

Surface Modification of Titanium Alloy with Laser Treatment

Nassier A. Nassir, Robert Birch, D. Rico Sierra, S. P. Edwardson, G. Dearden, Zhongwei Guan

Abstract—The effect of laser surface treatment parameters on the residual strength of titanium alloy has been investigated. The influence of the laser surface treatment on the bonding strength between the titanium and poly-ether-ketone (PEKK) surfaces was also evaluated and compared to those offered by titanium foils without surface treatment to optimize the laser parameters. Material characterization using an optical microscope was carried out to study the microstructure and to measure the mean roughness value of the titanium surface. The results showed that the surface roughness shows a significant dependency on the laser power parameters in which surface roughness increases with the laser power increment. Moreover, the results of the tensile tests have shown that there is no significant dropping in tensile strength for the treated samples comparing to the virgin ones. In order to optimize the laser parameter as well as the corresponding surface roughness, single-lap shear tests were conducted on pairs of the laser treated titanium stripes. The results showed that the bonding shear strength between titanium alloy and PEKK film increased with the surface roughness increment to a specific limit. After this point, it is interesting to note that there was no significant effect for the laser parameter on the bonding strength. This evidence suggests that it is not necessary to use very high power of laser to treat titanium surface to achieve a good bonding strength between titanium alloy and the PEKK film.

Keywords—Bonding strength, laser surface treatment, PEKK, titanium alloy.

I. INTRODUCTION

FIBRE metal laminates (FMLs) are high performance structures, developed by the Delft University of Technology. FMLs consisting of alternating stacking layers of fiber reinforced composites and metal alloy offer a great promise as fatigue resistance materials. Based on the reinforcement of the polymer composite, these laminates can be GLARE (glass reinforced metal laminate), CARALL (carbon reinforced metal laminates), and ARALL (Aramid reinforced metal laminates). GLARE FMLs are the most commonly used to manufacture the upper fuselage of the A380 Airbus aircraft [1], [2]. FMLs combine the high toughness and impact energy offered by metals and the high

specific properties associated with fiber reinforced composite. It has been demonstrated that FMLs have superior in-plane tensile strength and tension fatigue properties compared to those for aluminum alloys [3]. In recent years, a number of researchers have investigated the response of aerospace fiber laminates under different load conditions [4]-[7]. The mechanical properties of FMLs of Aluminum layers and tough glass fiber reinforced polypropylene was investigated by Reyes et al. [4]. Their results indicated that incorporating surface roughness on aluminum layers and inserting resin film at the interface between the aluminum and composite plies lead to increase the fracture energy of these laminates. The response of glass fiber reinforced epoxy and aluminum FMLs under low velocity impact was investigated by Jan et al. [6]. The results showed that the impact resistance of FMLs is high compared to the plain composite. The results also indicate that the specific energy absorption of FMLs can be increased by increasing the plies number of metal and composite. Titanium-based FMLs combine the advantage of titanium sheets and high temperature fiber reinforced composites. These hybrid laminates offer the good mechanical advantages of the traditional fiber metal laminates and it can be used at high temperature applications. However, the bonding strength between the titanium and the polymeric materials is still a major challenge which limit their applications and needs to be solved [8]. The literature showed that the nature of the surface treatment of titanium has a significant effect to improve the bonding strength of these hybrid laminates. Recently, different surface treatments were conducted to enhance the surface roughness of the titanium alloy prior to bonding, and these being mechanical, chemical, electrochemical, plasma, and laser treatments [8]. Although these pre-treatment processes showed good bonding behavior, it is not environmental friendly. Laser surface treatment demonstrates a good joining strength resulted from good surface roughness, formation of a thin oxide layer, good surface cleaning and modification. The aim of this study is to investigate the influence of the laser treatment on the titanium foils in terms of surface roughness, residual tensile strength and bonding strength.

II. EXPERIMENTAL PROCEDURE

A. Manufacturing Procedure

The lap shear specimens examined in this study were based on 0.14 mm thick layers of titanium 15-3-3- β alloy foil from TICOMP (California, USA) and A 50 μ m film of PEKK (ARKEMA, France) was placed between foils of titanium alloy to ensure good bonding between the constituent materials. Prior to manufacturing, titanium alloy sheets were

Nassier A. Nassir is with the School of Engineering, University of Liverpool, Liverpool L69 3GH, UK and Department of Materials Engineering, University of technology, Baghdad, Iraq, (phone: 447417413264; e-mail: N.A.N.Altmtteri@liv.ac.uk).

Robert Birch is with the School of Engineering, University of Liverpool, Liverpool L69 3GH, UK (e-mail: rsb123@liverpool.ac.uk).

