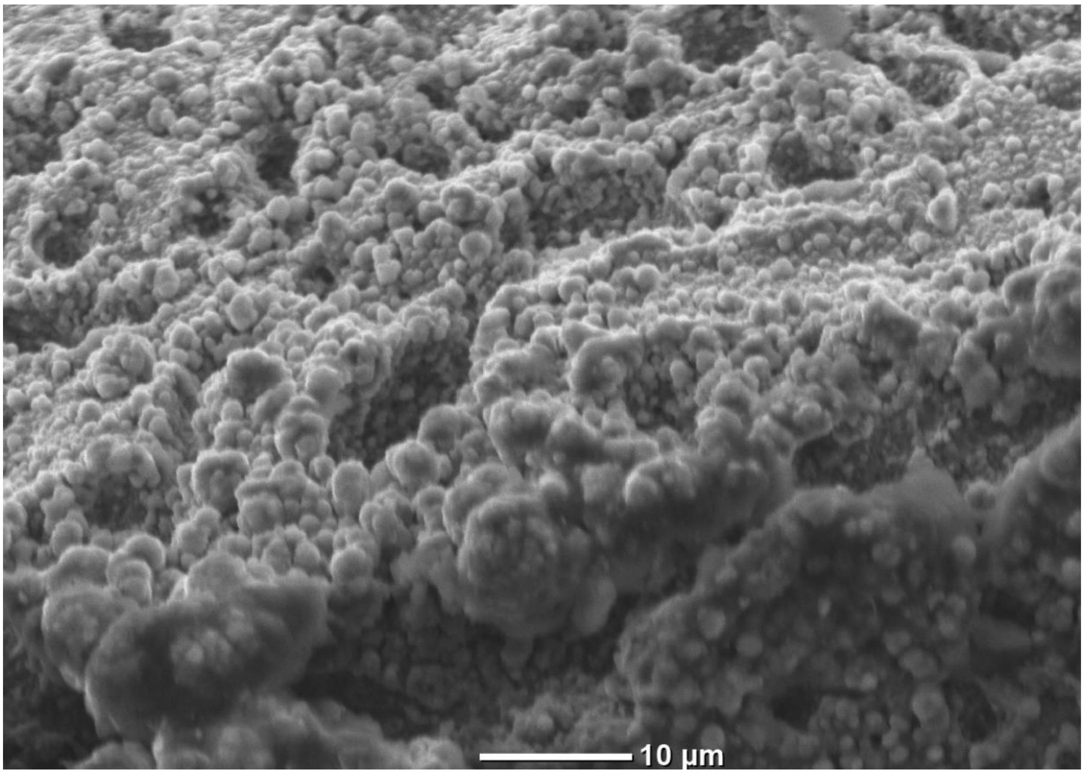


Fig. 5: Top view of the corona-induced silica layer visible after SiC exposure to the corona discharge system.

