Available online at www.sciencedirect.com
SCIENCE DIRECT®
Biomaterials 24 (2003) 3611–3617
Biomaterials
www.elsevier.com/locate/biomaterials

The removal torque of titanium screw inserted in rabbit tibia treated by dual acid etching

Sung-Am Cho*, Kyung-Tae Park

Department of Prosthodontics, College of Dentistry, Kyungpook National University, 101 2 Gu Dong-In Dong, Jung-Gu, Daegu, South Korea

Received 26 October 2002; accepted 12 March 2003

Abstract

Chemical acid etching alone of the titanium implant surface have the potential to greatly enhance osseointegration without adding particulate matter (e.g. TPS or hydroxyapatite) or embedding surface contaminants (e.g. grit particles). The aims of the present study were to evaluate any differences between the machined and dual acid etching implants with the removal torque as well as topographic analysis.

A total of 40 custom-made, screw-shaped, commercially pure titanium implants with length of 5 mm and an outer diameter of 3.75 mm were divided into 4 groups, 10 screws in each, and chemical modification of the titanium implant surfaces were achieved using HF and HCl/H_2SO_4 dual acid etching.

The first exposure was to hydrofluoric acid and the second was to a combination of hydrochloric acid and sulfuric acid. The tibia metaphysics was exposed by incisions through the skin, fascia, and periosteum.

One implant of each group was inserted in every rabbit, 2 in each proximal tibia metaphysics. Every rabbit received 3 implants with acid etched surfaces and 1 implant with a machined surface.

Twelve weeks post-surgically, 7 rabbits were sacrificed, Subsequently, the leg was stabilized and the implant was removed under reverse torque rotation with a digital torque gauge (Mark-10 Corporation, USA) (Fig. 1). Twelve weeks after implant placement, the removal torque mean values were the dual acid etched implants (24% $HF + HCl/H_2SO_4$, group C) required a higher average force (34.7 Ncm), than the machined surface implants (group A) ($p = 0.045$) (Mann-Whiteney test).

Scanning electron micrographs of acid etching of the titanium surface created an even distribution of very small (1–2 μm) peaks and valleys, while machining of the titanium surface created typical microscopically grooved surface characteristics.

Nonetheless, there was no difference in surface topography between each acid etched implant groups.

Therefore, chemically acid etching implant surfaces have higher strengths of osseointegration than machined implant surfaces. There is less correlation between removal torque and the difference in HF volume%.

Keywords: Removal torque; Titanium screw; Rabbit tibia; Dual acid etching

1. Introduction

Through the study of clinical and experimental data, it is clear that the long-term clinical success of dental implants depends largely on osseointegration of the implant. The importance of osseointegration was first described by Branemark et al. In addition, the original microstructural and macrostructural aspects of the standard Branemark implant (Nobel Biocare, Goteborg, Sweden) were initially considered essential to ensure adequate bone formation around any implant [1,2]. The term ‘integration strength’ refers to the force required to break the bond between the implant and the bone. The surface morphology and bone–implant interactions determine the predictability of endosseous dental implant–bone integration.

To obtain osseointegration of an implant, several factors are of importance: biocompatibility, the surface quality, the surgical technique, the status of the host tissue, and the loading conditions [3]. The 1996 World Workshop in Periodontics concluded that the surface characteristics of an implant, particularly roughness, may direct tissue healing. However, these macrorough surfaces could potentially cause failure of the implant

*Corresponding author. Tel.: +82-53-422-6128; fax: +82-53-421-6898.

E-mail address: sacho@kyungpook.ac.kr (S.-A. Cho).

0142-9612/03/$ - see front matter © 2003 Elsevier Science Ltd. All rights reserved.
doi:10.1016/S0142-9612(03)00218-7

