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Measurement and Image Processing Evaluation of Surface Modifications of Dental Implants G4 Pure Titanium Created by Different Techniques

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Abstract. Foreign substances and organic tissue interaction placed into the jaw in order to eliminate tooth loss involves a highly complex process. Many biological reactions take place as well as the biomechanical forces that influence this formation. Osseointegration denotes to the direct structural and functional association between the living bone and the load-bearing artificial implant's surface. Taking into consideration of the requirements in the manufacturing processes of the implants, surface characterizations with high precise measurement techniques are investigated and thus long-term success of dental implant is emphasized on the importance of these processes in this study. In this research, the detailed surface characterization was performed to identify the dependence of the manufacturing techniques on the surface properties by using the image processing methods and using the scanning electron microscope (SEM) for morphological properties in 3D and Taylor Hobson stylus profilometer for roughness properties in 2D. Three implant surfaces fabricated by different manufacturing techniques were inspected, and a machined surface was included into the study as a reference specimen. The results indicated that different surface treatments were strongly influenced surface morphology. Thus 2D and 3D precise inspection techniques were highlighted on the importance for surface characterization. Different image analyses techniques such as Dark-light technique were used to verify the surface measurement results. The computational phase was performed using image processing toolbox in Matlab with precise evaluation of the roughness for the implant surfaces. The relationship between the number of black and white pixels and surface roughness is presented. FFT image processing and analyses results explicitly imply that the technique is useful in the determination of surface roughness. The results showed that the number of black pixels in the image increases with increase in surface roughness.

Keywords: Biomedical surfaces, Image Processing, Stylus Profilometer, SEM
PACS: 87.85.Pq

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INTRODUCTION

Compared to the conventional pure Ti-Grade 4 surface, many different surface topography modifications are applied in order to improve bone and implant interactions. There are different mechanical and chemical applications for instance, grit blasting with various types and sizes of abrasives, acid etching and electrochemical processes with different solutions, plasma-sprayed coating methods by organic or inorganic materials or hybrid techniques which contains combination of some of them [1, 2]. All of these surface modification methods fundamentally aim to control chemical interaction, surface energy, contact angle and contact area between tissue and implant for a good osseointegration. For an acceptable implant procedure an optimum interlocking between the organic tissue and titanium surface has been proposed as an osseointegration by establishing a good contact between bone and Ti-implant [3- 5].

By microscopic and laboratory researches it is obtained that by alternative surface modification techniques such as sand blasting, acid etching, coatings presented better bone formation around the implant surfaces compared to the Cp titanium surfaces [6-8]. Many different topographical suspects assumed to be effective for early implant fixation and better long term osseointegration have been searched. These 3D geometrical properties are also contributing to better chemical and mechanical performances of titanium implants. In researches it is founded that the implant surface energy founded to be sensitive to the titanium surface area. Fixation of the implant is strongly correlated with all of these parameters for instance, surfaces with higher areas and smaller contact angles implant presents stronger interactions of the biological cells with the implant surface [8, 9]. Especially rough surfaces showed better initial fixation because of stronger interactions and the interfacial shear strength correlated positively with the degree of surface roughness [10]. As it is known that titanium dental implant surface is a multidimensional, and can be grouped according to their surface geometrical characteristics in macro, micro, nanometer scales. For long term osseointegration submicron surface properties were assumed to be the most effective parameter [11].

Researchers used many different image observation and measurement techniques to obtain these 2D and 3D surface topographical properties accurately [12]. Previous studies indicated that accurate and precise surface characterization had a vital effect for understanding the performance of different titanium implant surfaces manufactured for osseoteintegration.

The aim of the present study was to determine, by means of comparing different techniques, the surface properties of different dental implant surfaces manufactured using sand blasted, acid etched and ceramic blasted techniques. In this research, obtaining the precision of these surface properties was assumed to be the key method for the characterization of 2D and 3D topographies using different measurement devices such as SEM and SP.