D. Rico Sierra, S. P. Edwardson, G. Dearden are with Laser Engineering Group, School of Engineering, University of Liverpool, L69 3GQ, UK

Zhongwei Guan is with the School of Engineering, University of Liverpool, Liverpool L69 3GH, UK, and from School of Mechanical Engineering, Chengdu University, Chengdu 610106, China (e-mail: zgguan@liverpool.ac.uk).

cut as sheet and subjected to laser treatment to enhance the surface roughness as well as the interface bonding strength.

The testing specimens were manufactured by stacking a pair of surface treated titanium stripes which adhesively bonded using PEKK film (0.1 mm) with an overlapping area of $23 \times 5 \text{ mm}^2$ as shown in Fig. 1. Then, the samples were inserted between two metal sheets. The mold was then heated in a Meyer hydraulic hot press to 330°C at a heating rate of about $3^\circ\text{C}/\text{min}$, maintained at this temperature for 30 min before cooling to room temperature. A pressure of 2 bars was applied to the laminates during the processing cycles.

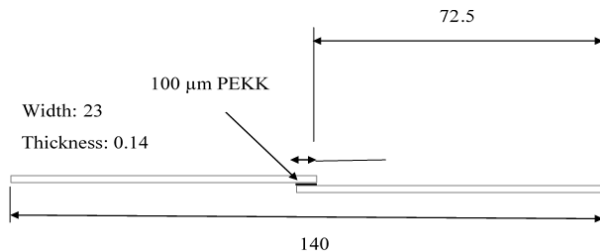


Fig. 1 Lab shear specimens (dimensions in mm)

B. Laser Surface Treatment

Laser pre-treatment of titanium surface was carried out using different power parameters of laser to investigate the influence of these parameters on the surface roughness, residual tensile strength of titanium alloy and metal-resin bonding strength. Firstly, titanium alloys were cut to required sizes and cleaned with acetone previously to the laser treatment. A nanosecond pulsed laser (SPI 20W G4 HS L Type) was used to modify the surface microstructure of the material. The laser pulsed system works with 1064 nm wavelength, a variable pulse width of 9-200 ns, 20 W of maximum output power, and a pulse repetition rate of 25-500 kHz. The spot size of the focused beam is $45 \mu\text{m}$. A line pattern microstructure was created; the space between the lines was set as $29 \mu\text{m}$. The processing area was treated with the parameters on Table I. The parameters were used in order to create overlap between the laser pulses modifying the roughness of the surface, an example of the scanning technique is shown in Fig. 2. After that, surface was characterized by an optical profiling system (Wyko NT1100) to measure the mean surface roughness. Fig. 3 shows the influence of laser power on the surface roughness of titanium alloy.

TABLE I LASER TREATMENT PARAMETERS USED FOR TITANIUM ALLOY	
Laser fluence	4.09, 4.54, 5 and $5.45 \text{ J}/\text{cm}^2$
Repetition Rate	70 kHz
Pulse Length	200 ns
Scan Speed	2.380 mm/s

C. Tensile Test

Tensile tests were conducted to investigate the influence of laser treatment on the residual tensile strength of titanium alloy. The tests were undertaken using Instron 3369 testing

machine. An extensometer with 25 mm gauge length (GL) was attached to the coupons in the middle to measure the sample displacement. The specimen's geometry and dimensions are shown in Fig. 4. Tests were undertaken at a constant crosshead speed of 0.5 mm per minute.

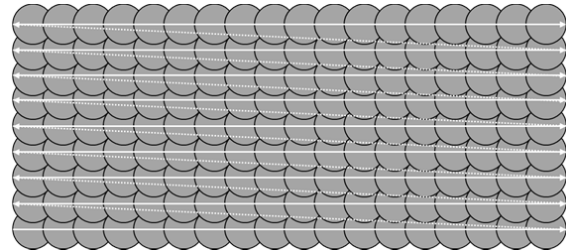


Fig. 2 Scanning path of the laser, horizontal distance between pulses of $34 \mu\text{m}$, and a vertical distance between lines of $29 \mu\text{m}$



Fig.3 The effect of the parameter of the laser treatment on the surface roughness of titanium alloy

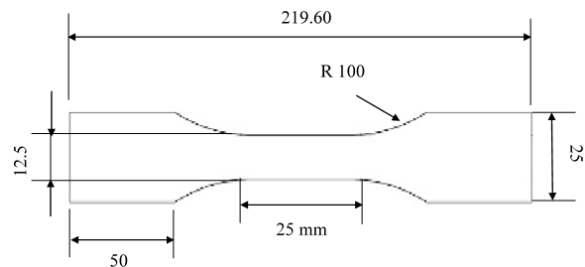


Fig. 4 Specimen geometry (dimensions in mm) [9]

D. Lab Shear Test

Single-lab shear tests were performed to examine the shear strength between PEKK film and modified surface titanium alloy. Here, shear tests were conducted on the specimens described in Fig. 1. The maximum shear (bonding) was measured using an Instron model 3369 universal testing machine equipped with a load cell with capacity of 50 kN, as shown in Fig. 5. The tests were carried out at a constant loading rate of 1 mm/min. Three samples were tested for each laser parameter and the average values were obtained.



