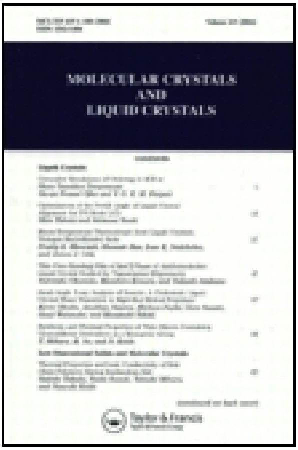

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Dental Implants Coated with Laser Deposited Hydroxyapatite Films - Physical Properties and In-vivo Study

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Version of record first published: 29 Oct 2010

Mol. Cryst. Liq. Cryst., Vol. 374, pp. 599-604

1058-725X/02 $12.00 + .00

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Thin films of hydroxyapatite were created by laser deposition on real tooth prostheses. Overwiev of physical and biomedical tests, including unloaded and loaded osseiontegration of experimental minipigs is presented.

Keywords :

laser deposition; thin film; implants; hydroxyapatite, in-vivo tests

1. INTRODUCTION

For orthopedic and dental prostheses the metal or metal alloy implants are usually used. Several authors claim that the metal implants cause failure due to corrosion [1-5], inflammation [6], tumors [7] or assault of lungs [8]. Because of living organisms generally reject any foreign matter, prostheses appear to be of natural or non- foreign (biocompatible) material. The best known biocompatible material is hydroxyapatite (HA) - $Ca_{10}(PO_4)_6(OH)_2$. But HA has a low tensile strength, is brittle and suffers from fatigue. Problem can be solved by coating a bulk metal material with a thin layer of biocompatible coating. Coated implants connect advantages of good mechanical properties of metal with convenient chemical and biological properties of coating. Various attempts have been made to deposit HA layers onto metals, as plasma spraying, radio frequency sputtering, ion- beam deposition, flame spraying, powder sintering, electrophoretic deposition etc. Problem of mentioned methods is in lack of control over Ca/P ratio, lack of control over ratio of amouphous/crystalline structures, poor adhesion, creation of non- coated area, etc. One of the promising method for coating of implants with HA layers is pulsed laser deposition (PLD).Using PLD the crystalline, amourphous, or partly crystalline HA films can be created, having good adhesion to implant. Also Ca/P ratio can be modified by changing of deposition conditions. In this work we are presenting results of our study on coating of tooth implants with HA, physical and biomedical tests and in vivo study on experimental minipigs in unloaded and loaded regimes.

2. EXPERIMENTAL AND RESULTS

For deposition KrF excimer laser was used. Targets were made from HA powder (MERC). One group of target was prepared by ram pressing and second one by isostatic pressing. At first the films were created on polished flat titanium substrates. Various deposition conditions in connection with film crystalinity, stoichiometry and adhesion were tested. Films were created in vacuum, pure H_2O vapors (from 2 x 10^{-3} mbar to 2 x 10^{-1} mbar), in Ar- H_2O vapor mixture (water vapor flow was varied in region 0.7 sccm – 10 sccm, Ar flow from 9 sccm to 18 sccm), for target-substrate distances 3 cm – 9 cm, and substrate temperatures T_s in range 200 °C – 780 °C. Laser energy density on the HA target was changed in region 3 Jcm^{-2} – 13 Jcm^{-2}. Films crystallinity (by X- ray diffraction), stoichiometry (by Rutherford basckscattering), morphology, adhesion and hardness were studied [9-11]. From results of physical tests the set of suitable deposition conditions was eliminated. For this set the films exhibited nearly the same stoichiometry as target, were partly amorphous and partly crystalline, and also adhesion to substrate was good. On such films we started with biomedical in- vitro tests (T- lymphocytes proliferation, fibroblasts, macrophages changes, metabolic activity) [12-16]. Coming out from results of physical and biological characterization of HA flat films the deposition conditions was chosen to cover real dental implants. Layer on implants were created in mixed Ar- water vapor atmosphere at T_s round 500°C. Energy density of KrF excimer laser beam on HA target was 3 Jcm^{-2}. In such deposition conditions the films exhibited good adhesion to implants and favorable biocompatible properties. Deposition scheme is in Fig.1. Special ceramic holder was screwed into the dental implant. It made possible to rotate and decline implant during deposition and to cover the whole implant surface. The implant used was cylindrical shape, 3.5 mm in diameter and 12 mm long. Substrate was heated with CO_2 laser. The Gaussian beam from the CO_2 laser was expanded using a ZnSe diverging lens and passed through a copper reflective beam homogenizer to obtain rectangular shape of laser beam having cross-section of dental implant. The thickness of PLD created hydroxylapatite layer was about one micrometer.

