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Влияние лазерного текстурирования поверхности титанового сплава на адгезионную прочность клеевых соединений

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В работе рассмотрены вопросы, связанные с влиянием лазерного текстурирования поверхности титанового сплава на характеристики клеевого соединения «титан – углепластик». Иттербиевым импульсным волоконным лазером на поверхности титанового сплава были созданы текстуры с линейной структурой (0°-0° и 90°-90°) и сетчатой структурой (0°-90°, $\pm 30^\circ$, $\pm 45^\circ$, $\pm 60^\circ$). Были определены значения шероховатости поверхности в двух перпендикулярных направлениях и сделаны микрошлифы, по которым можно охарактеризовать морфологию поверхности титанового сплава. Для определения адгезионной прочности соединения, между собой склеивались образцы с одинаковой текстурой поверхности. Образцы склеивались по ОСТ 1-90281-86. Склеивание проводилось в течение 24 ч после лазерной обработки поверхности. Перед склеиванием обработанная поверхность очищалась изопропиловым спиртом. Площадь клеевого соединения S = 300 мм². В качестве адгезива использовался трехкомпонентный клей ВК-9 на основе эпоксидной и полиамидной смолы. Лазерная обработка поверхности титановых сплавов увеличивает прочность клеевого соединения более чем на 70 % относительно необработанной поверхности. Это может свидетельствовать о том, что главными механизмами повышения прочности клеевого соединения являются увеличение площади контакта поверхности и адгезива, а также химическая модификация, которая активирует поверхность. Текстура обработки в меньшей степени влияет на адгезионную прочность при условии одинаковой удельной поверхностной энергии лазерной обработки. При лазерной обработке стоит уделять большое внимание выбору текстуры поверхности, потому что определенные текстуры могут дать прирост прочности на 20–30 %. Если тип нагрузки в элементах ферменных конструкций известен, то лучше использовать линейные текстуры, направленные перпендикулярно направлению нагрузки (для сдвига – текстура 0°-0°; для кручения – текстура 90°-90°). При смешенных нагрузках лучше использовать сетчатые структуры $\pm 30^{\circ}$, $\pm 45^{\circ}$, $\pm 60^{\circ}$, которые сопротивляются нагрузкам в двух направлениях.

Ключевые слова: лазерное текстурирование, повышение прочности клеевого соединение, титановые сплавы.

The effect of laser texturing of the surface of a titanium alloy on the adhesive strength of adhesive joints

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The paper examines issues related to the influence of laser texturing of the surface of a titanium alloy on the characteristics of the titanium-carbon fiber adhesive joint. Using an ytterbium pulsed fiber laser, textures with a linear structure (0°–0° and 90°–90°) and a mesh structure (0°–90°, $\pm 30^{\circ}$, $\pm 45^{\circ}$, $\pm 60^{\circ}$) were created on the surface of a titanium alloy. The surface roughness values in two perpendicular directions were determined, and microsections were made, which can be used to characterize the surface morphology of the titanium alloy. To determine the adhesive strength of the joint, samples with the same surface texture were glued together. The samples were glued together according to OST 1-90281–86. Bonding was carried out within 24 hours after laser surface treatment. Before gluing, the treated surface was cleaned with isopropyl alcohol. Adhesive joint area $S = 300 \text{ mm}^2$. Three-component adhesive VK-9 based on epoxy and polyamide resin was used as an adhesive. Laser surface treatment of titanium alloys increases the strength of the adhesive joint by more than 70 % relative to the untreated surface. This may indicate that the main mechanisms for increasing the strength of an adhesive joint are an increase in the contact area between the surface and the adhesive, and chemical modification that activates the surface. The processing texture has a lesser effect on the adhesive strength, provided that the specific surface energy of the laser processing is the same. When laser processing, you should pay great attention to the choice of surface texture, because certain textures can give an increase in strength by 20–30 %. If the type of load in the truss load elements is known, then it is better to use linear textures directed perpendicular to the direction of the load (for shear – texture $0^{\circ}-0^{\circ}$; for torsion – texture $90^{\circ}-90^{\circ}$). For mixed loads, it is better to use mesh structures $\pm 30^{\circ}$, $\pm 45^{\circ}, \pm 60^{\circ}$, which resist loads in two directions.

