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Surface Properties of Different Heat Treated Titanium Alloy Dental Implants

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Abstract: ***Titanium alloy surface properties have an essential role in the interaction of dental implants with bone, and alteration of the surface of the implant could improve osseointegration. This study was designed to investigate the effect of different heat treatment temperatures on titanium alloy surface properties for dental implants. The effect of different temperatures of heat treatment (750°C, 850°C, 950°C and 1050°C) were investigated on the surface topography, surface chemistry, titanium oxide layer thickness, blood contact angle, & blood drop diameter of titanium alloy. The disks were prepared from titanium alloy (Ti-6Al-4V) and the samples were divided into five groups depending on the different temperatures of heat treatment. The heat treatment at 1050 °C for 30 minutes significantly enhanced the titanium alloy surface characteristics; surface topography, titanium oxide layer thickness, surface chemistry, blood contact angle, and blood drop diameter. This may result in faster and stronger bone formation around dental implants.***

Keywords: Titanium Alloy, Heat Treatment, Surface Properties

1. Introduction

Implant surface modifications continue to undergo advancement to achieve osseointegration, with quicker formation of stronger bone. Also, better stability with accelerated bone formation favor immediate or early loading of dental implants in these areas.[1]

The future success of dental implants depends on the reaction of the dental implant surface with living human tissue. Therefore, the surface properties and the reaction of the dental implant with the tissue is critical in determine the success of implantation. Biologically, the host bone response is influenced by the titanium implant surface. This surface is merely part of the dental implant that contacts the host bone, and differs from the main body of the dental implant in many aspects.[2] This uniqueness of the dental implant surface could manage the healing process at the interface involving the bone and the implant.[3]

There are three methods of modification of the titanium implant surface; mechanical, chemical, and physical, thus altering titanium surface topography, chemistry, and structure. The goals of such methods are to improve the bio-mechanical properties of the titanium implant like removal of surface contaminants, improvement of wear and corrosion resistance, and stimulating bone formation.[4,5]

Osseointegration may be affected by the surface chemistry, surface topography, surface wettability, and thickness of titanium oxide layer.[1,6] Wettability of the surface may influence the rate and the strength of osseointegration and could be determined by the measurement of contact angle that forms between the drop of liquid and the surface of titanium alloy.[7-9]

The surface of titanium is mainly covered by a spontaneous titanium oxide layer that is thin (5-10 nm) and forms immediately after being subjected to air. The oxide layer on the titanium surface is responsible for the titanium biocompatibility since it protects the underlining metal from corrosion and inhibits the release of any metal particles that could be toxic and could result in failure of the implant.[4]

Several researches were conducted to investigate the ability of heat treatment to improve the titanium alloy surface properties. It was found that the heat treatment at 750°C for 30 minutes was effective and produced better surface characteristics, and better biocompatibility of the TiO2 layer.[4,10] Another study conducted by Abass[11] investigated the influence of heat treatment at different durations on surface properties of titanium alloy. In this study the titanium alloy showed improvement of its surface properties with increase in the duration of heat treatment. Duration of 90 minutes at 750°C showed the greatest improvement in the surface properties.

This research was intended to study the effect of several heat temperatures (750°C, 850°C, 950°C and 1050°C) for 30 minutes on the thickness of the titanium oxide layer (TiO2), surface chemistry, surface topography, contact angle, and blood drop diameter of grade 5 titanium-aluminum-vanadium alloy (Ti-6Al-4V).

The hypothesis for this research was that as the temperatures increase the surface properties of the titanium alloy would improve.

2. Materials and Methods

Disk shaped titanium samples of 6mm x 1mm thickness were prepared from a Titanium-Aluminum-Vanadium (Ti-6Al-4V) alloy. These disk shaped titanium samples underwent finishing with the use of 400 and 1200 silicon carbide grit paper. The cleaning process included immersion of the titanium samples for 10 minutes in acetone and later ethanol with rinsing in deionized water in between and after each solvent.[10] Afterwards, the samples were stored in a desiccator.[12]

There were five sample groups in this study and each group included five samples. Thus, there was the control group (**I**) with no treatment, group (**II**) treated with heat at 750 C° for 30 minutes, group (**III**) treated with heat at 850 C° for 30 minutes, group (**IV**) treated with heat at 950 C° for 30 minutes, and group (**V**) treated with heat at 1050 C° for 30 minutes. A heavy duty box furnace (CARBOLITE, Parsons Lane, Hope, England) was used for the treatment with heat.

