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TEM and zeta potential titration as suitable techniques for investigating the joining of modified ceramic surfaces

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ABSTRACT

Transmission Electron Microscopy (TEM-EDS) and Zeta potential titration of ceramic surfaces allowed researchers to go inside their adhesion mechanism when joined with an adhesive. The case study concerned the plasma Corona treatment to improve the joint strength between SiC surfaces joined with an epoxy adhesive. The formidable mechanical and chemical properties inherent to silicon carbide pose challenges in applying conventional methods like mechanical machining and wet etching for surface texturing. Exploring alternative strategies, plasma Corona treatment which is an Atmospheric Pressure Plasmas (APPs) process emerges as a potential solution. The mechanism of chemical interaction and mechanical interlocking between plasma-treated surfaces and the epoxy adhesive used for the joining was never explored in detail and proven. The presence of layers with different crystallographic natures and chemical compositions was assessed by TEM-EDS: an amorphous silica layer was produced by the corona activation treatment and it created a mechanical anchoring system when penetrated by the adhesive. The zeta potential titration as a function of pH evidenced that untreated SiC had a surface with amphoteric functional groups, while the Corona treatment induced the formation of surface functional groups (OH) with a strong acidic behavior on SiC. The investigated epoxy adhesive exposed a prevalence of basic amino groups. Zeta potential titration curves highlighted the chemical interaction between the treated SiC surfaces and the epoxy adhesive: the large electrostatic attraction between the OH groups of the treated SiC and the amino groups of the epoxy adhesive had a role in the mechanical strength of the joining. TEM and Zeta potential titration can be proposed as suitable techniques to evaluate the effectiveness of the plasma corona discharge system in modifying the surface of SiC and to solidify its standing in comparison to other established surface modification techniques to improve SiC-based joints.

1. Introduction

Indirect joining is one of the most popular strategies for joining ceramic materials, but it comes with a major issue. The joining material has different properties compared to the substrates and interfaces are formed that act as weak points when a load is applied to the joined component or if it is subjected to thermal stresses. Therefore, besides the need to choose a suitable joining material, it is critical to strengthen the joint interfaces as much as possible. This can be done through surface treatments that modify the surface chemistry, its roughness, or both of them.

In particular, silicon carbide (SiC) is attracting a great deal of interest in advanced technological applications, because of its outstanding thermomechanical properties and lightweight. Nevertheless, the application of SiC-based materials depends on the ability to join them, in many cases, because manufacturing these materials as large components, with complicated shapes, is extremely difficult and expensive. A critical issue for the broader use of SiC is thus the development of reliable and user-friendly joining methods to assemble large components in complex structures [1–5].

Several joining methods, such as mechanical fastening, brazing, diffusion bonding, and the use of adhesives have been studied and developed to join SiC materials. In this context, adhesive bonding for SiC is increasing for similar and dissimilar joints; it offers many advantages, compared to mechanical connection (e.g. more uniform stress distribution, reduced weight, and cost), but requires effective processing

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methodologies. Surface pre-treatments are often used to functionalize a surface before gluing, and they may result in bonds that show an improvement in mechanical strength on joined parts [6]. Some authors [7,8] report on the feasibility of plasma or laser technology as a SiC surface pretreatment before bonding. The surface modification treatment relies on the following effects, or a combination of them: surface cleaning, surface topography modification, and activation of chemical groups [9–13].

Characterization of the surface before and after the treatment is a key point to understanding the results obtained downstream of the joining process. Firstly, surface treatments can affect the crystallographic phases of the surface at different depths, and they must be carefully investigated layer by layer. TEM equipped with EDS is a useful technique in this respect. Changes in roughness can be easily detected via profilometry of the modified surface and Scanning Electron Microscopy (SEM) observation of the cross-sections of the joined samples, but only TEM can image penetration of the adhesive at the joined interface. Furthermore, evaluation of the chemical affinity between the surface, pristine or treated, and the adhesive can be very tricky, requiring multiple characterization techniques from Energy Dispersive spectroscopy (EDS) to X-ray Photoelectron Spectroscopy (XPS). In particular, EDS can be used for a quick analysis of the modified surfaces, but it provides information limited to elemental analysis. EDS has a micrometric penetration depth and modifications limited to the outermost surface layer can be not detected. XPS is a chemical analysis limited to nanometric penetration depth and gives information about the functional groups but not about their chemical reactivity (strong or weak acids or bases, for instance). What is useful for this specific aim is a chemical analysis able to provide any insights about the functional groups exposed by the outermost layer of a surface and their chemical reactivity.

