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Laser surface treatment for enhanced titanium to carbon fiber-reinforced polymer adhesion

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Abstract The adhesion between carbon fiber-reinforced polymer (CFRP) and titanium parts can be improved by laser surface texturing before gluing them together. Here, a pulsed Nd:YAG laser was employed before bonding of the textured surfaces using an epoxy paste adhesive. To investigate the influence of the laser parameters, the roughness of the surfaces were characterized and correlated to the wettability, by measuring the contact angles of water droplets. Scanning electron microscope (SEM) characterization was also performed showing that under careful conditions, the fiber structure could be maintained. Tensile tests were performed to evaluate the influence of the laser treatment parameters on the joining efficiency. The lap-joint tensile strength tests presented the best results when the titanium surfaces were treated using 5.03×10^{-3} J pulses and the CFRP sample treated with 5.03×10^{-4} or 5.03×10^{-3} J or even using a virginal CFRP surface. Using a virginal CFRP surface, the critical load of fiber delamination was increased and as a consequence the tensile strength attains the highest level of 8.2 MPa.

Keywords Laser surface treatment · Adhesive bonding · Titanium · Carbon fiber-reinforced polymer

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1 Introduction

The aerospace sector is known for the use of high reliability of tough and low-density materials. Over the last 10 years, carbon fiber-reinforced polymers (CFRP) have become increasingly common in the aerospace sector, because they combine low weight components with excellent tensile strength [1]. However, the fracture toughness of CFRP is not as high as that of some metals, such as aluminum, steel and titanium. As the resistance to crack propagation is an important property for many aircraft components, some aircraft components are composed of metal-to-composite bonds. For example, the leading edge of the wings is subject to impacts from birds, ice and other solid particles and for this reason needs to be shock resistant. As a result, CFRP is not applicable for the wing head surfaces and thus many aerospace industries apply titanium alloys at the leading edge [1]. CFRP and titanium alloys are very dissimilar structural materials, but need to be connected to each other in some way. Mechanical fastening has been made using screws or rivets, but they introduce an extra weight and may cause stress concentrators, delamination and fiber chipping around the holes. Some industries prefer to bond CFRP and Ti alloys using a high-strength glue, such as epoxy, although the surfaces preparation before gluing could be tricky. Here, to improve the adhesion performance, a laser treatment of the two connecting surfaces was proposed. This technology is a step ahead of the ordinary instructions of guarantee of “clean, degreased and dry surfaces before gluing”, because the contact surfaces will be lasered just before joining. It will be shown that multiple laser pulses can be used to control the roughness of a given solid surface by treating the whole surface [2] or by producing a raster of flat-shaped holes [3].

Since the CFRP and Ti phases must be well wetted by the glue so as to assure a strong bond, the contact angle must be optimized. The influence of roughness on the adhesion depends on the adhesion mechanism. Not all adhesives work in the same way and some benefit more than others from a rough surface [4]. One of the adhesive mechanisms that experience benefits by a rougher surface is the mechanical interlocking. In this mechanism, the adhesive spreads into cracks and pores after which it solidifies and anchors to the surface. This mechanism works less for flat surfaces and may benefit from laser surface texturing [5]. Other mechanisms such as interdiffusion, adsorption and surface reaction rely more on a chemical adhesive bonding rather than a mechanical one. Interdiffusion relies on atomic diffusing into other materials and the effect is similar to that of mechanical locking, except the fact that it happens on a much smaller (sub-nanometer) scale. For adsorption and surface reaction, adhesive mechanisms must have a chemical bond, such as donor–receptor interactions or Van der Waals forces to connect to the substrate.

An increased contact angle often means a lower wettability and lower adherence. However, when using adhesives, the adhesive force is also related to other factors such as the surface area. Lim et al. [6] have shown that increasing the surface area by increasing the roughness of an Al–Cu junction by 10%, the adhesive force is increased by 50%. Uehara and Sakurai [7] investigated the influence of five different types of adhesives on the tensile, shear and peeling strength of carbon steel plates. An optimum condition was found in the range of 3–6 μm R_a . This value can be slightly different for different types of adhesives and, according to the authors [7], for the peel-off force the roughness did not seem to matter. Bandoki et al. [8] showed that a laser-induced roughness increase on 316 L stainless steel and titanium was able to increase the contact angle from about 80°–120°. Yilbas et al. [9] demonstrated that for a treated surface, the contact angle may diverge on different places of a sample. Otherwise, Lawrence et al. [10] showed that Al–Si laser-treated surface resulted in a smaller contact angle. Sakata et al. [11] investigated laser texturing of galvanized steels before adhesive bonding. These authors showed that samples processed with 0.3 mJ green pulses presented a lap-joint tensile stress of 88 MPa, compared to 23 MPa of the untreated samples.

