# Osseointegration between Oxide Layer of Titanium and Bone by Different Heat Treatments

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## 열처리에 따른 티타늄 Oxide Layer와 Bone 사이의 Osseointegration에 대한 연구

대구카톨릭의과대학 치과학교실

이 규 복

본 연구의 목적은 순수 티타늄에 열처리를 통해 서로 다른 두께의 산화막을 형성하여 온도차이에 따른 골결합에 대한 영향을 관찰하고자 하는 것이다.

총 52개의 임플란트를 제작하여 열처리를 하지 않은 26개의 임플란트를 대조군으로, 600°F(316℃)에서 10분간 열처리한 13개의 임플란트를 실험군 1로, 1200°F(649℃)에서 10분간 열처리한 13개의 임플란트를 실험군 2로 하여 13마리의 가토의 경골에 식립한 후 12주 후의 removal torque를 측정 비교함으로써 다음과 같은 결과를 얻었다.

1. 실험군 2에서는 결정구조가 생성된 것이 관찰되었으나, 실험군 1에서는 무정형이었다.
2. 대조군에 비해 열처리를 한 실험군들이 더 큰 removal torque 값을 나타냈다($p=0.0059$, $0.0039$).
3. 실험군 2가 실험군 1에 비해 큰 removal torque 값을 나타냈다($p=0.0488$).

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## Ⅰ. INTRODUCTION

Currently, various implants with different materials, designs, and surface structures are used and it was reported that the success rates are also highly variable. Although strenuous efforts have been made to identify contributing factors for implant success or not, the mechanism is not fully understood yet.

Various factors must be considered for successful osseointegration. Among the factors, it is significantly important that the physical and chemical properties of the implant surface. Especially, the oxide layer is known to have a direct effect on implant success by helping the attachment of cells and matrixes to the bone-implat interface[4-6].

The effect of the oxide layer has been clearly demonstrated by Larsson et al[7,8] who showed that rougher implant surface and thicker oxide layer showed more favorable osseointegration. Hazan et al[9] and Ha et al[10] also reported that thicker oxide layer revealed higher removal torque. According to Hazan et al[9], there was higher shear strength in implants with heat treatment compared to those without heat treatment, and maximal enhancement of bone growth was achieved at 280℃.

Our observations differ somewhat from the results by Hazan et al[9] which reported that the thickness of oxide layer doubled at 280℃ and there was even more increase in oxide layer at 550℃, on the other hand, bone growth reached the highest point at 280℃ and rather decreased at 550℃. But direct comparison is difficult due to the fact that our study was conducted for 10minutes while Hazan et al[9]'s study was conducted for 3hours.

Olin et al[11] and Ask et al[12] reported that heat treatment or electrochemical oxidation resulted in increased thickness of oxide layer in pure titanium. Radegran et al[13] showed that the temperature strongly influences the thickness of the oxide layer.

Many researches have been demonstrated the relationship between the temperature of heat treatment and the quality of the oxide layer formed[5,9-19]. However, the ideal temperature and the proper oxide layer thickness to achieve maximum biocompatibility and osseointegration are not well understood.

In this study, it was performed that the effect of the heat treatment temperature in implants on the thickness of the oxide layer and the osseointegration.

## Ⅱ. MATERIAL AND METHODS

### 1. Implant fabrication

A total of 52 screw-type pure titanium(ASTM

Grade2) implants with 5.0mm in lenght, 3.75mm in diameter, and 0.6mm in pitch height were fabricated and divided into three groups. In Group I, 13 implants were treated at 600°F(316℃) for 10minutes in furnace. In Group II, 13 implats were treated at 1200°F(649℃) for 10minutes in furnace. In Control Group, 26 implats were prepared without heat treatment(Fig. 1).

