The International Journal of
Prosthodontics

Special reprint

Mandibular Bone Response to Plasma-Sprayed Coatings of Hydroxyapatite

*H.W. Denissen, DDS, PhD**

*W. Kalk, DDS, PhD***

*H.M. de Nieuport, DDS****

*J.C. Maltha, PhD*****

*A. van de Hooff, MD, PhD******

*University of Nijmegen and
University of Amsterdam, The Netherlands*

1

VOLUME 3
JANUARY/FEBRUARY 1990

ITEMS : 5D 5E 5F

Mandibular Bone Response to Plasma-Sprayed Coatings of Hydroxyapatite

*H.W. Denissen, DDS, PhD**

*W. Kalk, DDS, PhD***

*H.M. de Nieuport, DDS****

*J.C. Maltha, PhD*****

*A. van de Hooff, MD, PhD******

University of Nijmegen and University of Amsterdam, The Netherlands

Sintered hydroxyapatite ceramic particles can be applied as a coating on a titanium substrate using a plasma-spraying technique. The biological and mechanical properties of implants with such a coating were studied in the mandible of a dog. The results indicated that a very strong and direct bonding between the hydroxyapatite coating and the mandibular bone developed. The shear strength of the bone/ceramic interface was higher than the interfacial strength between ceramic and titanium substrate. From the radiologic, macroscopic, and microscopic observations it was concluded that the biological properties of plasma-sprayed coatings of hydroxyapatite are the same as those of sintered hydroxyapatite ceramic. As a result of mechanical failure of the coherence of the hydroxyapatite particles at the outer layer of the coating, free particles of hydroxyapatite were observed in the surrounding bone tissue. *Int J Prosthodont 1990;3:53–58.*

Dense "hydroxyapatite" (HA) ceramic has been tested extensively in animals and clinical research.[1-3] The biologic properties of implants made of sintered HA ceramic are excellent. However, HA ceramic cannot be used in load-bearing situations because of its poor mechanical properties. Only recently have reports been published on a composite implant consisting of a titanium (Ti) substrate coated with a bioactive surface of HA by a plasma-spraying technique.[4] Plug implant studies were done in femurs of dogs.[5,6] From these studies it was concluded that a strong bond between HA and bone formed very rapidly and that close apposition between bone and HA coating after implantation was not necessary. The physical strength of implants with plasma-sprayed coatings of HA on Ti, including the fatigue strength, was far superior to that of sintered HA alone. Biocompatibility and biostability appeared the same as those of sintered HA.[4]

It is felt, however, that data on reactions of the femurs of dogs cannot be applied directly to mandibular bone, because mandibular bone undergoes a continuous resorption during the postoperative nonfunctional period while the femur maintains its function. It is also possible that the healing process of the edentulous mandible differs from that of the femur from a histomorphologic point of view, because the mandible is developed by intramembranous bone formation while the femur is derived by endochondral bone formation. Furthermore, implantations in fresh extraction sites in the mandible differ from implantations in specially prepared implant sockets in the femur.

*Associate Professor, Department of Oral Function and Prosthetic Dentistry, University of Nijmegen School of Dentistry, PO Box 9101, 6500 HB Nijmegen, The Netherlands.

**Professor and Head of the Research Programme, Department of Oral Function and Prosthetic Dentistry, University of Nijmegen School of Dentistry.

***Research Associate, Department of Oral Function and Prosthetic Dentistry, University of Nijmegen School of Dentistry.

****Associate Professor, Department of Oral Function and Prosthetic Dentistry, University of Nijmegen School of Dentistry.

*****Professor, Department of Histology and Bone Biology, University of Amsterdam.

This study evaluated the bone response to implants with plasma-sprayed coatings of HA placed directly after extraction of teeth in dog mandibles.

Materials and Methods

Implants with Plasma-Sprayed Coatings of Hydroxyapatite

The principle of the plasma-spraying procedure has been described by Bunshah[7] and more recently by De Groot et al.[4] An electric arc is struck between two electrodes, while a stream of gases passes through this arc. This results in an ionized gas, called a plasma. This plasma reaches a temperature of 30,000 °C and approaches the speed of sound.

