258. Research of microstructure and steadfastness of bioceramic coatings formed by magnetronic method

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Abstract. Paper presents the research of microstructure and steadfastness of polyethylene medical implants coated with ceramic and other materials. After 2500 loading cycles it was noticed that the strongest implant ceramic coatings are Co+TiO_2 and Co-Cr-Mo. However the roughness of Co-Cr-Mo surface is far larger than Co+TiO_2. The largest and the highest collections of granules are formed in the Co-Cr-Mo coating, and the medium – in Co+TiO_2 coating. Consequently, more durable implant coating is Co+TiO_2.

Keywords: bioceramic coatings, medical implant, microstructure

1. Introduction

Materials used in the production of endoprotheses must be distinguished for inertness, good biocompatibility with the tissues of organism, perfect mechanical and tribological features. Such materials as titanium, Co-Cr-Mo, polyethylene, TiO_2 [1] are frequently used in the production of endoprotheses. However, it was noticed that artificial polyethylene joints marked for good compatibility with the tissues of organism are nondurable and more suitable for elderly patients. One of the promising groups of materials for the production of artificial joints is ceramic oxide coatings [2,3,4].

The previous research [5,6] has shown that when the elasticity module of the material of a hip endoprosthesis socket is small and the stiffness of femoral stem is large, the pressure at contact surfaces is reduced considerably, lubrication conditions are improved and the durability of the hip's endoprosthesis is increased. Research [7] has shown that when coating the polyethylene surface of the socket with durable coatings TiO_2 and Co-Cr-Mo, on one side the contact surface is hardened, but on the other side it is coated on a resilient base and allows one to increase the contact surface. Such a solution helps to reduce stresses and to considerably improve wear-and-tear resistance. The above combination can be applied in fields where it is crucial to extend the service life of frictional parts and units featured for their restricted lubrication, so as to undertake damping of vibrations and impacts.

Therefore, it is important to analyze the microstructure of polyethylene coated with ceramics and metals as well as adhesive features with polyethylene.

2. Methods

Chirulen (ISO5834-2, *POLY Hi Solidur MediTECH*), special design (for medical purposes) high molecular weight polyethylene has been used for investigation purposes.

Coatings were obtained by using a magnetronic method. In the pilot studies polyethylene implants were coated Co+TiO_2, Co-Cr-Mo and Zr. When coating polyethylene with ceramics and metals using a magnetronic method the process takes place under low temperatures (less than 70°C). Affected by the electric field the electrons are expedited and ionize the process gas (e.g. Ar, O_2) under the cover. During the discharge under the effect of interperpendicular electric and magnetic fields an annular plasma forms over the cathode (target). Under the effect of electric and magnetic fields electrons start moving in cycloids. With the help of magnetic field the plasma of electrons – ions is accumulated over the target surface, therefore, when ions and electrons strike the target for a number of times they knock out atoms or molecules out of the target.

The surface of samples has been measured by the scanning probe microscope Dimension 3100 produced by company Digital Instruments.

Coatings have been tested for their steadfastness and durability. For this purpose a special rubbing device generating laoding cycles has been designed and manufactured. The device is intended for operation in the German made FP-10 tensioning equipment [7]. The device is used for simulation of real movements taking place in the human hip joints. During experiment a number of cycles have been repeated at first 1500 times and later 2500 times.

The roughness of coatings surfaces was analyzed with digital profiler DEKTAK 6M. Measures have been taken to record surface roughness height R_z and the mean value R_a of profile deviations prior to coating, following coating procedure and upon completion of 2,500 cycles in order to evaluate durability and wear-out rate of surfaces coated with different coating materials.

3. Results

Photos of polyethylene surface coated with $Co+TiO_2$ until tests is shown in Fig. 1.

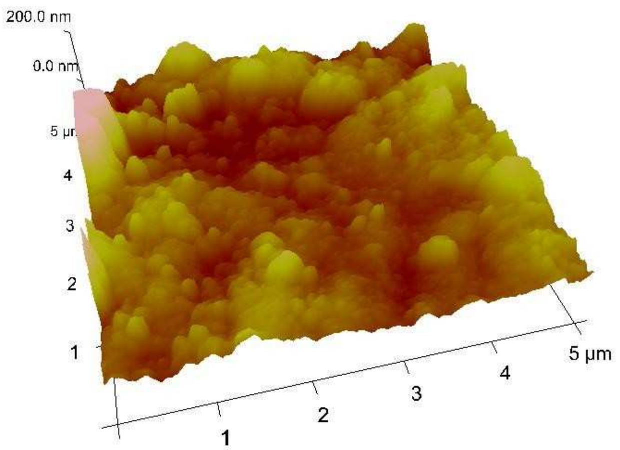

Fig. 1. Photos of polyethylene surface coated with $Co+TiO_2$ until tests

After coating by magnetronic method the following was estimated: granule size of $Co+TiO_2$ coatings ranges from 140 to 416 nm and the surface is rough; whereas clearly seen sharp form roughness of the surfaces with Co-Cr-Mo coatings (Fig. 2), where granule size ranges from 100 to 366 nm and they are oval; granule size of Zr coatings (Fig. 3) ranges from 75 to 300 nm and surface is least grainy and rough.