Keywords: laser texturing, increasing the strength of the adhesive joint, titanium alloys.

Introduction

In modern spacecraft (SC), truss structures are widely used, which provide high strength and rigidity of the product with minimal weight. One of the key tasks in the development of such structures is to achieve optimal mechanical characteristics and geometric accuracy of products to ensure safe and efficient functioning of the SC. To solve this problem in the field of SC production, new technologies and materials for the creation of truss structures with improved characteristics are being developed [1–3].

For example, in the Millimetron space observatory, the main mirror truss structure consists of a set of carbon fiber rods connected in nodal elements (fittings) made of VT6 titanium alloy [4]. The design diagram is shown in Fig. 1. The nodal elements 3 are manufactured using selective laser melting (SLM) technology, then carbon fiber rods 1 with titanium end pieces 2 glued into them are attached to these nodes.

The biggest problem encountered in the manufacture of this unit is related to ensuring a reliable connection when gluing the carbon fiber rod to the titanium tip. This is a common problem for structures in which it is necessary to make a composite-to-metal connection [5; 6].

Titanium alloys, from which the tips are made, are difficult to glue materials, due to an amorphous oxide film on their surface, which does not allow the adhesive to form a strong bond with the titanium surface. As a result, in order to increase the strength of the adhesive joint, the surface of the titanium alloy must be preprocessed before gluing [7]. Traditional methods for increasing the strength of the carbon fiber – titanium adhesive joint are preliminary mechanical or electrochemical treatment of the

titanium alloy surface [8; 9] or a combined method of mechanical and chemical treatment [10]. Mechanical treatment (grinding, turning, milling) increases the contact area between the metal surface and the adhesive, and chemical treatment transforms the amorphous oxide film into a crystalline oxide layer with high valence, which provides better adhesion to the adhesive.



Рис. 1. Ферменная конструкция КА: *I* – углепластиковый стержень; *2* – титановая законцовка, вклеенная в стержень; *3* – узловой элемент, изготовленный технологией SLM; *4* – фиксатор законцовки в узловом элементе

Fig. 1. Truss structure of the spacecraft:
l – carbon fiber rod; 2 – titanium tip glued into the rod; 3 – nodal element manufactured by SLM technology; 4 – end clamp in the nodal element

In addition, alternative methods of surface preparation are being developed, such as treatment with concentrated energy sources (plasma flows, electron beams, laser radiation), application of coatings (including electrochemical, gas-thermal, etc.), and ultrasonic surface treatment [7].

With the development of modern technology, lasers are increasingly used in surface processing in mechanical engineering along with other highly concentrated energy sources, including for welding, cutting, and the creation of functional surface structures (FSS) [11].

In industrial production, laser processing is a more economical and environmentally friendly compared to mechanical and electrochemical processing. In addition, laser processing is easy to implement when automating the process [12; 13]. However, the technological modes of laser preparation and the factors affecting the quality of the adhesive joint have not been sufficiently studied at present, which prevents its widespread implementation in production. An increase in the strength of the adhesive joint due to laser processing of the metal surface is a combination of several factors, such as an increase in the contact area between the surface and the adhesive, a change in the chemical composition of the surface, and the creation of a complex surface relief consisting of grooves and microcavities into which the glue gets and creates a mechanical "locking" effect. Thus, the study of the effect of laser processing on the formation of FSS and the determination of surface processing modes before gluing is the goal of this work.

The following main parameters of laser processing, which affect the adhesive properties of the metal surface, can be distinguished: laser power, processing speed and frequency, processing texture.

Surface texture is a local deviation of the surface from a perfectly flat surface. The measure of surface texture is usually defined in terms of its roughness, waviness, and shape. Laser processing can create such texture elements as microgrooves, microholes, micropillars, porous and hierarchical structures, and surface ripples [11]. Various combinations of these elements create an unlimited number of surface textures that can be created by laser processing.

The concept of "processing texture" includes the following parameters: the step between the lines of the laser movement trajectory, the angle between these lines, the number of laser passes along one line, the overlap coefficient. The totality of all laser processing parameters affects the surface energy density of the laser E_p , which determines the macro- and microstructure of the surface, its chemical and phase composition, surface wettability, etc.

In [14], we investigated the effect of laser processing of the titanium alloy surface on the strength properties of the adhesive joint under shear. We identified processing modes for the most characteristic type of texture – unidirectional, across the shear direction.

