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LASER PROCESSING; REMELTING, ALLOYING, PULSED LASER DEPOSITION

Key words

laser, remelting, alloying, deposition, microstructure, texture, residual stress

Abstract

Laser application in technology is growing exponentially. In modern materials engineering, lasers are used for surface remelting and alloying as well as for explosive ablation of target materials and fabrication of thin mono- or multilayers. Origin of microstructure modification by laser remeling or laser alloying is attributed to specific solidification fine structure comprising often high concentration of alloying elements. Application of laser to produce thin films is very sophisticated. The method based on laser ablation of material and plume transfer to substrate seems to be very perspective. Any materials from pure elements to multicomponent compounds can be deposited using this method. The basis for laser surface modification as well as for pulsed laser deposition are given in this work basing on the own research work performed in this field. Wide spectrum of possible applications of laser technology in materials engineering is also presented.

Streszczenie

W ostatnich latach obserwuje się wykładniczy wzrost wykorzystania laserów w technologii. W nowoczesnej inżynierii materiałowej, lasery wykorzystywane są do nadtapiania i stopowania powierzchni, a także do odparowywania tarczy różnych materiałów na drodze eksplozyjnej ablacji i wytwarzania mono- lub wielowarstw. Podstawę do modyfikacji mikrostruktury na drodze laserowego przetapiania lub laserowego stopowania stanowi drobnokrystaliczna struktura często zawierająca duże stężenie dodatków stopowych. Metoda bazująca na laserowej ablacji materiału i transporcie strugi na podłoże postrzegana jest jako perspektywiczna. Z wykorzystaniem tej techniki osadzać można prawie nieograniczone spektrum materiałów; od czystych pierwiastków do wieloskładnikowych związków. Podstawy laserowej modyfikacji powierzchni oraz osadzania laserem impulsowym przedstawione zostały w pracy i zilustrowane wynikami badań własnych w tym zakresie. Podano szerokie spektrum możliwości wykorzystania technologii laserowych w inżynierii materiałowej.

1. Introduction

Rapid solidification process (RSP) is the application of high cooling rates or high undercooling to obtain high solidification rate (u>1 cm/s) [1-3]. RSP is the basis for microstructure selection in laser modification of material surface layer. Thus, understanding of the peculiarities of laser-surface interactions relies on the knowledge of the intrinsic properties of both lasers and surfaces [2-4]. The development of specific microstructure depends upon the solidification conditions and the shape of the solid-liquid interface, the transformation or even reaction processes which occur in the solid during the process of cooling

down to the room temperature. Depending on the solidification conditions, various microstructures are formed, namely, planar, cells, dendrites. All these morphologies may be obtained in single-phase solidification or in multiphase (eutectic) solidification (Fig.1) [5].
Nucleation and growth of the solid phase is closely correlated with the applied laser processing, cooling rate and type of material [6].
Laser melting can be used to create supersaturated and highly alloyed materials with novel structures. Most of the beneficial effects of laser treatment can be attributed to specific types of solidification fine structure. The high-melt-pool temperature, resulting from a high power density of laser beam, enables the dissolution of even thermodynamically stable intermetallic phases and the formation of metastable phases due to the high cooling rates [4-6].
Stability of solidification front is also observed at high solidification rates. It is possible to show that for a given alloy and for the positive temperature gradient, the plane solidification front is formed when the growth rate is sufficiently high. The transition from the plane to cellular, dendritic and again cellular and plane solidification front occurs when the growth rate increases at small temperature gradient. The plot of temperature gradient versus solidification rate and solidification morphology showing a range of stable interface morphology is presented in Fig.2 [6]. Different metastable phases of various morphology (dendrite, eutectic, icosahedral) can be formed. Thus, knowledge of a phase diagram is important as it allows one to predict the microstructure formation. As properties of the laser modified surface are related to the formed microstructure, it is of great importance to

understand the nucleation and growth of micro-scale crystals during solidification in the layer subjected to laser melting.

Fundamentals of Solidification

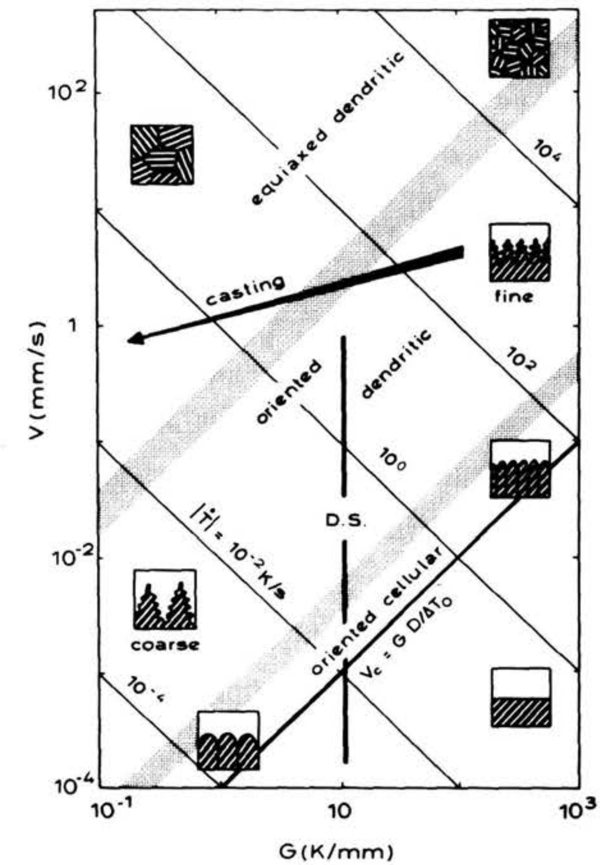

Fig. 1. Scheme of single-phase solidification morphologies

Continuous or pulsed lasers are used in the technologies of material surface treatment by remelting or alloying. In most cases, CO_2 (10600 nm) or Nd:YAG (1064 nm) lasers and recently diode lasers (800-900 nm) of high power are used for laser melting or alloying. The surface to be melted is shrouded by an inert atmosphere and the main characteristics of the process are: moderate to rapid solidification rates producing fine, near homogeneous structures (i); little thermal penetration, resulting in little distortion and the possibility of operating near thermally sensitive materials (ii); surface finishing of around 25 μm is fairly easy to obtain and can be significantly reduced by post-processing (iii); process flexibility (iv), due to software control and possibilities in automation (v).

