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Alkali Etch And Heat Treatment, Heat Treatment As A Subtle Treatment For Dental Implant Surfaces.

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ABSTRACT

INTRODUCTION

This study was conducted to evaluate the effect of different surface modifications on titanium topography, the aim of this study was to explore the effect of treating titanium dental implant surface with alkali etching and heat treatment, heat treatment on titanium dental implant topography.

MATERIALS AND METHODS

Pure polished titanium discs were immersed in 5.0 M Sodium hydroxide (NaOH) in a 60 ˚C water bath for 24 hours. Alkali etched discs were heated to 600 ˚C for 1 hour in a furnace.

Titanium discs were heat treated in furnace at 750 ˚C. for 1 hour.

Surface roughness was measured using profilometry. Topography was examined using scanning electron microscopy. X-ray photoelectron spectroscopy was used to determine the percentage of elements on titanium surfaces.

RESULTS

A dramatic changes in the morphology of titanium discs was visible to the naked eye.

heat treated surfaces were rougher than alkali etched/heat treated surfaces (higher Ra value) with highly significant differences between them $P<0.001$.

The percentage of element composition in heat treated titanium surfaces showed decreased surface contamination with carbon and increased percentage content titanium when compared with polished titanium control. While carbon percentage in alkali etched/heat treated titanium surface was more than control polished titanium surface. These increases in carbon occurred at the expense of oxygen content.

CONCLUSIONS

Titanium is an amenable material and it is possible to modify the surface using a variety of protocols and modification of titanium surfaces using alkali etching/heat treatment, heat oxidation produced subtle changes in surface roughness.

I. INTRODUCTION

Dental implants have become an important option in treatment plans within dentistry to replace missing teeth. However, implant failure and peri-implant diseases are still a problem facing implantologists. The long term success of an implant is hieghly dependent upon the ability of the material to integrate with the surrounding bone and connective tissue.[1] Many efforts have been made to develop materials that can accelerate osseointegration,[2] enhance gingival attachment to provide a soft tissue seal that prevents bacterial invasion,[3] and resist bacterial adhesion and colonization and/or having bactericidal effects.[4]

Good adhesion of gingival tissue to a dental implant can be a factor in determining the long term success of the implant. The gingival adaptation is mediated by the junctional epithelium and it is important to have a strong attachment of the implant surface not only to the surrounding bone but also to the gingival tissue.[5]

The ideal transmucosal part of the dental implant should inhibit bacterial adhesion and also enhance good epithelial and connective tissue adhesion.[4,5] There are many factors that can affect gingival attachment like the surface characteristics (such as topography and hydrophobicity). These in turn will affect cellular adhesion, proliferation, differentiation, and functional changes of cells.[5] A certain amount of roughness (pore or micro texture surface) is essential for promotion of tissue ingrowth. A surface roughness value of 0.2 µm seems to be the ideal surface roughness for the transmucosal part of a dental implant (threshold roughness). This roughness has been suggested to be the most suitable roughness to obtain a stable soft tissue seal around the supragingival part of a dental implant. If it is smoother than this it will prevent cell attachment.[5,6]

Several attempts have been made to form a biologically active bone like apatite layer directly from the titanium material to overcome the drawback of coating the titanium with a bioactive material. The treatment of titanium and titanium alloy with 10 M NaOH or 10 M KOH at 60 ˚C for 24 h, followed by heating in furnace to 600 ˚C for 1 h, dramatically increased apatite formation on the surface compared with untreated titanium.[7] A similar observation was reported later by Yan *et al* who in their *in vivo* animal studies showed that these modified active surfaces developed an intimate contact with the newly formed bone after 8 weeks, in which the gaps were filled with woven and lamellar bone. [8]

To investigate the importance of heat treatment following alkali etching on the bone bonding ability, Nishiguchi *et al.,* conducted a study with an animal model. They found that an alkali etched modified surface alone had no bone-bonding ability. The results of a histological study showed that the alkali etching and heat treated titanium specimens had a direct bone contact without any fibrous tissue at week 8 while for those having alkali treatment alone there was a thin fibrous layer present between the bone and titanium at week 8.[9]

