Modification of Surface Including Charged Particle Beams and Photon and Plasma Fluxes
Модифицирование поверхности, в том числе пучками заряженных частиц, потоками фотонов и плазмы

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Effects of ion-plasma treatment temperature of the aluminium coating on the structure and phase composition of the VT6 titanium alloy

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Abstract. In this study, we studied the effects of aluminum coating treatment temperature on the microstructure and phase composition when applied to a VT6 titanium alloy substrate within a low-pressure arc discharge plasma environment. The ion-plasma treatment was conducted at 450 and 500 °C, employing argon shielding, while the aluminum coating was deposited using the vacuum-arc process, resulting in a coating thickness of ~3 μm. Microstructural analysis was performed using a scanning electron microscope, and the structural and phase composition were examined using *X*-ray diffraction (XRD) imaging in symmetric imaging mode with CuK_α radiation. Our findings demonstrate that the application of the aluminum coating initiates the formation of a near-surface α-stabilized layer, extending up to 2.5 μm in thickness due to the heat generated during the ion cleaning process. Subsequent ion-plasma treatment further results in the development of a $TiAl_3$ intermetallide site, reaching thicknesses of up to 1.5 μm, while the α-stabilized region expands to 5.5 μm. Higher temperatures during the treatment process contribute to an increase in the thickness of these aforementioned layers and also lead to the emergence of an intermediate TiAl intermetallic layer.

Keywords: ion-plasma treatment, intermetallide coatings, gradient coatings, titanium alloys

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Влияние температуры ионно-плазменной обработки алюминиевого покрытия на микроструктуру и фазовый состав титанового сплава ВТ6

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Аннотация. Представлены результаты исследования влияния температуры обработки поверхности алюминиевого покрытия на титановом сплаве ВТ6 в плазме дугового разряда низкого давления на микроструктурные и фазовые изменения. Ионно-плазменную обработку проводили в плазме дугового разряда низкого давления при температурах 450 и 500 °C в среде аргона. Алюминий наносили вакуумно-дуговым методом, толщина покрытия составляла ~3 мкм. Микроструктурные изменения исследовали с помощью растровой электронной микроскопии. Структурно-фазовый состав определяли по результатам расшифровки дифрактограмм, полученных при симметричной съемке в CuK_α-излучении. Показано, что после нанесения алюминиевого покрытия в результате нагрева при ионной очистке формируется приповерхностный α-стабилизированный слой толщиной до 2,5 мкм. Последующая ионно-плазменная обработка приводит к формированию интерметаллидной области $TiAl_3$ толщиной до 1,5 мкм, α-стабилизированная область увеличивается до 5,5 мкм. Выявлено, что повышение температуры обработки приводит как к увеличению толщины указанных выше областей, так и к появлению промежуточной интерметаллидной зоны TiAl.

Ключевые слова: ионно-плазменная обработка, интерметаллидные покрытия, градиентные покрытия, титановые сплавы

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Introduction

Titanium and its alloys fnd extensive application in the aerospace industry and medicine [1–3]. However, their wear resistance is low, with most titanium friction parts being susceptible to diffusion interaction between the contact surfaces and subsequent wear [4; 5]. There is a demand to enhance the wear resistance of titanium alloys.

One efficient approach to achieve this enhancement is through ion nitriding [6–8] and alloying [9; 10] of the surface layer, as well as applying coatings [11–13]. In the case of titanium alloys, ion nitriding requires high temperatures and extended holding periods [14; 15], while the low-temperature process [16; 17] proves to be less efficient. The application of coatings enables to create super-hard films on surfaces based on nitrides, carbides, and oxides of transition metal [18–20]. However, these coatings may experience delamination when subjected to impacts [21; 22], inevitably leading to accelerated surface wear, intensified by detached coating particles.

Gradient coatings made from alloys with high impact resistance and resistance to aggressive media show better performance due to the absence of a distinct substrate-coating interface. In steel products, a combination of nitriding and coating (Duplex Treatment) is commonly employed [23; 24]. However, the application of duplex treatment to titanium alloys does not improve performance. In this scenario, the nitrided layer exhibits significantly lower hardness and depth compared to the same treatment time and temperature.

Titanium forms intermetallic compounds with various metals, and these compounds typically have enhanced physical and mechanical properties compared to pure titanium and its non-hardened alloys. Ti–Al intermetallides have a low specific gravity (3.3–4.2 g/cm^3) and show high hardness, heat resistance, oxidation resistance, and corrosion resistance. Some aerospace applications of this material have been reported by Lazurenko D. et al. [25]. Nevertheless, due to the high brittleness, particularly the $TiAl_3$ phase, intermetallides are unsuitable for manufacturing solid parts. Zhang Y. et al. [26], Liu Y. [27], and Parlikar C. [28] have investigated the application processes and resulting intermetallide coatings, reporting a significant increase in strength (up to 20 %) and wear resistance for coating thicknesses less than 16 µm.