Zeta potential [14] is widely used to measure the surface's charge of nanoparticles and their dispersions' stability. Over the last decades, this analysis has been extended to the study of treated surfaces for several applications [15] like biomaterials, semiconductors, and polymer activation, to determine their characteristic functional groups and even their chemical reactivity such as acidity or basicity [16,17]. However, it has not yet been used in the characterization of surfaces for joining applications as well as a potential tool for the prediction of joining effectiveness.

In a previous work [9], the effectiveness of a Corona plasma treatment as a pre-joining step has been assessed for silicon carbide (SiC) substrates. The treatment induced a chemical and textural change on the substrates. The change in texture was remarkable: the as-sintered irregular surface evolved in a cauliflower-like textured silica layer after being plasma treated. The new Corona-induced texture proved to be beneficial when bonded with the epoxy adhesive known as Hysol EA9391 [18,19].

Therefore, such plasma treatment has proved to be effective in increasing adhesion at the ceramic/adhesive interface but the mechanism of adhesion is not completely known.

Moreover, since the plasma treatment is carried out at atmospheric pressure, it offers the possibility of treating large areas with a certain degree of automation and can also easily be used for complex-shaped geometries, being viable to be employed by manufacturing industries. In the present research, zeta potential measurements, in combination with TEM, were applied to characterize SiC surfaces and adhesive to understand more in-depth the eventual chemical interaction between the surface and the adhesive, proposing for the first time, to the best of the authors' knowledge, this technique for characterizing surfaces and adhesives before joining.

2. Materials and methods

The substrate material is an as-sintered Mersen Boostec SiC [20] (France). It is a polycrystalline alfa-SiC (>98.5 wt% SiC) obtained using pressureless sintering. The adhesive material is a bicomponent epoxy resin commercialized as Loctite Hysol EA9391, reinforced with aluminum particles (Henkel Corporation, USA). The Corona treatment was carried out with Tantec SpotTEC equipment [21] (Tantec, Denmark) with the same conditions described in Ref. [9].

Samples were surface treated using a corona-plasma generator, equipped with one treatment head containing two electrodes. The following parameters have been used: voltage: 230 V, frequency 50 Hz, output power 550 W, output power: 2 × 6,5 kV, output frequency 25 kHz. Air was fluxed between the electrodes to transport the plasma filaments onto the designated treatment surface. The treatment head was continuously moved over the SiC surface at a distance of around 5 mm for 5 min to improve the uniformity of the surface treatment.

Corona-treated and untreated SiC joints were analyzed using a Transmission Electron Microscope (TEM) Titan Cubed G2 60–300 (FEI), equipped with a field emission electron gun (X-FEG) Schottky high brightness source with a monochromator, at AGH University of Science and Technology (Kraków, Poland). Images were taken in the bright field mode and electron diffraction patterns were obtained using Selected Area Electron Diffraction (SAED) analysis. The chemical composition was investigated by means of Energy Dispersive X-ray spectroscopy (EDX) with a ChemiSTEM EDX system based on 4 windowless Silicon Drift Detectors (Super X).

The Focused Ion Beam (FIB) technique was used to prepare samples for TEM and STEM investigations directly from the selected area. A Zeiss Crossbeam 350 dual-beam device was used in the preparation of the samples. The sputtering sample surface was achieved by using Ga ions emitted from a liquid metal source. To protect the investigated volume, a thin Pt layer was deposited on a sample surface over the region selected for investigation. The sample was mounted in a TEM-dedicated copper grid to perform the finishing lamellae thinning, down to a level of several dozen nanometers. This procedure minimizes the occurrence of artifacts during sample preparation, and it is used to obtain thin lamellae of a uniform thickness, suitable for high-resolution imaging and quantitative TEM-EDX analyses.

Zeta potential analyses were performed with an electrokinetic analyzer (SurPASS - Anton PAAR) equipped with an adjustable gap cell. The measurement was performed on SiC tiles before and after Corona treatment and on cured epoxy. Hysol was cured in the air at 85 °C for 1 h, as done in Ref. [9]. Acid and basic titration curves were obtained in 0.001 M KCl varying the pH with 0.05 M HCl and 0.05 M NaOH by means of the instrument's automatic titration unit. A freshly prepared new set of samples was used for acid and basic titration for SiC while the same set of samples was used for both titrations in the case of epoxy.

The sticky nature of the glue made it infeasible to collect information on the behavior of each component via zeta potential before curing. Therefore, only cured epoxy was analyzed.

Chemical groups of the cured epoxy adhesive were investigated using FTIR-ATR - Fourier Transformed Infrared Spectroscopy in Attenuated Total Reflectance mode (Nicolet iS50 FTIR Spectrometer, Thermo Scientific, Waltham, MA, USA).

