HYDROXYAPATITE COATINGS DEPOSITED FROM ALCOHOL SUSPENSIONS BY ELECTROPHORETIC DEPOSITION ON TITANIUM SUBSTRATE

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Non-calcined and calcined, synthetic powders of HAp were deposited by electrophoretic (EPD) process from alcohol medium (methanol, ethanol, propanol and butanol) on the Ti-plate cathode within one or several cycles. The quality and the rate of the coating deposition were evaluated with respect to applied direct voltage (5, 10 and 20 V) and the concentration of suspension. The optimal conditions bring the medium of methanol and a direct voltage of 10 V. The coating thickness attained within several seconds tens of micrometers. The deposition time 150-180 s brings the layer thickness 150-200 μm. The growth rate of coating is affected mostly by the current density and duration of process, i.e. by charge density. The suspension concentration, conductivity of suspension and applied voltage are the main determining factors of the current density.

INTRODUCTION

Metal implants coated by thin ceramics layer based on apatite or Ca-P glassceramic are used in stomatology and orthopedic surgery. Ceramic bioactive coatings consist of mainly hydroxyapatite (HAp; $Ca_{10}(PO_4)_6(OH)_2$), which is a basic component of bones. The role of apatite layer is to inhibit negative interaction of living tissue with metal implants, which are implemented on the defective place and improve its biointegration, i.e. speeds up the healing process, penetration of bone tissue.

The quality of synthetic HAp, prepared by simple precipitation of Ca^{2+} ions from the solutions containing PO_4^{3-}, depends on concentration and stoichiometry of Ca^{2+} and PO_4^{3-}, pH, temperature and duration of stabilization of precipitate in solution. These factors are crucial for HAp-production otherwise complex precipitate can be obtained that contains additional phases e.g. deficient, amorphous calcium phosphate, calcium hydrogenphosphate ($CaHPO_4$), octacalcium phosphate ($Ca_8H_2(PO_4)_6 \cdot 5H_2O$) a.o. [1-3].

Synthetic HAp prepared by precipitation from inorganic solutions is calcined at temperatures of 400-1050°C. Higher temperatures and non-controlled atmosphere with low water partial press as well as impurities stimulate the HAp decomposition into simple phosphates [4]. HAp powders can be applied on the metal substrates by plasma sprayed, magnetron sputtering, electrophoretic deposition, enameling techniques and other methods [5].

Practice shows that for good adhesion and overgrowth of implant with bone a hydroxyapatite layer of only a few micrometers represent a sufficient thickness. Therefore the research is oriented on electrochemical and electrophysical coating processes (magnetron sputtering, electrophoresis, etc.), dipping implants into various solutions, for example HAp-sols or into body fluids simulating solutions (biomimetic treatment). In order to elucidate the nucleation and growth of apatite crystals the interactions of metal substrates with Ca^{2+} and PO_4^{3-} ions are studied [6-10].

Electrophoretic deposition (EPD) and electrolytic deposition (ELD) are simple techniques for substrate coating of complex shapes by thin layers of ceramic materials [11]. Layers of a thickness of a few micrometers as far as several tenths of micrometers can be deposited from HAp suspension depending on process conditions. The conditions of ELD/EPD mentioned by various authors [11-13] are very different. The quality of coating is determined by properties of solutions/suspensions like pH, conductivity, size of particles, kinds and amounts of ions, etc. as well as electrode materials (anode-Pt, C and other, cathode-Ti, Ti - alloys, resistant steel et.al.), electrode voltage, and so on. In case of water solutions and suspensions electrode voltage up to 2 V is recommended [11]. Much higher voltages (20-100 V, even 800 V) were used for deposition of HAp precipitate from alcohol- based suspensions [12-14].

Final hardening and densification of coatings is achieved by sintering at temperatures of 900-1050 °C. Sintering at these temperatures results in interaction of

substrate with HAp, too. On the other hand, such conditions can induce partial decomposition of HAp and formation of cracks. The cracks can develop not only during HAp-layer sintering but also during the heating and cooling periods. The reason for the formation of cracks is the increasing strain between the substrate and layer due to different thermal expansion of materials, the thermal expansion coefficient of Ti being $8.0\text{-}12.2 \times 10^{-6}$ K^{-1} and that of HAp 14.0×10^{-6} K^{-1} [15].

The present work describes the deposition of HAp powder onto the titanium plate by electrophoretic process from the alcohol-based suspensions (methanol, ethanol, propanol and butanol). The effects of dispersing media and of the kind of HAp powders on the growth rate and quality of coating of Ti-substrate were studied.

EXPERIMENTAL

Hydroxyapatite powder was prepared by precipitation of Ca^{2+} ions from supersaturated $Ca(OH)_2$ solution using 0.3 mol/l H_3PO_4. Under permanent stirring the acid was poured into the cell until Ca : P molar ratio of 1.67 was attained, which corresponds to stoichiometry of $Ca_{10}(PO_4)_6(OH)_2$. After stabilization of suspension for 24 hours, the pH was adjusted to 8 adding NH_4OH. Ageing of precipitate was carried out under controlled pH = 8-8.5 at the temperature of 37°C for 5 days. The precipitate was filtered, washed with distilled water and dried. A part of the precipitate was calcined in air at temperatures of 400 or 900 °C for 2 hours, assigned as HAp400 or HAp900. The rest of the sample, assigned as HApN, was used as prepared.