Currently, primary Ti–Al intermetallic coating technologies include aluminizing [29], magnetron [30], and vacuum-arc [31] sputtering, as well as laser [32] and electron-beam [33] surfacing, ion implantation [34], often combined with subsequent heat treatment [35–37]. It is, however, challenging to control the phase composition of the resulting layers during coating deposition and ion implantation. When aluminum and/or Ti–Al coatings are deposited, followed by heat treatment at the aging temperature of the titanium alloy, the final coating consists only of the $TiAl_3$ phase. Subsequent ion-plasma treatment can intensify the formation of the TiAl and Ti_3Al intermetallides, which are more ductile compared to $TiAl_3$.

Our study focuses on a combination of vacuum-arc deposition used to create pure aluminum coatings, followed by plasma treatment with low-pressure non-self-powered arc discharges. The objective of this study is to analyze the effects of the ion-plasma treatment temperature on the aluminum coating on the structure and phase changes of the VT6 titanium alloy surface layers.

Materials and methods

We prepared samples in the form of 20 mm diameter disks, each measuring 4 mm in thickness, obtained from a titanium bar (VT6 alloy). The combined treatment involved two stages. In the initial stage, we deposited a pure aluminum layer, approximately 3 µm thick, onto the surface of the titanium disk using vacuum arc deposition. The sample surface underwent an initial ion cleaning process in an Ar plasma (40 A discharge current, 800 V bias voltage) for 30 min, raising the surface temperature to 450 °C. Then it was exposed to Al plasma for 1 min, generated by a 60 A arc evaporator current. We monitored the surface temperature using a chromel-copel thermocouple and an AST250+ IR pyrometer (Accurate Sensors Technologies, India). In the second stage, the surface was treated in a PINK plasmatron with an incandescent cathode (ISE, Russia) [38] for 1 h under argon shielding. The bias voltage varied depending on the temperature, but the discharge current was always 50 A. We processed the samples at both 450 and 500 °C. Then the samples were vacuum-cooled in argon atmosphere at 1 Pa.

Following the treatment, we examined polished section structure of the samples using a Mira scanning electron microscope (Tescan, Czech Republic) operating in the secondary electron mode. For *X*-ray diffraction analysis (XRD), we used an Ultima IV diffractometer (Rigaku, Japan) with CuK_α-radiation in the symmetrical imaging mode.

Results and discussion

To investigate the interaction between the aluminum coating and the VT6 titanium substrate, we examined how the ion-plasma treatment temperature influenced changes in the structure and phase composition of the surface layers. Following the deposition of the aluminum coating onto the rough substrate surface (Fig. 1, *a*, *b*), an α-stabilized region I, approximately 2.5 ± 0.5 μm deep, is formed within the near-surface layer of the substrate. This formation results from the diffusion of the coating elements during the initial stage of deposition when the surface of the titanium alloy is still heated to 450–470 °C. The α-stabilized region can be distinguished from the substrate due to the dissolution of small recrystallized β-particles