3. Results and discussion

The different crystallographic phases of Corona-treated SiC were observed and analyzed by means of TEM-SAED. Fig. 1 shows the cross-section of the Corona-treated SiC surface (Fig. 1a), where the presence of a SiO_2 layer that was formed after the surface treatment is evident. SAED analysis confirmed the crystalline nature of SiC (Fig. 1b) while the diffraction pattern of SiO_2 (Fig. 1c) proved its amorphous structure. The silica layer thickness ranges between 200 and 300 nm.

The presence of the silica layer was also confirmed by means of EDX elemental maps (Fig. 2). The cracks that can be found at the surface could be related to the cutting of the sample, before the investigation. Regardless, small internal cracks, as depicted in Fig. 1, are not expected to be critical for the mechanical strength, as the adhesive can infiltrate them during the joining process. Enhancing adhesive retention at the

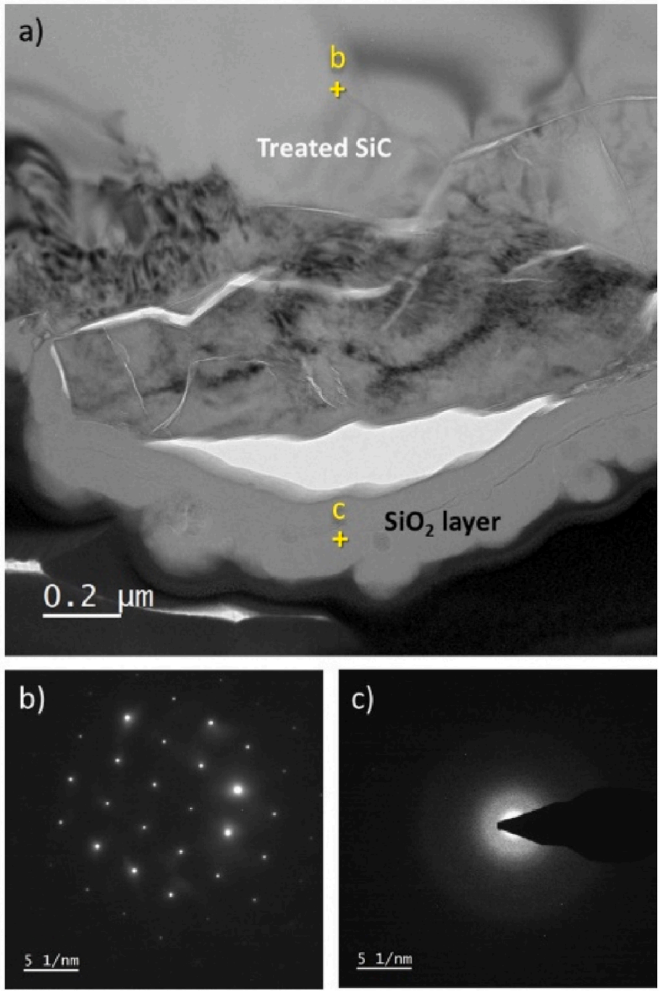


Fig. 1. Bright field image of the cross-sections of treated (a) SiC. Electron diffraction patterns of SiC (b) and SiO_2 layer (c) were obtained. It is possible to notice the presence of the amorphous SiO_2 reaction layer, confirmed by SAED analysis.

joined interface is desirable, as well as fostering the formation of anchoring points on the joint surface.

TEM analysis was also performed on both untreated and Corona-treated SiC joined with the epoxy adhesive, and their cross-sections are reported in Fig. 3 a-b, respectively. The observation on untreated joined SiC (Fig. 3a) showed distinctly the presence of the epoxy adhesive and the underlying SiC. In the untreated SiC joined with the epoxy adhesive a reaction layer between the two components is absent, while, in the Corona-treated SiC joint, the silica layer at the interface between the SiC and the epoxy adhesive can be detected (Fig. 3b). It was not possible to detect any specific morphology of the formed silica layer, contrary to what has been observed in laser-structured SiC surfaces, where the formation of a silica-based nanostructured columnar layer was detected [7]. In any case, the interface between silica and the adhesive was sound and homogeneous; the epoxy adhesive infiltrated effectively the silica structure providing anchoring, as expected. SAED analysis was conducted on the samples, confirming the crystallinity of SiC (Fig. 3c), the amorphous structure of SiO_2 (Fig. 3d) and the epoxy adhesive (Fig. 3e).

As a comparison, the critical issue of the laser treatment used for SiC in Ref. [7] was the formation of a thin graphite layer that was detrimental to the joint strength. Accordingly, the composition of the underlying layer of the plasma-treated material was analyzed by TEM-EDS in detail (Figs. 4 and 5). It can be noticed that under the SiO_2 layer a small region (light grey colour area, highlighted by red square in Fig. 5a) can be identified. From compositional analysis, it was possible to detect that carbon content in this area changes and the carbon concentration is slightly higher (Fig. 5c). However, compared with the research reported by Suess et al. [7], there was no formation of a graphite layer. It can be speculated that, in the indicated area, there is a zone enriched in carbon content. Further investigation will be addressed to verify the possible formation of a Si–C C-rich layer below the SiO_2 amorphous layer.