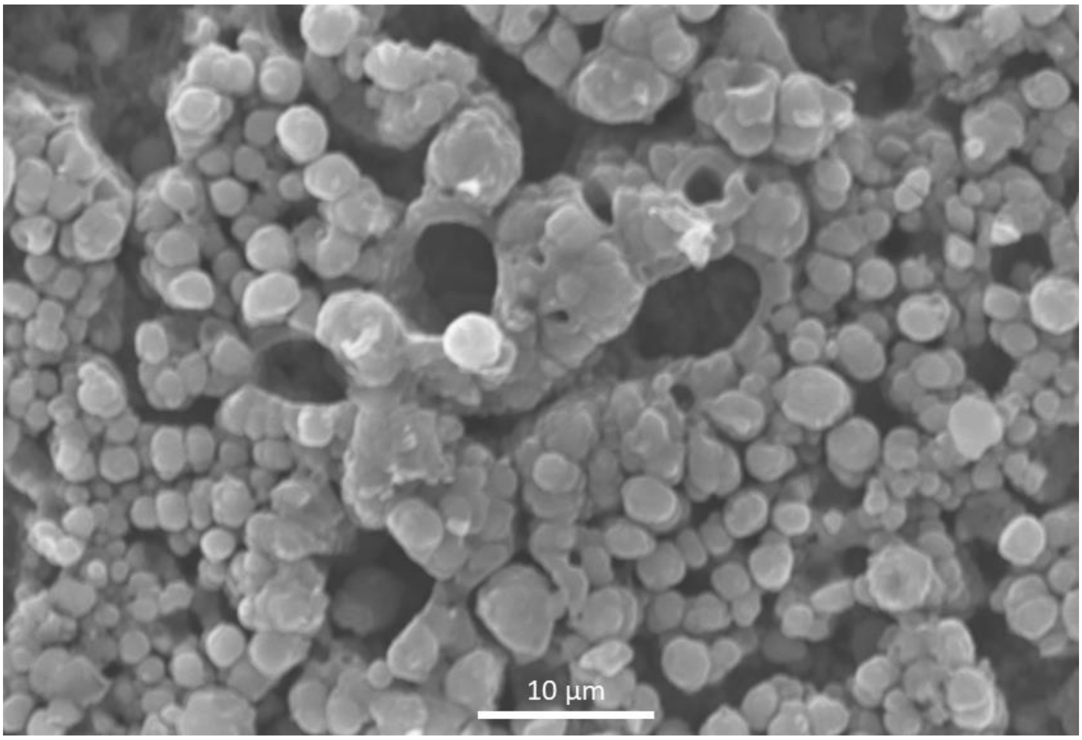


Fig. 6 (color): Comparison of the fracture surfaces. Adhesive failure for the untreated Hysol-joined SiC sample (left) and cohesive failure for the corona-treated Hysol-joined SiC sample (right). The adhesive is gray in color.

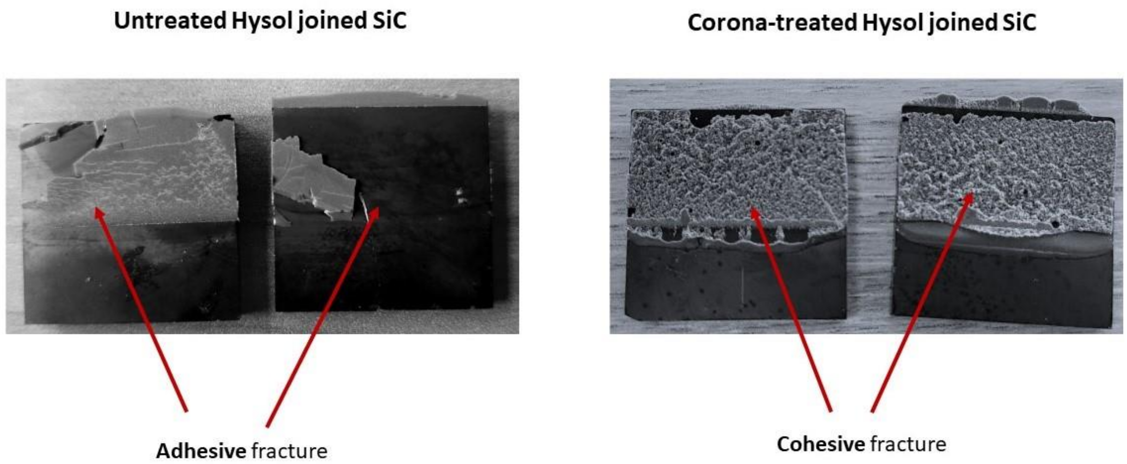


Table 2: Settings of corona plasma generator.

Main Voltage/ Frequency	Output power/ Output voltage	Output frequency	Power consumption
230 V, 50 Hz	550 W / 2 x 6,5 kV	25 kHz	600 VA (max. value)

Table 2: Joint strength and failure modes for different surface treatments.

	Corona plasma treatment	Pulsed laser irradiation [14]	Low-pressure plasma treatment [13]	Untreated	Untreated [14]
Apparent Shear Strength [MPa]	68.8 ±2.3	34.8 ± 3.4	44.3 ± 2.4	61.5 ± 5	41.6 ± 0.9
Failure mode	Cohesive	Adhesive	Cohesive	Adhesive	Adhesive
Curing treatment	85°C, 1 h	R.T, 7 days	R.T, 7 days	85°C, 1 h	R.T, 7 days