The objective of this work is to examine if the adhesion of CFRP and titanium alloy can be improved by a laser surface treatment. To achieve this goal, tensile tests were elected to evaluate the degree of adhesion on the CFRP and titanium alloy sheets after being glued together.

2 Experimental

The base materials were carbon fiber-reinforced polymer (CFRP) and grade 5 titanium alloy (Ti-6Al-4V). The CFRP characteristics were the following: carbon fiber diameter = 8 μm , carbon fibers bunch diameter = 1 mm, number of pre-peg layers = 9, pre-peg fabric type = shed double layer, carbon fiber mesh and type of polymer = epoxy. The coupons were electron discharge machined (EDM) to the dimensions: height 6.2 mm, width 20 mm and length 100 mm. The grade 5 titanium alloy had the following composition: Ti-6.58Al-4.13V-0.21Fe (wt%) and is furnished in as-annealed condition. The Ti sheets had 1 mm thickness and were machined to width 20 mm and length 100 mm.

The glue was Hysol Loctite EA 9394 QT AERO. The glue comes in two parts that have to be mixed to cure at room temperature. The two parts, A and B, were mixed with a weight ratio of 100 to 17, respectively. Before applying the glue, the treated surfaces are cleaned by a strong nitrogen gas flow. The glue was added to an area of $2 \times 3 \text{ cm}^2$ and the samples were pressed by a 25 N force to be in contact for 24 h. After this period, the samples were left to dry for 7 days in a 35% humidity, 30 °C, controlled chamber.

A scanning electron microscope Tescan Vega 3 was used for the sample analyses. The roughness was measured by a Holmes TR200 Digital Surface Roughness Tester. The roughnesses of the samples were measured six times for each laser setting. To take into account the anisotropy, each measurement was done in different directions to avoid errors. The tensile tests were performed on a Kratos Machine, capable of applying a maximum load of 5000 kgf. The tensile test was performed in the lap shear configuration, as schematically depicted in Fig. 1. The applicable standard is ASTM D 1002 [12]. Each surface condition was tested three times.

Water droplets were used to measure the contact angle of the treated surfaces. A digital camera was used to take photos of the droplet on the surface. The contact angle was measured by means of a specific software (QuantDim). The contact angle of a droplet on a surface depends on the equilibrium between the liquid–solid, liquid–vapor and solid vapor interfaces as given elsewhere [13]. The contact angle θ defines the wettability of the water drop as give

Fig. 1 A schematic view of the lap-joint tensile tests with the arrows indicating the load

Fig. 2 Photos of the CFRP surfaces after laser treatment in two different laser conditions: hydrophilic (a) and hydrophobic (b)

Table 1 Laser parameters used in this work

Condition	I (W/cm ²)	E (J)	N	P_1 (W)
A	1×10^7	2.51×10^{-4}	1	1.3
B	2×10^7	5.03×10^{-4}	1	2.5
C	5×10^7	1.26×10^{-3}	1	6.3
D	1×10^8	2.51×10^{-3}	1	12.5
E	2×10^8	5.03×10^{-3}	1	25.1
F	5×10^8	1.26×10^{-2}	5	12.5
G	1×10^9	2.51×10^{-2}	5	25.1
H	2×10^9	5.03×10^{-2}	20	12.5
I	4×10^9	1.01×10^{-1}	20	25.1

in Fig. 2. If the angle is above 90°, the surface is called hydrophobic, otherwise it is called hydrophilic, as also shown in Fig. 2.

