Mechanical Evaluation of Pure Titanium Dental Implants Coated with a Mixture of Nano Titanium Oxide and Nano Hydroxyapatite

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ABSTRACT

Background: The type of dental implant surface is one of many factors that determine the success of implant restoration. This study aimed to study the effect of mixture of nano titanium oxide with nanohydroxyapatite coating of screw shaped CPTi dental implant on bond strength at bone implant interface by torque removal test related to two healing periods (2 and 6 weeks).

Materials and methods: Dip coating process was performed to get an even coating layer on CPTi screws. X-ray diffraction (XRD) analysis and microscopical examination were performed on the coating surfaces of the CPTi. The tibia of 10 white New Zealand rabbits was chosen as implantation sites. The tibia of each rabbit received two screws, one was coated with mixture of nano$Tio_2$and nanoHA and the other was coated with nanoHA and a total of 40 screws were implanted. Torque removal test was performed to measure bond strength between implant and bone, after 2 and 6 weeks healing periods. .

Results: The results revealed, that the mean removal torque recordings for the mixture of nano$Tio_2$and nanoHA coated screws was significantly greatly than the nanoHA covered screws over the two periods of time(2and6 weeks). There was an increase in the torque value with time.

Conclusion: Commercially pure titanium implant coated with mixture of nanoTio_2andnanoHApresented an increasing bond strength at bone implant interface than nanoHA , after 2 and 6 weeks (20.13±4.4 N.cm , 26.47±4 N.cm.) in comparison to nanoHA coating after 2 and 6 weeks (15.16±2.5 N.cm , 20.12±2.3 N.cm) .

Key words: NanoTio_2, nanoHA, dental implant coating, Torque removal test. (J Bagh Coll Dentistry 2016; 28(3):38-43).

INTRODUCTION

Dental implants are the preferable treatment modality for restoring missing teeth, they made from commercially pure (CP) titanium that forms bio-inert titanium-oxide (Tio_2) on its surface, so multiple surface modifications have been made to improve the speed of bone attachment with the implant surface. These modified implant surfaces will improve the rate of osseointegration. One of those is a calcium-phosphate ceramic coating, which changes the bio-inert Tio_2surface into a bio-active surface.

The HA coating is associated with a number of problems, including delamination of the coat, cohesion and adhesion failures, and disintegration with the formation of particulate debris; these problems may be due to the porosity and the thickness of the coating, weak interfacial bonding, and the resistance of HA particles to biodegradation [(1)]. It is found that the good biological effect of a titanium implant is related to its passivating nature and the production of titanium oxide layer on the implant surface. There are many foundations that the titanium oxide layer is responsible for a good attachment of an implant to its surrounding tissue [(2,3)].

HA has a great interest as coating compound contributed to its high osteoconductivity. Many methods have been used to coat the metallic objects with HA and other calcium phosphate coated materials e.g., plasma spraying, sol-gel method, electron beam sputtering process, and ion beam sputtering process, all these methods possess weak results concerning coating with HA on complex-designed implants[(4)].

Titanium and its alloys were used in many bio implant applications. But they have some disadvantages, such as poor osteoinductiveability and decreased corrosive-wear resistance. Efforts to avoid the weak osteoinductivity included coating Titanium and its alloys with the bioceramic material as hydroxyapatite (HA), which is the same constituent of bone and a very good osteoinductor.

Coatings with TiO_2 act as active chemical barriers to prevent ions release from the metal implants. A mixture layer of HA–TiO_2 coating on titanium implant has best chemical stability, bioactivity, and mechanical integrity [(5)].

MATERIALS AND METHODS

Sample preparation for an in vitro experiments

Commercially pure Titanium (grade 2) was used as the substrate for coating. The titanium was cut into small circular discs (29mm diameter and 2 mm thickness) by lathe machine**.**

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Implant preparation

Forty screw shaped implants, 3.0 mm in diameter and 8mm in length (threaded part is 5mm and smooth part is 3mm) and pitch height is 1mm, were machined from Cp Ti rods using Lathe machine. The head of the implant had a slit to fit the screwdriver during insertion and removal by torque meter during mechanical testing

Preparation of coating solution

The coating solution of nanohydroxyapatite consists from dissolution of 0.01g of phosphorous pentoxide (p_2o_5) [3] in 50 ml of absolute alcohol (ethanol) with continuous mixing and heating (45° C)on a hot plate stirrer for half an hour, after that we added the nanohydroxyapatite powder(7g) to the solution, the temperature is maintained in the range of (45°C)and the mixture was left for halve an hour. The same procedure was done to prepare the (coating solution of mixture nano hydroxyapatite and nano titanium oxide), the amount of nanohydroxyapatite powder (5.27g)and nano titanium oxide powder (2.68g) [18,19].The material weighting was done by using analytical balance (Figure 1).

