INFLUENCE OF Ti6Al4V ALLOY SURFACE PREPARATION ON FORMATION OF DLC LAYERS

VPLYV PRÍPRAVY POVRCHU ZLIATINY Ti6Al4V NA TVORBU DLC VRSTIEV

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Abstract

This paper deals with surface preparation influence on deposition characteristics and morphology of thin DLC (Diamond-Like-Carbon) layers on Ti6Al4V titanium alloy. Layers deposition after various surface metallographic preparation steps was performed. The influence of surface roughness and stresses led into surface during preparation were also investigated.

Continuous ahdesive nanocrystalline DLC layers were successfully deposited on prepared surfaces. The mean grain size of nanocrystals produced was depending on number of impulses during deposition. With increasing of number of impulses increased the estimated mean grain size, from some tenth nanometers to about 100 nm. Surface roughness and surface stresses had no influence on DLC layers formation.

Článok sa zaoberá vplyvom prípravy povrchu titánovej zliatiny Ti6Al4V na depozičné charakteristiky a morfológiu tenkých DLC (Diamond-Like-Carbon) vrstiev. Vykonaná bola depozícia vrstiev na vzorkách po rôznej metalografickej príprave. Sledovaný bol aj vplyv drsnosti povrchu a vplyv napätí vnesených prípravou do povrchu vzoriek na následnú tvorbu vrstiev.

Na pripravených povrchoch boli úspešne vytvorené súvislé nanokryštalické DLC vrstvy. Stredná veľkosť vytvorených nanokryštálov bola závislá od počtu impulzov pri depozícii. S rastúcim počtom impulzov stredná veľkosť nanokryštálov narastala, od niekoľko desiatok po asi 100 nm.

Key words

titanium alloy, metallographic preparation, sample surface, DLC layer

zliatina titánu, metalografická príprava, povrch vzorky, DLC vrstva

Introduction

The demand for application of implant and prosthetic surgery is steadily increasing, particulary as the average age of the population continues to rise. For many years, the metals and alloys are used in prosthetic devices to replace or repair parts of the human body, mainly due to their unique mechanical properties. Especially for orthopeadic surgery the pure titanium and titanium alloys are widely used. Their application as a implant material is based on their high inertia, light weight, excellent biocompatibility and good fatigue resistance [1-3].

A large amount of research in last decades has been devoted to surface modification of implant alloys. The main reason was to improve their biocompatibility, enhance bone bonding and reduce wear or corrosion in body fluids. To the surface coatings that can markedly improve the biological compatibility and wear resistance of implants belong also thin diamond and other carbon- based films [1, 2]. There are many methods for diamond film deposition. Good deposition rates and uniform coatings are attainable by plasma assisted techniques [2, 4].

The aim of this paper was to evaluate the influence of surface preparation on deposition characteristics and morphology of thin DLC films produced on Ti6Al4V alloy. The influence of surface roughness and stresses led into surface during individual metallographic procedures as well as layer stresses were also investigated.

Ti6Al4V titanium alloy

Ti6Al4V (ASTM B265) titanium alloy is most used titanium grade. It is a two phase $\alpha+\beta$ titanium alloy, with aluminium as the α stabiliser and vanadium as the β stabiliser. The properties of Ti6Al4V are depending on proportion of both phases in alloy microstructure and temperature of processing. A typical proportion of β phase in equilibrium state is from 10% up to 18% [3, 5].

The alloy is obviously produced by primary melting using vacuum arc, electron beam or plasma arc melting. Remelting is achieved by one or two vacuum arc steps [3, 6].

The main features of Ti6Al4V titanium alloy can be summarised as follows below [3, 7]:

- *Mechanical properties*. Ti6Al4V is a high-strength alloy, with typical ultimate strength at room temperature up to 1180 MPa. Other tensile properties of this alloy at room and elevated temperatures are shown in Fig. 1. Hardness in annealed condition is 30-34 HRC and about 35- 39 HRC in aged condition.
- *Physical properties*. Ti6Al4V has a low specific weight of 4,47 kg/m^3. Its melting range is 1538-1649°C, β transus temperature is 999°C (±14°C). Other physical properties are listed in Table 1.

