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Alloying of surface layer of the Ti-6Al-4V titanium alloy through the laser treatment

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ABSTRACT

Purpose: The purpose of this paper is modification of the Ti-6Al-4V titanium alloy surface layer properties through laser alloying technology.

Design/methodology/approach: Laser treatment was performed on the samples coated by graphite and BN powders in stream of nitrogen. Thopography of the surface of laser melted layer was investigated. Microstructure,fracture surface and chemical composition analysis were made by using Epiphot 300 optical microscope and Novascan 30 scanning electron microscope equipped with EDS X-ray detector. Phase composition was determined using X-ray diffractometry (Philips) with $Cu_{K\alpha}$ radiation. The Vickers hardness under load of 1.96 N and thermo-electric power was measured on the surface of cross-sections.

Findings: Laser treatment has produced a surface layer which consists of hard ceramic TiC, TiN and TiB particles spaced in ductile martensitic matrix. Under the layer, the heat affected zone containing martensitic Ti_{α}' phase is present. The hardness obtained on cross-sectioned layer increases clearly in comparison with the base material. The high hardness level (HV 920 - 570) can be attributed to the formation of TiN, TiC and TiB phases. The thermoelectric power decreases noticeably with hardness increase and enables alloying process evaluation.

Research limitations/implications: Research range was limited to investigation of microstructure, phase composition and hardness effects of laser alloying process. In order to estimate the influence of the laser alloying technology on durability of the layer, supplementary wear resistance tests will be performed in future research.

Practical implications: The surface alloying by laser irradiation is investigated as a process capable to produce coatings composed of metallic matrix reinforced by ceramic particles and this can increase durability of elements made of titanium alloys.

Originality/value: The wide range of investigations contained microstructure, phase and chemical composition analysis as well as fractographic and thermo-electric power estimation enables in-depth analysis of alloying process efficiency.

Keywords: Surface treatment; Titanium alloy; Laser treatment; Microstructure; Phase composition

1. Introduction

Titanium and its alloys are extensively used in the aeronautical industry, medicine and engineering industry due to their specific properties such as light weight, high strength-to-weight ratio, corrosion resistance and excellent high temperature properties. Surface engineering of titanium alloy components provides means by which the desirable bulk properties may be retained in conjunction with enhanced wear resistance [1, 2]. Various superficial layers technologies are applied in order to obtain surface hardening of machine elements made from titanium alloys. These layers posses larger hardness and wear resistance in comparison with the base material [3-7]. The laser surface melting treatments represent a particularly attractive solution where sufficient deep protection is received. Laser surface alloying exemplifies much perspectives but it is still insufficiently developed method of increasing wear resistance. Relatively large quantity of experimental works has been devoted to study the effect of laser nitriding [8-12]. Titanium nitrides formed in laser melted zone bring about hardening and increase wear resistance of titanium alloys, but nitrided layers contain pores, porous edges and cracks, so fatigue life of the material decreases rapidly [11, 13- 16]. The tendency for cracking in laser-melted zones may be reduced by the dilution of the alloy gas with a carrier gas or by a combination of laser alloying and pre-heating of the substrate. This provides of decreasing tensile residual stresses inside the laser treaded zone. Moreover there are many independent variables (e.g. laser power, beam size, configuration and wavelength, travel speed of the work-piece, surface condition, processing atmosphere, pre-coating or powder injection method used), which influence dimensions and properties of laser treated layers. There is yet no experimentally developed process of optimum processing parameters for surface hardening and producing layers devoid of cracks and considerable roughness on the surface. Over a wide range of laser processing conditions complex microstructures are developed in the solidified melt pool [17-19].

Titanium alloys can be boronised efficiently, but use of conventional methods is limited due to relatively long processing times and small layer thickness. Laser boronising is a new effective method to increase hardness and wear resistance of titanium alloys. Effect of laser alloying of B, B_4C, TiB_2, MoB, and MoB_2 powders with titanium alloys has been studied as well as the BN powder addition [20,21]. Laser boronising of Ti-6Al-4V titanium alloy brings about increase of hardness of surface layer (almost three times). Alloyed zone was free from cracks and porosity. The results of investigations revealed the formation of TiB, Ti_3B_4 and Ti_2N in effect of laser remelting.

