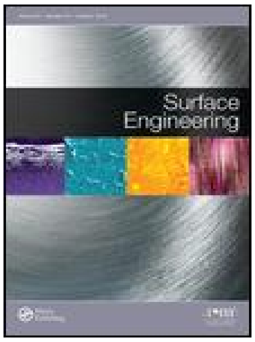


Surface Engineering

ISSN: 0267-0844 (Print) 1743-2944 (Online) Journal homepage: https://www.tandfonline.com/loi/ysue20

Experimental study of plasma nitriding dental implant surfaces

C. L. B. Guerra Neto, M. A. M. da Silva & C. Alves

To cite this article: C. L. B. Guerra Neto, M. A. M. da Silva & C. Alves (2009) Experimental study of plasma nitriding dental implant surfaces, Surface Engineering, 25:6, 430-433, DOI: 10.1179/174329408X271570

To link to this article: https://doi.org/10.1179/174329408X271570

Published online: 19 Jul 2013.

Submit your article to this journal

Article views: 23

View related articles

Citing articles: 6 View citing articles

Full Terms & Conditions of access and use can be found at
https://www.tandfonline.com/action/journalInformation?journalCode=ysue20

Experimental study of plasma nitriding dental implant surfaces

C. L. B. Guerra Neto*, M. A. M. da Silva and C. Alves, Jr

Titanium samples have been used as the substrate that was submitted to a plasma discharge nitriding for the surface treatment of dental implants. Two different arrangements regarding the electrode shapes were tested: planar and hollow cathode. The treated samples were evaluated regarding surface phases, texture, roughness, layer thickness, wettability and visual appearance. The roughness of the surface of all samples submitted to the plasma nitriding process was lower compared to those treated by commercially available technology plasma spray, on the other hand, better wettability was achieved. Some commercially available implants were also treated under the best conditions found with the experiment at titanium samples. After plasma nitriding these implants, tests were carried out to evaluate both wettability and surface texture. As a result the treated implants had both their surface texture and wettability improved.

Keywords: Implants dental, Plasma nitriding, Osseointegration, Biomaterials

Introduction

Dental implants have been used successfully in dentistry for many years. Recent research carried out in the topic has offered dentists useful information for reliable and efficient implant treatment.[1–4] In the consolidation of the clinical use of osseointegrated implants, research in implantology has focused on reducing or even eliminating the bone healing period established by Branemark's protocol.[5] To achieve this objective the main research topics have aimed engineering novel surfaces and examining their topographical characteristics. The surface to which bone cells are exposed at bone implant interfaces significantly affects osseointegration. Biocompatibility is another fundamental aspect to be considered.[6,7] It takes into account the response of the biological environment to biochemical stimuli and changes induced by foreign materials, as well as the physical and chemical response of the implanted material with respect to the biological environment.[7,8]

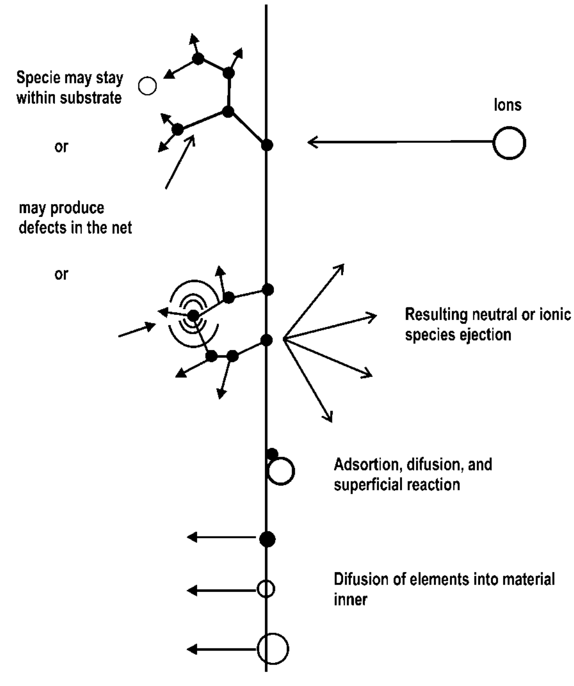
Different types of surface have been studied to fulfill such conditions. In particular, plain titanium[9] or titanium alloys have been submitted to different surface treatment.[10] In the present study, a technique new to the field of dentistry has used plasma as the energy source to modify titanium surfaces for potential dental implant applications. The formation of nitride layers instead of oxide surfaces and the use of a plasma atmosphere is a new approach to implant modification. In plasma nitriding, metallic surfaces are placed in contact with a plasma environment generated by applying a voltage between two electrodes. The electrodes are placed in a sealed reactor and surrounded by a nitriding gas (usually N_2–H_2 mixtures) at pressures varying from 1 to 10 mbar. The use of a hollow cathode configuration increases the plasma ion density making it more active. As a voltage is applied between the electrodes, electrons are accelerated towards the cathode, colliding with atoms and molecules of the gas, ionising atoms and exciting other species that form the plasma. Under these conditions, the plasma surrounds the cathode and the component is nitrided. As ions are accelerated towards the surface of the cathode, a myriad of effects is produced, including heating, creation of surface defects and surface sputtering,[11] as schematically illustrated in Fig. 1.