Sintered HA particles are then suspended in a carrier gas stream, which is fed into the plasma flame. Because of the high temperature, the HA particles explode and form new smaller particles of 1 to 5 μm. The particles start to melt at the surface and are then sprayed on the roughened surface of the titanium core of the implant. The result of the spraying procedure is a chemical bonding between the HA particles and the titanium-oxide layer on the Ti core. The HA coating is applied in successive layers of 5 to 10 μm and has a final thickness of approximately 50 μm.[4]

For the evaluation of our experimental results, it is important to know that the outer layer of the coating (10 to 15 μm) is not as dense as the inner layer (35 to 40 μm). It is porous to a minor degree and granular. Other important properties can be summarized as follows:

1. The HA coating consists of 90% pure HA and 10% calcium phosphates without apatite structure. This is the same composition as our sintered bulk HA.[2]
2. Fatigue failure of the coating does not occur even after 10 million loads of 100 to 6,000 N.[4]
3. Strength and other properties are not adversely affected by sterilization with ethylene oxide or gamma irradiation.[5]

Two different designs of implants were used (Dyna® Dental Engineering, Bergen op Zoom, The Netherlands). One type had a straight cylindrical design. The second was also cylindrical, but had a grooved surface. The grooves increase the potential bone/HA contacting area. A total of 16 implants with plasma-sprayed coatings of HA was placed. Six implants were cylindrical, while ten implants had a grooved surface (Fig 1).

Fig 1 The two experimental plasma-sprayed–hydroxyapatite (PS-HA) designs used in this study. The implant on the left has a grooved surface, while the implant on the right has a smooth cylindrical design with a rounded apex. The occlusal surfaces (not visible) are not coated with HA ceramic.

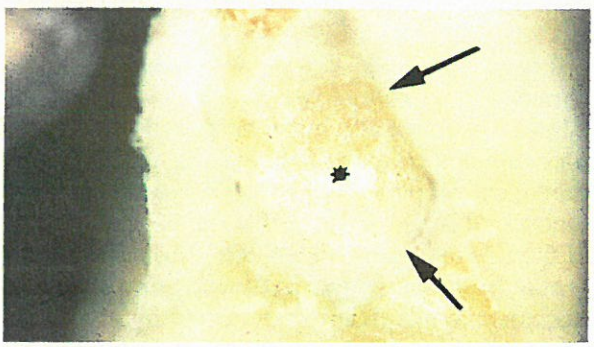

Fig 2 Mandible cross-sectioned at the site where a cylindrical implant has been extracted. Bone closely follows the contour of the implant (arrows). The HA coating is still strongly attached to the bone (*). A microscopic image of such a HA/bone interface is shown in Fig 6.

Surgical Procedures

Two beagle dogs were used for this investigation. They were approximately 10 years of age, and weighed 13 and 15 kg. The animals were caged under normal laboratory conditions in the Central Animal Laboratory of the University of Nijmegen, The Netherlands. Prior to surgery, the animals were premedicated intramuscularly with 0.5 mg atropine and 1 mL Thalamonal. Subsequently, the animals were anesthetized intravenously with 15 mg/kg Pentothal. After intubation, anesthesia was maintained with a mixture of ethane, N_2O, and oxygen. The third and fourth premolars of the mandible were extracted. The apical parts of the sockets were reamed using a cylindrical bur with internal cooling. The bur had a diameter of 3 mm, the same as the plasma-sprayed–hydroxyapatite (PS-HA) implants. The operation field was continuously flooded with physiological saline. The gingiva was mobilized and sharp bony ridges were removed. Next, the implants

were placed into the sockets without hammering or screwing. The tissue was closed with sutures, using Vicryl 4.0. Six cylindrical and ten grooved implants were randomly chosen. The animals were kept on a soft diet for the first week after surgery.

Radiologic Procedures

Standardized extraoral radiographs were prepared using a specially developed animal cephalostat.[8] For standardized intraoral radiographs, an individual splint carrying a holder for dental x-ray film was used.[1] This provided the possibility of a rapid and reproducible orientation of the film in the mandibular premolar region.

Morphologic Procedures

After an experimental period of 9 months, the animals were sacrificed by an overdose of Nembutal. Perfusion with physiological saline was followed by a 10% buffered formalin solution. Areas of the mandible carrying the implants were dissected and immersed in 10% buffered formalin solution for further fixation. Portions of the tissue blocks were decalcified in 20% formic acid with 5% sodium citrate. After decalcification, the implants were removed from the bone, and the tissue blocks were dehydrated and embedded in paraffin. Serial sections of 10 μm were cut on a base sledge microtome. The sections were stained with hematoxylin and eosin. The other tissue blocks were embedded undecalcified in poly(methyl methacrylate). Sections of 50 μm were prepared and stained according to the Van Gieson technique. The remnants of the blocks used for sawing were sputter coated with gold and studied with a scanning electron microscope.