Figure 1: Coating solution preparation

Discs and screws coating

The coating of CPTi discs and screws was accomplished by dipping them in the nano coating solution for (5) seconds and withdrawal the specimens for one minute and drying them by hair dryer (Figure 2) then returned the specimens to the coating solution and repeated this method three times in the same way (Figures3,4)

Figure 2: Disc dryness by hair dryer

Heat treatment

Sintering of the coated specimens (thermally treated specimens) was carried out for densification using carbolated furnace (tube furnace).The treatment carried out for one hour under inert gas (argon), to prevent oxidation of the specimen. Best results were obtained at 400°C[20].

Figure 3: Coated CP Ti discs for in vitro study

Figure 4: Coated screws

Sterilization of screws

The screws were then sterilized with gamma irradiation at 2.5-3.0 megarad using gamma cells 220 withCo60source**.**

Tests performed

A. Microscopical examination: The appearance of each sample for each type of coating (two samples) was examined by using optical microscope.

B. X-Ray phase analysis: Phase analysis was employed on CP Ti discs before and after coating with different materials using (Shimadzu LabX-XRD- 6000). The peak indexing was carried out based on the JCPDS (joint committee on powder diffraction standards).

Sample grouping

A. Control group (20 screws): This group includes 10 screws for each healing interval (2 and 6weeks), coated with Nano HA.

B. Experimental group (20 screws): This group includes 20 screws for each healing interval (2 and 6 weeks), coated with mixture of Nano HA and Nano titanium oxide.

Surgical procedure

The total animals were divided into 2 groups for each healing interval (2 and 6 weeks) each group consists of 5 animals for mechanical test (torque removal test). Four implants (2:nano HA coated and 2: mix of nanoHA and nano Tio2 coated) were implanted in the tibia, (each tibia received one implant coated with nanoHA and the other one coated with mix of nanoHA and nano Tio2) consequently starting from the proximal to the distal side for each experimental tibia.

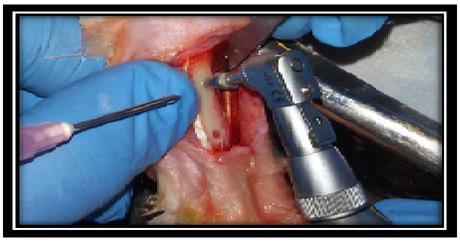

Figure 5:Hole preparation

Figure 6:Screw insertion (distance 1cm between 2 screws)

Mechanical testing

The stability of implants was checked. A torque removal test was done by engaging the head of torque meter into the slit in the head of the implant to determine the peak torque necessary to loosen the implant from the bone bed.

Radiographic evaluations

This test performed to the rabbit tibia after 2and 6 week after implantation to check if there were any problems in bone healing around the implanted screws.

RESULTS

Optical microscopical findings

The micrographs demonstrated the microstructure of nanoHA and a mixture of NanoHA and NanoTio$_2$and illustrated homogeneous thickness layer over the surface of titanium disc. There are no cracks in the coating layer and there are two phase in the layer of mixed material appeared (Figures 7,8).

Figure 7: Optical micrographic Nano HA coated disc surface

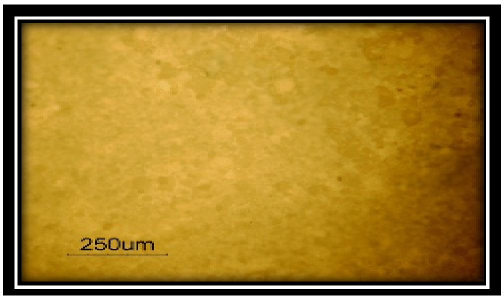

Figure 8: Optical micrographic mix. of Nano HA andnanoTio$_2$coated disc surface

<u>X-ray Diffraction of nanocoated Samples</u>

After dip coating, it is evident from figure (9) that the surface of specimen is well covered with HA and mix. of HA and Tio$_2$,because most of diffraction peaks could be indexed to HA and Tio2 phase according to JCPDS file, # 9-432 for HA file # 44-1294 for titanium, #21.1276 for Tio$_2$.