It is well established that wetting precedes the entire interactive surface/biomolecule/bone cells process necessary for osseointegration. Dynamic hydration of the surface and adhesion of biomolecule layers create a favorable scenario for the interaction of tissue and surrounding cells.[7,12] This process takes place because hydrophilic surfaces are strongly bonded to water. Proteins involving by water capsules consider the surface similar to water and interact with the material, thus remaining in their normal conformational state. As a result, a glycoproteic film is formed and attracts osteoblastic cells. Conversely, hydrophobic surfaces yield conformational changes and/or protein denaturation, resulting in growth of undesirable superficial cicatrisation tissue.[13] In summary, water intermediates osseointegration, confirming the importance of wetting to the process. Results obtained by nitriding Ti implant surfaces in plasma reactor are described in the present paper. Improved wetting can be achieved as compared to commercial implants, as a result of the topographical changes on the surface, characterised by microcavities and a columnar structure.

Universidade Federal do Rio Grande do Norte Centro de Tecnologia, Departamento de Engenharia Mecânica, Labplasma, Campus Universitário s/n, Lagoa Nova Natal, 59072970, Brazil

*Corresponding author, email custodioguerra@yahoo.com.br

© 2009 Institute of Materials, Minerals and Mining
Published by Maney on behalf of the Institute
Received 31 March 2007; accepted 12 October 2007
DOI 10.1179/174329408X271570



1 Mechanism of titanium plasma nitriding

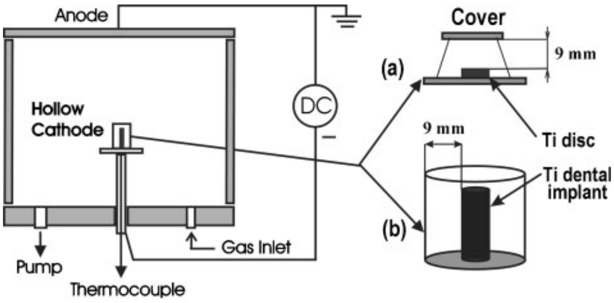
1 Mechanism of titanium plasma nitriding

Materials and methods

First, titanium samples (second grade) of 4 × 2 mm (diameter × height) were polished treatment until their average surface roughness R_a, reached values of 0, 22 μm. Next, the samples were cleaned in an acetone ultrasonic bath and then introduced into the plasma nitriding system. The search for surfaces having better wettability and higher surface texture led us to use two different electrode configurations for plasma nitriding: planar and hollow cathode. As a result, different ionisation grades were achieved. Following the nitriding process, the Ti discs were characterised according to their wettability, adhesion, thickness and the hardness of the nitrided layer and its visual appearance.

For the second part of the present study dental implants manufactured by Neodent (Curitiba, Brazil), 3·3 × 15 mm (diameter × height) made of titanium (second grade) were used. For them, no additional cleaning was necessary as they were supplied already sterilised using gamma rays. These implants were treated according to the best overall results achieved for the above test samples.

The equipment used was similar to that shown in Fig. 1. There are 400 × 400 mm (diameter × length) electrodes inside the stainless steel chamber which, in turn, is closed by two stainless steel flanges fitted with holes used as passageways for pressure probes and in/out gas flow. The chamber is also fitted with a polarised cathode electrode where a disc (or dental implant) is set with either the planar or hollow cathode configuration (Fig. 2).



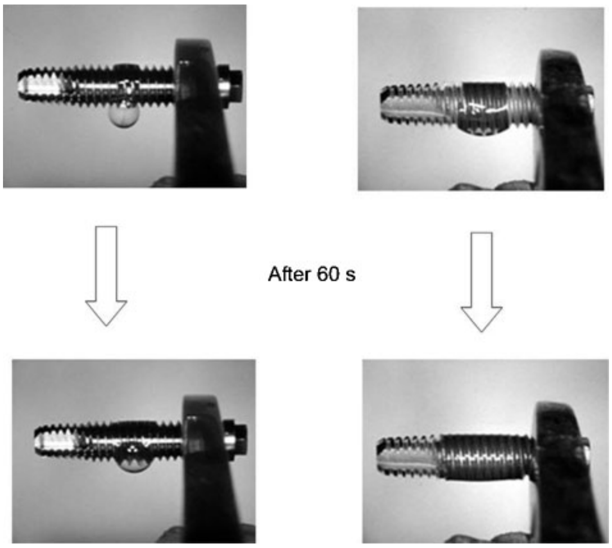
2 Experimental set-up used for hollow cathode plasma nitriding of *a* Ti disc and *b* dental implants

