



# Laser nitriding of the surface layer of Ti6Al4V titanium alloy

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## ABSTRACT

**Purpose:** The purpose of this paper is modification of the surface layer of the Ti6Al4V titanium alloy microstructure and properties by laser remelting in nitrogen atmosphere.

**Design/methodology/approach:** Laser treatment was performed on the samples in stream of nitrogen. Microstructure of laser treated layer was investigated by using Epiphot 300 optical microscope and Novascan 30 scanning electron microscope. Phase composition was determined using X-ray diffractometry. The roughness of surface of treated material was examined using topography scanning system T 8000 made by Hommelwerke GMBH. The Vickers hardness under load of 1.96 N was measured on the cross sections of surface layer. The wear properties of alloyed zone were tested on the testing machine T 08M using 'pin on disc' test.

**Findings:** Laser remelting process has produced a surface layer consists of hard ceramics particles of TiN and Ti<sub>2</sub>N phases spaced in martensitic matrix. The hardness of surface layer increases clearly in comparison with untreated alloy due to formation of TiN and Ti<sub>2</sub>N particles and depends on the volume fraction of nitrides. Their maximum value of the hardness (1500 HV 0.2) occurs on the surface of laser treated zone. Wear resistance of laser nitrided layer increases considerably in relation to base alloy.

**Research limitations/implications:** Research range was limited to microstructure, phase composition, hardness, fractography and wear resistance investigations. To estimate the influence of the laser nitriding process on corrosion resistance of the layer additional examinations will be performed in future research.

**Practical implications:** Laser remelting of titanium alloy in nitrogen atmosphere makes possible to obtain coatings composed of ceramic particles spaced in metallic matrix characterised by high hardness and wear resistance.

**Originality/value:** The range of investigation included microstructure, phase composition, hardness as well as fractographic estimation and wear properties enables analysis of laser nitriding process efficiently.

**Keywords:** Metallic alloys; Microstructure; Wear resistance; Surface Treatment; Laser technology

## PROPERTIES

### 1. Introduction

The laser surface treatment techniques use mainly CO<sub>2</sub>-gas, continuous action lasers or YAG, impulse and continuous action lasers (rarely glass lasers), which power is within 0.5 – 5 kW. YAG lasers emit radiation of 1.064 μm wavelength, in case of molecular lasers the wavelength is 10.6 μm. Since the coefficient

of radiation absorption by the alloy surface decreases with increasing wavelength, especially in case of CO<sub>2</sub> laser treatment, the surface should be properly prepared (coarsened, oxidized or covered with protective layers) in order to improve its laser radiation absorbability. Continuous-action lasers enable possibility of smooth regulation of the speed of heating or cooling the surface layers of the material through the change of laser beam speed in relation to the surface under treatment. It is

especially important in thermal treatment of many materials, especially tool steel and titanium alloys [1-9]. Laser nitriding of titanium and its alloys is usually conducted with continuous-action CO<sub>2</sub> lasers. Continuous action or impulse-action Nd-YAG lasers or excimer lasers are rarely used. The radiation power density changes within 10<sup>8</sup> – 10<sup>10</sup> W m<sup>2</sup>. The basic technique here is blowing pure nitrogen or its mixture with argon or helium into the alloy layer. The microstructure of the nitrided titanium alloy layers depends on laser nitriding conditions. However, and author's own research revealed the presence of dendrites and needle-shaped release and included the analysis of the impact of laser machining on their morphology, size, distribution. However, the conclusions on the phase composition were formulated solely on the basis of the measurement of hardness and the data available in the existing literature. On this basis the researchers conclude that the dendrites are created by TiN, and needle-shaped release is the Ti<sub>6</sub>(N) phase of various nitrogen content in the solid solution. In some cases the presence of a dendrite form of TiN in Ti-6Al-4V after the laser nitriding (continuous action) is also confirmed by an XRD analysis [10]. The results of further research, have been based on the newest version of the Ti-N phase equilibrium state [11] and have confirmed the presence of other titanium nitrides in the nitrided layer. The critical review by H.A. Wriandt and J.L. Murray [11], who also provided analysis of the data on the Ti-N phase equilibrium, assumes that for the temperatures below 800°C there is a range of ε-Ti<sub>2</sub>N phase and a regular nitride δ'-TiN<sub>y</sub> occurrence between the α-phase and δ-phase zones. Moreover, W. Lengauer and P. Ettmayer stated that there are two additional nitrides: ξ-Ti<sub>4</sub>N<sub>3-x</sub> and ζ-Ti<sub>3</sub>N<sub>2-x</sub> [12,13] in that system. The analysis of the phase composition of the laser-nitrided layers, based on the data on the Ti-N phase equilibrium system, should not neglect the fact that phase transitions during laser machining occur under ultrahigh heating and cooling rate, which may lead to creation of metastable phases [14-16].

## 2. Material and experimental

The material tested was vacuum melted martensitic two-phase α+β 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 α+β 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.

The sample surfaces were treated by a 1 kW CO<sub>2</sub> 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. Nitrogen gas was supplied at a flow rate of 10 l/min. The nitrogen was used 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%HNO<sub>3</sub>.

