The Interfacial Strength in Sputtering-Hydroxyapatite-Coating Implants with Arc-Deposited Surface

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ABSTRACT. Titanium columns (Ti-6A1-4V) were treated with arc-deposition to roughen the surface enough to anchor the bone, and then coated with hydroxyapatite (HA) at a thickness of 5 µm by the sputtering technique. Columns were implanted into dog femurs, and fixation of columns to bone due to bone-ingrowth was assessed histologically and with the push-out test. The HA-coated columns were inserted in the shafts of the right femurs of 2 dogs. As a control, columns that were only arc-deposited (non-coated columns) were inserted into the left femurs. The interfacial strength was higher for the HA-coated columns than for the non-coated columns. Coating a rough surface with an HA layer using a sputtering technique reinforces interfacial strength between bone and implants.

KEY WORDS: arc-deposition, hydroxyapatite, sputtering.

NOTE

Surgery

Total hip replacement (THR) is a highly effective procedure for restoring function in diseased canine coxofemoral joints [2]. The prosthesis can be cemented or cementless, with cemented THR being the more frequently performed procedure. However, complications have been observed after cemented THR, including formation of fibrous tissue at the surface of the bone, breakage of the cement, tissue disorders due to exothermic repolymerization of the polymethylmethacrylate (PMMA), and infection. To avoid such complications, cementless prostheses have been developed for use in humans.

Recently, hydroxyapatite (HA) has been used to coat surfaces of cementless prostheses in order to promote primary fixation. Favorable findings of primary bone-ingrowth have been reported for HA-coated prostheses, and clinical applications have been reported [1, 3, 9].

The sputtering technique is a useful method for preparation of a homogenous and dense thin film less than 1 µm thickness. In recent years, it has been used for coating HA on metals such as titanium in the biomaterials field [10]. The role of HA should only be to accelerate bone-ingrowth into the porous layer, whereas bone anchoring should be the mechanism of long-term fixation [4].

In the present study, titanium columns (Ti-6A1-4V) were treated with arc-deposition to roughen the surface enough to anchor the bone, and then coated with HA at a thickness of 5 µm using a sputtering technique. Columns were implanted into dog femurs, and fixation of columns to the bone due to bone-ingrowth was assessed histologically and with the push-out test.

Sixteen titanium columns (Ti-6A1-4V; φ4.2-mm, 10 mm) were treated with arc-deposition of titanium to make a rough surface. Eight of these columns were coated with HA (thickness, 5 µm) using a sputtering technique by glow discharge, and the remaining 8 were used as control columns (arc-deposition only) [7, 12]. The 8 HA-coated columns were inserted into the shafts of the right femurs of 2 dogs (4 per dog), and the 8 control columns were inserted into the left femurs (4 per dog).

Two Siberian husky dogs aged 6 and 7 years, respectively, which were raised in the kennel as experimental animals were used. These dogs were cared for according to the principles of the ‘Guide for the Care and Use of Laboratory Animals’ prepared by Rakuno Gakuen University. Animals were sedated intravenously using a mixture containing 0.025 mg/kg butorphanol tartrate (Stadol, BMS, Tokyo, Japan), 10 µg/kg medetomidine (Domitor, Meiji Seika Co., Tokyo, Japan) and 150 mg/kg midazolam (Dormicum, Yamanouchi Pharmaceutical Co., Tokyo, Japan), and anesthetized intravenously with 5 mg/kg ketamine (Ketalar, Sankyo Co., Tokyo, Japan). Anesthesia was maintained with N2O-O2-sevoflurane (Sevofrane, Maruishi Pharmaceutical Co., Osaka, Japan). The hindlimbs were washed, shaved, and disinfected with povidone-iodine. A longitudinal incision was made on the surface of right and left femora. Four holes were drilled in the shaft of the femur at intervals of 2 cm using a φ4.8-mm drill bit. Columns were implanted in the holes perpendicular to the bone surface. After implantation of the columns, the soft tissues were closed in separate layers using polydioxanone. After surgery, 22 mg/kg cefazoline sodium (Cefamezin, Fujisawa Co., Osaka, Japan) and 10 mg/kg aspirin (Bufferin, Lion Co., Tokyo, Japan) were prescribed twice daily for 1 week. Euthanasia was performed 4 and 8 weeks after surgery by barbiturate overdose. Columns were extracted from 1 dog 4 weeks after surgery, and from the remaining dog 8 weeks after surgery. Each femur with columns in situ was removed and wrapped in saline-moistened gauze. For the push-out test [10], the distal three columns were formed into
half-cylindrical portions by cutting the cortical bone perpendicular to the cut surface. The columns were pushed out at a crosshead speed of 0.5 mm/min using an Instron universal testing instrument model 1130 (Instron Co., Canton, U.S.A.). The results of push-out tests are shown as the average of shear strength of the each column. Then, after the push-out test, scanning electron microscope (SEM; DS130, Akashi Co., Kobe, Japan) and energy dispersive x-ray spectrometry (EDAX; PV-9100), EDAX Inc., Mahwah, U.S.A.) were performed.

