RESONANCE FREQUENCY ANALYSIS OF IMPLANTS WITH ANODIZED SURFACE OXIDES

Jeong-Won Choi, D.D.S., M.S.D., Seong-Joo Heo, D.D.S., Ph.D., Ik-Tae Chang, D.D.S., Ph.D., Jai-Young Koak, D.D.S., Ph.D., Jong-Hyun Han, D.D.S., Ph.D.*, Yong-Sik Kim, D.D.S., Ph.D., Seok-Hyung Lee, Dr med dent., D.D.S.***, Soon-Ho Yim, D.D.S., Ph.D.*****

Department of Prosthodontics, Graduate School, Seoul National University

*Yonsei University, Yongdong Severance Hospital, **University of Ulsan, Asan Medical Center, ***Department of Prosthodontics, Samsung Medical Center, College of Medicine, Sungkyunkwan University

The present experimental study was designed to address two issues. The first was to investigate whether oxidation voltage of titanium implants influenced bone tissue responses after an in vivo implantation. The second aim was to investigate secondary stability change after 1 to 3months period.

Screw-shaped implants with a wide range of oxide properties were prepared by electrochemical oxidation methods, where the oxide thickness varied in the range of 3-15㎛. The micro structure revealed pore sizes of 1-3㎛, the crystal structures of the titanium oxide were amorphous, anatase and a mixture of anatase and rutile type.

Bone tissue responses were evaluated by resonance frequency measurements that were undertaken 1 to 3months after insertion in the rabbit tibia.

It was concluded that no statistical difference of RFA values was found between the groups, RFA gains after 1month and 3months were calculated.

Key Words

Anodic oxidation, Resonance frequency analysis, Titanium implant, Voltage

The importance of implant surface properties for successful osseointegration was first pointed out by Albrektsson et al.[1] At a state-of-the-art meeting on tissue integration held in 1985, the importance of implant surface properties for biological response were further emphasized in a consensus agreement that stated; surface properties are important for and may be used to facilitate tissue integration.[2] However, a number of

※ This work was supported by a grant from the Korea Health 21 R&D Project, Ministry of Health & Welfare, Republic of Korea (02-PJ3-PG6-EV11-0002)

questions have followed regarding the important role of the surface properties of titanium implants during dynamic build-up of the osseointegration process.[3] Interest in the surface oxide properties of titanium implants has increased with the development of methods to characterise such surfaces. Moreover, the possibility of surface modification of titanium implants to improve tissue responses is an outstanding feature in metal implantology research.

In ultrastructural studies of the implant interfacial zone, it has been observed that the tissue elements do not directly border the bulk titanium, but rather the 'native' oxide layer of the metal. This thin oxide layer was shown to be in 'contact' with remodeled mineralised bone.[4] The interface itself was a dense amorphous layer of approximately 100～500nm thickness. Studies of implants that have been retrieved from patients have demonstrated that both the thickness and the nature of the thin oxide layer change during implantation. Successfully osseointegrated titanium implants showed an increase in oxide thickness of up to 200nm, as well as incorporation of Ca and P into the oxide layers over an insertion time of six years.[5] However, in the case of failed titanium implants that were retrieved from patients, there were no changes in the oxide thickness or oxide composition during a period of function of up to eight years.[6]

Knowledge is still lacking about the role of surface oxide properties during the dynamic build-up of an osseointegration process. A very few in vivo studies have investigated the bone tissue responses to the surface oxide properties of c.p. titanium implants.[7] Heat treated titanium implants that were placed for eight days in the distal femoral condyles in a rat model were reported to result in an increased shear strength in comparison to titanium implants that had native oxide layers.[8]

Do the oxide properties of an implant surface have an effect on osseointegration? If so, which oxide properties of the implant surface will play an important role during the dynamic osseointegration process? The main purpose of the present study was to try to answer these questions. Earlier methodological studies for surface oxide preparation indicated that the electrochemical growth behaviour of the oxide film on c.p. titanium metal was strongly dependent on the particular electrolyte used and, for a given electrolyte, on the anodic process parameters that were employed, such as the applied current density, the electrolyte concentration, the electrolyte temperature, agitation speed, and cathode- to-anode surface area ratios.[9] The present study investigated a variety of surface oxide properties, such as oxide thickness, pore configuration (porosity, pore size distribution, etc.), crystal structure, chemical composition and surface roughness, by controlling such electrochemical parameters in a standardized manner. The current paper focuses on the in vivo investigations, especially those that were evaluated with biomechanical tests, i.e. resonance frequency analysis.

