Original Article

Surface changes of anodic oxidized orthodontic titanium miniscrew

Sung-Hwan Choi[a]; Jung-Yul Cha[b]; Uk-Hyon Joo[c]; Chung-Ju Hwang[d]

ABSTRACT

Objective: To evaluate the structural stability of anodic oxidation treatment of miniscrews during a self-drilling procedure and an initial loading period.

Materials and Methods: Eight orthodontic miniscrews with a machined surface and an anodic oxidized surface were placed in the mandible of two beagle dogs. With all miniscrews, an orthodontic force was applied immediately after placement and was continued for 12 weeks. After beagle dogs were sacrificed, the miniscrews were carefully removed from decalcified bone fragments. Miniscrews were evaluated by comparing and quantitatively analyzing changes in surface roughness of unused and used miniscrews (machined surface vs anodic oxidized surface) utilizing both scanning electron microscopy (SEM) and atomic force microscopy (AFM).

Results: SEM revealed that only a thread edge close to the tip of the used anodic oxidized miniscrew became smooth by smearing, compared with the unused anodic oxidized miniscrew. No definite changes were observed in the thread valleys of the two groups after placement. AFM measurements demonstrated that all surface roughness parameters of thread edges of the used anodic oxidized miniscrews were significantly reduced compared with the unused anodic oxidized miniscrew ($P < .05$). A middle thread edge of the used anodic miniscrew surface was rougher than the unused and used machined surface miniscrews ($P < .05$).

Conclusion: Anodic oxidized miniscrews had improved surface characteristics compared with machined surface miniscrews, even if the surface texture was changed by the self-drilling procedure and during the initial loading period. (*Angle Orthod.* 2012;82:522–528.)

KEY WORDS: Anodic oxidation; Miniscrew; Surface roughness; Atomic force microscopy

INTRODUCTION

Anchorage control, an important factor in the prevention of unwanted tooth movement, directly affects the outcome of orthodontic treatment. Recently, orthodontic miniscrews have been used as orthodontic anchors.

It is important to ensure the biological stability of an orthodontic miniscrew because this reduces its micromovement, which supports the healing process surrounding the bone. Therefore, various miniscrews based on prosthetic surface–treated implants are currently being developed and marketed to improve biological stability.

Among these implants, anodic oxidized implants have greater surface roughness than original machined surface implants, and thus possess relatively large surface areas. Higher osseointegration of anodic oxidized implants is observed during the initial healing period[1,2] because osteoblasts more easily attach to a rough surface than a smooth surface.[3,4] The quantity of calcium and phosphorus in the surface of the anodized oxide implant is greater than that of machined surface implants.[5,6] Additionally, differences in porosity allow for potential drug incorporation and release around titanium implants.[7,8]

However, to improve biological stability, changes in the unique surface characteristics should be minimal during self-tapping or self-drilling insertion through cortical bone, during which the miniscrew is exposed to friction and heat. For example, shiny spots appear at the sandblasted large-grit, acid-etched (SLA) surface

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when damaged during the handling or packaging process, or when removed from the sterile ampule. Scanning electron microscopy (SEM) demonstrated that these shiny spots correspond to smearing of the soft surface with alteration of the microtopography.[9] This trait is not specific to the SLA surface but rather is common to all etched surfaces. The etching process carves the titanium surface at a depth of 2 to 5 μm and leaves a soft and smearable surface.[10] If any surface changes are observed in an anodic oxidized miniscrew after placement by self-tapping or self-drilling, the effects of these changes on stability and miniscrew retention must be determined.

SEM, surface profilometry, and atomic force microscopy (AFM) can be used to observe changes before and after placement of an anodic oxidized miniscrew; SEM requires proper sample preparation such as special coatings or vacuum treatment and provides only a two-dimensional image of the surface morphology.[11] Surface profilometry can induce sample damage and is difficult to use when overall surface roughness is measured because it analyzes the topography of a single line in a preselected area.[12]

In contrast, AFM scanning is noninvasive and provides three-dimensional information regarding surface morphology and mechanical properties[13]; quantification of surface roughness can be useful for quantitative analysis.[14–17]

The aim of this study is to evaluate the structural stability of anodic oxidation treatment on miniscrews before evaluating its biological stability under a self-drilling procedure and during the initial loading period by comparing and quantitatively analyzing changes in surface roughness of unused and used miniscrews (machined surface vs anodic oxidized surface) utilizing both SEM and AFM.

