



Electrodeposition of porous hydroxyapatite/calcium silicate composite coating on titanium for biomedical applications



Yong Huang^a, Shuguang Han^a, Xiaofeng Pang^{a,b,*}, Qionqion Ding^a, Yajing Yan^a

^a Institute of Life Science and Technology, University of Electronic Science and Technology of China, Chengdu 610054, China

^b International Centre for Materials Physics, Chinese Academy of Science, Shenyang 110015, China

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ABSTRACT

A novel method of electrolytic porous hydroxyapatite/calcium silicate (HA/CaSiO₃) composite coating was conducted on pure titanium in a mixed solution of nano-SiO₂, Ca(NO₃)₂ and NH₄H₂PO₄. SEM observation showed that the composite layer was porous, thereby providing abundant sites for the osteoblast adhesion. XRD results showed that the composite coating was mainly composed of HA and CaSiO₃. Bond strength testing exhibited that HA-CaSiO₃/Ti had higher bond strength than HA/Ti. The HA/CaSiO₃ coating was more corrosion resistant than the HA coating based on the polarization tests. In vitro cell experiments demonstrated that both the HA and HA/CaSiO₃ coatings showed better cell response than the bared titanium. In addition, the proliferation of MC3T3-E1 osteoblast cells grown on the HA/CaSiO₃ coating were remarkably higher than those on the bared Ti and pure HA coating.

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1. Introduction

Metallic implants such as medical-grade titanium in the dental and orthopaedic prostheses are gaining distinct superiority in the recent years because of their superior mechanical features that match with the human bone, their corrosion resistance and biocompatibility [1]. To ensure their long-term clinical success, metallic implants are often coated with osteoconductive or/and osteointegrating biomaterials such as hydroxyapatite (HA) ceramics [1–3]. It has been established that HA is one of the most widely used bioactive ceramics in the surgical repair of hard tissue trauma and disease [1–4]. HA has been successfully applied clinically in a series of surgical specialties, namely, bone substitute materials in bony defects repair in orthopaedic surgery, sinus occlusion and ossicular chain reconstruction in otolaryngological surgery, as well as craniofacial augmentation in plastic surgery [4].

However, HA shows a relatively slow rate of osseointegration in vivo [5]. Silicon (Si) is considered to be necessary for normal cartilage and bone growth [6]. Recently, Si element was doped into HA ceramic by different methods to further promote osseointegration further [7]. Furthermore, calcium silicate (CS; CaSiO₃) ceramic shows good biocompatibility, osteoconductivity, and degradability

[8]. Some studies have shown concluded that CS ceramic accelerates the proliferation and differentiation of osteoblast cells compared with brushite ceramics [9]. However, CS rapidly degrades in vivo and in vitro [8], which restricts its clinical application as a bone replacement. Therefore, to enhance the cytocompatibility and corrosion resistance further, a CS and HA (HA/CS) composite coating has been proposed for clinical applications. The HA/CS composite coating can improve the integration of implant materials and body tissue. The superiority of HA/CS is significant because current implant devices, such as artificial hips/knees, spines, and so on face challenge of dislocation, which is partially caused by poor integration of the device with body tissue [10]. The coating technique of cathodic electrodeposition (CED) recently has attracted considerable attention [3,11].

To take advantage of the CED technique and the benefits of porous HA/CS film, a new approach to incorporate CS into the HA has been developed. In this work, porous HA/CS was electrochemically coated onto pure titanium (Ti) substrate. The structural, mechanical, and electrochemical behaviour of the coatings was studied. Moreover, osteoblast adhesion and proliferation on the HA/CS composite-coated Ti substrate were elementarily evaluated quantitatively to assess its biocompatibility.

2. Materials and methods

Ti substrate was preprocessed according to our previous report [12]. Subsequently, the samples were immersed and stirred in a solution of 5 M NaOH at 65 °C for approximately 12 h, washed with

* Corresponding author at: Institute of Life Science and Technology, University of Electronic Science and Technology of China, No. 4 of Section 2, Jianshe North Road, Chengdu, Sichuan 610054, China. Tel.: +86 28 83202595; fax: +86 28 83202595.

E-mail address: pxf2012@yahoo.com.cn (X. Pang).

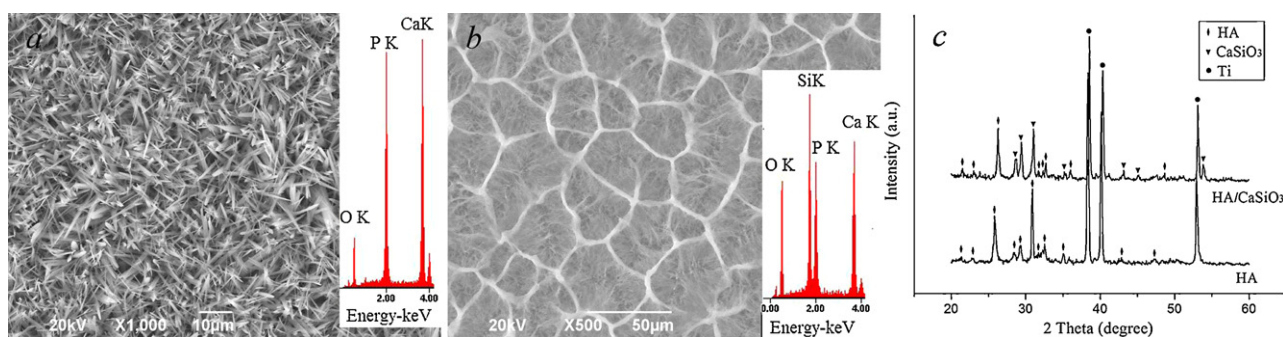


Fig. 1. (a) SEM image of the HA film; (b) SEM image of the HA/CS film; (c) XRD patterns of HA coating and HA/CS coating on titanium.

distilled water, and then dried in air. CED was carried out using an electrochemical workstation (LK2005A, China). The NaOH pretreated Ti sheets were used as the working electrode, a platinum foil was the auxiliary electrode, and a saturated calomel electrode (SCE) was the reference electrode. The HA/CS coating was conducted in an electrolyte composed of 1 g/l nano-SiO₂ (typical size $\approx 15 \pm 5$ nm), 0.042 M Ca(NO₃)₂, and 0.025 M NH₄H₂PO₄ under the following conditions: pH 4.2, 0.8 mA/cm² current density, 30 min, and 65 °C. Pure HA coating was designated as a control. After deposition, the samples were annealed at 300 °C for 2 h in a vacuum environment.