The oxidizing effect of corona plasma discharge is well-known for silicon wafers [22]. and for applications in cleaning and polymer surface activation. However, understanding the reaction of SiC to oxygen presents challenges, as it has traditionally been observed with significantly higher energy sources (e.g., laser) or elevated temperatures. Currently, the corona-induced oxidation of SiC may tentatively be attributed to a localized and substantial temperature increase provided by plasma filaments. While the growth of $Si0_2$ is typically reported in dry air within a temperature range of 800 °C–1400 °C, the corona discharge-induced oxidation has been noted even at room temperature [23]. It is essential to acknowledge that species generated by the ionization of air induced by corona discharge, such as oxygen ions, exhibit high reactivity [24]. These reactive ions can induce oxidation at temperatures significantly lower than 800 °C. Thus, the combined presence of energetic plasma filaments and highly reactive ions may explain the observed oxidation of SiC.

Concerning the carbon identified in the area near the surface of the treated SiC, it can be attributed to the decomposition of SiC into silicon and carbon; it can be hypothesized that part of this carbon does not escape as CO_2 from the material's surface but remains inside the SiC, thus leading to a C-enriched SiC zone.

The zeta potential titration curves of SiC samples before and after Corona treatment and of the cured epoxy are reported in Fig. 6. FESEM images of the surface of SiC before and after the Corona treatment are shown in Fig. 7. It is possible to observe a significant difference in the top view of the two samples: on the treated SiC (Fig. 7b) a porous coating can be detected, according to the microstructure reported in Ref. [9]. Homogenous distribution of the silica-grown region over the entire surface is revealed by SEM in Corona treated sample and an open porosity of the silica structure can also be appreciated. Such a structure leads to an extensive increase in the surface area that suggests a possible improvement in the adhesion of the glue and joint strength. A comparable structure was noted in a silica layer produced on SiC through laser treatment, as documented in Ref. [7].

Untreated SiC had an isoelectric point close to 4, as typical of surfaces without the prevalence of functional groups with a specific acidic or basic chemical reactivity [16]. The presence of two plateaus, with an onset respectively at about pH 3 and 6, revealed that basic and acidic functional groups were exposed by the SiC's surface. According to the measured isoelectric point, these functional groups were balanced on the surface without a net prevalence of one of them. These data gave another experimental basis to the hypothesis that SiC is an amphoteric surface with both acidic and basic functional groups derived by the partial oxidation of the surface and formation of silicon oxycarbides, as already reported in Ref. [25].

After plasma Corona treatment, a significant shift of the isoelectric point through more acidic values (significantly lower than 3, not measurable from the instrument) can be observed. This shift can be associated with a surface enrichment in acidic functional groups, following the observed formation of a silica layer after the plasma treatment of silicon carbide surfaces [7,9]. The formed acidic functional groups were presumably OH groups exposed by the surface silica layer: they were easily deprotonated in an aqueous environment at any pH higher than the isoelectric point inducing a negative zeta potential. No evident plateau was observed because the exposed functional groups did not have a defined acidic strength and they were progressively deprotonated as much as the pH of the solution increased. It is of interest that the slope of the titration curve around the isoelectric point was significantly reduced after the Corona treatment. This effect is due to the