because of increased bacterial aggregation on the rough surface or breakdown of the hydroxapatite [4]. The interaction between implants and surrounding tissues can be affected by the macroscopic surface topography as well as by the surface morphology or roughness at a microscopic level [5,6]. Although machined commercial pure titanium surface were recommended for many years to ensure best results, more recent studies suggest that other materials and surface characteristics may also be beneficial [1]. Various procedures have been tested to improve the anchorage strength and mechanical interlocking of root-form dental implants by modifying implant characteristics, especially the implant surface texture, by roughening, coating or chemical treatment [7–10]. The development and use of these surfaces modifications have been based on the theory that improved osseointegration can be achieved by increasing the topography or roughness of the implant surface [11,12]. Several investigators have found that roughened surfaces had increased implant surface areas that resulted in greater surface coverage by bone when compared to smooth-polished surfaces [13,8]. Also, several studies have shown that a rough implant surface helps to promote osseointegration more than a smooth machined surface. Buser et al. reported that an increased surface area positively correlated with an increased bone–implant contact. Several investigators have demonstrated that implant surface roughness enhances the biomechanical anchorage (osseointegration) of implants to bone. They determined that this fact through torque removal tests [5,14–16].

Surface blasting and acid etching can increase the rate and amount of bone formation on the implant surface [5,16,9,17]. Moreover, one of the more effective bone–implant interfaces is sandblasting, followed by acid-attacking surfaces [5,18]. No negative effects on cell adhesion have been shown using this technique [5,18]. Grit blasting followed by acid etching has produced surface characteristics resulting in the greatest bone contact percentage of metal surfaces tested. This appeared superior to grit blasting, acid-pickled and titanium plasma sprayed surfaces alone [5]. However, several investigators have reported that grit particles can remain impregnated in the implant material, and are potentially a causative agent in observed tissue breakdown [19].

A new surface that produces a microroughness similar to the blasted/etched surface but uses only special high-temperature dual acid etching without grit blasting has been developed. Chemical modification (acid etching alone) of the titanium implant surface is of particular interest because it appears to have the potential to greatly enhance osseointegration without adding particulate matter (e.g. TPS or hydroxyapatite) or embedding surface contaminants (e.g. grit particles). The purpose of dual etching is to produce a microrough surface that provides rapid osseointegration, while maintaining the long-term success associated with a machined implant surface.

The aims of the present study were to evaluate any differences between the machined and dual acid etching implants with the removal torque as well as topographic analysis.

2. Material and methods

2.1. Animals and anesthesia

Ten adult white rabbits weighing 3–3.5 kg were used in the present study. The animals were anesthetized with a combination of ketamine (Ketalar® 45 mg/kg of body weight, YuHan, Korea) and xylazine (Rompun®, 7 mg/kg of body weight, Bayer, Korea) intramuscularly. Prior to surgery, 1.8 ml of 2% lidocaine were injected locally into the tibia metaphases. Post-operatively, the animals received antibiotics at a dose of animal for 3 days. Twelve weeks after surgery, the animals were sacrificed using an overdose of carbon dioxide.

2.2. Implant surface treatment

A total of 40 custom-made, screw-shaped, commercially pure titanium implants with length of 5 mm and an outer diameter of 3.75 mm were used in this study. They were divided into 4 groups, 10 screws in each, and chemical modification of the titanium implant surfaces were achieved using HF and HCl/H_2SO_4 dual acid etching. The groups were as follows:

Group A: 10 implants, left as machined.

Groups B–D: 30 implants, (10 implants per group) acid-etching surface treatment.

Group B: 12% hydrofluoric acid (HF) + 70% hydrochloric acid/sulfuric acid (HCl/H_2SO_4).

Group C: 24% hydrofluoric acid + 70% hydrochloric acid/sulfuric acid.

Group D: 48% hydrofluoric acid + 70% hydrochloric acid/sulfuric acid.

Chemically treated implant surfaces were accomplished by soaking the implants for 120 s in HF at each different volume as well as five more minutes in 70% HCl/H_2SO_4 at a high temperature (80°C). [20] The first exposure was to hydrofluoric acid and the second was to a combination of hydrochloric acid and sulfuric acid. All implants were cleaned with a steam cleaner as well as an ultrasonic cleaner and then sterilized in an autoclave.