Lee *et al.,* also modified titanium surfaces by alkali etching and heat treatment using 5.0 M NaOH solution, and heat treated in a furnace at 600°C.[10] Similar improvement in bioactive behavior of titanium alloys was showed in their *in vitro* study. Kokubo *et al.,* in their review described titanium after modifying with alkali etching and heat treatment, “Bioactive Metal” because of ability to form a bond to living bone. Spontaneous bone-like apatite layer could be formed on the surface of titanium *in vivo* using alkali etching and heat treatment, and demonstrated a bond between the bone and the apatite layer.[11]

The thermally oxidized titanium surface is probably the most cost effective surface treatment.[1] The thermal oxidization of titanium in air for 60 min at a temperature of 100-450 ˚C will result in a rutile oxide layer. The oxide thickness increase with increasing oxidation temperature and time duration. Simultaneously, surface contamination decreases and this might be related to decrease in the affinity for adsorption of hydrocarbons.[12]

This study was conducted to evaluate the effect of different surface modifications on titanium topography, the aim of this study was to explore the effect of treating titanium dental implant surface with alkali etching and heat treatment, heat treatment on titanium dental implant topography.

II. MATERIAL AND METHOD

Discs of 5 mm in diameter (± 0.1 mm) were punched out from 100 x 100 x 1 mm of annealed titanium sheets (99.6+%) (Goodfellow Cambridge Limited, Huntingdon, England). Discs were cleaned in an ultrasonic bath at room temperature with ethanol for 15 min then allowed to dry at room temperature.

Polishing procedure had been explained previously.[13]

Alkali etching and heat treatment

Titanium discs were immersed in 5.0 M Sodium hydroxide (NaOH) (BDH laboratory supplies, Poole, England) in a 60 ˚C water bath for 24 hours. They were then rinsed with distilled water and put in an oven at 40 ˚C for 1 h before allowed to dry at room temperature for 24 hours.[10]

Alkali etched discs were placed on an Alumina plate (PI-KEM LTD, Advanced Materials, Shropshire, England) and heated to 600 ˚C for 1 hour in a furnace[10] (ELITE thermal system limited furnace). The temperature was increased gradually at a rate of 5 ˚C/min until it reached 600 ˚C and was kept at this temperature for 1 hour. The discs were then allowed to cool inside the furnace.

Heat treatment

Titanium discs were heat treated using an ELITE thermal system limited furnace. The discs were placed on an alumina plate (PI-KEM. LTD, Advanced Materials, Shropshire,England) in the furnace. There was a gradual controlled increase in temperature at a rate of 5 ˚C/min until the furnace reached 750 ˚C. This temperature was maintained for 1 hour before being allowed to cool to room temperature within the furnace.[14]

After each surface modification the discs were ultrasonicated in ethanol for 5 min and then distilled water for 10 min at room temperature before being put in the oven at 40 ˚C for 1 and then allowed to dry at room temperature.

Scanning electron microscopy (SEM examination)

Topographic inspection was conducted using a scanning electron microscope (SEM Tech Ltd, Bonsall, Derbyshire, U.K.). All samples were attached by adhesive to aluminum SEM stubs and examined at 20 kV in the secondary emission mode in a PC-controlled ISI 60 scanning electron microscope.

Measuring surface roughness

A non contact optical Proscan profilometry was used to measure the surface roughness (Proscan 2000, Scantron Industrial Products Ltd. Monarch centre, Taunton, England). Four discs per surface were measured.

The measurement was conducted in an X and Y direction, and the scanned area was 2 x 2 mm. The measurement was taken for 20 lines in the Y axis; 250 spot in each line were measured twice. The final analysis was conducted in an area of 1.2 mm x 1.2 mm in order to avoid edge effect.

X-ray photoelectron spectroscopy (XPS) was used to determine the percentage of elements on titanium surfaces.