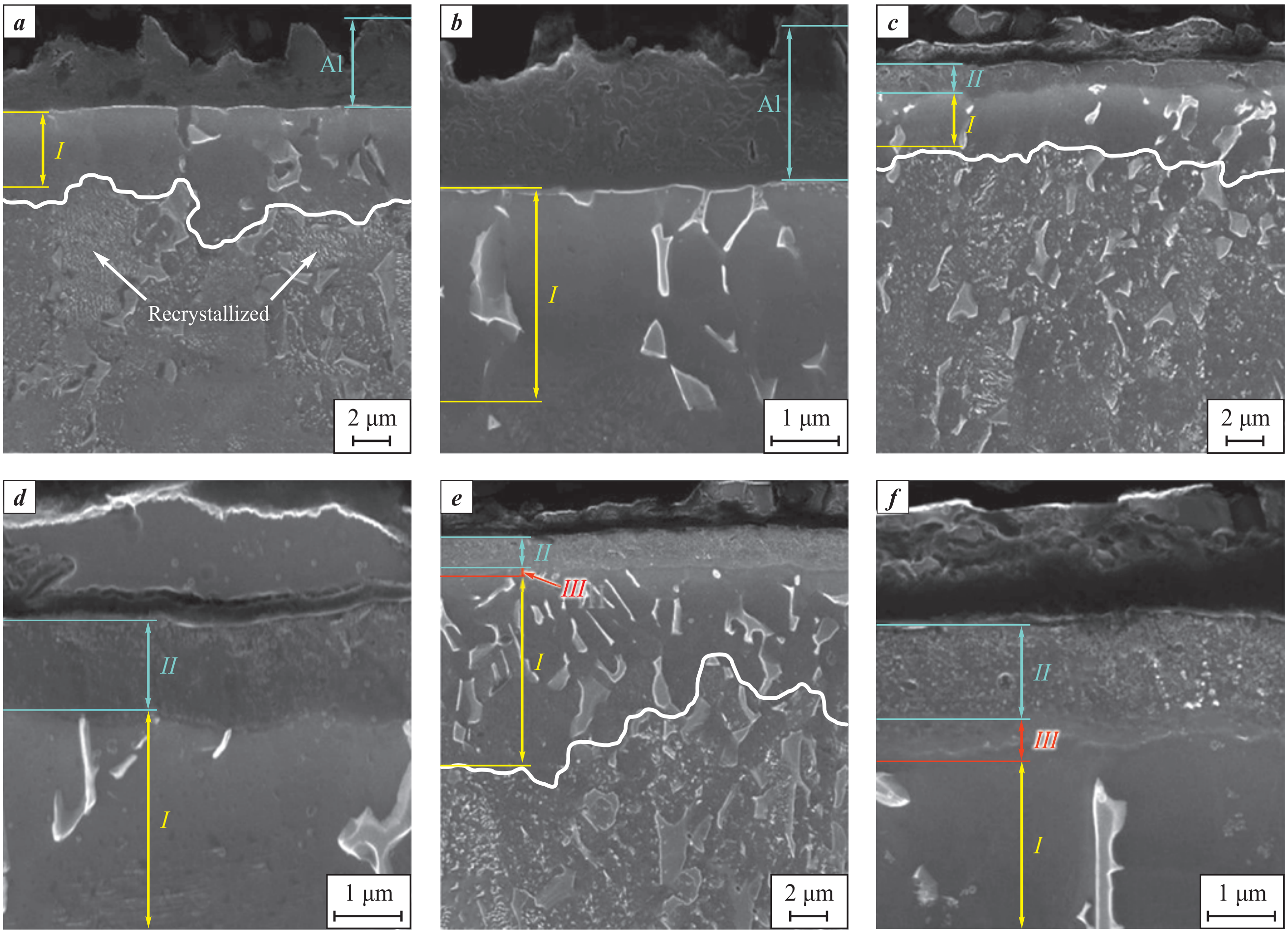

Fig. 1. Polished section structures of the aluminum-coated titanium samples
a, ***b*** – initial coating; ***c***–***f*** – after ion-plasma treatment at 450 (***c***, ***d***) and 500 °C (***e***, ***f***)

Рис. 1. Изображения структуры поперечного сечения образцов титана с покрытием из алюминия
a, ***b*** – исходное покрытие; ***c***–***f*** – после ионно-плазменной обработки при 450 (***c***, ***d***) и 500 °C (***e***, ***f***)

within the α-grains, a consequence of aluminum diffusion deep into the surface layer, and the α-phase stabilization facilitated by the presence of Al, which is known to be an α-stabilizing element.

The ion-plasma treatment conducted at 450 °C (Fig. 1, *c*, *d*) also led to the formation of an intermetallic site comprising the $TiAl_3$ phase with a high aluminum content. The total length of the modified site increased to 5 ± 0.5 μm. Plasma etching reduced the thickness of the aluminum coating to ~2 μm. The coating became brittle, as evidenced from its fracturing during our sample section polishing. This brittleness can be attributed to the presence of the brittle $TiAl_3$ intermetallic phase and its large volume concentration in the surface layers. Elevating the temperature to 500 °C resulted in the emergence of a transition layer between the substrate and layer *III* (Fig. 1, *e*, *f*). This transition layer, as reported by Ramos A. et al. [35] and Garbacz H. et al. [36], primarily consists of the TiAl intermetallide. As indicated by the gradient of aluminum concentration, the layer above it consists mainly of the $TiAl_3$ phase, while the layer below it comprises Ti_3Al.

This assumption finds confirmation in the XRD analysis, as depicted in Fig. 2. The surface layers following treatment at 450 °C mostly contain the $TiAl_3$ intermetallide. No aluminum peaks were detected, possibly due to the low Al concentration in the surface layer of the coating. Additionally, aside from the intermetallic phases, a solid solution forms as aluminum substitutes into the titanium lattice, evidenced by the shift of titanium peaks towards larger diffraction angles, signifing a decrease in the lattice period. As the treatment temperature increased to 500 °C, the TiAl intermetallide was found in the surface layers, while no Ti_3Al peaks were observed. The intensity and number of the $TiAl_3$ phase grew, which correlates with the observed microstructure changes.