2.3. Implant placement

Prior to surgery, the legs were shaved, washed and decontaminated with a mixture of Betadine and 70% ethanol. The tibia metaphysics was exposed by incisions through the skin, fascia, and periosteum. The flat surface on the anteromedial aspect of the tibia was selected for implant placement. One implant of each group was inserted in every rabbit, 2 in each proximal tibia metaphysics. The screws were alternately inserted in the left or right leg and in a distal or proximal position. In addition, each group was subdivided into Sections 1 and 2. Section 1 received implants inserted into the proximal part of the tibia, while Section 2 received implants in the more distal part of the tibia. By intermittent drilling using a low rotary speed and profuse saline irrigation, 2 holes were drilled 7 mm apart in the central portion of each tibia and sequentially enlarged to 3.2 mm. The implants were gently screwed into place, without tapping the sites, until the implant shoulder was level with the bone surface. All implants were allowed to penetrate the first cortical layer only. Every rabbit received 3 implants with acid etched surfaces and 1 implant with a machined surface. After the implants were seated and stable, the fascia and skin were closed in separate layers using resorbable sutures.

2.4. Removal torque measurements

Twelve weeks post-surgically, 7 rabbits were sacrificed, while the other 3 rabbits die of infection. The implant sites were surgically exposed via sharp dissection and the bone and soft tissues that had formed on top of the implants were carefully removed. Subsequently, the leg was stabilized and the implant was removed under reverse torque rotation with a digital torque gauge (Mark-10 Corporation, USA) (Fig. 1). This machine was used to measure the peak value of resistance to reverse torque rotation, in Ncm.

Fig. 1. Digital torque gauge (Mark-10).

2.5. Statistical analysis

Mean values of removal torque were calculated and subjected to the Mann-Whitney test and Kruskal-Wallis test. A comparison between the machined (group A) and 24%HF + 70%HCl/H_2SO_4 (group C) groups was accomplished with the Mann-Whitney test. On the other hand, a comparison between the dual acid etched implants was accomplished using the Kruskal-Wallis test. These tests evaluated the differences in removal torque between each group. All statistical testing were carried out at the 5% significance level.

3. Results and discussion

3.1. Removal torque

Twelve weeks after implant placement, the average removal torque was 12.2 Ncm (A1) and 36.1 Ncm (A2) for the machined implants, 18.5 Ncm (B1) and 31.6 Ncm (B2) for the 48%HF + HCl/H_2SO_4 acid etching implants, 25.8 Ncm (C1) and 64.7 Ncm (C2) for the 24%HF + HCl/H_2SO_4 acid etching implants and 27.6Ncm (D1) and 44.1Ncm (D2) for the 12%HF + HCl/H_2SO_4 acid etching implants. For the Mann-Whiteney test and Kruskal-Wallis test, the removal torque mean values were of 15.2, 34.3, 34.7 and 38.7 Ncm for groups A, B, C and D, respectively. The highest removal torque corresponded to the 24%HF + HCl/H_2SO_4 acid etching implants, while the lowest was demonstrated by the machined implants. In the Mann-Whiteney test, the dual acid etched implants (24%HF + HCl/H_2SO_4, group C) required a higher average force (34.7 Ncm), than the machined surface implants (group A). This was statistically significantly ($p = 0.045$). However, the data between dual acid etched implants were not statistically significant ($p < 0.05$). The mean values obtained for maximum removal torque forces for each implant surface are shown in Tables 1 and 2 as well as Figs. 2 and 3.

3.2. Topographic evaluation

Scanning electron micrographs of the chemically etched and machined surfaces demonstrated microscopic differences in surface topography. Acid etching of the titanium surface created an even distribution of very small (1–2 μm) peaks and valleys, while machining

Table 1
Removal torque values in Newton's

Rabbit No.	Machined	48%HF	24%HF	12%HF
	(RP,AI)	(RD,BI)	(LP,CI)	(LD,DII)
1		37.8	12.4	52.9
2	8.8	12.0		
3	14.9	34.3	42.4	35.9
4	13.0	42.3	22.5	43.6
Mean values	12.2	31.6	25.8	44.1
Rabbit No.	48%HF	Machined	12%HF	24%HF
	(RP,BI)	(RD,AI)	(LP,DI)	(LD,CII)
5	34.4	62.4	41.4	124.7
6	12.0	15.4	14.3	37.3
7	9.2	30.6	27.0	32.0
Mean Values	18.5	36.1	27.6	64.7

RP: proximal part of the right tibia. RD: distal part of the right tibia. LP: proximal part of the left tibia. LD: distal part of the left tibia.