The powders were characterized by X-ray diffractometers with Ni-filtered CuK radiation (XRD) (MIKROMETA II), scanning electron microscopy and energy dispersive spectroscopy (SEM/EDS) (Hitachi S4700).

The Ti plates of size 10×12×0.5 mm were cut from rolled sheet, polished using SiC paper (# 800 and # 1200) ultrasonically cleaned in water bath, rinsed three times in distilled water and dried at 40°C for 2 hours.

Deposition of HAp onto Ti cathode was carried out in Cr-Ni-steel cell serving as anode. Its radius was 16 mm with a spherical bottom. The anode surface area was 1600 mm^2. The electrodes were connected to DC power source.

The coating was deposited onto Ti plate from suspensions containing from 1 up to 5 wt.% of HAp powders. The suspensions were prepared 24 hours before deposition. The powders were dispersed in pure alcohols: methanol (p.a. 99.5 %), ethanol (p.a. 99.5 %), propanol (p.a. 99.7 %), butanol (95 %), Each suspension has been ultrasonically dispersed for 10 min. 24 hours thereafter the suspensions were characterized in terms of their pH and conductivity (Table 1). Conductivity was measured using ORION 115+ conductometer (Thermo-electron corporation).

The experiments were carried out at voltage of 5, 10 and 20 V at temperatures of 25 and 40°C. The suspensions have been ultrasonically agitated before EPD process for 10 minutes while deposition was carried out without stirring. Coating was deposited either in one cycle or in several consecutive cycles. One cycle comprised deposition (15 or 30 s), sample drying at 35-40°C and weighing. The morphology of coatings was observed using light microscope (Neophot 32, Carl-Zeiss Jena) and the thickness of the layers was determined from depth of focus measurements.

Table 1. Basic properties of alcohols and suspensions.

Dispersion medium		Methanol p.a. (CH_3OH)	Ethanol p.a. (C_2H_5OH)	Propanol (i) p.a. (C_3H_7OH)	Butanol (C_4H_9OH)
Abbreviate		M	E	P	B
Density ρ (kg/m³)		791	789	785	812
Dynamic viscosity η 10³ (Pas)		0.59	1.19	2.20	3.95
pH of alcohol	0% HAp	7.4	7.3	6.1	6.0
and suspension	1% HApN	7.1	6.0	7.0	6.3
before process	1% HAp400	5.8	4.9	6.0	5.2
of EPD	1% HAp900	7.5	6.9	6.2	6.0
Conductivity	0% HAp	2.5	0.3	0.175	1.02
of alcohol	1% HApN	14.8	N/A	N/A	N/A
and suspension	1% HAp400	108	29.3	4.9	5.5
σ (µS)	1% HAp900	19.8	N/A	N/A	N/A

RESULTS AND DISCUSSION

The broad lines of non-calcined HAp in the X-ray diffraction pattern shown in Figure 1 suggest that the sample has a defective structure and/or particles are very small. The calcination process carried out at 400°C for 2 hours improves crystallinity of HAp. The diffraction lines of HAp400 become sharper and the diffraction pattern is nearly identical with the commercial HAp supplied by Merck. The higher calcination temperature of 900°C is causing partial decomposition of HAp to TCP ($Ca_3(PO_4)_2$). These phases were identified in the XRD patterns of HAp900 sample.

The size of particles was measured using laser particle analyzer (Sympatec Helos) equipped with CLCELL unit in water suspension of 0.1 mol/l $Na_4P_2O_7$. The maximum size of particles of HAp400 was up to 40 μm. The distribution of particles size is 35 % under 2 μm, 45 % in the range of 2-10 μm and 10 % in the range of 15-40 μm.

The SEM micrographs of HAp400 coating on Ti-substrate deposited by EPD process show that the dominant fraction is represented by particles of about 50-100 nm (Figure 2 a-c). The particles on SEM images are by 2 orders of magnitude smaller as the results of the particle size analyzers.

According to [12] the smallest particles reached the highest electrophoretic velocity, and therefore these were the preferential deposited. It is probably the reason of the discrepancy between the results of the particle size analyzer and SEM images. Another possibility need to be taken into account which is the sedimentation of particles over 10 μm that is especially significant for longer-time deposition over 30 s.

