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Research Article

Cold Plasma Pretreatment of Carbon Fibre Composite Substrates to Improve Adhesive Bonding Performance

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The purpose of the paper is to investigate the effects of low pressure plasma treatment on wettability of carbon fibre reinforced polymer samples and on shear properties of adhesive bonded joints based on these substrates. In particular, two plasma process parameters, exposure time and power input, were optimized, performing contact angle evaluation on lap-shear tests. The plasma treatment was also compared with a conventional mechanical abrasion and untreated and only degreased specimens. The experimental results show that choosing the optimal parameters is possible to improve the wettability of composite substrates and reduce the contact angle.

1. Introduction

The use of composites is a growing reality in many industrial fields, from civil structures [1–3] to transport industry and especially in aeronautics components [4–9]. Some of their advantages are stiffness, ability to be tailored into complex shapes, strength, corrosion resistance, fatigue properties, and lightweight. In particular, the possibility to decrease the final weight of a manufactured structural component is essential in terms of fuel consumption reduction [10, 11].

Composites are primarily integrated in structures by means of mechanical fastening or adhesive bonding. Adhesives have many advantages in joining composite materials. Perhaps the most significant is that adhesive bonding does not require the composite to be drilled or machined. In fact, traditional techniques of mechanical fastening require the presence of metallic inserts for entering screws and rivets, which makes the manufacturing of the components more complex and does not allow modifications during construction. The use of bonding techniques also allows a better stress distribution as well as durable, lightweight, and aesthetic joints [12, 13].

One of the most important processes to be set before realizing polymer based composite adhesive bonding is the pretreatment of the surface, due to the low surface energy showed by polymers. Recommended preparations of many composite adherends simply consist of a solvent wipe in order to remove dirt and oil followed by a mechanical abrading operation [12–15]. Another widely used technique to solve the problem of composite pretreatment is the peel ply [16]. Many studies have been performed on the preparation of composite substrates also using nonconventional techniques, such as laser [17, 18] or plasma treatments [19–23]. In particular, the aim of a plasma treatment, which can be considered as a physical-chemical procedure, is the functionalization of the specimen surfaces in order to increase surface energy and promote adhesion by providing specific interactions between the adhesive-adherend interfaces. The ionized gas generated by plasma discharge allows not only a deep cleaning of the samples exposed but also the activation and oxidation of polymeric surfaces without affecting bulk properties [12, 13, 24–29]. If we consider the different plasma treatments (corona, low-pressure glow discharge, atmospheric, etc.), the low-pressure glow discharge plasma, also called cold plasma, allows complete control of the processing parameters, and this leads to good homogeneity and reproducibility. Furthermore, it promotes a remarkable increase in adhesive properties of polymer films in terms of wettability of the surface [30, 31]. It also offers a more long-lasting adhesion performance increase than any other treatment [29, 32].

In the context of this study, cold plasma treatment was employed to modify polymer based composite surfaces. The wettability of the specimens was estimated for untreated, solvent degreased, abraded, and plasma treated specimens. The improvement in adhesion properties of these materials after plasma treatment was correlated with lap-shear strength of adhesive bonded joints.

2. Materials and Methods

2.1. Materials. The composite substrate used for this study was an epoxy resin reinforced with carbon fibres, supplied in sheets of thickness 1.5 mm. The sheets were fabricated by laying up woven prepregs and cured in autoclave with a vacuum bag according to a confidential process. A plastic release film was used to remove the laminate from the mould. In this way, the surface of the composite was not contaminated by waxes or silicones, which could compromise the secondary bonding.

A two-part, low viscosity epoxy adhesive developed by 3 M was used to manufacture the composite joints. It has a work life of approximately 70 minutes and a tack-free time of about 3 hours and is fully cured after 48 hours at room temperature [33].

2.2. Surface Pretreatment. In this paper two types of preliminary surface treatment were compared: standard abrasion and plasma pretreatment. Every treatment came after a preliminary cleaning process with acetone, in order to eliminate grease or pollutant particles from the surfaces.