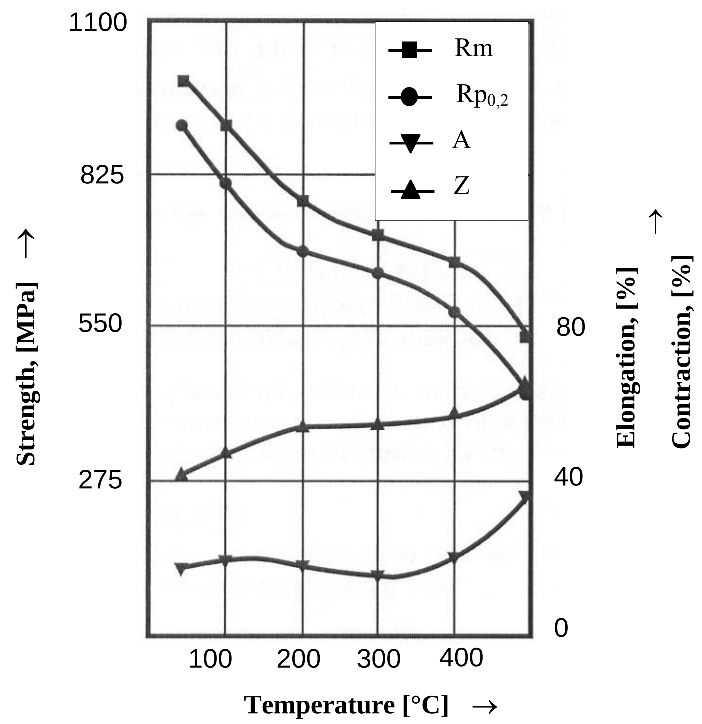


Fig. 1. *Typical tensile properties of Ti6Al4V alloy at room and elevated temperatures [3]*

PHYSICAL PROPERTIES OF TI6Al4V TITANIUM ALLOY [3]

Table 1

Coefficient of	
thermal conductivity	*at 25°C*: 7,2 W/mK
thermal expansion	*at 0- 100°C*: 8,6 . 10^{-6} K *at 0- 300°C*: 9,2. 10^{-6} K

- *Technological properties*. Ti6Al4V alloy is difficult to form at room temperature even in the annealed condition. Therefore several forming operations such as bending or stretching are performed on annealed material at temperatures up to 650°C, without affecting mechanical properties. Hot sizing or shaping can be done in 540-650°C.
  Ti6Al4V can be machined with slow speeds and large amounts of cutting fluid.
  Thereinafter, alloy is easily welded in annealed condition or partially aged condition, with ageing being completed during the post weld heat treatment. Precausions must be taken to prevent oxygen, nitrogen and hydrogen contamination. Fusion welding can be done in inert gas filled chambers or using inert gas welding. Spot, seam and flash welding can be performed without resorting to protective atmosphere.
- *Application*. Ti6Al4V can be used from cryogenic temperatures to about 430°C. It is used in the annealed condition and in the solution treated condition.
  Some of Ti6Al4V applications include compresor blades, discs and rings for jet engines, airframe and space components, pressure vessels, rocket engine cases, helicopter rotor hubs, fasteners, critical forgins requiring high strength-to-weight ratios, medical and surgical devices.

Experimental

The chemical composition of investigated Ti6Al4V alloy is given in Table 2. Rolled 500 x 1000 x 30 mm titanium alloy plate was cut in order to produce samples 10 x 10 x 3 mm in size. Five individual metallographic procedures were selected for sample surface preparation. These procedures and corresponding sample labelling are listed in Table 3. Typical microstructure of experimental alloy is shown in Fig. 2.

CHEMICAL COMPOSITION OF Ti6Al4V Alloy [wt. %] Table 2

Ti	*Al*	*V*	*C*	*Fe*	*others*
min. 87,7	5,5- 6,75	3,5- 4,5	max. 0,08	max. 0,3	max. 0,4

METALLOGRAPHIC METHODS EMPLOYED FOR TI6AL4V ALLOY SURFACES
Table 3

Sample	*Metallographic procedure*
1	wet grinding using SiC papers up to 1200 grain size
2	wet grinding + mechanical polishing using diamond suspension with 6 μm particle grain size
3	wet grinding + mechanical polishing using diamond suspensions with particle grain size up to 3 μm
4	wet grinding + mechanical polishing using diamond suspensions with particle grain size up to 0,7 μm
5	wet grinding + mechanical polishing using diamond suspensions + mechano- chemical polishing using STRUERS suspension consisting of 60% OP-S and 40% hydrogen peroxide