Results

Gross Observations

The roots of the premolars were very difficult to remove due to ankylosis. The socket walls were often damaged because of the removal procedure. Therefore, the implants initially were very ill-fitting in the root sockets.

Two of the grooved-surface implants were removed because there was a dehiscence of the overlying mucosa. The remaining implants (six cylindrical and eight grooved) were retained in situ, covered with mucosal tissue. Exfoliation during the observation period of 9 months did not occur, and the alveolar ridges appeared healthy. After retrieval, the implants appeared to be fully incorporated in the mandibular bone. Macroscopically, all implants were strongly attached to the bone. This bonding resembled the bonding of HA bulk implants to dog mandibular bone.[1] During the process of cutting the implant/bone specimens into two halves, it also appeared that all the implants were very strongly bonded to the adjacent bone. It was not possible to remove the implants with the HA layer intact. Extraction of the implants always resulted in detachment of the HA coating from the Ti core. The interface between HA and bone, however, remained intact (Fig 2). These gross observations warrant the conclusion that the HA coating was intimately and strongly bonded to the mandibular bone. (5E)

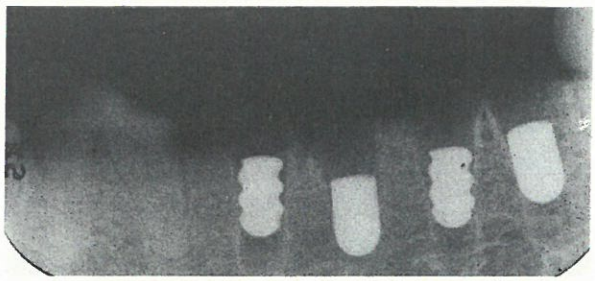

Fig 3 Radiograph made directly after implantation of PS-HA implants of both designs in the root sockets of the third and fourth premolar. Radiolucencies are present around the implants because of the lack of adaptation of the implants to the extraction sites.

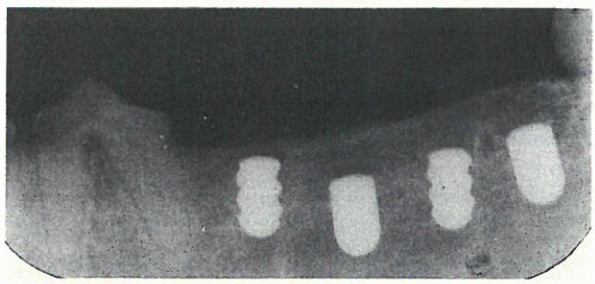

Fig 4 Radiograph of the same implants as in Fig 3. The radiolucencies have disappeared and the implants are well tolerated. No bone resorption is apparent; only bone apposition has taken place.

Radiographic Results

From the radiographs made directly after implantation, it could be deduced that large radiolucencies were present around the implants (Fig 3). Follow-up radiographs showed that mandibular bone was deposited in very close proximity to the implant surfaces and that the radiolucencies disappeared completely (Fig 4). In this respect, it seems that no

substantial changes occurred up to 9 months after implantation, ie, at the time of sacrifice of the animals. Depending on the initial height of the implant with regard to the crest of the alveolar ridge, the implants appeared on the radiographs to be completely, partially, or not at all covered by bone.

Microscopic Findings

The tissues adjacent to both types of implants showed identical characteristics. In the experimental period, bone was deposited in the extraction socket. Large surface areas of the HA coating were in close contact with the bone. The bone was of the lamellar type and it was mostly laid down as secondary osteons, although deposits directly against HA surfaces were also observed (Figs 5 and 6). In those areas where no connection between HA and bone was established, fatty bone marrow was found. No signs of inflammation were observed. At the apical aspect of the implant there was no bone or only a very thin layer of lamellar bone deposited. Most often the apex protruded freely into the fatty bone marrow (Fig 7). There was no bone deposition occlusal to the implants, and the implants were covered with a fibrous capsule (Fig 8). The outermost layer of the HA coating appeared to be unstable under the experimental circumstances. Clusters of globular HA particles were found in the fatty bone marrow. Usually such a cluster was in the vicinity of a nucleus, suggesting that the particles might have been ingested by macrophages. However, because of the masking effect of the HA particles, it is not possible to decide whether indeed they are located intracellularly (Fig 9). In several areas, free HA particles were also located at bone surfaces and incorporated within the bone matrix. Detachment of the HA coating from the Ti core, but a bonding between the coating and mandibular bone, was observed with the scanning electron microscope (Fig 10).

