Wettability behavior of nanotubular TiO_2 intended for biomedical applications

Molhabilidade de nanotubos de TiO_2 para aplicações biomédicas

La humectabilidad de nanotubos de TiO_2 para aplicaciones biomédicas

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Abstract

Nanotubes have been subject of studies with regard to their ability to promote differentiation of several cells lines. Nanotubes have been used to increase the roughness of the implant surfaces and to improve bone tissue integration on dental implant. In this study TiO_2 nanotube layer prepared by anodic oxidation was evaluated. Nanotube formation was carried out using Glycerol-H2O DI(50-50 v/v)+NH4F(0,5 a 1,5% and 10-30V) for 1-3 hours at 37ºC. After nanostructure formation the topography of surface was observed using field-emission-scanning-microscope (FE-SEM). Contact angle was evaluated on the anodized and non-anodized surfaces using a water contact angle goniometer in sessile drop mode with 5 µL drops. In the case of nanotube formation and no treatment surface were presented 39,1° and 75,9°, respectively. The contact angle describing the wettability of the surface is enhanced, more hydrophilic, on the nanotube surfaces, which can be advantageous for enhancing protein adsorption and cell adhesion.
Descriptors: Titanium; Nanotubes; Wettability.

Resumo

Nanotubos têm sido objeto de estudos no que se refere à sua capacidade de promover a diferenciação de várias linhagens celulares. Nanotubos têm sido utilizados para aumentar a rugosidade da superfície dos implantes e melhorar a integração do tecido ósseo. Neste estudo foi avaliada camada de TiO_2 de nanotubos preparada por oxidação anódica. Nanotubos foram formados utilizando glicerol-H2O DI (50-50 v / v) + NH 4 F (0,5 a 1,5% e 10-30) durante 1-3 horas a 37ºC. A topografia da superfície foi avaliada quanto à quantidade, diâmetro e altura dos nanotubos crescidos por meio de Microscópio Eletrônico de Varredura (MEV) de alta resolução (FEG-SEM). A molhabilidade foi avaliada por meio da mensuração do ângulo de contato obtido na superfície utilizando-se Goniômetro 300-F1 (Ramé-Hard Inst.Co) no modo de gota séssil com 5 gotas/microlitro. Resultados apontaram valores médios de 39,1 ° para superfície recoberta por nanotubos e 75,9° para superfície não-anodizada (controle). O ângulo de contato que descreve a capacidade de umedecimento da superfície mostrou-se aumentado sobre as superfícies de nanotubos. A maior hidrofilicidade pode se mostrar mais vantajosa para melhorar a adsorção de proteínas e adesão celular.
Descritores: Titânio; Nanotubos; Molhabilidade.

Resumen

Los nanotubos se han estudiado con respecto a su capacidad para promover la diferenciación de diversos linajes de células. Los nanotubos se han utilizado para aumentar la rugosidad de la superficie de los implantes y mejorar la integración del tejido óseo. En este estudio, TiO_2 nanotubos se preparó por oxidación anódica. Los nanotubos fueron formados utilizando glicerol-DI H2O (50/50 v / v) + NH4F (0,5 a 1,5%, y 10-30) durante 1-3 horas a 37 ° C. La topografía de la superficie se evaluó en cuanto a la cantidad, el diámetro y la altura de los nanotubos cultivadas por microscopio electrónico de barrido (SEM) de alta resolución (FEG-SEM). La humectabilidad se evaluó midiendo el ángulo de contacto obtenido en la superficie usando goniómetro 300-F1 (Ramé-Hard Inst.Co) en el modo de la gota sésil con 5 gotas / microlitro. Los resultados mostraron valores medios de 39,1 ° a la superficie cubierta por nanotubos y 75,9 ° en la superficie no anodizado (control). El ángulo de contacto que describe la capacidad de amortiguación de la superficie se incrementó en las superficies de los nanotubos. La mayor hidrofilicidad puede resultar más ventajoso para mejorar la adsorción de proteínas y la adhesión celular.
Descriptores: Titanio; Nanotubos; Humectabilidad.