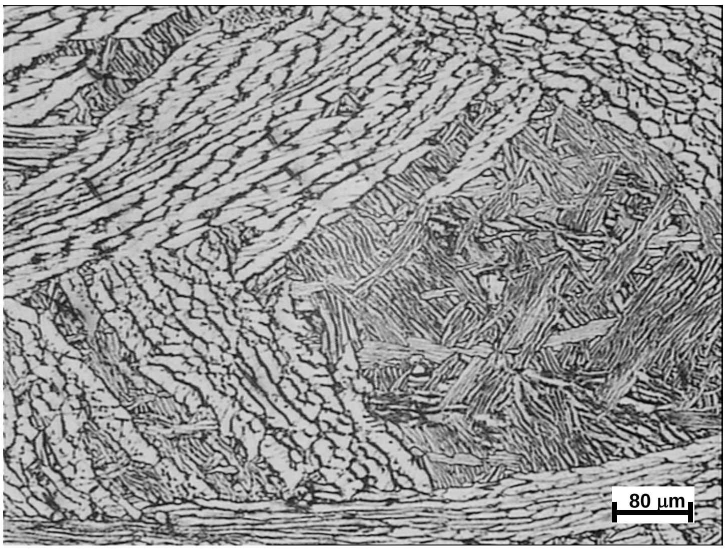


Fig. 2. *Microstructure of experimental titanium alloy*

Metallographically prepared surfaces were documented using NEOPHPOT 30 light microscope, equipped by image analyser. Subsequently the roughness measurements were performed, using SURTRONIC 3+ profilometer made by Rank Taylor Hobson. The surface characteristics were given according to EN ISO 4287 standard [8].

Stresses led into sample surfaces during metallographic preparation were also measured. These measurements were carried out in PHILIPS PW 1710 X- ray diffractometer by $\sin^2\Psi$ tensiometric method [9]. The analysis was performed using a CoKα target. The voltage 40 kV, current 30 mA, angular range Ψ: -30° ÷ 0° and 0,05° 2Θ step (with 3 seconds per step) were used during the measurement.

DLC layers were deposited in UVNIPA-1-001 vacuum system. Samples entering were sputtered in one vacuum cycle. All samples were first cleaned 10 minutes by Ar ions. DLC layers were deposited at low deposition temperature (about 150 °C). The pulse sputtering of graphite target was f = 10Hz, with number of impuses ranging from 5 to $30x10^3$. Nitrogen with 300, respectively 400 sccm flow rate was added during layer deposition into working chamber. Substrates were planetary rotating trough all deposition steps for homogeneous deposition.

The morphological evaluation of produced DLC layers was carried out in International Laser Centre (ILC) Bratislava using LEO scannning electron microscope operating at 10 kV.

Results and discussion

Sample microstructures

Typical surface micromorphologies obtained after individual surface preparation steps are given in Fig. 3. Metallographic preparation of titanium and its alloys is accompanied by intense surface strengthening and in consequence by pulling out the surface particles, as was well documented after sample grinding (Fig. 3a).

The application of mechanical polishing steps using diamond polishing suspensions led to elimination of observed grinding surface defects, Fig. 3 b-d. Mechano-chemical polishing first produced the smooth sample surface (Fig. 3e).

Roughness measurements

The surface roughness of the substrate is important for the adhesion of diamond films [10]. Therefore in our investigation the attention was also paid to sample roughness measurements.

Results of roughness measurement are summarised in Tab. 4. The roughness and also a total profile heigth on prepared surfaces was evaluated. Measurements were carried out from areas close to the top and bottom as well as from middle specimen region. The total evaluated length for each sample was 12 mm.