Fig. 5 Single-lab shear test setup

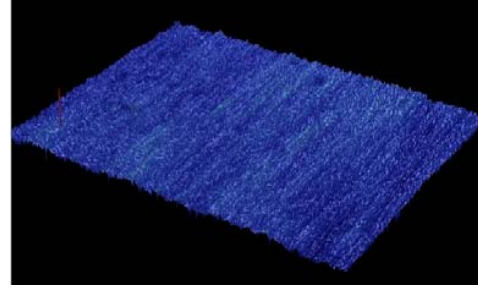
III. RESULT AND DISCUSSION

The influence of the laser treatment with various power parameters on creating rough structure on the surfaces of the titanium alloy samples was illustrated in Table II. It can be seen that a relatively smooth surface of 0.309 microns without hierarchical structure was observed on the as-received samples compared to 1.72 microns to those treated with laser fluence of 5.45 J/cm². Here, during laser process, the materials surface will be moved and removed by the laser throughout melting and evaporation, resulting significant increment of the surface roughness [10]. In the other word, the change in the surface roughness is due to the laser interaction with the material, this creates a microstructure with material removed due to the laser ablation and the re-deposition of the molten material due to the thermal component of the nanosecond pulse. The melting of the material creates a structure in the borders of the spot size helping to increase the roughness of the material. Increasing the fluence on the laser increases the ablation rate and the amount of molten material in the surface.

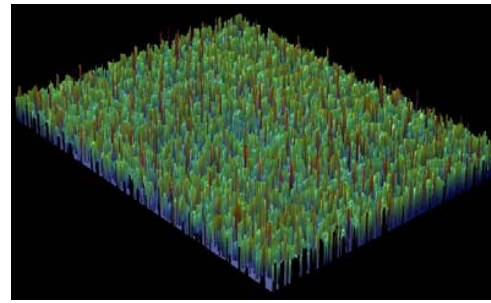
TABLE II
MEAN ROUGHNESS OF TITANIUM SURFACE AGAINST LASER FLUENCE

Laser fluence (J/cm ²)	Mean Roughness (μm)
Untreated Surface	0.309
4.09	1.19
4.54	1.43
5	1.63
5.45	1.72

The measurement of the surface roughness was performed on separate specimens for each laser power parameters to characterize the surface texture of pristine titanium foils and after laser treatment using different parameters of laser fluence. Fig. 6 shows the three dimensional (3D) profile of titanium foils treated under different laser power parameters. Clearly, different rates of laser power led to different roughness on these sample surfaces. Here, higher laser power led to coarser texture on the sample surface compared to as-received one. As mentioned previously with increasing the laser power, the material removal and the depth of the micro pits increased.



(a)



(b)

Fig. 6 3D profile of a titanium surface under different power parameters, (a) as received surface, (b) surface treated with laser fluence of 4.54 J/cm²

The next part of this study was to investigate the influence of the laser surface roughness on the residual strength of the titanium alloy. Here, tensile tests were undertaken on titanium (0.14 mm thick) foils treated with various laser fluences of 0, 4.09, 4.54, 5 and 5.45 J/cm². Here, zero value corresponds to the virgin specimens. Fig. 7 shows the variation of the tensile strength as a function of the surface roughness. An examination of this bar chart indicates that the tensile strength values are varying between 1122 MPa for untreated specimens with surface roughness of 0.309 micrometers and 1069 MPa for specimens with roughness of 1.72 micrometers. The evidence presented in Fig. 7 indicates that there is no significant change in tensile strength after laser treatment. This is a useful observation, suggesting that laser treatment parameters can be optimized based on better bonding strength between modified titanium alloy and PEKK film.

Fig. 8 shows the variation of the metal-resin bonding strength with surface roughness at different laser power parameters. Clearly, the curve can be divided into two regions, i.e. the linear proportional one (I) in which the bonding strength between the titanium alloy and PEKK film increased with increasing the surface roughness increment to a specific roughness due to formation of the microporous structure on the treated surface which filled with the resin resulting good metal-resin bonding strength. After this point, it is interesting to note that there is no significant effect for the laser power parameters on the bonding strength (II). The above results suggest that the surface treatment with laser influence of 4.54 J/cm² seems to be an optimum parameter in which good

bonding strength can be achieved. Therefore, this parameter will be used to treat all the titanium foils investigated in this study.