The electrical power source used for polarising the electrode is a continuous current type, capable of being regulated up to 1200 V and 1·5 A. Beyond the chamber and its power source, there are vacuum and gas flow circulation systems to complete the plasma nitriding apparatus. In order to start the running procedure, the chamber was evacuated until it reached 10^{-2} mbar in pressure. Then the working gas ($20N_2$–$80H_2$) was introduced under a constant flow rate for all experiments. The power supply was activated and adjusted to 500 V. The treating conditions applied to the samples are summarised in Table 1.The temperature was measured by a chromel–alumel thermocouple inserted in the substrate holder and controlled by varying continuously the voltage and current between the electrodes.

During the wettability tests, all samples were submitted to a rigid cleaning and sterilisation protocol: fat, protein and carbohydrates were removed by an enzymatic detergent acting for 10 min (twice), followed by absolute alcohol washing for 10 min (twice), which in turn was followed by bidistillated water washing for 10 min (twice). All procedures involved the application of ultrasound. Finally the implants were dried and sterilised in an autoclave appliance at 121°C for 20 min. After the implants had been sterilised, they were submitted to a wettability test using the physiological serum drop technique. The physiological substance used was Eurocolins (glucose solution of 3·57%) that is responsible for simulating human body liquids. This kind of substance is also used, for instance, for preserving human organs during transport for transplant surgery. The drop was allowed to accommodate and register its status at both 5 and 60 s. This was recorded by a Dental EYE photo camera fitted with a macrolens of 100 mm and attached to a tripod support

Table 1 Characteristics of samples treated under optimised plasma nitriding conditions

Sample	Visual aspect	Behaviour in ultrasound bath	Wettability, °	Roughness R_a, μm
P5T2/7P	Dark, uniform	Good	20	0·18
P3T2/7P	Dark, uniform	Good	17	0·19
P5T2/7DC9	Dark, uniform	Good	21	0·44
P2,8T1·5/7DC9	Yellow, dark, heterogeneous	Bad	18	0·65
P2,8T1/7DC9	Yellow, dark, heterogeneous	Bad	20	1·24
P2,5T1/7DC9	Yellow, uniform	Good	18	0·80
P2,5T1/6DC9	Yellow, bright, uniform	Good	17	0·50
P1,5T1/5DC9	Yellow, bright, uniform	Good	15	0·84



3 Typical aspect of sessile drop on nitrided implant and non-nitrided implants

thus guaranteeing a better standard of pictures. The wettability tests were carried out and their results were compared with those achieved by both national and international market available implants. Further observations were carried out by means of an Olympus BX60M model optical microscopy and a Phillips XL30 model MEV.

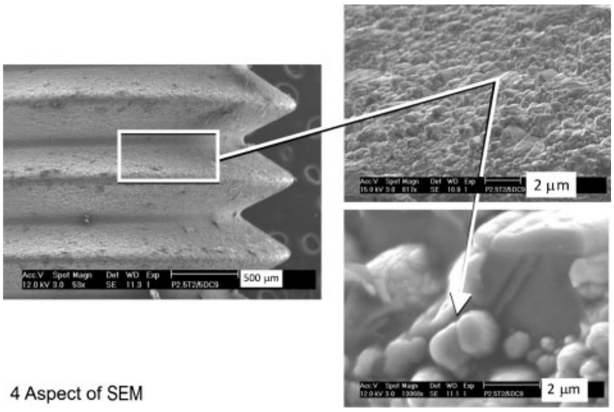
Results and discussion

Samples treated at 700°C for 2 h using the standard plasma configuration at high pressures exhibited dark coloration and showed little layer adherence. The layer detached itself during the ultrasound cleaning process resulting in esthetically and mechanically unsuitable materials for dental implant use. The samples nitrided under pressures greater than 2·5 mbars, using the hollow cathode configuration, were discarded due to the presence of heterogeneous layers and dark discoloration. This was necessary despite the fact that they showed good wettability.

Sample nitrated at 700°C under 2·5 mbars of pressure for 1 h using the hollow cathode method, ended up showing a uniform yellow coloured layer and good. Samples nitrided at 500 and 600°C were light yellow, with acceptable adherence, in addition to enhanced wettability (*see* Table 1).