Osseointegration is a treatment concept based on stability where achievement and maintenance of clinical rigidity of implants are prerequisites for the successful long-term clinical function of an implant supported prosthesis. Implant stability can be divided into primary and secondary stability. Primary stability is determined by the density and quantity of the bone, the surgical technique, and the design of the implant. Secondary stability refers to implant stability after primary healing ; it is determined by primary stability and any gain in stability that results from bone formation and remodeling at the implant-bone interface. If primary stability is high, for instance when implants are placed in dense cortical bone, it is likely there will be only a modest increase in stability because most of the implant surface is engaged with bone from the start. On the other hand, in situations with low bone density and poor primary stability, healing can add markedly to secondary stability because a greater part of the implant surface is not in contact with bone at placement. Only short healing periods are usually needed for implants with good primary stability. Implants with poor primary stability need longer healing periods to achive sufficient gains in secondary stability.

Surgical preparation of an implant site initiates a preprogrammed tissue response aimed at complete repair of the defect. The early phase of the healing process includes migration and differentiation of mesenchymal cells, proliferation of vessels and bone marrow, and formation of primary woven bone. During later healing, the primary bone is replaced by lamellar bone through a remodeling process. The time needed for remodeling and maturation depends on many factors, including the size of the defect and the state of the local bone and the host. However, it is much longer than the 3-6 month healing period commonly used for implants - probably up to 18 months.

In this study, RFA value was measured at 1st op. for primary stability and measured after 1 and 3 months for secondary stability.

The purpose of this study is to compare primary and secondary stability of anodically oxidated implants under different voltage.

MATERIALS AND METHODS

2.1 Implant preparation : design and surface oxide

A total of 152 square screw-shaped implants, which had a pitch-height of 0.9mm, an outer diameter of 4.3mm, a length of 8.0mm, a external hexa head and an inner threaded hole of 4mm, were turned from 5mm rods of commercially pure titanium (ASTM Grade 2). All surface oxides used in the present study were prepared with the use of a dc power supply in electrolyte solution. For convenience, the implant samples were divided into five groups according to the anodic forming voltage, as follows: (Fig. 1)

* Group I implants (n=38) had electrochemically prepared surface oxides up to the forming voltage of 300V, with an oxide thickness of 3.1㎛
* Group II implants (n=38) had electrochemically prepared surface oxides up to the forming voltage of 400V, with an oxide thickness of 7.8㎛
* Group III implants (n=38) had electrochemically prepared surface oxides up to the forming voltage of 500V, with an oxide thickness of 13.5㎛
* Group IV implants (n=38) had electrochemically prepared surface oxides up to the forming voltage of 550V, with an oxide thickness of 14.6㎛

In brief, surface morphology a porous structure in Groups I-IV. In Groups I, the pore size was 1㎛ in diameter, while the pore size distribution (PSD), which was measured by opening area, increased according to the forming voltage that was applied, i.e. 2㎛ in Group II, 3㎛ in Group III and IV. The crystallinity of the titanium oxide was assigned to the anatase phase for Group I (a mixture of anatase and rutile phase as analysed by Raman spectroscopy) and a mixture of anatase and rutile phase for Group IV (as analysed by thin-film X-ray diffractometry, TF-XRD). The chemical composition in each groups was mainly TiO2, with small variations in trace elements such as C, Ca, Na and Si. The surface roughness was in the range of 0.4㎛~2.2㎛. The various oxide properties in this study are summarized in Table I.

