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# DUPLEX SURFACE TREATMENT OF Ti6Al4V ALLOY

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**ABSTRACT**
*Described technology of Ti6Al4V alloy plasma surface treatment is based on combination of both plasma nitridation of the component in micropulse plasma when nitridation atmosphere consisting nitrogen and hydrogen is used and subsequent deposition of thin $TiO_x$ film.. The process is performed so that a layer of nitrides and other chemical components may be formed on the surface of nitrided components. The formed layer is characterized by very good mechanical properties, e.g. an excellent adhesion and. higher layer hardness.*
**Keywords:** plasma nitriding, Ti6Al4V alloy, chemical composition, structure, properties

## 1. EXPERIMENTAL

Ti $\alpha+\beta$ alloy Ti6Al4V (3.7165, grade 5, DIN 17851) was used for experiment. Chemical composition according to DIN standard and measured by EDXS method for selected chemical elements is presented in Table 1. Plasma nitriding process was carried out on the PN 60/60 equipment according to parameters in Table 2. For experiment two samples were used.  Parameters of subsequent coating process are in Table 3.

*Table 1. Chemical composition of used Ti6Al4V alloy*

| Method | Chemical composition (weight %) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Ti | Fe | Ni | O | Al | C | V | H |
| DIN standard | rest | 0.25 | 0.05 | 0.20 | 5.50 - 6.75 | 0.08 | 3.5 - 4.5 | max. 0.015 |
| EDXS | rest | 0.13 | 0.06 | - | 5.61 | - | 3.75 | - |
| *Parameters of EDXS analysis: U = 30 kV, M = 100 x, I = 67 pA, WD = 20.50 mm, detector UltraDry* | | | | | | | | |

Chemical composition of substrate alloy was measured by EDXS method (Noran system Six), depth profiles were evaluated by GDOES/QDP (SA2000 spectrometer). Calibration of nitrogen: JK41-1N and NSC4A standards. Microstructure and surface morphology was evaluated by electron and light microscopy (Vega TS 5135 electron microscope and Olympus digital camera on the Neophot 32 light microscope), respectively. Surface structure was tested by 3D topography method (TALYSURF CLI 1000) with confocal gauge before and after treatment. Mechanical properties, such as layer thickness and microhardness were measured by indentation method (M400 microhardness tester). Other properties (adhesion, corrosion resistance) were evaluated, too. Relations among chemical composition, structure and diffusion layer properties were briefly discussed.

*Table 2. Parameters of plasma nitriding process*

| **Sample 1 – two stage process (short time-higher temperature)** | | | |
|---|---|---|---|
| **Parameter** | **Plasma cleaning** | **Nitriding-stage 1** | **Nitriding-stage 2** |
| Temperature (°C ) | 525 | 540 | 570 |
| Time/Duration (min, h) | 30 min | 8 h | 8 h |
| Flow $H_2$ ($l.min^{-1}$) | 20 | 25 | 8 |
| Flow $N_2$ ($l.min^{-1}$) | 2 | 5 | 24 |
| Voltage (V) | 800 | 520 | 510 |
| Pulse length (μs) | 110 | 90 | 90 |
| Pressure (Pa) | 80 | 270 | 260 |
| **Sample 2 – one stage process (long time-lower temperature)** | | | |
| **Parameter** | **Plasma cleaning** | **Nitriding** | |
| Temperature (°C ) | 510 | 520 | |
| Time/Duration (min, h) | 30 min | 40 h | |
| Flow H2 (l.min¯1) | 20 | 24 | |
| Flow N2 (l.min¯1) | 2 | 8 | |
| Voltage (V) | 800 | 510 | |
| Pulse length (μs) | 100 | 100 | |
| Pressure (Pa) | 80 | 280 | |

*Table 3. Parameters of subsequent coating process*

| **Coating** | Temperature (°C)/ Duration (min) | Current density ($mA.cm^{-2}$) | Washing (water) |
|---|---|---|---|
| $TiO_x$* | 15-25/5-10 | 2-4 | 2 min |

## 2. RESULTS AND DISCUSSION

Depth profiles of plasma nitrided layers (Figure 1 and Figure 2) for both nitrogen and titanium are in good agreement with the proposed plasma treatment regimes. Nitrogen content decreases along the layer depth (from surface to substrate). As for nitrogen concentration, there is local maximum about one micrometer from the surface. Existence of this maximum was verified by microstructure evaluation, too. Surface morphology for samle 1 and sample 2 is the same type, but with different roughness and other surface structure parameters (Figure 3 and Figure 4). This result is in conformity with metalographic measuring and next additional (e.g. 3D surface topography) evaluation. Qualitative and quantitative results of 3D surface topography measurements are in Figures 5 and Figure 6. The most important parameters of surface structure are presented at the same time.