Some untreated and only solvent degreased specimens were used as a basis for comparison. The mechanical abrasion was performed using a P240 grain carbide paper.

To evaluate the effect of cold plasma treatment, the samples were exposed to radio frequency (RF) low pressure plasma, using air as working gas. A glow discharge RF generator operating at 13.56 MHz (model name: Tucano by Gambetti Kenologia, Italy) was used. The plasma is generated in a vacuum chamber between two electrodes: one that also acts as support for the samples to be treated and the other, positioned in the upper part of the chamber, allowing the samples to be completely crossed by the plasma beam. The chamber dimensions are: diameter 150 mm, length 330 mm; the total volume is about 5.5 L. This kind of treatment allows treating of more components at once and this is an advantage when the treatment of a large number of small components is required.

In particular, the effect of plasma power input and exposure time as working parameters was investigated. The flow rate of 25 $\mathrm{cm}^3\ \mathrm{min}^{-1}$ for the air input and a 0.5 mbar working pressure were kept fixed. Table 1 summarizes all the surface treatments compared in this study and gives more details about the plasma pretreatments working parameters.

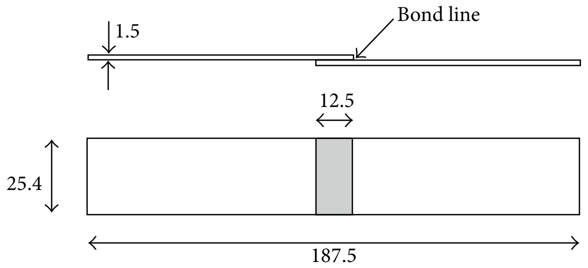


Figure 1: Dimension (in mm) of the specimens realized according to ASTM D5868.

Table 1: Surface treatment summary and plasma working parameter details.

Surface treatment	Description	
No treatment	Samples as-received	
Degreasing	Acetone wiping	
Abrasion	Acetone wiping + P240 grain carbide paper abrasion	
Plasma	Acetone wiping + plasma discharge (with different set-up parameters)	
Set-up parameters	Power input (W)	Exposure time (s)
	100, 200	5, 30, 60, 180, 300, 450, 600

2.3. Evaluation of Contact Angle. Since polymeric based material wettability is very poor [12, 13, 34, 35], tests were carried out to verify the effects of cold plasma treatment on this parameter, compared to untreated and abraded specimens.

In particular the wettability of the substrate was evaluated by contact angle measurement. Demineralised water was dropped onto the surface of the sample using a calibrated pipette. All the measures were performed using a Leica Digital Microscope and X-Pro Software. At least three drops were measured and averaged on the samples treated.

2.4. Lap-Shear Test. Rectangular adherends, having dimensions $100 \times 25.4 \times 1.5\ \mathrm{mm}^3$, were prepared with different types of treatment and parameters and bonded for single tensile lap-shear tests. An overlapping of 12.5 mm was realized. The dimensions of the specimens refer to ASTM D 5868 [36] standard, as well as the lap-shear test conditions; five samples for each parameter setup were tested and averaged. Shape and dimension of the specimens are reported in Figure 1. All the specimens were realized with a specific equipment in order to maintain the same adhesive thickness of 0.5 mm and to have a high repeatability rate. Standard deviation was also calculated in percentage.

After the lap-shear test, the failure mode was analysed in order to evaluate the percentage of cohesive and adhesive failure. This investigation was carried out using a microscope and a specific function of its software.

3. Results and Discussion

3.1. Effects of Pretreatment on Contact Angle. As expected, the hydrophilic behaviour of the surfaces increased from the untreated to the abraded ones, reaching the minimum

Table 2: Contact angle on CFRP surfaces.

Surface treatment		Mean contact angle (°)	Standard deviation (%)
No treatment		54	9
Degreasing		48	6
Abrasion		52	10
Plasma			
Power input (W)	Time (s)		
100	5	21	9
100	60	20	5
100	180	19	10
100	300	16	15
100	450	14	20
100	600	12	18
200	5	17	6
200	60	14	20
200	180	10	10
200	300	7	25
200	450	6	23
200	600	5	30

values of contact angle for the samples exposed to the plasma treatment.

