Diamond-like carbon coatings for biomedical applications

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Abstract

The results of experimental studies on amorphous diamond carbon layers obtained by a new method of r.f. dense plasma chemical vapour deposition onto orthopaedic pins and screws are presented. Research on this subject which has been carried out over many years allows us to draw optimistic conclusions concerning the biomedical applications of diamond-like carbon (DLC). In particular, preliminary medical research on a new DLC-steel substrate system developed in 1992, which has just been concluded, is extremely promising.

1. Introduction

Biomaterials, materials used in the human body, should satisfy the following requirements [1]: non-iniation of reactions in the tissues surrounding an implant, biotolerance (sometimes biocompatibility), and a specific set of mechanical properties. It seems that carbon is one of the best biomaterials. It has been used for a long time as a construction element, first of all in the form of highly oriented pyrolytic graphite (HOPG) and composites containing carbon fibres [1]. A relatively new material is diamond-like carbon (DLC) [2]. Attempts to use it in implants have appeared to be successful [3, 4]. Of particular importance are those properties of carbon in the form of DLC which allow us to use this material as a barrier against metalosis [5].

2. Experimental details and results

Investigations of optimization of the production of superhard carbon layers, known as diamond-like carbon (DLC), resulted in the development of a new technology, called a dense r.f. methane plasma method [6]. The idea of this method, described in detail elsewhere, is to excite a plasma in methane or other hydrocarbons in an r.f. electric field at a relatively high gas pressure of about 100–300 Pa. Figure 1 shows the r.f. plasma region with orthopaedic screws placed in it. The significant role of electrons in this process should also be emphasized [7].

Amorphous diamond coatings produced by the dense r.f. CH₄ plasma method on steel AISI-316L used in surgery were investigated to determine their suitability as biomaterials satisfying the requirements stated in the introduction.

2.1. Tribological investigations

Results of tribological investigations are presented in Table 1. Differences between the properties of DLC layers obtained by the dense plasma r.f. method [6] and that applied previously by the authors [2] should be stressed. The hardness of DLC layers obtained previously was about 3000 VHN, their critical load did not

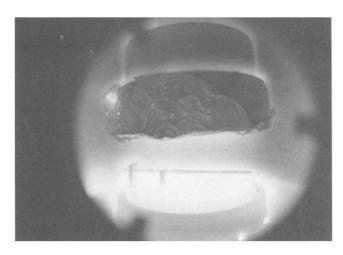


Fig. 1. The r.f. plasma region with orthopaedic screw placed in it.

TABLE 1. Results of tribological investigations of AISI 316L steel implants coated with ultrafine grained diamond

Test	Result
Microhardness	8400 ± 1000 VHN
Adhesion-scratch test	$50 \pm 5 \text{ N}$
(critical load)	
Adhesion energy of the	4 Jm ⁻²
layer-substrate interface	
Grindability	> 100 times
(number of screwings without visible changes)	

exceed 2 N, and the adhesion energy of the layer-substrate interface was $0.09 \text{ J m}^{-2} \lceil 8 \rceil$.

The grindability test was performed by screwing the orthopaedic coated screws into animal bones. Figure 2 presents an optical micrograph of such a screw with a coating after 100 subsequent screwings into bone.



Fig. 2. The orthopedic screw with DLC coating after 100 subsequent screwings into bone.

2.2. Tests of corrosion resistance

Tests of the corrosion resistance of steel AISI 316L coated with superfine diamond layer were carried out in Tyrod solution according to ASTM STP 684 standard. The Tyrod solution simulates fluids present in the human body. Samples stored in the Tyrod fluid at temperatures of 277 K, 310 K, 337 K and 355 K did not reveal any traces of surface degradation. Scanning electron microscopy observations were taken after 1 week, 1 month and 3 months. The investigations are continuing [5].

2.3. Breakdown test

The breakdown voltage was measured in the Tyrod fluid. The values of this potential V = 1300 show that the DLC layers can provide efficient protection against galvanic processes in the human organism.

2.4. Investigation of biotolerance

Investigations of the biotolerance were carried out using stainless steel AISI 316L passivated and coated with a carbon layer and implanted in laboratory animals.

The aim of the research was to reveal toxic and allergic reactions occurring after placing the implants in animal tissue. The research was carried out according to ASTM 981-86 standard. In the investigations guinea pigs were chosen as animals which react to toxicologic and allergic factors similarly to humans.

Implants in the form of disks 5.0 mm in diameter and 1.0 mm thick of weight $0.750 \pm 0.010 \text{ g}$ were investigated. The disks were polished electrolytically and fitted [2]. They were coated with a carbon layer [6]. The disks were implanted into chest muscles, subcutaneously between shoulder blades, and into the tibia bone. The investigations were carried out for 52 weeks.

On the basis of the research it was found that in subcutaneous tissue, muscles and bones thin connective tissue capsules built from fibrocytes and collagen fibres were formed. No phagocytic reaction was observed in the wall of the capsule, and no products of corrosion were found. Internal organs (liver, kidneys, spleen) did not show any pathomorphological changes. The observations were taken using an optical microscope (see Figs. 3–5).

The histopathological investigation showed a very good biotolerance of the implants coated with the DLC layers. The coating is efficient protection against corrosion and metalosis.

3. Conclusions

The coating made from AISI 316L steel consisting of ultrafine grained diamond bound to the substrate by carbides seems to be a very good biomaterial.

The present stage of the investigations allows us to

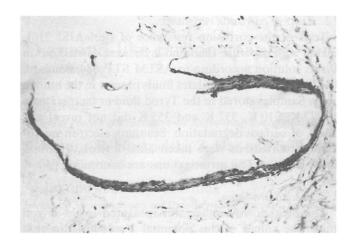


Fig. 3. Implant into subcutaneous tissue, showing a thin connective tissue capsule. Optical micrograph, original magnification $160 \times$.

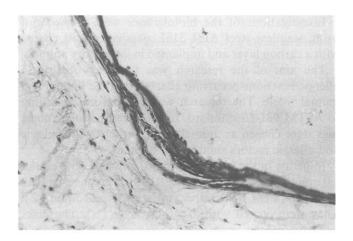


Fig. 4. Implant into muscles, showing thin connective tissue capsule adhering to loose connective tissue. Optical micrograph, original magnification $160 \times$.

draw optimistic conclusions concerning the potential applicability of carbon coatings obtained by the dense r.f. CH₄ plasma method.

Naturally, the specificity of the investigations and medical tests requires long-term observations after which the applicability of coatings can be determined, but the material can already be estimated favourably.

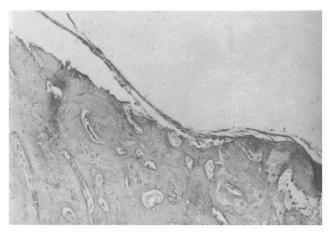


Fig. 5. Implant into bones, showing thin connective tissue capsule adhering to the bone. Optical micrograph, original magnification $100 \times$.

Acknowledgments

This work was partly sponsored by project 3.3602.91.02 of the Polish State Committee for Scientific Research.

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