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The Evaluation of Calcium Phosphate Coated Implants by Ion-beam Assisted Deposition (IBAD) Method in Dogs : A Preliminary Study

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Rough surfaced implants have been reported to favor early bone healing than smooth surfaced implants. Among various methods introduced to roughen the implant surface, coating with calcium phosphate(CaP) is one of them. The purpose of this study was to evaluate the bone healing response of anodized implants with two different coating thickness of CaP(200 nm and 500 nm) deposited by ion beam assisted method. CaP coated implants were placed on circumferencial coronal defects of four mongrel dogs and the amount of defect fill was evaluated histologically after 8 weeks. The amount of coronal defect filling varied among the implants according to its surface characteristics. The CaP coating thickness of 500 nm showed the greatest amount of defect fill than implants with 200 nm of CaP coating thickness and non coated implants. Within the limits of this study, when coating implants with CaP by ion-beam assisted deposition method, coating thickness of 500 nm seemed to be effective.

Key words: Dental implants, Calcium phosphate coating, Ion beam assisted method

Introduction

Restoring missing teeth with dental implants has become a widely accepted and predictable modality.[1] The success of implant treatment, especially during bone healing process, is highly related to bone quality and implant surface topography. Several studies have shown variability in success rate with implants placed in different bone quality, presenting that implant sites with higher bone quality have higher success rate than sites with poorer bone quality.[2-6] Several attempts such as using longer or wider implant, placing additional implants and using surface modified implants have been made to increase the success rate of implants placed in poor quality bone.[4]

Surface modification of dental implants has presented to increase osseointegration to bone.[7-9] Rough surfaced implants increase bone to implant contact and speed up bone apposition than smooth surfaced implants.[4] Among several methods being introduced to produce a rough surfaced implant, coating with calcium phosphate is one of them.

Calcium phosphates(CaP) or hydroxyapatite(HA) is a biocompatible and an osteoconductive material which has been commonly used as a coating material to dental implants. It has a similar chemical property to inorganic component of bone tissues and improves cellular response thereby increasing bioactivity.[10-13] Among various coating techniques, plasma spraying is the primary method in coating CaP to dental implants. However, this method has shown problems such as low bonding strength and nonuniformity of the coating layer presenting controversy outcomes.[14-16]

The dissolution rate of the coating layer is related to bone cell activity, and is affected by Ca/P ratio and cyrstallinity of the CaP particles. The more the crystalline phase formation, there is more reduction in dissolution rate, which increases the stability of the coating surface.

Ion-beam-assisted deposition(IBAD) method is a recently introduced CaP coating technique and research has shown that CaP coating by IBAD method produces a coating layer with low dissolution rate by increasing Ca/P ratio.[17,18]

The objective of the present study was to evaluate the bone healing response of CaP coated implants by IBAD method with two different coating thicknesses in surgically created defects of dogs.

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Materials and Methods

Implant sample preparation

Anodized implants were coated with CaP by IBAD method. The CaP coating layer was coated to a thickness of either 200 nm or 500 nm. The detailed procedure for CaP coating followed the method of our previously published papers.[17,19] Briefly, pure titanium implants were anodized at 270 V for 3 minutes. CaO(Cerac, USA) and HA(Alfa, USA) were sintered at 1200 ^{o}C for 2 hours in air to prepare calcium phosphate evaporates. An electron beam evaporator (8.5 Kv rated power supply, Telemark, USA) and an end-hall type ion gun(Commonwealth Scientific, USA) were employed for ion deposition.

Animals

Four male mongrel dogs were used and all had intact dentition with healthy periodontium. Animal selection, management, and surgical procedures followed a protocol approved by the Animal Care and Use Committee, Yonsei Medical Center, Seoul, Korea.

Surgical procedure

All mandibular premolars and the first molar were extracted and were healed for 8 weeks. A crestal incision was made followed by a mucoperiosteal flap refection, and implant ostectomy was prepared with additional circumferencial coronal defect creation (2 mm gaps) by use of a customized paralleled step drill. Three implants (Implantium®, Dentium, Korea) with a diameter of 3.4 mm and 10 mm of length were placed on each mandible. The implant were divided into three groups according to their surface treatment. The control group received anodized implants, experimental group 1 received anodized implants with CaP coating of 200 nm thickness and experimental group 2 received anodized implants with CaP coating of 500 nm thickness. The flaps were sutured with a resorbable suture material and were removed after 10 days. After 8 weeks of healing, the animals were sacrificed and block sections were obtained for histological analysis.