Table 2 shows the values of contact angles for untreated, only degreased, abraded, and plasma treated CFRP substrates using a power input of 100 W and 200 W and different treatment times. The standard deviation in percentage is also reported as an indication of the repeatability of the results. Each value was measured as soon as the samples were withdrawn from the reactor.

The degreasing surface pretreatment does not affect the surface wettability of the composite substrate in any way. The contact angle remains almost unchanged for untreated, degreased, and abraded treated specimens, being close to 50°.

On the contrary, the application of a plasma discharge, even at low treatment times, produces a remarkable increase in surface wettability, reducing the contact angle by more than half compared with the untreated surface. Concerning the 100 W treatments, the best result in terms of surface wettability was obtained for treatments longer that 300 s, even if at 180 s the result is considerable.

Figure 2 shows the variation of the demineralised water contact angle values of the low pressure plasma treated CFRP as a function of treatment time (from 5 to 600 s). The graph also includes three horizontal lines representing the average value of contact angle of untreated, only degreased, and abraded substrates.

As it is easy to observe, the plasma treated sample surfaces experencied a significant decrease of the contact angle even using very short exposure times, while for longer times the advantage is not so substantial.

This is due to the fact that, during the first seconds of the plasma treatment, the free radicals produced by the action of the plasma gas have high instability and reactivity and they could insert polar species and activate the surfaces [37].

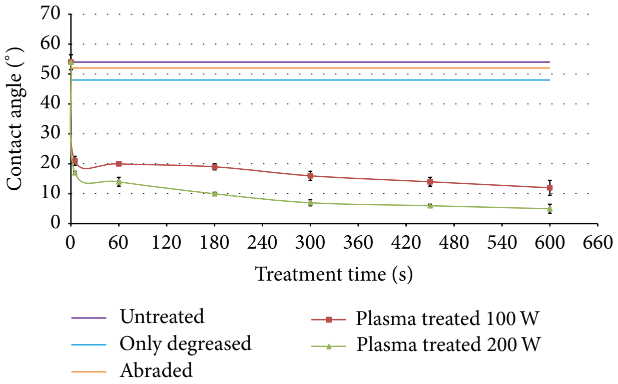


Figure 2: Variation of contact angle measured on sample surfaces in terms of treatment time and kind of treatment.

The plasma power is linked with the potential to produce the functionalized layer on the surface. Because of the hydrophilicity of the functional group constituting the layer, the drops of water are easily absorbed into substrate surface and the contact angle decreases. This fact is being reported by [38]. In a previous work we have also reported an effective functionalization of polyethylene surfaces using the same plasma reactor [29].

These effects are generally supposed to be responsible for establishing strong interactions with adhesives, and their presence could be quantified by the surface energy polar component. Several authors calculate all the surface energy components in their works and express the link between plasma treatment and polar component [10, 21, 26, 28, 30, 37, 38].

Indeed from the results, it is possible to observe that the surface wettability improved even using short plasma treatment time, and using a high power level it is possible to minimize contact angle values.

3.2. Effects of Plasma Pretreatments on Lap-Shear Strength. Adhesive joints between two CFRP substrates were realized, comparing only degreased, abraded, and plasma treated surfaces with untreated ones. Single lap-shear tests were conducted as mechanical characterization, using a 1.3 mm/min test speed.

Figure 3 reports shear strength of the joints in terms of plasma treatment time, with two power input levels, 100 W and 200 W, being fixed. Each point of the curve represents the average strength of five joints with the corresponding time-power setup and error bars. The graph also includes three horizontal lines representing the average value of shear strength of untreated, only degreased, and abraded substrates. Table 3 reports the numerical values of mean shear strength and standard deviation of these samples.