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* *Anodzing in $H_2SO_4$ based electrolyte. (50-400 g/l water solution).*

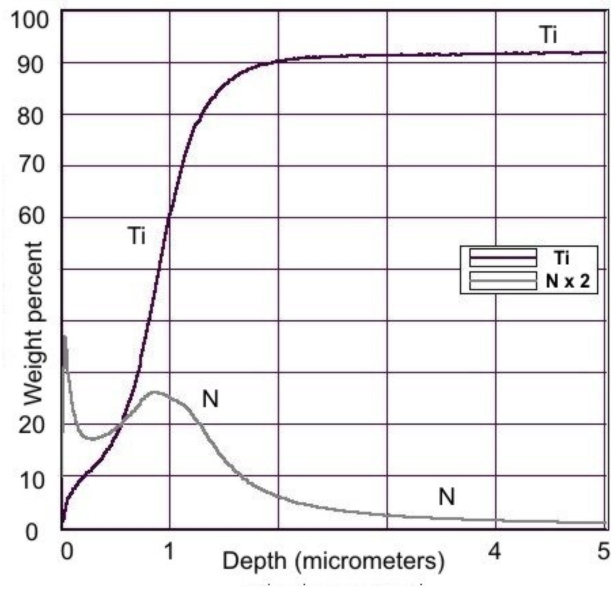



*Figure 1. Quantitative depth profiling (GDOES/QDP), after plasma nitriding, sample 1*

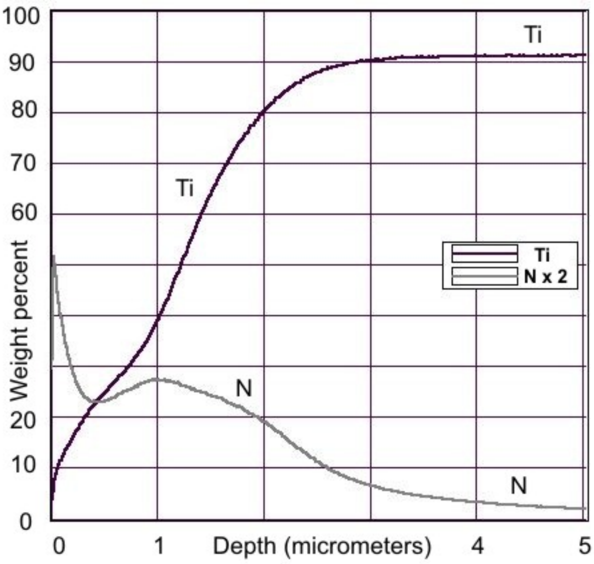



*Figure 2. Quantitative depth profiling (GDOES/QDP), after plasma nitriding, sample 2*