Microstructural characterization of the deposit was performed by X-ray diffraction (XRD; BEDE D1 SYSTEM) with Cu K α radiation (35 kV, 30 mA) at a scanning speed of 1 °/min. The morphology was identified by scanning electron microscopy (SEM; JSM-6490LV). An attached energy dispersive X-ray spectroscopy system (EDS) system (GENESIS 2000 XMS) was used to analyse the chemical composition of the deposit. An electronic universal testing machine (INSTRON-5567) was used to measure the bond strength (BS) between the apatite film and substrate according to the ASTM F1044-05 international standard [12,13]. Tensile tests were carried out at a crosshead speed of 1 mm/min. For each testing material, six parallel samples with an average value were used to report the BS date. After the BS test, cross-sectional examination of the coatings was performed using the SEM-EDS system.

Potentiodynamic polarization tests were conducted using an electrochemical workstation (LK2005A, China). The working cell was a classical three-electrode cell, in which a platinum foil was the counter (auxiliary) electrode, a saturated calomel electrode (SCE) was the electrode, and the uncoated or coated samples (exposed surface area of 1 cm²) were the working electrode. The polarization curves in artificial saliva were obtained at a scan rate of 10 mV/s. The artificial saliva was composed of the following [12]: NaCl (400 mg/l), KCl (400 mg/l), CaCl₂·2H₂O (795 mg/l), NaH₂PO₄·H₂O (690 mg/l), KSCN (300 mg/l) and Na₂S·9H₂O (5 mg/l). Lactic acid was added to achieve a pH of 5. The samples were immersed in the artificial saliva for 20 min to obtain a stable open circuit potential.

The cell responses to the films were evaluated in terms of the cell proliferation. The MC3T3-E1 osteoblast-like cell line was inoculated onto each sample for 1 and 7 days at a cell density of 5×10^4 cells ml⁻¹. Bared Ti substrate was also investigated for comparison. After incubation, the cells were separated with a trypsin-EDTA solution, and the living cells were calculated using a haemocytometer. To determine the cell morphology, cells were cleaned with PBS three times to eliminate nonadherent cells, and then treated by a 2 h fixation in 2.5% (w/v) glutaraldehyde, gradual dehydration in 40–100 vol.% alcohol, and critical point drying in CO₂. The specimens were sputter-coated with gold and examined with SEM.

All data were recorded as the mean \pm standard deviations. Significant differences were analysed by one-way (ANOVA) and

subsequently calculated by the Student–Newman–Keuls post-hoc test, and $p < 0.05$ was considered significant [12].

3. Results and discussions

XRD was conducted on the coating to identify its phase constitution, as shown in Fig. 1c. The XRD result indicated that the HA/CS coating was predominantly composed of HA (JCPDS file card #9-432) and CaSiO₃ (JCPDS file card #72-1396). The representative surface morphology of the films is shown in Fig. 1a and b. Both the HA and HA/CS films formed uniform layers on the Ti substrate without cracking or shedding on the surface. The SEM microstructure revealed that the HA/CS coating had a porous structure (interconnected network-type structure) whereas the HA coating had a relatively dense surface structure (needle/rod-like crystals). The pore size distributions ranged from 20 μ m to 50 μ m (Fig. 1b). The bone regeneration rate for the porous HA was depended on the porosity [14]. The porous structure of the HA/CS coating may be benefit to the initiation of bone formation, providing abundant sites for the growth of osseous tissue [14].

EDS analysis conducted on the coating (in Fig. 1b), demonstrated that the HA/CS surface was mainly composed of O, P, Ca and Si, and the Si content was 7.77 wt.% \pm 0.39 wt.%. The generation mechanism of the electrodeposited HA can be summarised as a series of electrochemical and chemical reactions according to a previous study [12]. Meanwhile, the formation process of CaSiO₃ may be expressed as follows:

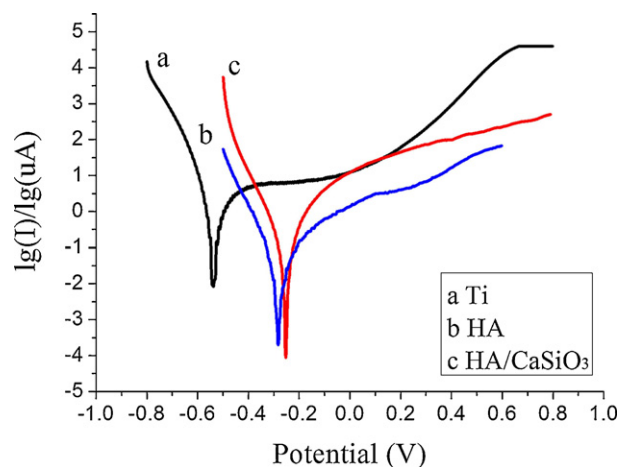
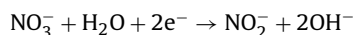


Fig. 2. The polarization curves of the bared Ti and the coating covered Ti in artificial saliva.

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