Table 2
Mean values of the removal torque in each group (UNIT: Ncm)

	Group A	Group B	Group C	Group D
Mean values	15.2	34.3	34.7	38.7
SD	20.1	14.3	20.4	13.6

Group A: 10 implants left as machined.
Group B: 12% hydrofluoric acid + 70% hydrochloric acid/sulfuric acid.
Group C: 24% hydrofluoric acid + 70% hydrochloric acid/sulfuric acid.
Group D: 48% hydrofluoric acid + 70% hydrochloric acid/sulfuric acid.

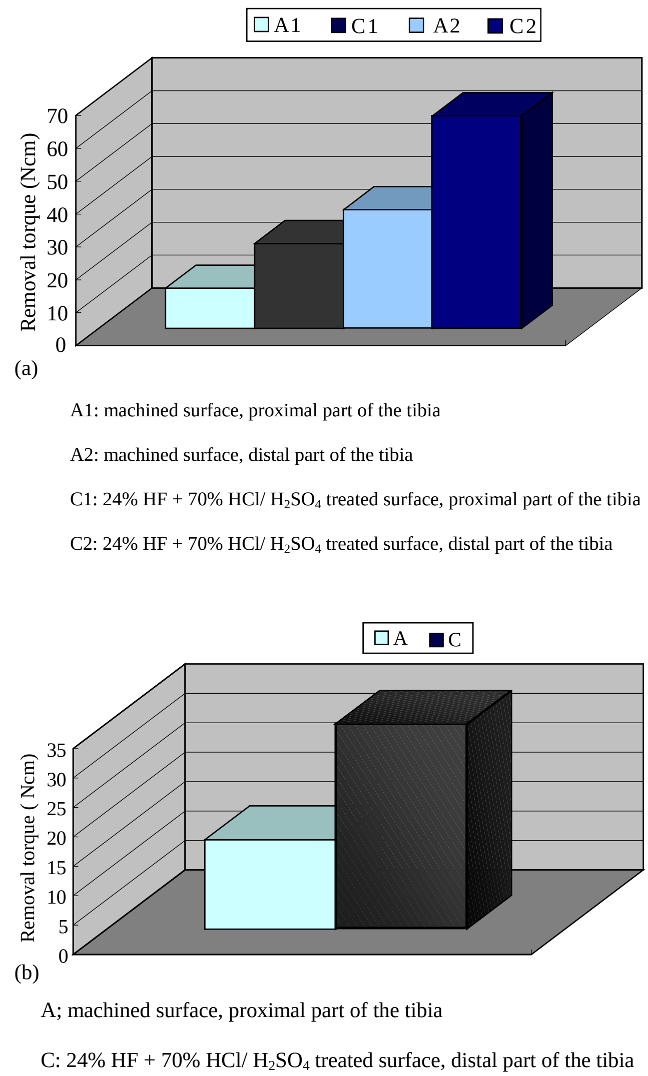

Fig. 2. (1) Machined versus 24%HF + 70%HCl/H_2SO_4, (2) Machined versus 24%HF + 70%HCl/H_2SO_4.

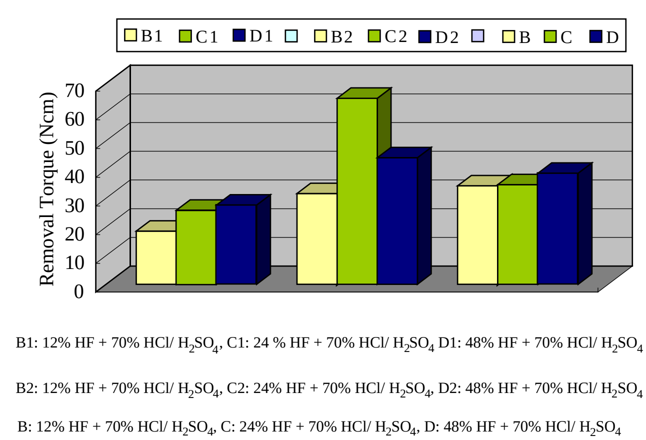

Fig. 3. Comparison of removal torque as difference of volume% HF.

of the titanium surface created typical microscopically grooved and relatively smooth surface characteristics. In addition, acid etched implant groups appeared rougher than the machined implant groups. Nonetheless, there was no difference in surface topography between each acid etched implant groups (Figs. 4–7).