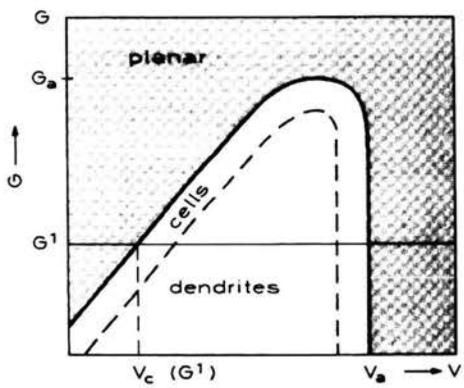

Fig. 2. The range of stable (planar) interface morphology

Pulsed laser deposition (PLD) belongs to modern technology which allows to produce thin layers of a nanostructure type [7,8]. Deposition of virtually any materials, from pure elements to multicomponent compounds, on various substrate, makes PLD very suitable technology in fabrication of thin layers for wide range of application. A typical set-up for the deposition of metallic alloys and multilayers consists of a target holder and a substrate holder housed in a vacuum chamber (Fig.3). In an UVH chamber ablated targets are struck at an angle of 45^0 by a pulsed and focused laser beam. The atoms and ions ablated from the target are deposited on substrates climbed on a heater. High-power pulsed lasers with nanosecond pulses mainly Nd:YAG and excimers are used as an external energy source to vaporize materials and to deposit thin films. A sequence of particular processes occurs during laser ablation from the target and deposition of the ablated material on the substrate surface are as follows: absorption of laser intensity, target heating , ablation of material, plasma formation, plasma expansion, instantaneous deposition rate in connected with a laser pulse. One of important advantage of the PLD method is stoichiometry transfer from target to substrate and production of multilayered materials. It could be also possible to influence on the transferred plume producing a special environment in the reactive chamber, thus producing a layer of a requested phase.

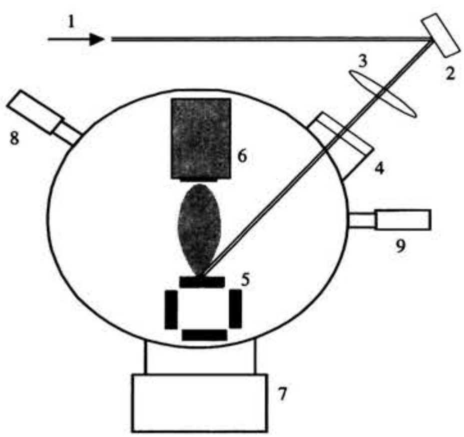

Fig. 3. Scheme of pulsed laser deposition; 1-laser, 2-mirror system, 3-focusing lenses, 4-laser port, 5-targets, 6-substrate with heater, 7-vacuum pomp system, 8,9- vacuum valves and gas supporting system

2. Experimental

2.1. Laser remelting and laser alloying

Aluminum alloys Laser treatment of aluminum and its alloys is somewhat difficult due to their low absorption of laser radiation. In laser treated Al-Si alloys (between 4wt.% and 20wt.%Si), the hypoeutectic alloys showed aluminum dendrites surrounded by flake-like eutectic regions [9]. Examinations of rapidly solidified Al-Fe alloys subjected to laser surface melting revealed different types of microstructures as to the growth rate i.e. at low growth rates, cellular/dendritic structures and at high growth rates, a banded structure consisting of a succession of light and dark bands which lay approximately parallel to the solid-liquid interface [10]. Examinations of Al-Zn alloys [11] revealed that extensive segregation of solute atoms took place, even during rapid solidification of laser melted surface layer. This turns out to be advantageous for resolving details of solidification processes in these alloys.

Steels The most beneficial effects of laser treatment of steels can be attributed to specific solidification structures, mostly of fine martensite in the case of laser melting [12]. Different alloying materials are used to improve the properties of various grades of steels, for instance chromium, nickel, tantalum as well as non metallic materials, like carbides or borides [13,14]].

Carbon steels The application of cw CO_2 laser to melt C45 revealed the formation of fine martensite microstructure. In the area of overlapping laser track, the martensitic microstructure was even finer

[10]. Laser alloying of carbon steels C45 with chromium or carbides B_4C and borides VB_2, CrB was used to improve in this way surface properties of bulk material [14]. A satisfactory correlation was observed between the results of structure examinations and the phase diagram Fe-C-B calculated with the Thermo-Calc program [15].

High speed steels Improved wear resistance can be expected from HSS with higher amount of primary and secondary carbides [16,17]. Surface melting techniques, like laser or electron beam melting, offer new possibilities for the modification of the structure in near-surface regions. The accompanying rapid solidification modifies the microstructure by structure refinement and by the supersaturation of crystals. In this way, the content of strong carbide-forming elements can be increased significantly by laser alloying. It was observed that laser melting and subsequent tempering increased hardness and improved wear resistance in the conditions of friction cause by static load. However, the resistance to dynamic loads was diminished [18,19].

Titanium alloys Titanium and its alloys are very attractive materials due to their low density, high mechanical strength, good corrosion resistance and biocompatibility. Nevertheless, the spectrum of applications of titanium alloys is actually rather narrow. The main reason is that these alloys have poor tribological characteristics during dry sliding, i.e. high wear and high coefficient of friction [20-22]. Experiments were performed on titanium alloys of the following grades: Ti1Al1Mn (wt.%, alloy of α type) and Ti6Al4V (wt.%, alloy of $\alpha + \beta$ type) [23]. Two types of lasers were used, namely, cw Nd:YAG laser (1064 nm) and cw diode laser (810 nm). Nitrogen environment was used in laser melting. Overlapping 1.5 mm wide laser tracks and individual laser track 7 mm wide were obtained for Nd:YAG or diode laser, respectively. The microstructure of the cross-section of the Ti1Al1Mn alloy, melted with Nd:YAG laser in nitrogen environment is presented in Fig.1.