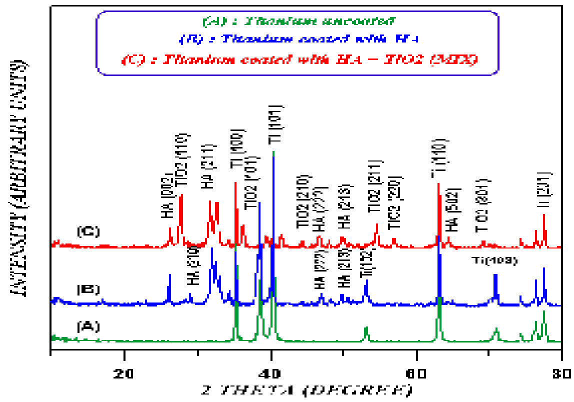

Figure 9:X-ray diffraction patterns of nano HA and mix of nano Tio$_2$and nanoHA coated CPTi specimen in comparison with the uncoated specimen.

<u>Mechanical testing</u>

The removal torque mean value nano mixture coated is (20.131N.cm) compared to the torque mean value of nanoHA coated implants (15.162 N.cm) after 2 weeks healing period. While after 6 weeks of implantation, a higher torque was required to remove the screws covered with a mix of nanoHA and nanoTiO_2mean recording(26.475 N.cm) compared to the torque mean value of nanoHA coated screws (20.121 N.cm). The summary of the differences in the torque mean values between all groups are shown inTable 1 and figure 10,the results revealed that the torque values increased with different healing periods for nano HA coated implants and a mix of nanoHA and nano Tio_2 coated implants

Table 1: Comparison of mean of torque value of mixture of nanoTio_2 and nano HA coated groups between 2 and 6 weeks healing periods

Types	Time	N	Mean	S.D	S.E	Min.	Max.
nano HA	2 weeks	10	15.162	2.562	0.810	12	19.6
	6 weeks	10	20.121	2.382	0.753	17.65	24.71
a mix of nano HA andnano Tio_2	2 weeks	10	20.131	4.412	1.395	17.65	13.77
	6 weeks	10	26.475	4.160	1.315	21.18	13.77

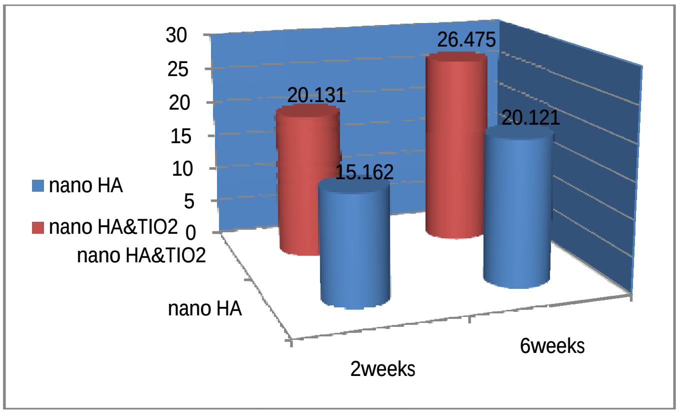

Figure10: Bar chart showed the summary of the differences in the torque mean values between all groups

t – test for equality of means of torque values demonstrated highly significant difference between nano HA and a mix of nano HA and nano Tio2 coated screws at 2 and 6 weeks healing periods**.** It was obvious that the torque value required to remove screws from the living bone bed was elevated with time (table 2).

Table2:t –test for equality of means of torque value for nanoHA and a mix of Nano HA and nano Tio_2 coated implants at 2 and 6 weeks healing periods

Types	Time	t-test	df	P value	Sig
nano HA	2 weeks x 6weeks	2.324	18	0.0003	HS
a mix of nano HA and nano Tio_2	2 weeks x 6weeks	4.028	18	0.0039	HS

Radiographic evaluations

The results of radiographic evaluation appeared that there were no areas of radiolucency between the nano coated implant and adjacent cortical bone in the radiographic examination and also there was an increase in thickness of cortical bone around the implants especially at 6 weeks healing periods as shown in figure11.