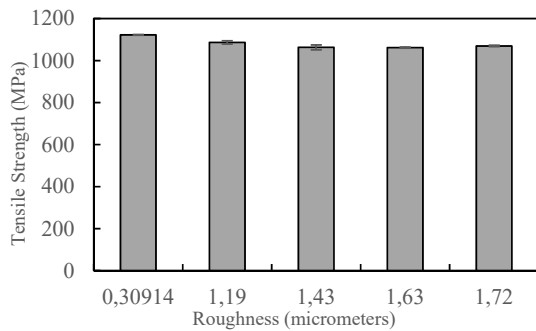


Fig. 7 Surface roughness versus tensile strength of titanium alloy foils

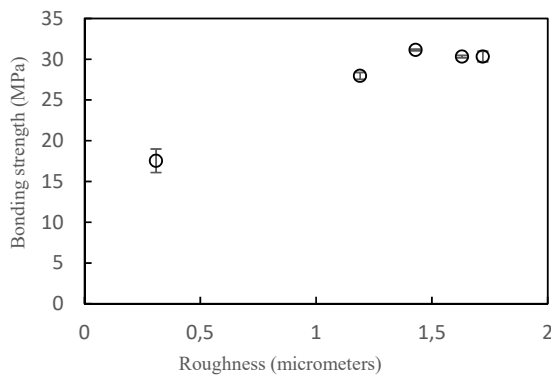


Fig. 8 The value of the shear strength as a function of the surface roughness for the titanium alloy

IV. CONCLUSION

The influence of the laser treatment parameters on the residual strength of titanium alloy and on the bonding strength between titanium alloy and PEKK film has been investigated. Tests on titanium alloys that treated with different laser power parameters under tensile loading showed that there was a low reduction in the residual tensile strength between 3 and 5% for laser treated specimens. For the metal-resin adhesion tests, laser power parameter of 4.54 J/cm^2 was selected to give an optimum bonding strength between the titanium alloy and plain composite.

ACKNOWLEDGMENT

This work is funded by the higher committee for education development in Iraq (HCED) (prime minster office), which is greatly appreciated. The authors would like to thank ARKEMA Company for providing the PEKK materials.

REFERENCES

- [1] F. D. Morinière, R. C. Alderliesten, and R. Benedictus, "Modelling of impact damage and dynamics in fibre-metal laminates - A review," *Int. J. Impact Eng.*, vol. 67, pp. 27–38, 2014.
- [2] P. Cortés and W. J. Cantwell, "Fracture properties of a fiber-metal laminates based on magnesium alloy," *J. Mater. Sci.*, vol. 39, pp. 1081–1083, 2004.
- [3] A. Vlot, "Low-velocity impact loading on fibre reinforced aluminium laminates (ARALL and GLARE) and other aircraft sheet materials," Delft University of Technology, 1993.
- [4] G. V. Reyes and W. J. Cantwell, "The mechanical properties of fibre - metal laminates based on glass fibre reinforced polypropylene," vol. 60, pp. 2–6, 2000.
- [5] J. G. Carrillo and W. J. Cantwell, "Mechanical properties of a novel fiber-metal laminate based on a polypropylene composite," *Mech. Mater.*, vol. 41, pp. 828–838, 2009.
- [6] J. Fan, W. Cantwell, and Z. Guan, "The low-velocity impact response of fiber-metal laminates," *J. Reinf. Plast. Compos.*, vol. 30, pp. 26–35, 2011.
- [7] J. Zhou, Z. W. Guan, and W. J. Cantwell, "The influence of strain-rate on the perforation resistance of fiber metal laminates," *Compos. Struct.*, vol. 125, pp. 247–255, 2015.
- [8] P. Molitor, V. Barron, and T. Young, "Surface treatment of titanium for adhesive bonding to polymer composites: a review," *Int. J. Adhes. Adhes.*, vol. 21, pp. 129–136, 2001.
- [9] E. Li and W. S. Johnson, "An Investigation into the Fatigue of a Hybrid Titanium Composite Laminate," *J. Compos. Technol. Res.*, vol. 20, pp. 3–12, 1998.
- [10] V. D. Ta, A. Dunn, T. J. Wasley, J. Li, R. W. Kay, J. Stringer, P. J. Smith, E. Esenturk, C. Connaughton, and J. D. Shephard, "Laser textured superhydrophobic surfaces and their applications for homogeneous spot deposition," *Appl. Surf. Sci.*, vol. 365, pp. 153–159, 2016.