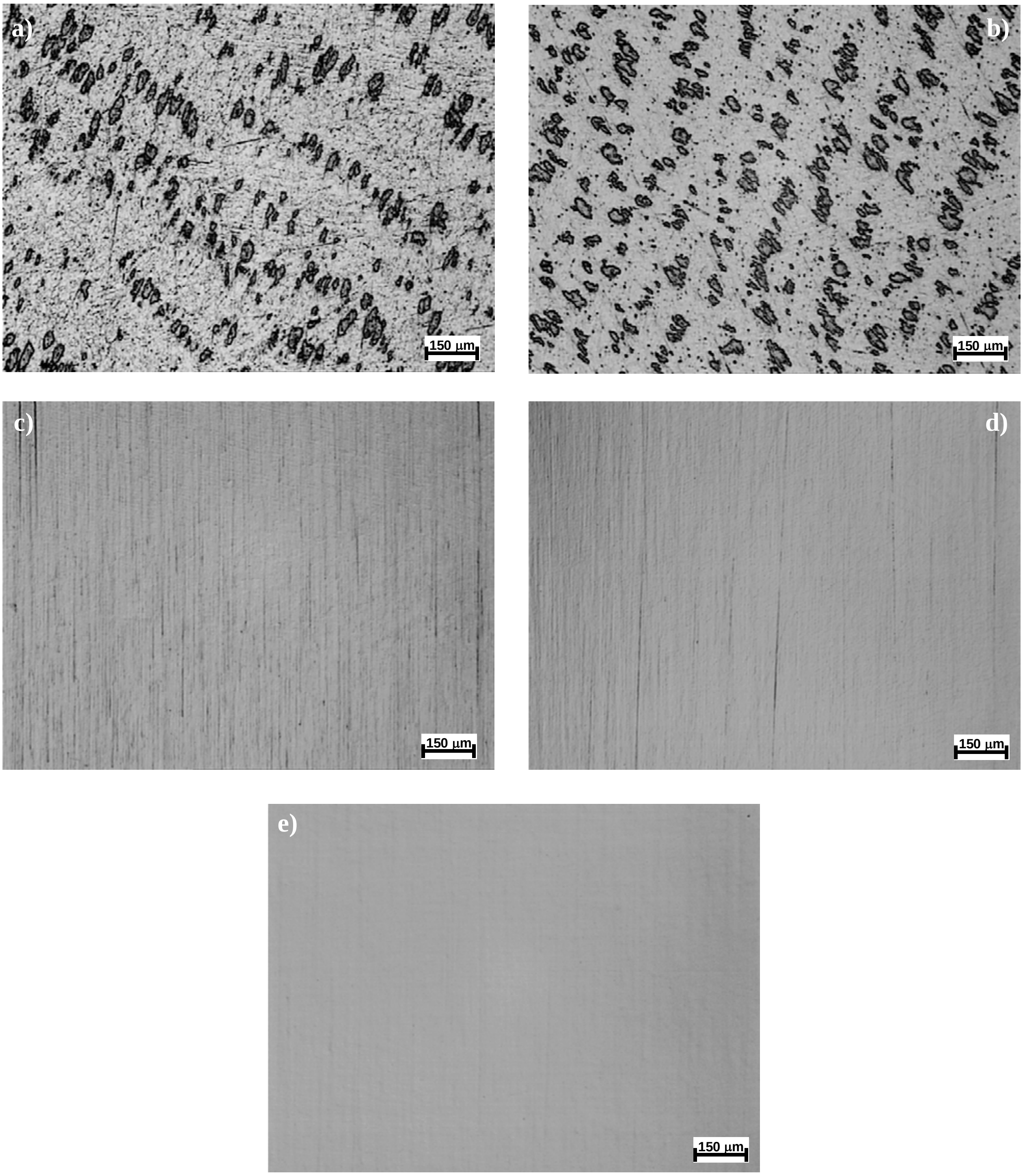


Fig. 3. *Ti6Al4V sample surfaces after individual metallographic preparation steps: a) sample 1; b) sample 2; c) sample 3; d) sample 4; e) sample 5*

Results of rougness measurement well corresponded to former microscopic observation of prepared surfaces. The maximum roughness of 0,13 µm and total profile heigth of 1,2 µm were measured after sample grinding. Eliminating of surface defects by application of mechanical polishing was accompanied by roughness decreasing, Table 4. After 0,7 µm diamond polishing the total profile heigth and surface roughness was approximately less by half to these after grinding.

Application of mechano-chemical polishing led into least surface roughness of about 0,035 µm.

RESULTS OF ROUGHNESS MEASUREMENTS ON PREPARED SURFACES (MEAN VALUE OF THREE MEASUREMENTS)

Table 4

Sample	*Roughness R_a [µm]*	*Total profile heigth R_t [µm]*
1	0,13	1,2
2	0,125	1,13
3	0,06	0,93
4	0,056	0,76
5	0,035	0,43

X-ray analysis

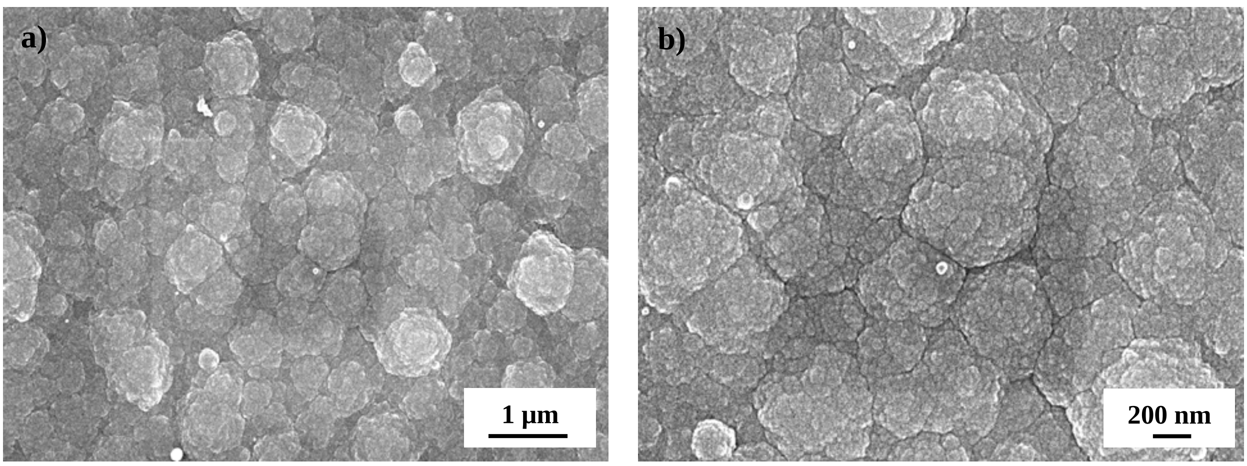
From tensiometric data measured on analysed samples the Young´s modulus E= 114 GPa and Poisson ratio ν= 0,342 were first calculated.

