

Early bone apposition in vivo on plasma-sprayed and electrochemically deposited hydroxyapatite coatings on titanium alloy

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Abstract

Three different implants, bare Ti–6Al–4V alloy, Ti–6Al–4V alloy coated with plasma-sprayed hydroxyapatite (PSHA), and Ti–6Al–4V alloy coated with electrochemically deposited hydroxyapatite (EDHA), were implanted into canine trabecular bone for 6 h, 7, and 14 days, respectively. Environmental scanning electron microscopy study showed that PSHA coatings had higher bone apposition ratios than those exhibited by bare Ti–6Al–4V and EDHA coatings after 7 days; however, at 14 days after implantation, EDHA and PSHA coatings exhibited similar bone apposition ratios, much higher than that for bare Ti–6Al–4V. The ultrastructure of the bone/implant interface observed by transmission electron microscope showed that the earliest mineralization (6 h–7 days) was in the form of nano-ribbon cluster mineral deposits with a Ca/P atomic ratio lower than that of hydroxyapatite. Later-stage mineralization (7–14 days) resulted in bone-like tissue with the characteristic templating of self-assembled collagen fibrils by HA platelets. Though adhesion of EDHA coatings to Ti–6Al–4V substrate proved problematical and clearly needs to be addressed through appropriate manipulation of electrodeposition parameters, the finely textured microstructure of EDHA coatings appears to provide significant advantage for the integration of mineralized bone tissue into the coatings.

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1. Introduction

Since their introduction in the 1980s, hydroxyapatite (HA) coatings on orthopedic implants have gained wide acceptance in orthopedic surgery [1]. It has been repeatedly demonstrated clinically that HA coatings have osteoconductive properties, and that the fixation of HA-coated implants is better than for uncoated implants following optimal surgical conditions [2]. Laboratory research also supports the conclusion that the early bone growth and apposition are accelerated by implants coated with HA [3].

Plasma-spraying is still the most popular technology commercially used for depositing HA coatings onto

titanium-base and other metal-base implants. However, plasma spraying is a high-temperature and line-of-sight process. Potential problems with this technology include exposure of substrates to intense heat, residual thermal stresses in coatings, and the inability to coat complex shapes with internal cavities [4]. Many other techniques have been explored to address these problems [5], including ion-beam deposition [6], chemical deposition [4], metallo-organic chemical vapor deposition [7], derivation from sol-gels [8], pulsed laser deposition [9], and electrophoresis [10].

Electrochemical deposition is one of the most promising new processes [11,12]. Compared to plasma spraying, the advantages of electrochemical deposition include good control of composition and structure of the coatings, relatively low processing temperatures that enable

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formation of highly crystalline deposits with low residual stresses, and the ability to deposit on non-line-of-site, porous or complex surfaces. The structure of the coating can be controlled by changing the composition, pH and temperature of the electrolyte, as well as the applied potential or current density. Various calcium phosphate coatings, including carbonated apatite [12], brushite [13], octacalcium phosphate [14,15], and HA [16,17], have been successfully applied to titanium-base and other alloys by electrochemical deposition.

The earliest stages of mineralization (3 h to 14 days) of new bone forming on plasma-sprayed HA (PSHA) coatings have extensively been studied at MIT by transmission electron microscopy (TEM) [18]. These studies showed that new bone formation on implants was accelerated in the presence of PSHA coatings, the rate of earliest-stage bone formation being influenced by the solubilities of the PSHA coatings. Coatings with higher crystallinity, which exhibited lower solubility *in vitro* and would be expected to have lower solubility *in vivo*, exhibited delayed formation of new bone compared to less crystalline coatings with higher solubility. Electrochemically deposited HA (EDHA) coatings exhibit deposition morphologies and crystallinities substantially distinct from those of PSHA. Therefore, the study of early bone formation on EDHA coatings can provide an additional perspective from which to understand the underlying mechanism of new bone formation, as well as an assessment of the relative osteoconductivity of EDHA coatings.

In this study, bare Ti–6Al–4V alloy (nominal composition in wt% alloy additions), Ti–6Al–4V coated with PSHA, and Ti–6Al–4V coated with EDHA were implanted into canine trabecular bone for 6 h, 7 and 14 days, respectively, in order to study the initial bone formation on these implant materials. The new bone apposition ratios at different time points were evaluated using environmental scanning electron microscopy (ESEM). The ultrastructures developing at the interface between host bone and HA coatings were studied by TEM.

2. Materials and methods

2.1. Materials

A Ti–6Al–4V ELI grade (ASTM F136-92) rod, 4.76 mm in diameter and 1.83 m long, was purchased from Titanium Industries, Inc. (Parsippany, NJ). This rod was machined into 10.0 mm-long sample rods. Thirty rods were plasma sprayed with HA by Bio-Coat, Inc. (Southfield, MI), in their entirety except for 2 mm at one end which was left uncoated. Thirty rods were electrochemically deposited with HA entirely. Thirty rods were left uncoated, presenting the as-received surface. All 90 rods were sealed in sterilization bags separately, sterilized by ethylene oxide for 2 h, and then left to aerate in laboratory air for at least 2 days before implantation.