The mechanical properties of the adhesive joints are greatly influenced by the surface preparation. The results show that, without surface preparation, the joints presented very low shear strength, but it increased three times after only

Table 3: Shear strength of adhesive joints with different surface treatments.

Surface treatment		Mean shear strength (MPa)	Standard deviation (%)
No treatment		1.24	83
Degreasing		3.36	26
Abrasion		5.28	34
Plasma			
Power input (W)	Time (s)		
100	5	3.62	24
100	60	4.36	7
100	180	4.79	5
100	300	4.34	7
100	450	—	—
100	600	—	—
200	5	3.92	19
200	60	5.67	12
200	180	5.77	8
200	300	7.30	6
200	450	7.28	11
200	600	5.79	8

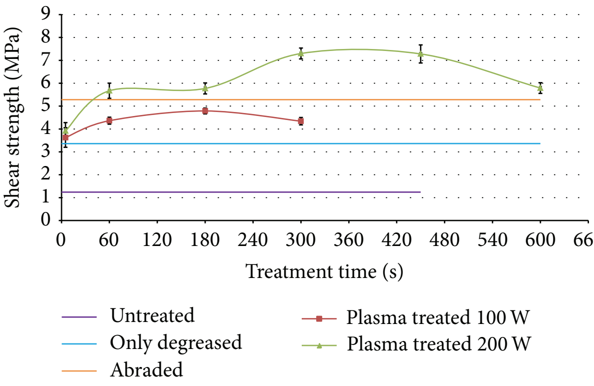


Figure 3: Comparison between shear strength of plasma treated and other samples.

Table 4: Mode of failure of the adhesive joints with different surface treatments of the substrates.

Surface treatment		Mode of failure	Standard deviation (%)
No treatment		100% adhesive	—
Solvent treatment		100% adhesive	—
Abrasion		25 % cohesive in adhesive	10
Plasma treatment			
Power input (W)	Time (s)		
100	5	100% adhesive	—
100	60	100% adhesive	—
100	180	100% adhesive	—
100	300	100% adhesive	—
100	450	—	—
100	600	—	—
200	5	15% cohesive in adhesive	5
200	60	48% cohesive in adhesive	8
200	180	53% cohesive in adhesive	12
200	300	95% cohesive in adhesive	4
200	450	98% cohesive in adhesive	2
200	600	52% cohesive in adhesive	13

degreasing the surfaces before bonding with acetone, even if this preparation was not satisfactory and it corresponded to adhesive failure of the joints tested.

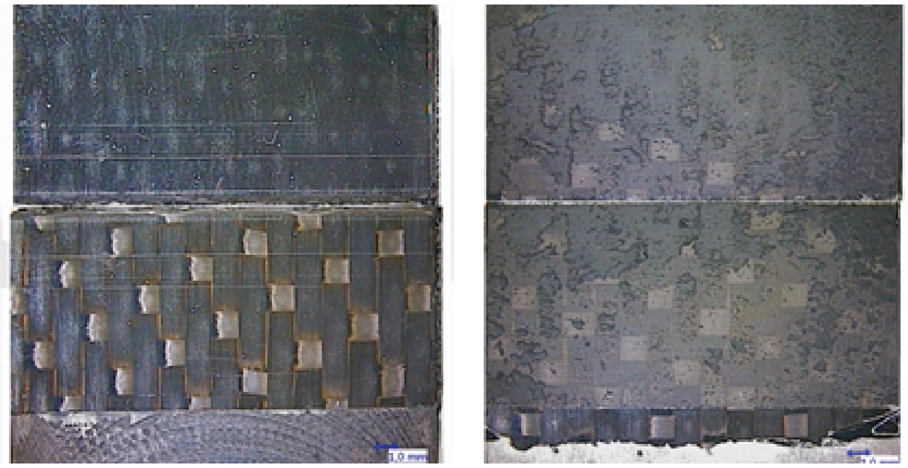
A typical mechanical pretreatment of abrasion of the surface gave a very good result. Being very careful not to reach the carbon fibre, it is in fact one of the most applied methods to increase the adhesion properties of the surface. This is confirmed by the failure mode, which is partially cohesive. Figure 4 compares the failure surfaces after lap-shear test of samples prepared with only degreasing (Figure 4(a)) and abrasion (Figure 4(b)).

