Surface Observation of Thin Hydroxyapatite-Coated Implants at 80 Months After Insertion

Tetsuya Sugiyama, DDS, PhD 1,2*
Yasuo Miake, DDS, PhD 3
Yasutomo Yajima, DDS, PhD 4
Kohji Yamamoto, DDS, PhD 5
Kaoru Sakurai, DDS, PhD 1

We observed surfaces and cross sections of thin hydroxyapatite (HA)-coated implants produced by the thermal decomposition method in a patient attending our clinic who underwent implant removal at 80 months due to fracture of the implants. On the implant surfaces of the removed sample, most of the HA had dissolved, and extensive osseointegration was observed where Ti had closely bonded to bone. This indicated that the HA coated on the implant surfaces had disappeared and osseointegration had been established where Ti directly bonded to the bone. In addition, calcium titanate (CaTiO₃) and HA layers formed by the thermal decomposition method showed no desorption. The results clearly indicate the positive clinical potential of thin HA-coating by the thermal decomposition method.

Key Words: hydroxyapatite, HA, dental implant, thin HA coating, surface observation

INTRODUCTION

Pure titanium is the standard material for oral implants. Various methods for treating the surface of such implants have been developed with the aim of enhancing bonding between implant and surrounding bone. Since the 1980s, coating the implant with hydroxyapatite (HA) in particular has drawn much interest, as doing greatly enhances biocompatibility, which leads to early stage synostosis by osteoconduction. 1–6 However, the advantages offered by HA remain to be fully exploited due to problems in its application. 7–10 Recently, in order to overcome these problems and obtain the full benefits offered by HA-mediated osteoconduction, a method of coating implants with an HA layer only a few micrometers in thickness has been developed and applied. 11–15

1 Department of Removable Prosthodontics and Gerodontontology, Tokyo Dental College, Chiba, Japan.
2 Department of Dysphagia Rehabilitation and Community Dental Care, Tokyo Dental College, Chiba, Japan.
3 Department of Ultrastructural Science, Tokyo Dental College, Chiba, Japan.
4 Department of Oral and Maxillofacial Implantology, Tokyo Dental College, Chiba, Japan.
5 Department of Removable Prosthodontics (Operative Dentistry), Division of Oral Functional Science and Rehabilitation, Asahi University, School of Dentistry, Gifu, Japan.

* Corresponding author, e-mail: tesugiya@tdc.ac.jp
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In this study, we observed thin HA-coated implants produced by the thermal decomposition method in a patient attending our clinic who underwent implant removal at 80 months due to fracture of the implants. We report our observations and analyses of the surfaces and cross sections of the removed implants.

**MATERIALS AND METHODS**

**Case**

The patient was a 37-year-old man. At initial presentation, his chief complaint was masticatory disorder due to lack of bilateral molars in the mandible. He had no significant systemic disease or local problem, the bone quality of the region of implantation was D2 and D3, and implantation was planned in accordance with the patient's wishes.

On May 24, 2001, 2 thin HA-coated implants (Platon Implant, Bio Type I, Platon Japan, Tokyo, Japan) were inserted to replace the left mandibular first and second molars using a single-stage procedure. On August 29, 2001, a superstructure was attached by cementation. Implant therapy was then conducted to replace the right mandibular first and second molars. After attachment of the superstructure, a good clinical course was observed on regular examination. However, on November 17, 2007, the 2 implants on the left side broke and desorbed along with the superstructure. The implants broke at sites subjected to stresses exerted by the tips of the abutment screws, and no procedures other than implant removal were considered. The implants were removed on February 7, 2008. The surfaces and cross sections of the removed implants were observed with the patient's consent.

**Analysis methods**

One of the removed implants and its surrounding bone was fixed with 10% formalin solution, after which it was dehydrated in an increasing series of ethanol solutions and embedded with polyester resin to obtain a specimen. This specimen was cut across the implantation axis and its surface ground with waterproof grinding paper for use as a sample (removed sample). Reflection images obtained with a scanning electron microscope (SEM) (JSM-6340F, JEOL Ltd, Tokyo, Japan) and Ca and Ti surface analysis images obtained with the electron probe microanalyzer (EPMA) (JXA-8200, JEOL Ltd) were observed at 15 kV of acceleration voltage. An unused implant (control sample) was also observed for comparison with the removed sample.

The other removed implant was fixed on the sample table while unembedded. Next, Pt-Pd sputter coating was conducted for surface protection, and electrical conduction and cross sections were prepared by focused ion beam (FIB) treatment. Sections were observed with a SEM (S-4700, Hitachi, Tokyo, Japan) at 10 kV acceleration voltage. Carbon was deposited on the treated area as a local protective coating to reduce damage caused by gallium ion beam irradiation.