Depending on the laser metal processing program, it is possible to create many different surface textures that will affect the strength of the adhesive bond. The overlap coefficient of the laser spot on the surface is an important criterion for laser processing, on which the surface texture depends. The overlap coefficients in two perpendicular directions may differ from each other. If along the processing trajectory the overlap coefficient (with a constant laser spot area) depends on the speed and frequency of the laser, then between the trajectory lines the coefficient depends only on the step between them [15].

The main loads arising in the power elements of truss structures, including connections of tubular rods and ends, are shear and torsion loads (Fig. 2, *a*). Shear tangential stresses are directed along the rod axis during shear, and tangentially to its radius during torsion. Microgrooves on the surface of the cylindrical end can contribute to an increase in the maximum load at failure. The direction of the surface texture will determine which types of load the connection will resist to a greater extent. The processing angle $\alpha = 0^{\circ}$ should contribute to an increase in shear strength, the processing angle $\alpha = 90^{\circ}$ should contribute to an increase in torsional strength, and intermediate processing directions $0^{\circ} < \alpha < 90^{\circ}$ should increase the strength under mixed loading (Fig. 2, *b*).



Рис. 2. Схема клеевого соединения цилиндрических деталей:
А – элемент трубчатого клеевого соединения с приведёнными нагрузками, где
I – углепластиковый стержень, 2 – металлическая законцовка, 3 – клей;
В – направление микроканавок на металлических цилиндрических законцовках

Fig. 2. Scheme of adhesive connection of cylindrical parts: A – element of a tubular adhesive joint with reduced loads, where I – carbon fiber rod, 2 – metal ending, 3 – glue; B – direction of microgrooves on metal cylindrical ends

The main objective of this study is to experimentally determine the characteristics of the adhesive bond of samples made of OT-4 titanium alloys with different textures obtained by laser processing, and to determine the processing texture of cylindrical ends in spacecraft elements.

Since testing of cylindrical parts is associated with the need to use additional equipment and the complexity of performing adhesive bonding of samples, it was decided to introduce some model simplification in the work, which consists in the fact that the surface of the cylinder is as if "unfolding" into a plane (Fig. 3) and at the same time flat samples can be tested according to OST 92-0949–74 (Fig. 4). Textures are characterized by the following parameters: α_1 – the angle of inclination of the first laser treatment, α_2 – the angle of inclination of the second laser treatment, δ – the step between the grooves.



Рис. 3. Развертка поверхности цилиндра (титановой заглушки) с канавками от лазерной обработки на плоскость





Рис. 4. Схема клеевого соединения плоских деталей: *А* – образец для испытания текстуры поверхности на адгезионную прочность, где *I* – пластина из ПКМ, *2* – титановая пластина с лазерным текстурированием, *3* – адгезив; *B* – схематическое изображение текстур на поверхности титана от угла лазерной обработки

Fig. 4. Scheme of adhesive connection of flat parts: A – sample for testing surface texture for adhesive strength, where I – PCM plate, 2 – titanium plate with laser texturing, 3 – adhesive; B – schematic representation of textures on the surface of titanium from the angle of laser processing

In this preparation, linear (unidirectional) and net (bidirectional) structures are selected as textures, in which the processing paths intersect each other at different angles $(0^\circ, 30^\circ, 45^\circ, 60^\circ \text{ and } 90^\circ)$.

For correct comparison of the results of adhesive bond strength with different textures, the surface laser energy density Ep must be constant on all selected textures. To form net structures, it is necessary to apply laser energy twice as much as for linear structures, therefore, to equalize this indicator, the linear structure must be processed twice.

Experiment

The influence of the processing angle on the strength of the adhesive joint was determined according to the following scheme:

- creation of various textures on the surface of titanium alloy samples by laser processing;
- study of the microrelief and surface roughness of the processed samples;
- gluing samples with the same texture for shear testing;
- conducting a shear test of the glued samples;
- analysis of the obtained results.

The samples were plates made of titanium alloy OT-4, which is widely used in spacecraft designs. The size of the samples was $70 \times 20 \times 2$ mm. The laser processing zone was 20×20 mm on one side of the plate.