A 100 W plasma preparation is not enough to overcome the abrasion treatment. Since the tests were performed step by step analysing the data acquired, this power input series investigation was interrupted at 300 s to switch to a higher power level. The poor result is also confirmed by the fact that, using this power input, the failure mode is completely adhesive for all the exposure times.

On the contrary, a 60 s exposure to the plasma discharge with a power input of 200 W is sufficient to reach and overcome the mechanical properties of the abraded specimens. Using this power input, the best results were obtained for two exposure times: 300 s and 450 s. Observing Figure 3 it is possible to note that, for this power level, the shear strength increases until a 450 s treatment, while for longer exposure time the effect is negative and the strength decreases. In fact, if the plasma treatment is too aggressive, the surface will be overetched and its uniformity will decline and might also be damaged by heat generated during the treatment.

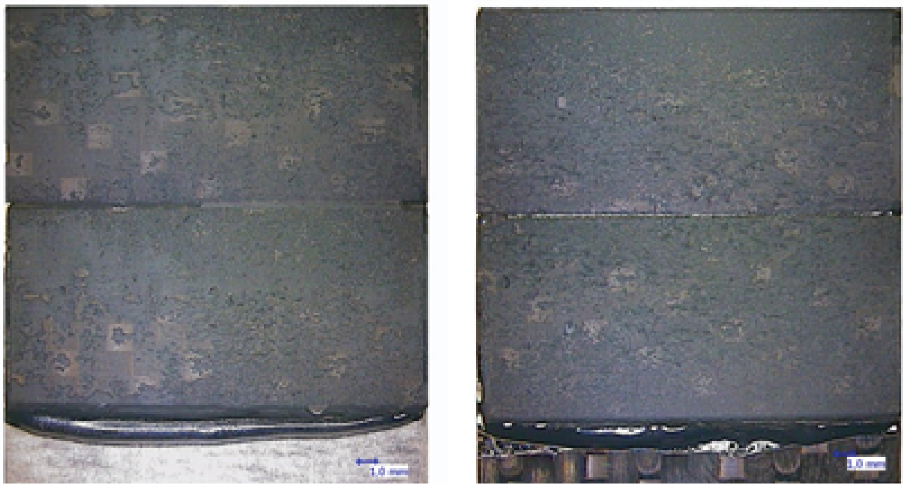
The good results of the bonded joints treated with the optimal parameter setup were confirmed by their failure mode.

Figure 5 reports two examples of failure surfaces of joints realized with plasma treated samples, with two different power-time setups. Comparing the images and still more by reading the data in Table 4, it can be observed that the effect of treatment time on the failure mode repeats exactly that already seen for the shear strength: the percentage of cohesive failure grows to 450 s and decreases for higher treatment times.



(a) (b)

Figure 4: Failure surfaces of joints realized with samples only degreased (a) and abraded (b) after lap-shear test.



(a) (b)

Figure 5: Failure surfaces of joints realized with plasma treated samples with different power-time setup: 200 W-180 s (a) and 200 W-450 s (b) after lap-shear test.

The literature on the relationship between the parameters used in the cold plasma treatment and shear properties of the joints obtained using the treated composite substrates is rather limited. However, the results of this research are comparable with some research conducted by Shanahan and Bourgès-Monnier [19] and Gude et al. [21] for an epoxy composite, by Saleema et al. in two of his works [32, 39] for aluminium supports, and by Anagreh et al. [23] and De Iorio et al. [27] for polymeric substrates.

Finally, it was found that there is a correlation between the shear strength properties of the adhesive joints and their failure mode and this is clearly visible comparing the results in Tables 3 and 4. This has already been analysed in several works and in particular by Gude et al. [21] for this substrate. In fact, it is already known that the worst mechanical performance takes place when the joints fail through the adhesive/substrate interface and the application of surface treatments is precisely used to avoid the adhesive failure mode. In the joints realized using 200 W plasma treated samples, the percentage of cohesive failure mode increases with the treatment time, in the same way as the shear strength. In fact, the best shear properties are obtained when the failure mode is 98% cohesive.

