MECHANICAL FAILURE OF HYDROXYAPATITE-COATED TITANIUM AND COBALT-CHROMIUM-MOLYBDENUM ALLOY IMPLANTS AN ANIMAL STUDY

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Defects in the hydroxyapatite (HA) ceramic coatings applied to metallic implant systems may occur at the time of insertion or at the time of in vivo loading. However, defects may also occur with time because of interaction with physiological fluids.

A canine study was performed to make a histological and biomechanical evaluation of HA-coated titanium and cobalt-chromium-molybdenum alloy implants in a non-weight-bearing model. Twelve cylindrical plugs were inserted into the medial femoral condyle on 6 mongrel dogs. HA-coatings of 80-120 μm thickness were applied to 6 Cr-Co-Mo implants and 6 Ti-6AI-4V implants. The implants were removed together with adjacent bone tissue after 4 weeks.

There were no statistically significant differences between the two groups for interface shear strength. No differences were encountered with HA-coating on Cr-Co-Mo or on Ti-6AI-4V, failure consistently being caused by disruption between the metal surface and the HA-coating, which remained fixed to the bone. At histologic evaluation the HA-coated implants exhibited a great amount of direct bone-to-implant contact. In spite of that, this study indicates that HA-coating on smooth metal surfaces might not be suitable for clinical use, because of low bonding strength between metal and coating.

Keywords : hydroxyapatite ; histologic evaluation ; implant-bone interface ; interface mechanics.

Mots-clés : hydroxyapatite ; étude histologique ; interface implant-os ; mécanisme de l'interface.

INTRODUCTION

Porous materials allowing direct bone ingrowth have been used for non-cemented joint implants. Fixation by bone ingrowth has been demonstrated both in humans and in animals (2, 3, 4, 5, 16, 20, 21). Some concern has been expressed about such high-tech implants because of beads detaching from sintered surfaces or diffusion-bonded Ti-fiber mesh loosening (6, 22). Porous coatings of hydroxyapatite (HA) have demonstrated a bioactive effect, with a large degree of direct bone-to-implant contact (1, 7, 9, 10, 14, 28, 29). However, the biomechanical properties of HA are weak, and some studies have shown HA degrading by interaction with physiological fluids, raising doubts about the long-lasting stability of the interface between the HA coating and metallic substrate (25, 26, 29).

The aim of the present study was to evaluate biomechanics and histology of HA ceramic coatings applied to smooth metallic surfaces of Cr-Co-Mo or Ti-6AI-4V alloy, in a transcortical push-out model in dogs, at 4 weeks.

MATERIALS AND METHODS

Twelve HA-coated cylindrical plugs of diameter 10.00 ± 0.05 mm and length 20 mm were implanted paired in 6 dogs (table I). Ceramic HA was plasma sprayed (Plasma Biotal, UK) on the smooth surface of Co-Cr-Mo or Ti-6AI-4V plugs, resulting in a coating of 100 μm (80-120 μm). The purity of HA after spraying

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was tested routinely by xray diffraction analysis and was found to be better than 97%, containing tricalcium phosphate as the only other major constituent. Data provided by the manufacturer indicated a minimum HA coating-substrate shear strength of 20 MPa in vitro tests. The test plugs were delivered by the manufacturers, and the coating was performed in a fashion similar to joint implants for noncemented use.

The 6 dogs (20-30 kg) were premedicated with propionyl promazine 0.1 mg/kg and atropine 0.02 mg/kg and anesthetized with thiomebumal sodium. Anesthesia was induced with 0.04 mg/kg of etorphine 0.125 mg/ml and acepromazine 0.4 mg/ml and maintained with supplemental injections.

The plugs were implanted in the medial femoral condyle via a transverse hole of diameter 10.0 mm, drilled by atraumatic technique, using water cooling and successive larger drills. All the implants were inserted with a press-fit technique and protruded just outside the periosteal borders. Prophylactic antibiotics, 1 gram ampicillin intravenously, were given for 3 days. All the animals bore full weight on the hind legs within 2 days, and none were infected.

