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Medical Implants Realized from the Sintered Compacts of Titan-Hydroxyapatite

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Abstract

The functionality of biomaterials must assure a good osteointegration of endobone implants used for the maxilo-facial implants. Materials must present a gradient for porosity and one for composition (presents of a bioactive phase in titan matrix).
The exterior porosity, in the contact zones with the bone tissue, favors the incipient cellular processes (the adhesion and attachment of osteoblastes, proliferation and cellular differentiation and nucleation of mineral bone). The pores must be open and with a specific surface more great. It's preferred the exterior intercommunicating porosity.

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Keywords: sintered compacts; phenomena at the interface; selective laser sintering; hydroxyapatite

1. Matrix of titan and hydroxyapatite particles

It is realized an implant material with bioactive behavior in bone tissue, with a titanium matrix and hydroxyapatite particles. Considering the low level of the ceramic phase properties, its presence is desirable only in the exterior zone of the implant, which is in contact with the bone.[2]

The presence of phosphates of calcium in the contact zone of the implant with bone favors the mineral kernel on bone. The report Ca/P of phosphates mineral bone has an ascending evolution towards 10/6 according to hydroxyapatite. On realized compacts sinterised titan-hydroxyapatite for study of the phenomena at the interface of sinterization. Has been used titanium dehydride powder with granulations greater than 100 microns. Hydroxyapatite powder was obtain by sol-gel technique and then ground. Diffractogram of hydroxyapatite is presented in figure 1.

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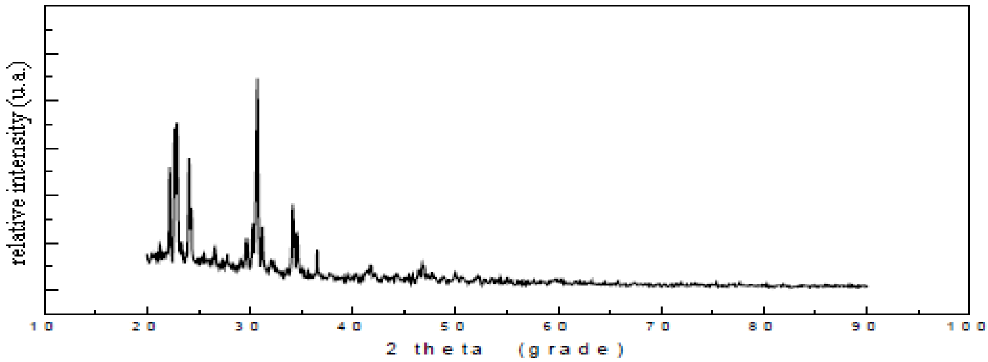

Fig. 1. Diffractogram of hydroxyapatite. [1]

It is realized a compozite powder titan with 5% hydroxyapatite (percentage by mass), which was homogenised 30 minutes. The powder was pressed into the mold to active section of 0,5 cm^2, without the introduction of lubricant. The mold walls and the punches were smeared with oil. Compacts was realized using the pressures of 400, 500, 600 MPa like in figure 2.

Fig. 2. Compozite samples titan-5% hydroxyapatite.[3]

The samples were sintered in vacuum below 10^{-4} after a complex regim multistage assuming maintenance isotherm: 250^0C for 30 minutes in order to remove water and any organic residues; 800^0C for 30 minutes under the transformation temperatures of metallic and ceramic phase matrix; 1160^0C for 60 minutes, the final stage of sintering. The samples were packaged separately in crucibles of zircona, to not be subject to any pollutions treatment.[3]

After cooling of sintering was conducted slowly, in vacuum, up to ambient temperature. The samples were measured before and after sintering. There was a general trend of swelling during sintering process. Interdifusion between components elements of the two initial constituients, demonstrated through careful chemical analysis, resulted in the appearance of Kirkendall effect. The phenomenon is demonstrated by sintering full samples with a compressive strength of 400 MPa, 500 MPa, 600MPa, also on the relative swelling conditions, like in figure 3.