There were detected zero values of deformation (or stresees, respectively) in surface layers of grinded and mechano- chemically polished samples. Only in mechanically diamond polished sample surfaces increased compression stresses were measured. The stress value measured was about 120 MPa.

Scanning electron microscopy

Scanning electron micrographs showing typical morphology of DLC layers are given in Fig. 4.

At prepared titanium alloy surfaces a continuous adhesing nanocrystalline films exhibiting „cauliflower“ morphology were prepared. The thickness of films measured was about 10 to 15 µm. The grain size of nanocrystals produced was depending on the number of impulses during deposition. With increasing of number of impulses increased the estimated mean grain size, from some tenth nanometers for $10x10^3$ impulses to about 100 nm for $30x10^3$ impulses.



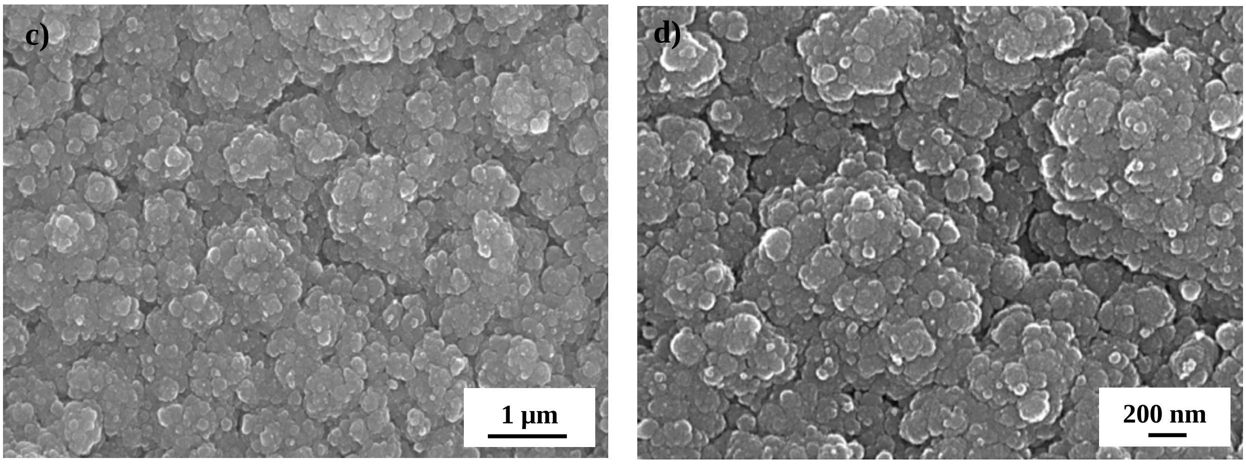


Fig. 4. Examples of „cauliflower" morphology of prepared DLC layers:
a) sample 2 at magnification 50 000x; b) sample 2 at magnification 100 000x;
c) sample 5 at magnification 50 000x; d) sample 5 at magnification 100 000x

Conclusions

The influence of various metallographic surface preparation steps on deposition characteristics and nucleation of thin DLC films produced on Ti6Al4V alloy was evaluated. Grinding, mechanical and mechano- chemical polishing were metallographic procedures chosen for substrate preparation for DLC layers deposition. The main conclusions are:

- continuous ahdesive nanocrystalline DLC films were successfully deposited on prepared surfaces. All layers exhibited the typical „cauliflower" morphology.
- there were detected zero values of stresses in surface of grinded and mechano- chemically polished samples. Stresses of about 120 MPa were measured only in mechanically polished sample surfaces. Their influence on layer formation was not indicated.
- the thickness of prepared films was about 10 to 15 μm. The mean grain size of nanocrystals produced was depending on deposition conditions and it was varying from some tenths nanometers to about 100 nm.

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