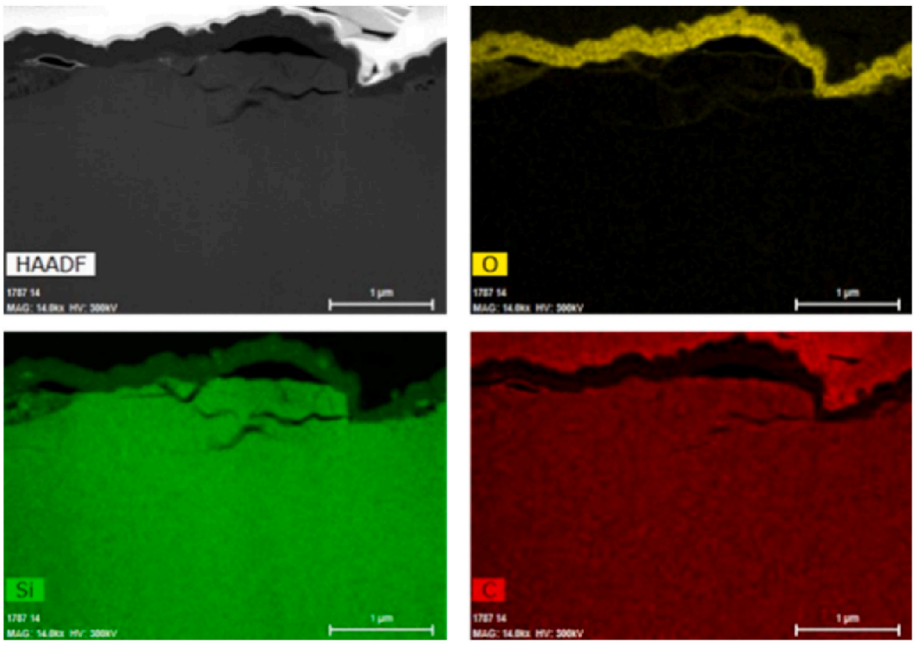


Fig. 2. Elemental maps of treated SiC from the image obtained with a high-angle annular dark field (HAADF) detector. It is possible to detect the SiO_2 layer.

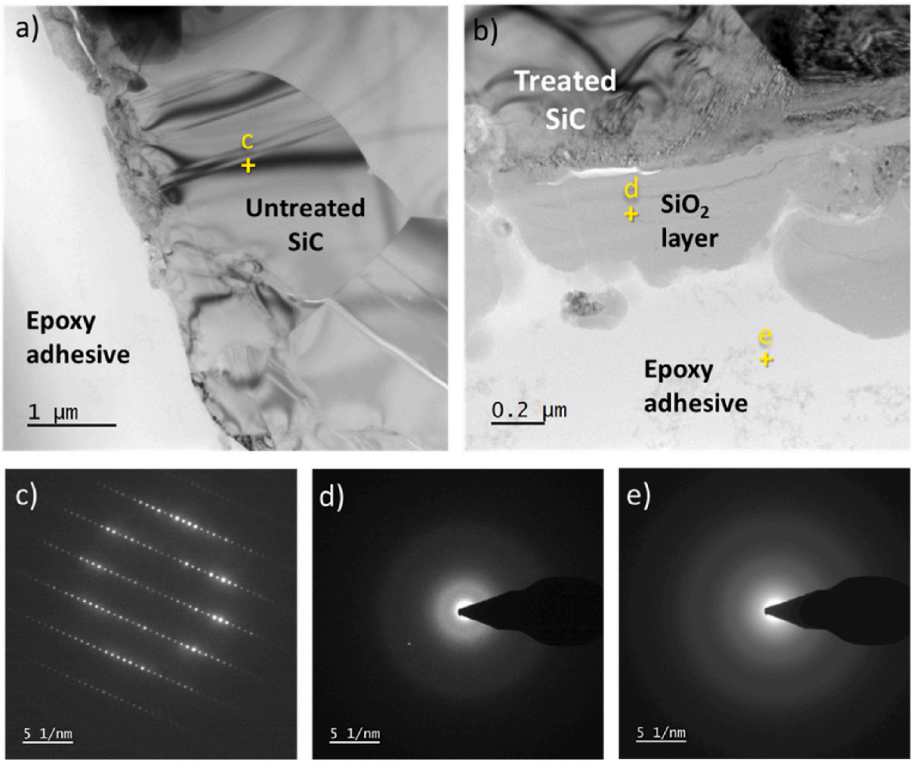


Fig. 3. Bright field images of the cross-sections of untreated (a) and treated (b) SiC-epoxy adhesive interface. Diffraction patterns of SiC grain (c), SiO_2 layer (d), and epoxy adhesive (e) were obtained for the treated SiC joint. It is possible to notice the presence of the SiO_2 reaction layer, confirmed by SAED analysis.

formation of a higher wettable surface. Water molecules are strongly attracted and hardly substituted, on a hydrophilic surface, by hydroxyl or hydroxonium ions from the electrolyte solution when the pH changes, during the zeta potential titration: as a consequence, the surface zeta potential does not strongly change. The opposite occurs on hydrophobic surfaces [17]. A higher wettability is expected to have a positive effect on joining. The low standard deviation recorded for the surfaces, both before and after the Corona treatment, suggested that the surfaces were chemically stable in the explored pH range. The shift of the IEP through more acidic values after plasma treatment was also reported for carbon fibers [26], which experienced oxidation, when Corona-treated, similarly to SiC.

