R. GAŁUSZKA, M. MADEJ, D. OZIMINA, A. KRZYSZKOWSKI, G. GAŁUSZKA

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THE CHARACTERISATION OF THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF DIAMOND - LIKE CARBON (DLC) FOR ENDOPROSTHESIS

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Preliminary Note – Prethodno priopćenje

The paper presents the results of research of DLC coating of a - C:H type obtained by using a technique of physical vapor deposition (PVD) on the surface of CoCrMo alloy, commonly used for the elements of the endoprosthesis. The surface has been observed using a scanning electron microscope (SEM). Analysis of the chemical composition and distribution of the different elements were performed using glow discharge optical emission spectrometry analysis (GDOES). It has been shown that the DLC elements are characterized by high hardness and good adhesion to the substrate.

Keywords: CoCrMo alloy, diamond - like carbon coatings, endoprosthesis, mechanical properties, microstructure

INTRODUCTION

The market of demand for medical implants, including hip prosthesis, steadily increases with declining age of the users. In recent years, there is noted a growth of the amount of implantation of medical implants, whose main goal is to replace diseased or traumatically damaged organs, such as hip replacement, knee, etc. [1, 2]. Biomaterials that are used to this are of different composition, structure and properties, but they share a common trait - they are biocompatible. These materials are subject to continual improvement, and the forthcoming new [3, 4]. The high demands that are placed on biomaterials cause that they are some of the most expensive man-made. This forces the need to use the latest materials and technologies in order to obtain prostheses for the best properties, especially high tribological and corrosion resistance, biocompatibility and mechanical properties appropriate [4]. The improvement of the operational properties of materials can be achieved by modifying the chemical composition, modification of the surface layer and the production of coatings having advantageous /desirable mechanical, tribological and corrosion properties [3, 5, 6].

An important role among biomaterials play carbon coatings, and their importance in medicine clearly increased with the mastery of techniques to produce by DLC [7, 8].

The amorphous carbon layers were received for the first time in 1971 by Aisenberg and Chabot [3, 9]. This discovery has become a landmark and technologies of their preparation are constantly being developed [10]. The amorphous carbon may be a mixture of carbon bonds sp^3, sp^2 even sp^1, with the possibility of the presence of hydrogen. It is characterized by a large variety of structures, and its properties depend mainly on the method and manufacturing parameters and the ratio of sp^2 bonds, occurring in graphite to sp^3 bonds present in diamond pattern [6, 8]. High hardness, low coefficient of friction and low wear tribological, chemical passivity and biocompatibility of diamond coatings, DLC made it posssible to use them in various industries [3, 6-10].

R. Gałuszka, The Jan Kochanowski University in Kielce, Poland
M. Madej, D. Ozimina, Kielce University of Technology, Poland
A. Krzyszkowski Kazimierz Pułaski University of Technology and Humanities in Radom, Poland
G. Gałuszka - School of Economics, Law and Medical Sciences in Kielce, Poland

MATERIALS AND METHODS

The coatings were deposited on CoCrMo alloy trade name Biodur CCM Plus, whose the chemical composition in Table 1, the most important mechanical proper-

Table 1 **The chemical composition of Biodur CCM PLUS / wt. %**

Co base	Cr 26 - 30	Mo 5 - 7	Mn 0,5	C 0,2 - 0,3
Ni 0,3	Fe 0,3	N 0,15 - 0,2		Si 0,6

Table 2 **The mechanical properties of the Biodur CCM Plus alloy**

Condition	Annealed	Warm Worked	Hot Worked
$R_{0,2}$ / MPa	585	930	760
R_m / MPa	1 035	1 310	1 100
Z / %	25	26	25
/ HRC	30	40	33
Modulus of Elasticity / MPa	240		
Poisson's Ratio	0,3		

ties are summarized and in Table 2. The alloy is produced by powder metallurgy method VIM - Vacuum induction melting.

The processes of applying a thin coating of diamond obtained with CVD technic run at elevated temperatures, which effects tempering its layers and lower hardness. Coating a - C: H was obtained in the process of chemical vapor deposition of plasma PACVD at a temperature of < 250 ° C

Observations of the structure was carried out by using a scanning electron microscope - SEM. In the study of the structure one used a microscope FEI XL30 SEM E-spectrometer equipped with an energy dispersive X-ray EDAX GEMINI 4000, with the possibility of testing in low vacuum.

A very useful technique for analyzing the chemical composition and distribution of individual elements was to analyze glow discharge optical emission spectrometry (GDOES) was carried out by using a spectrometer with glow discharge radio frequency (RF) signals produced by Jobin Yvon Horiba. Superficial quantitative analysis allows to introduce changes in the elemental composition of the sample, expressed as a percentage of atomic or weight, depth from the surface. Resolution into the GDOES analysis of the material is very high, measured in the nanoscale.

Micro-hardness measurements were made using Matsuzawa microhardness testers with Vickers and Knoop indentors. Using Knoop indenter allows using small loads to eliminate the hardness of the backing material. The study was conducted with a load of 98,07 mN force. The research of a grip and marking of signs of mechanical damage was done by scratch - scratch test using a REVETES CSM Instruments. The tests were performed with increasing loading force of 0 - 150 N at the following parameters: the speed of loading of 10 N/s, the speed of the indenter 10 mm/min, the length of cracks 10 mm type indentation - Rockwell diamond. The study was conducted on CoCrMo alloy, coated with DLC.