The samples were placed in a desiccator immediately after heat treatment till the time of testing.

Different dental implant surface characteristics were tested in this study; surface topography, titanium oxide layer thickness, surface chemistry, blood contact angle, & diameter of blood drop.

1) Surface topography: The surface topography was observed by using the scanning electron microscopy (VEGA Easy Probe SEM ™).
2) Titanium oxide layer thickness: In order to evaluate the thickness of the titanium oxide layer, the disk shaped titanium samples were placed in self-cured acrylic resin and then sliced in the middle to obtain a cross-sectioned view that could be examined by the scanning electron microscopy (VEGA Easy Probe SEM ™).[11]
3) Surface chemistry: The X-ray diffractometer (XRD-6000 Shimadzu X-ray Diffractometer) was used to assess the surface chemistry of the test groups.
4) Blood contact angle measurement: A drop of 2 µl volume of human blood was placed on the surface of the titanium disk sample. A picture was taken for the side view of the human blood drop at different intervals of time after placement on each disk sample surface. The Corel Draw X3 analyzing software was used for measuring of the contact angle. [11-13]
5) As for the diameter of the blood drop, a top view picture was taken for the blood drop formed over the surface of the titanium sample. The blood drop picture was analyzed with the software program Dimaxis version 2.3.3. to measure the diameter of blood drop formed on the titanium sample surfaces.

3. Results

Surface Topography

The surface topography of the control group showed an even and regular crystal pattern. The crystals of the surface of (group II) were rougher, irregular, and larger than (group I). The surface topography of (group III) was the same as (group II) but rougher and larger. The shape of the crystal (group IV) became more irregular, and pointed. In the (group V) the crystals had the same shape of (group IV) but more sparkler and larger in size, as shown in figure (1).

Figure 1: Surface topography for test groups I, II, III, IV, and V from left to right (SEM mag. 10 kx).

Titanium Oxide Thickness

The thinnest oxide layer was for test group I (1.99µm) and this significantly increased, $p<0.05$, as the heat treatment temperature increased, when compared to the control group, to became the thickest for test group IV (14.30µm), then it declined for group V (11.56 µm), figure (2&3) and table(1&2).

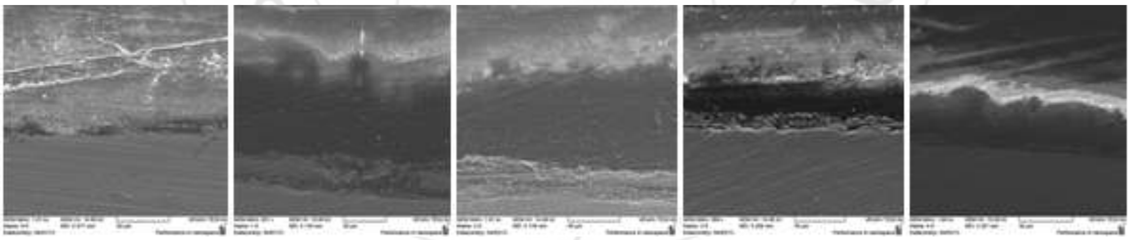

Figure 2: SEM pictures of cross section showing the titanium oxide thickness for test groups I, II, III, IV, V from left to right (SEM mag. 1.01 kx)

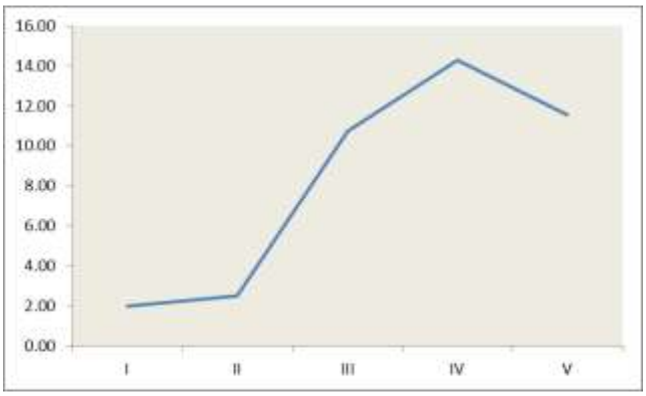

Figure 3: Oxide layer (TiO_2) thickness (µm) for all test groups.