The zeta potential titration curve of the cured epoxy adhesive presented an isoelectric point more basic than both SiC and Corona-treated SiC (6.2) and a plateau in the acidic region which were both related to the exposition of basic functional groups. The presence of basic

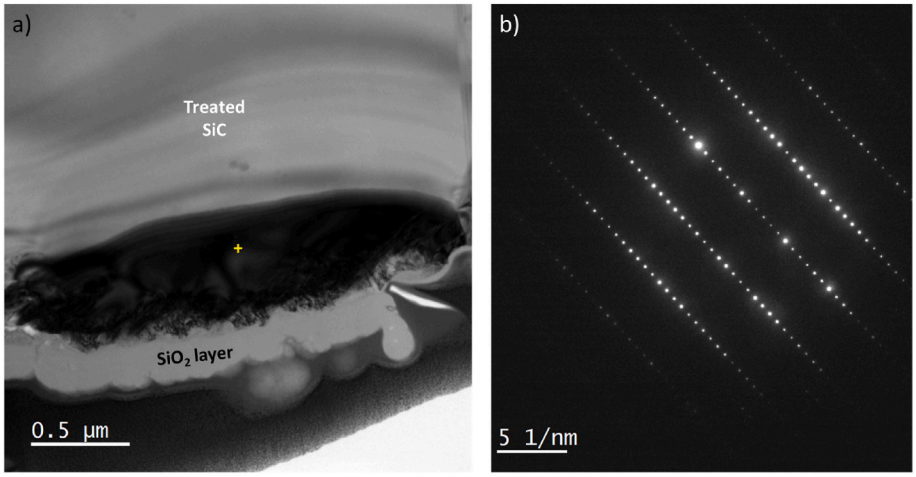
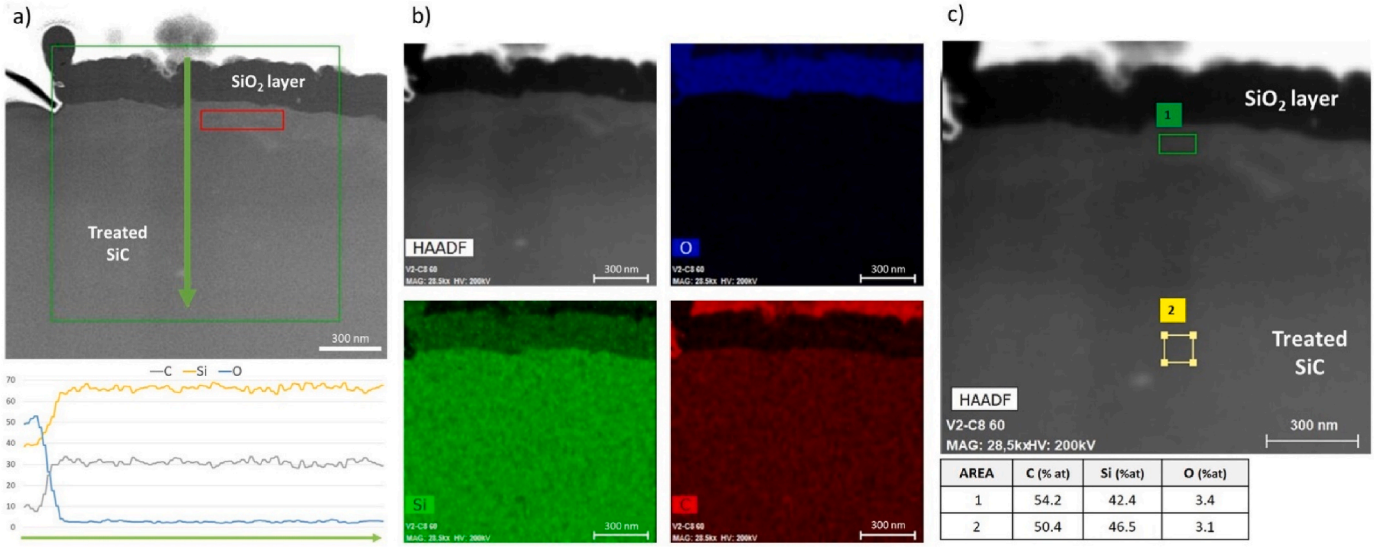


Fig. 4. TEM image obtained in bright field mode (a) showing the cross-section of a plasma-treated SiC, where the SiO_2 layer, a small dark area, and the treated SiC are identified. Image (b) reports the SAED pattern obtained for the dark area.



AREA	C (% at)	Si (%at)	O (%at)
1	54.2	42.4	3.4
2	50.4	46.5	3.1

Fig. 5. EDS Line scan (a), EDS Elemental maps (b) and EDS Area analyses (c) of plasma-treated SiC. The presence of the outer SiO_2 layer can be detected (yellow and blue curves), as well as the treated SiC below (yellow and grey curves). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

functionalities is in line with the resin composition, which foresees the presence of amines and with the basic pH measured for the adhesive components in the liquid form. More in detail, the amines present in the resin are triglycidyl-*p*-aminophenol, at around 40–50 wt% in component A [18], and C18 Fatty acid dimer, tall oil fatty acid, triethylenetetramine polymer (50–60 wt%), diethylenetriamine (5–10 wt), triethylenetetramine (1–5 wt%), and N-(3- (Trimethoxysilyl)propyl)ethylenediamine (1–5 wt%) in component B [19]. According to the titration curve of the adhesive, basic amino groups were prevalent on the surface of the cured adhesive giving a positive zeta potential at any pH lower than pH 6.2. Due to the complex chemical formulation of the adhesive, it can be supposed that some acidic functional groups were also present and they were deprotonated at a pH higher than 6.2 giving a negative overall zeta potential at pH values larger than 6.2.

