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DLC coatings on martensitic steel used for surgical instruments

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ABSTRACT

Purpose: The main aim of the work was evaluation of corrosion resistance, mechanical properties and topography of DLC coating formed by means of RF PACVD and magnetron method on the X39Cr13 martensitic steel used for surgical instruments.

Design/methodology/approach: The corrosion resistance tests of samples were carried out by means of potentiodynamic method registering anodic polarization curves in physiological Tyrode's solution at temperature 37±1^{o}C. Registering of anodic polarization curves was conducted at the pace of potential change equal to 1 mV/s. As the reference electrode saturated calomel electrode (SCE) was, the auxiliary electrode was platinium electrode. Mechanical properties were evaluated on the basis of the Vickers hardness test. The test was realized with the use of the CSEM NHT hardness tester. The topography observations of the surface with the DLC coating by means of the confocal laser scanning microscope LEXT OLS3000 Olympus were carried out. The following surface treatments were applied: barrel finishing, chemical passivation and deposition of DLC coating by means of the RF PACVD and magnetron method.

Findings: The investigations revealed diverse corrosion resistance and mechanical properties of the individual samples. The highest corrosion resistance was observed for the barrel finished, passivated and DLC coated samples obtained by means of magnetron method.

Research limitations/implications: Usefulness of DLC coatings will be evaluated in further research in in vitro conditions. Furthermore wear a measurements of surgical drills coated with DLC layer (RF PACVD and magnetron method) will be carried out.

Originality/value: The improvement of physico-chemical properties of surgical instruments made of martensitic steel will undoubtedly increase the safety of surgical procedures and reduce costs.

Keywords: Corrosion; Mechanical properties; Wear resistance; Tool materials

PROPERTIES

1. Introduction

The advantageous effect of carbon coatings on biotolerance of implants made of metallic biomaterials has recently been reported [1-8]. The interdisciplinary research headed by J. Marciniak and S. Mitura was also dedicated to these problems. The research was focused on problems of developing the passive-carbon coating on surfaces of implants made of the AISI 316L steel [9-14]. The electrolytic polishing and passivation technology was developed, ensuring the increase of the Cr concentration in the implant's surface about 6% and of Mo about 2% compared to the substrate. The carbon coating is deposited in the RF PACVD process onto the implant surface prepared in that way. On the basis of on the in vitro tests it was found that this coating guarantees to the implants a very good resistance to pitting, stress, and crevice corrosion.

Moreover, tests carried out in the experimental animals' tissues demonstrated their good biotolerance [10-12]. On the basis of the previous, positive results obtained for the passive-carbon layer deposited on Cr-Ni-Mo steel, the authors worked out the depositon conditions of DLC coating on surgicall tools, in order to increase their usage properties.

2. Methods

The research was conducted on the samples of size 20x15x1 mm made of X39Cr13 martensitic steel. The way of surface preparation of the samples contained mechanical processing (barrel finishing), chemical passivation and DLC coating using RF PACVD and magnetron method in conditions created by the authors [ ]. The following surface treatments were applied:

- barrel finishing and DLC coating by means of RF PACVD method,
- barrel finishing, passivation and DLC coating by means of RF PACVD method,
- barrel finishing and DLC coating by means of magnetron method
- barrel finishing, passivation and DLC coating by means of magnetron method.

The corrosion resistance tests of samples were carried out by means of potentiodynamic method (registering anodic polarization curves). The measurements were conducted in physiological Tyrode's solution at temperature 37±1°C. The research was realized by means of the system used for electrochemical research VoltaLab 2.1. As the reference electrode saturated calomel electrode (SCE) was, the auxiliary electrode was platinium electrode. The measurement of corrosion potential was realized in time of 120 min. Registering of anodic polarization curves was conducted at the pace of potential change equal to 1 mV/s.

Mechanical properties of the samples were evaluated on the basis of the Vickers hardness test. The test was realized with the use of CSEM NHT hardness tester. The measurements were realized for maximum penetration h_{max} = 50 nm.

Furthermore, the topography observations of the surface with the DLC coating by means of the confocal laser scanning microscope LEXT OLS3000 Olympus were carried out.

3. Results

The research of the samples made of X39Cr13 steel showed diverse corrosion resistance in simulated body fluids - Table 1, Figs. 1 and 2.