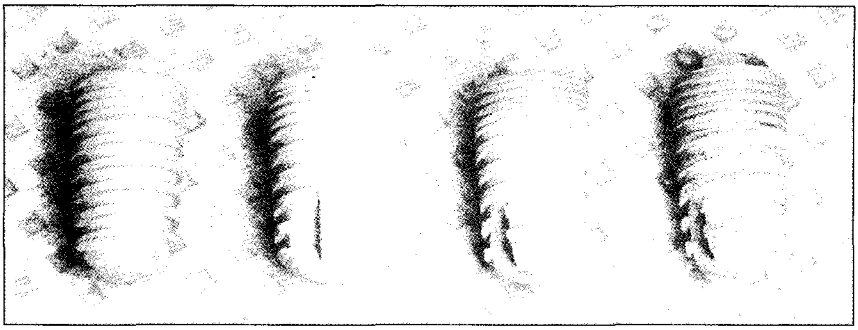

Fig. 1. Square screw shaped implants with microthread, group 1-4.

Table I. summary of oxide growth parameters and surface characteristics of the four different types of c.p. titanium implants

Oxide characteristics	Group I	Group II	Group III	Group IV
Anodic forming voltage	300V	400V	500V	550V
Oxide thickness	3.1㎛	7.8㎛	13.5㎛	14.6㎛
Morphology	porous	porous	porous	porous
Pore size	1㎛	2㎛	3㎛	3㎛
Crystallinity	Mainly anatase	Anatase + rutile	Anatase + rutile	Rutile form increase
Chemical composition	Ca, P	Ca, P	Ca, P	Slightly increase
Roughness (Ra)	0.4㎛	0.9㎛	1.7㎛	2.2㎛

2.2 Animals and surgical technique

38 mature (average age 10months old) New Zealand white rabbits of both sexes were included in this study. During surgery, the animals were anaesthetized with intramuscular injections of Ketamine and Rompune at a dose of 1ml per kg body weight. Prior to surgery, the shaved skin was carefully washed with a mixture of iodine and 70% ethanol. Local anaesthesia with 1.0ml of 5% lidocaine was administered, at the tuburositas tibiae part of the bone where the incision was planned, under aseptic conditions. The skin and facial layers were opened and closed separately. The periosteal layer was gently pulled away from the surgical area and was not resutured. During all surgical drilling sequences, low rotary drill speeds not exceeding 1000r.p.m and profuse saline cooling were used.

Each rabbit had two implants inserted in each tibia; these penetrated one cortical layer only. The implants were inserted in a predetermined randomised design that enabled multiple comparisons. The animals were kept in separate cages and immediately after surgery they were allowed to be fully weight-bearing.

Rabbits were sacrificed in CO_2 chamber at the scheduled time.

2.3 Resonance frequency measurements

Directly after implant insertion, the baseline resonance frequency analysis (RFA) was monitored on the implants. At the day of sacrifice, i.e. 1month or 3month after implant insertion, the RFA was again tested prior to removal torque tests. This method is a non-destructive technique that demonstrates the implant stability in terms of interfacial stiffness (Hz). The frequency response of the system was measured by attaching the transducer, i.e. an L-shaped cantilever beam, to a screw implant. The excitation signal was given over a range of frequencies (typically 5kHz to 15kHz with a peak amplitude of 1V) and the first flexural resonance was measured.[10] The resonance frequency was measured immediately after implant insertion and then again 1 or 3month later. (Fig. 2)

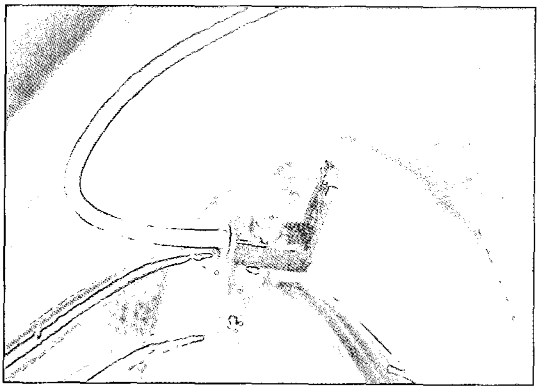

Fig. 2. Resonance frequency measurement after 1st op.