MATERIALS AND METHODS

For this study, miniscrews were placed in two beagle dogs (age, 1 year; weight, 10 to 13 kg). Their purchase, selection, management, and experimental procedures were carried out according to prescribed conditions of the institutional review board and the Animal Experiment Committee of Yonsei Hospital, Seoul, Korea. A self-drilling type of miniscrew (Biomaterials Korea, Seoul, Korea; diameter, 1.5 mm; length, 7 mm) was used. Both machined surface and anodic oxidized surface miniscrews were selected; a total of eight screws were used (Figure 1A).

The animals were injected subcutaneously with 0.05 mg/kg of atropine followed by an intravenous injection of rompun, 2 mg/kg, and ketamine, 10 mg/kg, to induce general anesthesia. Anesthesia was maintained with 2% enflurane, and each animal's temperature was maintained with a heating pad. Animals were monitored by electrocardiogram. When the miniscrew was placed, 2% hydrochloric acid lidocaine containing 1:100,000 epinephrine was infiltrated into the placement area. Before placement, gingival incision was made under saline solution irrigation, and complete placement of the screw into alveolar bone was confirmed. Sites selected were between the roots of the fourth premolar and the first molar in the mandible. In all miniscrews, an orthodontic force of 250 g was applied with a nickel-titanium (NiTi) coil spring engaged reciprocally after placement. Miniscrews were implanted in the inferior periosteum to prevent gingival inflammation around the miniscrews. Elastomeric tube covering a NiTi coil spring was used to reduce discomfort (Figure 1B).

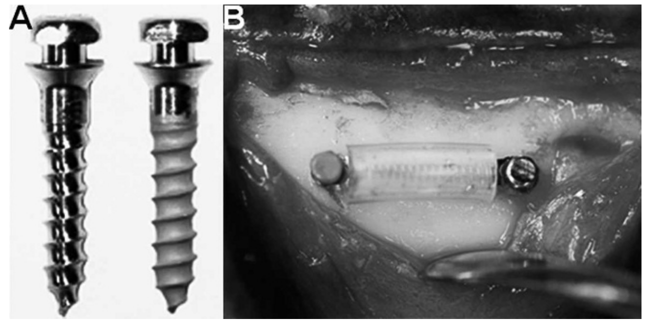

Figure 1. Images of tested miniscrews. (A) Machined surface miniscrew *(left)* and anodic oxidized miniscrew *(right)*. (B) Implantation sites.

The highest insertion torque was measured during an initial one-quarter turn using a torque sensor (MGT50, Mark-10 Co, New York, NY). Initial screw mobility was measured twice on each miniscrew with Periotest (Siemens AG, Bensheim, Germany) after insertion.

Twelve weeks after miniscrew implantation, the dogs were sacrificed and 2 cm × 2 cm bone fragments with miniscrews were collected. The bone fragments were decalcified in Calci Clear-Rapid (National Diagnostics, Atlanta, Ga) and were fixed with 10% formalin and neutral pH 7.4 after 4 weeks of decalcification. Miniscrews were removed from bone fragments carefully and were ultrasonically cleaned for 30 minutes to remove integuments around the miniscrews.

Noncontact mode AFM images were obtained using an NPX 200 (Seiko Instruments Inc, Chiba, Japan). The miniscrews were scanned in an air conditioner; scanned images were 5 μm × 5 μm in size. We obtained three images each of the following: a thread edge close to the screw head, a middle thread edge, and a thread edge close to the tip, for a total of nine images per miniscrew. For surface characterization of the two types of miniscrews, a total of 72 images were obtained (Figure 2).