In the present study, it was found that chemically acid-etched titanium implants achieved greater resistance to reverse torque rotation than machined implant surfaces 3 months post-surgery in the rabbit tibia. Resistance to reverse torque rotation has been used as a measure of implant anchorage or osseointegration. Greater torque rotation forces required to remove implants may be interpreted as an increase in the bone–implant contact leading to higher osseointegration strength.

In vivo experiments investigating bone growth into hollow test chambers lined with acid-etched and machined surface demonstrated that acid-etched surfaces support more and closer bone growth. Several in vivo studies have also reported that osseointegration is better facilitated by rough implant surfaces than by machined implants composed of identical material [5,21,22].

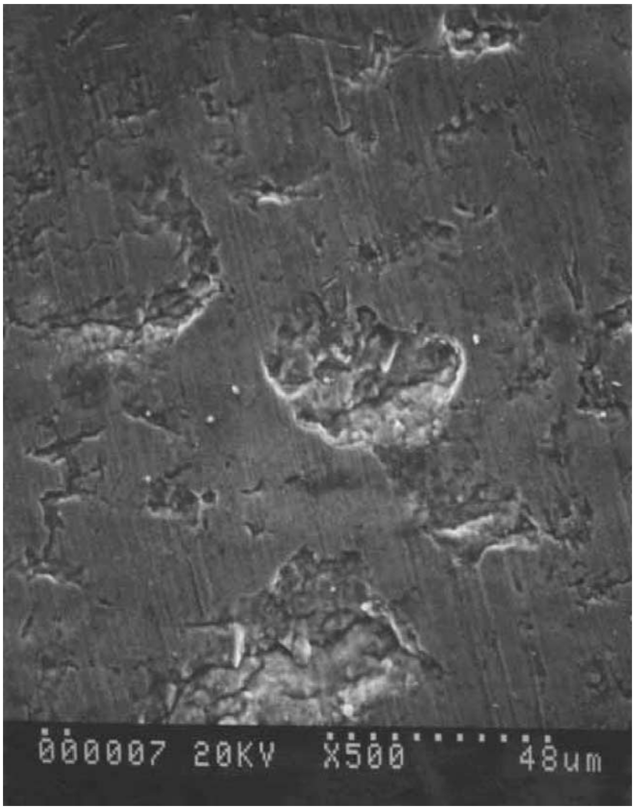

Fig. 4. Machined implant (Group A).

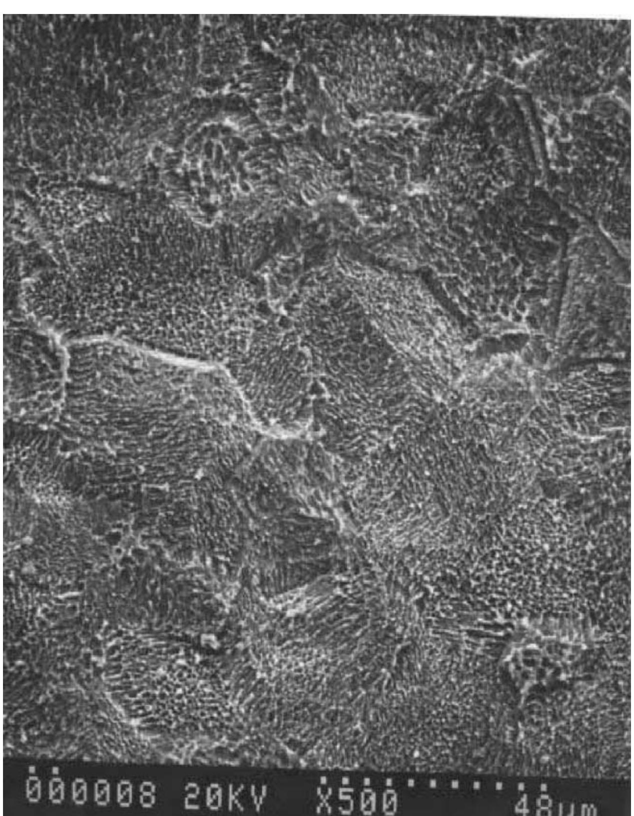

Fig. 5. 48% HF + HCl/H_2SO_4 (Group B).