Prior to electrodeposition, the exposed alloy surfaces were mechanically ground on 1000-grit SiC paper, followed by 30 min ball milling with 1 μ m alumina powder. The alloy rods were then cleaned ultrasonically in acetone. Electrodeposition was carried out in a standard three-electrode cell in which a platinum foil was used as the auxiliary (counter) electrode

and a saturated calomel electrode (SCE) as the reference electrode. The electrolyte was prepared by dissolving 0.61 mM $\text{Ca}(\text{NO}_3)_2$ and 0.36 mM $\text{NH}_4\text{H}_2\text{PO}_4$, both AR-grade from Merck (Darmstadt, Germany), in Millipore water (Milli-DI™, resistivity > 1 M Ω cm; Millipore Corporation, Billerica, MA). The acidity was measured using an InoLab pH/Oxi Level 3 meter (WTW GmbH, Weilheim, Germany) and adjusted to pH 6.0, so that the electrolyte was saturated with calcium and phosphate ions. During the electrodeposition process, CO_2 -free nitrogen gas (99.999% purity) was continuously purged into the electrolyte to minimize the risk of contamination of the deposits with carbonates. In addition, continuous stirring was carried out using a magnetic bar. A Lauda GmbH (Lauda-Königshofen, Germany) Ecoline model E-220T thermostatic bath was used to maintain a constant temperature of 85 ± 0.1 °C. An EG&G/PAR (Princeton, NJ) model 263A potentiostat/galvanostat operating in potentiostatic mode was employed to maintain the cathode potential at -1.4 V vs. SCE for 2 h.

The near-surface phase composition of the different implant sample types was studied by powder X-ray diffractometry (Rigaku model RU-200 diffractometer with 18-kW Cu rotating anode source, Rigaku Corporation, Tokyo, Japan). The surface morphology was studied by ESEM (Philips model XL30, FEI/Philips, Hillsboro, OR). The surface roughness was measured by a surface profilometer (Tencor model P-10, Tencor Inc., San Jose, CA). The solubilities of the coatings were assessed by immersing one sample from each type in 20 mL of distilled water at room temperature (nominally 25 °C). At various immersion periods of one, 24, 48, 72, 216 and 240 h, 1 mL of solution was removed and replaced by 1 mL of fresh distilled water. The volume removed was further diluted to 10 mL by distilled water, and the calcium concentration was measured by a direct current plasma atomic emission spectrometer (DCP-AES, SpectraSpan model III A, Applied Research Laboratories Inc., Valencia, CA).

2.2. Surgery

Samples were implanted into canine trabecular bone for three different periods, as follows, before sacrificing the animal for further analysis: 6 h, 7 and 14 days, respectively. For each period, seven PSHA-coated samples, seven EDHA-coated samples, and six Ti–6Al–4V bare samples were available for TEM/ESEM studies, thus summing to a total of 60 implant samples.

Animal protocols followed were those specifically approved by the institutional animal care and use committee (IACUC) of the Veterans Administration (VA) Hospital in Boston. All samples were implanted in the proximal and distal femora and proximal tibiae of the back legs of adult mongrel dogs, each weighing about 30 kg. A pilot study established that the cancellous bone at these three implant sites was of comparable density. Therefore, the data were not stratified on the basis of implant site or by animal [18].

2.3. ESEM

After sacrifice, tissues intended for ESEM examination were placed in 0.1 M sodium cacodylate-buffered 4% paraformaldehyde and 2% glutaraldehyde solution. Tissue around the rods was trimmed to about 1–2 mm thick. The trimmed samples were fixed in the same buffered solution for 24 h under partial vacuum at 4 °C. Next, the samples were washed in 0.1 M sodium cacodylate buffer solution twice for 10 min in order to remove the fixatives, then stored overnight in the same solution at 4 °C. The samples were subsequently fixed in 1% osmium tetroxide aqueous solution at 4 °C for 2 h. After rinsing in de-ionized water, the samples were dehydrated through a series of 50%, 75%, 95% and 100% ethanol aqueous solutions for 15 min each and then a final 15 min in 100% ethanol. The samples were agitated in 1:3, 1:2, 1:1 and 2:1 Spurr's resin (Ted Pella Inc., Redding, CA) dissolved in ethanol for 24 h each and then vacuum-infiltrated with pure Spurr's resin for 24 h. They were then embedded in fresh Spurr's resin for 24 h at 60 °C. The embedded samples were cut into 0.5 mm-thick slices with a slow-speed diamond saw. All slices were ground on 1200-grit SiC

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