For evaluation of bone repair and remodelling tetracycline 20 mg/kg/day was supplied orally as tablets. Nine days and two days before sacrifice, intravenous injections of calcein green, 15 mg/kg/day i.v., was given. The dogs were killed 4 weeks after implantation by an overdose of mebumal sodium i.v.

The femoral condyles were removed en bloc, dissected and immediately fixed by immersion in 10% buffered formalin solution. The position of the implants was determined radiographically, and bone-implant specimens were cut perpendicular to the long axis of the implant in three sections using a microtome (Acutom2®, Struers, Denmark). The middle section of 3-mm thickness with adjacent trabeculated cancellous bone was used for a mechanical push-out test. The superficial and the deep sections were prepared for histologic examination.

Push-tests were carried out on a test machine (Instron, UK) with a constant cross-head speed of 1 mm/min. The specimens were placed on a plate with an 11-mm circular opening, thus supporting the bone surrounding the implants to within 0.5 mm of the interface. The maximum force was determined from the load-displacement curve recorded. Shear strength was calculated by dividing the extraction force by the total bone contact area, using the formula : **Area** = $\pi . \varnothing^* \mathbf{h}$ ($\varnothing$ = diameter of the plug, h = thickness of the specimen).

After fixation the specimens were dehydrated in a graded series of ethanol 60, 70, 80, 90 and 99% with changes at 24-hr intervals and embedded in methyl methacrylate. The 300 -µm sections were cut with the microtome. All sections were then mounted on Plexiglas slides using cyanoacrylic glue (B210®, 3M Inc., USA) and ground to a thickness of 40 µm on a grinding machine (Exakt Apparatebau GmbH, Germany).

Half of the specimens were simultaneously stained with 0.3% basic fuchsin before embedding and thereafter rinsed in absolute acetone, changed 3 times in 10 days. Sections stained with basic fuchsin were later counterstained with 2% light green to show contrast between mineralized bone stained green and soft tissues stained red (19). The other half of the unstained sections was examined with a fluorescence microscope, and photomicrographs were made using an Orthomat® camera and Ectachrom 160® film (Kodak). Thereafter, the specimens were stained with van Gieson picrofuchsin/Stevenels blue, staining collagen green, bone orange and osteoid yellow-green, and cells and extracellular structures were visible in blue tones (16).

Histomorphometric evaluation, including the amount of direct bone-to-implant contact to the HA-coated implants, was estimated by using a conventional point-counting technique with a 100-point grid with gridlines that were 250 µm apart. Each section was evaluated by placing the grid at 5 randomly selected locations around the implant surface.

Statistical analyses were performed with a Wilcoxon test for paired data.

RESULTS

The results of the push-out tests are summarized in table I. No difference was found between the HA-coating on Cr-Co-Mo or Ti-6Al-4V ($p = 0.6875$, Wilcoxon test). The median interface shear strength was 0.3 MPa and 0.4 MPa, respectively.

All HA-coated implants failed between the metal core and the HA-coating, which remained fixed to the bone. In three plugs some HA-coating remained fixed to the metal (fig. 1).

Histological sections of the HA-coated implants showed no evidence of foreign body reaction or fibrocartilage (fig. 2). The newly formed bone adjacent to the implants had a morphology similar to the surrounding bone, and thus varied from thin trabeculae to dense Haversian bone. In one

case bone formation was observed between the metal substrate and HA-coating, which had been disrupted from the core (fig. 3). The histomorphometric analyses are summarized in table I.