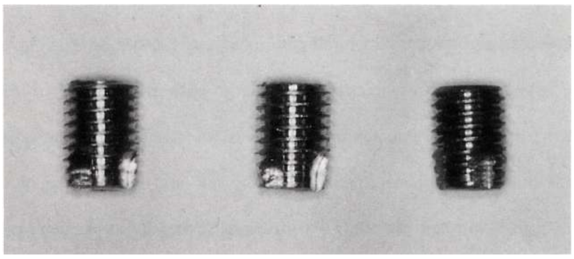

Fig. 1. Photograph of the implants : unheat treated(left), 600°F(middle) and 1200°F (right) heat treated implant.

## 2. Characterization of the implant

The morphology and thickness of the surface oxide layer in the heat treated implants were observed using scanning electron microscope (SEM, S-800, Hitachi).

## 3. Procedure

For the experiment, 13 adult rabbits weighing approximately 3kg were used. Surgery was performed in aseptic conditions and preoperative antibiotics was not used. General anesthesia was performed using 5mg/kg of Xylazire(Rompun®, Byel, Korea) and 35mg/kg of Ketamine(Ketara®, Yu-Han, Korea) by intramuscular injection. The surgical site was shaved and carefully cleaned with a mixture of iodine and 70% alcohol. And then, 2ml of 2% lidocaine was injected locally on the tibial metaphysis. Layer incision was made antero-medial to tibial tuberosity to expose tibial metaphysis which provided good bone quality and visibility.

Preparation of bone for implantation was performed using traditional Branemark system, and implantation was done with connection of the specific instrument. In this experiment, tapping drill and countersink drill were not used.

A total of 52 implants were placed in the left and right tibiae of 13 rabbits. One control group implant and one experimental group I implant were placed in the right tibia of each of the 13 rabbits. One control group implant and one experimental group II implant were placed in the left tibia of each of the 13 rabbits. To prevent fracture of cancellous bone, there was at least 7mm of space between two implants as shown in Fig. 2.

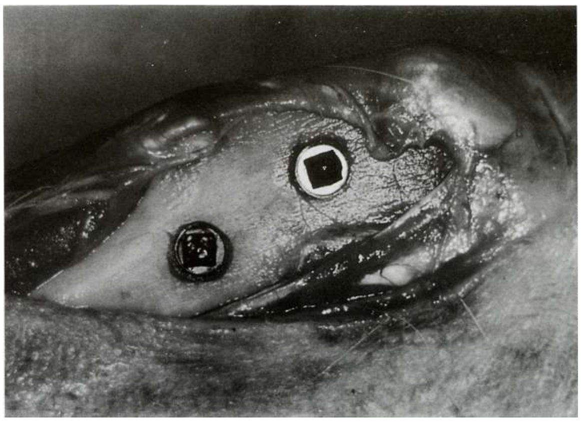

Fig. 2. After preparing the bone of threaded sites

It was used 4-0 absorbable suture material for layer suture. Intramuscular injection of 1ml of antibiotics (Baytril®, Byel, Korea) and 1ml of metabolic catalyst (Catosal®, Byel, Korea) was given. Elastic bandage was placed on the surgical site to prevent contamination.

## 4. Radiographic examination

To evaluate the implanted state, x-ray pictures were taken 12 weeks after implantation. The X-ray beam was directed perpendicular to the long axis of the implats to have a good image of the implant thread and its surrounding bone.

## 5. Measurement of removal torque

After 12 weeks, the rabbits were sacrificed. Incisions were made and periosteum was reflected to expose the implant site. Any overgrowth of bone over the implant was carefully removed with low-speed round bur to expose the implant head.

Tohnichi 15 BTG-N Torque Gauge Manometer (Tohnichi Mfg Co., Ltd, Tokyo, Japan) was used to measure the removal torque for each implant. A specially designed connector was used to connect the torque gauge to the implant. Any movement of the torque gauge was prevented by the use of a stabilizing device which holds the torque gauge in place. The removal torque value was measured at the moment when fracture took place between the bone and the implant.

## 6. Statistical analysis

The results of the experiment were statistically analyzed using the Wilcoxon signed-ranks test and the Duncan's multiple range test. The provability value(P) was evaluated at 5% level.