INTRODUCTION

It is well known that the rate and strength of osseointegration of dental titanium implants, which is related to the direct connection between living bone and implant surface, undergo a strong influence of several factors[1-3]. Surface properties, such as topography, wettability, charge, surface energy, crystal structure and crystallinity, roughness, chemical potential, strain hardening, impurities, TiO_2 passive film thickness and coatings play important role for long-term clinical success[4,5]. The type of bone that receives the biomedical material, the surgical technique, the health of patient, the characteristics of the prosthesis to be applied on the implants and loading conditions can also influence the clinical performance and the longevity of the dental implant[6-9].

Titanium and titanium alloys have become very attractive biomaterials for prosthetic components in dental surgery due to their light weight, high biocorrosion resistance, biocompatibility and good mechanical properties[1-3]. The naturally formed oxide layer on titanium and titanium alloys (mainly TiO_2) is amorphous and has few nanometers thickness (usually 4 to 6 nm). This oxide film presents high chemical stability, low solubility in physiological media and no toxicity under in vivo conditions. However, this layer does not safe enough the dental implant from corrosion when exposed to the adverse oral environment, because it does not provide sufficient long-term corrosion protection and it is not an efficient barrier for preventing the usual release of metal ions. Several researchers claimed that the protective function of coatings such as Ca-phosphates or bioactive glass can decrease and lose themselves with the resorption[9,10].

Many studies have indicated that coating of nanotubular TiO_2 layer could improve the biocompatibility and bioactivity of titanium and its alloys due its stable chemical behavior and ability to promote differentiation of several cells lines. The nanotubular TiO_2 layer formed by anodization creates a nanometer-scale topography which can allow the bone to grow into, presents good loading distribution and keeps the implant surface under high shear forces[11].

The process of anodic oxidation of titanium is an electrochemical method that increases both the thickness of TiO_2 layer and roughness. Some authors ensure that anodized surface becomes more biocompatible, with increased cell attachment and proliferation. For the formation of nanotubular oxide film on titanium by anodization, the sample is immersed in a suitable electrolyte (generally containing fluoride species) and acts as the anode in the electrochemical cell. Platinum is generally used as cathode. A voltage is applied between anode and cathode, resulting in the formation and growth of the oxide film[12,13]. The formation of TiO_2 nanotubes occurs due to two competitive reactions, anodic oxidation and oxide dissolution due to fluoride ions. The characteristics of the nanotubular oxide layer is mainly dependent on voltage, anodization time, electrolyte composition and temperature[14].

Our research described in this paper has shown the method for control the nanotube size and morphology for biomedical implant use. The control factors are applied voltage, alloying, current density, time, and the electrolyte medium. The wettability behaviour of nanotubular TiO_2 layer formed by anodization and defined by SEM analysis.

MATERIAL AND METHODS

Commercially available pure Ti (CP-Ti, Grade 4, 99.5% purity, Alfa Aesar), machined into disks with dimensions of 6mm (diameter) X 1mm (thickness), was used as the substrate. These disks were ground using 400-grit SiC sandpaper and cleaned ultrasonically in acetone, ethanol and de-ionized water. To growth TiO_2 nanotubes the specimens were carried out using potentiostat (Model 362, EG&G, USA) with a conventional three electrode configuration having a platinum counter electrode, a saturated calomel reference electrode, and working electrode. The sample was embedded with epoxy resin, leaving a square surface area of 10 mm^2 exposed to the glycerol electrolyte (glycerol DI-H_2O) with DC fields 50-50 v / v and NH_4F solution (0.5 to 1.5%) by applying a pulsed DC field to the specimen (10-20V) for a period of 3 hours at 37°C. After anodization, the samples were rinsed with (DI) water and dried in air. The topography of surface was observed using field-emission scanning microscope (FE-SEM/ Hitachi S-4700, Tóquio, Japão). Contact angle was evaluated on the FS laser textured surface using a water contact angle goniometer 300-F1 (Ramé-Hard Inst.Co) in sessile drop mode with 5 microliter/drops.