The EDS analysis showed that the Ca:P molar ratio of the deposed powder (2% suspension of HAp400-methanol) was 1.6. It corresponds to molar stoichiometry of 9.6 Ca : 6 P. The coating was compact and had
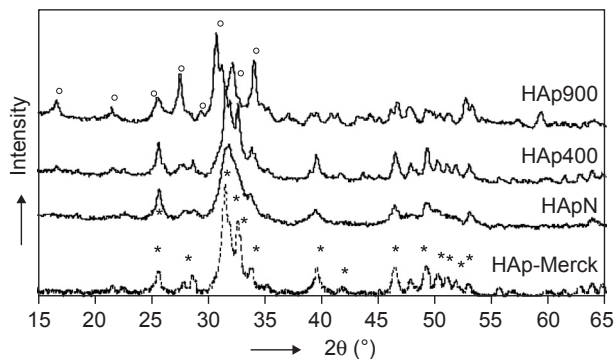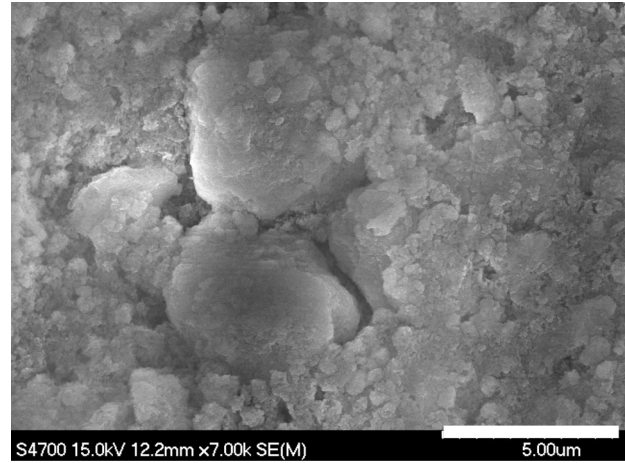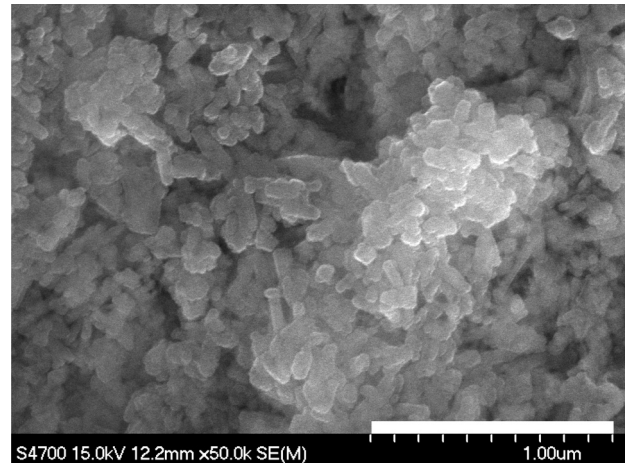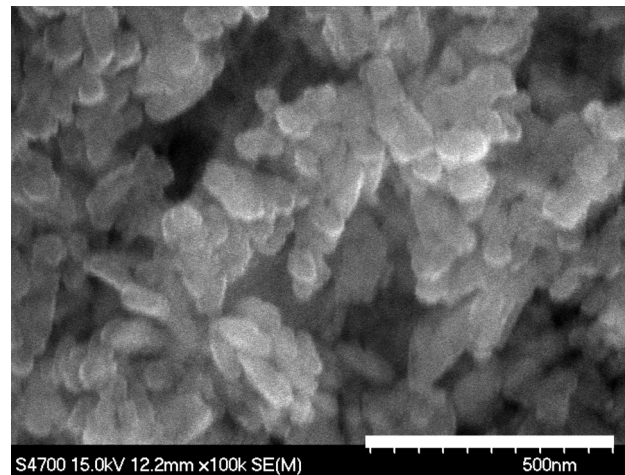

a)

b)

c)

Figure 1. X-ray diffraction patterns of HAp - f. Merck; HApN-synthetic; HAp400 - calcined at 400°C for 2 hours; HAp900 - - calcined at 900°C for 2 hours. (* - characteristic lines of HAp; o - characteristic lines of TCP).

Figure 2. SEM micrographs of HAp coatings on Ti-surface prepared via EPD from HAp400 suspension (2 %) in methanol: a) 5 μm; b) 1 μm; c) 500 nm. EPD parameters: U = 10 V; τ = 15 s.

a thickness of ca 35-40 μm. The thicknesses of the layers were determined on the several samples, on various areas from depth of focus measurements between surface of substrate and surface of coating using light microscope.

The differences in these results of granulometry suggest that the primary particles coagulate in water solutions. The dispersion of particles depends on the value of the surface tension, surface charge of particles, charge of HAp micelles and their electrokinetic potential. The above parameters are influenced mostly by the following factors: pH, dissociation and concentration of electrolytes in solution. As seen in Table 1, pH and conductivity values are different for each suspension which results in different stability of suspensions. In all cases the sedimentation of particles was very slow and several days took than solutions were semitransparence. The existence of agglomerates in suspensions affected the quality of coatings obtained from ethanol and butanol - containing suspensions.

The values of conductivity of suspensions (Table 1) are in correlation with data of current density. For the suspension of methanol and ethanol that show the highest values of current density, the highest changes of pH were observed comparing to the pure alcohols. It could

point to increase degree of dissociation in suspension.

The current density under identical conditions - electrode voltage 10 V, temperature of 25°C, surface SA of cathode and concentration of HAp400 suspensions - varied in several orders of magnitude depending on the type of suspension (Table 2). In addition, its value was unsteady during deposition.

The current density was affected by the deposition method. When the deposition was carried out in several consecutive cycles (cycle 30 s), the current density in the beginning of each subsequent cycle was lower than at the end of the previous one. Later on it raised and became steady (Fig.3). The stabilization of current density is probably related to the drying of coating and to its increasing thickness. Similar relationships as shown in Fig. 3 were observed also in case of ethanol suspensions.

The lower current density was attained the less dense coating was obtained. Therefore the coatings deposited from propanol and butanol were very susceptible to how quickly they were withdrawn from suspension. It often happened that at a rate of withdrawal of cca 2 mm/s the deposited layers were damaged or destroyed just due to the friction between the layers and the suspensions.

