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Procedia CIRP 33 (2015) 458 – 463

www.elsevier.com/locate/procedia

9th CIRP Conference on Intelligent Computation in Manufacturing Engineering - CIRP ICME '14

Effect of laser and plasma surface cleaning on mechanical properties of adhesive bonded joints

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Abstract

The use of adhesives in place of traditional joining techniques as welding and riveting is becoming increasingly common in structural design. Compared to other conventional joining processes, adhesive bonding offers several advantages: including acoustic insulation, vibration attenuation, structure lightening, corrosion reduction and uniform stress distribution. These benefits can be offset if the surfaces to be bonded are not carefully cleaned, degreased and prepared. Many approaches to surface treatments based on physical or chemical modifications have been developed in the years in order to improve the surface activity. Although widely used and very efficient, these techniques present several disadvantages. Physical methods based on mechanical abrasion are supposed to extend bonding area as they increase the roughness, but cause an extensive degradation to the specimens and they are often not easy to reproduce. On the other hand, chemical treatments are typically used with the aim of modifying both morphology and chemical structure of the substrates layers, but they present serious environmental problems of waste disposal, which has moved investigations to an industrial alternative to these processes. According, the use of energetic methods of surface cleaning, such as laser and plasma cleaning processes have been recently consolidated since they are useful to modify the topmost layers of the substrates, increasing their reactivity, without affecting the bulk material properties. These treatments offer an effective and environment-friendly processing of different materials, providing strong and durable bonds with adhesives through modification of the surface morphology and functionalities. In this study, the effects of laser and low pressure plasma treatments on mechanical properties of adhesive bonded joints has been investigated and compared performing lap-shear tests.

Selection and peer-review under responsibility of the International Scientific Committee of "9th CIRP ICME Conference"
Keywords: Laser surface cleaning, Low pressure plasma, Adhesive bonding.

1. Introduction

Adhesive bonded aluminium and its alloys are widely utilized in aviation and aerospace, transport, packaging, shipbuilding, construction industries, automotive sectors and other fields. Usually, in structural applications, a bonded assembly is expected to maintain a significant load also when the components are exposed to harsh environmental conditions in its service life. For this purpose, the pre-treatment for structural bonding of aluminium alloy are often required. In order to improve adhesion different kinds of surface treatment were proposed [1]. Traditionally, solvent degreasing, and acid pickling or alkaline etching followed by conversion coating, or anodising treatment, have been used to prepare aluminium surfaces for adhesive bonding [2]. Cleaning processes using organic solvents are undesirable since they are implicated in ozone-depletion [3], with much effort put into formulating non-ozone depleting alternative solvents [4]. Also mechanical treatment, like as fluidized bed machining [5], polymers or sand blasting [6], are usually adopted in order to remove the contaminants or to control the desired level of surface roughness and joint strength [6]. However, also for this methods drawback like difficulty in process and surface extension control or material contamination, can be occur. Dry cleaning processes, like as plasma or laser treatments, represent a promising alternatives to the aforementioned methods. Plasma cleaning on polymers and metals for adhesion increasing has been studied widely [7-13]. Initial plasma-etching to clean and or 'activate' the surface is normally an intrinsic part of such plasma deposition.

Selection and peer-review under responsibility of the International Scientific Committee of "9th CIRP ICME Conference"
doi:10.1016/j.procir.2015.06.054

Low-pressure plasma treatment with the aim to improve the surface wettability, has previously been studied [14-17]. While the effectiveness of plasma treatments on the adhesive bonding of aluminium were proved in [12, 18-21]. On the other hand, despite laser cleaning treatments are an emerging technology, there are many study that demonstrate the effectiveness of this treatment in the cleaning of surfaces and/or in joint preparation [22-28].