Histological analysis

The block sections were fixed in 10% buffered formalin. The specimens were dehydrated in ethanol, embedded in methacrylate, and sectioned in the mesio-distal plane. From each implant site, the central section was reduced to a final thickness of about 20 μm. The sections were stained in hematoxylin and eosin and histological analysis was performed using a stereoscope and microscope.

Results

Clinical findings

General healing was uneventful and implants were all well maintained throughout the healing period with no implant loss.

Histological findings

Three implants presented mild inflammation in the soft tissue adjacent to the implant surface, but did not affect the bone below the connective tissue area.

All the implants showed bone to implant contact at the apical area irrespective of the experimental groups. However, the amount of coronal gap filling varied among the groups. The experimental group 2 (500 nm thickness) presented the greatest amount of coronal defect filling than the two other groups (Figure 3). The newly formed bone lined the implant surface and was well integrated to the implant surface. Many osteoblasts surrounded the newly formed bone representing bone formation activity. Reversal lines were also observed at the bone distant to the implant surface and the density of the bone was comparable to the existing bone.

The experimental group 1 (200 nm thickness) and the control group presented limited coronal defect filling with no bone to implant contact even at the threads below the microthread area (Figure 1,2). Loose connective tissue was dominant with epithelial migration, which revealed failure in achieving osseointegration at the coronal defect area.

Discussion

In order to provide a favorable bone healing environment for early osseointegration of dental implants, surface properties of dental implants are important. Implant surface topography and chemical composition influence osteoblastic proliferation thereby encouraging bone ingrowth and apposition.[20,21] Early bone healing of dental implants is not only beneficial to the clinicians, but is also beneficial to the patients receiving dental implants which provides a shorter treatment time and less discomfort. Approaches in achieving surface modification includes, acid etching, blasting, oxidizing, plasma spraying, and coating the surface with osteoconductive materials.

Calcium phosphates(CaPs) are widely used osteoconductive material in coating implant surface. When CaP is coated on titanium implants, favorable mechanical and biological properties are combined.[22] Coating with CaP increases the surface area[23] and its biocompatibility and osteoconductive effect favor cellular activity, enhancing osseointegration.[10-13]

CaP coating on implant surface could be applied by various methods, including electrophoretic deposition, dip coating, hot isostatic pressing, flame spraying, plasma spraying, and pulsed laser deposition. The most commonly used method is plasma spraying, however, this method has presented nonuniformity and low adhesion of the coating layer to the implant surface.[14-16]

Ion beam assisted deposition(IBAD) is a recently developed method in coating CaP on titanium implant surface. CaP coatings by IBAD method allow formation of nano crystalline CaP coating layer which enhances cellular activity.[24] The nano