The superficial layer consisted of relatively ductile solid solution and dispersed titanium carbides, nitrides and borides caused both surface hardening and increase of wear resistance simultaneously with fatigue limit remaining unchanged. Thorough microstructure examinations, particularly determination of dendrites volume fraction and distribution in the matrix after laser melting in different areas of laser treated zone prove necessary. The aim of this study was the rough estimation of suitability of laser alloying under conditions of CO_2 laser remelting of graphite and BN coat for improvement of titanium alloys service properties. For this purpose investigation of microstructure and phase composition of laser reaction zone as well as microhardness and thermoelectric power tests were undertaken.

2. Material and research methodology

The material tested was vacuum melted martensitic two-phase $\alpha+\beta$ titanium alloy having following composition (%wt): 6.29 Al, 4.12 V, 0.18 Fe, 0.14 C, 0.1 Si, 0.01 Mn, 0.1 Mo, 0.02 Cu, 0.001 B, 0.1 Zr, 0.01 Sn, 0.02 Cr, 0.19 O, 0.0032 H, Ti- balance. The bars of 22 mm diameter were rolled at $\alpha+\beta$ temperature range and annealed at temperature 973 K in order to obtain fine-grained equiaxial microstructure Afterwards the disk shaped samples, 10 mm thick, were cut. For the laser boronising process the BN powder of micrometric grain size (Fig. 1) was mixed with graphite (in volume ratio of 50 to 50%) and water to obtain a steady deposit on the plain surface of the disks. After drying in hot air the thickness of deposit averaged to about 0.2-0.3 mm.

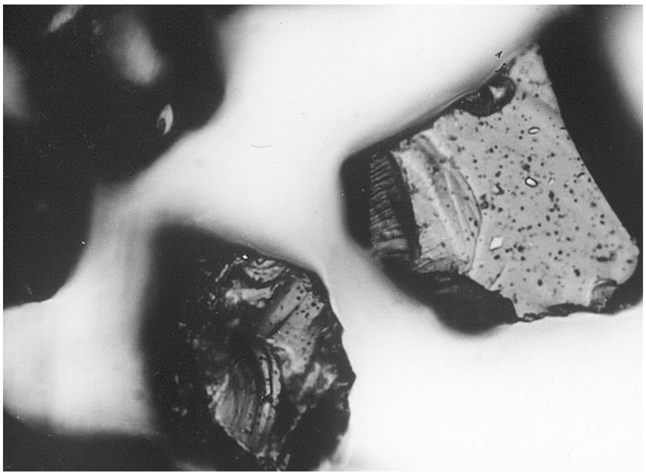

Fig. 1. Grains of BN powder used in laser alloyed process, mag. 1000×

The sample surfaces were treated by a 1 kW CO_2 laser with 10.63 μm wavelength. The raw beam was focused by 65 mm ZnSe lens. Using a X-Y translation stage, the specimen was traversed with respect to the laser beam. The beam diameter on the sample surface was about 2.0 mm. Single pass alloying trials were made with a scan speed 1 m/min. To prevent atmospheric contamination of the melt pool, nitrogen gas was supplied at a flow rate of 10 l/min. The nitrogen was used also as an alloying element, with the purpose of hard nitrides formation. The width of laser-melted zone was determined by transverse sectioning, polishing and etching the laser track using a solution of 2%HF and 2% HNO3.

Microstructural analysis was carried out using Epiphot 300 optical microscope and Novascan 30 scanning electron microscope equipped with EDS X-ray detector. The fracture surfaces of the laser treated areas of the specimens were observed using scanning electron microscope Novascan 30 with 7 nm resolution at 15 kV acceleration voltages. Fractografic examinations were indispensable for analysis of the damage process and brittleness of the alloyed zone estimation.

The phases were identified by X-ray diffractometry (Philips) with CuK_α radiation. The Vickers microhardness under the load of 1.96 N was measured. Value of thermo-electric power was measured in the same places of cross section using PMT-3 unit. It was calculated using the relation:

$$e = \frac{U}{1000 \cdot \Delta T} \tag{1}$$

where:

e - thermoelectric power, μV·/°C; U - voltage, mV;

ΔT - temperature difference between hot and cool junction, °C.