The most proximal column was observed under an optical microscope, and the bone apposition rate around the column was measured to determine the index of bone-ingrowth. The result of bone apposition rate was shown from the most proximal column, in each femoral bone.

Shear strength was higher at 8 weeks than at 4 weeks, for both types of column. Also, shear strength was higher for HA-coated columns than for non-coated columns (Fig. 1). Optical microscopic images of non-coated columns were compared with those of HA-coated columns (4 weeks, Fig. 2; 8 weeks, Fig. 3). Images of non-coated columns at 4 weeks showed an obvious gap between implant and bone and little bone-ingrowth. Images of HA-coated columns at 4 weeks also showed a gap between column and bone, but the gap was narrower than that of the non-coated columns, and bone-ingrowth was observed in which the column was in partial contact with the bone. Images of non-coated columns at 8 weeks showed that the column was in contact with bone, but a gap between bone and column was still obvious. Images of HA columns at 8 weeks showed that the porous area had been filled by bone-ingrowth. The percentages of bone-ingrowth into columns were 52% and 83%, respectively.

Thus, the bone apposition rate of HA-coated columns was 1.5 times that of non-coated columns at 8 weeks.

Following the push-out test, SEM and EDAX were performed on the HA-coated columns. The results at 4 weeks and 8 weeks are shown in Fig. 5 and Fig 6, with EDAX results in the upper panel and SEM results in the lower panel. At 4 weeks, a gap was observed between the HA coating and the column, and the HA coating was uniformly distributed on the bone surface. At 8 weeks, the HA coating had disappeared.

HA coating produces high bone conductivity, and is a method of surface treatment used to promote fixation. However, the bonding of the HA-titanium interface is very low, and it is therefore thought that HA can peel off implants and cause loosening of prostheses [13]. To overcome this prob-
lem, it was proposed that the surface of the implant should be treated to make it porous before coating it with HA. However, HA coating of a titanium surface that is covered with titanium beads or fiber mesh plugs up the spaces in these surfaces, which are meant to promote bone-ingrowth, even though HA coats only the surface of such structures and does not penetrate to the inner layers [6, 11]. It was reported that HA coating could prevent bone-ingrowth to the inside of such porous structures [4, 5, 8].

It was shown that arc-deposited surfaces do not experience problems such as plugging, non-uniformity, voids and cracking in the titanium surface when coated with HA [4]. Arc-deposition (which produces a macrostructure component surface) not only improves resistance to shear forces experienced by the acetabular component and increases initial stability, but also provides channels for bone-ingrowth [7]. Sputtering techniques for applying a titanium coating are currently being studied, because this technique may help overcome problems associated with plasma spraying. It is easy to control the thickness of HA layers using sputtering techniques, and very thin layers with a thickness about 2 to 5 µm can be applied. Furthermore, sputtering techniques maintain the roughness of the porous surface and are thought to be less likely to cause cracking of the HA layer.

In the present study, we examined the results of using implants consisting of a Ti-6Al-4V columns treated by titanium arc-deposition for maintaining a rough surface and coated by a sputtering technique with 5 µm of HA. In the push-out test, HA-coated columns had a higher interfacial strength than non-coated columns. Histologically, a gap was observed between the non-coated column and bone at 8 weeks, with a bone apposition rate of about 50%. In contrast, the HA-coated column exhibited much bone-ingrowth into the rough surface at 8 weeks, with a bone apposition rate of 80%. These findings were confirmed all around the column.

Hayashi et al. reported that the gap between the implant and the HA layer is a contributing factor in loosening of the implant in the long term [5]. In the present study, the results of the push-out test indicated that there was a gap between the HA layer and the column at 4 weeks. The high affinity of HA for bone is a factor in separation of HA from the column, as are the strength of binding of HA to titanium and the degree of rough surface of the titanium. However, we found
that the thin layer of HA was uniformly distributed over the surface of the bone, which indicates that the rough surface was not greatly reduced by application of a thin HA layer. At 8 weeks, the HA had disappeared completely, most likely because of resorption of the HA into the bone.

Together with previous findings [4, 5], the present results suggest that a 5 μm layer of HA applied to a metal implant with a porous surface might promote early bone-ingrowth and increase initial interfacial strength. At 8 weeks, the 5 μm HA layer had dissolved completely, and there was no decrease in interfacial strength, as measured with the push-out test. This indicates that separation of HA did not decrease interfacial strength, and that HA increased bone conductivity of the surface of the columns. It seems plausible that the coating of a porous implant surface with HA by the sputtering technique can reinforce interfacial strength between bone and implant, as suggested by the good bone-ingrowth on the rough surface that was maintained on the titanium treated with arc-deposition. It appears that the ultra-thin HA coating prevents separation of HA from the titanium and enables the bone to anchor into the rough surface immediately after the HA layer dissolves. Arc-deposition for titanium and the HA sputtering technique may be useful for bone-ingrowth to materials with a porous surface.

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REFERENCES