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. Fractographic 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<sub>α</sub> radiation. The Vickers microhardness under the load of 1.96 N was measured. The roughness of surface of treated material was examined using topography scanning system T 8000 made by Hommelwerke GMBH. The measurement was performed on the face of three adjacent tracks perpendicular to the laser beam relocate direction. The wear properties were tested on the testing machine T 08M using 'pin on disc' test.

## 3. Results and discussion

The alloyed surface topography examination (Fig. 1) specified roughness quantity. The plateau area curve (Fig. 2) describes usability of alloyed surface for mating in wear conditions. Microscopic investigations revealed presence of two characteristic zones attend laser treated area: zone with dendritic microstructure presents on the top of alloyed area and zone with acicular precipitations spaced in martensitic matrix occurring in depth of alloyed area (Fig. 3).

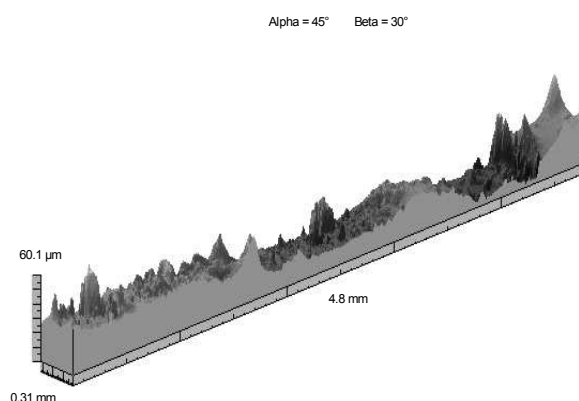


Fig. 1. Topography of the laser treated surface of Ti6Al4V alloy

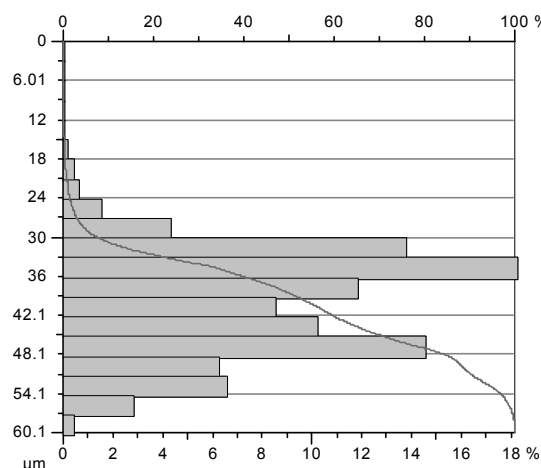


Fig. 2. The roughness bar chart and plateau area curve of the surface

The 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 TiN and Ti<sub>2</sub>N phases speaks for realisability of efficient nitriding of the surface layer of titanium alloy.

The hardness was measured on the surface and at different depths of the laser alloyed layer (Fig.4). The high level of the hardness was observed at alloyed surface (1500 HV0.2). It is attributed to the formation of hard nitrides. In deeper areas amount of martensitic Ti<sub>α</sub> phase increases, so hardness is reduced up to about 900 HV 0.2. 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 900 HV 0.2 to 600 HV 0.2 and dropped to the HAZ (420 HV 0.2) and finally to the base material (390 HV 0.2).

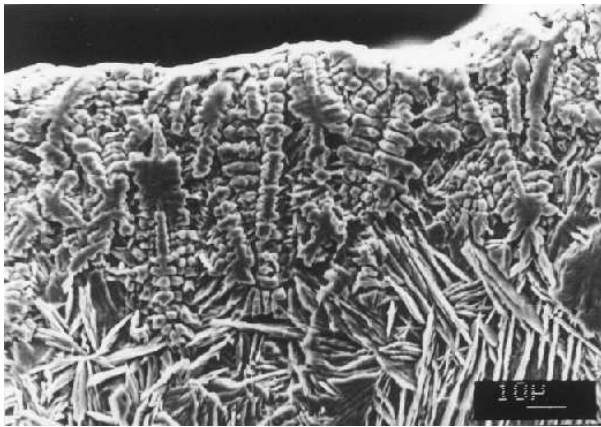


Fig. 3. Microstructure of laser melted surface layer of Ti6Al4V alloy

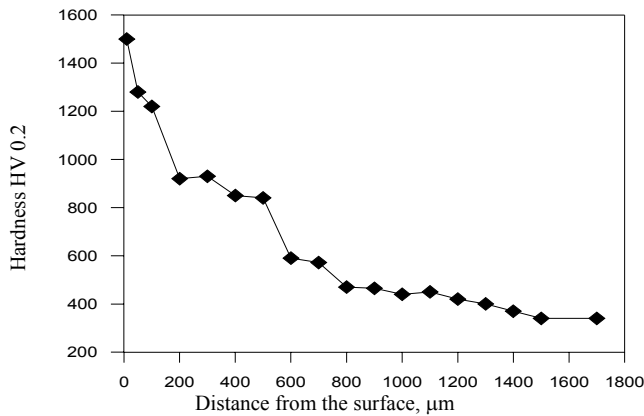


Fig. 4. Hardness of laser melted zone

There was a linear dependence between hardness and dendrites amount thus the hardness value may be used as laser alloying level index (Fig. 5). Results of hardness measurements confirm microstructural analyses as to the depth of individual areas present in laser treated zone. Thickness of laser alloyed area came to about 600 μm, heat affected zone – about 500 μm. It goes to show that laser alloying process may be efficiently used for improvement of surface properties.