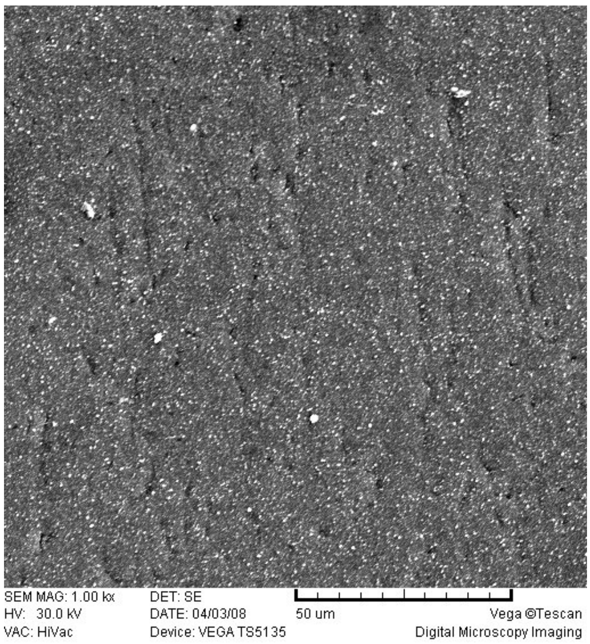



*Figure 3 Surface morphology after plasma nitriding, sample 1*

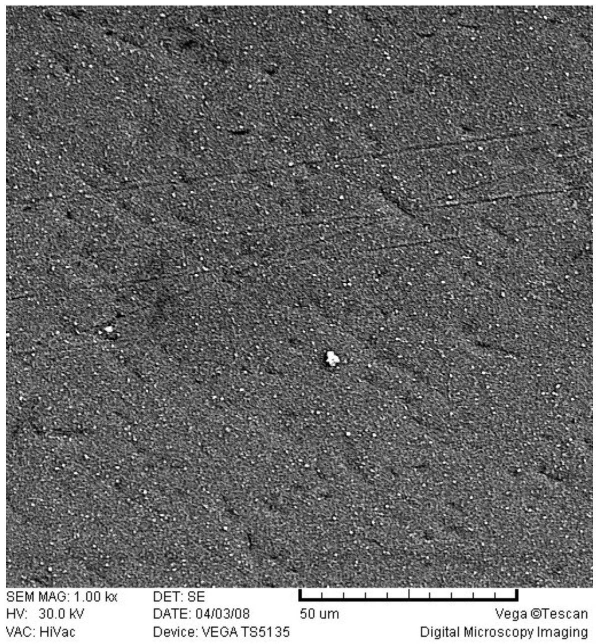
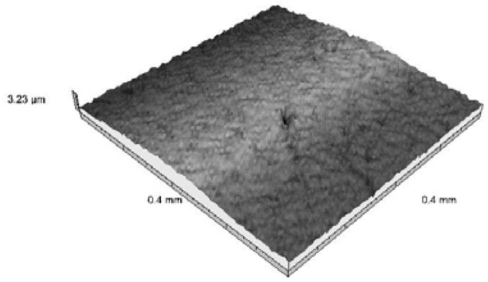
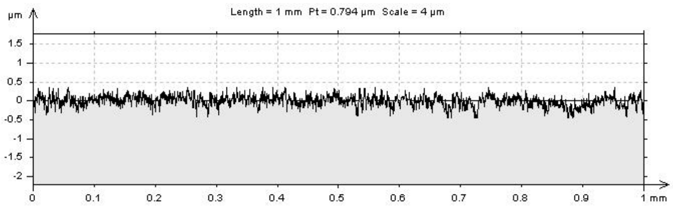


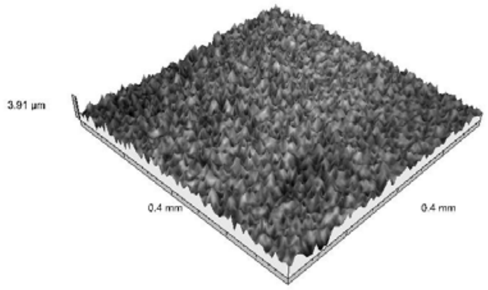

*Figure 4. Surface morphology after plasma nitriding, sample 2*

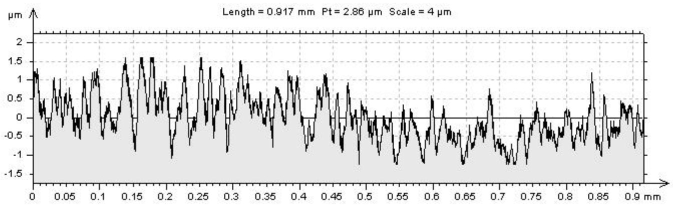





*Sa = 0.365 µm; Sq = 0.432 µm; St = 3.23 µm*
*Ra = 0.0904 µm; Rq = 0.116 µm; Rt = 0.769 µm*
*Wa = 0.0289 µm; Wq = 0.0306 µm; Wt = 0.14 µm*

*Figure 5. 3D surface topography after plasma nitriding, sample 1*






*Sa = 0.49 µm; Sq = 0.618 µm; St = 3.91µm*
*Ra = 0.412 µm; Rq = 0.509 µm; Rt = 2.60 µm*
*Wa = 0.321µm; Wq = 0.341µm; Wt = 0.961µm*

*Figure 6. 3D surface topography after plasma nitriding, sample 2*

## 3. CONCLUSIONS

Plasma nitride layer on the Ti6Al4V alloy surface at two different regimes and subsequent anodizing were carried out. The focus was on the relations among chemical composition, structure and properties of created duplex coating (layer/thin film). From GDOES measurements, it follows that a variable composition depth profile after plasma nitriding can be fabricated. To modify surface properties, $TiO_x$ type of very well adhesive thin film with thickness in units of micrometers was subsequently deposited. This thin film improves not only final surface structure and surface properties, but also perfect appearance of treated surface. It can be used as a buffer layer for deposition of next coating, e.g. HAP type biocompatible ceramic.

## 4. REFERENCES

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