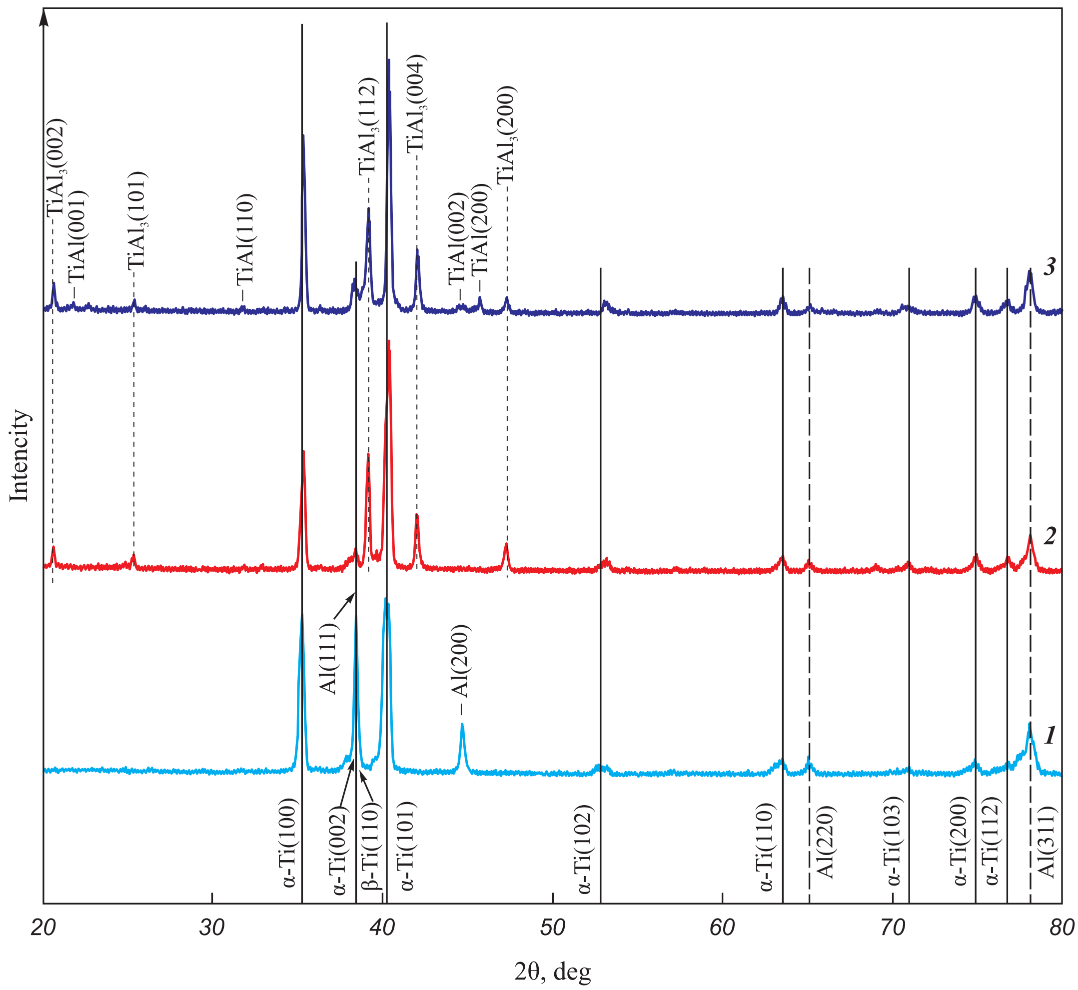

Fig. 2. XRD images of the samples

1 – initial coating; *2* and *3* – after ion-plasma treatment at 450 (*2*) and 500 °C (*3*)

Рис. 2. Дифрактограммы исследуемых образцов

1 – исходное покрытие; *2* и *3* – после ионно-плазменной обработки при 450 (*2*) и 500 °C (*3*)

Given the inherent brittleness of the coatings, we intend to explore the impact of ion-plasma treatment duration and the thickness of the initial aluminum coating on the elemental and phase compositions, as well as the wear resistance of these coating layers.

Conclusion

We conducted experimental investigations on intermetallic surface layers created through the deposition of an aluminum coating onto a titanium substrate, followed by low-pressure non-self-sustained gas plasma treatment.

Our findings reveal that such treatment results in the development of intermetallic and α-stabilized layers within the near-surface layer of the VT6 titanium alloy. Treatment at 450 °C yields an intermetallic layer, approximately 1.5 µm thick, composed only of the $TiAl_3$ phase. Raising the temperature to 500 °C leads to the formation of an additional TiAl intermetallide layer, measuring 300 nm in thickness, situated beneath the 1.8 µm thick $TiAl_3$ layer. Importantly, XRD analysis does not detect any Ti_3Al peaks.

The creation of the α-stabilized layer commences during the deposition of the aluminum coating itself. As the ion-plasma temperature increases, the layer's thickness increases as well, attributed to the accelerated diffusion rate of aluminum into the titanium substrate.

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