3. Results and their analysis

Laser radiation has an impact on phase transitions of powders and background-remelted layers as a result of considerable power density. Surface of melted tracks (Fig. 2) presents characteristic appearance and their topography is presented on the Fig. 3. Microstructure and phase composition development, which determines properties of remelted zone, proceeds under conditions of:

- unusually rapid heating and cooling after ceasing of laser radiation action bringing about considerable thermal gradients;
- brief heating and subsequent cooling process;
- high speed of convection stream provides intensive stirring and uniform distribution of BN and graphite inside of molten pool.

Intensive rabbling of alloyed substance in molten pool posses characteristic feature of laser alloying and proceeds by the action of thermoconvectional and gravitational movements. Thermo-capillary convection appears the most essential process responsible for the effect of mixing. It consists of motion of a liquid caused by dependence of surface tension on temperature, as well as heating heterogeneity of molten pool surface.

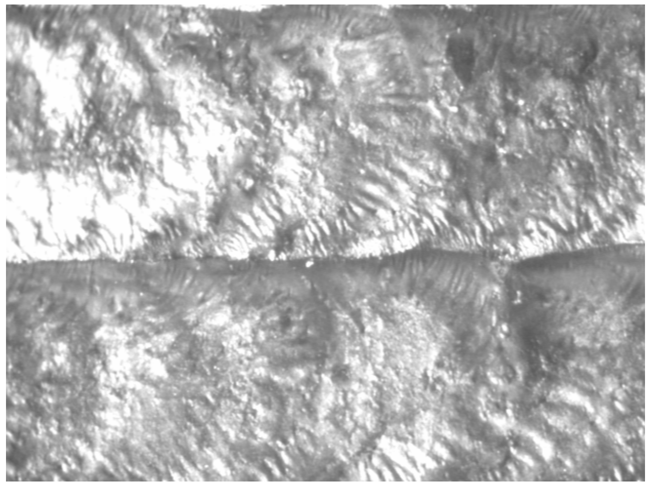

Fig. 2. Surface of laser melted tracks on the Ti-6Al-4V alloy, mag. 20×

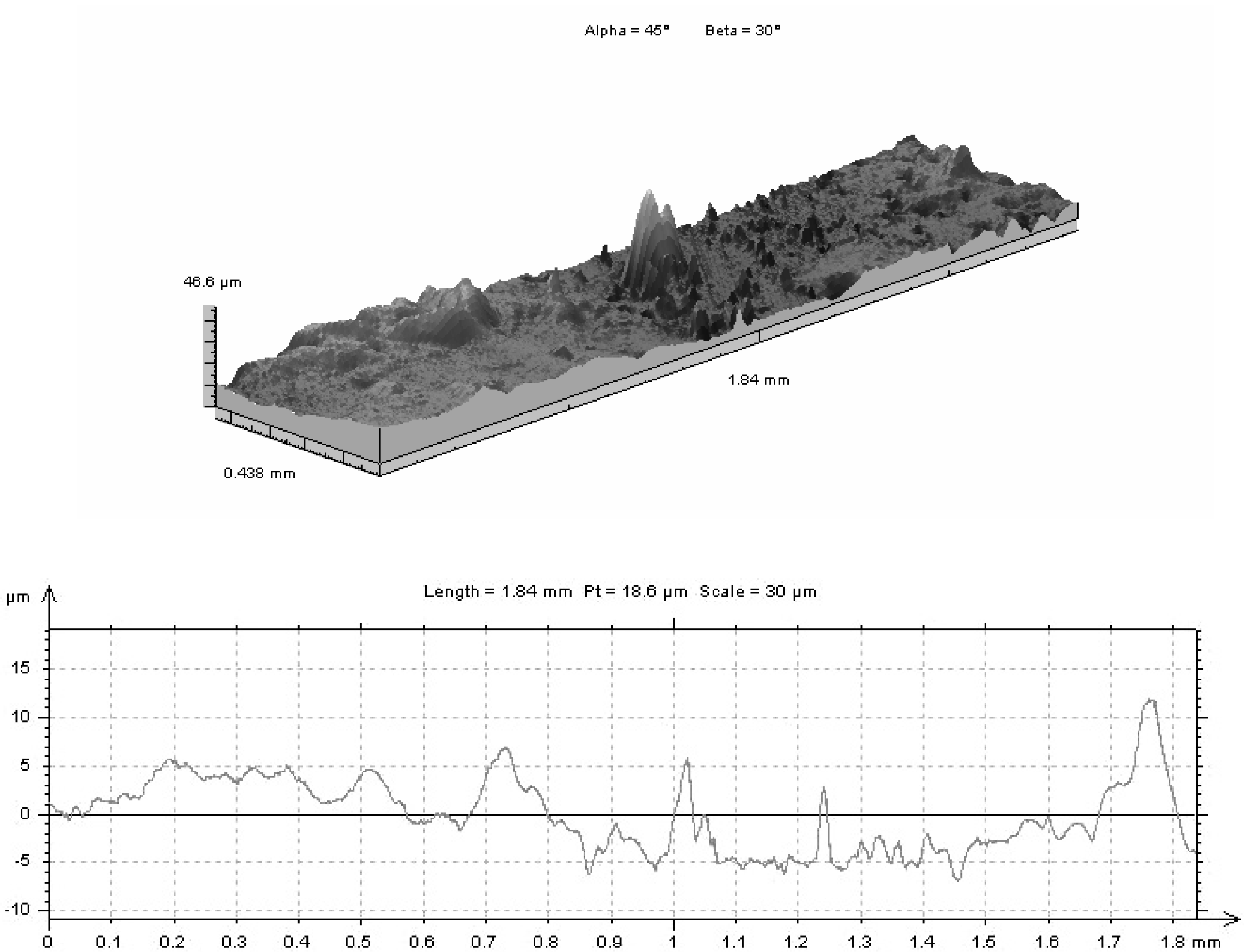