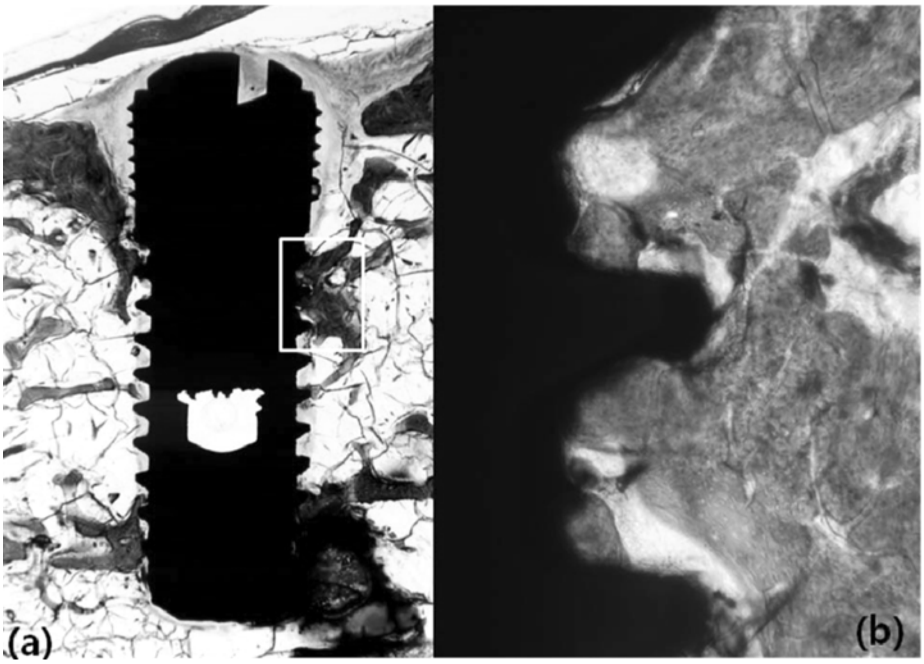

Figure 3. Histological view of experimental group 2 (CaP coating thickness 500 nm). (a) overall view, bone filling observed on micro-thread area (magnification × 10). (b) higher magnification, bone to implant contact observed on implant thread area (magnification × 100).

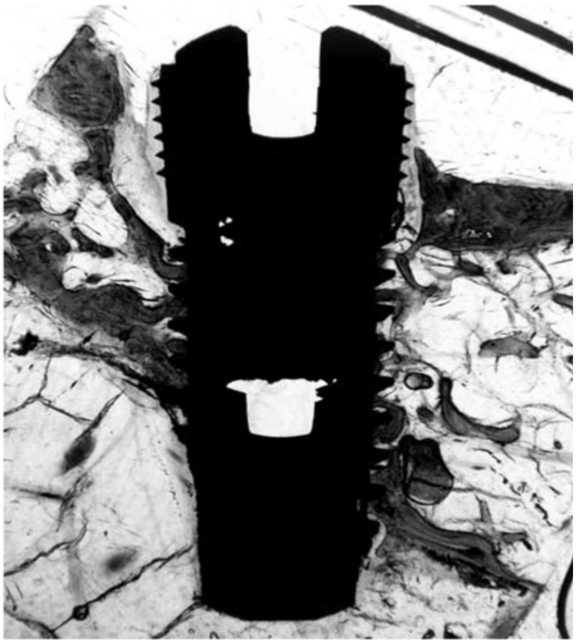

Figure 1. Histological view of the control group. Minimal amount of defect fill observed at the coronal defects with no bone to implant contact at the coronal micro-thread area (magnification × 10).

Figure 2. Histological view of experimental group 1 (CaP coating thickness 200 nm). Limited amount of bone formation observed at the coronal defect area (magnification × 10).

crystalline layer has low dissolution rate in physiological fluids and produces a stable coating layer compared to that of plasma spray method. In the experiment by Choi et al. HA was coated on titanium based metal substrate by IBAD method, and the results presented a remarkable increase in Ca/P ratio with greater crystalline HA phase formation.[17)]

In the present study, anodized implants coated with two different coating thickness of CaP, by using IBAD method were evaluated for its bone healing around circumferential coronal defects.

The thickness of the coating layer to implant surface varies from micro to nano size depending on selected coating technique. Hot isostatic pressing produces a coating thickness of more than 200 μm and plasma spray procedure produces surface coating thickness from 20 μm to 200 μm. Wet techniques such as sol-gel, and high temperature techniques such as radio frequency(RF) sputtering or ion beam method produce much thinner coating layer.[24] Therefore, the optimal thickness varies among the coating technique applied. However, whatever the coating method being used, the final CaP coating layer formed should have a thickness sufficient to compensate its dissolution process. If the coating layer is too thick, the outer layer may detach, and if the coating layer is too thin, the stability of the coating layer may decrease due to poor cyrstallinity. The optimum thickness of the commercially available plasma spray HA coated implants is approximately 50 μm[25,26] However, by IBAD method, this coating thickness could be reduced to nano size. And since cells interact only with the top coating layer, such nano size thickness is sufficient for cellular activity to take place.[24]