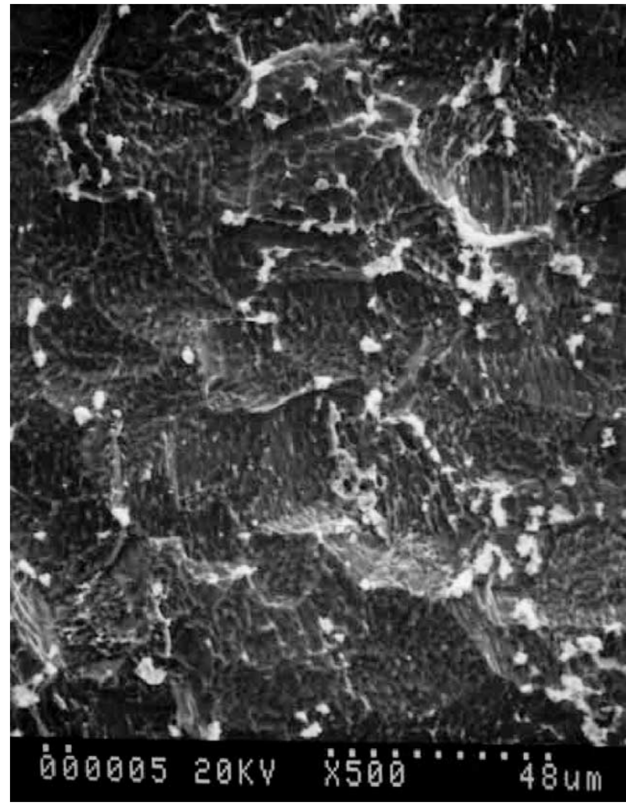

Fig. 6. 24%HF + HCl/H_2SO_4 (Group C).

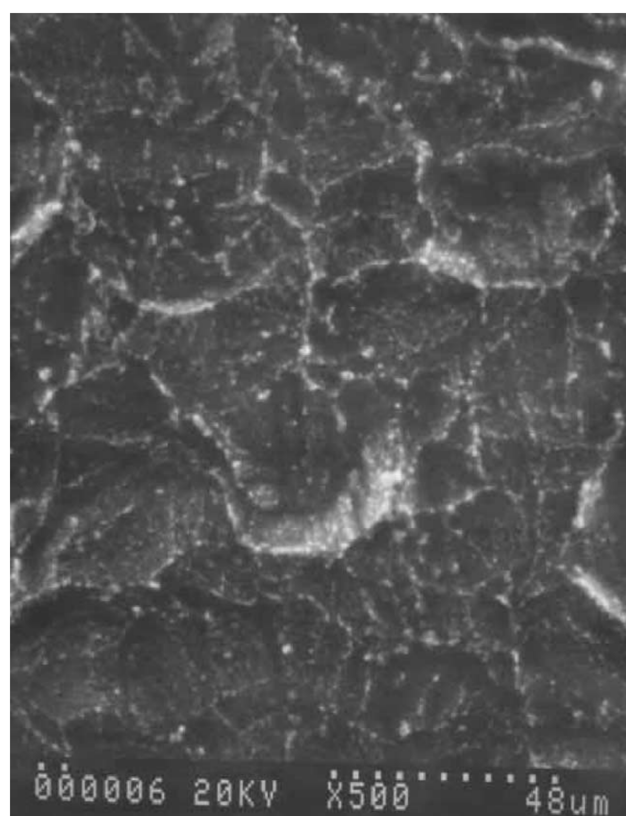

Fig. 7. 12%HF + HCl/H_2SO_4 (Group D).

In vitro, Bowers et al. also found that surface roughness significantly increases the degree of cellular attachment of osteoblast-like cells. At the cellular level, the differences between acid-etched and machined surfaces may be explained by the microrough topography of acid-etched surfaces, which could favorably affect angiogenesis as well as cellular migratory behavior, cell alignment, orientation, attachment, and finally, cell activity and function [23,9,24]. Wennerburg et al. also reported improvements in bone anchorage of cp titanium and titanium alloy implants when the surfaces were roughened with chemical etching [14].

