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Short Communication

Three-dimensional and chemical changes on the surface of a 3-year clinically retrieved oxidized titanium dental implant

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ABSTRACT

Objective: To report the main topographical features in the micro- and nano-scales and to assess implant chemical changes of the surface of a 3-year clinically retrieved oxidized titanium dental implant, and compare them with a similar, unused implant.

Materials and methods: The surface of the oxidized titanium dental implants was assessed by surface electron microscopy (SEM) analysis at increasing magnifications. X-ray photoelectron spectroscopy (XPS) measurement was performed to analyze the implants surface chemistry. XPS spectra were acquired before and after sputtering with an Ar^{+} ion etching of 3 keV.

Results: With a length of 10–40 μm, and a width of 0.05–0.1 μm, numerous cracks were ubiquitous along the implant surfaces. Chimney-like structures formed micropores between 1 and 5 μm, with up to 40% of them partially or totally broken in the retrieved implant. In relation to chemical composition, Ti and O were predominant in both the unused and in the retrieved implant. N was present in high concentrations (11.49 at%) at the retrieved implant surface, in contrast with those observed for the unused implant (1.14 at%). Also, C was present in higher concentrations in the retrieved implant surface, while drastically decreased following the sputter-cleaning process.

Conclusion: While cracks were ubiquitous present from the manufacturing, broken chimney-like structures forming micropores in the clinically retrieved implant may be attributable to excessive mechanical friction forces during the insertion of the implant.

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Chemical composition of the implant surface may be subjected to changes because of the *in vivo* environment, with increase of N and C, and decrease of Ca and P.

1. Introduction

Endosseous dental implants have become a popular and widespread method for oral rehabilitation in partially or totally edentulous patients, the vast majority of them composed of titanium and its alloys, due to their resistance to corrosion, biocompatibility and high osseointegration rate (Daood et al., 2011). Ultimately, the efforts of the clinician and the industry must be conducted to decrease as much as possible the probability of implant failure in any kind of conditions. Factors affecting implant failure are diverse, including those related to implant design and composition, to biological issues and also to surgical technique. However, the greatest influence is osseointegration.

Implant geometry in the macro-scale is based on the threaded screw and macropores, in a range of millimeters to tens of microns (Daood et al., 2011). Also, the combination of surface morphology and chemistry may play an important role in implant success, as corrosion, together with functional stress and bacteria colonization, may be factors in implant failure (Chaturvedi, 2009). Treated surfaces with increased roughness and enlarged area have an increased number of bone contacts and a higher resistance to torque forces in comparison to non-treated implants (Gotfredsen et al., 2000; Li et al., 2002). We have to look for a possible explanation to this observation in the ingrowth of bone into larger surface irregularities (Schüpbach et al., 2005). At the micro-scale, the morphology of the surface of treated implants and also the interaction of bone with the micropores and surface irregularities have not yet been fully documented.

One of the techniques used for increasing roughness of implant surface is anodic oxidation. Using this technique the titanium oxide layer is increased, while interconnecting pores are formed along the entire surface. Several authors have reported the benefits of oxidized surfaces in terms of bone union in humans (Ivanoff et al., 2003; Rocci et al., 2003). For immediate loading, Glauser et al. (2003) observed a 3% failure rate for oxidized implants in comparison to approximately 17% for turned implants. Also Rocci et al. (2003) found a survival rate 10% higher for oxidized implants than for turned implants.

Scanning electron microscopy (SEM) produces a focused beam of electrons that interacts with electrons of the sample, providing information about its surface topography and composition. Two main features must be highlighted: (1) it can produce very high-resolution images of a sample surface, revealing details of less than few nanometers; (2) it has a large depth of field yielding a characteristic three dimensional (3D) appearance. Within the field of dental implantology, SEM has been used for the analysis of implant surface topography and composition alone or in conjunction with the bone–implant interface.

The aim of this study was to study the main topographical and chemical features of the surface of a 3-year clinically retrieved oxidized titanium dental implant at the micro-scale using SEM and X-ray photoelectron spectroscopy (XPS), and also to compare them with those found in a similar new unused implant.

2. Material and methods

2.1. Clinical case

A partially edentulous 55 year-old male previously rehabilitated with dental implants in the upper maxilla and mandible three years before presented to our center (CICOM, Badajoz, Spain) complaining of pain and mobilization of an implant placed at the first maxillary molar. After careful examination a Brånemark System Mk IV TiUnite® ($4 \times 11.5\ mm^2$) (Nobel Biocare AB®, Göteborg, Sweden) dental implant was retrieved.

TiUnite® is titanium oxide rendered into an osteoconductive ceramic biomaterial through spark anodization. It has a porous surface, slightly rough ($Ra \approx 1.3$ micrometric) with titanium oxide (TiO_2). This crystalline surface is further enriched with phosphates and it is microstructured with no pronounced features and it is characterized by the presence of open pores or "craters" distributed in the low micrometer range.

The retrieved implant and a similar unused one were submitted for further analysis by SEM at the CIBER-BBN (Centre for Biomedical Investigation in Net in Bioingeniery, Biomaterials and Nanomedicine), Badajoz, Spain.

2.2. Implant cleaning

Prior to capturing SEM images, the implants were carefully cleaned using an antiseptic cleaner (DERQUIM DSF 11; Panreac Quimica S.A., Spain), gently rubbing with a smooth cotton cloth to remove any biological residue from the surface. Then they were rinsed repeatedly with distilled water and sonicated in distilled and deionized water (Milli-Q system). Finally, they were placed in alcohol for a few seconds and then allowed to air dry at room temperature.

2.3. Scanning electron microscopy (SEM)

SEM images of the implants were taken with a scanning electron microscope Quanta 3D FEG (FEI, Hillsboro, US) operated at different voltages (indicated at the bottom of each of the images) with secondary electrons. Images with magnification ranging from 150 to 50,000 were taken randomly in different sections of the implants.

2.4. X-ray photoelectron spectroscopy (XPS)

XPS measurement was performed for surface chemistry analysis and characterization of elements present in the