Nuclear Instruments and Methods in Physics Research B xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Evaluation of modified titanium surfaces physical and chemical characteristics

Magdalena Lukaszewska-Kuska *, Bartosz Leda, Przemyslaw Gajdus, Wieslaw Hedzelek

Department of Prosthodontics, Poznan University of Medical Sciences, Bukowska 70, 60-812 Poznan, Poland

A R T I C L E I N F O

Article history:
Received 29 June 2016
Received in revised form 18 January 2017
Accepted 6 March 2017
Available online xxxx

Keywords:
Titanium
Surface modification
SEM
EDS
Profilometry

A B S T R A C T

Development of dental implantology is focused, among other things, on devising active surface of the implant, conditioning acceleration of the implant's integration with the bone. Increased roughness, characteristic for group of implants with developed surface, altered topography and chemically modified implant's surface determines increased implants stability. In this study four different titanium surfaces modifications: turned (TS); aluminium oxide-blasted (Al_2O_3); resorbable material blasted (RBM); sandblast and then etched with a mixture of acids (SAE), were evaluated in terms of surfaces topography and chemical composition prior to in vivo analysis. Topography analysis revealed two groups: one with smooth, anisotropic, undeveloped TS surface and the second group with remaining surfaces presenting rough, isotropic, developed surfaces with added during blasting procedure aluminium for Al_2O_3 and calcium and phosphorus for RBM. Physical and chemical modifications of titanium surface change its microstructure (typical for SAE) and increase its roughness (highest for Al_2O_3-blasted and RBM surfaces). The introduced modifications develop titanium surface – 10 times for SAE surfaces, 16 times for Al_2O_3-blasted surfaces, and 20 times for RBM surfaces.

© 2017 Elsevier B.V. All rights reserved.

1. Introduction

Development of dental implantology is focused, among other things, on devising active surface of the implant, conditioning acceleration of the implant's integration with the bone. Implants with developed surface, such as titanium plasma-sprayed surface and sand-blast and/or etched surfaces, are rougher than the smooth machined surface [1–4]. In addition to roughness, also the topography, chemical composition, wettability, or TiO_2 layer thickness affect the surface's activity. Rough surfaces of implants with modified topography offer faster adhesion of ions and cells, forming a stronger bond with the bone or connective tissue [5–7]. In vitro studies have shown that rough surface also affects proliferation and differentiation of chondrocytes, osteoblasts, and matrix synthesis [8,9]. Osteoblasts are capable of distinguishing and reacting to various chemical compositions of the surface. Experimental enrichment of implant surfaces with calcium, phosphorus, magnesium, or sulphur facilitates formation of a biochemical bond between the implant and the tissue, resulting in accelerated osseointegration [10]. Ceramic materials, on the other hand, such a bicalcium phosphate (BCP) or hydroxyapatite (HA) are capable of dissolving in tissue liquids, leading to precipitation of apatite on the implant surface and to mineralisation of the bone tissue [11]. Osteoinductive properties of BCP and HA are also associated with the fact that precipitation of apatite is accompanied by precipitation of growth factors, such as bone morphogenetic proteins, from body fluids, and this results in differentiation of cells into osteoblasts [12]. Treatments applied to change physical and chemical properties of implant surfaces are aimed at inducing a biological response facilitating functional loading of integrated implants with the prosthetic structure.

2. Aim

The aim of the study was to evaluate physical characteristics of modified titanium surfaces in terms of topography and roughness along with the evaluation of surfaces chemical composition.

3. Materials and methods

To evaluate characteristics of active surfaces in in vitro studies, titanium discs of 8 mm in diameter and 1 mm thickness were used. The discs were made of commercially pure titanium grade IV using titanium rod machining method. Chemical composition includes titanium with admixtures of oxygen (0.285%), carbon (0.01%),

* Corresponding author.
E-mail address: m.lukaszewska.kuska@gmail.com (M. Lukaszewska-Kuska).

http://dx.doi.org/10.1016/j.nimb.2017.03.029
0168-583X/© 2017 Elsevier B.V. All rights reserved.

hydrogen (0.0055%), and nitrogen (0.007%). Titanium discs were manufactured by the Dental Implants Manufacturing Osteoplant, Foundation of Poznan University of Medical Sciences, Poland. In terms of titanium samples preparation method, surface types were divided into four study groups:

1. turned surface sample – TS
2. aluminium oxide-blasted surface sample – Al_2O_3-blasted
3. resorbable blast material surface sample – RBM
4. mixed surface sample: sandblast and then etched with a mixture of acids – SAE (Sandblast and Acid-Etched)

Turned surface (TS) is a result of titanium rod machining. Following completion of the machining process, no further modifications of the discs surface were made. Turned titanium surface samples were the control group in the study.

Aluminium oxide (Al_2O_3) blasted surface was obtained by bombarding the turned surface discs with aluminium oxide grains sized between 53 μm and 75 μm. Grain size was determined on the basis of experiments by Wennerberg and other researchers [13–17]. The sand used contained mainly Al_2O_3 (98.5%) and trace amounts of SiO_2 (0.18%), TiO_2 (0.01%), Fe_2O_3 (0.007%), and CaO (0.001%). Sandblasting was performed at the temperature of 22 °C and the pressure of 6 atm.