Table 2. Comparison of growth of HAp400-coating on the Ti substrate in alcohols as dispersion medium. Condition of EPD: concentration = 1 wt.% suspension of HAp400, U = 10 V, t = 25°C, $S_{A(Ti)}$ = 200 mm², $S_{anodeCr-Ni-steel}$ = 1 600 mm².

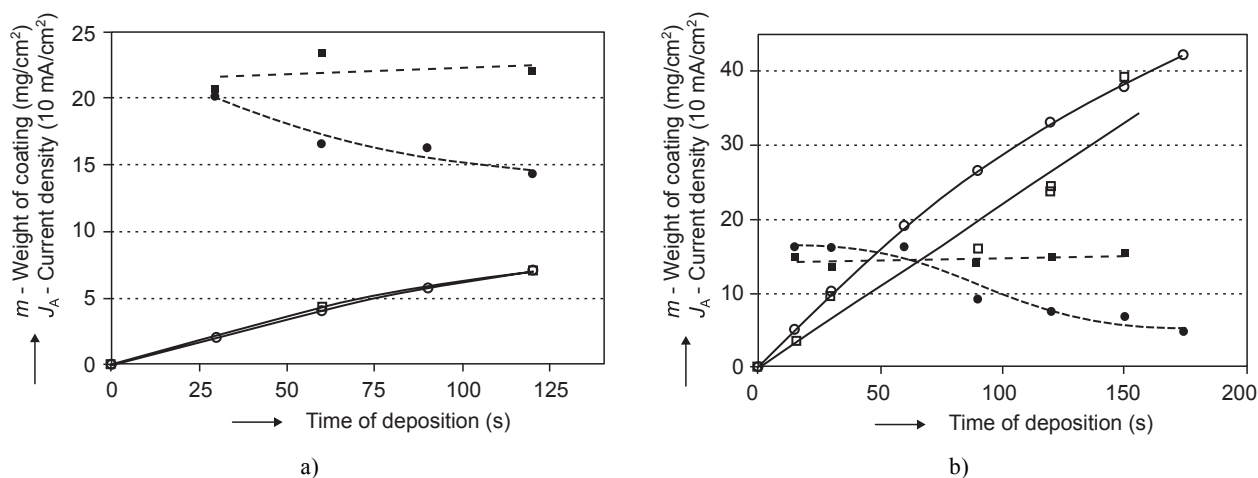| Medium	Current density J_A (mA/cm²)	Time (s)	Weight of coating (mg/cm²)	Average coating thickness (μm)
M	1.5 – 2.1	15		
E	0.6 – 0.8	90 – 120	1.5 – 2	20 – 25
P	0.025 – 0.035	900 – 1 100		
B	0.06 – 0.08	800 – 900		

Figure 3. Dependence of current density and weight of HAp-deposite as function of deposition time a) suspension of 1 wt.% HAp400 in methanol; b) suspension of 5 wt.% HAp400 in methanol. Conditions of EPD process: U = 10 V, t = 25°C. □○ - weight of coating, ●■ - current density (J_A); ○● - deposition in several cycles; □■ - deposition in one cycle.

The fluctuation of current density affected the weight of the deposited powder. Consequently the results following here below are described as dependence of the amount of HAp powder on current density J_A multiplied by time τ. The charge Q is defined as electric current (I) × time (τ).

Regardless of the kind of alcohol used in suspension, more or less identical amount of HAp400 powders was deposited on Ti plate by equal electrical charges (Figure 4). In other words it means that the amount of deposited powders is proportional to the charge passed between the electrodes. As the current density in methanol suspension is by orders higher than in other alcohols, the coating process is significantly faster in this medium (Table 2).

The current that passes between the electrodes for a certain time is affected by several factors like purity of alcohols, pH value, concentration and kind of powders dispersed in suspension, micelle charge, concentration of free ions, etc. The observed differences can be explained as the consequence of instability of the above factors.

The following results show how the deposition rate of coatings on Ti substrate and their quality are affected by electrode voltage, temperature and concentration and kind of HAp powder - see Figures 5-7.
The current density increases in direct proportion to the increasing electrode voltage and amount of the deposited HAp is a linear function of the charge density (Figure 5).

The change of temperature between 25-40°C had no measurable effect neither on current density nor on the amount of the deposited HAp coating. On the other hand, higher temperatures caused excessive evaporation