On the basis of the measurement it was stated that the corrosion potential value of samples with DLC coating (RF PACVD method) was in the range E_{corr} = -390 ÷ -330 mV. The recorded anodic polarization curves indicate the existence of passive range. The values of the breakdown potential equal to E_{np} = -60 ÷ +9 mV. When the current density reached 1 mA/cm^2 the direction of anodic polarization of samples was changed. The change of the polarization direction caused further increase of the current density. That course of the anodic polarization curves indicate the development of pitting corrosion. The values of the repassivation potential equal to E_{cp} = -343 ÷ -343 mV – Table 1, Fig. 1. On the basis of the Tafel curves the additional parameters describing the corrosion resistance of the samples were determined: polarization resistance R_p, corrosion current density i_{corr} and corrosion rate. Their values were in the range R_p, = 17,9 ÷ 22,5 kΩcm, i_{corr} = 0,50 ÷ 0,80 μA/cm^2 and W = 5,2 ÷ 7,8 μm/year respectively – Table 1.

No significant differences of corrosion potential of the barrel finished, passivated and DLC coated samples with respect to the unpassivated samples were observed (RF PACVD method). The values of corrosion potential were in the range E_{corr} = -369 ÷ -356 mV. The values of breakdown potential for these samples showed higher values (with respect to the unpassivated samples) equal to E_{np} = +118 ÷ +144 mV. The change of direction of polarization caused increase of the current density – Table 1, Fig. 1. The other parameters were in the range: R_p, = 21,9 ÷ 24,4 kΩcm, i_{corr} = 0,31 ÷ 0,51μA/cm^2 and W = 3,7 ÷ 5,1 μm/year.

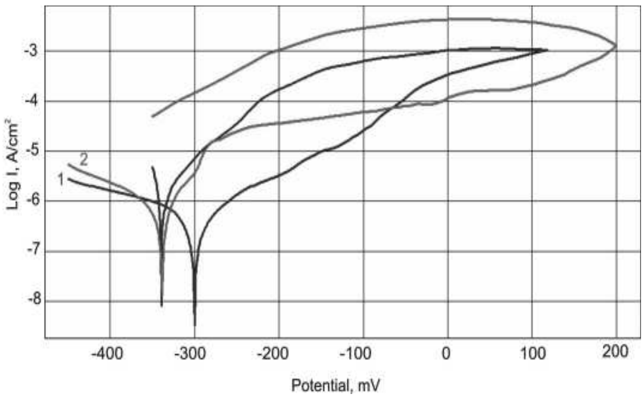

Fig. 1. Anodic polarization curves of samples made of X39Cr13 steel: 1 – barrel finishing with DLC coating (RF PACVD method), 2 – barrel finishing, passivation with DLC coating (RF PACVD method)

Further research was carried out on the samples coated with carbon by means of the magnetron method. The tests of the barrel finished and DLC coated samples showed that values of the corrosion potential were in the range E_{corr} = -374 ÷ -352 mV– Table 1. The values of breakdown potential equal to E_{np} = -112 ÷ +10 mV – Table 1. The change of anodic polarization direction caused the increase of current density up to 1,20 mA/cm^2. That course of polarization curve indicate the pitting corrosion – Fig.2.

The measurements of potential change in a function of time for barrel finished, passivated and DLC coated samples by means of magnetron method showed, than values of corrosion potential were in the range E_{corr} = -383 ÷ -308 mV – Table 1. The recorded anodic polarization curves of samples indicate the existence a very wide passive range. The values of the breakdown potential for these samples equal to E_{np} = +1050 ÷ +1124 mV and were among all research variants of samples the highest– Table 1, Fig. 2. The other parameters were in the range R_p, = 30,9 ÷ 38,2 kΩcm, i_{corr} = 0,18 ÷ 0,30 μA/cm^2 and W = 2,2 ÷ 3,5 μm/year.

As the comparative tests the corrosion resistance only of the barrel finished samples were carried out. Results of the corrosion tests are presented in Table 1.

Table 1.
Results of corrosion resistance investigations

The way of surface preparation	Corrosion potential E_{corr}, mV	Breakdown potential E_b, mV	Corrosion current density i_{corr}, μA/cm^2	Polarization resistance R_p, kΩcm	Corrosion rate W, μm/year
barrel finishing	-385 ÷ -349	-65 ÷ -29	0,53 ÷ 0,63	12,5 ÷ 19,6	6,1 ÷ 6,7
barrel finishing +DLC coating (RF PACVD method)	-390 ÷ -330	-60 ÷ +9	0,50 ÷ 0,80	17,9 ÷ 22,5	5,2 ÷ 7,8
barrel finishing +passivation +DLC coating (RF PACVD method)	-369 ÷ -356	+118 ÷ +144	0,31 ÷ 0,51	21,9 ÷ 24,4	3,7 ÷ 5,1
barrel finishing +DLC coating (magnetron method)	-374 ÷ -352	-112 ÷ +10	0,29 ÷ 0,47	25,9 ÷ 31,9	3,4 ÷ 5,5
barrel finishing +passivation +DLC coating (magnetron method)	-383 ÷ -308	+1050 ÷ +1124	0,18 ÷ 0,30	30,9 ÷ 38,2	2,2 ÷ 3,5

The measurements showed that the DLC coating deposited by means of RF PACVD on previously barrel finished and passivated samples are characterized by the highest mechanical properties. Hardness of the DLC coatings was in the range 711 ÷ 939 HV. The values of Young's modulus were in the range E = 138 ÷ 179 GPa – Table 2. Hardness of samples barrel finished was in the range 562 ÷ 576 HV.