The laser was a frequency-doubled diode-pumped Nd:YAG laser. The laser wavelength was 532 nm, the pulsing frequency was fixed at 5 kHz, the pulse length was 80 ns, the spot diameter 0.2 mm and the maximum power was 25.1 W at the workpiece. The distance between the centers of the two neighboring pulses was fixed at 0.2 mm. A 50% superposition between each laser pulse was assured by fixing the scanning speed of 500 mm/s. To investigate the influence of the laser parameters on the samples, both the titanium and the CFRP were treated with nine different intensities (I) as shown in Table 1. The pulse energy, E , is given by the intensity times the irradiated area (0.0314 mm^2) times the pulse length (80 ns). As the laser was unable to deliver a P above 25.1 W, conditions F to I require several runs (N) maintaining the laser power (P_1) below its limit. When multiple runs ($N > 1$) are necessary, each sequential run is perpendicular to the predecessor.

These conditions, together with the virginal material, called V hereafter, resulted in ten different surface conditions for each material. Each treated surface had the roughness, contact angle and the surface analyzed. From these results, two laser settings of each material were selected to be used for tensile tests.

3 Results and discussion

3.1 General aspect

Both the CFRP and the titanium substrates showed a change in optical appearance after laser treatment. In the case of the CFRP samples, A and B conditions produced a surface with little damage and almost intact epoxy. The underlying carbon fiber structure became visible when the energy (Table 1) was equal or above condition C. Under these conditions, most of the superficial polymer was ablated. Conditions D to I fully expose the carbon fibers and the processed surface showed many bristle spots. Severely damaged fibers were observed when the energy was equal or above 12.6 mJ, and the surfaces had a fluffy appearance.

For the titanium substrates, the most important modifications belonged to coloring. The two lower energy conditions (A and B in Table 1) seemed to only clean the surface and the final aspect was bright silver. From this point, the surfaces became continuously darker with gray levels. Conditions E, G, H and I presented a golden color. It is interesting to observe that sample F did not show this golden color even while the total energy was higher than that of sample E. The difference was that sample F was treated to a lower laser power, but with more runs, each one ablating the previous oxide coating.

3.2 Microstructural analyses

Figure 3 shows the CFRP sample surfaces after different conditions of the laser treatment. As can be seen, from the optical observation after laser treatments A and B, the plastic top layer is still intact. In treatment C, the first parts of the carbon fiber structure become visible. The fiber structure becomes increasingly exposed to higher laser intensities and at treatment E the fiber is fully exposed. At higher intensities, the fiber becomes more damaged layer by layer, as seen for laser treatment I.

In the titanium case, the laser impact was more visible as the energy increased, but the effect was less evident

Fig. 3 Scanning electron images of the CFRP surfaces compared to the virginal (V) condition. Treatments B, C, D, E and I

Fig. 4 SEM images of the virginal (a) and laser-processed surface at condition G (b), titanium samples

Table 2 The measured average roughness of the Ti and the CFRP samples

Sample	Ti roughness Ra (µm)	CFRP roughness Ra (µm)
V (virginal)	0.45	1.80
A	0.50	1.59
B	0.51	1.54
C	0.48	4.61
D	0.53	6.11
E	0.58	*
F	0.46	4.18
G	0.47	4.74
H	0.43	*
I	0.44	5.54

* Measure not possible

than that of the CFRP case. Figure 4 presents a comparison between the virginal (V) and type G lasered surface.

3.3 Surfaces roughness

The results of average roughness for each condition are presented in Table 2. The roughness of titanium surfaces increased from the virginal (untreated) to condition E, from 0.45 to 0.58 µm. Therefore, for one run, the Ti roughness was approximately proportional to the laser energy: $Ra \approx 0.47 + 19.95E$. For five laser runs over the same area, the average roughness reduced to about 0.46 and 0.47 µm for conditions F and G, respectively. Then, for 20 runs in conditions H and I, the final Ra was below the initial Ra of the virginal titanium surface, indicating polishing by ablation.

In the case of CFRP, the virginal surface average roughness was 1.80 µm, decreasing to 1.59 µm using 0.25 mJ pulses and to 1.54 µm using 0.50 mJ pulses. In this case, the carbon fibers were not exposed and the overall effect was polymer polishing. When the energy attained the values 1.26 and 2.51 mJ per pulse, the roughness attained 4.61 and 6.11 µm, respectively. This corroborates the full fiber exposure of the optical evidences. Condition E produced a surface too rough to be measured because it was covered by a fine hairy structure. The conditions F to I, using multiple runs, also produce very rough surface due to the exposition and ablation of the carbon fibers.