Table 1: One- way ANOVA of the oxide layer (TiO_2) thickness for all test groups

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	377.833	4	94.458	1613.13	0
Within Groups	0.586	10	0.059		
Total	378.419	14			

Table 2: Multiple Comparisons LSD for the oxide layer (TiO_2) thickness (µm) for all test groups

Test Groups	Mean Difference	Std. Error	Sig.
I-II	-.51459*	0.19758	0.026
I-III	-8.77409*	0.19758	0
I-IV	-12.30351*	0.19758	0
I-V	-9.56254*	0.19758	0

*. The mean difference is significant at the 0.05 level.

Surface Chemistry

XRD patterns of the heat treated samples at different temperatures revealed the appearance of additional peaks which may be related to the formation of new phases; anatase (II), anatase-rutile (III, IV), and rutile (V), as seen in figure (4). The rutile phase became more prominent than anatase in higher temperatures. The peak of titanium occurred in the XRD patterns for the control group and disappeared in the XRD patterns for the test groups. As the temperatures increased the peaks became more tapered and meant that the chemical structure of the titanium alloy changed toward the amorphous structure.

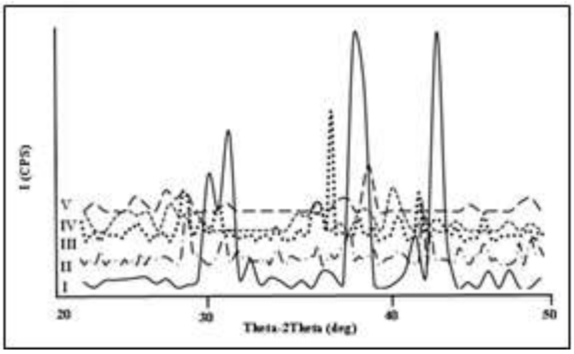

Figure 4: XRD patterns of test groups

Contact Angle

The highest blood contact angle was for group I and the lowest was for test group V, see figure (5) & (6). The blood contact angle significantly ($p<0.01$) decreased as the temperature of heat treatment increased (table 3&4).

Figure 5: Contact angle for all test groups; I, II, III, IV, V from left to right

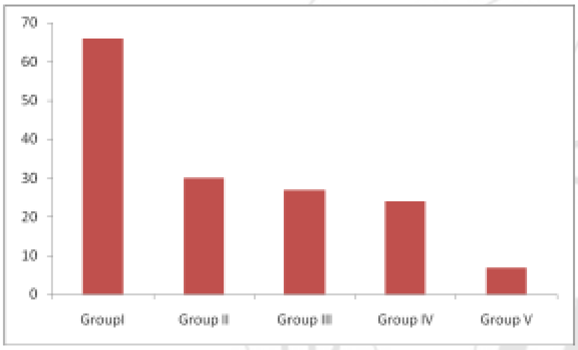

Figure 6: The mean of contact angle for all test groups.

Table 3: One- way ANOVA of contact angle for all test groups

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	9291.035	4	2322.759	293.695	0
Within Groups	158.175	20	7.909		
Total	9449.21	24			

Table 4: Multiple Comparisons LSD of contact angle for all test groups

Test groups	Mean Difference	Std. Error	Sig.
I-II	35.75000*	1.77862	0
I-III	39.10000*	1.77862	0
I-IV	41.50000*	1.77862	0
I-V	58.95000*	1.77862	0

*. The mean difference is significant at the 0.05 level.

Blood Drop Diameter

The mean of the diameter of blood drop (mm) increased as the temperature of heat treatment increased (figure 7 &8). This increase was highly significant according to ANOVA and LSD tests, $p<0.01$ (Table 5 & 6).

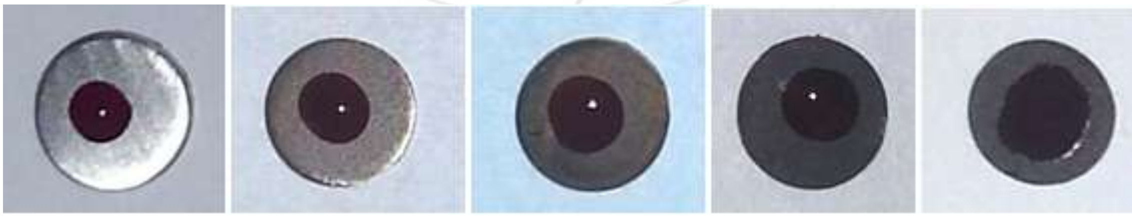

Figure 7: Blood drop diameter of test groups I, II, III, IV, V from left to right