RBM surface was obtained by blasting the turned surface of titanium discs with a mixture of hydroxyapatite (HA) and tricalcium phosphate (TCP) in the ratio of 7:3. Chemical composition also featured trace amounts of Na (1%), Mg (0.9%), Zr (0.05%), and Fe (0.035%). Grain size varied from 300 to 600 μm. Blasting conditions, temperature, and pressure were identical as in aluminium oxide blasting.

SAE surface was obtained in two stages. The first stage consisted in blasting of titanium discs with turned surface with an HA/TCP mixture in line with the procedure described above. Then, titanium samples were flushed in acetone (7 min) and distilled water (3.5 min). In the second stage, the samples were etched in a mixture of 36% hydrochloric acid (HCl) and 96% sulphuric acid (H_2SO_4) in the ratio of 1:6. The procedure took 10 min and was performed in room temperature. Finally, titanium samples were once again flushed with acetone and distilled water and left to dry.

24 titanium, discs, 6 per each of the four study groups, underwent typical preparatory treatments similar to the dental implant manufacturing process. Preparation of samples comprised degreasing, flushing with isopropanol, chemical disinfection in ultrasound cleaner, flushing with distilled water, and drying. Titanium discs were then sterilised by radiation (electron accelerator, radiation dose 25 kGy).

To determine characteristics of their active surfaces, the titanium samples were tested using the following methods:

1. scanning electron microscopy analysis of the surface's topography,
2. spectroscopic analysis of the surface's chemical composition,
3. 2D and 3D profilometric analysis of the surface's roughness.

Topography of the titanium discs surfaces was analysed using micrographs on Zeiss EVO25 (Carl Zeiss, Germany) scanning electron microscope (SEM).

The presence of aluminium, calcium and phosphorus, on the surface of titanium, as a result of the different chemical surface treatments, was analysed by energy dispersion spectroscopy (EDS). The analysis was performed using Quantax AXS detector (Brucker, Germany) combined with a scanning electron microscope (EVO Zeiss, Germany).

The sample roughness test was carried out without touching the surface through the use of a NT1100 interference microscope using Wyko Vision®32 (Veeco Instruments, USA) software. A five-fold magnification was used on a test surface of 0.9 × 1.2 mm. The surface of each sample was measured at five different sites and the results were presented as the mean of 5 values.

Standard profilometric measurements are taken in two dimensions (2D) on single cross-sections referred to as roughness profiles. Roughness degree is most frequently described using amplitude parameter Ra. Although Ra is stable, easy to measure, and allows comparison with other previously tested surfaces, it has its limitations. Most importantly, Ra – being a 2D parameter – does not fully reflect the characteristics of the three-dimensional active surface of implants. It gives no information on the profile shape or the spatial layout. Therefore, three-dimensional (3D) parameters were additionally used to characterise the roughness more fully [18–20]. 3D dimension pertains to the surface and is referred to as stereometric surface measurement. It is usually taken contact-free and then processed digitally. Spatial analysis provides detailed information on the shape and size of the irregularity, allowing also determination of functional parameters such as load bearing capacity, abrasion, or contact area [21,22]. In the present paper, 2D and 3D parameters are used to describe roughness.

List of parameters and their units used in the conducted study:

1. 2D parameters
   a. Ra – arithmetic mean of profile ordinates [μm]
   b. Rq – square mean of profile ordinates [μm]
   c. Rsk – profile asymmetry factor [–]
   d. Rt – total profile height [μm]
2. 3D parameters
   e. Amplitude
      i. Sa – arithmetic mean of surface ordinates [μm]
      ii. Sq – square mean of surface ordinates [μm]
      iii. Ssk – surface asymmetry coefficient [–]

   f. Spatial
      iv. Sds – density of surface roughness peaks [1/sq mm]
      v. Str – surface texture indicator [–]

   g. Hybrid
      vi. Sdq – square mean of surface slope [°]
      vii. Sdr – surface development [%]
      viii. Ssc – arithmetic mean of curvatures of surface roughness peaks [1/mm]

Statistical analysis using variance analysis method (ANOVA) was performed for the obtained results. In order to establish the difference between groups in study sample, post-hoc tests were performed. Profilometry results, on the other hand, were analysed using HSD test for unequal Ns, which is a modified Tukey's test. Calculations and presentation of data were performed using Statistica 10 (StatSoft Inc.) and Office (Microsoft) software packages.

In vivo evaluation of modified titanium surfaces influence on bone was also conducted. Results of this study are presented elsewhere.

4. Results

4.1. Surfaces topography analysis

Images of surfaces of the tested titanium discs recorded with the scanning electron microscope are presented on the figures (Figs. 1–4). In SEM images, topography of titanium surface samples upon modification has a different structure than the control turned

Download English Version:

https://daneshyari.com/en/article/8039548

Download Persian Version:

https://daneshyari.com/article/8039548

Daneshyari.com