ASSESSMENT OF SURFACE AREA CHARACTERISTICS OF DENTAL IMPLANTS WITH GRADUAL BIOACTIVE SURFACE TREATMENT

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Abstract
Since most of the implant surface is in direct contact with bone tissue, shape and integrity of said surface has great influence on successful osseointegration. Among other characteristics that predetermine titanium of different grades of pureness as ideal biomaterial, titanium shows high mechanical strength making precise miniature machining increasingly difficult. Current titanium-based implants are often anodized due to colour coding. This anodized layer has important functional properties for right usage and also bio-compatibility of dental implants. Physical method of anodizing and usage of anodizing mediums has a significant influence on the surface quality and itself functionality. However, basic requirement of the dental implant with satisfactory properties is quality of machined surface before anodizing. Roughness, for example, is factor affecting of time length of anodizing operation and so whole productivity. The paper is focused on monitoring of surface and area characteristics, such as roughness or surface integrity after different cutting conditions of miniature machining of dental implants and their impact on suitability for creation of satisfactory anodized layer with the correct biocompatible functional properties.

Keywords
dental implants, surface characteristics, bioactive surface, miniature machining

1 INTRODUCTION

Bone fusing to titanium was first reported in 1940 by Bothe et al. Branemark began extensive experimental studies in 1952 on the microscopic circulation of bone marrow healing. These studies led to dental implant application in early 1960; 10-year implant integration was established in dogs without significant adverse reactions to hard or soft tissues. Studies in human beings began in 1965, were followed for 10 years, and were reported in 1977. Osseointegration, as first defined by Branemark, denotes at least some direct contact of living bone with the surface of an implant at the light microscopic level of magnification. The percentage of direct bone-implant contact varies. [1, 2, 3]

The role of surface topography has been the interesting area of investigation in implant dentistry for several years. Several types of implant surface textures are currently available for clinical use. Some of these have the ability to enhance and direct the growth of bone and achieve osseointegration when implanted in osseous sites [2, 4, 5, 6].

Endosseous dental implants are available commercially with many different surface configurations. Most implant systems of this category are based on the fact that bone tissue can adapt to surface irregularities in the 1 – 100 microns range, and that altering the surface topography of an implant can greatly improve its stability [3, 7, 8, 9].

Surface treatments are normally carried out to modify yet maintain desirable properties of the substrate materials. The surface area can be increased remarkably by using proper modification techniques, either by addition or subtraction procedures [10, 11, 12, 13].

The additive methods employed the treatment in which other materials are added to the surface, either superficial or integrated, categorized into coating and impregnation, respectively. While impregnation implies that the material/chemical agent is fully integrated into the titanium core, such as calcium phosphate crystals within TiO2 layer or incorporation of fluoride ions to surface, the coating on the other hand is addition of material/agent of various thicknesses superficially on the surface of core material [10, 14, 15, 16, 17].

The coating techniques can include titanium plasma spraying (TPS), plasma sprayed hydroxyapatite (HA) coating, alumina coating, and biomimetic calcium phosphate (CaP) coating. Meanwhile, the subtractive techniques are the procedure to either remove the layer of core material or plastically deform the superficial surface and thus roughen the surface of core material. The common subtractive techniques are large-grit sands or ceramic particle blasts, acid etch, and anodical oxidation. [4, 18, 19, 20, 21, 22]

2 SETUP OF EXPERIMENTS

Experiment compares and closely examines surface structure and characteristics of milled area on crest module of dental implants with different level of surface treatment. All 5 samples of dental implants were made of Titanium Grade 5 (Ti6Al4V) by CNC machining center DIAMOND CSB 20. After machining, all implants (Sample
A, B, C, D, E) (Figure 1.) were passivated in 30% nitric acid for 20 minutes at 47°C to remove any dirt or remainings after machining tools. Furthermore, Samples B, C, D and E were then sand-blasted with beads of 100 µm diameter to produce a matte finish on the surface. Before further surface treatment, samples were soaked for 10 minutes in 70% ethanol and subsequently in deionized water for another 10 minutes. Samples C, D, E were then sand-blasted, anodized in 1.5 weight percent (wt%) hydrofluoric acid. During anodic oxidations, implant samples were placed by copper wires, connected to a voltage source, 2 cm away from titanium cathode. A constant voltage of 20V was applied for 5 minutes. After treatment with hydrofluoric acid, all samples were sonicated in 70% ethanol for 10 minutes, then in deionized water for 10 minutes, and then air-dried for an additional 10 minutes. Figure 1 shows resulting surfaces of implant samples (different surface treatments with various voltages change the color of titanium).