Three zones, about 80 μm thick, could be distinguished in the surface layer, where the observed microstructure was different from the parent material. The first zone was located close to the substrate and it was about 20 μm thick. It was the heat affected zone, where remelting did not take place, and it was characterised with a fine microstructure. This zone transformed continuously into the intermediate zone with a fine dendritic microstructure. This zone was about 20 μm thick. The third zone, close to the surface, contained well-developed dendrites with the tips directed from the surface into the parent material. This observation seems to be very important, because in the case of laser remelting, the dendrites formed close to the surface are usually directed at the surface, i.e. opposite to these observed in the examinations [24]. That could be related to the manner of changing the solidification rate in the laser remelting process, which varies from zero at the solid/liquid interface to the maximum at the surface. It should be stressed, that the metallic titanium alloy was being remelted by a laser beam, while the TiN phase was solidified. Its melting point is much higher than the temperature of the liquid metallic pool; liquidus of TiN is in the range of 3000 to 3500^{0}C, which is related to the nitrogen content in the TiN. The chemical reaction between titanium and nitrogen could lead to the formation of the TiN phase, which was directly undercooled just after its formation.

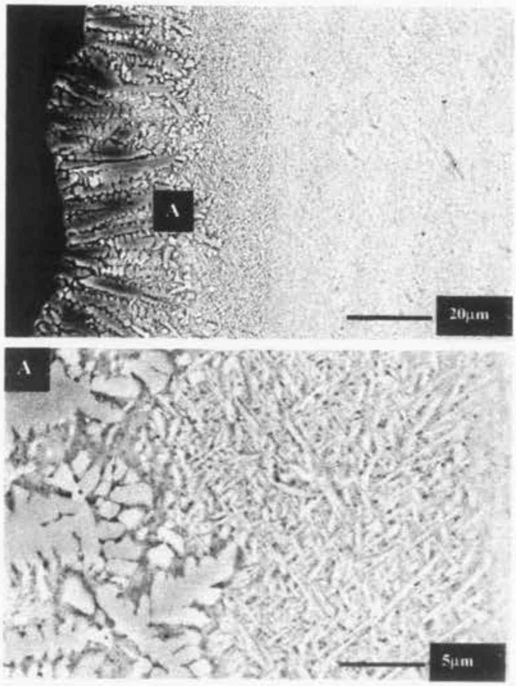

Fig. 4. SEM micrographs of a cross-section of the Ti1Al1Mn alloy subjected to remelting with Nd:YAG laser in nitrogen environment by overlapping laser tracks

Four different types of the microstructure of the TiN phase were observed (the TiN phase was verified by means of electron diffraction), namely, the single-crystal type was observed inside the dendrite arms and it was characterised by a high dislocation density; in the interdendritic regions one could observe elongated cells as well as equiaxial forms ; moreover, observations identified a fine-grained structure that yielded ring-shaped electron diffraction patterns (Fig.5).

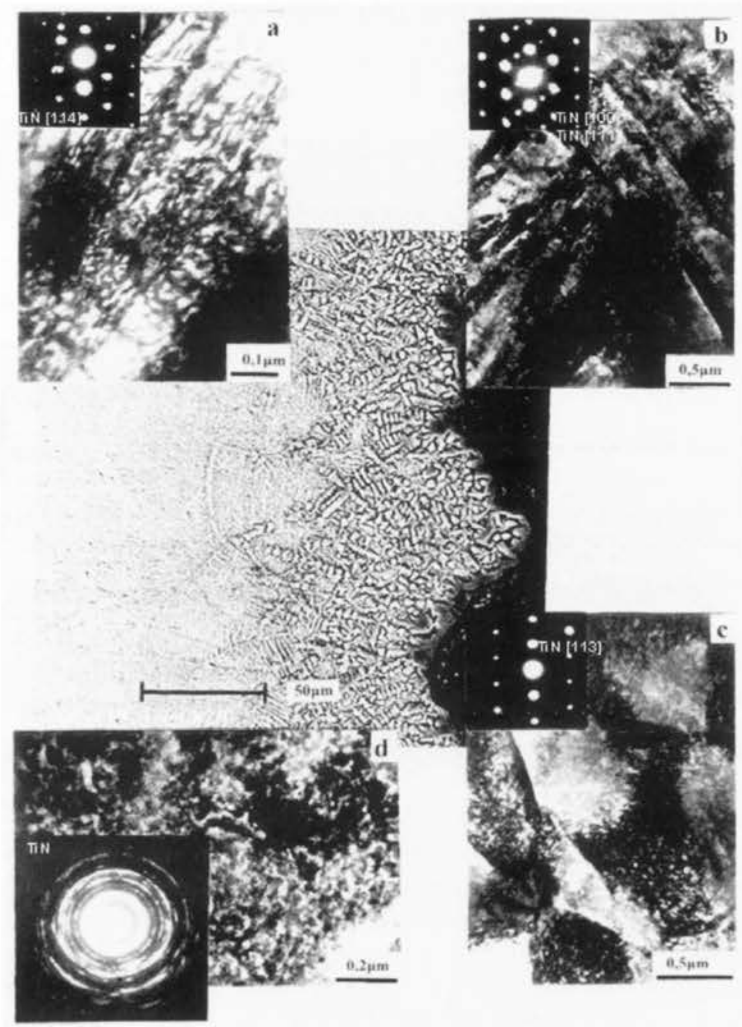

Fig. 5. SEM and TEM micrographs of a cross-section of Ti6Al4V alloy subjected to remelting with the diode laser in nitrogen environment, showing the cross-section of the remelted layer (centre) and details in the dendrite arm (a) and between arms (b,c,d)

The crystallographic texture was stated to be close to random orientation (Fig.6).