III. STATISTICS

Statistical analysis was carried out using Excel and SPSS (Statistical Package for Social Sciences). The statistical method uses to analyze and asses the results were: mean, standard error., ANOVA test.

After verification of the normal distribution and the homogeneity of the variance, an analysis of the variance (ANOVA) was used to asses any significant differences among selected group. For multiple comparisons test

(Post Hoc multiple comparisons) LSD (least significant difference) was used to determine the specific differences between the means of the group members.

The probability value (p-value) considered significant at $P < 0.05$ and highly significant if $P < 0.01$.

IV. RESULTS

The morphology and the surface roughness of titanium discs were analysed, and found to be different from one to another .

After heat oxidation, visible changes were observed on the titanium disc surfaces, in which the surface colour changed from bright mirror surface to bright, glassy, grey colour with smooth appearance.

While for the alkali etching and heat treatment the titanium disc surfaces changed from a bright mirror surface to a bright glossy-deep yellow, green, and purple according to light reflection which indicated an increase in oxide thickness.

Figure (1) illustrates the mean Ra value of heat treated and alkali etched/heat treated with polished surface and shows that heat treated surfaces were rougher than alkali etched/heat treated surfaces (higher Ra value) with highly significant differences between them $P<0.001$.

Figure (2) shows SEM images of these surfaces. The polished surface appears to be very smooth and homogenous with very few distinguishable features at low and high magnification. No obvious changes were seen in SEM pictures of polished titanium and alkali etched/heat treated titanium surfaces. While for heat treated titanium surfaces a creation of ridges across the surfaces were characterized.

The percentage of element composition in heat treated titanium surfaces showed decreased surface contamination with carbon and increased percentage content titanium when compared with polished titanium control Fig (3).

An increase in carbon percentage in alkali etched/heat treated titanium surface can be seen in Fig (3) carbon contamination was more than control polished titanium surface. These increases in carbon occurred at the expense of oxygen content.

V. DISCUSSION

The present results showed that different surface treatments produced different surface topographies; this is in agreement with previous work by Xavier *et al.*, Significant differences in surface roughness have been seen.[15]

In this study we also included some surface modifications which although not currently available on commercial implants, but have been reported to show promising characteristics in previous studies. Heat treatment and alkali etching/heat treatment are simple convenient, affordable procedures which appear to produce an enhanced bioactive titanium surface for osseointegration.

The aim of heat treatment is to produce a purely rutile oxide layer, as demonstrated by previous studies conducted by Gandhi [14] and Groessner-Schreiber *et al.*,[16] who produced purely rutile crystal surfaces on titanium surface. The Ra value for heat treated surface in this study was close to the values reported by Gandhi [14] who found an Ra value of 0.138 µm and Groessner-Schreiber *et al.*, [16] who reported Ra values of 0.11 ± 0.02. The results were slightly lower than those found by Größner-Schreiber *et al.*, [1]who found mean Ra values of 0.19

µm. However, these Ra values are in agreement with Chen *et al.*, who stated that the changes in surface roughness were on average of a tenth of micrometer. [17]

The Ra values for alkali etched/heat treatment were lower than that found by Nishiguch *et al.*, [9] 0.35 µm and this may be because their initial etched surface (0.32 µm) was rougher than our polished surface (0.047 µm). This may lead to an increase in surface roughness in two ways, the first explanation may be that the alkali etching/heat treatment procedure may start increase the original surface roughness affected by the original surface roughness, and the second explanation could be related to the fact that increase surface roughness enhance the action of etching. [19]

Heat treatment resulted in a remarkable decrease in the surface contamination, simultaneously with an increase in both titanium and oxygen levels. This has been reported previously by Olin *et al.*,[20]. Lausmaa showed that the amount of surface contamination decrease is in a linear relationship with temperature increase.[21]

The SEM images seen in this study were also different to those seen by previous authors [22,9,18] for the reasons stated above.