In the fluorescence microscope a 150-µm thick zone of tetracycline-labeled (newly formed) bone adjacent to the HA-coated implants with no interposed membrane was observed, and the demarcation line between old and newly formed bone was clearly visible (fig. 4). Calcein green produced a clearly outlined fluorescence in bone in areas which calcified at the time of administration ; its yellow color was distinctly different from the yellow of tetracycline. Single or double labeling of calcein green was observed, located in the tetracycline-labelled bone. The apposition rate, measured as the distance between two calcein green lines, was 30 to 40 µm/week.

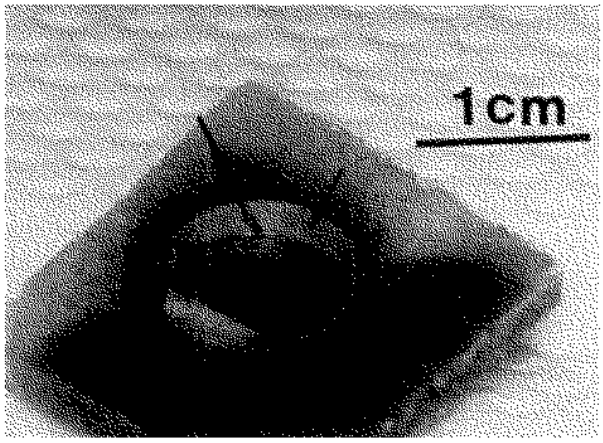

Fig. 1. — Photograph of a 3-mm section containing an HA-coated implant after the push-out test. Note that the HA-coating is fixed to the bone (small arrow) and only on a small spot is the coating still fixed to the implant (arrow).

Table I. — Results of histomorphometric evaluation and push-out test of HA-coated implants. N = number of samples. Median (Range)

Implant	Surface	Shear strength (MPa)	Bone-to-implant contact (%)
Ti-6AI-4V (Corin, Ltd.) N = 6	HA	0.4 (0.1-0.2)	32 (18-67)
Cr-Co-Mo (Corin, Ltd.) N = 6	HA	0.3 (0.1-2.2)	35 (9-67)

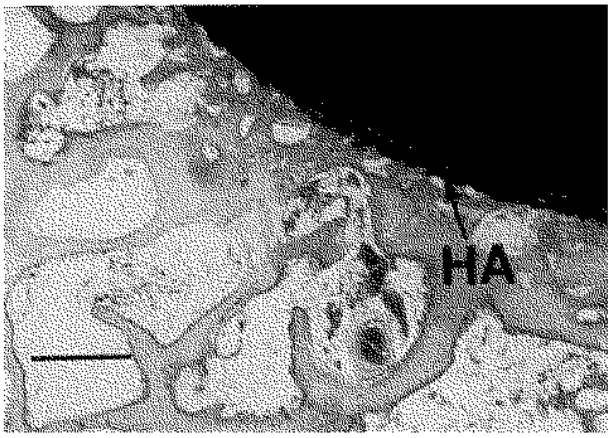

Fig. 2. — Photomicrograph of an HA-coated implant demonstrating bone apposition and mineralization directly onto the hydroxyapatite coating (HA), with no interposed fibrous tissue layer. Stained with basic fuchsin and light green (bar 500 µm).

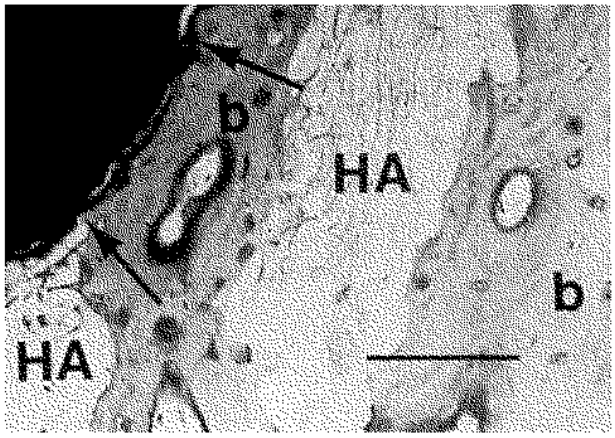

Fig. 3. — Photomicrograph of an HA-coated implant demonstrating bone (b) with a cortical structure and Haversian canals between the metal surface and HA-coating (HA), which had been disrupted at the insertion. Stained with Stevenels blue and van Gieson picrofuchsin (bar 100 µm).

