

Tribocorrosion Behavior of Anodic Titanium Oxide Films and Assessment of Cell-Materials Interactions

S.A. Alves^{abc}, R. Bayón^b, V. Saénz de Viteri^b, M.P. Garcia^c, A. Igartua^b, M.H. Fernandes^c, L.A. Rocha^{ad}

^a CT2M – Centre for Mechanical and Materials Technologies, Universidade do Minho, Guimarães, Portugal; ^b Fundación IK4-Tekniker, Eibar, Spain; ^c FMDUP – Faculdade de Medicina Dentária da Universidade do Porto, Porto, Portugal;

^d DEM – Department of Mechanical Engineering, Universidade do Minho, Guimarães, Portugal

Statement of Purpose: Titanium (Ti) has been widely used for implant applications due to an excellent biocompatibility and high resistance to corrosion provided by its highly stable passive oxide layer. However, Ti presents poor wear resistance which is a concern. Dental implants are frequently subjected simultaneously to a corrosive environment and cyclic micro-movements at implant/bone interface due to transmitted mastication loads. These may lead to the degradation of the implant material by tribocorrosion phenomena characterized by the liberation of wear debris and corrosion products/ions to the peri-implant site. These particles can induce a foreign body inflammatory response and, consequently, lead to periprosthetic osteolysis and implant loosening [1, 2]. Moreover, the lack of osseointegration immediately after implantation is also one of the most frequent causes of failure of these systems [3]. Hence, the main objective of this work was to modify the surface features of Ti in order to produce bio-multifunctional surfaces able to simultaneously display an improved tribocorrosion response and an enhanced biological performance.

Methods: Square samples of commercially pure (cp)-Ti grade 2 (Goodfellow Cambridge Ltd., Cambridge, UK) were submitted to Plasma Electrolytic Oxidation (PEO), an electrochemical technique that allows the production of oxide films on metal surfaces with high corrosion and wear resistances. Experiments were carried out using a 20 kW AC power supply (KT 20-50, Keronite International Ltd., Cambridge, UK). A commercial electrolyte (CE) containing sodium fluoride and a calcium and phosphate-based electrolyte (C β) were used in PEO treatments to improve materials bioactivity (Table 1). Untreated cp-Ti samples were used as the reference.

Table 1. PEO conditions used for anodizing treatments

Group of samples	Current density (A/dm ²)	Process duration (minutes)	Electrolyte
PEO_CE,8,5	8	5	CE
PEO_CE,8,10	8	10	CE
PEO_C β ,8,5	8	5	C β
PEO_C β ,25,10	25	10	C β

After PEO treatments, the roughness, thickness, morphology (by Scanning Electron Microscopy - SEM), composition (by Energy Dispersive x-ray Spectroscopy - EDS), crystalline phase (by X-ray Diffraction - XRD) of the anodic films were investigated. Reciprocating sliding tests were performed in a ball-on-plate tribometer. The tribocorrosion behavior was analyzed by Open Circuit Potential (OCP) measurements in artificial saliva (AS) and the coefficient of friction (COF) was monitored during sliding action. After tribocorrosion assays, the morphology of the wear scars was evaluated by SEM and wear volume measurements were also performed. The samples presenting the best tribocorrosion performances were submitted to biological assays in order to assess the interactions between MG63 bone cells and the produced

cp-Ti surfaces. The osteoblastic cells morphology and viability were evaluated, respectively, by SEM and MTT reduction assay for 1, 3 and 7 days of culture.

Results: Micro-porous anodic oxide films were produced on Ti surfaces by PEO treatments. The oxide films obtained presented different features, namely roughness, thickness, pore sizes and crystalline phases, according with anodizing conditions. The tribocorrosion behavior of cp-Ti was enhanced after anodizing treatments with both electrolytes. However, the OCP of C β -treated samples remained unaltered during the whole sliding duration and so, the wear/corrosion performance of these samples was significantly improved regarding to the ones treated with CE (Figure 1). Moreover, the wear volume of C β -anodized samples was significantly lower than the ones measured both on reference and CE-anodized samples.

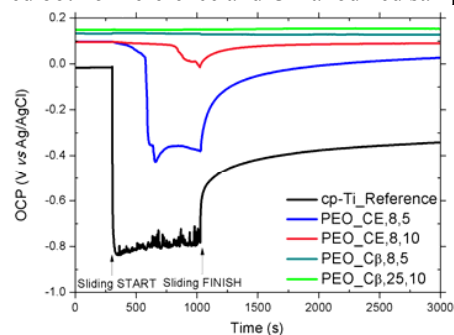


Figure 1. OCP evolution of treated and untreated cp-Ti samples prior, during and after reciprocating sliding tests. The osteoblasts were able to adhere and spread on untreated and treated samples. In agreement with SEM observations, MTT assay revealed that the cell proliferation increased throughout the culture time, with PEO_C β ,25,10 samples exhibiting the best performance.

Conclusions: In this work, porous anodic oxide films were produced on cp-Ti surfaces by PEO. The anodizing conditions significantly influenced the features of the anodic films produced on cp-Ti substrates. In addition, PEO treatments greatly improved the cp-Ti tribocorrosion performance. Furthermore, the electrolyte composition appears to play a crucial effect on wear/corrosion performance of anodized samples by decreasing the tendency to corrosion and mechanical damage. PEO_C β ,25,10 surface features significantly improved the tribocorrosion and biological performances of cp-Ti, leading to the development of bio-multifunctional Ti surfaces. The major highlight of this study relies on the development of the C β -treated samples by PEO as very attractive candidates for implant applications.

References:

- [1] (Vieira AC. Wear. 2006;261:994-1001.)
- [2] (Faghihi S. Nanotechnology. 2010;21:485703.)
- [3] (Tomisa AP. Int J Oral Maxillofac Implants. 2011;26: 25-44.)