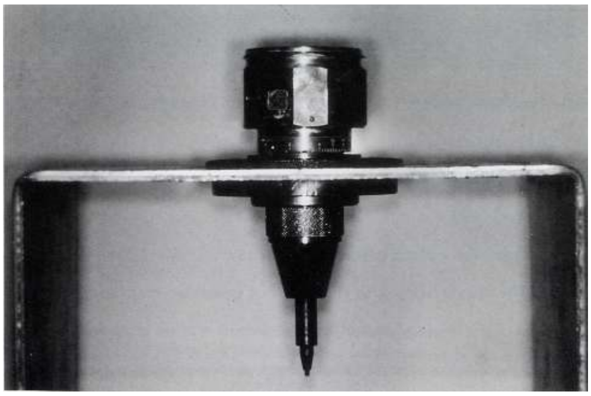

Fig. 3. Tohnichi 15 BTG−N Torque Gauge Manometer with a specially designed connector held in place by a stabilizing device.

# Ⅲ. RESULTS

## 1. Implant surface characterization

The thickness of oxide layer and the morphology of the implant surface were observed using SEM. The Group II implants with heat treatment at 1200°F(649℃) demonstrated some thickness of oxide layer, while the Group I implants with heat treatment at 600°F(316℃) showed no noticible oxide layer thickness(Fig. 4). A definite grain structure was observed on the surfaces of the Group II implants while not on the surfaces of the Group I implants(Fig. 5).

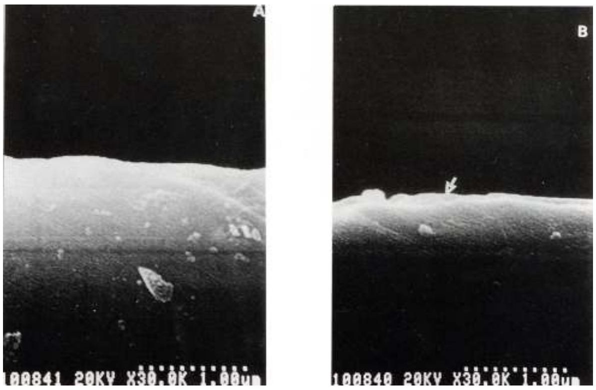


Fig. 4. SEM showing the titanium oxide thickness at 1200°F(649℃) heat treated sample (B), while not at 600°F(A).


Fig. 5. SEM showing the surface topography of samples : heat treatment at 600°F(A), heat treatment at 1200°F(B).

## 2. Radiographic findings

The upper one or two threads of the implants are embedded in the cortical bone and the remaining parts of the implants are in the cancellous bone. No radiolucency was observed around the implants.

## 3. Removal torque test

Removal torque was measured 12 weeks after the implant placement. One of the 13 rabbits died during experiment without any specific reason and was excluded from the statistics. Also 2 rabbits of the remaining had bicorticated implants, so they were excluded from the statistics. The results of the removal torque measurement were shown in Table 1. The mean values for implants with heat treatment at 600°F (Group I) and 1200°F(Group II) were 3.46Ncm and 5.12Ncm respectively, while those of the control group were 1.33Ncm in the right and 1.29Ncm in the left.