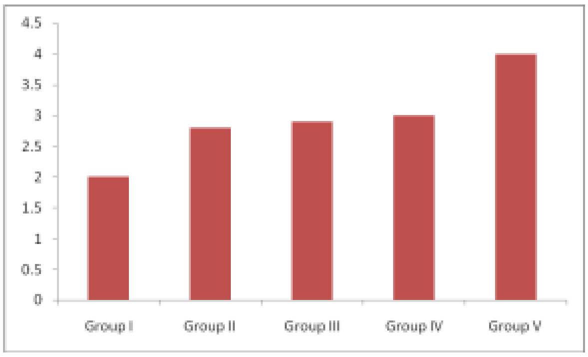

Figure 8: The mean of diameter of blood drop (mm) for all test groups

Table 5: One-way ANOVA of diameter of blood drop for all test groups

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	7.677	4	1.919	174.851	0
Within Groups	0.22	20	0.011		
Total	7.896	24			

Table 6: Multiple Comparisons LSD of diameter of blood drop for all test groups

Test groups	*Mean Difference (I-J)*	*Std. Error*	*Sig.*
I-II	-.65200*	0.06626	0
I-III	-.81000*	0.06626	0
I-IV	-.83400*	0.06626	0
I-V	-1.73400*	0.06626	0

*. The mean difference is significant at the 0.05 level.

4. Discussion

This research showed an improvement in the surface characteristics of titanium alloy involving the surface topography, titanium oxide layer thickness, surface chemistry, blood contact angle, & diameter of blood drop for all the test groups that were exposed to high temperature of heat treatment. There were variations in these improvements among these test groups. These improvements were amplified as the temperature of heat treatment increased, to achieve greatest improvement at 1050°C for 30 minutes. So the hypothesis of this research “the surface properties of the titanium alloy improved as the temperature of the heat treatment increased” was accepted.

Figure (1) showed that the surface irregularities of heat treated titanium alloy increased as the temperature of heat treatment increased, and the shape of the surface crystals became more irregular and sparkler and their size became bigger. At higher heat treatment temperature titanium alloy underwent transition into different crystal phases as noticed by Lee et al.[10] This coincided with the findings of Pookmanee & Phanichphant[14], Ninsonti et al.[15], & Abass[11] who all found that the heat treatment of titanium alloy was associated with increased irregularities of the surface topography.

As the temperature of the heat treatment increased the thickness of titanium oxide layer increased, figure (2). This may be clarified by the reality that the titanium medium fortified with oxygen and the solubility of oxygen to the titanium medium enhanced as the temperature of heat treatment got higher.[16] This coincided with the outcomes of Lee et al.[10] and Abass.[11]

Different crystalline phases, anatase & rutile, of titanium oxide appeared in the XRD patterns of the heat treated samples at different temperatures, figure (4). The rutile phase became more prominent than anatase phase in higher temperatures.[17] This may explain the color change of the surface of the heat treated titanium alloy. The discoloration of the surfaces of the heat treated titanium samples varied from beige to dark brown. This may be associated with the formation of rutile phase which increased as the temperature of the heat treatment increased, so the color became darker as the rutile phase increased. This was in agreement with the findings of Abdolldhi et al.[16], MacDonald et al.[18], lee et al.[10], and Abass[11].

As the temperature of the heat treatment increased, there was a decrease in the blood contact angle and an increase in the diameter of blood drop. The increase in the surface roughness and alteration in surface topography that was seen with the SEM (figure 1) could be responsible for the improvement in surface wettability of the titanium sample surfaces which could enhance its biocompatibility.[18,19,21] This finding is in agreement with results of Lee et al.[10] & Abass[11]. Also, it may be related to the presence of titanium oxide layer that increased wettability.[20] The oxide layer increased in thickness as the temperature of heat treatment increased as was confirmed in the SEM. This agreed with the Lee et al.[10], & Abass [11]. Another reason may relate to the fact that the crystallity of titanium surface increased as the temperature increased with change in the surface chemistry, as founded by the XRD. This change in the chemical structure and its effect on the wettability was also observed by Scharnweber et al.[21], Pegueroles et al.[22], and Abass [11].

5. Conclusions

Treating titanium alloy with heat at temperatures of at 1050 °C for 30 minutes allowed for improvement of many surface characteristics. Such enhancement could allow for better and faster formation of bone around the dental implant.

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