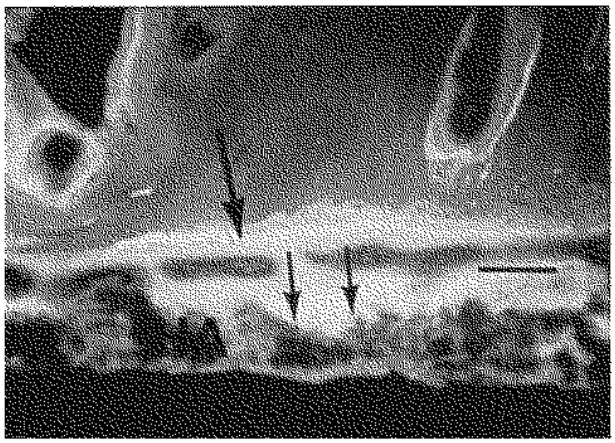

Fig. 4. — Fluorescence photomicrograph of an HA-coated implant showing active bone formation directly onto the hydroxyapatite coating (HA). Tetracycline (small arrows) and calcein green (big arrow) (bar = 100 µm).

DISCUSSION

A transcortical implantation has been used for evaluation of the interface shear strength of new implant materials by a push-out test, in order to measure the bonding strength between the implant and compact cortical bone (7, 10, 13, 27, 28, 29). This does not fully represent the clinical situation in which the bonding between the implant and trabecular bone is the problem. We have therefore evaluated the bone anchorage of impants in trabecular bone (24).

Interface shear strength for HA-coated implants has varied over a wide range in previous investigations, from 6-7 MPa (7, 24, 29) to approximately 50 MPa in transcortical models (10, 14). We found shear strength values much lower than previously reported. This might result from differences in animal species, surgical techniques, or the experimental design and characteristics of the HA-coated implants. Taking these facts into account, we do not think it is feasible to compare push-out results from different centers with each other. For instance the difference from the study of Soballe *et al.* (24) can be explained by application of HA-coating on a rough plasma-sprayed metallic surface. The HA-coatings in our experiments were applied to smooth metal surfaces as in the study of Geesink (10) and de Groot (13).

The maximum shear strength obtained was about 1/50 the strength at the time of manufacture, indicating that the HA-metal bond degrades in vivo, and that the in vivo results are not comparable with in vitro results.

We observed failures occurring at the interface between metal and coating and found no differences between metal cores of titanium or chromium-cobalt alloy. Our results correspond well with Shen *et al.* (23) in that the mechanical failure occurred in 15 out of 18 cases at the interface between the hydroxyapatite coating and the titanium core in a weight-bearing canine model. In contrast numerous reports have described the failure of the HA within the material itself, leaving both the metal-coating and coating-bone interfaces intact (26) or with disruption at the interface between coating and bone (9, 10, 11, 19, 22). Thomas *et al.* (29) observed failures at the metal-coating interface ; however they occurred only at the crests of the grooves on the implants and the follow-up period was 32 weeks.

Our studies indicate deficiencies in the bonding strength of the HA-coating to the metallic implant, especially as failures in this state cannot be detected radiologically during clinical use. The limited clinical experience shows that it might be a potential risk factor.

Several studies have concluded that HA is inert as an implant material and does not show signs of bioresorption (1, 15, 17, 18). However an in vitro study (26) showing that plasma-sprayed HA is susceptible to degradation by interaction with physiological fluids raises doubts about the stability of the metal-coating interface. A case study showed that the HA-coating became fragmented in many places and disappeared in others under infectious conditions (9). Dhert *et al.* (8) reported an experiment where cylindrical HA-coated titanium alloy implants were laced in goats in a non-weight-bearing model. The HA was plasma-sprayed to a coating thickness of 50 µm. Scanning electron microscopic investigation at 12 weeks showed degradation of the HA coating.