METHODS AND MATERIALS

Implant Specimens

In this experimental research, the specimens were made from 6 mm cylindrical Grade 4 titanium (ASTM B 348, ISO 5832/2). The specimen geometries were ready to use with geometrical dimensions of 4.7 mm diameter and 16 mm length. Implant specimens were divided into three different groups according to their manufacturing methodologies; sand blasting, acid etching and ceramic blasting.

For this experimental study specimen groups were selected especially to investigate topographical differences on their surfaces. At the beginning of the study, all specimens were blasted to increase surface roughness. From recent in vivo and in vitro studies it can be easily understood that increasing surface roughness enables bone colonization on the implant which provides implant fixation [13, 14]. Grit blasting method enabled proper geometries on the implant surface for tissue formation [15, 16]. In this experimental study two different blasting materials were selected. Particle geometries and chemical composition for these two specimen groups were different. The first group was TiO_2 blasted. In literature, it is founded that the blasting surfaces with different materials were also created by different chemical composition on the implant surfaces. For these reasons, the third group was modificated with a ceramic material which includes Ca-P, HA. It is well known that HA is a good biocompatible material [17, 18]. In vivo studies especially these two materials are known for their good osteoconductivity (for the early stage of osteogenesis). They enable direct binding to bone tissue in vivo [1, 19]. From this point of view, the second group was selected as sand blasted and then acid etched which decreases the shot particle geometry sharpness on the titanium implant surfaces.

These different modification techniques were selected for this experimental study, thus observing the macro and micro scale differences more slightly. All of these selected surface modification techniques are using in today's dental implants market, and approved to have reasonable osseointegration performance.

First two groups were blasted with TiO_2 particles of 7–220 µm. This procedure was applied by jets from a 20 mm distance and the TiO_2 particles hit the surface with nearly 90 degree. A radial forceps held the test specimens during the blasting procedure to enable homogeneous blasting procedure. After this procedure the specimens were stored in isolated containers before other surface modifications.

A positive correlation was found in a research between increasing grain size of the TiO_2 particles, and the degree of functional attachment. This correlation was true for grain sizes between 7.5 and 220 µm. Particles with distorted geometries found out to be having not an impact on improvement for the retention capacity of the implants [13]. In our study first group specimen blasted with titanium oxide (TiO_2) particles approximately 150 µm in diameter with a blast pressure of 4 Bar for 40 seconds. Second group was the blasted and etched group, had their surfaces waited in a hydrofluoric acid (HF) bath. These specimens had two step modification procedures so called hybrid surface treatment. All of specimens firstly had sand blasting procedure with same parameter of the first group and then the acid was applied to the surface. After sand blasting, the implants were waited in a hydrofluoric acid bath for 12 seconds.

The third group specimens were HA/B-TCP Biphasic Calcium Phosphate blasted. According to the material technical report the composition includes >65% hydroxyl apatite, <35% b-TCP, A-TCP and TTCP, <5% Ca-P materials. The particles' were >95% bigger than 300 µm and <5% smaller than 300 µm. The blasting period was 50 second and the pressure was 3.5 Bar. For the blasting procedure a nozzle with 1.5 mm diameter was used.

MEASUREMENT

In this study, three different implant groups were investigated by means of the evaluation of the roughness measurements as well as the analysis of the images captured from scanning electron microscope with the help of image processing technique called FFT-Dark Light Technique. The analyses performed on the surfaces of the titanium dental implant which had the same geometry and material besides their surface topography was individually treated.

Contact Stylus Type Profilometer

The contact roughness measurement of the dental implant surfaces was performed by the stylus type profilometer (Form Talysurf Intra 50 profilograph) with µltra software (FTS Iµ) illustrated in Table 1. according to the ISO 4287 [9] by mapping the readings taken in a direction perpendicular to the direction of lay by calculating of the parameters Ra from a standard spectrum of roughness. Table 1 represents the specifications of the contact stylus profilometer. During the stylus measurement procedure, gauss filter with 0.8 mm cut off value, and 0.8 mm length was selected for assessing the 2D profile roughness measurement. Same measurement parameters were used for all groups for enabling comparison within groups.

The measurement was conducted in the apical part of the dental implants in linear direction and 0.8 mm length. The maximum, minimum and mean roughness values were recorded.

