

Characterization of chemically treated Ti-Zr system alloys for dental implant application

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ABSTRACT

Materials and surfaces developed for dental implants need to withstand degradation processes that take place in the oral cavity. Therefore, the aim of the study was to develop and evaluate the topographical, mechanical, chemical, electrochemical and biological properties of Ti-xZr alloys (x = 5, 10, and 15 wt%) with two surface features (machined and double acid etched). Commercially pure titanium (cpTi) and Ti-6Al-4V alloy were used as controls. Surface characterization was performed using dispersive energy spectroscopy, X-ray diffraction, scanning electron microscopy, atomic force microscopy, profilometry and surface energy. The mechanical properties were assessed using Vickers microhardness, elastic modulus and stiffness. The electrochemical behavior analysis was conducted in a body fluid solution (pH 7.4). In addition, MC3T3-E1 cells were used to determine the impact of material and surface treatment on cell morphology by SEM analysis. Data were analyzed by two-way ANOVA and Bonferroni test ($\alpha = 0.05$). Ti-Zr alloys showed lower surface roughness, elastic modulus and stiffness, as well as higher hardness and surface energy when compared to cpTi. Ti-Zr system increased the polarization resistance values and significantly decreased the capacitance, corrosion current density (i_{corr}), and passivation current density (i_{pass}) values. The acid treatment increased the resistance and corrosion potential of the oxide layer. SEM data analysis demonstrated that Ti-Zr alloys displayed normal cell attachment/spreading and slightly changed cell morphology in the double etched surface. In conclusion, Zr addition and surface treatment altered surface, mechanical, biological and electrochemical properties of Ti material.

1. Introduction

Titanium (Ti) alloys have emerged in dentistry as viable options for rehabilitation with dental implants [1], especially in situations where the use of commercially pure titanium (cpTi) is not feasible. Applications involving extensive defect treatments, areas under high loads, or

cases that require implants with reduced dimensions [2,3] are conditions that demand materials with superior properties, generally achieved by adding chemical elements to Ti. Besides that, implants are exposed to complications of a mechanical or biological nature [4], such as fracture [5], the stress shielding effect [6], overloading, osseointegration failure, peri-implantitis [7], and corrosion and wear

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degradation processes that can lead to inflammation and allergenic or toxic reactions [8].

For a long period, Ti-6Al-4V alloy was the first choice for clinically challenging situations, mainly because of its enhanced mechanical performance. However, this material has been considered to have a highly stimulatory nature due to the release of ions and debris, which may play a critical role in stimulating peri-implant inflammation and osteolysis [9] due to its toxic potential [10]. Thus, an ideal material must offer high levels of mechanical stability without releasing metal ions into the human body. In this context, the binary Ti-Zr alloy has gained much attention, since this system guarantees increased mechanical strength with extensive applications [2,11], showing a remarkable decrease in ion release [12], which demonstrates its more inert and biocompatible behavior in comparison with cpTi [13,14]. Additionally, its homogeneous α -phase microstructure ensures that surface modification processes used for enhancing the osseointegration of Ti can still be applied to Ti-Zr implants [2,15].

This fact is of great concern, since the optimization of implant surface chemistry and topography has been crucial to promote cell adhesion, differentiation, and proliferation, as well as to increase the corrosion resistance [16]. Among the surface treatments developed in recent years, the process involving sandblasting followed by acid etching (SLA) of the implants is one of the most successfully applied in clinics [17]. Treatments that produce surfaces similar to those obtained by SLA but without the grit blasting process have been proposed. These surfaces are achieved by means of double acid etching, which appears to have the potential to greatly enhance osseointegration without adding particulate matter or embedding surface contaminants [18]. The biological [18,19] and corrosion behaviors [20] of this type of surface have been evaluated when applied in cpTi and Ti-Al-V alloys. However, the effect of such etching treatment on promising Ti-Zr alloys with different Zr concentrations has not yet been studied.

Therefore, this study developed and accessed the surface, mechanical, electrochemical, and biological properties of binary Ti-xZr ($x = 5, 10, \text{ and } 15 \text{ wt\%}$) alloys considering two surface features: machined and double acid etched. CpTi and Ti-6Al-4V alloys, which are widely used for the manufacture of dental implants, were used as controls.

2. Materials and methods

2.1. Experimental design

Fig. 1 shows the experimental design of this study. The machined surface was obtained by a polishing process, while the double etched group was chemically treated with acids (nitric, sulfuric, and hydrochloric acid) according to standard procedures (Military Institute of Engineering – IME, Rio de Janeiro, Brazil). CpTi and Ti-6Al-4V alloys were obtained in the shape of discs, 10 mm in diameter and 2 mm thick (Implalife Biotecnologia, Jales, SP, Brazil). Ti-x%Zr ($x = 5, 10, \text{ and } 15$) alloys were fabricated from pure metals.