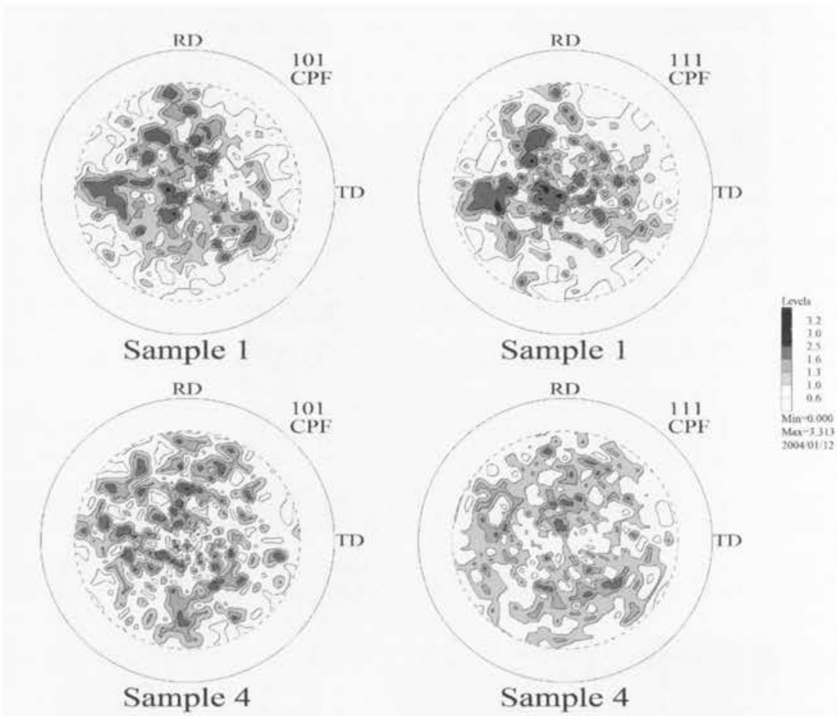

Fig. 6. Pole figure of crystallographic texture measured in the surface layer of titanium alloys subjected to laser remelting in nitrogen atmosphere (nitride phases); (101) pole figure corresponds to Ti_2N tetragonal phase while (111) pole figure corresponds to TiN cubic phase; a) Sample 1 - Ti1Al1Mn alloy subjected to laser remelting by Nd:YAG laser using overlapping laser tracks; b) Sample 4 - Ti6Al4V alloy subjected to laser remelting by diode laser using individual laser track.

2.2. Pulsed laser deposition – PLD technique

Among technologies of surface layers fabrication, one of the premier places belong to the methods which base on the photon beam in the form of a coherent laser radiation of the given wavelengths. The UV radiation is thus preferable due to low absorption. The laser beam can be applied in materials engineering for: annealing, remelting and sputtering or ablation. The technologies basing on annealing or remelting are connected with the substrate, while ablation is used with application of pulsed lasers [1-4,7,8].

Fabrication of coating by means of the laser ablation belongs to PVD (Physical Vapour Deposition) methods. In all these technologies, the process comprises three stages:

a. Evaporation of the material from the target
b. Transfer of the material from the target to the substrate
c. Deposition of the material on the surface of the substrate

There are used different methods for evaporation like: resistance, inductive, arc, electron and laser. The pulsed laser deposition (PLD) is based on the laser ablation. The focused laser beam on the target leads to the ablation of the material. Interaction between the laser radiation and the evaporated material causes an increase of the energy of this material and its expand. The originated plume comprises: atoms , molecules, electrons, ions, clusters, micron size particles and dropplets. The mean collision ways are small inside the plume after irradiation with laser beam of the high fluence and short pulses which results in expand of the plume in the vacuum having characteristic close to the hydrodynamic flow. Properties of the plume are related closely to the fluence and pulse repetition. When the temperature in the plume increase, the energy of the particle rises which positively influences on the quality of the deposited layers. The mechanism of material ablation is also related to the physico-chemical properties of the ablated target. At absorption of the laser radiation by the surface of the solid material, the electromagnetic energy is transferred into the thermal, chemical and even mechanical energy leading to evaporation, ablation, excitation, plasma formation and exfoliation. The processes proceeding by deposition are joined with [7,8]: absorption (i), target heating (ii), ablation (iii), plasma formation (iv), expand of plasma (v), kinetic energy of the deposited particles (vi) and temporary deposition rate (vii).

The basic functional elements of the deposition system are: laser with electric supporting system, reactive chamber, vacuum system, technological gas supporting system and valve system. Author of this paper working at different institutes is experienced in PLD method due to the international co-operation with the Laser Center in Leoben (Austria) and Institute of Physics in Praque (Czech Rep.). At present, it has been set up a system for PLD at the Institute of Optoelectronic Military University of Technology in Warsaw which starts to deposit tribological and biomedical coatings. The system based on the excimer laser ArF (193nm; 0.7 J/puls) .

TiN coatings on metallic titanium were produced by pulsed laser deposition using a system working with a Nd:YAG laser for ablation of titanium target. Nitrogen environment was applied in the reactive chamber in flow of 30sccm. Structure examinations X-ray diffraction (XRD), atomic force microscopy (AFM) and transmission electron microscopy (TEM) were performed to study surface morphology (AFM) and microstructure of the cross-section (TEM) as well as crystallographic texture and residual stresses (XRD).

Contribution of roughness of the metallic titanium sheet to the developed crystallographic texture and its generation by the layer as well as the residual stress in the substrate and the deposited layer was examined. Three materials were under examination i.e. not treated surface of the titanium sheet just after rolling with average roughness 0.9229 μm, mechanically ground, roughness 0.6409 μm and mechanically polished roughness 0.3483 μm. The heritage of the crystallographic texture in the substrate and the deposited layer was discussed (Fig.7).