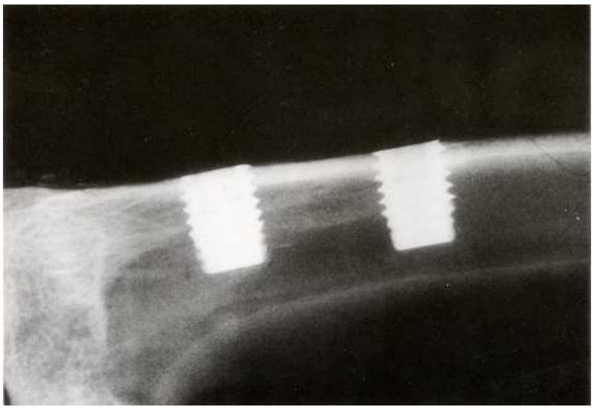

Fig. 6. Radiograph of the experimental implants

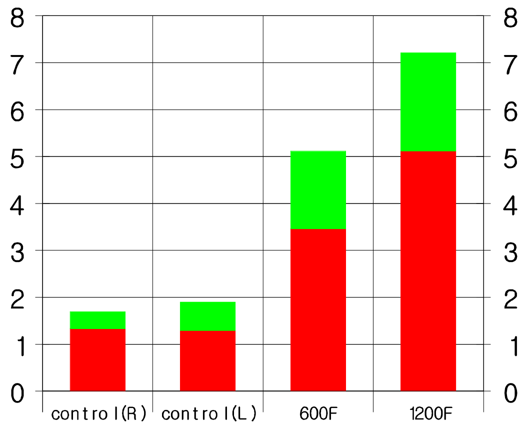


Fig. 7. Bar graph of the removal torques

Table 1. Removal torque measurement for the experimental group

| Sample No. | Removal Torque(N·cm) | | | |
|---|---|---|---|---|
| | control (R)* | control (L)** | Group I*** | Group II |
| 1 | 1.0 | 1.0 | 5.4 | 6.1 |
| 2 | 1.8 | 2.4 | 4.3 | 4.5 |
| 3 | 1.0 | 1.0 | 3.7 | 8.2 |
| 4 | 1.5 | 1.0 | 3.2 | 6.7 |
| 5 | 1.0 | 2.0 | 1.5 | 6.1 |
| 6 | 1.0 | 1.0 | 4.4 | 3.8 |
| 7 | 1.8 | 2.0 | 3.2 | 1.2 |
| 8 | 1.5 | 1.0 | 5.7 | 6.2 |
| 9 | 1.0 | 0.5 | 0.3 | 2.6 |
| 10 | 1.7 | 1.0 | 2.9 | 5.8 |
| mean | 1.33 | 1.29 | 3.46 | 5.12 |
| S.D | 0.362 | 0.611 | 1.656 | 2.091 |

* : p=0.0059 compared with group I

** : p=0.0039 compared with group II

*** : p=0.0488 compared with group II

Heat treated samples showed significantly higher removal torque value than control(right tibia ; p=0.0059, left tibia ; p=0.0039).

And 1200°F(649℃) heat treated samples showed significantly higher removal torque value than 600°F (316℃) heat treated samples(p=0.0488).

## Ⅳ. DISCUSSION

Various methods, including the examination of the histologic feature[20-25], the measurement of shear strength[26,27], and the measurement of removal torque[10,20-26], have been proposed to evaluate the bonding pattern between the implant and bone. The removal torque measurement, since first introduced by Johansson et al[22] to measure the bonding strength between the screw-type implant and bone, has become the most widely used method today. This experiment also adopted it because the advantage indicated by the theory of the Anusavice et al[27]. The removal torque was measured by the use of the torque gauge as in the experiment of Ha et al[10].

Branemark et al[14], Machnee et al[5] and Klauber et al[15] reported the oxide layer of titanium surface measured 3-5nm after various cleaning and sterilizing procedures, while Keller et al[16] reported the oxide layer of 3-25nm. According to Machnee et al[5], the thickness of the oxide layer which remained unchanged under 370℃ kept increasing above the temperature. But, commercially pure titanium lost its structure and characteristics and became weak above 545℃ with the increase of oxygen absorption.

Smith et al[18] and Bignolas et al[19] reported that the increase in thickness of oxide layer at low temperature followed logarithmic time law, and it was impossible to study under 100℃ due to the high contamination rate of titanium, and oxygen dissolution had tendency to increase above 400℃, and it was very difficult to study above 600℃ because of severity of oxygen dissolution and contamination from such as sulfur and chlorine.

Fukuzuka et al[17] observed changes in color of titanium surface with increase in oxide layer following the increase of time and temperature, and titanium became severely oxidized and so brittle with formation of porous oxide film above 800℃.