### Table 1. Surface treatments of experimental implant samples

<table>
<thead>
<tr>
<th>Material</th>
<th>Initial surface finish</th>
<th>Surface treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>passivation HN\textsubscript{2}O\textsubscript{3}</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>passivation HN\textsubscript{2}O\textsubscript{3}</td>
<td>sand-blasted</td>
</tr>
<tr>
<td>C</td>
<td>passivation HN\textsubscript{2}O\textsubscript{3}</td>
<td>sand-blasted, anod. in H\textsubscript{2}SO\textsubscript{4}</td>
</tr>
<tr>
<td>D</td>
<td>passivation HN\textsubscript{2}O\textsubscript{3}</td>
<td>sand-blasted, anod. in H\textsubscript{2}SO\textsubscript{4}. anod. in HF</td>
</tr>
<tr>
<td>E</td>
<td>passivation HN\textsubscript{2}O\textsubscript{3}</td>
<td>sand-blasted, anod. in H\textsubscript{2}SO\textsubscript{4}. anod. in HF, etching in HF</td>
</tr>
</tbody>
</table>

### 3 RESULTS OF EXPERIMENTS

On the resulting surfaces of experimental dental implant samples were made photographs and measurements of profile, roughness, surface properties using Alicona InfiniteFocus device. It works on the principle of non-contact, optical, three-dimensional measurement based on focus variations in Table 2 and 3.

### Table 2. Measurement of surface area roughness

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Sample C</th>
<th>Sample D</th>
<th>Sample E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sa [µm]</td>
<td>0.786</td>
<td>1.113</td>
<td>0.995</td>
<td>1.546</td>
<td>0.863</td>
</tr>
<tr>
<td>Sq [µm]</td>
<td>0.978</td>
<td>1.597</td>
<td>1.299</td>
<td>1.954</td>
<td>1.133</td>
</tr>
<tr>
<td>Sp [µm]</td>
<td>4.07</td>
<td>23.74</td>
<td>13.46</td>
<td>10.75</td>
<td>14.93</td>
</tr>
<tr>
<td>Sv [µm]</td>
<td>2.98</td>
<td>15.05</td>
<td>7.25</td>
<td>6.87</td>
<td>5.539</td>
</tr>
<tr>
<td>Sz [µm]</td>
<td>7.06</td>
<td>38.80</td>
<td>20.71</td>
<td>17.62</td>
<td>20.47</td>
</tr>
<tr>
<td>S10z [µm]</td>
<td>6.53</td>
<td><strong>30.44</strong></td>
<td><strong>16.43</strong></td>
<td><strong>15.27</strong></td>
<td><strong>19.43</strong></td>
</tr>
<tr>
<td>Ssk</td>
<td>0.39</td>
<td>1.52</td>
<td>0.106</td>
<td>0.904</td>
<td>0.794</td>
</tr>
<tr>
<td>Sku</td>
<td>2.75</td>
<td>13.84</td>
<td>4.82</td>
<td>3.12</td>
<td>11.87</td>
</tr>
<tr>
<td>Sdq</td>
<td>0.114</td>
<td>0.329</td>
<td>0.229</td>
<td>0.107</td>
<td>0.136</td>
</tr>
</tbody>
</table>

- Sa – Average height of the selected area
- Sq – Average quad. height of the selected area
- Sp – Maximum peak height in the selected area
- Sv – Maximum depth of the pit in selected area
- Sz – Maximum height in the selected area
- S10z – Ten-point height of the selected area
- Ssk – Obliquity of the selected area
- Sku – Spikiness of the selected area
- Sdq – Average quadratic gradient
- Sdr – Expanded ratio of interface

### Table 3. Measurement of surface area texture

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Sample C</th>
<th>Sample D</th>
<th>Sample E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sk [µm]</td>
<td>2.550</td>
<td>3.20</td>
<td>3.07</td>
<td>4.97</td>
<td>2.76</td>
</tr>
<tr>
<td>Spk [µm]</td>
<td>0.830</td>
<td>2.631</td>
<td>1.511</td>
<td>1.552</td>
<td>1.116</td>
</tr>
<tr>
<td>Svk [µm]</td>
<td>0.856</td>
<td>1.671</td>
<td>1.459</td>
<td>1.877</td>
<td>1.255</td>
</tr>
<tr>
<td>Smr1 [%]</td>
<td>9.880</td>
<td>10.91</td>
<td>10.31</td>
<td>7.740</td>
<td>9.110</td>
</tr>
<tr>
<td>Smr2 [%]</td>
<td>90.20</td>
<td>89.09</td>
<td>89.29</td>
<td>88.94</td>
<td>89.81</td>
</tr>
<tr>
<td>Vmp [ml²]</td>
<td>0.041</td>
<td>0.135</td>
<td>0.075</td>
<td>0.078</td>
<td>0.055</td>
</tr>
<tr>
<td>Vmc [ml²]</td>
<td>0.903</td>
<td>1.145</td>
<td>1.085</td>
<td>1.721</td>
<td>0.957</td>
</tr>
<tr>
<td>Vvc [ml²]</td>
<td>1.205</td>
<td>1.596</td>
<td>1.484</td>
<td>2.188</td>
<td>1.264</td>
</tr>
<tr>
<td>Vvv [ml²]</td>
<td>0.105</td>
<td>0.173</td>
<td>0.158</td>
<td>0.232</td>
<td>0.135</td>
</tr>
<tr>
<td>Vvc/Vmc</td>
<td>1.333</td>
<td>1.393</td>
<td>1.367</td>
<td>1.271</td>
<td>1.321</td>
</tr>
</tbody>
</table>