Fig. 3. Topography and surface roughness profile of the laser track

Therefore, under the influence of these motions of liquid, material moves from the centre of surface of melting pool to edge of the pool and from the edge to the centre along the diffusion front. Hence, paths of the flow take the circular character. Microscopic investigations revealed presence of two typical areas attend laser treated area: alloyed zone and heat-affected zone (Fig. 4).

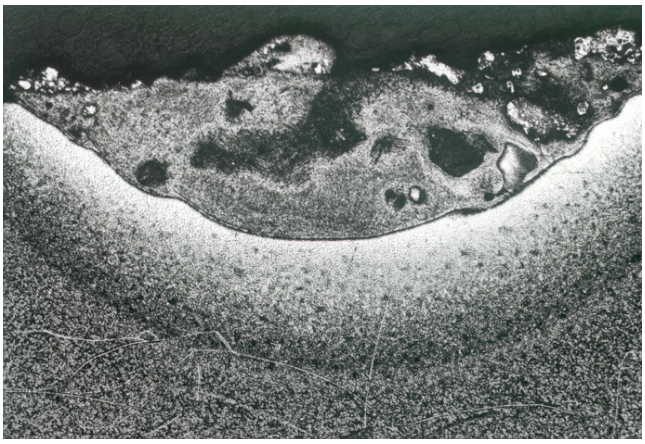

Fig. 4. Cross section of single alloyed track, mag.50×

Microstructure analysis points at dendritic microstructure in top of alloyed zone (Fig. 5-7). Dendrites grow chaotically even from surface inwards melting pool. Ordered, parallel orientation of dendrites is observed in separated local zones only. Dendrites grow along temperature gradient, so theirs different orientation points to complex character of heat transfer owing to convecting motions and brings about efficient stirring of material in melted pool. The microcraks and pores weren't revealed in laser-melted zone, which proves its satisfactory plasticity. Below dendritic zone appears acicular particles spaced in martensitic matrix (Fig. 8, 9). Under the alloyed zone, the heat-affected zone was presented, containing needles of $Ti_{\alpha'}$ phase spaced inside of primary β phase grains (Fig. 10).