After 2500 loading cycles et was noticed that on the surface of implants coated with $Co+TiO_2$, 2-3 white spots (from 160 to 500 nm size) have appeared on the research area (5×5μm), i.e. on attrition process as surface coated with ceramic (Fig. 4); 4 white spots have appeared on surfaces with Co-Cr-Mo coatings (Fig. 5); and on surfaces coated with Zr – about 10-13 white spots (Fig. 6).

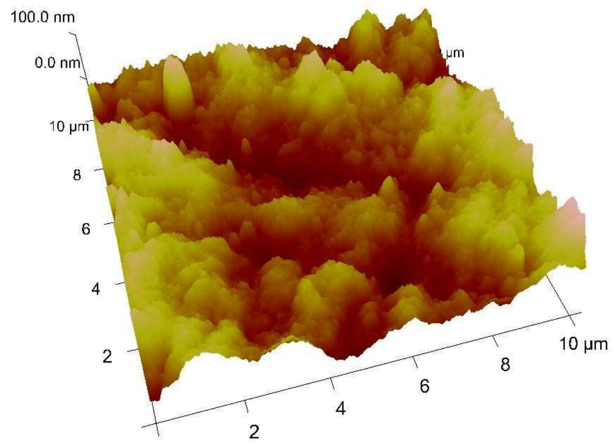

Fig. 2. Photos of polyethylene surface coated with Co-Cr-Mo until tests

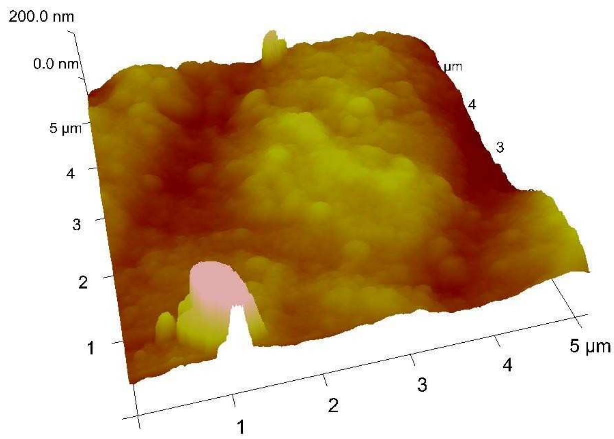

Fig. 3. Photos of polyethylene surface coated with Zr until tests

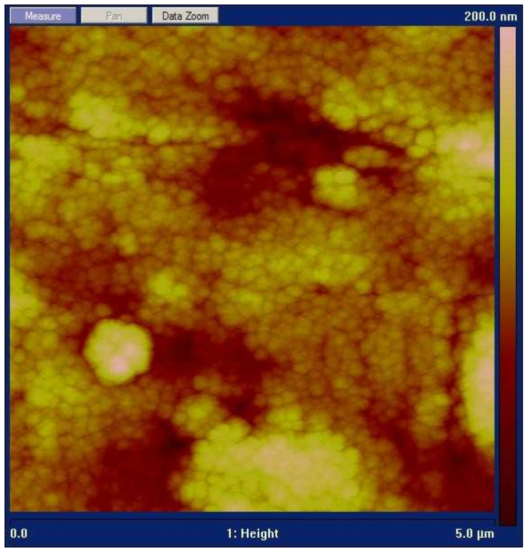

Fig. 4. Photos of polyethylene surface coated with $Co+TiO_2$ after 2500 loading cycles

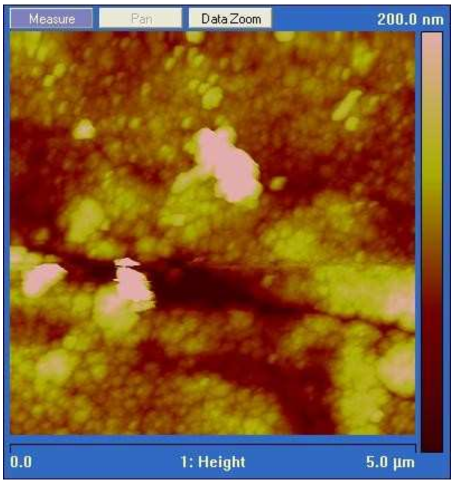

Fig. 5. Photos of polyethylene surface coated with Co-Cr-Mo after 2500 loading cycles