2.2. Fabrication of experimental alloys

The three experimental binary Ti-Zr alloys (Ti-5Zr, Ti-10Zr, and Ti-15Zr) (wt%) were melted from pure metals (Ti and Zr presented degrees of purity equal or superior to 99.0%) (Sigma-Aldrich, St. Louis, MO, USA) in an arc-voltaic furnace with a water-cooled copper crucible under an argon atmosphere. The ingots were remelted five times to ensure homogeneity of the samples [11,21]. All ingots were heated to 1000 °C and hot-swaged to form bars $\approx 11 \text{ mm}$ in diameter. Then, ingots were fitted in quartz tubes, heat-treated at 1000 °C for 1 h, and air-cooled to improve the alloys' mechanical behavior [21]. Ti-Zr bars were machined into discs (10 mm in diameter and 2 mm thick).

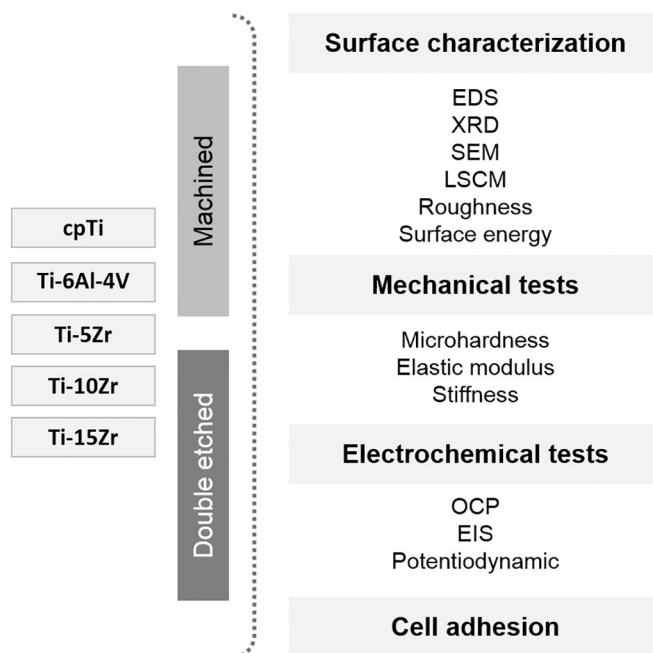


Fig. 1. Schematic diagram of the experimental design. EDS = energy-dispersive spectroscopy; XRD = X-ray diffractometry; SEM = scanning electron microscopy; LSCM = laser scanning confocal microscopy; OCP = open circuit potential; and EIS = electrochemical impedance spectroscopy.

2.3. Surface preparation

All discs were polished with #320-, #400-, and #600-grit SiC abrasive papers (Carbimet 2, Buehler, Lake Bluff, IL, USA) in an automatic polisher (EcoMet 300 Pro with AutoMet 250; Buehler, Lake Bluff, IL, USA). The surfaces that resulted from this process were considered as the machined surfaces.

The double etched surfaces were obtained according to proprietary standards (IME, Rio de Janeiro, Brazil). After being polished, the discs were chemically treated with a double acid etching (sulfuric and hydrochloric acid) process for 1 h and 50 min [20]. After surface preparation, all samples were ultrasonically cleaned/degreased with deionized water (10 min) and 70% propanol (10 min) (Sigma-Aldrich, St. Louis, MO, USA) and dried with warm air.

2.4. Surface characterizations

The samples' chemical compositions (in the order of $1 \mu\text{m}^3$) were checked by energy-dispersive spectroscopy (EDS) ($n = 1$). The microstructural analysis of the cpTi and Ti alloys were determined by X-ray diffractometry (XRD; Panalytical, X'Pert3 Powder, Almelo, The Netherlands) using Cu-K α ($\lambda = 1.540598 \text{ \AA}$) radiation and operating at 45 kV and 40 mA at a continuous speed of 0.02° per second and a scan range from 20° to 80° ($n = 1$). Scanning electron microscopy (SEM; JEOL JSM-6010LA, Peabody, MA, USA) was used to confirm the material microstructural phases after a specific polishing protocol that is followed by the surface etching with Kroll's reagent (5% nitric acid, 10% hydrofluoric acid, and 85% water) (Sigma-Aldrich, St. Louis, MO, USA) [21]. SEM and 3D Laser Scanning Confocal Microscope (LSCM, VK-X200 series, Keyence, Osaka, Japan) were used to observe the surface morphology and topography ($n = 1$). The LSCM images were obtained with lenses of $50\times$ and $150\times$ magnifications. VK-Analyzer software (Keyence v3.3.0.0) was used for image processing and surface area acquirement, which was achieved by the extrapolation of its measurements in images of $100 \times 100 \mu\text{m}$ ($50\times$ magnification) [22]. The average roughness (Ra) ($n = 5$) and surface free energy ($n = 5$) were evaluated by a profilometer (Dektak 150-d; Veeco, Plainview, NY,

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