X-ray analysis was performed on the surface of laser melted track and in depth of 0.2 mm. It gives indications of alloying process occurrence. Presence of TiB, TiC and TiN phases speaks for realisability of efficient enrichment of the surface layer of titanium alloy. The phases identification was rather difficult because of the nearness of some phases peaks (for example TiN and TiC phases) (Fig. 11). Presence of TiO_2 phase points to appearance of oxidation process owing to insufficient nitrogen protection. Reduction of peaks height in spectrum from the surface points to influence of surface roughness.

Chemical composition investigation was performed on the selected areas of the transverse sectioned surface. It was enabled identification of dendrites existing in the top of alloyed zone as TiC and TiB phases (Fig. 12). Acicular particles situated in areas below dendritic zone were recognized as TiN and TiC phases (Fig. 13). In a few areas precipitates of TiB phase appeared inside deep part of alloyed zone.

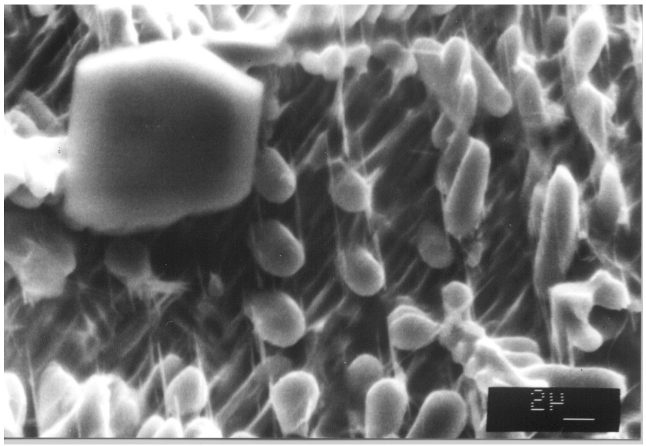

Fig. 5. TiC and TiB particles in bottom part of alloyed zone

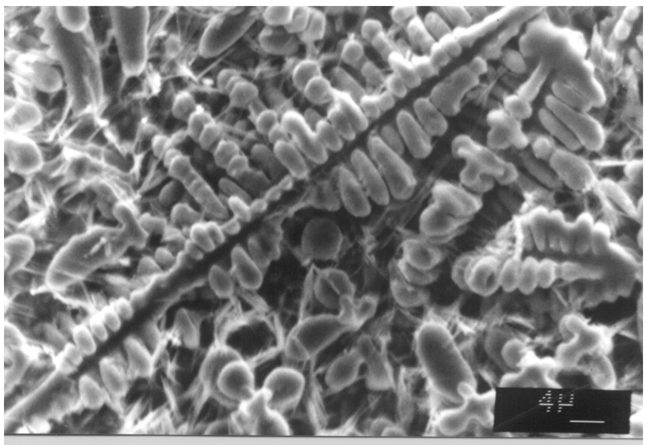

Fig. 6. TiC dendrite in alloyed zone

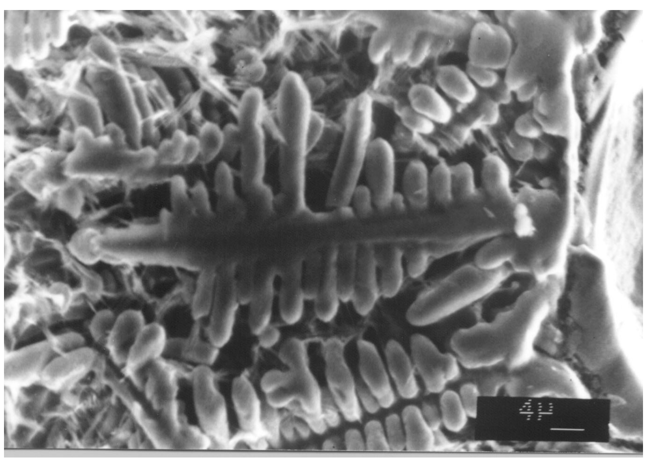

Fig. 7. TiC and TiN dendrites in top part of alloyed zone

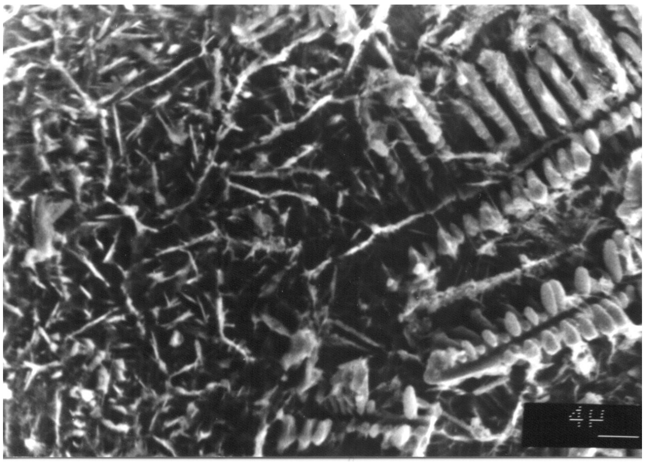

Fig. 8. Microstructure of transition zone into alloyed area

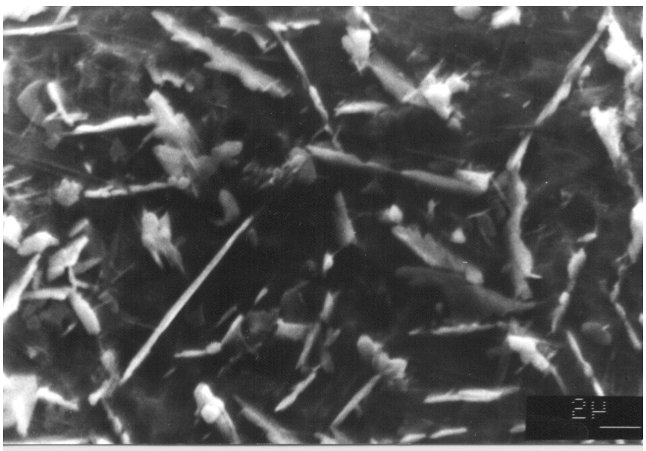

Fig. 9. Needle-shape particles of TiC and TiB phases in bottom part of alloyed zone