3.4 Contact angle

The results of the contact angle measurements are listed in Table 3. The table shows that the laser-treated surfaces have a large increase in contact angle. The increase is most visible for the CFRP samples. The difference in contact angle

Table 3 The measured water contact angles for lasered titanium and CFRP

Sample	Contact angle Ti (°)	Contact angle CFRP (°)
V (virginal)	64.5	66.5
A	72.5	81
B	75.5	80.5
C	71	80.5
D	80.5	101
E	79	112
F	81.5	126.5
G	76.5	127.5
H	77.5	125
I	73	107.5

V is the virginal material

Fig. 5 Ti and CFRP contact angles for the different total laser energies. The horizontal dashed lines are the virgin materials' contact angle

is clearly shown in Fig. 2, which represents conditions B and F of the CFRP-treated substrates.

A higher contact angle is what one would expect for a rougher surface. From the data available, there seems to be a clear correlation between the roughest and contact angle, something which could be expected. When looking at the laser energy with respect to the contact angle, there is clear evidence that laser treatment produced an increase in the hydrophobic behavior (Fig. 5). For the CFRP samples, the increase after exposing the fiber structure is clearly visible. The interesting fact is that both Ti and CFRP show a decrease in contact angle for the highest energies because of the laser polishing effect.

As a point of discussion, it should be realized that in the aforementioned Wenzel approach [13], the rough surface was supposed to be completely wetted and unwetted sharp grooves are ignored. In fact, a similar model was proposed by Cassie and Baxter [14] taking into account the area fraction of an uncontacted solid–liquid interface on the solid. Unfortunately, neither of these theories can predict correctly the experimental contact angles as a function of roughness and the parameters of area fractions are not easily experimentally accessible either. Moreover, the corresponding equations are only correct for radial grooves with a liquid droplet spreading radially, for which an equilibrium state can be reached. As a result, we have revised these approximations by considering random roughness at a submicron length scale. In particular, it is shown that the apparent contact angle depends critically on the roughness exponent and on the long wavelength ratio of roughness amplitude and correlation length. For details, reference is made to [15, 16].

3.5 Tensile tests

With 10 different titanium and 10 different CFRP samples, there would be 100 possible combinations to test the strength of the adhesive force. This would result in too many samples and tests to explore the possible combinations. Therefore, it was decided to make a selection of three surfaces for each material, including the virgin (V) condition. Consequently, for the tensile tests, two promising conditions were selected for each material, Ti and CFRP. The conditions selected for Ti were “E” and “F”, called hereafter TE and TF, respectively. The conditions selected for CFRP were “B” and “E”, called hereafter CB and CE, respectively. The virgin surfaces were named TV and CV, for the Ti and CFRP, respectively.

For the CFRP, only samples A and B had no carbon fibers exposed. Thus, it would be interesting to compare one sample with fully intact fiber structure and one with exposed fiber structure. Considering the intact fiber, laser parameter B was chosen because this setting has more impact on the structure than A. For the exposed fiber it was not possible to choose a setup, which requires too many laser cycles. The main reason for this is that the laser treatment will take much time, which from an industrial point of view is not attractive. The second reason is that when exposing the CFRP to the laser for too long, the samples may experience burning as the temperature of the samples rises over time. So, for exposed carbon fiber, laser parameter E was chosen, which was a balanced option between exposure and processing time. An exposed fiber structure is not favored in the industry, since it could easily cause damage to the fibers. However, it is interesting to see how the samples would hold compared to the ones which have no fiber structure exposed.

For the titanium alloy samples, the choice of convenient parameters was less simple from the data gathered so far. There were no observable differences in roughness or large differences in wettability compared to the virgin material. Due to the change in color, the parameter setting E was chosen. Considering process time, parameter setting F was also chosen, which requires not more than five runs, but uses a lower laser power compared to settings from E.

Before the joined samples were tested, the titanium and CFRP samples were tested separately. A standard glued test

Table 4 Shear test results of the test samples

Sample	Load at failure (kgf)	Stress at failure (MPa)
Ti alloy untreated	1100	1078.73
CFRP untreated	2300	363.80
Conventional glued samples	235	3.84

sample was also tested for the sake of comparison. This had several reasons. First, the glued test sample was made to see if the gluing could be successfully performed. Second, the glued area could be chosen so that the case where the glue is actually stronger than the samples themselves can be avoided. Then the dimensions of the glued area were around 20×20 mm. The results from the tensile tests are tabulated in Table 4. The loading forces to break for both Ti and CFRP were about the same and a minimum of ten times higher than the conventional glued sample, which is a necessary condition for a proper lap-joint tensile test. For stress at failure, the thickness of the Ti and CFRP was considered and for the lap-joint glued sample the adhesion area was 20×30 mm².