TABLE 1. The Specification of contact stylus type profilometer

Measurement Method	Stylus Profilometer
Spatial Resolution	1-2 µm
Z Resolution	3 -16 nm
Range Z	3 -16 nm

Scanning Electron Microscope-Image Acquisition

In many experimental researches, SEM was used for its high image capability [20, 21]. In these study scanning electron microscope (Zeiss Evo) was used for revealing characteristic differences at the micro level according to the surface modification methods used for implant samples. For observation of surfaces SE mode with an acceleration voltage of 10 kV was selected. The specimen were maintained under $P<1 \times 10^{-5}$ Torr vacuum pressure.

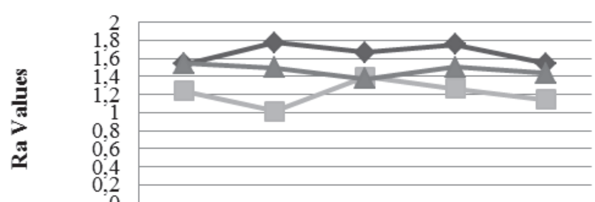
All specimens were captured from 7 mm distance and with same magnifications of 200X. All implant images were technically taken in the near region of the screw thread by means of electron microscopic scanning analysis.

Images of the specimens were taken using a CCD camera. The resolution of CCD camera is 54 Megapixels. Before all image analyses, images were resized into 1024X768 pixel sizes in order to standardize images. The captured images of the specimens were analysed using image processing technique such as FFT-Dark Light Technique. In analyses, RGB images were converted into gray level. A threshold is applied to the images and all images were binarised. FFT algorithm was applied to all images. Then the numbers of white, black and total pixels for each image were computed.

RESULTS AND DISCUSSION

The surface topography of each implant group roughness measurement results of Ra values which were taken from the Form Talysurf Intra 50 profilograph were given in Table 1. Tables 3 and 4 represent the number of black and white pixels computed from the Fourier domain image and corresponding mean Ra values.

TABLE 2. Roughness values of specimen surfaces



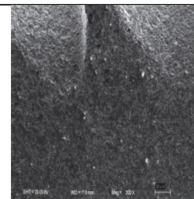
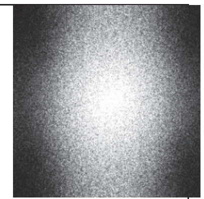
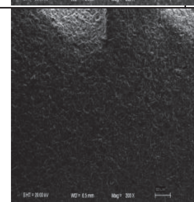
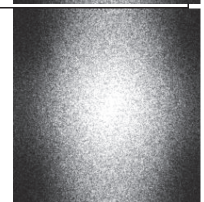
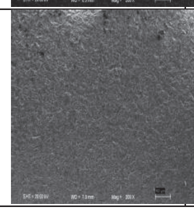
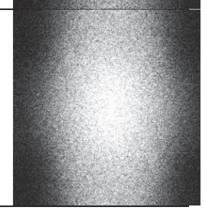
	1	2	3	4	5
BCP	1,535	1,775	1,667	1,753	1,545
Acid Etched	1,238	1,014	1,394	1,265	1,144
TiO2 Blasted	1,545	1,5	1,375	1,505	1,435

TABLE 3. Number of Black Pixels computed from the Fourier domain image and corresponding mean Ra values

Type of Manufacturing	**Number of Black Pixels**	**Mean Ra (µm)**
TiO_2 Blasted+ Acid Etched	132843	1.211
TiO_2 Blasted	138470	1.472
BCP	139944	1.655

TABLE 4. Original and FFT transformed images for BCP Surface, TiO_2 Blasted Surface and TiO2 Blasted + Acid Etched Surface

Surfaces	Original Images	FFT Images

BCP Surface		
TiO_2 Blasted Surface		
TiO_2 Blasted + Acid Etched Surface		

In this paper, the roughness of the implant surfaces has been investigated by using image processing technique (FFT). The texture of these implant surfaces were observed and their captured images from 3D digital microscope and SEM were processed to investigate their surface topographies. Original and FFT transformed images for the implant surfaces with different manufacturing techniques are illustrated in Table 2-4, which illustrates the original and FFT transformed images for BCP surface, TiO_2 blasted surface and TiO_2 blasted + acid etched surface.