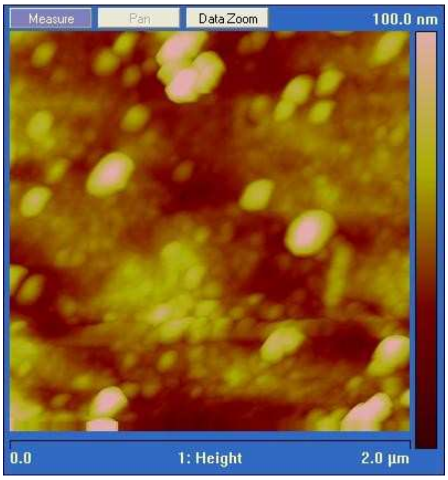

Fig. 6. Photos of polyethylene surface coated with Zr after 2500 loading cycles

The largest collections of granules are formed in Co-Cr-Mo coating (about 7 µm length, 2 µm width and 14 µm high). Also large collections of granules (4.5×4.5µm) are formed in Zr coating but the surface has minimal roughness. The coating Co+TiO_2 surface has middle roughness and the collections of granules exceed 0,8×0,6µm.

Average values of granules size are presented in the table 1.

Table 1. Average values of sizes of coatings granules, nm

Sample No.	Co+TiO_2		Co-Cr-Mo		Zr	
	min	max	min	max	min	max
1	140	416	100	366	75	300
2	125	400	100	350	80	310
3	130	410	95	345	85	300
Average arithmetic value	131,6	408,6	98,3	353,6	80	303,3

Fig. 7 shows the dependence of roughness parameter R_z of coatings on the number of load cycles.

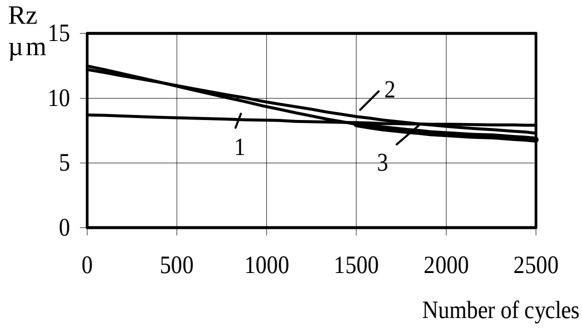

Fig. 7. The dependence of roughness parameter R_z of coatings on the number of load cycles: 1 - Co+TiO_2, 2 - Co-Cr-Mo; 3 - Zr

The dependence of roughness parameter R_a on the number of load cycles is presented in Fig. 8.

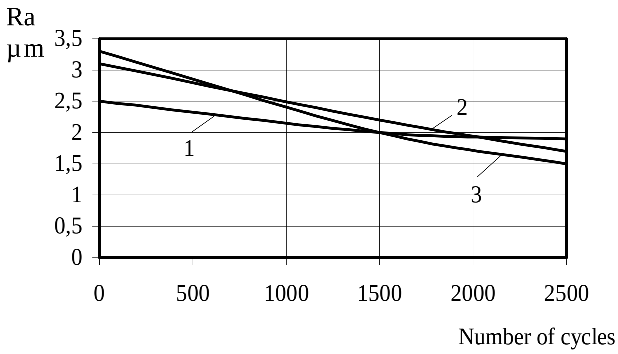

Fig. 8. The dependence of roughness parameter R_a of coatings on the number of load cycles: 1 - Co+TiO_2, 2 - Co-Cr-Mo; 3 – Zr

Therefore, the strongest implant ceramic coatings are Co+TiO_2 and Co-Cr-Mo. However the roughness of Co-Cr-Mo surface is far larger than Co+TiO_2. Consequently, less worn implant coating is Co+TiO_2.

Conclusions

The research results have proved the following:

1. The most rough and with the largest collections of granules is the surface of Co-Cr-Mo coating, medium roughness and minimal granule collections has the surface of Co+TiO_2 coating, and finally the least roughness with largest collections of granules is Zr coating;
2. The largest granules noticed in Co+TiO_2 coating (from 131 to 408 nm), the smallest ones – in Zr coating (from 80 to 303 nm);
3. Co+TiO_2 coating is the steadiest and the most durable. And coatings of Co-Cr-Mo and Zr have very similar durability.

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