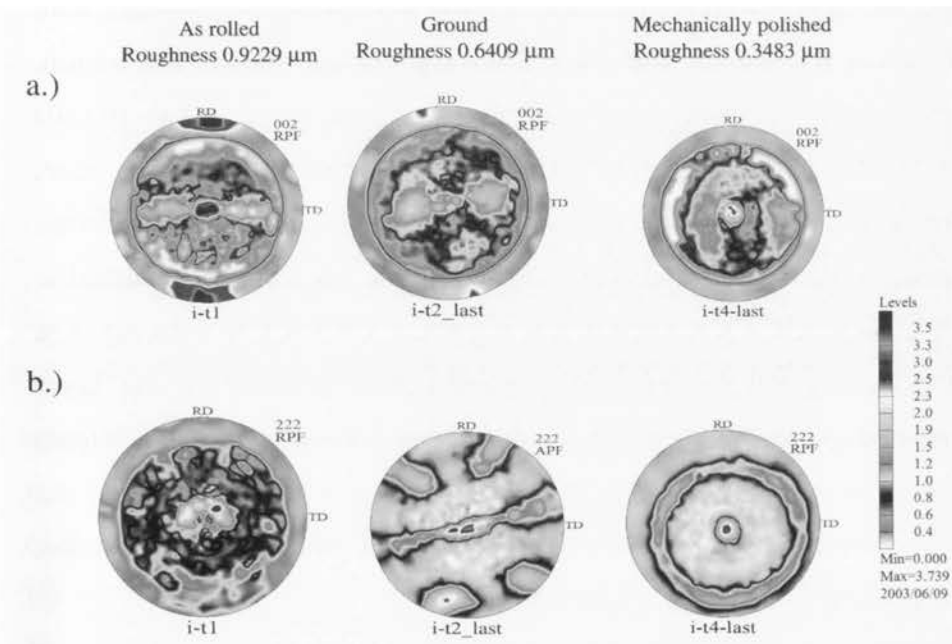

Fig. 7. Pole figures of : a.) titanium substrate; b.) TiN coating

Texture in the substrate in all cases are different and it could cause on the texture of the layer. In the first case when the TiN was deposited on as rolled substrate with 0.9229 μm roughness the maxima of the substrate and the layer are in the same position. The character of both pole figures is axial and well generated. In the second layer the orientation is strong in spite of the high disturbance of the orientation of the substrate. No generation is observed. The texture character of the TiN layer deposited on mechanically polished substrate (roughness 0.3483 μm) is axial, similar to the substrate. Inheritance is good. Application the position sensitive detector allows to draw pole figures of residual stress distribution (Fig.8). The most uniform and isotropic stress distribution was observed for the layer deposited on the substrate with the lowest roughness and the axial type of the stress distribution. Variation of the compressive residual stress in the range of 4 to 10 GPa measured in the TiN coating was stated in respect to the surface state. Thin foil examination on TEM revealed a blurred character between the nanocrystalline deposited TiN coating to the polycrystalline metallic titanium (Fig.9). Electron diffraction pattern achieved by selected area diffraction technique revealed nanocrystalline structure of the layer deposited on metallic which could be associated with the uniform distribution of the particles. In the samples with Ti substrate the interlayer has a blurred character from the TiN coating to the substrate, which could confirm good adhesion.

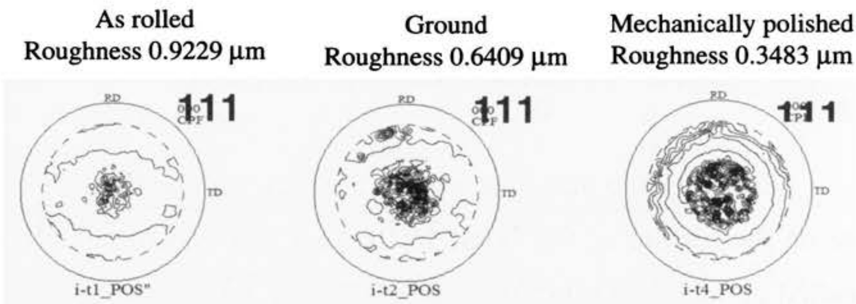

Fig. 8. Residual stress distribution in the layer

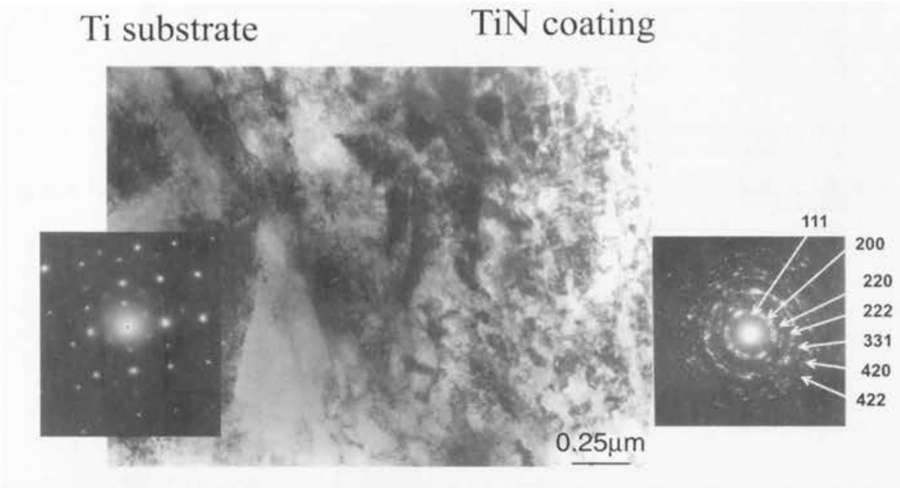

Fig. 9. TEM micrograph of TiN deposited on Ti

Perspective development of tribological coatings of new generation seems to be expected in functionally gradient materials. Appearing high value of stress in monolayer tribological coatings leads in many cases to micro-cracks formation. One of elimination method of this disadvantage is expectation in stress reduction due to interlayer application of super-elastic material which separates super-hard layers and could moreover block cracks propagation during exploitation.