In the present study, CaP was coated on anodized implants with a thickness of 200 nm and 500 nm. The results of the histological studies revealed better coronal defect fill with 500 nm of CaP coating thickness than 200 nm of thickness (Figure 2,3). However, comparable defect filling was observed with 200 nm CaP coated anodized implants and non coated implants (Figure 1,2). The reason for observing such results is assumed to be due to low crystallinity of CaP particle. Several researches presented that post heat treatment to CaP coated implants by IBAD method increases crystallinity of the CaP particles.[27,28] In the study by Li *et al.*[18] post heat treatment(350 ^{o}C) to CaP coated anodized implants showed a grow in particle size and a change in crystalline phase to HA phase, and concluded that a coating layer stable in physiological fluid could be achieved after post heat treatment. Kim *et al*. also presented improved characteristics of bone healing with CaP coated implants when post heat treatment was performed, with observation of no separation of the coating layer from the implant surface.[29] Moreover, post heat treatment also showed beneficial bone healing response.[30] When anodized implants coated with CaP by IBAD method were heat treated at 430 ^{o}C and implanted on circumferential coronal defects of beagle dogs, higher bone to implant contact was observed after 12 weeks.

Although the number of animals used in the present study was limited and only histological evaluation was performed, the results of the present experiment suggest that CaP coating thickness of 500 nm to be effective for its stability in physiological fluids, and post heat treatment is recommended for better stability when using IBAD method. However, further experiments are needed with a larger sample size and with post heat treated implants in order to confirm such findings.

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Reference

1. N. H. Creugers, C. M. Kreulen, P. A. Snoek and R. J. de Kanter. "A systematic review of single-tooth restorations supported by implants." *J Dent*. **28**, 209-217, (2000).
2. R. Adell, B. Eriksson, U. Lekholm, P. I. Branemark and T. Jemt. "Long-term follow-up study of osseointegrated implants in the treatment of totally edentulous jaws." *Int J Oral Maxillofac Implants*. **5**, 347-359, (1990).
3. P. I. Branemark, B. Svensson and D. van Steenberghe. "Ten-year survival rates of fixed prostheses on four or six implants ad modum Branemark in full edentulism." *Clin Oral Implants Res*. **6**, 227-231, (1995).
4. D. L. Cochran. "A comparison of endosseous dental implant surfaces." *J Periodontol*. **70**, 1523-1539, (1999).
5. T. Jemt. "Fixed implant-supported prostheses in the edentulous maxilla. A five-year follow-up report." *Clin Oral Implants Res*. **5**, 142-147, (1994).
6. T. Jemt and U. Lekholm. "Implant treatment in edentulous maxillae: a 5-year follow-up report on patients with different degrees of jaw resorption." *Int J Oral Maxillofac Implants*. **10**, 303-311, (1995).
7. L. Carlsson, T. Rostlund, B. Albrektsson and T. Albrektsson. "Removal torques for polished and rough titanium implants." *Int J Oral Maxillofac Implants*. **3**, 21-24, (1988).
8. K. Gotfredsen, T. Berglundh and J. Lindhe. "Anchorage of titanium implants with different surface characteristics: an experimental study in rabbits." *Clin Implant Dent Relat Res*. **2**, 120-128, (2000).
9. C. J. Ivanoff, C. Hallgren, G. Widmark, L. Sennerby and A. Wennerberg. "Histologic evaluation of the bone integration of TiO(2) blasted and turned titanium microimplants in humans." *Clin Oral Implants Res*. **12**, 128-134, (2001).
10. H. W. Kim, H. E. Kim and J. C. Knowles. "Fluor-hydroxyapatite sol-gel coating on titanium substrate for hard tissue implants." *Biomaterials*. **25**, 3351-3358, (2004).
11. H. W. Kim, Y. H. Koh, L. H. Li, S. Lee and H. E. Kim. "Hydroxyapatite coating on titanium substrate with titania buffer layer processed by sol-gel method." *Biomaterials*. **25**, 2533-2538, (2004).
12. L. Gan, J. Wang, A. Tache, N. Valiquette, D. Deporter and R. Pilliar. "Calcium phosphate sol-gel-derived thin films on porous-surfaced implants for enhanced osteoconductivity. Part II: Short-term in vivo studies." *Biomaterials*. **25**, 5313-5321, (2004).
13. H. Q. Nguyen, D. A. Deporter, R. M. Pilliar, N. Valiquette and R. Yakubovich. "The effect of sol-gel-formed calcium phosphate coatings on bone ingrowth and osteoconductivity of porous-surfaced Ti alloy implants." *Biomaterials*. **25**, 865-876, (2004).
14. C. P. Klein, P. Patka, H. B. van der Lubbe, J. G. Wolke and K. de Groot. "Plasma-sprayed coatings of tetracalciumphosphate,