Discussion

The most remarkable radiologic and microscopic finding was that the HA-coated implants originally placed in large extraction defects in the mandible were completely surrounded by newly formed bone and could not be extracted without fracturing the HA coating from the Ti substrate. Although the reliability of extraction tests is questionable, these tests nevertheless gave an indication of the strength of the bond between the mandibular bone and HA coating. Since after extraction tests the HA coating

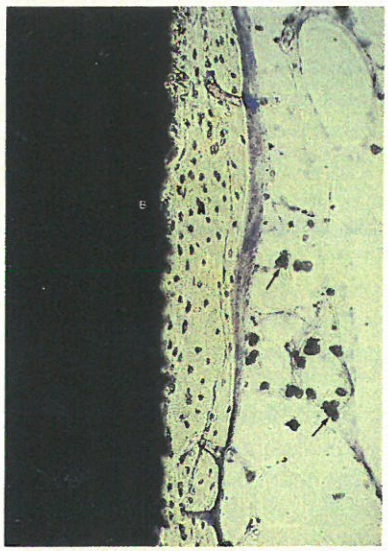

Fig 5 Undecalcified section of methacrylate-embedded mandibular bone (A) adjacent to a cylindrical implant (B) that has been in situ for 9 months. A very tight connection between the lamellar bone and the HA coating is found. Clusters of HA particles are present in the bone marrow (arrows). (Sawed section, Van Gieson stain, original magnification × 200.)

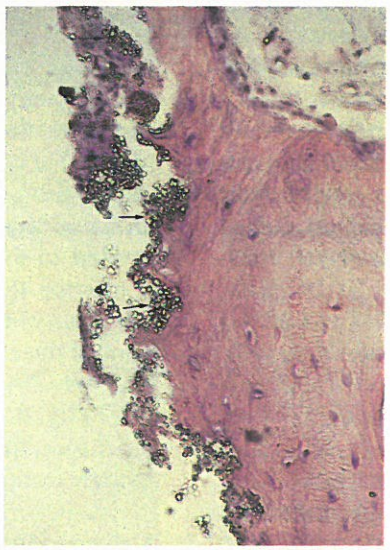

Fig 6 Decalcified paraffin section after removal of the implant. Remnants of the HA coating (arrows) that have survived the decalcification remain attached to the bony surface. (H&E stain, original magnification × 500.)

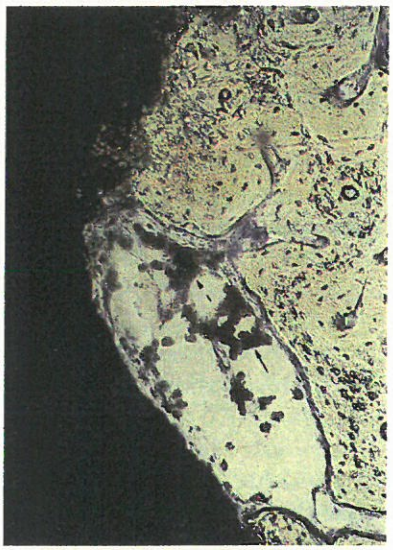

Fig 7 Technique as in Fig 5. In areas where no bone is in direct contact with the implant, fatty bone marrow is found. Clusters of the HA particles (arrows) are found within the marrow. (Original magnification × 200.)

appeared to be detached from the Ti rather than from the bone, it can be concluded that the union between the coating and the bone is higher than that between the HA coating and the Ti substrate. This is in agreement with the results of studies on plasma-sprayed coatings in dog femoral bone. In fresh samples of dog femur, the shear strength of the HA/bone interface appeared to be 30 MPa.[5,6] The shear strength of the HA/Ti interface was only approximately 22 MPa, while the shear strength of the cortical bone itself was 45 MPa.[4,9,10] These data render it feasible to conclude that it is the interface between the coating and Ti substrate that fails first.