The results showed that the number of black pixels in the image increases with increase in surface roughness.

CONCLUSSIONS

Image processing method analysis suggested that there are critical topographical differences on the individual treated dental implant surfaces.

From the SEM image-FFT analysis the macroscopic structure of TiO2 Blasted+ Acid Etched surface founded to be smoother from other groups (Table 2). The images proved that only applying TiO2 blasting procedure caused more roughened surfaces. The SEM images exhibit that grit blasted surfaces showed irregular morphology with different sized cavities. From literature roughened surfaces claimed to show better osseintegration as mentioned before [22- 24]. But the positive influence of acid shower was thought to be that the additional chemical process let no residues on the surfaces after blasting process as seen on SEM figure (Table 4). This is an advantage when the dissolution of the modification material to the body unwanted.

Etching in acid process decreased the roughness, sharp edges and decreased the residual on the surface. In the blasted surfaces the residual particles were easily identified using backscattered SEM as they appeared darker than the titanium itself Table 4. The BCP ceramic particles and TiO2 particles identifies from SEM images.

The roughest surface observed from the third group biphasic calcium phosphate ceramic blasted surfaces. It is assumed that increasing grain size of the blasting material resulted in increased roughness of the surfaces, but also caused different topographical characteristics. Blasted titanium surfaces exhibited an irregular rough morphology. It can be deduced from SEM images BCP surfaces and TiO2 blasted surfaces exhibit similar irregularities but BCP surface images showed wider cavities 10-25 µm produced by the blasting process in which micro pits, about 300 µm in diameter. The TiO2 blasted surface images showed cavities 0.5-15 µm in diameter.

All specimens were blasted and the blasted groups had irregularities in the form of pits from residuals stripes which caused from surface modification procedure or left from machining process. Also flat areas identified in the micron level which decreases in surface contact angle. In the macron level not irregular substantial flat areas identified which means homogeny blasted surfaces.

The relationship between the number of black and white pixels and surface roughness is presented. FFT image processing and analyses results explicitly imply that the technique is useful in the determination of surface roughness.

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REFERENCES

1. Aparicio C, Gil FJ, Fonseca C, Barbosa M, Planell J A. Corrosion behaviour of commercially pure titanium shot blasted with different materials and sizes of shot particles for dental implant applications. Biomaterials. 2003, 24(2), p.263-73.
2. Citeaua A, Guicheux J, , Vinatier C, Layrolle P, T. Nguyen P, Pilet P, Daculsi G.I n vitro biological effects of titanium rough surface obtained by calcium phosphate grid blasting. Biomaterials. 2005, 26(2), p. 157-165.
3. Kim B S, Kim J. S., Park Y M, Choi B Y, Lee J. Mg ion implantation on SLA-treated titanium surface and its effects on the behavior of mesenchymal stem cell. Materials Science and Engineering. 2013;33, p. 1554-60.
4. Guéhennec Le L, Soueidan A, Layrolle P, Amouriq Y. Surface treatments of titanium dental implants for rapid osseointegration, Dental Material. 2007;23, p. 844-854.
5. Ince A, Schütze N T, Hendrich C R, Thull, Eulert J, Löhr J F. In Vitro Investigation of Orthopedic Titanium-Coated and Brushite-Coated Surfaces Using Human Osteoblasts in the Presence of Gentamycin. The Journal of Arthroplasty. 2008; 23(5), p.762-71.
6. Buser D, Schenk R K., Steinemann S, Fiorellini J P, Fox C H, Stich H. Influence of surface characteristics on bone integration of titanium implants a histomorphometric study in miniature pigs. Journal Biomedical Material Resouce. 1991; 25, p.889-902,
7. Darimont G L, Cloots R, Heinen E, Seidel L, Legrand R. In vivo behavior of hydroxyapatite coatings on titanium implants: a quantitative study in the rabbit. Biomaterials. 2002; 23(12), p. 2569-75.