hydroxyl-apatite, and alpha-TCP on titanium alloy: an interface study." *J Biomed Mater Res*. **25**, 53-65, (1991).
15. K. Hayashi, T. Inadome, T. Mashima and Y. Sugioka. "Comparison of bone-implant interface shear strength of solid hydroxyapatite and hydroxyapatite-coated titanium implants." *J Biomed Mater Res*. **27**, 557-563, (1993).
16. E. A. McGlumphy, L. J. Peterson, P. E. Larsen and M. K. Jeffcoat. "Prospective study of 429 hydroxyapatite-coated cylindric omniloc implants placed in 121 patients." *Int J Oral Maxillofac Implants*. **18**, 82-92, (2003).
17. J. M. Choi, H. E. Kim and I. S. Lee. "Ion-beam-assisted deposition (IBAD) of hydroxyapatite coating layer on Ti-based metal substrate." *Biomaterials*. **21**, 469-473, (2000).
18. Y. Li, I. S. Lee, F. Z. Cui and S. H. Choi. "The biocompatibility of nanostructured calcium phosphate coated on micro-arc oxidized titanium." *Biomaterials*. **29**, 2025-2032, (2008).
19. I. S. Lee, C. N. Whang, H. E. Kim, J. C. Park, J. H. Song and S. R. Kim. "Various Ca/P ratios of thin calcium phosphate films." *Materials Science and Engineering C*. **22**, 15-20, (2002).
20. V. Sampathkumaran, M. R. DeGuire and R. Wang. "Hydroxyapatite coatings on titanium." *Advanced engineering materials*. **3**, 401-405, (2001).
21. M. Jayaraman, U. Meyer, M. Buhner, U. Joos and H. P. Wiesmann. "Influence of titanium surfaces on attachment of osteoblast-like cells in vitro." *Biomaterials*. **25**, 625-631, (2004).
22. V. M. Frauchiger, F. Schlottig, B. Gasser and M. Textor. "Anodic plasma-chemical treatment of CP titanium surfaces for biomedical applications." *Biomaterials.* **25**, 593-606, (2004).
23. K. A. Gross and M. Babovic. "Influence of abrasion on the surface characteristics of thermally sprayed hydroxyapatite coatings." *Biomaterials*. **23**, 4731-4737, (2002).
24. R. Narayanan, S. K. Seshadri, T. Y. Kwon and K. H. Kim. "Calcium phosphate-based coatings on titanium and its alloys." *J Biomed Mater Res B Appl Biomater*. **85**, 279-299, (2008).
25. K. de Groot, R. Geesink, C. P. Klein and P. Serekian. "Plasma sprayed coatings of hydroxylapatite." *J Biomed Mater Res*. **21**, 1375-1381, (1987).
26. R. G. Geesink, K. de Groot and C. P. Klein. "Chemical implant fixation using hydroxyl-apatite coatings. The development of a human total hip prosthesis for chemical fixation to bone using hydroxyl-apatite coatings on titanium substrates." *Clin Orthop Relat Res*. 147-170, (1987).
27. J. Chen, W. Tong, Y. Cao, J. Feng and X. Zhang. "Effect of atmosphere on phase transformation in plasma-sprayed hydroxyapatite coatings during heat treatment." *J Biomed Mater Res*. **34**, 15-20, (1997).
28. Y. Yang, K. H. Kim, C. M. Agrawal and J. L. Ong. "Influence of post-deposition heating time and the presence of water vapor on sputter-coated calcium phosphate crystallinity." *J Dent Res*. **82**, 833-837, (2003).
29. M. K. Kim, J. Y. Choi, I. S. Lee, T. Inoue and S. H. Choi. "The Effects of Ion Beam-Assisted Depoistion of Hydroxyapatite on the osseointegration of Endosseous Implant Surface." *Key Engineering Materials*. 330-332, 597-600, (2007).
30. J. C. Chae, U. W. Jung, S. M. Jung, I. S. Lee, K. S. Cho, C. K. kim and S. H. Choi. "Healing of surgically created circumferential gap aroung Nano-coating surface dental implants in dogs." *Surface and Interface Analysis*. **40**, 184-187, (2008).