Table II. Mean and SD of RFA values

		300V	400V	500V	550V
initial	mean	72.18	72.71	71.29	69.92
	SD	3.91	3.04	2.93	4.33
1month	mean	75.53	75.89	73.42	75.37
	SD	4.4	2.79	3.79	2.65
3month	mean	77.37	79.05	74.68	77.63
	SD	5.42	5.7	7.06	5.59

2.4 Statistics

Multiple comparisons between all groups were performed using two-way analysis of variance and the Tukey test.

RESULTS

The mean baseline RFA values were 72.18 for Group I implants, 72.71 for Group II implants, 71.29 for Group III implants and 69.92 for Group IV. The mean RFA values 4 weeks after implant insertion were 75.53 for Group I implants, 75.89 for Group II implants, 73.42 for Group III implants and 75.37 for Group IV implants. The mean RFA values 3 months after implant insertion were 77.37 for Group I implants, 79.05 for Group II implants, 74.68 for Group III implants and 77.63 for Group IV implants.(Table II, Fig. 3)

There were no statistically significant difference in RFA when all the Groups I-IV were compared with each other (P>.0.05). However, as shown in Fig. 3, there was a trend for RFA to increase as healing period from group I to group IV after implant insertion(P<0.05 in group II, IV).

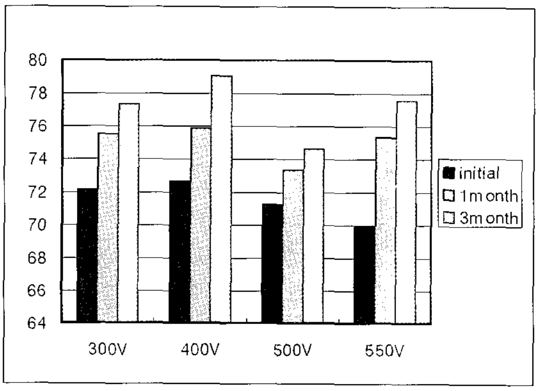

Fig. 3. RFA values of each group at 1st op, after 1 and 3months.

Table III. Mean and SD of RFA value gain after 1 and 3months

		300V	400V	500V	550V
1month	mean	2.79	2.47	1.42	5.79
gain	SD	5.77	2.32	3.79	5.12
3month	mean	5.53	7.05	4.11	7.37
gain	SD	6.09	7.16	6.4	5.61

From the data above, RFA gains after 1month and 3months were calculated, and also there was no statistically significant difference between groups. (Table III, Fig. 4)

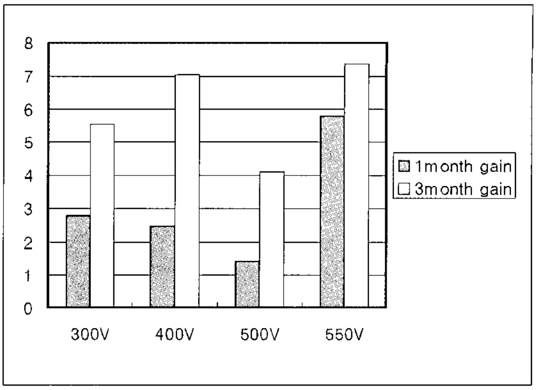

Fig. 4. RFA gain after 1 and 3months of each group.

DISCUSSION

Rough surface implant showed great success rate clinically, and took the place of prototype implant - machined implant. Many surface treating techniques were developed, and being developed now - sand blasting, acid etching, ion deposition, laser graphy, anodic oxidation etc..

In this study, among above techniques, we focused on anodic oxidation technique.

'Anodic oxidation' - micro arc oxidation - is a large description technically. Static current method, static voltage method, fluctuating method, and many techniques for change of electrolytes. Ti-Unite (Nobel biocare) implant is produced under static current method. In this case, voltage is changing continuously, generally growing up as surface oxide getting thicker. We used static voltage technique, and some fluctuation was used for thicker oxide layer.

Anyway, 'what surface character is working' is still unanswered question.