The presence of amino groups in the cured resin was confirmed by FTIR measurements (Fig. 8). Peak attribution was performed according to Refs. [27,28].

These results suggested that the Corona treatment, in addition to a topographical modification able to increase mechanical interlocking between the adhesive and the SiC surface, allows the exposition of acidic functional groups on the SiC surface which can have a strong electrostatic interaction with the basic functional groups exposed by the adhesive. The acidification of the surface was reported to be beneficial for improving the bonding of SiC and Hysol EA9391 by Levallois et al. [29]. The larger zeta potential difference between the Corona-treated surface and the adhesive concerning the untreated SiC can explain a larger electrostatic attraction and higher mechanical adhesion through an acid-basic mechanism.

These data well fit and explain the mechanical behavior of the joining of untreated and treated SiC with the epoxy adhesive. The average joint strength recorded for the Corona-treated SiC joined was approximately 10 % higher than the values registered for the untreated samples. In addition, it was reported [9]. That all the Corona-treated samples underwent cohesive failure, while the untreated samples underwent adhesive failure. The investigation of the surface by zeta potential measurement can be used as a powerful tool to characterize the surface modified by plasma and, generally speaking, the surfaces for joining.

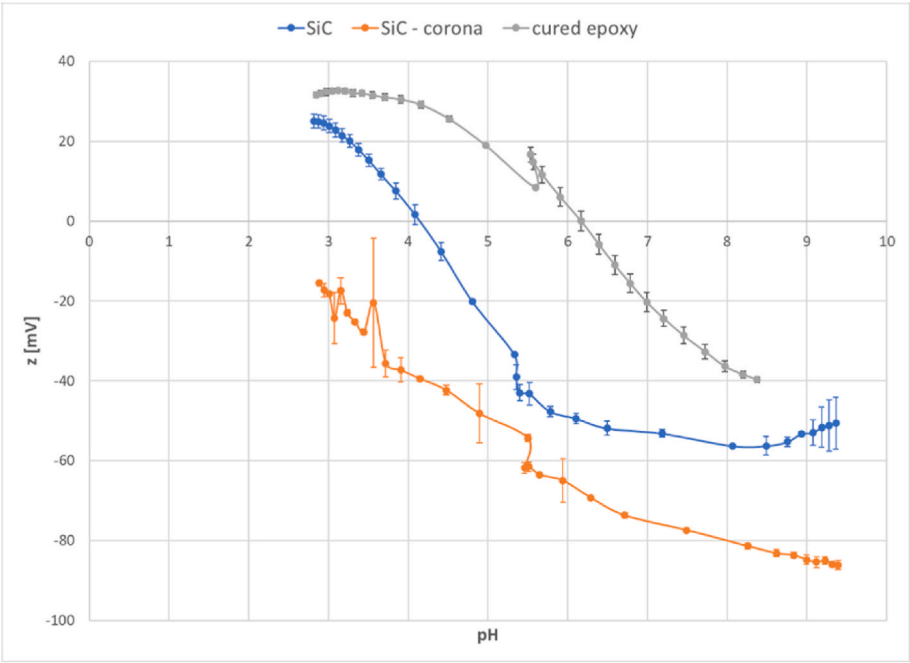


Fig. 6. Zeta potential titration curves for SiC (blue), Corona-treated SiC (orange), and cured epoxy (grey). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

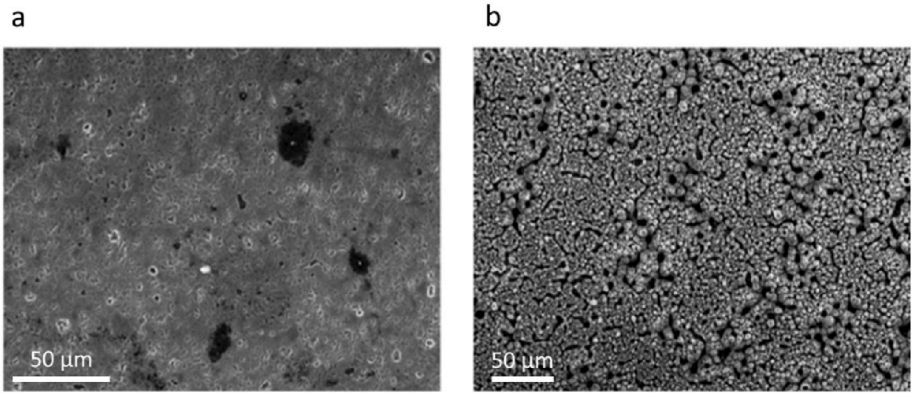


Fig. 7. Top view of SiC before (a) and after the Corona treatment (b).