Final list of sample surface treatments is stated in Table 1.
Sk – depth of core roughness, height of core material
Spk – Reduced peak height, medium peak height above core material
Svk – Reduced pit height, medium pit height below core material
Smr1 – Peak’s material component, a part of the surface that consists of peaks above the core material
Smr2 – Peak’s material component, a part of the surface that bears the load
Vmp – Volume of topographic surface peak’s material
Vmc – Volume of topographic surface core material
Vvc – Volume of spacing in the surface area
Vvv – Volume of pits in the surface area

In Figure 2, we can see a comparison of the achieved surface area roughness with parameter Sa, where the lowest value of the parameter is obtained by the Sample A, where $Sa = 0.786 \mu m$. This value gradually changes with more surface treatments. The highest Sa value was obtained by Sample D, where $Sa = 1.546 \mu m$. We can also see, in figure 2 the comparison of achieved depth of core roughness and height of core material – Sk, where the lowest value is obtained by Sample A, where $Sk = 2.55 \mu m$. This value gradually changes with more surface treatments as well. The highest Sa value was obtained by Sample D, where $Sk = 4.97 \mu m$. By studying the parameters Svk and Vvv, we found, that surface treatments have positive influence on pits (Figure 3).

Figure 2. Average height of the selected area - Sa and depth of core roughness, height of core material – Sk

Figure 3 Reduced pit height, medium pit height below core material – Svk and Volume of pits in the surface area – Vvv

The surface texture was then monitored by the scanning method for the relief recognition. Figure 6 refers layout of the pits (dark red and purple) and peaks (yellow and orange). Given pits are necessary to ensure porosity and hydrophobic behavior of the surface. In Figure 4, we can see layout and homogeneity of sample surfaces. The most favorable results appear on Samples B and E.
Figure 4. Scanning of surface with graphic distinction of relief

An enlarged view of Sample A shows, that after passivation of titanium surface, traces after milling tool are well visible. Passivation as such does not change the character of the machined titanium surface, it only removes iron impurities and debris left after machining. When sanding a machined titanium surface (Sample B) with particles of 100 µm diameter, machining track are removed and the Ti6Al4V grain structure starts to appear. This creates the basis for further surface treatments, creating finer structures. The first anodic oxidation using H2SO4 (Sample C) begins to transform the surface character, first signs of the porous structure appear. After anodic oxidation using HF (Sample D), a regular structure of small pores with 1-2 µm diameter appears on the sample surface. Etched Sample E has a similar structure to Sample D, with nanometer-sized elements. The pore structure is slightly more pronounced Figure 5.

Sample A

Sample B

Sample C

Sample D

Sample E

Figure 5 Surface texture created by different treatment methods

CONCLUSION

This paper presents a study of the process of creating special surfaces for invasive dental implants which provide surface functionalization and specifically improvement of properties such as implant-tissue biocompatibility as well as rapid healing of the site by investigating surface functionalization in the form of porosity and improvement of surface treatment for more hydrophobic behavior. Process of surface treatments was studied by application of various technologies such as passivation, sand-blasting, anodic oxidation and etching of titanium alloy TiGr5, material commonly used in biomedicine. Process of surface transformations was performed in controlled environment, using various acids and etchants. Improvement of surface treatments was mainly focused on surface smoothing and removal of surface layers after machining. The significance of this result is obvious given the already good initial roughness of the material for previous machining. Real achieved value of Average height of the selected area was Sa = 0.7861 µm for untreated surface of Sample A. Application of new surface treatments gradually changed area roughness as well, from Sample E (Sa = 0.8635 µm) through Sample C (Sa = 1.113 µm) up to Sample D (Sa = 1.5461 µm).

As far as fulfillment of surface functionalization in the form of porosity is concerned, that is characterized by parameters such as Sk – Depth of core roughness, height of core material, Spk – Reduced peak height, medium peak height above core material and Smr1 – Peak's material component, a part of the surface that consists of peaks above the core material. From the measured values of the individual parameters, it is possible to determine the change in surface properties, where the highest change was recorded for samples B and C and the most positive changes in sample D. The changes of surface characteristic such as treatment for more hydrophobic behavior are possible to study via area texture parameters such as Svk – Reduced pit height, medium pit height below core material, Smr2 – Peak's material component, a part of the surface that bears the load, Vvv – Volume of pits in the surface area. By observing the given parameters, it was found that the largest depths were obtained by sample D. The very fact of the occurrence of porosity with hydrophobic behavior can also be noticed in surface visualization, where we can clearly confirm the formation of pore-shaped nanotubes. These nanotubes have a high assumption of significantly higher hydrophobic surface, especially in samples D and E. Creating of controlled surfaces with a high degree of hydrophobic behavior is an excellent opportunity for drug dosing directly in the texture of the surface, thereby positively influence biocompatibility, but in particular, increase the efficiency of implant acceptance without complications. Presented study has shown, that the gradual anodization of Ti6Al4V using sulfuric acid, and subsequently hydrofluoric acid, produces a unique microporous surface with tubes of 1-2 µm diameter, which promotes osteoblast density, alkaline phosphatase activity and calcium deposition while retaining attractive color coding properties.
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**Used sources**