Thus, it has been suggested that even minimal changes in material or design of an implant may have a significant impact on the clinical outcome. The mean torque values obtained in this study are comparable with mean torque values observed for similar healing times in other studies using screw shaped implants in the rabbit [25–27]. Improved bone–implant interface and greater resistance of failure has been reported in studies that tested an acid etched implant surface in combination with other surface treatments. Specifically, screw shaped implants with surfaces that were sandblasted and acid etched (HCl/H_2SO_4) achieved higher resistance to reverse torque rotation than screw shaped implants with surfaces that were electro-polished, sandblasted and acid pickled (HF/HNO_3) or titanium plasma spray coated [16]. This is in agreement with the present findings.

In a histomorphometric study, implants with surfaces that were sandblasted with large grit (25–50 μm) and acid etched were found to have a 50–60% mean value of bone–implant contact at 6 weeks while implants that were titanium plasma sprayed had a 30–40% mean value of bone–implant contact for the same time period [16].

A direct comparison of sandblasted (large grit 25–50 μm) implant surfaces with those that were sandblasted (25–50 μm) and acid etched (HCl/H_2SO_4) revealed a significant increase in bone contact for surfaces that were sandblasted and acid etched [16].

The authors also stated that the increase can most likely be attributed to the acid treatment with HCl and H_2SO_4. This demonstration is in agreement with this experiment. In this study, the gradient difference of HF volume% was no statistically significant ($p<0.05$), but the difference in the removal torque between the machined implants and acid etched implants was significant ($p<0.05$). Thus these results may indicate that HF is less effective in acid etching treatment than HCl/H_2SO_4.

Increased surface roughness may enhance the mechanical interlocking between the macromolecules of the implant surface and the bone, resulting in a greater resistance to compression, tension and shear stress, as some authors have demonstrated [13].

A rougher surface has been shown to result in a faster as well as stronger bone integration [13]. However, there has been some confusion about what is rough and what is smooth due to a lack of an appropriate measuring technique and evaluation system especially for screw-shaped oral implants [21]. In this present study, the acid etched implants yielded higher removal torque values than the machined implants [17,18]. The results are in agreement with those of Klokkevold et al., who reported that chemically etched implant surfaces conferred a resistance to reverse torque removal in the rabbit femur that was 4 times greater than that of machined surfaces 2 months post-placement [16]. The greater removal torque values achieved with the acid-etched group may be related primarily to the higher bone-to-implant contact. Several investigators demonstrated this positive correlation between the degree of bone in contact with the implant and removal torque [25,17].

4. Conclusions

This study indicates that rough acid etched implants achieve greater resistance to reverse torque removal than machined surface implants. In addition, there is less correlation between removal torque and the difference in HF volume%. The greater torque rotation forces required to remove implants may be interpreted as increases bone–implant contact, leading to higher strengths of osseointegration.

Therefore, chemically acid etching implant surfaces have higher strengths of osseointegration than machined implant surfaces. Further research is needed to study chemical subtraction methods to define the optimal implant surface topology that enhances osseointegration.

Acknowledgements

This study was supported by a grant of the Korea Health 21 R&D Project, Ministry of Health & Welfare, Republic of Korea (00-PJ-PG3-31400-0053).

References

[1] Branemark P-I, Breine U, Adell R, Hansson BO, Lindstrom J, Olsson A. Intraosseous anchorage of dental prosthesis I. Experimental study. Scand J Plast Reconstr Surg 1969;3:81–100.

[2] Branemark PI, Hansson BO, Adell R, et al. Osseointegrated implants in the treatment of edentuluous jaw experience from a 10-year period. Scand J Plast Reconstr Surg 1977;16(Suppl): 7–127.

[3] Albreksson T, Zarb G, Eriksson AR. The long-term efficacy of currently used dental implant; A review and proposed criteria of success. Int J Oral Maxillofac Implants 1986;1:11–5.

[4] Consensus Report Implant therapy I. Ann Periodontol 1996;1(1): 792–5

[5] Buser D, Schenk R, Steinemann S. Influence of surface characteristics on bone integration of titanium implants. A histomophometric study in minature pigs. J Biomed Mater Res 1991;25:9–902.

[6] Wennerberg A, Albrektsson T, Lausmac J. Torque and histomophometric evaluation of cp titanium screw blasted with 25- and 75 μm-sized particles of Al_2O_3. J Biomed Mater Res 1996;30:251–60.