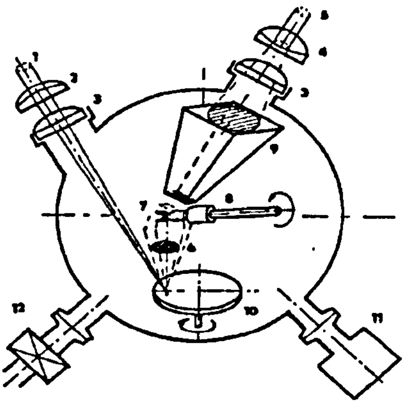

Fig.1. Scheme of laser deposition of HA layers onto real dental prostheses (1- KrF excimer laser beam, 2- focusing lens, 3- windows, 4- ZnSe lens, 5- CO_2 laser beam, 6- plasma plume, 7- dental implant, 8- ceramic holder, 9- homogenizer, 10- target, 11- gauge, 12- Ar/water vapor input).

Mini-pigs were used for in-vivo unloaded and loaded osseointegration investigation. In ***unloaded*** experiment (prostheses without crowns) the HA covered implants, including reference (uncovered titanium implants), were implanted into mandibula of minipigs. Three minipigs, two years old, 40 kg weight each, were used in this investigation. In the first step the premolars from lower jaw of each pig were extracted. The schedule treatments was the following :

- extraction of premolars,
- 10 weeks healing,
- implantation of 18 implants (16 HA coated, 2 non- coated),
- 16 weeks load- free osseointegration; sacrification of minipigs,
- histological evaluation and evaluation in polarized light,
- computer analysis of osseointegration.

Photograph of lower jaw of minipigs with implants is in Fig. 2. Newly formed bone and the percentage of bone- implant contact were determined by standardized radiography, micrography and by transmission and fluorescent microscopy. The level of osseointegration of HA coated implants and control (non coated- titanium) group was different, but the difference was not too high. The area of bone- implant contact was moved from 75.5 % for non coated implants to 77.3% for HA coated [14][17].

For ***loaded*** in- vivo experiment four minipigs were used. 24 cylindrical dental implants were covered with HA layers and 4 non- coated titanium implants were used for reference. The schedule of treatment was firstly similar as for unloaded regime. After healing process the implants were inserted and healed without loading for 16 weeks. Than the metaloceramic crowns were mounted- see Fig.3. Six month after loaded regime the experimental animals were sacrificed. Osseointegration of HA films and control titanium group was similar. The area bone/implant contact moved from 62.5 % for titanium implants to 77.5 % for HA films. The active bone formation was observed in both cases [18-20].

3. CONCLUSION

We have confirmed that PLD is a suitable method to cover substrates with thin layer of biocompatible material, like hydroxyapatite. By varying deposition conditions it is possible to "tailor" layer properties for various medical applications in dependence on hard or soft tissue (crystallinity, Ca/P ratio, film thickness, morphology) and to create films which are exhibiting good adhesion to the substrate can be created. Using special deposition scheme it is possible to cover real tooth prostheses with HA layers. In unloaded and loaded experiments with minipigs very good results were reached.

4. ACKNOWLEDGEMENT

The work was supported by the Internal Grant Agency of Ministry of Health of the Czech Republic No. 4092-3 and by the grant of GA ASCR No. A1010110/01.

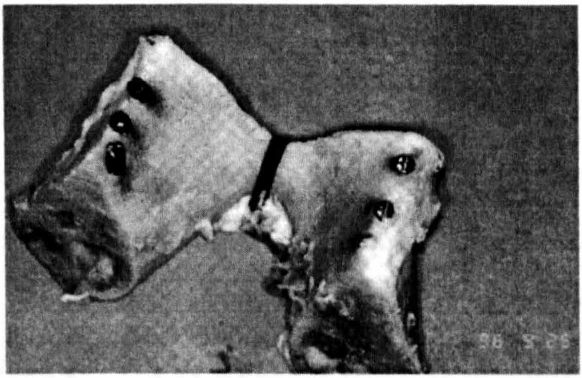

Fig.2. Photograph of lower jaw of minipig with implants.

