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Improvements in or Relating to Functional Characteristics of Biocompatible Plasma-sprayed Implant Coating

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It was proved that special treatment of biocompatible powder before plasma spraying consisting in fixing fine particles on major grains facilitates smoothness of porous structure, solidity, coating open porosity, and high level of its surface morphology, as well as enlargement a contacting area of joined surfaces (implant and bone tissue) in the process of the coating additory nanopatterning.

Keywords: Plasma spraying, Biocompatibility, Implant.

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The purpose of the work is improvement of functional characteristics of biocompatible plasma-sprayed implant coating by means of smoothing their porous structure and stabilizing their crystalline texture.

There is a technique of powder quality improvement based on its grain size distribution resulting in elimination of ultrafine and fine fraction consisting in thermomechanical processing (TMP) by means of prolonged annealing followed by a slight grinding [1-4]. During TMP fine and the most active particles of the original powder adhere together and to the larger particles and in the process of the following slight grinding do not detach as independent particles.

Large conglomerates (60-70 μm) which are not active during TMP dissolve to finer particles of the base size in the process of grinding. Thus, previously annealed and grinded powder becomes less polydispersed than the basic one and results in a more solid structure of a porous frame.

In the process of spraying ~ 40 μm fine particles they are warmed up greatly, though possessing low kinetic energy they lose shape very little when bumping against the support and do not adhere to it firmly as a result [5]. When increasing particle size above 40 μm their mass and inertial energy grow up, that is why the particles slow down less and bump against the support with a higher speed. This leads to a measurable deformation, contacting area extending, increase of tension and adhesion between the coating and the support as the final result.

Based on the above the proposed TMP of endosteal implant biocompatible powder consisting in creation of base powder combined particles for spraying by means of fastening (immobilization) of ~ 40 μm fine particles to larger ones leads also to increase of adhesion to titanium-based coating with an intermediate layer of titanium powder.

In the process of plasma spraying in high temperature stream heat-conducting path from a fine particle to a large one preserves a number of fine particles from a complete meltdown. Also when bumping against the support a combined particle splits by separation a fine particle from a large one. Here with it is assumable that a fine particle possessing kinetic energy and tension of a large particle breaks into nanosized particles. Inclusion of biocompatible coating of finer nanoscaled ceramic particles into the structure is reasonable considering its functional characteristics improvement due to increase of active contacting area between the implant and the bone.

The developed technology of Al_2O_3 powder TMP may also be used during preparation of calcium phosphate ceramic biocompatible powders before plasma spraying [1].

TMP of hydroxyapatite polydispersed powders before plasma spraying executed for the purpose of grain distribution and, subsequently, porous structure levelling was conducted in temperature range between 800 and 1000 °C. The powders were annealed in muffle furnace during 3 h and then crumbled in a ceramic mortar box during 20 min. Temperature control was maintained with the help of thermocouple.

The resulting powders were sprayed onto Ti Grade 1 titanium samples with an intermediate layer of titanium powder composed of 250 μm particles.

Metallographical and fractographical analysis of the coating conducted with the usage of biological light microscope (model 10) and upgraded research metallographic microscope (model 8M) and profilograph-profilometer (model 170623) showed that TMP appliance by 800, 900 and 1000 °C results in a smoother structure with larger pores, with their size increasing by anneal temperature rise adequately to surface morphology mutation (Table 1).

With an anneal temperature rise during TMP particle coarsening typical for TMP results in a more developed coating surface morphology and increase of open pore channel size.

Anneal temperature impact tests were also conducted during HA powder TMP concerning the powder structure, crystallinity, phase composition, and binding properties through X-ray diffraction and electron-microscopic analysis. X-ray diffraction and phase powder analyses were executed with the usage of general purpose X-ray diffractometer (model 3). Sprayed sur-

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face morphology and laminate pattern were examined with a MIRA II LMU scanning electron microscope (SEM) released by TESCAN company (Czech Republic). For this purpose a thin aurum conductor layer (10-20 nm) was spread onto the samples applying magnetron sputtering method.

TMP of HA base powders in a temperature range between 800 and 900 °C does not lead to powder phase composition mutation but leads to crystallinity degree change and inner stress decrease. HA in basic state is considerably amorphous and possesses an unstable structure. In the process of anneal in a temperature range between 800 and 1000 °C HA crystallizes and its stresses are measurably lowered. Anneal by 1000 ºC results in a new phase formation – tricalcium phosphate – β – $Ca_3(PO_4)_2$ which is defined by an occurance of a new reflex on HA diffraction pattern.

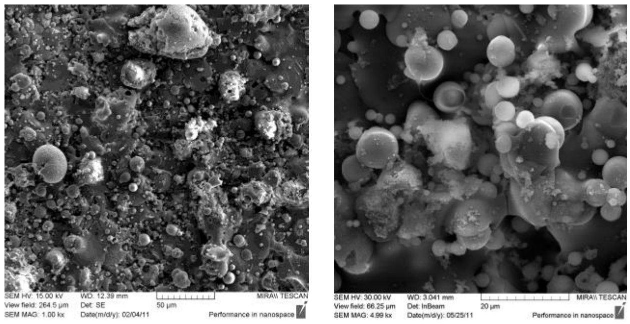
Considerable particle enlargement of biocompatible material with an HA base by TMP temperature 1000 ºC leads to depreciation of sputtering quality caused by formation of large open pore channels (Table 1) and, subsequently, notable uncovered areas of the intermediate layer.

Table 1 – Impact of hydroxyapaxite powder anneal temperature on asperity (*Rz*) and size of open pore channels of a plasma sprayed surface

Sintering temperature TMP, *T*, °C	Parameter $^*Rz$, μm	Open pore channel size, $^{**}d$, μm
No processing	45,8	5-7
800	49,9	7,8-19,6
1000	104	20-39,2

Thus a recommended HA powder TMP anneal interval lies between 800 and 900 °C.

Smoothing hydroxyapatite coating structure after TMP by 800 °C is proved also by electron microscopical structure tests (Fig. 1) [6]. At the Fig. 1 traces on large particles can be seen where finer particles immobilized during TMP separated after bumping against the support. As a result, the structure of hydroxyapatite coating consists not only of 180 nm nanoparticles formed by splitting HA particles in the process of spraying, but also specific finer 40-60 nm nanoflakes (Fig. 1b). Splitting of HA particles that underwent TMP is presumably caused by their more crystallized structure.



a b

Fig. 1 – SEM-images of plasma sprayed hydroxyapatite coating without TMP (a) and after TMP anneal by 800 °C (b)

So, the technologies of biocompatible aluminium oxide and hydroxyapatite powders were developed. The technologies lie in a prolonged anneal followed by grinding which provide elimination of polydispersed powder fine fraction, increase of an average particle size, and consequently an average pore size, and levelling the porous structure. It was established that the developed method of structure smoothing provides increase of strength and service life of biocompatible aluminium oxide coating. It was proved that hydroxyapatite powder TMP results in levelling the porous structure and forming a stable crystalline texture of plasma sprayed coating which facilitates its functional characteristics.

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