Surface contamination with a layer of different material (usually with organic material) is something that cannot be avoided. This layer is dynamic and its composition changes depending on the type of environment surrounding these surfaces.[21]

The presence of carbon as a contaminant material is normal as it can be adsorbed from air or may be a remnant of solvents used for cleaning.[12] The carbon percentage in the modified titanium surfaces ranged from 26.7-49.1% which was a relatively high percentage. Still this was lower than the range of carbon percentages found in commercially available implants (29.8-71.9 %).[23] This might indicate that the modifications in this study were better and less contaminated as they contained lower carbon contamination percentage. It is of note that commercially available implants have a roughness higher than the surfaces used in this study. This roughness sometimes leads to the over-estimation of the elements percentage value due to angular effects.

VI. CONCLUSIONS

Titanium is an amenable material and it is possible to modify the surface using a variety of protocols and modification of titanium surfaces using alkali etching/heat treatment, heat oxidation produced subtle changes in surface roughness.

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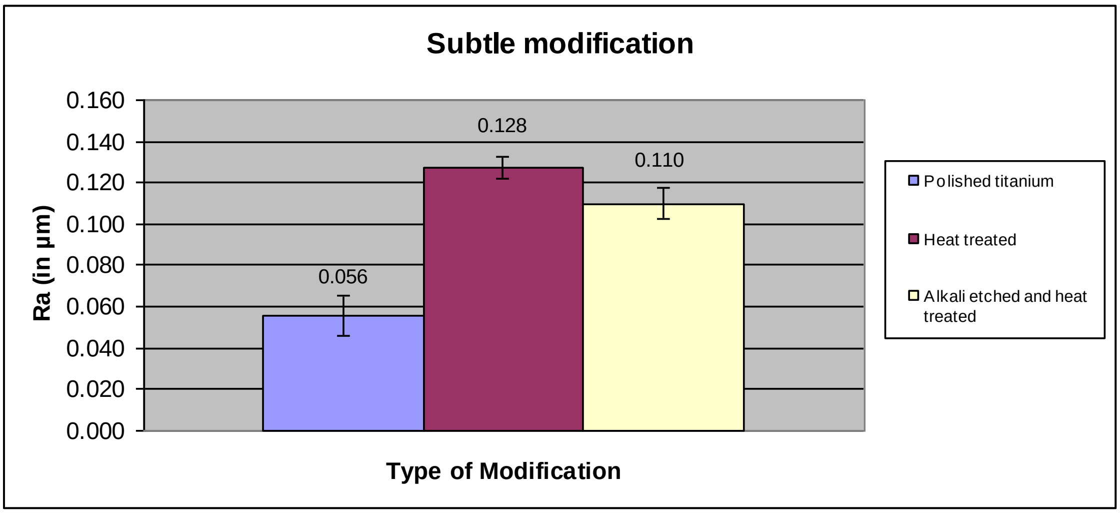


Over all P<0.001

Figure 1 Average Ra value (in µm) and standard error of polished titanium surface; heat treated titanium surface and alkali etched/heat treated titanium surface.

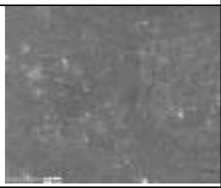
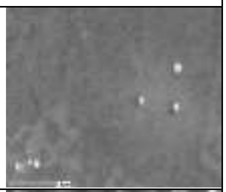
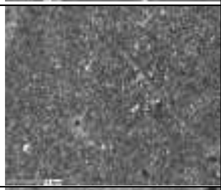
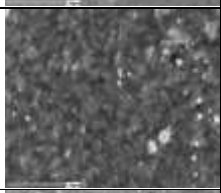
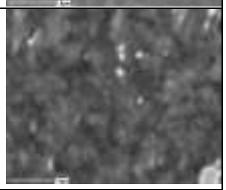
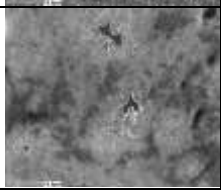
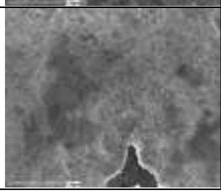
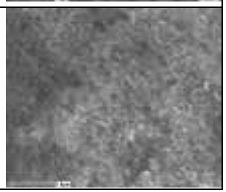
magnification / modification type		500	1500	5000	10000
1	Polished titanium				
3	Heat treated				
4	Alkali etched/heat treated				

Figure.2 SEM images of different modified titanium surfaces at different magnifications.