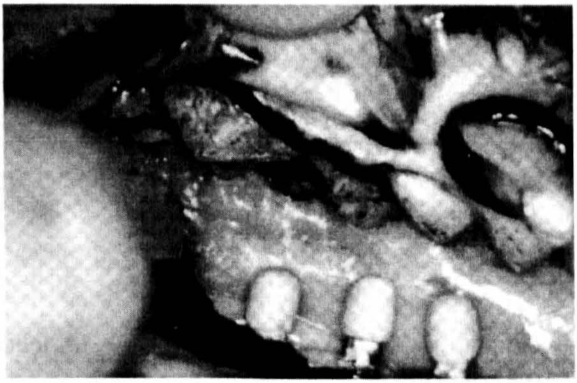

Fig.3. Lower jaw of minipig with mounted metaloceramic crowns

Fig.3. Lower jaw of minipig with mounted metaloceramic crowns

5. REFERENCES

[1] R. Reclau, J.M Mayer, J.Dent. **22**, 159 (1994).

[2] A. Thompson, F.C. Law, N. Rushton, Biomaterials **12**, 37 (199

[3] C.M. Cotell, Appl. Surf. Sci. **69**, 140 (1993).

[4] D.F. Williams, J.Mater. Sci.: Materials in Medicine **5**, 303, (199

[5] T.L. Parker, K.G. Parker, S.M. Howdle, E.N. Antonov, Bargarashvili, V.K. Popov, E.N. Sobol, C.J. Roberts, C Engineering, **91**, (1996).

[6] V.N. Bargatashvilli, E.N Antonov, E.N. Sobol, V.K. Popov Howdle, Appl. Phys. Lett. **66**, 2451 (1995).

[7] W.J. Gillespte, C.M.A. Frampton, R.J. Henderson, P.M. Rvan, J.Bone Joint Surg. **B 70**, 539 (1988).
[8] H. Schliephake, G. Reiss, R. Urban, Int. J.Oral Maxilofac. Implants **8**, 502 (1993).
[9] M Jelínek, V. Olšan, L. Jastrabík, V. Studnička, V. Hnatowicz, J. Kvítek, V. Havránek, T. Dostálová, Y. Zergioti, A. Petrakis, E. Hontzopoulos, C. Fotakis, Thin Solid Films **257**, 125 (1995).
[10] M. Jelínek M., T. Dostálová, C. Fotakis, V. Studnička, L. Jastrabík, V. Havránek, C. Grivas, V. Hnatowicz, J. Kadlec, A. Patentalaki, V. Peřina, M. Pospíchal, Laser Physics **5**, 144 (1996).
[11] M. Jelínek, V. Olšan, L. Jastrabík, T. Dostálová, L. Himmlová, J. Kadlec, M. Pospíchal, C. Fotakis, Proc. SPIE **2498**, 72 (1995).
[12] T. Dostálová, L. Himmlová, M. Jelínek, J. Bártová, Cells and Materials **5**, 255 (1995).
[13] T. Dostálová, M. Jelínek, L. Himmlová, V. Pešáková, M. Adam, Cells and Materials **6**, 117 (1996).
[14] T. Dostálová, M. Jelínek, L. Himmlová, C. Grivas, Laser Physics **8**, 182 (1998).
[15] M. Jelínek, T. Dostálová, C. Fotakis, J. Bártová, C. Grivas, L. Himmlová, Proc. SPIE **2629**, 308 (1996).
[16] T. Dostálová, M. Jelínek, C. Grivas, Proc. SPIE **2922**, 151 (1996).
[17] T. Dostálová, M. Jelínek, L. Himmlová, C. Grivas, Proc. SPIE **3192**, 73 (1997).
[18] T. Dostálová, M. Jelínek, L. Himmlová, C. Grivas, Proc. SPIE **3593**, 81 (1999).
[19] L. Himmlová, V. Dorazilová, T. Dostálová, M. Jelínek, Proc. SPIE **4166**, 245 (1999).
[20] T. Dostálová, L. Himmlová, M. Jelínek, Journal Biomedical Optics, 2001, in print.