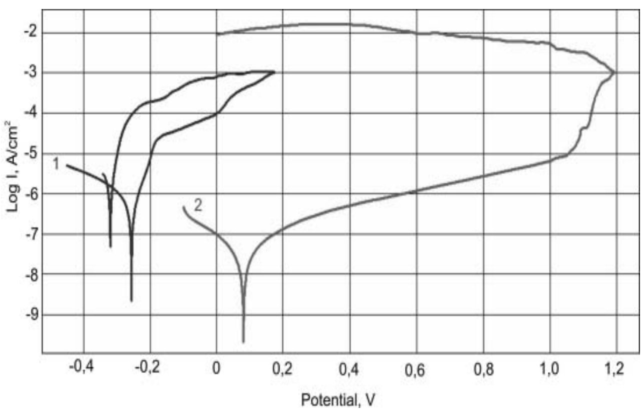

Fig. 2. Anodic polarization curves of samples made of X39Cr13 steel: 1 – barrel finishing with DLC coating (magnetron method), 2 – barrel finishing, passivation with DLC coating (magnetron method)

Observations of the samples by means of confocal laser scanning microscope showed similar topography of the carbons coatings deposited by means of the two methods. For the barrel finished, passivated samples with DLC coating (RF PACVD) the surface roughness was in the range R_a = 0,06 ÷ 0,09 μm. The highest values of leveling depth were in the range R_p = 0,07 ÷ 0,16 μm. For the DLC coated samples obtained by means of magnetron method the roughness parameters were in the range R_a = 0,06 ÷ 0,11 μm and R_p = 0,08 ÷ 0,18 μm.

Table 2.
Results of mechanical properties investigations

The way of surface preparation	Hardness HV, MPa	Young's modulus E, GPa
barrel finishing + passivation + DLC coating (rf PACVD)	711 ÷ 939	138 ÷ 179
barrel finishing + passivation + DLC coating (magnetron)	607 ÷ 649	99 ÷ 104

4. Conclusions

Recently the authors have made an attempt to work out the conditions of forming layers on surfaces of instruments used in bone surgery. The aim of the work is an improvement of corrosion resistance, mechanical properties and wear resistance of tools. The improvement of service life of surgical tools will undoubtedly increase the safety of surgical procedures and reduce costs.

The research revealed that the suggested variants of surface treatment influence the corrosion resistance of the X39Cr13 steel intended for surgical drills. The highest corrosion resistance was observed for the barrel finished, passivated and DLC coated samples obtained by means of the magnetron method – Table 1, Fig. 2 The increase of the corrosion resistance is mainly caused by the applied chemical treatment (the passivation process). The DLC coating deposited by means of the RF PACVD method was characterized by the best mechanical properties. Hardness of these coating was in the range 711 ÷ 939 HV – Table 2. Reasearch on the surface topography by means of the confocal laser scanning microscope showed, that suggest surface treatment (barrel finishing, passivation and DLC coating by means of RF PACVD and magnetron method) ensure smooth surface of samples.

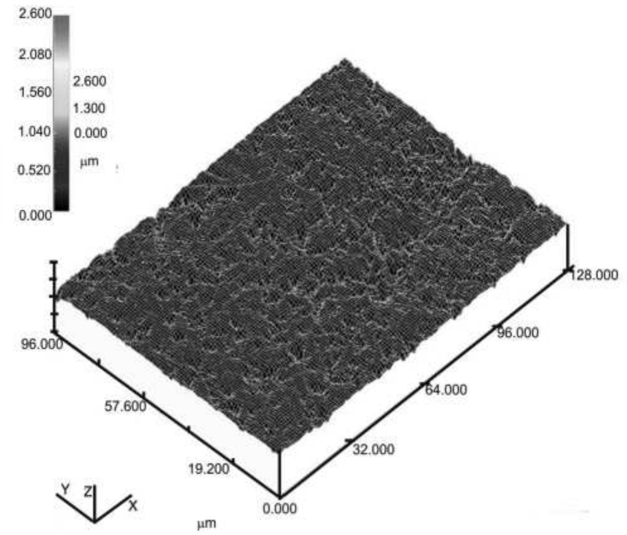

Fig. 3. Topography of the surface barrel finishing, passivation with DLC coating of samples by means of the magnetron methods

The work presents preliminary results of appling DLC coating in surgical tools. Further research will be focused on durability of surgical drills DLC coated by means of RF PACVD and magnetron method in in vitro conditions.

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