RESULTS AND DISCUSSION

Figures 1 shows FE-SEM micrographs of the titanium implant with anodized surface. Figure 2 exhibit nanotube morphology of treated sample. The untreated and treated surfaces were evaluated for wettability. Anodized surface TI-CP was presented 39.1º for contact angle (Figure 3) against 75.9 º for no treatment surface. Results obtained in this experiment suggest that modification of implant surface with nanostructure and microstructure can promote bioactivity, bone ingrowth and mechanical fixation. It is confirmed that contact angle is related with the surface roughness. Therefore, nanotube formation is good for biocompatibility of implant materials cause dental implant quality depends on the chemical, physical, mechanical, and topographic characteristics of the surface[14]. These different properties interact and determine the activity of the attached cells that are close to the dental implant surface. Jeong et al.[15] found also small values for the wetting angle by anodized titanium alloys (Ti–30Nb–xZr and Ti–30Ta–xZr). However, we got smallest nanotubes diameter. Dental implants have been designed to provide textures and shapes that may enhance cellular activity and direct bone apposition. Several evaluations have demonstrated that implants with rough surfaces show better bone apposition and bone-to-implant contact (BIC) than implants with smooth surfaces[16,17]. Wettability and surface energy influence the adsorption of proteins, and increase adhesion of osteoblasts on the implant surface. The cell behavior on a hydrophilic surface is completely different from that on a hydrophobic one. A hydrophilic surface is better for blood coagulation than a hydrophobic surface. The expressions of bone-specific differentiation factors for osteoblasts are higher on hydrophilic surfaces. Consequently, dental implants with high hydrophilic and rough implant surfaces which in turn exhibited better osseointegration than implants with smooth surfaces are highly interesting[18-20].

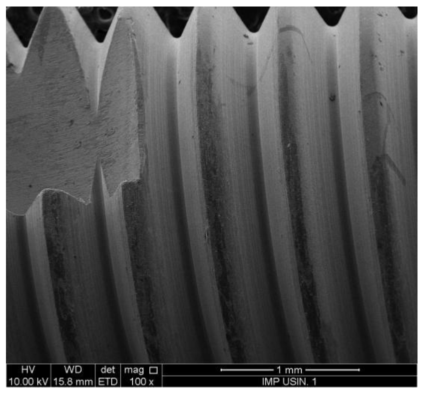

Figure 1. Scanning electron microscope (SEM) micrograph of the anodized implant

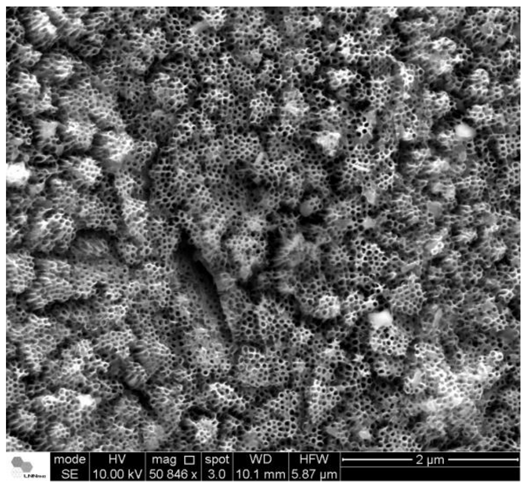

Figure 2. Scanning electron microscope (SEM) micrograph of the Ti specimens after electrochemical anodization treatment

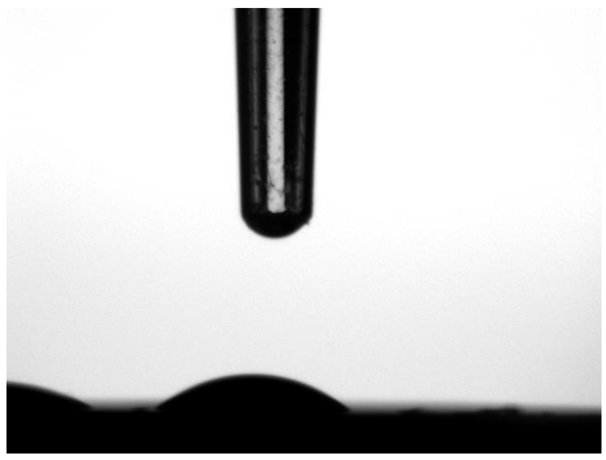

Figure 3. Contact angle values of the Ti specimens after electrochemical anodization treatment

CONCLUSION

The present outcomes suggest that the growth of nanotubes by anodic oxidation on the surface of Ti-CP was effective in experimental conditions and allowed higher wettability with the consequent increase in hydrophilicity.

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CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest

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