2. Equipment, material and experimental procedures

2.1. Plasma Equipment

Plasma treatment was carried out in a radio frequency (RF) low pressure plasma. In particular, a glow discharge RF generator operating at 13.56 MHz (Gambetti Kenologia, Italy) was used. The chamber volume is approximately 5.5 l and the chamber dimensions are a diameter of 150 mm x 330 mm of length. The system operate at a pressure 0.5 mbar with the gas admitted through a needle valve. The power, as measured on the RF supply, can be selected in the range of 10-200 W. The operating conditions led to little or no significant heating of the plates on their removal shortly after the plasma was extinguished. The plasma can work with different gas. Here, standard grade Argon and Oxygen were used and a flow rate of 25 $cm^3\ min^{-1}$ was used for gas input.

2.2. Laser Equipment

The experimental tests were performed using a 30 W Master Oscillator Power Fibre Amplifier (MOPFA) and a Q-switched pulsed Ytterbium fibre laser (YLP-RA30-1-50-20-20 from IPG). The laser beam was directed by means of two galvanometer mirrors placed in a scanning head (by LASIT) and focused by a “flat field lens" onto the workpiece. In Fig 1, a scheme of the laser cleaning process is illustrated. The laser system was computer controlled, which allows the generation of the geometric patterns (Fig. 2) and the setting of the process parameters: power percentage (P%), that is the percentage of the maximum available average power; pulse frequency (F); scan speed (Ss). Table 1 shows the detailed characteristics of the laser system. Before the testing the laser source was characterised in term of average power, pulse energy and pulse power. The average power was measured using a power meter (F150A-SH thermal head and a NOVA display by OPHIR) at different power percentage and pulse frequency. And the pulse energy and the pulse power were calculated as the average power/pulse frequency and pulse energy/duration ratio. In Fig. 3 the pulse energy and the pulse power are reported as a function of the power percentage, at different pulse frequency. It can be noticed that both Pe and Pp increase as frequency increases.

2.3. Material

The investigated material was the Aluminium alloy 6061-T6 (al6061, UNS A96061; ISO AlMg1SiCu) in form of rolled sheets 1.6 mm in thickness. The al6061 is a precipitation hardening aluminium alloy, containing magnesium and silicon as its major alloying elements, usually adopted in aircraft structures, automotive parts, yacht and boats construction, and scuba equipments construction thanks to the its workability, weldability and corrosion resistance.

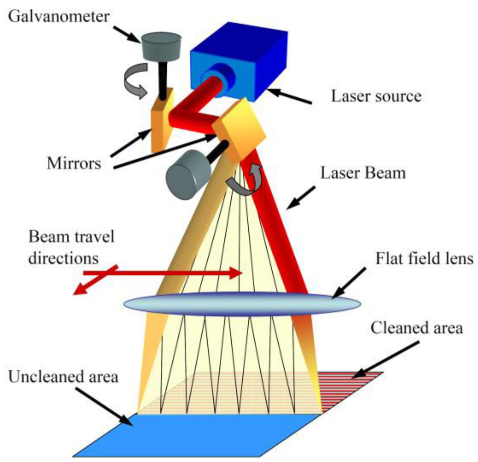

Fig. 1. Scheme of the laser cleaning process.

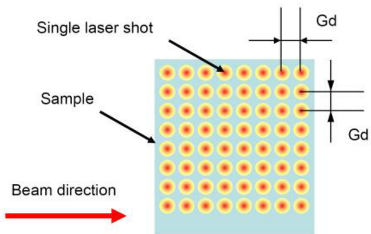

Fig. 2. Illustration of the grid dimension significance.

Table 1: Laser system characteristics.

Characteristics	Symbol	Value	Unit
Wavelength	λ	1064	[nm]
Nominal average power	Pm	30	[W]
Maximum pulse energy*	P_e	1	[mJ]
Maximum peak power*	P_p	20	[kW]
Pulse frequency	F	30÷80	[kHz]
Pulse duration	D_r	50	[ns]
Scan speed	Ss	1 ÷ 5000	[mm/s]
Mode	TEM	00	--
	M^2	1.2 ÷ 1.5	--
Focused spot diameter **	--	≈ 100	[µm]
Beam motion	by galvo mirror scanner		--
Working area**	100 x 100		[mm^2]
Power consumption***	160		[W]

*At Pm = 30 W and F = 30 kHz.
** For a “Flat field” lens, with a focal length of 160 mm.
*** At the max. power output.