The plasma effect was shown to be both a physical and a chemical modification. About the latter, the Corona treatment was found to be effective in modifying the SiC surface, where it forms a silica cauliflower structure, as shown in Ref. [8]. This structure can help the infiltration of the joining material and the formation of a stronger bond with the adhesive. The zeta potential technique can be used to detect the effectiveness of the chemical surface modification. A detailed investigation of the surface properties would aid in understanding the adhesion mechanism, i.e. by helping the optimization of adhesive formulations and surface modification or by choosing the most suitable adhesives.

The presented activity shows that surface modification is beneficial for increasing the chemical attraction of epoxy-joined SiC and it better explains the higher joint strength. The study can be expanded to all the adhesives able to maintain basic functional groups after curing or can be used as an analysis to predict the best coupling between a treated SiC surface and a specific adhesive.

Moreover, the zeta potential measurement can be a complementary method of surface characterization to the roughness detection method (i.e. profilometer …). The joining process can be optimized by splitting the chemical and the mechanical/physical behavior of the facing surfaces.

4. Conclusion

SiC surfaces, before and after plasma Corona treatment, a cured epoxy adhesive for SiC surfaces, and joined surfaces were characterized. TEM-EDS investigation reported the presence of a silica layer on the surface of Corona-treated SiC and at the interface between them and the epoxy adhesive in joints. The effectiveness of the plasma treatment to strengthen the joining can be attributed to the formation of this thin silica film, which creates a mechanical anchoring system when penetrated by the adhesive.

The analysis of zeta potential in the function of pH highlighted the exposition of an amphoteric surface SiC before the Corona treatment. In contrast, there was an evident prevalence of strong acidic functionalities on SiC surfaces after plasma treatment. The epoxy adhesive had basic functionalities. These results highlighted an electrostatic interaction between the modified surfaces and the adhesive which can increase the adhesion in addition to mechanical interlocking.

Zeta potential measurements and TEM-EDS analyses resulted in

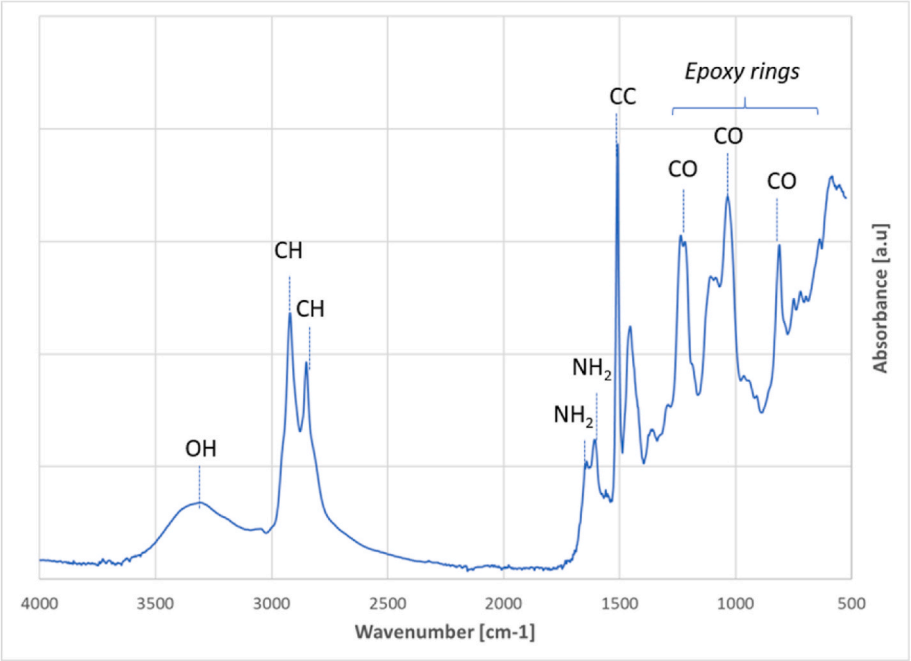


Fig. 8. FTIR-ATR spectrum of the cured epoxy adhesive.

suitable techniques for both the characterization of surface modifications for joining applications and the prediction of joining ability.

Author contributions

The manuscript was written through the contributions of all authors. All authors have given their approval to the final version of the manuscript.

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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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Abbreviations

SiC	Silicon carbide
TEM	Transmission electron microscopy
SAED	selected area electron diffraction
EDX	energy dispersive X-ray spectroscopy

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