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. 6),

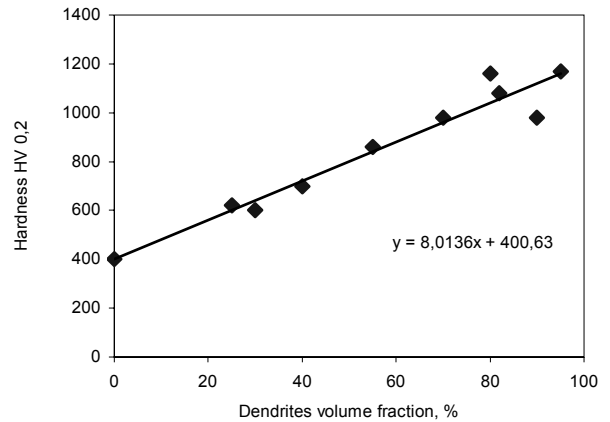


Fig. 5. Hardness versus dendrites volume fraction in laser treated zone of Ti6Al4V alloy

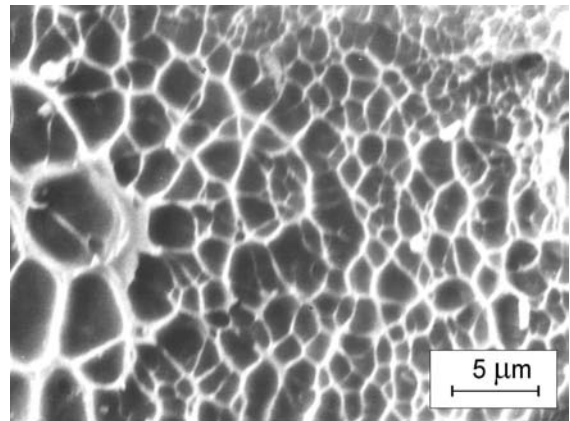


Fig. 6. Fracture surface of heat affected zone

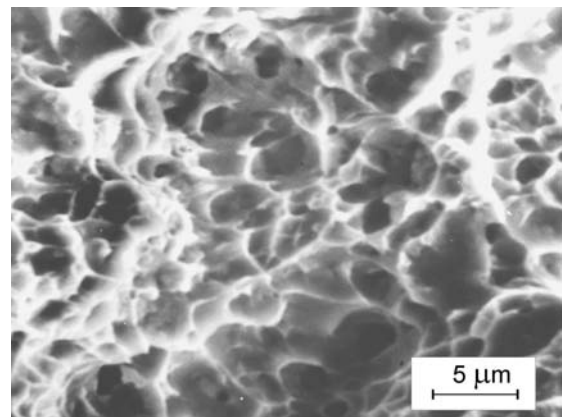


Fig. 7. Fracture surface of laser melted zone

whereas inside of alloyed zone occurred the complex character of fracture (Fig. 7). 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 TiN and Ti<sub>2</sub>N phases. The microcracks and pores weren't revealed on fracture surface, which proves its satisfactory plasticity.

Wear tests revealed decreasing of linear wear during dry friction on 6000 m sliding path in case of laser treated surface (Fig. 8). The increase the abrasion resistance takes place in spite of increasing the friction factor value – from 0.44 (untreated alloy) to 0.69 (laser nitrided).

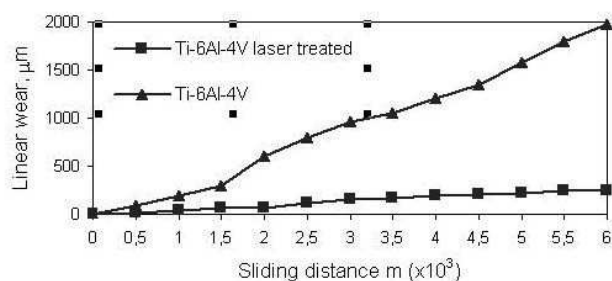


Fig. 8. Linear wear of both laser treated and untreated Ti6Al4V alloy

#### 4. Conclusions

Laser remelting of the Ti-6Al-4V titanium alloy has produced surface layer which consists of hard ceramic TiN and Ti<sub>2</sub>N 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<sub>α</sub> matrix. Below, the heat affected zone containing martensitic Ti<sub>α</sub> phase is present. The hardness obtained on cross-sectioned layer increases clearly in comparison with the base material. The high hardness level (1500 HV 0.2) can be attributed to the formation of TiN and Ti<sub>2</sub>N phases. Wear resistance improved considerably. The surface roughness compared to thickness of alloyed zone makes efficient utilization possible.

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