of alcohols changing the concentration of the suspensions. As it can be seen in Figures 3 a, b and 6, it is concentration of suspension which governs the rate of coating formation. The current density increases with increasing concentration of suspension. Oppositely, current density gradually decreases with the number of cycles/increasing deposit thickness. In case of EPD from the methanol suspension with 5 % HAp400 the current density at the end of the last deposition cycle (after 180 s when the layer was about 150 μm thick) decreased to about 2/3 of its starting value. The decrease of current density is due to increasing thickness of deposit that increases resistivity. Also in cause of cyclic deposition the soaking of dried coating after its immersion into suspension affects value of current density.
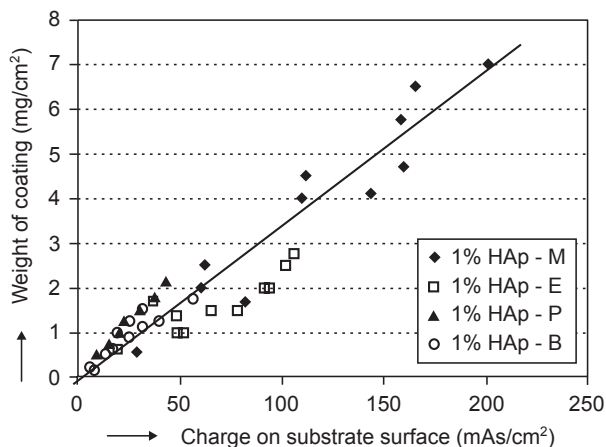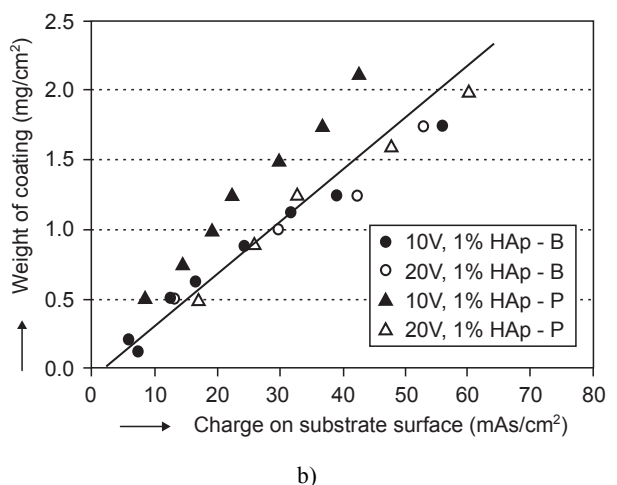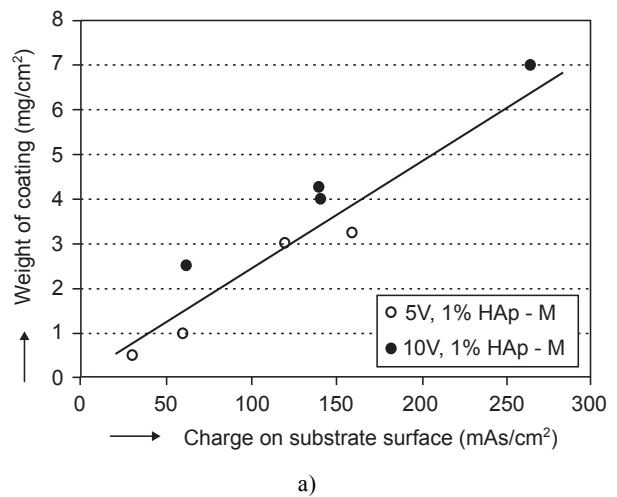

a)

Figure 4. Comparison of various dispersion medium and the relative amount of deposited powders at constant conditions during the EPD process: concentration of suspensions = 1 wt.% HAp400 in alcohol, U = 10 V, t = 25°C. M - methanol; E - ethanol; P - propanol; B - butanol.

b)

Figure 5. Dependence of the amount of the deposited HAp400 as a function of voltage in a) methanol (U = 5 and 10 V); b) propanol and butanol (U = 10 and 20 V) in one cycle.

Homogenity, density and adhesion of coatings to substrates increased with prolonged time of deposition as well as with increasing concentration of suspensions in methanol. Coatings of weight of over 30 mg cm^{-2} were obtained by EPD from 5 % suspension of HAp400 in one cycle (process duration was 150 s) while coatings of weight of over 40 mg cm^{-2} were obtained in 6 cycles with total process duration of 180 s (6 × 30 s). The coatings were dense, homogenous and over 150 μm thick. The coatings prepared in several cycles were more dense than those prepared in one cycle.

The same importance as the suspension concentration has the kind of powders, their phase composition and granulometry (Figure 7). Calcination of HApN

changed the composition of powders which further affected pH value of suspensions, their conductivity and stability (Table 1).

Even if the amount of the deposited HAp from the 1% suspensions of HApN and HAp900 in dependence on charge density is higher in comparison with the weight of HAp400 (Figure 7), deposition of HApN and HAp900 was less effective because the formation of the coatings was slower. In 30-60 s only very thin coatings of HApN were obtained and all look alike. Although their surface appeared to be smooth the cracks developed during drying even at room temperature (Figure 8). Cracks appeared rarely in coatings prepared from HAp400 and HAp900.
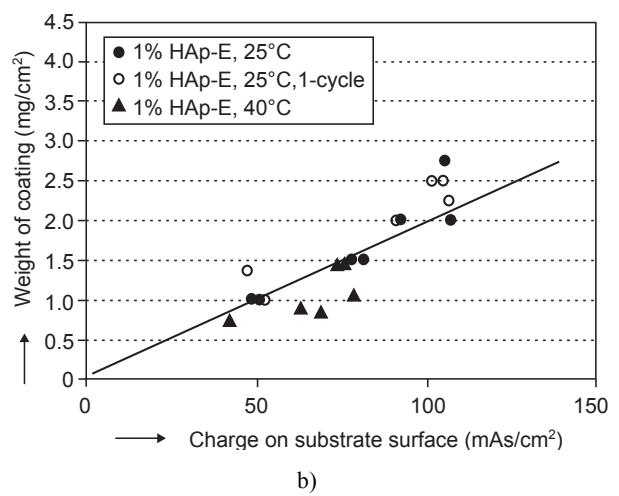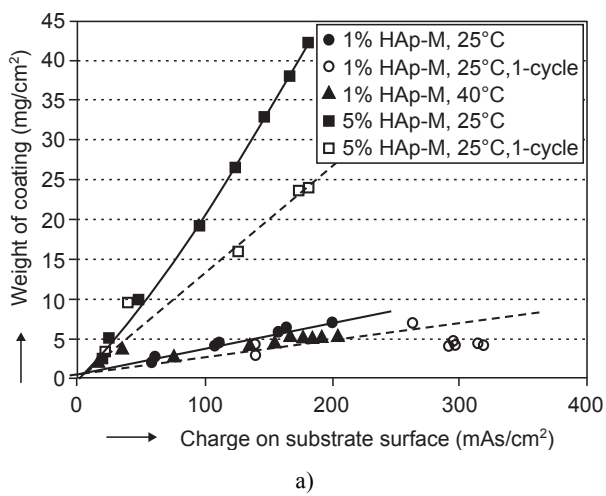