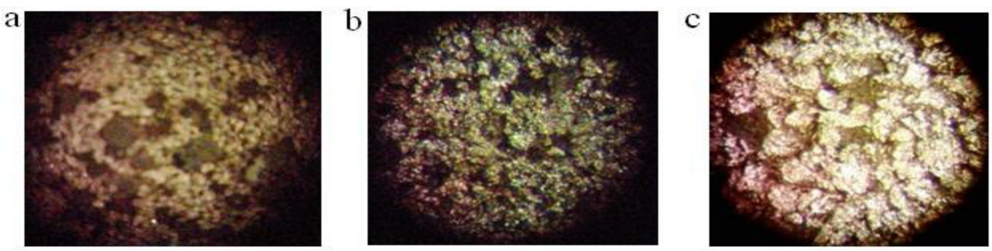

Fig. 3. Front surface composite Ti-5% hydroxyapatite, (a) - 400MPa, (b)-500MPa, (c) -600MPa (x 40).

2. Electron microscopy SEM realized for the composite samples of Ti-5% HA

The samples were sectioned transversely on the halfway of generatrix, sanded and polished with alumina (< 50 microns) and attacked with Kroll's reagent. The samples were studied by SEM microscopy to remark how the interface has been made between calcium phosphates and titan matrix, depending on the pressure of compaction as in figure 4.[4]

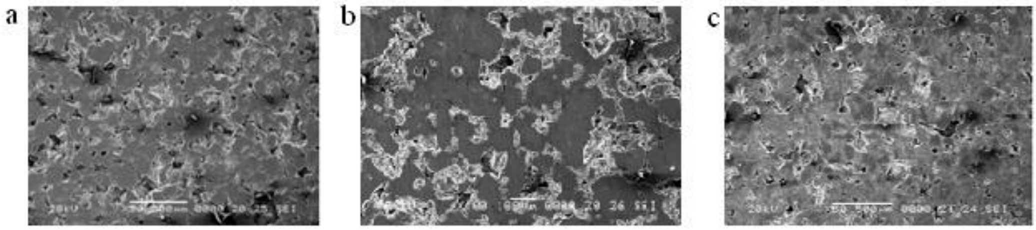

Fig. 4. Electron microscopy SEM realized for the samples Ti-5%HA, (a) - 400MPa, (b)-500MPa, (c) -600MPa (x 50).

During the process of preparation of samples for microscopy, ceramic granules were plucked from the surface to study. On remark the existance of large goals and typical form. Through the analysis of the areas where it was plucked phase ceramics were put in evidence the interface diffusion. The interface diffusion between hydroxyapatite and titan matrix is put in evidence by EDX spectrum like in figure 5.

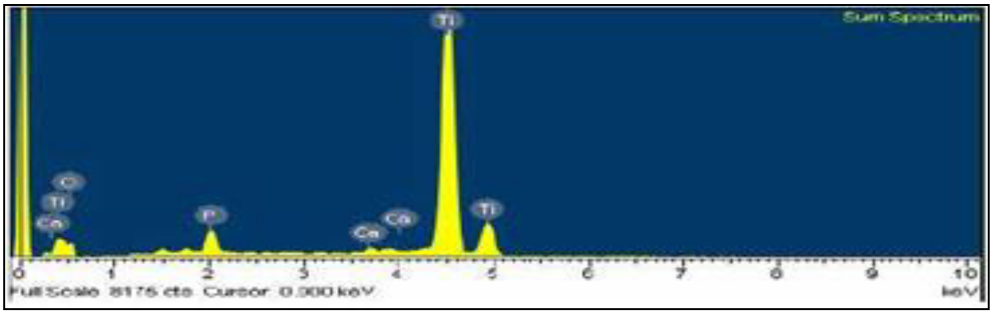

Fig. 5. EDX spectrometry realized for the samples Ti-5%HA, -500MPa.

3. Conclusion

Using the selective laser sintering and the tomographe images, on obtain the 3D virtual models and by sinterisation on produces the real models.

For en implants it is important that only the parts who is in contact with the bone must present a hydroxyapatite phase, like bioactive phase with osteoconductive importance. The presence of bioactive phase permets realizations of new calcium phosphates.

In the future will realize composite implants Ti-HA to fully integrate the implant in human bone tissue. Also, will be performed for this research in vivo and in vitro in order to point out the way how to make the connections between human bone tissue and implant.

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