Multilayer coatings basing on the Cr/CrN and Ti/TiN were fabricated using PLD method with application of a Nd:YAG laser working in basic mode 1064 nm with Q-switch generating nanosecond pulses of 10 ns with 50 Hz repetition. Cross-section of the multilayer coating Cr/CrN/Cr/CrCN was investigating by means of transmission electron microscopy (TEM) in order to solve their growth mechanism and microstructure (Fig.10).

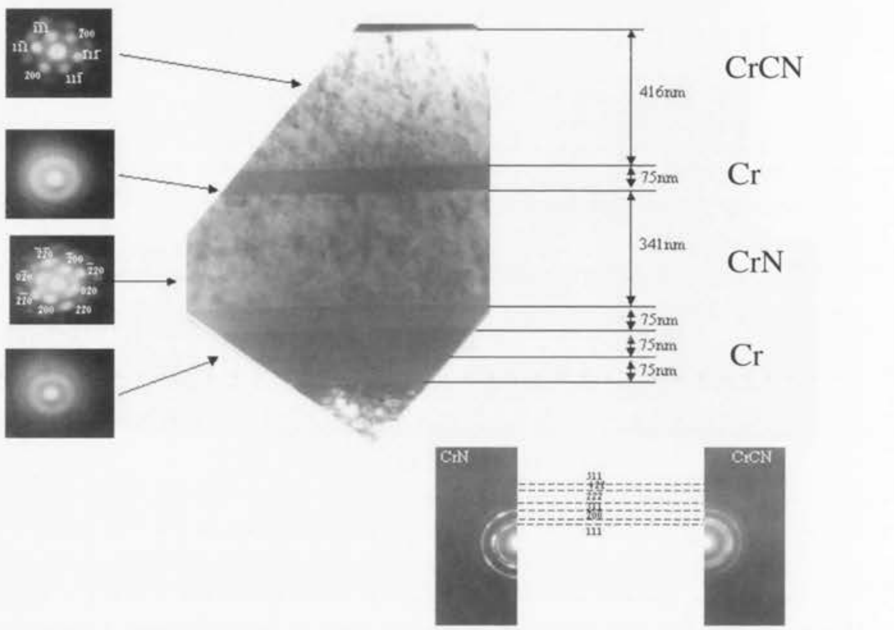

Fig. 10. TEM microstructure of the cross-section coating of the Cr/CrN/Cr/ CrCN fabricated by means of PLD method

The ion cutting with a focused gallium ion beam (FIB) was used as a preparation technique. Diffraction rings obtained by selected area diffraction technique as well as micro-diffraction obtained by the focused beam technique informed that CrN and CrCN have nanocrystalline structure. The blurred rings of the Cr layers could make an assumption that they have close to amorphous character. Technology of deposition was as follows: pure chromium (Cr) was deposited in inert atmosphere, then chromium nitride (CrN) in nitrogen atmosphere, then again pure chromium and at the end chromium carbo-nitride. The last layer was deposited in methane low pressure gas atmosphere. Initially, three chromium buffer layers were produced to increase the adhesion of the multilayer coating to the substrate. In the case of examined foil, it was possible to distinguish differences in the contrast in these layers (Fig.10 bottom). The first one was deposited with the highest kinetic energy i.e. 2 minutes in 20 sccm Ar atmosphere (initially vacuum was produced in the chamber), the next Cr layer was deposited 3 minutes in 25 sccm Ar atmosphere, and the last Cr layer was deposited 5 minutes in 30 sccm atmosphere Ar. The performed EDX analysis showed that each first chromium buffer layer had different amount of Cr atoms. The chemical analysis exhibited a high amount of oxide in all the layers, which should be explained that contamination of chromium (Cr) and chromium nitride (CrN_x) is usually observed in PVD processes. The high energetic pulsed plasma in the PLD process results in Cr and CrN_x deposition in conditions of a high oxygen trapping in the coating, changing the microstructure.

Details of the microstructure of the Cr buffer interlayer close to the substrate can be seen in the high resolution electron micrographs (HREM) presented in Figs.11-13. The images revealed that the pure chromium buffer layers have a nonocrystalline character (Fig.11). Different amount of crystallites in each layer influenced on differences in contrast in the first three chromium layers. The higher number of crystallites was in the first layer. The higher magnification of microstructure shows the interface between first and the second chromium layer (Fig.11 bottom) and the second and the third chromium layer (Fig.11 bottom).