Figure 6. Dependence of the amount of the deposited HAp400 as a function of suspension concentration and temperature. a) 1 wt.% and 5 % HAp400 in methanol; b) 1 wt.% HAp400 in ethanol. Conditions of EPD U = 10 V; ●■▲ - deposition in several cycles (4-6 cycles); □○ - deposition in one cycle.
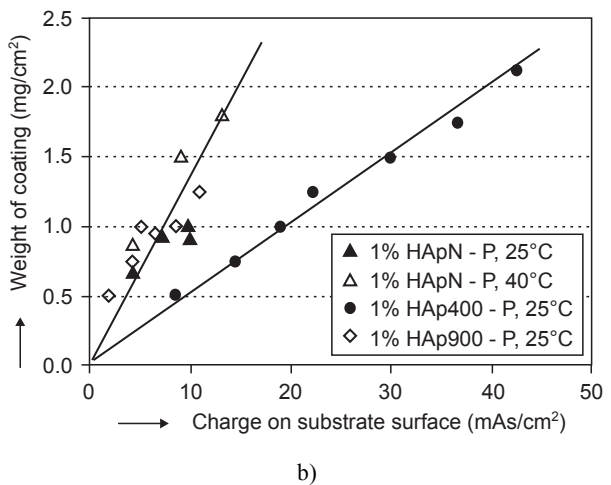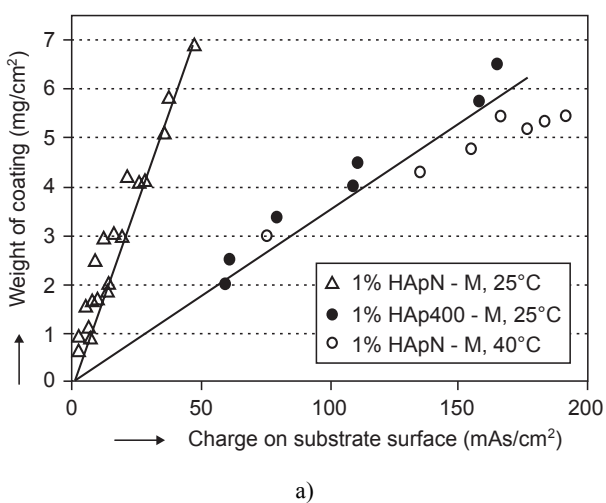