RESULTS AND DISCUSSION

Figure 1 shows a view of DLC coatings in cross-section together with its thickness measured.

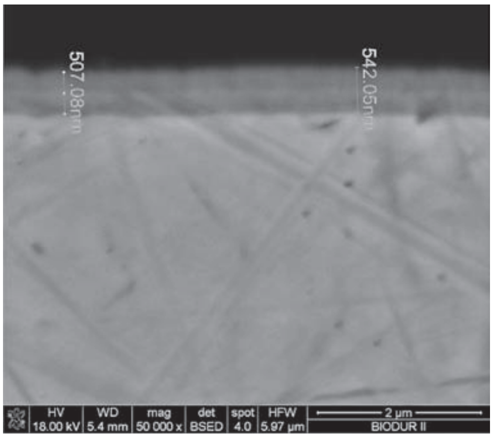


Figure 1 The microstructure of DLC coatings.

a)

b)

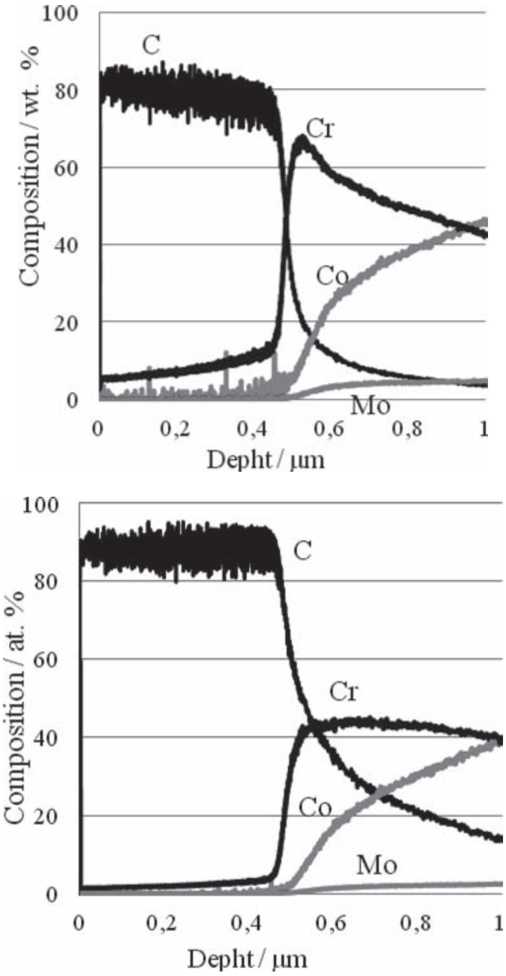


Figure 2 Elemental analysis of the DLC coating: a) mass; b) atomic.