8. Bacchelli B, Giavaresi G, Franchi M, Martini D, Pasquale V D, Trirè A, Fini M, Giardino R, Ruggeri A, Influence of a zirconia sandblasting treated surface on peri-implant bone healing: An experimental study in sheep. Acta Biomaterialia. 2009; 5(6), p. 2246-57.
9. EN ISO 4287:2009, Geometrical Product Specifications (GPS) - Surface texture: Profile method - Terms, definitions and surface texture parameters (ISO 4287:1997 + Cor 1: 1998 + Cor 2: 2005 + Amd 1:2009) (includes Corrigendum AC:2008 and Amendment A1:2009).
10. Eliasa C N, Oshida Y, Ca J Lima H, Mullere C A. Relationship between surface properties (roughness, wettability and morphology) of titanium and dental implant removal torque. Journal of The Mechanical Behavior of Biomedical Materials. 2008, p. 234-242.
11. Davies J E, Ajami E, Moineddin R, Mendes V C. The roles of different scale ranges of surface implant topography on the stability of the bone/implant interface. Biomaterials. 2013; 34 p. 3535-46.
12. Osanna P H, Durakbasa M N, Yaghmaei K, Kräuter L. Quality Control and Nanometrology for Micro/Nano Surface Modification of Orthopaedic/Dental Implants. Institute of Measurement Science Slovak Academy of Sciences. 2009; p.167-172.
13. Geesink R G T. Osteoconductive coatings for total joint arthroplasty. Clinical orthopaedics and related research. 2002; p. 53-65.
14. Traini T, Mangano C, Sammons R L, Manganod F, Macchi A, Piattelli A. Direct laser metal sintering as a new approach to fabrication of an isoelastic functionally graded material for manufacture of porous titanium dental implants. Dental Materials. 2008; 24, p.1525-33.
15. Esposito M, Hirsch J M, Lekholm U, Thomsen P. Biological factors contributing to failures of osseointegrated oral implants. European Journal of Oral Sciences. 1998; 106, p. 527-551.
16. Lavenus S, Rozé J, Hoornaert A, Louarn G, Layrolle P. Emerging Nanotechnologies in Dentistry Processes, Materials and Applications. Impact of Nanotechnology on Dental Implants. 2012; 5, p.71-84.
17. Nguyen H Q, Deporter D A, Pilliar R M, Valiquett, N, Yakubovich R, The effect of sol-gel-formed calcium phosphate coatings on bone ingrowth and osteoconductivity of porous-surfaced Ti alloy implants. Biomaterials. 2004; 25, p.865-876.
18. Piattelli, A, Scarano, A, Mangano C. Clinical and histologic aspects of biphasic calcium phosphate ceramic (BCP) used in connection with implant placement. Biomaterials. 1996;17(18), p.1767-70.
19. Yoshinari M, Oda Y, Inoue T, Matsuzaka, K, Shimono M. Bone response to calcium phosphate-coated and bisphosphonate-immobilized titanium implants. Biomaterials.2002;23(14), p.2879-85.
20. Gaggl A, Schultes G, Müller W D, Karcher H. Scanning electron microscopical analysis of laser-treated titanium implant surfaces. Biomaterials. 2000; 21(10), p.1067-73.
21. Luo X P, Silikas N, Allaf M, Wilson N H F. AFM and SEM study of the effects of etching on IPS-Empress 2TM dental ceramic. Surface Science. 2001;491(3), p. 388–394.
22. Han C H, Johansson C B, Wennerberg A, Albrektsson T. Quantitative and qualitative investigations of surface enlarged titanium and titanium alloy implants. Clinical Oral Implants Resources. 1998(9), p.1-10.
23. Feighan J E, Goldberg V M, Davy D, Parr J A, Stevenson S. The influence of surface-blasting on the incorporation of titanium-alloy implants in a rabbit intramedullary model. , J Bone Jt Surg Am. 1995; 77(9) p.1380-1395.
24. Rønold H J, Ellingsen J E. Effect of micro-roughness produced by TiO2 blasting-tensile testing of bone attachment by using coin-shaped implants. Biomaterials. 2002; 23(21), p.4211-4219.