SURFACE MODIFICATION OF PURE TITANIUM
BY HYDROXYAPATITE IMPLANTATION

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Abstract Fixation of orthopedic titanium implants to living bone is one of keys to achieve a successful result but still developing. Enhancement of bone formation around the implants can make a considerable contribution to long-term stable fixation. In this study, spherical HAP ceramics (φ 400 – 500 μm) were implanted in the surface of cylinder shape pure titanium by the loading up to 1 kN at 1173 K to enhance bone formation around the implant. By this method, the spherical HAP ceramics were mechanically held on the surface of the pure titanium.

INTRODUCTION

Fixation of orthopedic titanium implants, such as artificial tooth roots, to living bone is one of keys to achieve a successful result but still developing. Enhancement of bone formation around the implants can make a considerable contribution to long-term stable fixation. A practical way to stimulate the bone formation around the implants is plasma spray coating of hydroxyapatite (Ca_{10}(PO_{4})_{6}(OH)_{2}, HAP) that is a main inorganic component of bone and teeth and a widely accepted material as bone
grafts.\textsuperscript{8-12} However, plasma-sprayed HAP coating is a kind of ceramics thin layer with brittleness, meaning an anxiety for loosening of the implants, although plasma-sprayed HAP coating contributes to initial fixation of the implants.\textsuperscript{5, 13, 14} Therefore, establishment of a HAP fixation on the implants with long-term stability has been a deep interest.

We studded bioactive spots on the surface of the implant to achieve stable fixation. First, spray-dry-formed HAP granules (\(\phi 32 - 38 \mu m\)) were implanted on a superplastic titanium alloy (Ti-4.5Al-3V-2Fe-2Mo) to obtain a Ti alloy - HAP hybrid that has both sufficient mechanical strength and bone affinity.\textsuperscript{15, 16} This method suggested the possibility of the HAP implantation for the surface modification of orthopedic implants. However, aluminum, vanadium and molybdenum in the alloy are unwilling elements. Furthermore, crystallinity of the spray-dry-formed HAP granules is insufficient for long-term stability in the body. In this study, well-sintered spherical HAP ceramics with approximate diameter of 400 - 500 \(\mu m\) were implanted in the surface of cylinder shape pure titanium for the sake of dental implants use.

**EXPERIMENTAL**

Pure titanium rod (\(\phi 3 \mu m\), ASTMB348-GR2) was cut into a length of 19 mm for the substrate. Eight locating center punches were grooved on the surface of the substrate with a \(\phi 300\) drill at every 2 mm to keep the HAP ceramics pieces at regular intervals.

HAP ceramics were sintered at 1343 K, then quenched to break into small pieces by a thermal shock. As broken HAP pieces were polished into spherical shape by No. 400 diamond polishing sheets to secure HAP ceramics pieces during the implantation. The spherical HAP ceramics were screened into \(\phi 400 - 500 \mu m\).
The implantation was performed by a hot press with a vacuum furnace (JTT, Japan). The substrate with the spherical HAP ceramics adhered on the grooves with HP-500 vacuum grease (Dow Corning Asia, Japan) was set between upper and lower silicon nitride ceramics dice, then set between unconfined compression stage in the vacuum furnace (Fig. 1). After the evacuation of the vacuum furnace up to 0.4 Pa, the vacuum furnace was heated up to 1173 K at a heating rate of 573 K/h. At 1173 K, the dice with the substrate was loaded at 0.05 kN/min up to 0.25 kN and held for 5 minutes, and then loaded at 0.075 kN/min up to 1 kN and held for 5 minutes. The HAP-implanted substrate was washed by ultrasonic washing machine for 5 minutes.

The surface and the cross section of the HAP-implanted substrate were observed by a light microscope. Cross section of the HAP-implanted substrate was prepared by a low speed diamond saw, then polished by diamond paste.

FIGURE 1. Schematic diagram of the implantation.
RESULTS AND DISCUSSION

Eight spherical HAP ceramics were successfully implanted in the surface of the substrate by the loading up to 1 kN at 1173 K (Fig. 2). The implanted spherical HAP ceramics returned to its original color by the 5-minute ultra sonic washing. The spherical HAP ceramics were slightly exceeded the periphery of the substrate, providing the surface of the substrate with HAP spots (Fig. 3). In like manner as the case of spherical HAP implantation in the flat surface of pure titanium\textsuperscript{15, 16, 17}, only infinitesimal titanium moved over the upper hemisphere of the spherical HAP ceramics. Therefore, major HAP-locking factor was considered to be friction between the spherical HAP ceramics and the substrate.

![Figure 2](image1.png)

**FIGURE 2.** The HAP-implanted pure titanium rod.

![Figure 3](image2.png)

**FIGURE 3.** The cross section of the implanted spherical HAP ceramics.

Shaping HAP ceramics into spherical shape enables the HAP ceramics to be screened in narrow size distribution. Spherical HAP ceramics in narrow size distribution could contribute to prevent stress concentration on a certain spherical HAP ceramics, resulting in preventing cracks in the spherical HAP ceramics.

Pure titanium below the spherical HAP ceramics was deformed along an external shape of the spherical HAP ceramics without cracks and major gaps between the substrate and implanted spherical HAP ceramics. In like manner as HAP coatings, even well-sintered HAP ceramics will gradually dissolves as the time...
proceeds, resulting in providing the implants with cavities having a spherical bottom. Because cavities of 400 - 500 μm are reportedly suitable to induce bone tissue ingrowth, the cavities made by the implantation are expected to induce the ingrowth that can function as microanchoring of the implant.

The HAP-implanted pure titanium implant is considered to have positive prospects as orthopedic and dental implants. The implanted HAP ceramics are expected to bond to bone much faster than pure titanium, resulting in faster initial fixation of the implant. Furthermore, each implanted spherical HAP ceramics are independent, meaning that some dropouts or cracks of the implanted spherical HAP ceramics will not propagate across the surface of the implant.

SUMMARY

The present study showed a method to provide titanium implants with bioactive spots for the sake of dental implant use. Eight spherical HAP ceramics with diameter of φ 400 – 500 μm were successfully implanted in the surface of cylinder shape pure titanium by the loading up to 1 kN at 1173 K. Damages on the HAP ceramics will not suffer the fixation of the implant, because each implanted spherical HAP ceramics are independent. The cavities made by the implantation are considered to establish microanchoring of the implant by bone tissue ingrowth that contributes to long-term stable fixation of the implants.

REFERENCES


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