Before the tensile tests on conventional glued samples were performed, the thickness of the glue was measured. For all the samples, the thickness was between 0.1 and 0.3 mm. Within this range, the thickness of the adhesive should have no influence [17].

Table 5 presents the results of the tensile strength in MPa for nine different surface conditions with associated standard deviations. The column represents different CFRP finishings and the lines different Ti finishings. Each combination was repeated four times. The combination of untreated surfaces CV and TV can be used to produce the conventional glued samples (Table 4). Table 5 presents a comparison among the possible surface conditions to produce adhesive bonded sets.

From Table 5, it can be seen that the TV performed the worst and the TE sample gave the best. The TE condition was better on joining to virginal or CE condition. However, when the composite surface was CB, both TE and TF titanium presented the same result. This is because the carbon fiber structure delaminates at around 420 kg, which is before the TE and TF samples detach from the adhesive. A similar behavior occurred in the CE samples. The main difference is that the force needed for the delamination was higher and the adhesion on the TF samples was not always strong enough to delaminate the carbon fiber. The virginal samples were not strong enough to delaminate the carbon fiber and were always the first to detach from the adhesive and showed the worst performance. As seen in the fracture presented in Fig. 6, part of the composite remained at the Ti side as a result of delamination.

Table 5 Summary of tensile strength (in MPa) for each joint combination

		Composite side		
		CV	CB	CE
Ti side	TV	5 ± 2	4 ± 2	5 ± 2
	TE	8 ± 1	7.0 ± 0.2	8.2 ± 0.2
	TF	7 ± 1	7.0 ± 0.1	6.4 ± 0.6

Fig. 6 Delamination of the carbon fiber structure for tensile coupon CB and TE. The adhesive still connects the Ti sample and the top part of the CFRP sample which was delaminated

When looking at the average adhesion strength for each sample in Table 5, one observes that for the Ti samples laser treatments E and F both provided a large increase in adhesion. The difference is larger than the average results shown because TE and TF are limited by the delamination of the carbon fiber for the laser-treated samples. When comparing the CFRP samples, the virginal material performs slightly better when looking at the average. However, only the virginal CFRP was able to use the full potential of the titanium–TE adhesion. This is because laser-driven exposed fibers delaminate around 7 MPa. Besides causing the delamination, the laser treatment can also cause other complications such as exposure or damage of the fiber structure at high energy levels, such as condition E. Although it was conjectured that an increase in contact angle usually lowered the adhesion between two glued solid surfaces, the effect of pressing one against the other seemed to hide any surface energy equalization between them.

4 Conclusions

The present work concentrates on the effect of laser surface treatment on the adhesion between CFRP composites and grade 5 Ti. Different laser intensities, and associated pulse energies, were investigated. The damage threshold for the CFRP was 5×10^8 W/cm², when the fibers were damaged by the incident intensity. For the Ti samples, the unique distinctive features belong to the coloring which varied from silver to dark gray.

The surface roughness was observed to be a function of the energy imposed. For one laser run, the roughness of the titanium increased linearly by the laser energy. For 5 or 20 runs, the laser polishing effect was noticeable in the samples. For the CFRP samples, 2.51×10^{-3} J was the maximum energy to expose the fibers before damage, and an Ra roughness of 6.11 μ m, compared to 1.80 μ m of the base material, was attained.

The wettability of the Ti surfaces did not change significantly with laser energy. However, the contact angle of

the CFRP surfaces changed widely with the experimental conditions, attaining 127.5° for the condition where $E = 2.51 \times 10^{-2}$ J. This hydrophobic behavior is due to the fine C-hairy structures resulting from fiber damage at high energy.

Lap-joined shear strength tests presented the best results when the titanium surfaces were treated using 5.03×10^{-3} J pulses for a CFRP sample treated at 5.03×10^{-4} J or 5.03×10^{-3} J or even using a virginal surface. Using a virginal CFRP surface, the critical load when the fiber delaminates was increased and thus the tensile stress attained the highest level of 8.2 MPa.

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