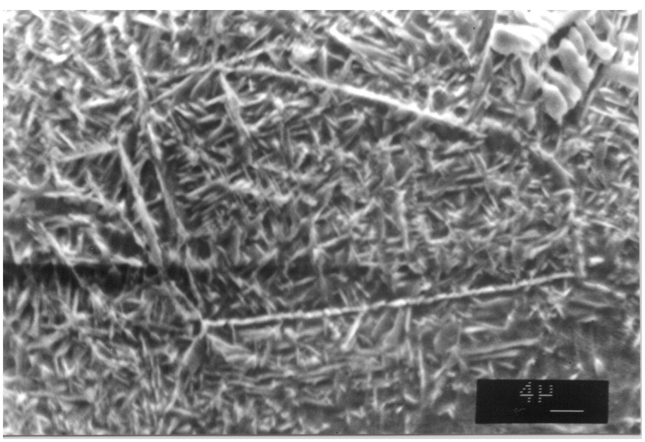

Fig. 10. Mirostructure of top of heat affected zone

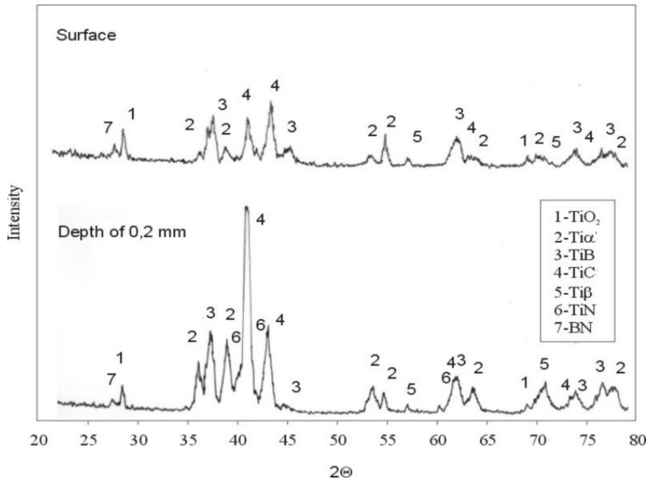

Fig. 11. XRD analysis of the alloyed zone of Ti-6Al-4V alloy

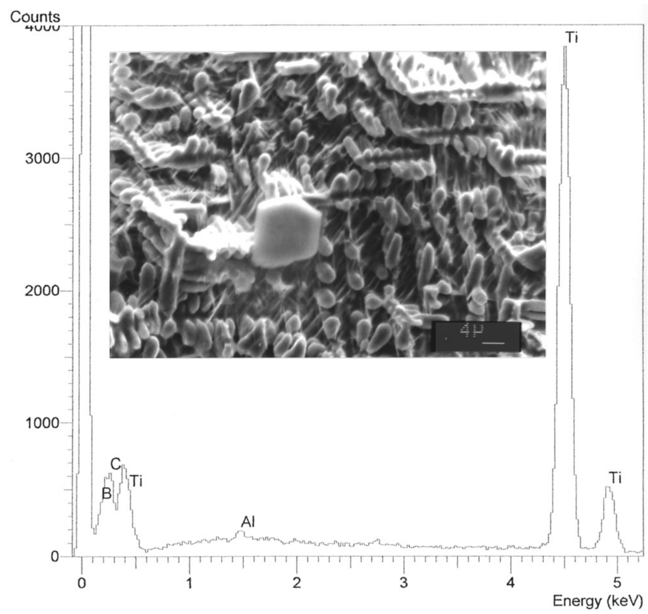

Fig. 12. EDX of dendritic area of alloyed zone

The hardness and thermoelectric power were measured on the surface and at different depths of the laser alloyed layer (Table 1, Fig. 14). The high level of the hardness was observed at alloyed surface (920 HV). It is attributed to the formation of hard carbides, borides and nitrides. In deeper areas amount of martensitic $Ti_{\alpha'}$ phase increases, so hardness is reduced up to about 600 HV. Relatively large variation of hardness inside the alloyed region can be attributed to the heterogeneity of phases arrangement. The hardness inside the alloyed zone varied from 650 HV to 500 HV and dropped to the HAZ (426 HV) and finally to the base material (390 HV). The value of thermoelectric power changes from 15.9 μV/°C (base metal) to 11.7 μV/°C (top of alloyed zone) (Table 1). This method can be used for measurement of alloying effects on element surface as a non-destructive testing.

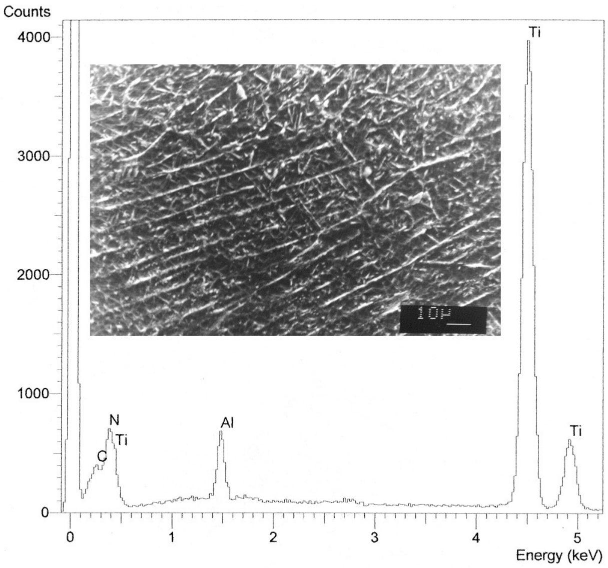

Fig. 13. EDX of deep area of alloyed zone

Table 1.
Results of thermoelectric power and hardness investigations

Zone		e_{av}, μV/°C	HV 0,2
Base metal		15.9	390
Heat affected zone		15.5	426
Alloyed zone	Bottom	15.1	570
	Centre	12.8	644
	Top	11.7	920