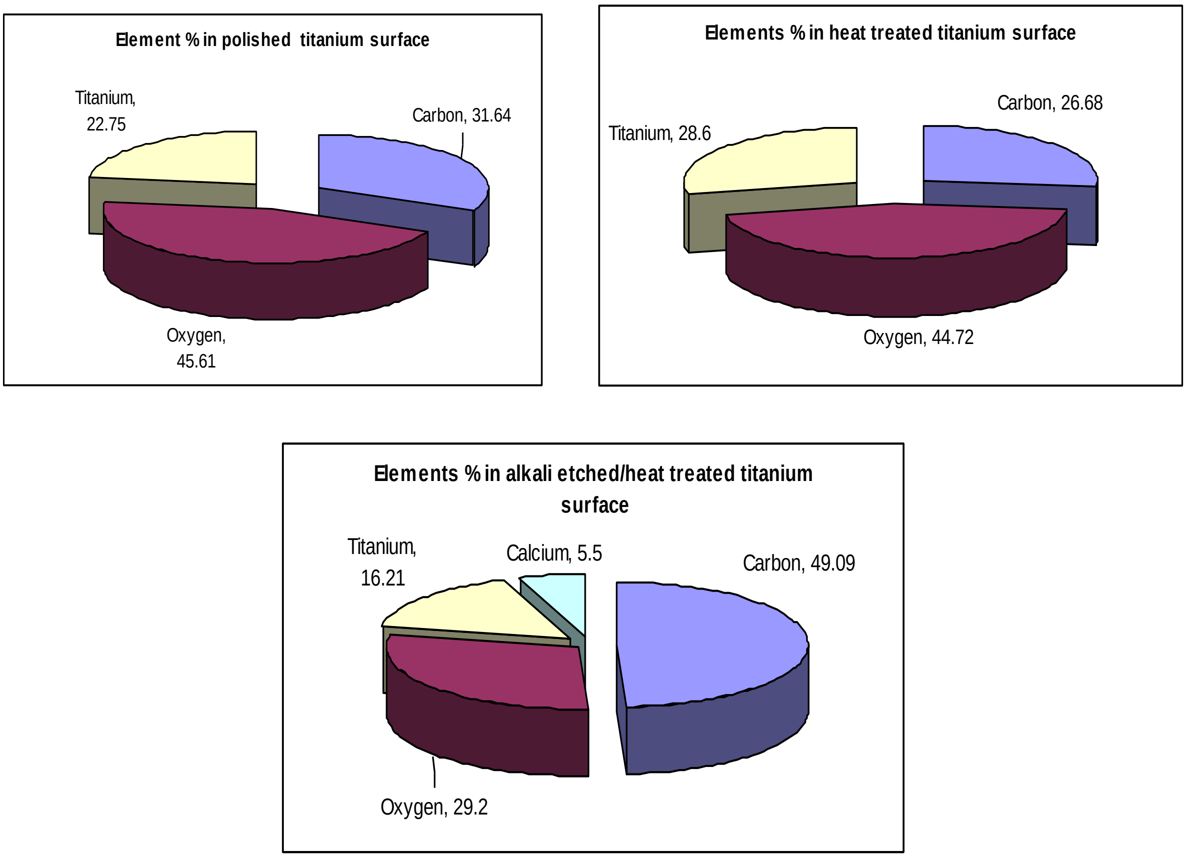


Figure 3. Percentage of elements in polished titanium, alkali etched/heat treated, heat treated titanium surfaces.