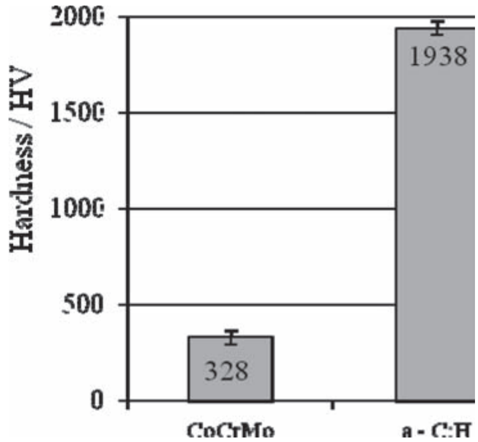
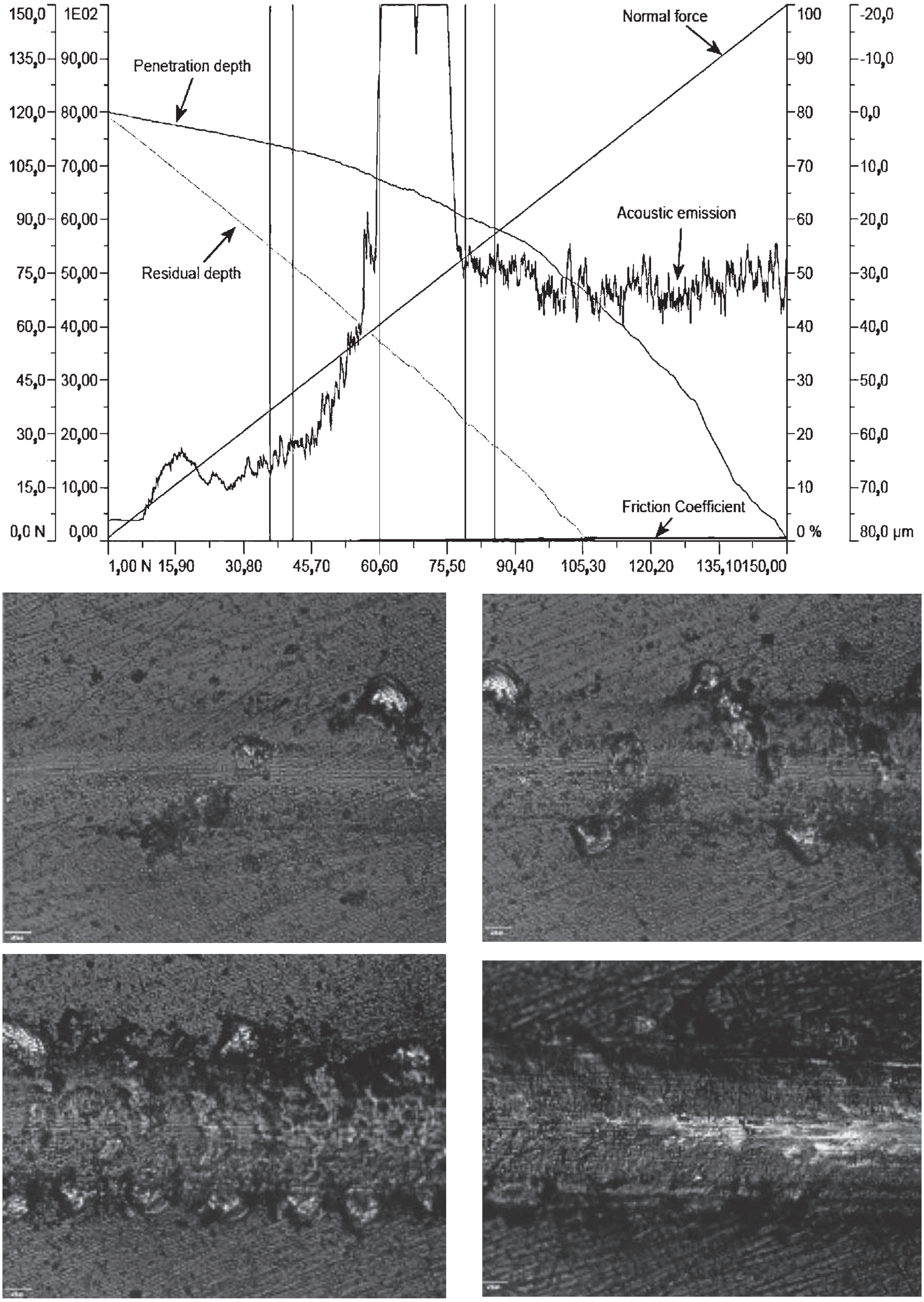


Figure 3 Microhardness of DLC coatings.

The resultant DLC coating has a thickness of about 525 nm. During follow-up there were no defects and discontinuities structure. GDOES analysis of a coating a - C: H (Figure 2) showed that it consists of the sub-coatings. The layer on the surface is mainly the carbon with a small extent of chromium. An interlayer lying between the substrate and the layer of a - C:H forms subcoat consisting essentially of chromium, as evidenced by a marked increase in the peak originating from the element.



a) b) c) d) e)

Figure 4 Scratch test: a) tangential force, friction coefficient and acoustic emission signal as a function of road paintings and damage the load: b) L_{C1} = 36,53 N, b) L_{C2} = 41,46 N, c) L_{C3} = 60,57 N and L_{C4} = 79,53 N.

On the basis of microhardness tests confirmed a clear increase in the hardness of the coating compared to the substrate (Figure 3).

The lowest force at which the coating degradation called critical load L_C which is a measure of adhesion of the coating to the substrate were measured by the scratch test. The results are shown in Figure 4.

The value of the critical load was determined from the variation of the acoustic emission. The study was accompanied by observations made at the optical microscope coupled with a measuring device.

The graph (Figure 4a) shows clearly the signal of acoustic emission. The sharp jump in its value corresponds to the first crumbling out and first cracks of coating. The images of scratches on the coating surface after the test are summarized in Figures 4b-4e.

The critical load L_{C1} is registered on the chart depending on the friction force and acoustic emission load, as first, the small jump of acoustic emission signal. Wheres critical loads L_{C2} - L_{C4} refer to places where occur cracks, chipping, delamination outside and inside the crack and delamination of the coating is proceeded.

DLC coatings for the first crack and accompanying removal of the coating from the substrate was observed with a load of 4,46 N (Figure 4a). The introduction of the chromium layer as an interlayer contributed to the strengthening of the shell and increase the critical load to almost 80 N (Figure 4d). The coating, even at very high pressures of contact, is still firmly fastened to the substrate (Figures 4b and 4c).

CONCLUSIONS

Observations have shown compliance of the construction of the structure of the coatings with the one that was assumed during the processes for their preparation, and glow discharge spectroscopy GDOES gave information about the chemical elemental composition of the coatings was verified and their thickness measured in the tests on the microscope SEM.

DLC coatings obtained by PECVD on CoCrMo alloy elements have a greater hardness than the material of the substrate.

The working test results indicate that the tested type of DLC coating a - C: H have good adhesion to the substrate.

The obtained parameters of mechanical properties of the applied coatings should guarantee good performance.

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Note: The responsible translator for English language is Przemysław Szczepańczyk, Kielce, Poland