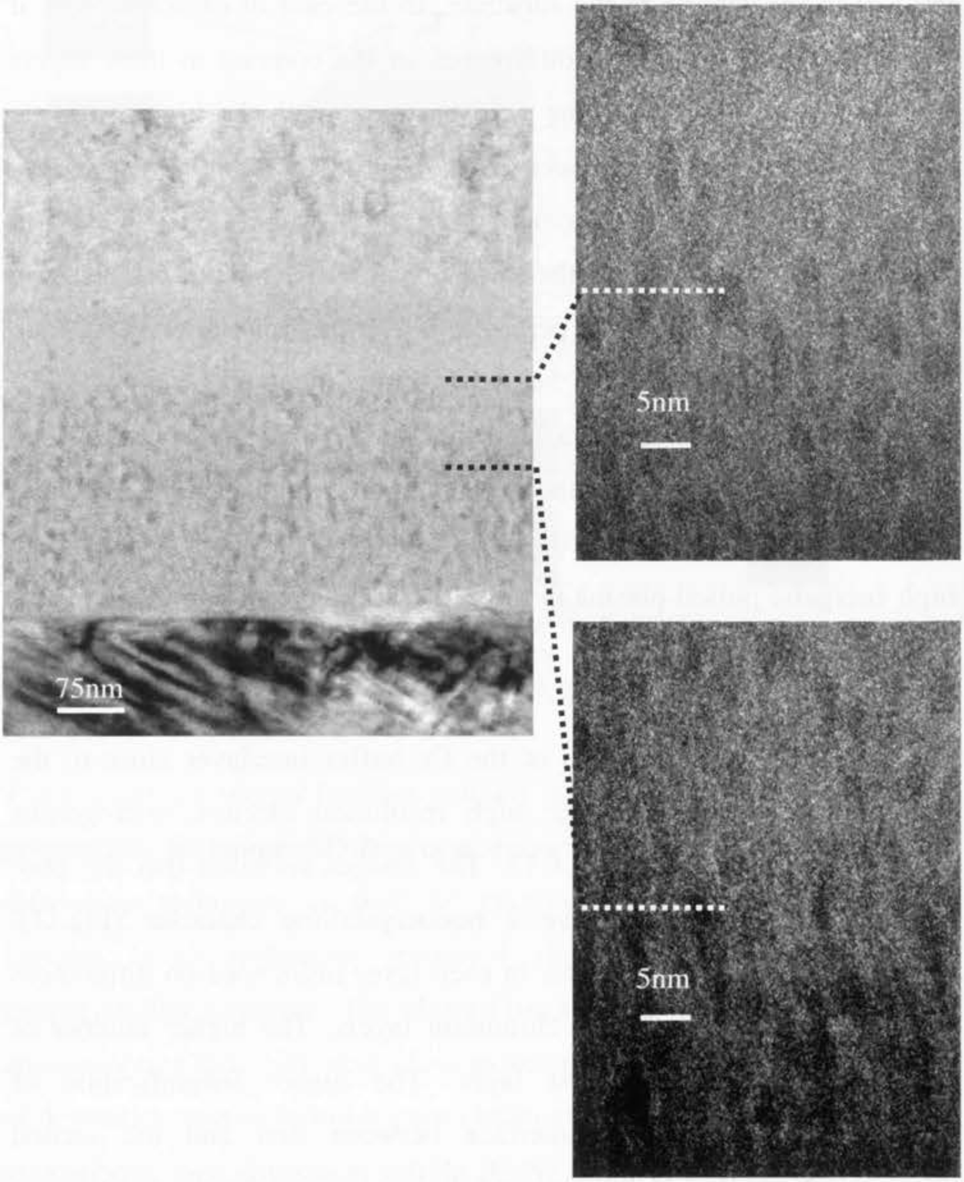

Fig. 11. HREM image of the Cr buffer layer

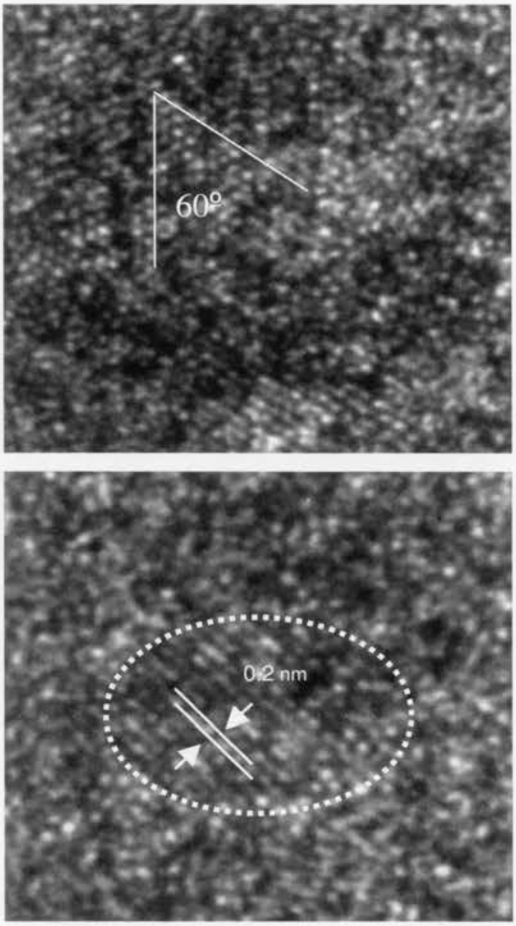

Fig. 12. HREM image of the Cr grain in the buffer layer

The elongated character of crystallites informs that the crystallization occurred during the deposition process. Based on the fact that the columns of atoms formed an angle of 60^0, the [111] orientation was found which was typical for Cr grains (Fig.12).

Crystallites of CrN layer, were also elongated, however, they were bigger than Cr once (Fig.13). The most visible area of the CrN crystallites was selected for the phase analysis. On the basis of the

Fourier transformation, the bcc structure was detected, which is characteristic for the CrN.