The surface treatment of the samples was carried out using an ytterbium pulsed fiber laser (IPG, YLPM-1-4×200-20-20) at room temperature in a standard atmosphere. The laser treatment parameters correspond to mode No. 1 from [14] (Table 1), since this mode is used to create structured microgrooves with a depth of 40 and 60 μ m with single and double treatments, respectively [16].

The texture schemes are presented in Table 1, where $\alpha 1$ is the tilt angle of the first laser pass, $\alpha 2$ is the tilt angle of the second laser pass. The step between the grooves in each texture is $\delta = 66.7 \mu m$.

Table 1

N⁰	1 2		3	4	5	6	
Texture scheme							
α_1	0°	90°	0°	30°	45°	60°	
α_2	0° 90°		90°	-30°	-45°	-60°	
Laser power, W	10						
Processing speed, mm/s	200						
Number of stripes per 1 mm, mm ¹	15						
Laser wavelength, µm	1.064						
Pulse repetition frequency, kHz	40						
Pulse width, ns	200						
Energy per pulse, mJ	er pulse, mJ 1						

Laser texturing modes

To ensure static reliability of the study, tests on three samples with each selected texture were carried out.

The samples were glued according to OST 1-90281–86. Gluing was carried out within 24 hours after laser surface processing. Before gluing, the processed surface was cleaned with isopropyl alcohol. The area of the adhesive joint $S = 300 \text{ mm}^2$. Three-component glue VK-9 based on epoxy and polyamide resin was used as an adhesive. Shear tests were carried out 7 days after gluing for complete polymerization of the glue in air at room temperature.

To determine the adhesive strength of the joint, samples with the same surface texture were glued together. Bonding titanium to carbon fiber does not allow determining the adhesive strength of the adhesive joint, since in this case cohesive failure occurs on the carbon fiber.

Determination of the adhesive bond shear strength (Single-Lap Shear Test) was carried out on a universal tensile testing machine (Eurotest T-50, S.A.E.IBERTEST, Spain) at a speed of 5 mm/min.

An optical metallographic microscope (Neophot-32, Carl Zeiss, Germany) was used to analyze the microrelief of the processed surfaces.

The roughness of the treated surface was measured using a profilometer (TR110, TIME Group Inc., China) in two perpendicular directions X and Y (Table 2).

Results and discussions

Figure 5 shows the surface roughness values of the titanium alloy after laser processing in two perpendicular directions.

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Fig. 5. The value of titanium surface roughness depending on the texture of laser processing in two perpendicular directions

Table 2 shows the morphology of the surface structure according to the laser processing texture.

Figure 6 shows the results of average values of shear strength of adhesive joints with different surface textures. The nature of failure for all samples is cohesive by adhesive.

The measurement showed that the roughness value of linear textures ($0^{\circ}-0^{\circ}$ and $90^{\circ}-90^{\circ}$) differs significantly depending on the direction. The roughness value of net textures depends on the processing direction less than that of linear textures. Textures $0^{\circ}-0^{\circ}$ and $90^{\circ}-90^{\circ}$, as well as $\pm 30^{\circ}$ and $\pm 60^{\circ}$, are identical textures rotated relative to each other by 90°. Textures $0^{\circ}-90^{\circ}$ and $\pm 45^{\circ}$ are identical, but rotated by 45° relative to each other. The roughness value in one direction for identical textures corresponds to the roughness value in the perpendicular direction for another identical texture rotated by 90°. The surface roughness under the action of laser processing increases by 4–12 times relative to the unprocessed surface.

Metallographic texture analysis (see Table 2) shows that the texture parameters correspond to the direction of the software surface processing and the specified step between the processing trajectories. The laser beam, moving along the metal surface, locally melts it, squeezes it out from the center to the periphery and evaporates it, creating a "microgroove" up to 40 μ m deep. Repeated laser action on the same place deepens the groove. The molten material squeezed out from the center of the groove hardens and creates a ridge between the laser processing trajectories. For net structures, the first pass of laser processing at an angle of α_1 creates a ridge between the trajectories, but the second pass at an angle of α_2 destroys it at the intersections of the trajectories.