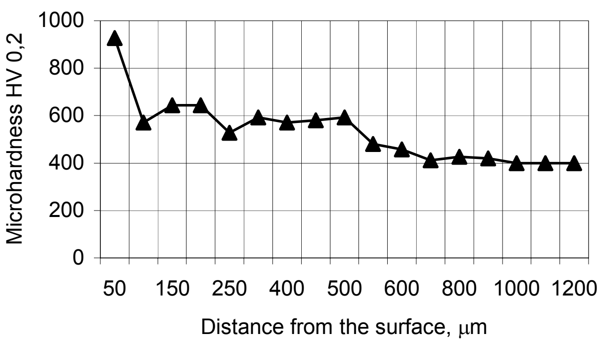

Fig. 14. Microhardness of laser alloyed zone

Fractographic examination of the fracture surfaces of the laser treated zone showed that the Ti-6Al-4 alloy in rapid cooling conditions (heat affected zone) displays ductile fracture(Fig. 15), whereas inside of alloyed zone occurred the complex character of fracture (Fig. 16). In the fine-grained material the grain boundary fracture is greatly reduced and zones of plastic deformation can be seen. Propagating crack penetrates the area of martensitic phase and can be stopped on particles of TiC, TiB and TiN phases. The microcraks and pores weren't revealed on fracture surface, which proves its satisfactory plasticity.

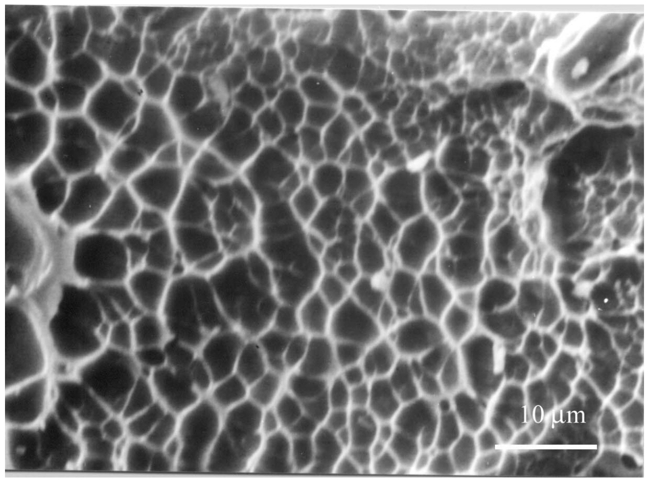

Fig. 15. Fracture surface of heat affected zone

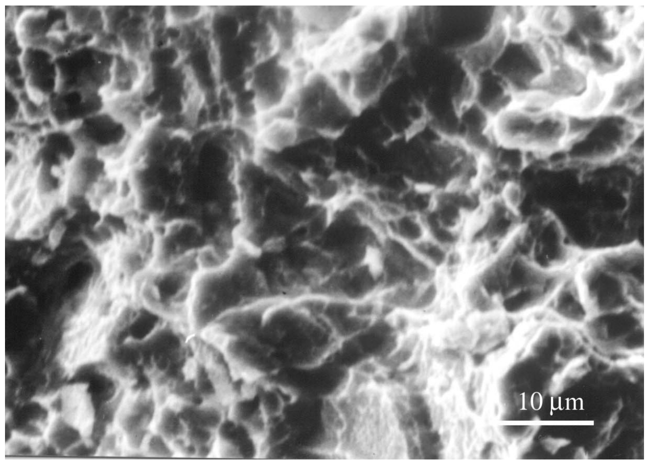

Fig. 16. Fracture surface of alloyed zone

4. Conclusions

Laser melted BN + C coating on the Ti-6Al-4V titanium alloy has produced surface layer which consists of hard ceramic TiC, TiN and TiB particles spaced in ductile martensitic matrix. In the upper part of alloyed zone regions of dendritic microstructure are present. During solidification dendrites grow along the maximum temperature difference direction. The orientation of dendrites within the melt pool is dependent on the stream direction. Underneath occur acicular particles placed in $Ti_{\alpha'}$ matrix. Below, the heat affected zone containing martensitic $Ti_{\alpha'}$ phase is present. The hardness obtained on cross-sectioned layer increases clearly in comparison with the base material. The high hardness level (HV 920 - 570) can be attributed to the formation of TiN, TiC and

TiB phases. The thermoelectric power decreases clearly with hardness increase and enables alloying process evaluation.

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