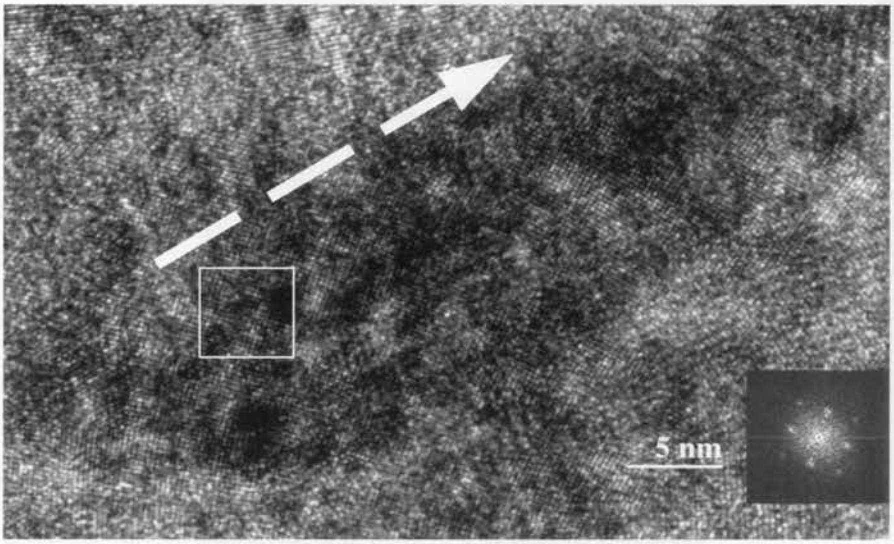

Fig. 13. HREM image of the CrN grain

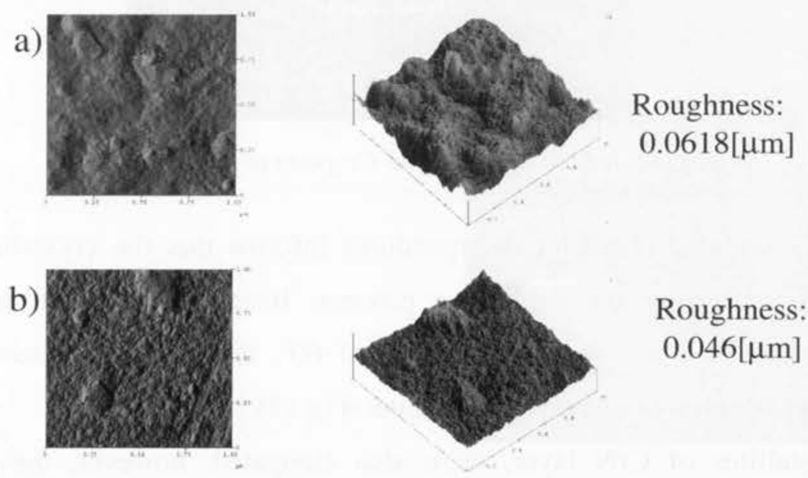

Fig. 14. AFM morphology of surface coatings produced by PLD;CrN (a) and TiN (b)

A mean roughness was in order of hundredths of micrometer which verified the PLD as the method producing high quality surfaces (Fig.14). Layers produced by pulsed laser deposition technique are characterized by high, compressive residual stress 12 and 4.5 GPa for Ti/TiN/Ti/TiN and Cr/CrN/Cr/CrCN, respectively. The experimental results obtained on materials with different numbers of layers based on Ti, presented that residual stress decreases with increasing number of layers (Fig.15).

The tribological wear tests were performed using the pin-on-disc method with application of 9.81N load and the obtained results showed the friction coefficient of order of 0.2 for Ti/TiN/Ti/TiN system. Moreover, the wearing process of super-hard TiN and compensation Ti layers could be examined by variation of the friction coefficient (Fig.16).

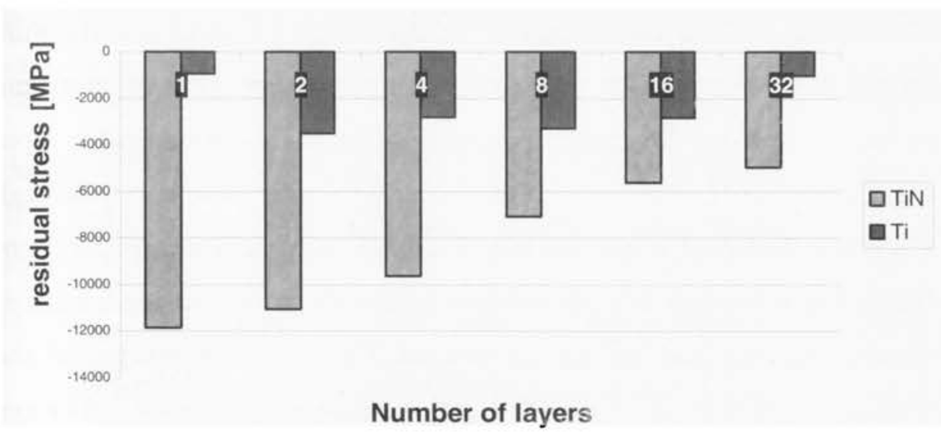

Fig. 15. Residual stress measured in the coating with different number of layers

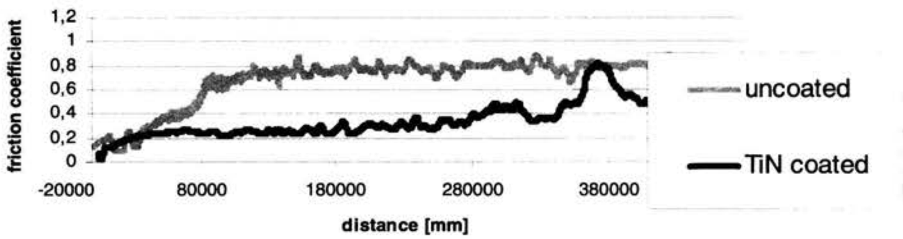

Fig. 16. Friction coefficient variation in the wear test for the Ti/TiN/Ti/TiN system

3. Concluding remarks

Optical energy is a convenient tool for surface engineering and it can be used as a localized method, which avoids damaging the component yet is capable of modifying selected areas. Understanding the peculiarities of laser-surface interaction in laser melting and alloying, as well as understanding of the details of rapid solidification processes both rely on the knowledge of intrinsic properties of lasers and surfaces. The use of laser technologies to study fundamental rapid solidification and diffusion control processes seems to be very practical. Materials treatment examines the possibility of practical application of fundamental changes of plain surface induced by laser radiation, such as heating, melting and plasma generation. Moreover, structuring of the surface in terms of controlled crystallization processes, ablation, generation of periodic structure and generation of thin films opens great perspectives for technical applications. The advantages offered by laser processing are the following: highly localized and clean nature of the process, low distortion and good quality of finish, economy of the process. The development of laser devices and reliable more compact lasers is now both economically and technologically feasible to use lasers in industry for surface treatment processes.

Titanium nitride layers will be potentially used for the implantable biomaterials. When blood comes in contact with biomaterial surface it is necessary to guarentee a degree of the roughness. It is necessary to form natural biolayer formed by blood proteins. The PLD method allows to control the surface roughness follows from the current experience and achieved results. A goal of the performed research examinations of the multilayer tribological materials of new generation has been a search for materials which could be used in miniature elements of medical equipment, namely. Fulfilling a non-failure service of elements at difficult exploitation conditions makes a new challenge for research workers. Application of the PLD method for fabrication of the multilayer tribological system seems to be helpful.

Acknowledgement

The financial support from the Polish Committee for Scientific Research project Eureka E!2841, No 62 E-88/SPB/Eureka T-08DZ 348/2002-2004 is greatly appreciated.

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