The observation, using scanning electron microscopy, of large areas with a discontinuity between the HA coating and Ti core of the implant is further evidence of the strong bonding between HA and bone. It is well known that histologic procedures, and especially dehydration, induce shrinkage of biologic structures. Because no shrinkage will take place in the implant material, forces might be generated at the border between the implant and tissue. If those forces are large enough, fracturing can occur. It then appears that the attachment between bone and HA is stronger than the attachment between HA and Ti. The reason for the strong bonding of the coating to the cortical bone must also be attributed to factors such as old age and the degree of difficulty of the extractions (in fragments) in these particular dogs, which indicated some measure of ankylosis of the natural tooth roots. Such an environment is favorable for implant success. In other words, the quality of the bone is very important.

Research with smooth Ti implants has indicated that uncoated Ti surfaces have no bonding properties.[11] The highest shear strength value ever found was only 0.6 MPa. Although bone is deposited very closely to the bioinert Ti surface, it is not chemically bonded.[12] Therefore, the retention of a Ti implant must be achieved through mechanical retention, by using an undercut surface such as a screw or blade shape. By coating Ti with HA ceramic, a dental implant with a 50-times-higher shear strength to mandibular bone can be obtained. Porous Ti/bone interfaces can achieve a shear strength of approximately 20 MPa. This value comes closer to that of HA coating and is practically equal to the shear strength of the HA coating/Ti interface. Plasma-sprayed coatings, however, have the following advantages:

1. Contrary to other implant systems, a good fit of the HA-coated implant in the implant socket is not necessary. This is considered a great advantage over other implant systems, which are dependent on mechanical retention by

Fig 8 Technique as in Fig 5. The occlusal surface of the implants, which was not coated with HA, is covered with a fibrous connective tissue capsule (arrows). (Original magnification × 200.)

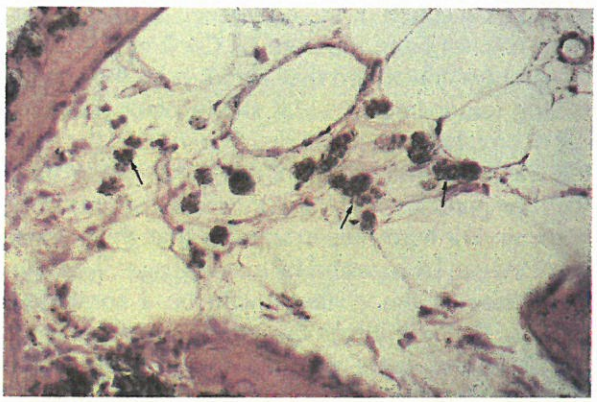

Fig 9 Technique as in Fig 6. Clusters of HA particles (arrows) are related to nuclei, indicating a possible ingestion of the particles by macrophages. (Original magnification × 500.)

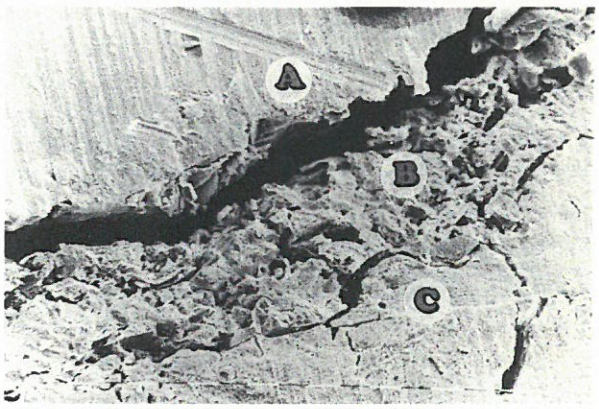

Fig 10 Scanning electron micrograph of the interface between the Ti core of the implant (A), the HA coating (B), and the bone (C). As a result of histologic processing procedures, a large crack is found between the Ti core and the HA coating. The interface between the HA coating and bone is almost intact. Smaller cracks are also observed in the HA coating itself. (Original magnification × 1,250.)

osseointegration. These later systems need a very precise initial fitting of the screw-shaped implant surfaces to the mandibular bone, without which they fall. The HA-coated systems do not require a complicated procedure and expensive precision instruments to obtain this fitting, because in a prepared cylindrical socket, mandibular bone grows directly up to the coating regardless of whether the implant has a straight cylindrical or grooved surface.