Figure 7. Dependence of the amount of the deposited HAp as a function of kind of powder 1 wt. % a) in methanol and b) propanol. Conditions of EPD: U = 10 V, HApN - non-calcined, HAp400 - calcined at 400°C for 2 hours, HAp900 calcined at 900°C for 2 hours, ●■▲ - deposition in several cycles (4-7 cycles); □○ - deposition in one cycle.
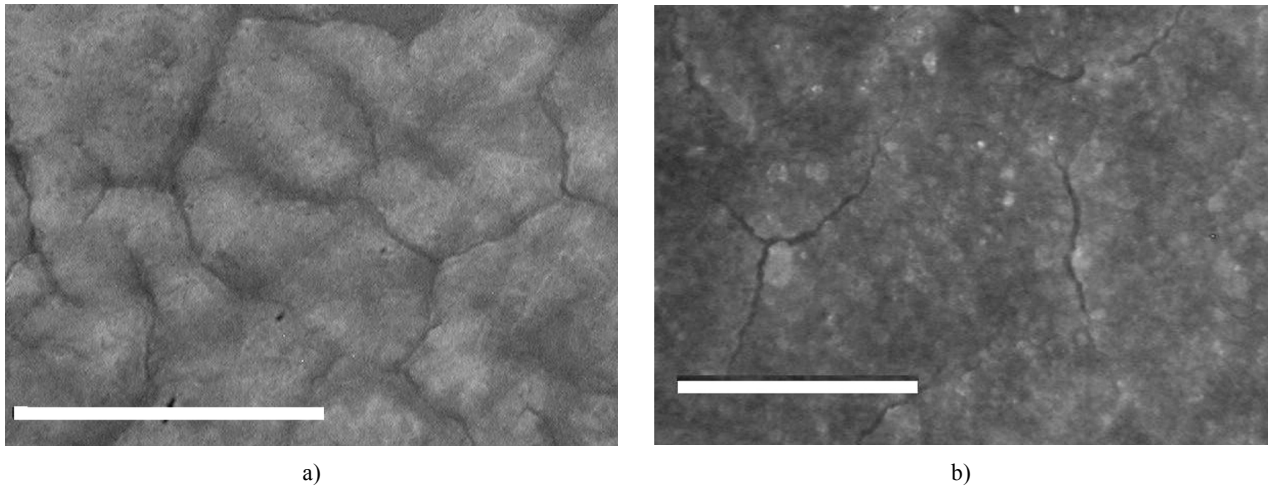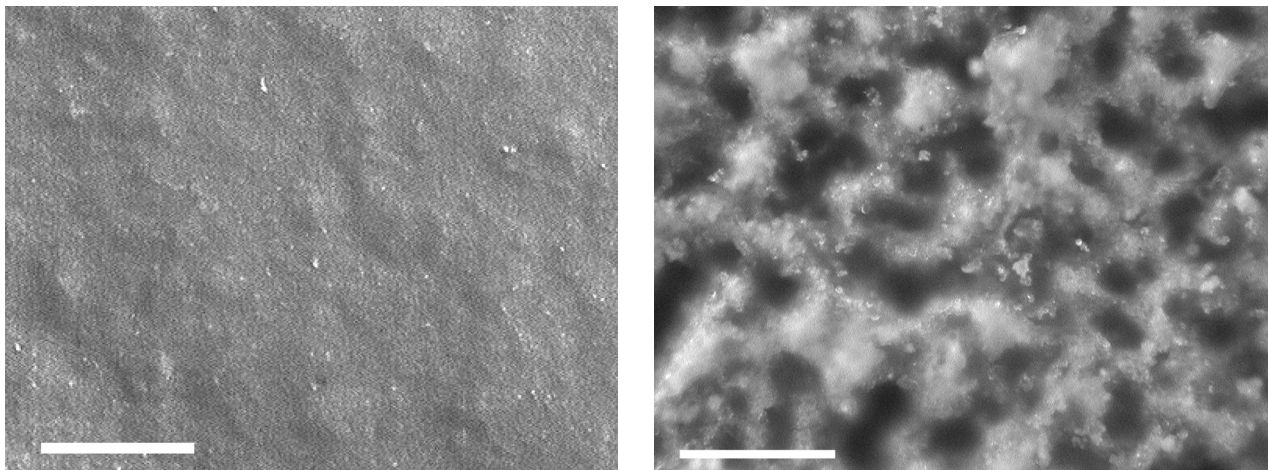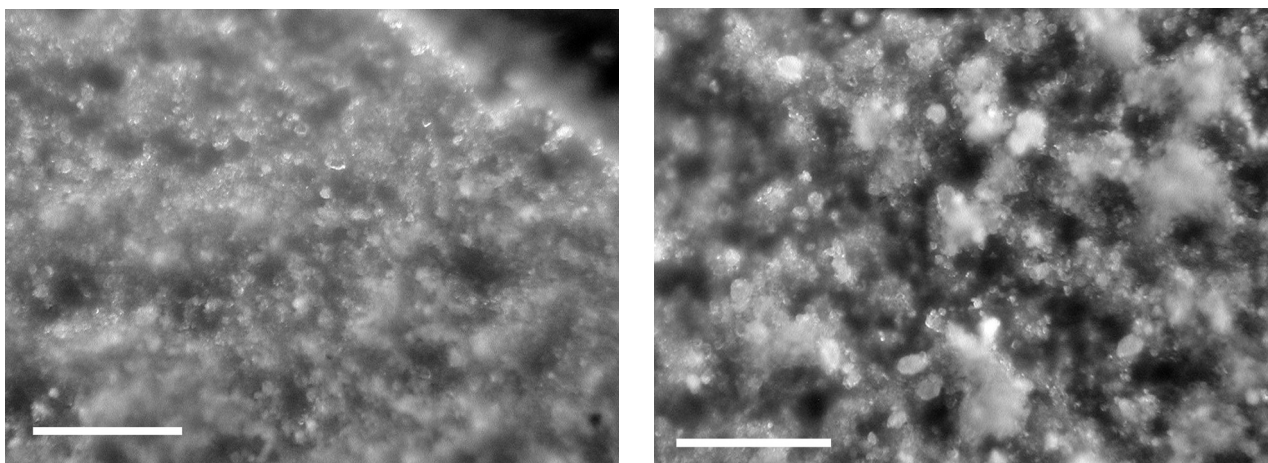
a)

b)

Figure 8. Optical micrographs of coatings on Ti substrate deposited by EPD from 1 wt.% HApN suspension in methanol. Scale - 100 µm.

a) Coating deposited from methanol,time of exposition = 90 s, thickness 40-45 µm

b) Coating deposited from ethanol, time of exposition = 210 s, thickness 30-40 µm

c) Coating deposited from propanol, time of exposition = 900 s, thickness 25-30 µm

d) Coating deposited from butanol, time of exposition = 600 s, thickness 20-25 µm

Figure 9. Optical micrographs of coatings on Ti substrate deposited by EPD method from 1 % HAp400 suspension in methanol. Scale - 100 µm.