Our observations about low adhesive strength between HA coating and metals and reports of other mechanisms of failure lead to misgiving about widespread clinical use of HA-coated im-

plants, and clinical series should be closely followed and reported. HA coating on rough surfaces might, however, be feasible.

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SAMENVATTING

L. NIMB, K. GOTFREDSEN, J. STEEN JENSEN. Mechanische defekten van titanium en cobalt-chroom-molybdeen implantaten, bedekt met hydroxyapatite.

Defekten van de hydroxyapatite (HA) keramieke bedekking van metalen implantaten, kunnen gebeuren bij de plaatsing of tijdens de belasting in vivo. Deze defekten kunnen ook voorkomen na verloop van tijd, door interaktie met de fysiologische vochten.
Een studie op de hond werd uitgevoerd ter histologische en biomechanische evaluatie van cobalt- chroom-molybdeen legering en titanium implantaten, bedekt met hydroxyapatite in een simulator zonder lichaamsbelasting. Twaalf cylindrische pluggen werden in de mediale femurcondyl van 6 honden geïmplanteerd. Zes Cr-Co-Mo-implantaten en 6 Ti-6AI-4V-implantaten waren bedekt met een laag HA van 80 à 120 µm. De implantaten werden na 4 weken verwijderd, samen met het omliggend bot.
Er was geen statistisch verschil tussen de 2 groepen wat de resistentie van de interface betreft. Er was geen verschil tussen de met HA bedekte Cr-Co-Mo en Ti-6AI-4V implantaten. Er werd geregeld een defekt gezien, veroorzaakt door een spatievorming tussen metaal oppervlak en HA bedekking, die met het bot gefixeerd werd. De histologische studie toonde bij de met HA bedekte implantaten een uitgebreide oppervlakte met goed contact bot-implantaat. Deze studie toont desalniettemin aan dat de HA-bedekking op zeer effen metallische oppervlakten niet geschikt is in klinisch gebruik, gezien de zwakke binding tussen metaal en bedekking.

RÉSUMÉ

L. NIMB, K. GOTFREDSEN, J. STEEN JENSEN. Défauts mécaniques des implants en titanium et des implants en alliage cobalt-chrome-molybdène recouverts d'hydroxyapatite. Étude animale.

Des défauts mécaniques du recouvrement d'hydroxyapatite (HA), appliqué sur les implants métalliques peuvent se produire au moment de l'insertion où lors de la charge survenant in vivo. De pareils défauts peuvent également survenir à l'usage, suite à des interactions avec les liquides physiologiques.
Les auteurs ont pratiqué une étude sur le chien afin d'évaluer à l'aide d'un simulateur sans mise en charge le comportement des implants en titanium et des implants en alliage cobalt-chrome-molybdène recouverts d'hydroxyapatite. Douze implants cylindriques furent placés dans le condyle fémoral interne de 6 chiens. Un recouvrement d'HA de 80-120 µm d'épaisseur, fut appliqué sur 6 implants en Cr-Co-Mo et sur 6 implants en Ti-6AI-4V. Après 4 semaines, les implants furent extraits avec le tissu osseux adjacent. On ne nota pas de différences statistiquement significatives entre les 2 groupes quant à la résistance de l'interface. Les revêtements d'HA sur Cr-Co-Mo et sur Ti-6AI-AV, présentaient tous 2 cas une déhiscence entre la surface métallique et le revêtement qui restait fixé à l'os. Une étude histologique des implants tapissés de HA montre un contact étendu entre l'os et l'implant. Cette étude montre néanmoins qu'en clinique, le recouvrement d'une surface polie par HA n'est pas fiable à cause de la faible adhérence du recouvrement au métal.