From the results of this research and from literature, it is clear that plasma power input and treatment time significantly affected the mechanical properties of adhesive bonding joints. Finding the optimal combination of these parameters it is possible to obtain joints characterized by very high strength.

4. Conclusions

This study focused on the effect of cold plasma treatment on CFRP substrates. The improvement in adhesion properties of plasma treated polymers has been described in terms of wettability, evaluated by contact angle measurement, lap-shear strength of the adhesive bonded joints realized using treated surfaces, and failure mode that occurred after these tests. The results were also compared with untreated, only degreased with acetone, and abraded ones.

The results have primarily emphasized how critical the surface preparation is to obtain good joints. The plasma treatment has proven to be quite effective and in particular the following occurs.

(i) Surface wettability improves as the plasma exposure time increases, as shown by the reduction of the contact angle. This is due to the almost total removal of pollutants and to surface activation.

(ii) The plasma treatment effectiveness is also confirmed by a significant improvement of the strength as power input and exposure time increase. It is worthy of notice that it is possible to exceed the shear limit obtained with abraded treated joints even within quite short time periods (60 s).

(iii) The fraction of cohesion failure increases by using plasma instead of abrasion and it is possible to reach the best result choosing the appropriate parameters.

The activation by plasma represents a fast and eco-friendly technology to more traditional methods, as only degreasing or degreasing followed by mechanical abrasion.

Conflict of Interests

All the authors declare that there is no conflict of interests regarding the publication of this paper.

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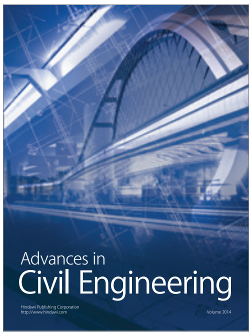
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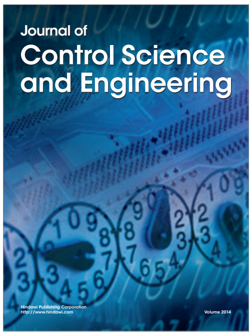
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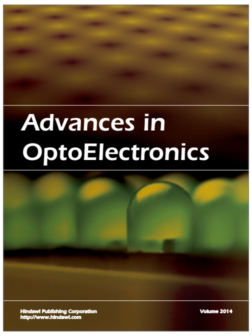
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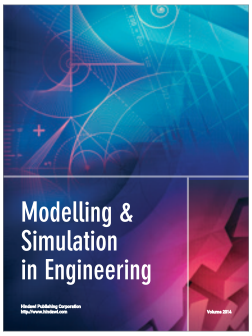
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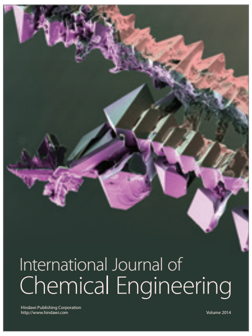
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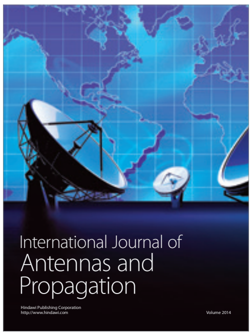
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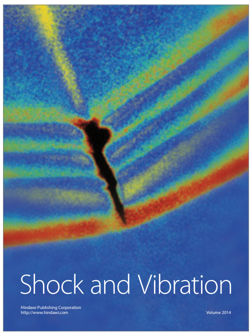
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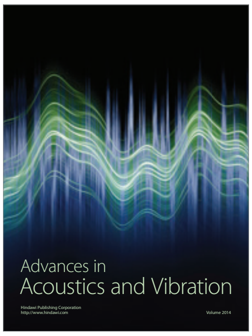
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