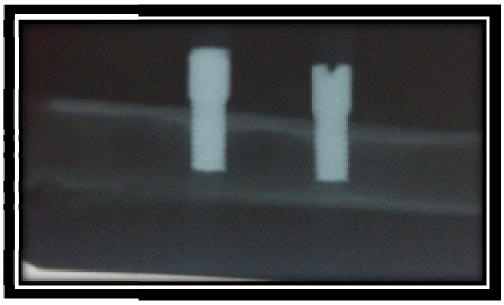

Figure 11: Radiographic view showed nano coated implant 6 weeks post implantation

DISCUSSION

Dip-Coating

Dip-coating process provides many advantages over other coating modalities like flexibility, control of coating morphology, chemistry and composition. If selected additives are used in this methodsphosphorouspentoxide (P_2O_5) added to the suspension as a thickening material) [6]

Improving the osseointegration of the metallic implants can be achieved by coating with thin biocompatible ceramic film [7]**.** Dip-coating method results in a homogeneous and pure coating also the lower processing temperature avoids the phase transition**.** It was found that the major problem of metallic implants coatings was their weak attachment to the metallic objects, the greatvariation between their thermal coefficients leads to the formation of tensile stresses at the contact zone. The dip-coating method and the electrodeposition process are considered as the best methods for producing ceramic coatings [8].

In the present study, dip-coating technique showed that the coating thickness increased with deposition time. The dip coating showed a thin continuous, uniform thickness layer of ceramic coating material proving that the dip process produces constant-thickness deposits on the implant surface. Ethanol was used for the dilution of the sols in this study to improve their stability, reduce the initial values of viscosity and avoid the agglomeration of particles, this in agreement with other findings [9-11].

Opticalmicroscopical observations

The microscopical testing carried out on the implants removed by the torque meter after 6 weeks of implantation, showed parts of bone adhered to the implants after unscrewing by torque meter, mainly the screws coated with mixture of nano TIO_2 and nano HA (5.27g of nano HA and 2.68 g of nano TIO_2), this could be a clear indication of good adherence and high bonding strength between the living bone and implant surface[9,12,13].

XRD phase analysis

It is evident from the figure of the XRD (Figure 9) that the surface of the specimens was well covered with nanoHA and mix of nano Tio_2-andnano HA layer because most of the diffraction peaks could be indexed to HA and Tio2**.**This finding agreed with others[9-11].

Radiographical examination:

It demonstrated direct contact between bone and implant, there was no radiolucent areas or any abnormal reaction to the implant. There are some increase in the thickness of cortical bone at experimental implant sites indicating increased bone formation and maturation around the(mixture of nano HAandnanoTio_2) coated implants after six weeks duration of implantation, this observation agree withother findings [7,8].

Mechanical test

Effect of coating materials on torque removal test: nano HA coating and nano mixture (HAandTio2) coating

The mixture of nano HA and nano Tio_2 coated cp Ti screws placed in rabbit bone recorded a higher mean of removal torque value than nanoHA coated screws at 2 weeks. This indicated increased bond strength at the bone–implant interface, the mixture of nano HA and nano Tio_2 stimulated bone formation more than nano HA.

Al-Mudarris [12] concluded that both the surface topography in the sub micrometer scale and oxide thickness influence the bone response to titanium. in this study good attachment is created between nano HA as" bone- like" material and nanoTIO_2as anticorrosive (oxide layer), the result be high torque value gained from screws coated by this material's compared to screws coated by nano HA only in both time 2 and 6 weeks. The higher amount of new bone formed by of mixture of nano HA and TIO_2 at 6 weeks was related to the fact that the new bone transformed to mature bone at 6 weeks together with the higher amount of new bone formation may reflect the higher bond strength at the implant bone interface and higher resistance to removal torque than the nano HA .

Effect of healing intervals on torque removal test

The present results illustrated that there was an increase in the removal torque value with time which may be due to progressive bone formation in bone-metal contact with time and remodeling around the implant during healing period that consequently improved the mechanical property of nano coated implant. This agreed with a removable torque studies in rabbit conducted by many authors [7,9,10,11,13-16].The force required to remove the implant from the living bone was greater with the increased implantation time. This could be related to increased shear strength, which resulted in stress transfer from the implant to the bordered bone, an even stress distribution between the implant and living bone, and reduced stresses in the implant [15].

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