Coatings obtained from suspensions of HAp400 in methanol, ethanol, propanol and butanol and prepared under identical conditions are different as shown in Figure 9. Homogenous and smooth coatings were obtained by deposition of HAp400 from methanol, rougher coatings were deposited from propanol suspensions. Very coarse, non-uniform coatings were obtained from ethanol and butanol based suspensions. The coatings where the time of deposition was short appear to be gritty what confirms that HAp was not properly dispersed in these alcohols.

All coatings are porous (Figures 2, 9). With increasing thickness, especially in case of HAp400 in methanol, coating appeared to be homogeneous and improved adhesion to the surface of Ti-substrate. They are easily removable from the smooth polished Ti substrate by stronger scratching or repeated abrasion. In case of coarse surface of Ti substrate HAp coating is anchored with better adhesion.

Hardening of coatings can only be attained by sintering process at high temperatures of 800-1000°C under conditions that supress the thermal decomposition of HAp. At the same time together with sintering of HAp particles chemical interaction between coating and metal substrate takes also place.

CONCLUSION

It was confirmed that the amount of deposited HAp from alcohol suspensions by EPD is a function of passed charge (current passed × time). The current density is proportional to the voltage set. In addition, its value significantly depends on the dispersion medium and of the kind of dispersed powder. The coatings are of better quality the deposition is faster when the fine powder is well dispersed in the medium and the particles surface is able to acquire higher charge. The coating is compact and smooth but always is porous because the HAp particles are bound by electrostatic and Van der Waals forces.

The process was most effective from methanol suspension which showed the best conductivity and highest current density.

The deposition process of HAp400-powder (calcined at 400°C) was faster from methanol and ethanol suspension than from propanol or butanol suspension. The HAp400-coating is deposed, in dependence on the concentration of suspension and electrode voltage, in a few seconds. Its thickness can be up to several tenths of micrometers.

Deposition of HApN- (non-calcined) and HAp900-powder (calcined at 900°C) was slow and coating of the same thickness as for HAp400-powder could be obtained in 10-30 min.

The voltage of 5 -20 V was, in case of methanol suspension, sufficient for EPD due to the high current density that was, in other works [12-14], attained at much higher voltages.

Acknowledgement

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ELEKTROFORETICKÁ DEPOZÍCIA HAP Z PROSTREDIA
ALKOHOLOVÝCH SUSPENZIÍ NA Ti-SUBSTRÁT

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Elektroforetická depozícia (EPD) je jednou z možných metód, ktoré dovoľujú jednoduchým spôsobom naniesť na tvarovo zložitý implantát len niekoľko mikrometrov tenkú vrstvu HAp. Spevnenie a zhutnenie povlaku sa dosahuje až následne spekaním za teplôt okolo 800°C.

Prášky HAp pripravené synteticky (HApN - vysušený precipitát, HAp400 a HAp900 - kalcinovaný pri 400 a 900°C) sa nanášali elektroforézou z disperzného prostredia alkoholov (metanolu, etanolu, propanolu, butanolu) na katódu z titánu. Povlaky HAp sa nanášali na Ti-katódu v jednom cykle a aj opakovane, vo viacerých za sebou nasledujúcich cykloch. Sledoval sa efekt opakovaného nanášania na kvalitu povlaku, vplyv napätia (5, 10, 20 V) a koncentrácie suspenzií na množstvo deponovaného HAp a rýchlosť tvorby povlaku na Ti-substráte. Prúdová hustota suspenzií bola v korelácií s hodnotami nameranej vodivosti. Pre relatívne rýchle vytvorenie kvalitnej vrstvy, niekoľko mikrometrov hrubej, je vhodná prúdová husto-ta 1.5-2 mA/cm².

Deponované množstvo HAp a tým aj hrúbka vrstvy povlaku bola úmerná náboju, t.j. prúdu, ktorý prešiel suspen-ziou za čas. Za niekoľko sekúnd sa zo suspenzie HAp400 v metanolu vytvorila na povrchu substrátu súvislá niekoľko de-siatok µm hrubá vrstva. Za 150-180 sekúnd depozície HAp400 sa získali z koncentrovaných suspenzií (5 hm.%) nadmerne hrubé povlaky, a to až 150-200 µm. Takéto hrubé vrstvy tvorí množstvo prášku okolo 35-40 mg/cm². Výrazne pomalšie sa pokrýval povrch HAp400 v prípade depozície zo suspenzií butanolu a propanolu. Príčinou je o dva rády nižšia prúdová hustota v týchto suspenziách ako v metanole. Povlaky nanesené zo suspenzií s disperzným prostredím etanolu a butanolu neboli ani tak homogénne a hladké.

Nanášanie povlaku po vrstvách, v opakovaných cykloch, dáva možnosť aj v prípade pomalého nanášania pri nízkej hus-tote docieliť kvalitnejšie povlaky.

Množstvo deponovaného HAp z prostredí alkoholov počas EPD a tým aj hrúbka povlaku je tak funkciou koncentrá-cie suspenzie, jednosmerného napätia, predovšetkým ale typu HAp prášku/disperzného média (schopnosti častíc HAp disper-govať v kvapalnom médiu) a prúdovej hustoty.