2. The HA coating results in a more rapid bonding to the bone than uncoated surfaces. Three months after implantation, the bonding strength has already attained its maximum value.[5]

The authors have not studied the thickness of the HA coating after the sacrifice of the animals. Disappearance of part of the coating because of mechanical failure of the coherence of the HA particles has not been reported by Cook et al.[13] Other researchers concluded that after 2 years the coating of dog femur implants was still in place, but they did not exclude the possibility that some of the coating was lost.[5] The results of this investigation show that free particles of HA are present in the fatty bone marrow. Furthermore, it appears that those loose particles can be ingested by macrophages. Mechanical failure of the coating might be a continuous process that will affect the entire coating in time, but it is also quite possible that this process is restricted to the more porous outer layer (10 to 15 μm) of the coating. The attachment of the bone to the coating appears not to be dependent on the surface structure of the coating. This implies that, after loss of the outer layer of the coating, bonding between the bone and the more compact inner layer of the coating can be established.

Our animal experiments are comparable to the first phase of the so called two-phase implantation procedure in patients, because the implants also remain under the mucosa during the healing phase. However, the healing phase in patients is less complicated, because the implants are placed in specially prepared implant sockets instead of extraction cavities as in our animal experiments.

Conclusions

From these experiments it can be concluded that plasma-sprayed coatings of HA ceramic become strongly bonded to mandibular bone. This occurs even when they are implanted in fresh extraction sites. No adverse biologic effects of the implants were observed. It is possible that the thickness of the coating has diminished because of loss of particles from the surface. We can only conclude that the coating was still there after the sacrifice of the animals. However, animal research of a longer duration is necessary to evaluate the stability of the HA coating in mandibular bone. In the event of loss of the HA coating, which is only 50 μm thick, this would result in direct contact between the Ti core and bone, negating all advantages.

References

1. Denissen HW, De Groot K: Immediate dental root implants from synthetic dense calcium hydroxylapatite. *J Prosthet Dent* 1979;42:551–556.
2. Denissen HW, Veldhuis AAH, Jansen HBW, Van Den Hooff A: The interface of permucosal dense apatite ceramic implants in humans. *J Biomed Mater Res* 1984;18:147–154.
3. Denissen HW, Kalk W, Veldhuis AAH, Van Den Hooff A: Eleven-year study of hydroxyapatite implants. *J Prosthet Dent* 1989;61:706–712.
4. De Groot K, Geesink RGT, Klein CPAT, Serekian P: Plasma sprayed coatings of hydroxyl-apatite. *J Biomed Mater Res* 1987;21:1375–1381.
5. Geesink RGT, De Groot K, Klein CPAT: Bone bonding to apatite coated implants. *J Bone Joint Surg* 1988;70B:17–22.
6. Geesink RGT, De Groot K, Klein CPAT: Chemical implant fixation using hydroxyl-apatite coatings. *Clin Orthop* 1987;225:147–170.
7. Bunshah RF (ed): *Deposition Technologies for Films and Coatings*. Park Ridge, NJ, Noyes Publications, 1981.
8. Maltha JC: *The Process of Tooth Eruption in Beagle Dogs*. PhD thesis, University of Nijmegen, The Netherlands, 1982.
9. Carter DR, Speugler DM: Mechanical properties and composition of cortical bone. *Clin Orthop* 1978;135:192–217.
10. Albright JA: Bone, physical properties, in Albright JA, Brand RA (eds): *The Scientific Basis of Orthopaedics*, East Norwalk, Conn, Appleton, 1979, pp 135–184.
11. Ronningen H, Solheim LF, Langeland N: Bone formation enhanced by induction; Bone growth in titanium implants in rats. *Acta Orthop Scand* 1985;56:67–71.
12. Gerner BT, Barth E, Albrektsson T, Ronningen H, Solheim LF, Wie H: Comparison of bone reactions to coated tricalcium phosphate and pure titanium dental implants in the canine iliac crest. *Scand J Dent Res* 1988;96:143–148.
13. Cook SD, Thomas KA, Jarcho M: Interface mechanics and histology of titanium and hydroxylapatite coated titanium for implant applications. *J Biomed Mater Res*, accepted for publication.