2.4. Experimental procedures

To characterize both the technique and to detect which factors affect the bonding efficiency, two experimental planes were developed according to the Design of Experiment (DOE) technique. The first experimental plane was developed for the Plasma treatments. It was a 2^3 full factorial design. This plane permits to analyse the main factor, the two and three-factor interactions without any confounding. On the basis of background experience on other materials and various references [7-14, 19-21], the following control factors were adopted: the gas (Gas), the power (P) and the treatment time (Tt). For each treatment, four replications were carried out. In Table 2, the adopted process conditions are summarised. Before the test, the sample were rinsed by acetone in order to avoid the contamination of the plasma chamber.

In the case of Laser treatment, a 2^2 full factorial design was selected for the experimental design. The presence of pure quadratic terms was checked by replicating 10 times the centre point. Such a design will help to analyse the main factor, the two and three-factor interactions without any confounding. As control factor the pulse power and the grid dimension were adopted. This because in pulsed laser, pulse energy and power play a central role since they determine the laser beam-material interaction mode, amount of worked material, and thermal effect [29-32]. On the other hand, in order to fill the sample area, a square grid of dimples (where each dimple was created by a single laser shot, Fig. 2) were used thanks to a proper combination of the pulse frequency and scan speed values. In Fig. 4 examples of the dimples at different process condition are reported. Accordingly, this study focuses on pulse power and the grid dimension. In Table 3, the adopted process conditions are summarized, while in Table 4 the complete design for the laser treatments is reported.

In order to investigate the effect of the treatments, single lap joint were produced bonding the sample using a 2-component epoxy adhesive. A bond area of 12.5 mm × 25.4 mm and a nominal bondline thickness of 0.5 mm were adopted. The sample were realized and tested using the same procedure adopted in [6-8, 33], in agreement with ISO 4587 standard. In Fig. 5, an image of the specimen is reported. The assembled joints were left for 1 week at room temperature to completely adhesive cure before performing the mechanical tests. In order to discriminate the treatment effect, the ultimate shear stress (τmax), calculated as the ratio between the maximum load and the bonded area, was used. Furthermore, untreated specimen as well as specimen only cleaned with acetone were performed and tested for comparison.

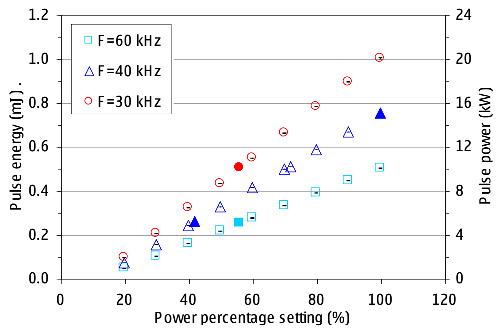

Fig. 3. Pulse energy (Pe) and pulse power (Pp) as a function of power percentage setting. The adopted experimental conditions are highlighted by the filled symbols.

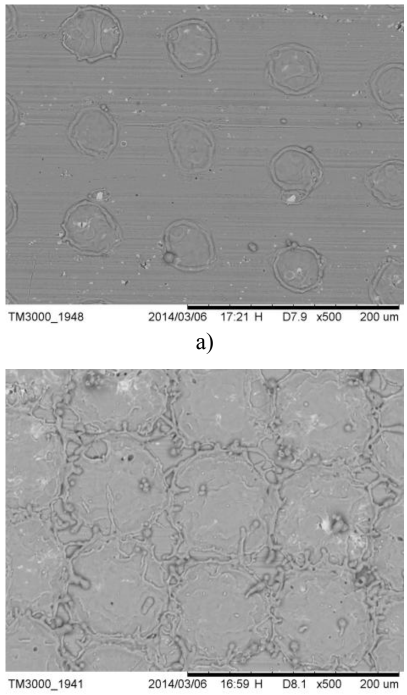

Fig. 4. Examples of the dimples present on the sample surface after fibre laser cleaning a) Pp=5 kW, Gd=100 µm, b) Pp=15 kW, Gd=100 µm. The images were done at 500x.