Data acquisition and image processing were performed with a Nanopics 2.10 (Seiko Instruments Inc). For roughness analysis, a plane correction process

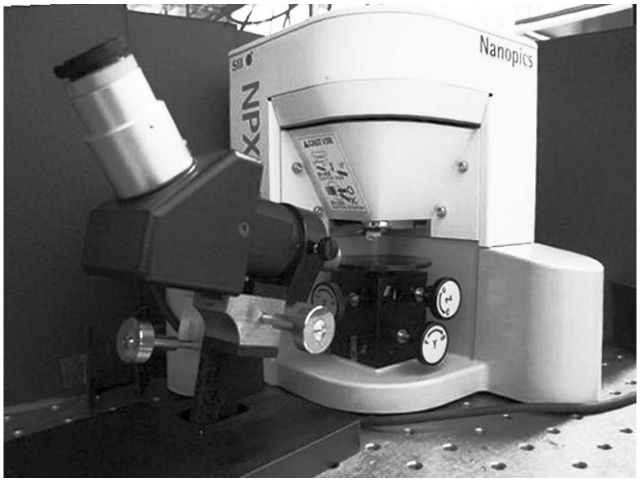

Figure 2. Atomic force microscope.

was performed on all of the AFM topographic images. Surface roughness on a nanometer scale was quantitatively analyzed using Nanopics 2.10. To analyze quantitatively, the three roughness parameters of roughness average (Ra), peak-to-peak height (Rmax), and root mean square (RMS) were used. Ra represented the average of the z-axis height values and could be seen as the most common parameter. Rmax represented the values of the z-axis height of the high-low, and the difference indicates that the smaller was smoother. RMS represents the root mean square value of the z-axis height; this calculation had the advantage of mathematical convenience.

After AFM measuring, the miniscrews were processed for SEM analysis for characterization of their morphologic condition and surface changes after placement. They were coated with platinum by ion sputter (IB-3, Eiko Engineering, Ibataki, Japan) 6 mA for 6 minutes and were examined and photographed with a Hitachi S3000N scanning electron microscope (Hitachi, Tokyo, Japan) at 20 kV acceleration voltage and X35-1000 magnification.

The Wilcoxon signed test was used to compare surface roughness changes before and after insertion. The Kruskal-Wallis test was used to identify significant differences between multiple groups. Analyses were carried out with statistical analysis software (Statistical Package for the Social Sciences [SPSS], version 15.0; SPSS Inc, Chicago, Ill). A probability of $P < .05$ was considered significant.

RESULTS

Measurement of Insertion Torque and Mobility

Insertion torque and initial mobility were not significantly different between anodic oxidized and machined surface miniscrews (Table 1).

SEM Analysis

When the used machined surface miniscrew was scanned at 1000 times the original magnification, the surface of a thread edge close to the tip of the used miniscrew was rougher than that of the unused machined surface miniscrew. No definite surface changes in thread valleys were noted (Figure 3).

A thread edge close to the tip of the used miniscrew had a rougher surface than that of the thread edge close to the head; no definite surface changes in thread valleys were noted (Figure 4).

The anodic oxidized miniscrew had a surface composed of numerous nanotubular and open pores. However, after placement, the open pore shape of a thread edge close to the tip was predominantly erased by smearing as compared with an unused miniscrew (Figure 3).

A thread edge close to the tip of the used miniscrew had a smoother surface than a thread edge close to the head, but no definite surface changes in thread valleys were observed (Figure 4).

AFM Analysis

The surface of the machined surface miniscrew showed scratches produced by the mechanical machining process. However, the anodic oxidized miniscrews did not show the scratches induced on the titanium surface by the machined surface process as a result of the anodic oxidation process (Figure 5).

Table 2 shows the changes in surface roughness parameters of anodic oxidized and machined surface miniscrews by AFM topography images ($5\ \mu m \times 5\ \mu m$). All surface roughness parameters of the used anodic oxidized miniscrews were significantly reduced compared with the unused miniscrews ($P < .05$). Surface roughness parameters of middle thread edges of the used machined surface miniscrew were significantly increased compared with those of the unused miniscrews ($P < .05$). The middle thread edge of the unused anodic oxidized miniscrew had the roughest surface (Table 2), exhibiting many deep and wide valleys.