**Results**

Observation of SEM reflection electron images of the removed samples at low magnification revealed close and extensive bonding to bone (Figure 1). Partial magnification revealed that the implant and bone were light and slightly darker gray, respectively, and that the implant had bonded closely to the bone (Figure 2). At this site, percentage of bone-to-implant contact at the calcium titanate (CaTiO$_3$) boundary was 99.1%. On the other hand, in the control sample, a few micrometers of slightly darker gray HA coating was observed on the surface of the light gray implant (Figure 3).

The Ca and Ti surface analysis conducted with the EPMA revealed Ca layers coated with HA on the implant surface in the control
sample (Figure 4). In the removed sample, Ca showed distribution throughout the bone that had bonded to the implant (Figure 5). Ca showed an even distribution, and no increase in Ca density on the Ti surfaces made it impossible to clarify whether the Ca was present in the HA or the bone.

On the other hand, on the implant surfaces of the removed sample, the area where no bone formation had occurred was very small. In the magnified reflection electron images, the implant was light gray, and the area where no bone formation had occurred was dark gray (Figure 6). The EPMA

**Figure 1.** Reflection electron image of removed sample at low magnification: dense bone (Bo) formation was observed around the implant (Ti).

**Figures 2–5. Figure 2.** Reflection electron image of removed sample: implant (Ti) showed close bonding to bone (Bo). **Figure 3.** Reflection electron image of control sample: hydroxyapatite (HA) layers a few micrometers thick were observed on Ti surfaces. **Figure 4.** Ca surface analysis of the control sample by electron probe microanalyzer (same area as in Figure 3). Light area on Ti surfaces is Ca, which indicates HA layers. **Figure 5.** Ca surface analysis of removed sample by electron probe microanalyzer (same area as in Figure 2). Light area on Ti surfaces is Ca, which indicates bone (Bo).
analysis of the same area revealed no Ca distribution on the Ti surfaces of the implant (Figure 7).

When the cross sections of the surfaces of the removed implant treated with FIB were observed at high magnification with a SEM, a slightly darker gray layer considered to be titanium dioxide (TiO$_2$) was observed on the Ti surface layer (Figure 8a), and a molding layer believed to be CaTiO$_3$ was observed on top of that (Figure 8b).

In addition, a light gray Ti surface layer and a slightly darker gray layer considered to be a layer of TiO$_2$ on top of the Ti layer (Figure 9a) were observed around the top of the thread. Another layer believed to be CaTiO$_3$ overlapped these layers (Figure 9b), with a further layer considered to be HA (Figure 9c) on top of that.

**DISCUSSION**

According to numerous studies, root form titanium oral implants, including those developed by Branemark et al,$^{16}$ are highly predictable therapies. One of the drawbacks of implant therapy, however, is the long treatment period required, and a number of methods have been developed to resolve this problem. One method is to modify the titanium surfaces with HA, and many types of implant now include titanium surfaces...
coated with HA, which offers excellent biocompatibility and osteoconduction. However, implants coated with HA by plasma-spraying or flame-spraying suffer from problems related to the thickness of the HA film, uneven film composition, and rough inner structures, all of which can lead to loss of implant due to film breakage or desorption.\textsuperscript{4,6–10,17}

Recently, thin HA coatings of only a few micrometers in thickness have drawn much attention as a potential solution to this problem. In this study, the implants analyzed were coated with CaTiO$_3$ and HA at the relatively low temperature of 600$^\circ$–650$^\circ$C by the thermal decomposition method, which allows high purity and HA layers of 3–5 μm in thickness. Moreover, the double-layered coating of CaTiO$_3$ and HA provides strong adhesive strength.\textsuperscript{11,12,14}

Where no bone formation had occurred on the surfaces of the removed sample, observation by EPMA revealed no Ca distribution in contrast to the control sample. This indicated that the HA layers on the implants had disappeared. Where bone formation had occurred, Ca showed an even distribution and no increase in density on the titanium surface. This indicated the disappearance of HA from the Ti surfaces in the overall implant, suggesting a transition from biointegration to osseointegration.

Observation of the FIB-treated samples at high magnification revealed bonding of CaTiO$_3$ layers to the TiO$_2$ layers on the Ti surface layer, and layers considered to be HA were also partially observed. This indicated that, as reported by Kawamura et al,\textsuperscript{14} CaTiO$_3$ layers and HA layers formed by the thermal decomposition method are thin with excellent adhesion, making desorption and breaking of the coating layers rare.

In thin HA-coated implants at 80 months after placement, most of the HA had dissolved, and osseointegration with close and extensive bonding of Ti was observed. In addition, CaTiO$_3$ layers and HA layers formed by the thermal decomposition method showed no desorption, indicating the usefulness of the thin HA-coating method.

**ABBREVIATIONS**

CaTiO$_3$: calcium titanate  
EPMA: electron probe microanalyzer  
FIB: focused ion beam  
HA: hydroxyapatite  
SEM: scanning electron microscope  
TiO$_2$: titanium dioxide

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**REFERENCES**