Table 2: Control factors and levels adopted for Plasma treatment.

Control factors	Labels	Low (−)	High (+)	Unit
Gas	Gas	Argon	Oxygen	
Nominal power	P	100	200	[W]
Treatment time	Tt	60	300	[s]

Table 3: Control factors and levels for Laser treatment.

Control factors	Labels	Low (−)	Midle (0)	High (+)	Unit
Pulse power	Pp	5	10	15	[kW]
Grid dimension	Gd	50	75	100	[µm]

Table 4: Experimental design matrix for laser treatment with coded and values.

Std. Order	Replication	Coded Variables		Actual variables	
		Pp	Gd	Pp [kW]	Gd [µm]
1	4	-1	-1	5	50
2	4	1	-1	15	50
3	4	-1	1	5	100
4	4	1	1	15	100
5	10	0	0	10	75

The ANOVA method was applied in order to test the statistical significance of the main effects and the two-factor interactions for τmax. The analysis was carried out at a 95 % confidence level (α = 0.05). For both the experimental plane diagnostic checking was successfully performed via graphical analysis of residuals in agreement with what reported in [34]. However, these results were not reported here for sake of briefness.

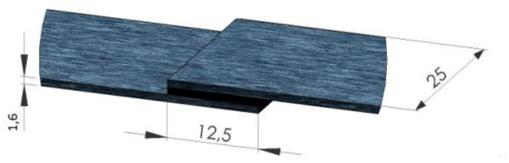

Fig. 5. Geometry and dimension of the adopted specimen.

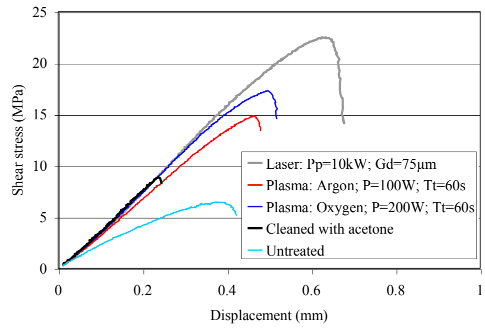

Fig. 6. Typical behaviour of shear stress vs displacement, for different treatments.

3. Experimental results and discussion

In Fig. 6, the typical response in term of shear stress vs displacement for Argon and Oxygen plasma, and Laser are compared to the untreated and only cleaned with acetone samples response. From the figure it is possible to observe that, excluding the untreated sample, all the curves show a similar slope in the first part. This is due to the different failure modes: indeed, while the untreated samples have a fully de-adhesive failure mode, the samples subjected to any kind of pre-treatment present partially de-cohesive failure modes, but never arriving to the complete de-cohesive fracture. Fig. 7 highlights that both plasma treatments have almost the same failure behaviour, while the laser treated specimens failure confirm the good results in terms of shear stress. In Table 5, the ultimate shear stress (τmax) obtained by plasma and laser treatments are compared with those obtained by untreated and acetone cleaning in term of maximum, minimum and average values together to the standard deviation. From the table, both the treatments produce higher values compared to the untreated or acetone cleaned samples. The values of the two plasma treatments are very close. Furthermore, the laser treatment shows the highest values of the τmax in term of maximum, minimum and average values.

In Tables 6 and 7 the ANOVA results are reported for the Plasma and Laser treatment respectively. On the basis of assumptions made, a control factor, or a combination of control factors, is statistically significant if the *p*-value is less than 0.05. Then the significant effects are highlighted by bolt underlined text. From Table 6, no factor affecting the plasma treatment are present. On the contrary, for the laser treatment, Table 7, the ANOVA indicates all the factors and their interaction. In order to understand the effect of the significant factors, the main effects plot and the interaction plot are reported in Fig.s 8-10. In the figures the significant factors are highlighted using continuous line. In Fig. 8 the main effects plot for Plasma treatment are reported. It is interesting to note that, despite for the plasma treatment do not result statistically significant factors, the τmax tends to increase increasing the power or the treatment time, or, again, changing the treatment gas from argon to oxygen.