For anodic oxidation, surface crater made by micro arc during oxidation is very unique structure, and the crater size is a interesting topic for better biologic response - 'How large crater bone loves? Osteoblast and vessel cell like big crater? Or big pore?' Big crater size means big pore size, and more roughness. This study shows that as if the crater, and pore is produced by micro arc, the size doesn' t matter! Bigger crater does not facilitate bone foramation more than small crater.

We tested RFA values at 1st surgery, after 1 month, and after 3 months and the gain was statistically significant for all groups, and the gain of 3months compared with 1month is as big as the 1st month gain. Immediate loading is spotlighted in many clinical researches, but osseointegration - secondary stability- is still getting mature after 3 months even in rabbit.

Interestingly, as you can see in figure 3, initial stability was lower at the high voltage group. It was just a tendency and statistically not significant, but it can be suggested that too rough surface means smaller contact area at 1st operation bone site, and it cause the lower ISQ value. This problem may need more study.

CONCLUSION

1. There was RFA gain after 1month, and more gain after 3months in all groups.
   The gain after 3 month is almost the double of 1st month gain.
2. There was no statistically significant differ-

ence of RFA gain between groups of different voltage.

3. Bigger crater and more roughness did not show more osseointegration.

REFERENCES

1. Albrektsson, T., Brånemark, P.I., Hansson, H.A. & Lindström, J. Osseointegrated titanium implants. Acta Orthopaedica Scandinavica 1981;52:155-179.
2. van Steenberghe, D., ed. Proceedings of the InternationalCongress on Tissue Integration inOral and Maxillo-Facial Reconstruction. New York: ExcerptMedica 1986.
3. Albrektsson, T., Brånemark, P.I., Hansson, H.A., Kasemo, B., Larsson, K., Lindström, I., McQueen, D. & Skalak, R. The interface zone of inorganic implants in vivo: titaniumimplants in bone. Annals of Biomedical Engineering 1983;11: 1-27.
4. Sennerby, L., Thomsen, P. & Ericson, L.E. Ultrastructure of the bone-titanium interface in rabbits. Journal of Materials Science: Materials in Medicine 1992;3: 262-271.
5. McQueen, D., Sundgren, J.E., Ivarsson, B., Lindström, I., Ekenstam, B.F., Svensson, A., Albrektsson, T. & Brånemark, P.I. Auger electron spectroscopic studies of titanium implants. In: Lee, A.J.C., Albrektsson, T. & Brånemark, P.I., eds. Clinical Applications of Biomaterials, 1982;179-185. New York: John Wiley & Sons.
6. Esposito, M., Lausmaa, J., Hirsch, J.M. & Thomsen, P. Surface analysis of failed oral titanium implants. Journal of Biomedical Materials Research 1999;48: 559-568.
7. Larsson, C., Thomsen, P., Aronsson, B.O., Lausmaa, J.,Rodahl, M. & Kasemo, B. Bone response to surface- modified titanium implants: studies on the early tissue response to machined and electropolished implants with different oxide thickness. Biomaterials 1996;15: 605-616.
8. Oron, U. & Hazan, R. Method of treating a metal prosthetic device prior to surgical implantation to enhance bone growth relative thereto following implantation. US Patent Number 5 080 671, 1992.
9. Sul, Y.T., Johansson, C.B., Petronis, S., Krozer, A., Jeong, Y., Wennerberg, A. & Albrektsson, T. Characteristics of the surface oxides on turned and electrochemically oxidized pure titanium implants up to dielectric breakdown: The oxide thickness, micropore configurations, surface roughness, crystal structure and chemical composition. Biomaterials 2002;23: 491-501.
10. Meredith, N., Shagaldi, F., Alleyne, D., Sennerby, L. & Cawley, P. The application of resonance frequency measurements to study the stability of titanium implants during healing in the rabbit tibia. Clinical Oral Implants Research 1997;8: 234-243.

Reprint request to:
SEONG-JOO HEO
DEPT. OF PROSTHODONTICS, COLLEGE OF DENTISTRY
SEOUL NATIONAL UNIVERSITY
28-1 YEONGUN-DONG, CHONGNO-GU, 110-749, SEOUL KOREA
heosj@plaza.snu.ac.kr