In our experiment, just like the color changes observed by Fukuzuka et al[17], we were able to observe the color change into light blue at 1200°F(649℃) while no change had occurred at 600°F(316℃).

According to Hazan et al[9], there was higher shear strength in implants with heat treatment compared to those without heat treatment, and maximal enhancement of bone growth was achieved at 280℃.

Ha et al[10] reported that the forming of oxide layer with different time frame during heat treatment at 980 ℃ and when observed 12 weeks after the implantation was shown that there was increase in thickness of oxide layer and removal torque relative to time.

Although there was a good result from the study of Ha et al[10] at high temperature, comparing those studies[5,9,17-19] above, it might be necessary to form the adequate thickness of oxide layer without being brittle at the temperature below 800℃. This is the reason for setting up the temperature at 600°F(316℃) and 1200 °F(649℃) in this study.

In this experiment, we were able to observe the difference in removal torque measurements with changes in the temperature of heat treatment. It had similar values of removal torque between the left and right control groups, and 600°F(316℃) and 1200°F (649℃) heat treated groups showed significantly higher removal torques than each control group. Also, it was shown that significantly higher removal torques at 1200°F(649℃) than 600°F(316℃) heat treated group.

These observations differ somewhat from the results by Hazan et al[9] which reported that the thickness of oxide layer doubled at 280℃ and there was even more increase in oxide layer at 550℃, on the other hand, bone growth reached the highest point at 280℃ and rather decreased at 550℃. But direct comparison is difficult due to the fact that our study was conducted for 10minutes while Hazan et al[9]'s study was conducted for 3hours.

Also when the results of this experiment was compared to Ha et al[10]'s study, it was shown the same result that heat treated samples had higher removal torques than unheat treated samples. But the samples of heat treatment at 600°F(316℃) and 1200°F(649℃) for 10minutes in our study gave rather higher torque value than the samples of heat treatment at 980℃ for 10minutes, although it had thicker oxide layer at 980℃. For these, it could all be contributing reasons such as the difference in thickness of oxide layer, the difference in length of fixture, the difference in implantation site, the difference in thickness of cortical bone and possibility of bicortication. So there is no significance of direct comparison, but when it is compared to Fukuzuca et al[17], it might be very porous and too brittle when the oxide layer formed at 980℃. This factor can be suggested one of the reasons for it's low torque value than our study.

In many cases of our study, there was overgrowth of bone over the implant and filling inside of pitch at the top of fixture. In order to measure the removal torque, it was required to remove overgrown bone from pitch and careful removal using a scalpel without harming the fixture during this process. And biocorticated fixtures were excluded from statistical analysis but we believe there were cases that it would not be identified the bicortication through radiographic pictures or with naked eyes. For these reasons, in order to achieve more accurate experimental results, using cover screws to prevent overgrowth of bone over the implant and using shorter fixtures or animals with bigger bone to reduce the possibility of bicortication are considered to be desired.

According to these results, the increase of the oxide layer by heat treatment of implants brings increase of the removal torque, and oxide layer gives direct influence to osseointegration. But it will be recommended to further stydy in order to find out the most appropriate thickness of oxide layer for osseointegration and the most appropriate temperature which forming the thickness of oxide layer.

## V. CONCLUSIONS

In this study, it was investigated that the influence of oxide thickness, which depend on the temperature of heat treatment, on the osseointegration.

Total 52 implants were manufactured, 26 implants without heat treatment were used as control group. Another 26 implants were devided two groups as follows :

Experimental group I : 13 implants with heat treatment at 600°F(316℃) for 10minutes

Experimental group II : 13 implants with heat treatment at 1200°F(649℃) for 10minutes.

Twelve weeks after implantation, the removal torque was measured.

The results were as followed:

1. Experimental group II showed definite grain structure, while not on the group I.
2. Samples heat treated demonstrated higher removal torque values than control(right tibia ; p=0.0059, left tibia ; p=0.0039).
3. Experimental group II demonstrated higher removal torque value than experimental group I(p=0.0488).

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