[7] Mustafa K, Lopez S, Hultenby K, Wenerberg A. Attachment and Proliferation of human oral fibroblasts to Ti surfaces blasted TiO_2 particles. Clin Oral Implants Res 1998;19:195–207.

[8] Wennerberg A, Albrektsson T, Andersson B. An animal study of cp titanium screws with different surface topographies. J Mater Sci: Mater Med 1995;6:302–9.

[9] Wennerberg A, Albrektsson T, Andersson B. Bone tissue response to commercially pure titanium implants blasted with fine and coarse particles of alumium oxide. Int J Oral Maxillofac Implants 1996;11:38–45.

[10] Young Jun Lim, Yoshiki Oshida, Carl JA, Marti TB. Surface charaterization of variously treated titanium materials. Int J Maxillofac Implants 2001;16(3):333–42.

[11] Han C, Johansson C, Wennerberg A, Albrektsson J. Quantitative and qualitive investigation of surface enlarged titanium and titanium alloy implants. Clin Oral Implants Res 1998;9:1–10.

[12] Wagner WC. A brief introduction to advanced surface modificationtechnologies. J Oral Implantology 1992;18(3):231–5.

[13] Thomas KA, Kay JF, Cook SD, Jarcho M. The effect of surface macrotexture and hydroxyapatite coating on the mechanical strengths and histologic profiles of titanium implant materials. J Biomed Mater Res 1987;21(12):1395–414.

[14] Wennerberg A, Albrektsson T, Andersson B, Krol J. A histomorphometric and removal torque study of screw-shaped titanium implants with three different surface topographies. Clin Oral Implants Res 1995;30:251–60.

[15] Ivanoff CJ, Sennerby J, Lekholm U. Influence of mono and bicortical anchorage on the titanium implants. A study in the rabbit tibia. J Oral Maxillofac Sur 1996;25:229–35.

[16] Klokkevold PR, Nishimura RD, Adachi A, Caputo A. Osseointegration enhanced by chemical etching of the titanium surface; A torque removal study in the rabbits. Clin Oral Implants Res 1997;8(6):442–7.

[17] David B, Robert M, Robert O'Neal. Rate of pullout strength gain of dual-etched titanium implant; A comparative study in rabbits. Int J Oral Maxillofac Implants 1999;14(5):722–8.

[18] Conchran DL, Nummikoski PV, Higginbottom FL. Evaluation of an endosseous titanium implant with sandblasted and acid-etched surface in the canine mandible; Radiographic results. Clin Oral Implants Res 1996;7:240–52.

[19] Darvell BW, Samman N. Contamination of titanium castings by aluminum oxide blasting. J Dent 1995;23(5):319–22.

[20] Park IG, Hwang BN, Lee JB, Lim JH, Kim JH, Choi SG. Studying for the Surface Treatment of the AVANA Dental Implant. J Korea Acad General Dentistry 2000;2(1):44–50.

[21] Wennerberg A, Hallgren C, Johansson C, Danelli S. Histomorphometric evaluation of screw-shaped implant each prepared with two surface roughness. Clin Oral Implants Res 1998;9:11–9.

[22] Schroeder A, van der Zypen E, Stich H, Sutter F. The reaction of bone, CT and epithelium to endosteal implants with sprayed titanium surfaces. J Max Fac Surg 1981;9:15–25.

[23] Boyan BD, Hummert TW, Dean TT. Schwartz, Role of material surfaces in regulation bone and cartilage cell response. Biomaterials 1996;17:137–46.

[24] Rich A, Harris AK. Anomalous preferences of cultured macrophages for hydrophobic and roughened substrata. J Cell Sci 1981; 50:1–7.

[25] Johansson C, Albrektsson T. Integration of screw implants in the rabbit: A 1-year follow-up of removal torque of titanium implant. Int J Oral Maxillofac Implants 1987;2:69–75.

[26] Carlsson L, Rostlund T, Albrektsson B. Removal torque for polished and rough titanium implants. Int J Oral Maxillofac Implants 1998;3:21–4.

[27] Giampiero C, Zeina M, Adriano P, Antonio S. Removal torque and histomorphometric investigation of 4 different titanium surfaces. An experimental study in the rabbit tibia. Int J Oral Maxillofac Implants 2000;15(5):668–74.