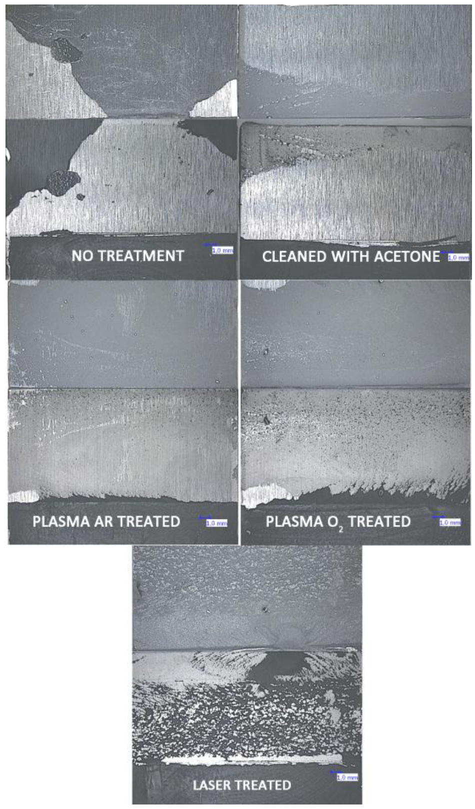

Fig. 7 Typical failure surfaces for different treatments.

Table 5: Ultimate shear stress for the different treatments.

Treat. / **Value**	**Untreated**	**Cleaned (Acetone)**	**Plasma**		**Fiber Laser**	**Unit**
			Ar	**O_2**		
Max	6,45	9,63	17,91	19,18	24,36	[MPa]
Min	5,12	7,38	10,51	10,39	12,28	
Average	5,77	8,42	14,39	15,13	19,35	
St. Dev.	0,55	1,02	2,14	2,57	2,95	

Table 6: ANOVA table for Plasma treatment.

Source	D.o.F	Seq. SS	Adj. SS	Adj. Ms	F-value	p-value
Gas	1	6,647	6,647	6,647	1,33	0,260
P [W]	1	9,113	9,113	9,113	1,82	0,190
Tt [s]	1	2,931	2,931	2,931	0,59	0,452
Gas*P	1	0,237	0,237	0,237	0,05	0,829
Gas*Tt	1	3,325	3,325	3,325	0,66	0,423
P*Tt	1	11,496	11,496	11,496	2,30	0,143
Gas*P*Tt	1	1,855	1,855	1, 855	0,37	0,548
Error	24	120,116	120,116	5,005		
Total	31	155,721				

Table 7: ANOVA table for Laser treatment.

Source	D.o.F	Seq. SS	Adj. SS	Adj. Ms	F-value	p-value
Pp [kW]	1	33,70	33,70	33,703	15,79	**0,001**
Gd [µm]	1	74,58	74,58	74,579	34,95	**0,000**
Pp*Gd	1	22,75	22,75	22,748	10,66	**0,004**
Error	21	44,82	44,82	2,134		
Total	25	217,12				

Although plasma treatment times were similar to those reported by some references on the plasma treatment of aluminium alloys [16, 17], the insensitivity of plasma treatment response to the factor changing demonstrates that the plasma treatment exhausts its effect (i.e. saturates) for time lower than 60s. However, the effect of the plasma is confirmed by the fact that the samples treated by plasma shown a τmax higher than the untreated or acetone cleaned samples. The main effects plot and significant two way interaction for laser treatment are reported in Fig.s 9-10. From Fig. 9A an increase of pulse power produces an increase of τmax. The opposite occurs when the grid dimension increases, Fig. 9B. Both the effects were expected, because to the increase of the pulse power it corresponds an increase of the effective spot dimension (i.e. the dimples printed on the surface) and then the treated area, as visible comparing Fig. 4a with Fig. 4b. On the other hand, the increase of the grid dimension, produces a reduction of the number of pulse per unit area and then a reduction of the treated area. This effect is reduced if a large dimple is obtained, such as in the case of high pulse power, as visible in the interaction plot, Fig. 9. The last sentence justifies the indication of ANOVA for the interaction effect. Furthermore, it explains also the center point behaviours and in particular the high τmax values obtained in correspondence of it, red points in Fig. 9. Indeed, when the factors are set at the centre point, the combination, in term of dimple dimension and step, is such as to permit the whole surface treatment, as visible in Fig. 11.