Table 2

Texture parameters

	α_1	α2	Overhead view		Microsection (side view)		
1	0°	0°		<u>у</u> 100µт.			
2	90°	90°		100µm			
3	0°	90°					
4	30°	-30°					
5	45°	-45°					
6	60°	-60°					



Рис. 6. Прочность клеевого соединения от угла текстурирования поверхности



The results of the shear adhesion strength tests showed that regardless of the type of texture obtained during surface treatment, the adhesive bond strength increased by more than 70%. This may indicate that the main mechanisms for increasing the adhesive bond strength are an increase in the contact area of the surface and the adhesive, as well as chemical modification that activates the surface. The processing texture has a lesser effect on the adhesive strength under the condition of the same specific surface energy of laser treatment.

The best result of adhesive joint shear strength $\tau_{cp} = 28.9$ MPa was shown by the 0°–0° texture. If compared with the strength of the adhesive joint without the "titanium–titanium" processing from work [14], which is 14.15 MPa, then the 0°–0° texture increased the strength by 104%. The strength of the adhesive joint with the 0°–90° texture showed the lowest result $\tau_{cp} = 24.2$ MPa relative to other textures, but the increase relative to the untreated surface is 71 %. The results of the study showed that it is possible to increase the strength of the adhesive joint by 20 % only due to the surface texture.

The $0^{\circ}-0^{\circ}$ texture is more resistant to shear loads than the other textures studied. This is facilitated by the linear structure of the microgrooves in the perpendicular direction of the shear. The ridges of the grooves, which were formed under the the laser pressure, are a micro-stop for the glue, preventing the movement of the glue under shear load.

In the net structures $\pm 30^{\circ}$, $\pm 45^{\circ}$, $\pm 60^{\circ}$ the stop for the adhesive is the ridge formed by the intersection of the laser trajectories. In the $\pm 60^{\circ}$ texture the ridge is the narrowest of the net structures. This explains the low strength value.

In the $90^{\circ}-90^{\circ}$ texture there is no stop in the shear direction, but because of a larger contact area due to a deeper groove than in net structures and the absence of a destroyed ridge it shows relatively high strength values.

The $0^{\circ}-90^{\circ}$ texture showed the worst result among the studied textures. The ridge, which could have been a stop for the glue, was destroyed by the second pass of laser processing.

Having determined the average groove size based on the microsection from Table 2, the macrostructure of the surface of the $0^{\circ}-0^{\circ}$ and $0^{\circ}-90^{\circ}$ textures (Fig. 7, *a*, *b*) was modeled in the CAD system. In this modeling, the surface microrelief was not taken into account due to its randomness. The $0^{\circ}-0^{\circ}$ texture model showed that the surface area increases by 94 % relative to the untreated surface.



And for the $0^{\circ}-90^{\circ}$ texture, only by 16 %. The small increase in the surface area of the $0^{\circ}-90^{\circ}$ texture can explain the reason for the low value of the adhesive joint strength relative to other textures.

Рис. 7. Модели макроструктуры поверхности с лазерной обработкой: A – текстура 0°–0°; B – текстура 0°–90°; C – модернизированная текстура 0°–90°

Fig. 7. Models of surface macrostructure with laser processing: A – texture 0°–0°; B – texture 0°–90°; C – modernized texture 0°–90°

In order to improve the strength characteristics of the adhesive bond of the $0^{\circ}-90^{\circ}$ structure, it is proposed to deepen the microgroove, which is formed across the direction of the shear load, by increasing the number of passes of the laser beam along the trajectory. It is proposed to double the step for perpendicular processing, thereby preserving part of the ridge, which contributes to an increase in the shear strength (Fig. 7, c). The surface area of the modernized $0^{\circ}-90^{\circ}$ texture increased by 72 % relative to the untreated surface. This modernized $0^{\circ}-90^{\circ}$ texture will require a higher specific surface energy than the textures in this study.

Conclusion

Laser treatment of titanium alloy surfaces increases the strength of adhesive bonds by more than 70% compared to untreated surfaces. When laser processing, it is important to pay attention to the choice of surface texture, because certain textures can increase strength by 20–30 %. If the type of load in truss elements is known, it is better to use linear textures directed perpendicular to the load direction (for shear $-0^{\circ}-0^{\circ}$ texture, for torsion $-90^{\circ}-90^{\circ}$ texture). For mixed loads, it is better to use net structures of $\pm 30^{\circ}, \pm 45^{\circ}, \pm 60^{\circ}$, which resist loads in two directions.

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