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Metal release from ceramic coatings for dental implants

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ABSTRACT

Objectives. Two types of ceramic coatings on commercially pure titanium for dental implant applications with different Ca/P ratios in the range from 1.5 to 4.0, and two different thicknesses (~5 and ~15 μm) were examined with the aim of underpinning the effect of coating composition, thickness and microstructure on the corrosion behavior and hydroxyapatite forming ability in SBF.

Methods. Bioactive coatings were formed on Ti by plasma electrolytic oxidation (PEO). The composition, structure, and morphology of the materials were characterized before and after the immersion in simulated body fluid solution (SBF) at 37 °C for up to 4 weeks. All the materials were screened with respect to metal ion release into SBF.

Results. Only thick PEO coating with overstoichiometric Ca/P ratio of 4.0 exhibited capacity to induce the precipitation of hydroxyapatite over the short period of 1 week. Long term Ti⁴⁺ ion release from all PEO-coated materials was 2–3 times lower than from the uncoated Ti. Metal ion release is attributed mostly to chemical dissolution of the coating at initial stages of immersion.

Significance. The long term stability was greater for thin PEO coating with overstoichiometric Ca/P ratio of 2.0, which exhibited ~95 ng cm⁻² of Ti⁴⁺ ions release over 4 weeks. Thin PEO coatings present economically more viable option.

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1. Introduction

Titanium and its alloys are well known as artificial biomaterials used for manufacturing of dental and orthopedic implants due to their high corrosion resistance and good mechanical stability [1,2]. Their biocompatibility is related to the chemical stability and structural integrity of the surface oxide layer (TiO₂) [3]. However, the thin native oxide layer does not form a strong bond with human bones since it is bio-inert [4]. To overcome this drawback, the structure, composition, and chemistry of the titanium surface needs to be modified [5–7].

Plasma electrolytic oxidation (PEO), also known as micro-arc oxidation (MAO), is a relative newcomer in the biomedical

field, a convenient technique for forming porous ceramic-like bioactive coatings on surfaces of titanium [8–10]. The porous oxide layers containing a mixture of anatase and rutile as well as bioactive elements, such as Ca, P, Si, Mg and Ag originating from the electrolyte, accelerate calcium phosphate formation on their surface in simulated body fluid (SBF) and promote the formation of hydroxyapatite (HA) [11–15]. A further advantage of PEO coatings is their good adhesion, since coating formation involves conversion (oxidation) of the substrate [16,17]. In addition, both *in vitro* [18] and *in vivo* [19] evaluations have revealed that PEO treatment considerably improves the bioactivity of titanium [20] and results in accelerated osseointegration of an implant. The latter is being

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Table 1 – Typical physiological media.

Material	Physiological solution	Reference
Ti6 Al4V	SBF	[38]
Ti–23Nb–0.7Ta–2Zr–O	Ringer's solution	[39]
Ti6Al4V and Ti6Al6Nb	Hank's balanced salt solution	[40]
Ti–6Al–4V and Ti–6Al–7Nb	Hank's naturally aerated solution	[41]
Ti–Mo	Fluoridated physiological serum	[42]
Ti CP, Ti6 Al4V and Ti–15Mo	Ringer's solution	[43]
Ti–6Al–4V	SBF	[44]
Ti	Ringer's solution	[45]
Ti	Artificial saliva	[46]
Ti	Artificial saliva	[47]
Ti	Fusayama and Meyer's artificial saliva	[48]
Ti–0.5Si–0.65C	Phosphate buffered saline	[49]
Sinteredporous Ti–10Mo	SBF	[50]
Ti–6Al–4V	Ringer's solution	[51]

attributed to specific topography of PEO coatings as well as to the presence of Ca and P in their composition. Since one of the characteristics of the bioactivity of a metallic implant for bone reconstruction is its apatite-forming ability in simulated body fluid, latest research on bioactive PEO coatings focuses in the generation of HA or compounds which act as precursors of HA using one-step approach, where Ca–P phases are generated during PEO, or involving post-treatments [21–30].

One of the most important characteristics of an implant is its service life, which must be extended as long as possible. This will largely depend on the corrosion resistance of the metal implant material and the release of metal ions: these are the critical factors that can adversely affect the mechanical integrity and biocompatibility [31–33]. It has been reported that the stability of the titanium surface oxide layer may be affected in physiological environments *in vivo* [34], increasing the metal ion release. This has been a source of concern due to potentially harmful effect of Ti^{4+} ions *in vivo*, because they have been shown to affect both proliferation and synthesis of extracellular matrix *in vitro* [35–37]. Therefore, the electrochemical behavior of titanium and its alloys in biological media is of increasing interest.

Several types of physiological fluids such as SBFs, artificial saliva and phosphate buffer solutions are used in electrochemical studies performed at body temperature (Table 1). In these biological media, PEO coatings have shown higher corrosion resistance than unmodified titanium alloy substrates [52–58]. The composition and, in particular, the thickness of bioactive PEO coatings may vary greatly in different studies. Even in case of today's only commercial PEO-coated dental implant TiUnite® (Nobel Biocare), which contains about 5–8% of phosphorus, the thickness, reportedly, varies from ~2 to ~9 μm [59,60]. Due to the mechanism of PEO coatings formation, their composition will depend on the treatment time, and therefore, on the coating thickness. The fabrication of thicker coatings requires greater energy consumption, and consequently, is more costly.

In this study two types of PEO coatings on commercially pure titanium with different Ca/P ratios and two different thicknesses each, are being examined with the aim of underpinning the effect of coating composition, thickness and microstructure on the corrosion behavior and hydroxyapatite forming ability in SBF.

2. Experimental methods

2.1. Materials

Specimens of dimensions $30 \times 20 \times 0.5 \text{ mm}$ were cut from commercially pure (c.p.) Grade I titanium foil (0.2 Fe, 0.18 O, 0.03 N, 0.015 H, and 0.18 C (max wt%)), degreased in isopropanol and rinsed in distilled water. Following subsequent pickling for ~20 s in a mixture containing 12 mL HF (40 wt%), 40 mL HNO_3 (70 wt%) and 48 mL H_2O at room temperature, the specimens were rinsed in distilled water, dried and masked with Lacquer 45 resin (McDermid plc.) to isolate a working area of 3 cm^2 .

2.2. Surface treatment

PEO treatment was carried out for 90 and 600 s using a 2 kW regulated AC power supply (EAC-S2000, ET Systems electronic). A square waveform voltage signal was applied with a positive-to-negative pulse ratio of 490 V/60 V at 50 Hz frequency and initial ramp of 60 s to achieve the voltage

Table 2 – Composition per litre of the immersion solution.

Reagents	Purity (%)	m-SBF ^a
NaCl	>99.5	5.403 g
NaHCO_3	>99.5	0.504 g
Na_2CO_3	>99.5	0.426 g
KCl	>99.5	0.225 g
$\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$	>99.0	0.230 g
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	>98.0	0.311 g
0.2 M–NaOH	–	100 mL ^b
HEPES ^c	>99.9	17.892 g ^b
CaCl_2	>95.0	0.293 g
Na_2SO_4	>99.0	0.072 g
1.0 M–NaOH	–	15 mL

^c HEPES = 2-(4-(2-hydroxyethyl)-1-piperazinyl) ethanesulfonic acid.

^b HEPES previously was dissolved in 100 mL of 0.2 M–NaOH aqueous solution.

^a Buffered at pH 7.4 at 36.5 °C with HEPES and 1.0 M–NaOH aqueous solution.

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