Finally, in Table 8, the treatment time for laser cleaning of 100x100 mm^2 is reported. The data are referred to the condition adopted here. The table allows to highlight some differences with respect to the plasma treatment. Despite treatment times between laser and plasma are very similar, the plasma treatment allows to treat more components at once. This is an advantage when the treatment of a large number of small components is required. On the contrary, if components of remarkable dimension must be treated, the plasma treatment application is limited by reactor size. This limitation does not exist in the case of the laser, that can be used also on large structures if a portable equipment is adopted.

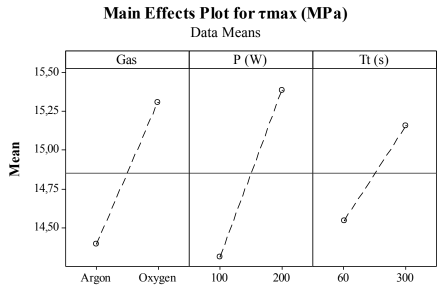

Fig. 8. Main effects plot for Plasma treatment.

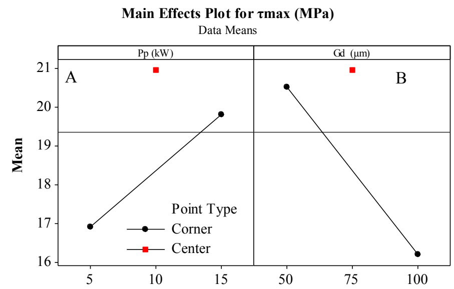

Fig. 9. Main effects plot for Laser treatment.

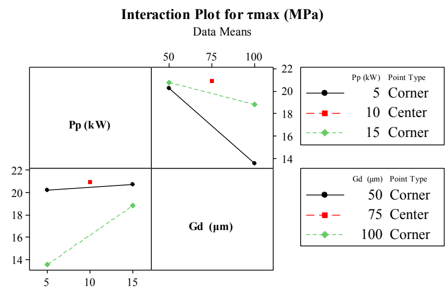

Fig. 10. Interaction plot for Laser treatment.

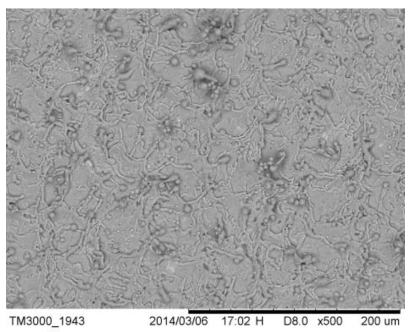

Fig. 11. Surface appearance obtained at Pp=10 kW, Gd=75 µm.

Table 8: Treatment time in second for laser cleaning of 100x100 mm^2.

	Pulse power [kW]		
Gd [µm]	**5**	**10**	**15**
50	**67**	**np**	**100**
75	**np**	**60**	**np**
100	**25**	**np**	**25**

np= not performed.

Conclusions

Plasma and Laser surface treatments were performed on Al6061 alloy in order to improve the adhesion of bonded joints. The effectiveness of the treatments were discriminate using single lap joint test. Untreated specimen and specimen cleaned with acetone were produced and tested as reference. Two factorial designs were developed for the experiments. ANalysis Of VAriance was applied in order to test the statistical significance of the main effects and the two-factor interactions. The obtained results showed that either the treatments, compared to the reference samples, are able to increase the joint strength. The laser treatment showed the highest values: up to the double compared to the reference. Though, for this technique the treatment time depend on the surface dimension. On the other hand, plasma treatments allow treating more components at once. However, when compared to the acetone cleaning, it yields an increase in strength of only 1.75 fold.

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