The contact angles between the titanium sample (pure or nitrided) and the EuroCollins solution was estimated by the sessile drop technique. A 0·5 mL drop of EuroCollins solution was placed onto the surface and photographed after 5 and 60 s. The angles measured less than 20°. The best condition obtained with the test samples were used to plasma nitride the commercially available Ti implants. These conditions are presented in Table 2.

The dental implant nitrided at a pressure of 2·5 mbars and 700°C for 1 h had a yellow coloration, uniform layer and good wettability, but exhibited a fragile structure, making it unsuitable for dental implantation. The implants nitrided at 500 and 600°C at 2·5 mbars for 1 h had a yellow coloration, a uniform layer, good rugosity and good wettability.



4 Aspect of MEV

It was impossible to accurately measure contact angles because of extensive spreading in the nitrided dental implants. On the other hand, the contact angle measured on the untreated implants after a waiting period of ~60 s was limited to 50° (Fig. 3). All the nitrided implants had better wettability when compared to the untreated implants.

The surface texture of the nitrated implants was also examined to estimate the mechanical performance of the nitrided implants. An increase in both the overall surface area between the existing surfaces and the number of contact points was observed. This phenomenon has happened, particularly, in the region of microcavity formation. As a consequence of the high reactivity of the hollow cathode plasma discharge and the ionic bombardment in different directions, non-columnar film coating was formed which had a uniformly distributed row of microcavities on the implant surface, thus increasing the specific surface area. This would suggest that they would be suitable for osseointegration in a short period of time compared to their corresponding original surface. Scanning electron microscope images of nitrided implant surface as well as details regarding the aspects of the microcavities are shown in Fig. 4.

Conclusions

After Ti samples and implants were plasma nitrided. The resulting surfaces were characterised with the aim of estimating osseointegration periods. The following conclusions can be drawn from the present study.

1. Under the same time/temperature condition layers produced by hollow cathode discharges were thicker than those produced using a standard planar plasma configuration.

2. By using hollow cathode discharges it was possible to nitride Ti surfaces at temperatures as low as 400°C

Table 2 Nitriding condition used in implant

Implants	Pressure, mbar	Duration, h	Temperature, °C	Configuration
P2,5T1/7DC9	2·5	1	700	Hollow cathode DC9
P2,5T1/6DC9	2·5	1	600	Hollow cathode DC9
P1,5T1/5DC9	1·5	1	500	Hollow cathode DC9

producing the same effects conventionally obtained at 650°C using the planar configuration.

3. Plasma nitriding Ti samples using either hollow cathode discharges or planar configuration reduced the contact angle of Eurocolins drops from 50 to 20° thus improving wettability displayed by commercial implants treated by other methods, including plasma spray, chemical etching or sandblastering.

4. Improved wettability was attributed to the high reactivity and random bombardment which produce uniformly distributed microcavities which have the potential of reducing the osseointegration of Ti dental implants.

References

1. F. H. Jones: *Surf. Sci. Report*, 2001, **42**, 75–205.
2. T. Hanawa: *Mater. Sci. Eng. A*, 1999, **A267**, 260–266.
3. U. Joos, H. P. Wiesmann, T. Szuwart and U. Meyer: *J. Oral Maxillofac. Surg.*, 2006, **35**, 783–790.
4. L. Le Gu'ehennec, A. Soueidan, P. Layrolle and Y. Amouriq: *Dental Mater.*, 2007, **23**, (7), 844–854.
5. P.-I. Bränemark, R. Adell, U. Breine, B. O. Hansson, J. Lindström and A. Ohlsson: *Scand. J. Plast. Reconstr. Surg.*, 1979, **3**, 81–100.
6. D. Buser, T. Nydegger, T. Oxland, D. Cochran, R. Schenk, H. Hirt and L. Nolte: *J. Biomed. Mater. Res.*, 1999, **45**, 75–83.
7. V. Pešáková, D. Kubies, H. Hulejová and L. Himmlová: *J. Mater. Sci.*, 2007, **18**, 465–473.
8. U. M. Gross: *J. Dent. Educ.*, 1988, **52**, 798–803.
9. P. P. Binor, D. J. Weir and S. J. Marshall: *Int. J. Oral Maxillofac. Impl.*, 1992, **7**, 168–175.
10. D. M. Brunette: *Int. J. Oral. Maxillifac. Impl.*, 1998, **3**, 231–246.
11. B. Edenhofer: *Heat Treat Met.*, 1974, **1**, 23–28.
12. D. Buser, R. K. Shenk, S. Steinemann, J. P. Fiorelleni, C. H. Fox and H. Stich: *J. Biometed. Mater. Res.*, 1991, **25**, 889–902.
13. B. Kasemo: *Surf. Sci.*, 2002, **500**, 656–677.