The used anodic oxidized miniscrew surface was significantly smoother than that of the unused anodic oxidized miniscrew ($P < .05$). However, a middle thread of the used anodic miniscrew surface was rougher than both unused and used machined surface miniscrews ($P < .05$) (Figures 5 and 6).

The surface of a thread edge close to the head and a middle thread edge of the unused and used anodic oxidized miniscrews were significantly rougher than the surface of a thread edge close to the tip ($P < .05$) (Figure 7). Table 2 showed that surface roughness parameters of a thread edge close to the tip were significantly lower than in other regions.

Table 1. Range of Periotest Values (PTV) and Insertion Torque for Initial Loading of Mandible

	Periotest Value (PTV)		Insertion Torque, Ncm	
	Mean	SD	Mean	SD
Machined surface miniscrew	−7.63	0.3	21.68	3.56
Anodic oxidized miniscrew	−7.83	0.26	21.53	6.05

[a] SD indicates standard deviation.

DISCUSSION

The healing time for dental implants without surface treatment is longer than that for implants with treated surfaces.[1,2] With smooth surfaces, biological processes at the bone implant interface are slower, and the properties of the native titanium oxidized layer take longer to be affected. To minimize mineralization time, titanium surface treatment is carried out to accelerate microadhesion formation between implant and bone.

However, to maximize the advantages of the surface treatment on implants or miniscrews, surface changes should be minimal after placement. In this study, before evaluating biological stability, we aimed to determine whether any surface changes are observed in anodic oxidized miniscrews after placement, and if they occur, what their characteristics are and how they affect surface roughness of the miniscrews.

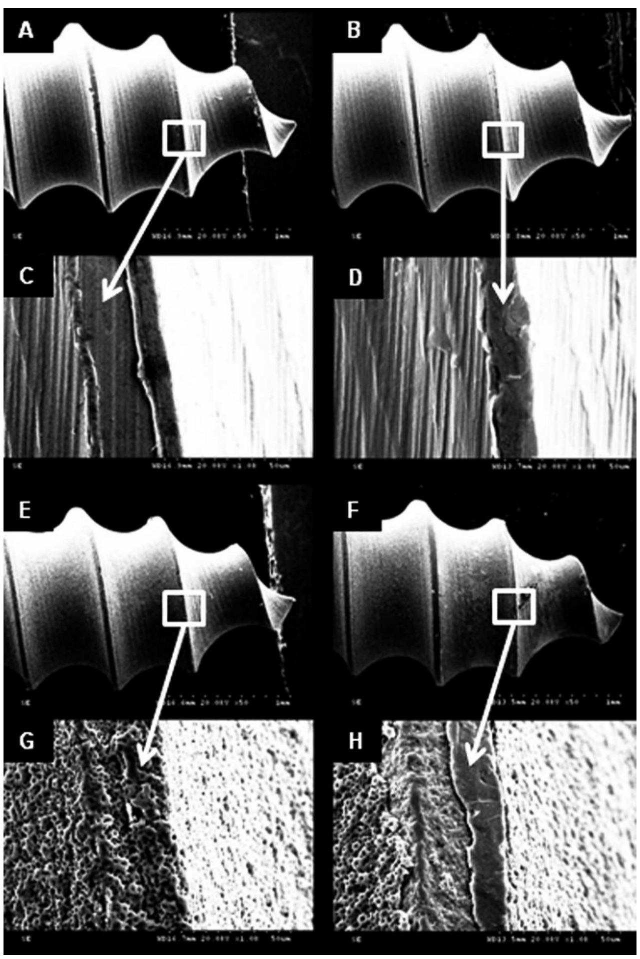

Figure 3. Scanned images of machined surface miniscrews (A through D) and anodic oxidized miniscrews (E through H). (A) Unused miniscrew, ×50. (B) Used miniscrew, ×50. (C) Unused miniscrew, ×1000. (D) Used miniscrew, ×1000. (E) Unused miniscrew, ×50. (F) Used miniscrew, ×50. (G) Unused miniscrew, ×1000. (H) Used miniscrew, ×1000.

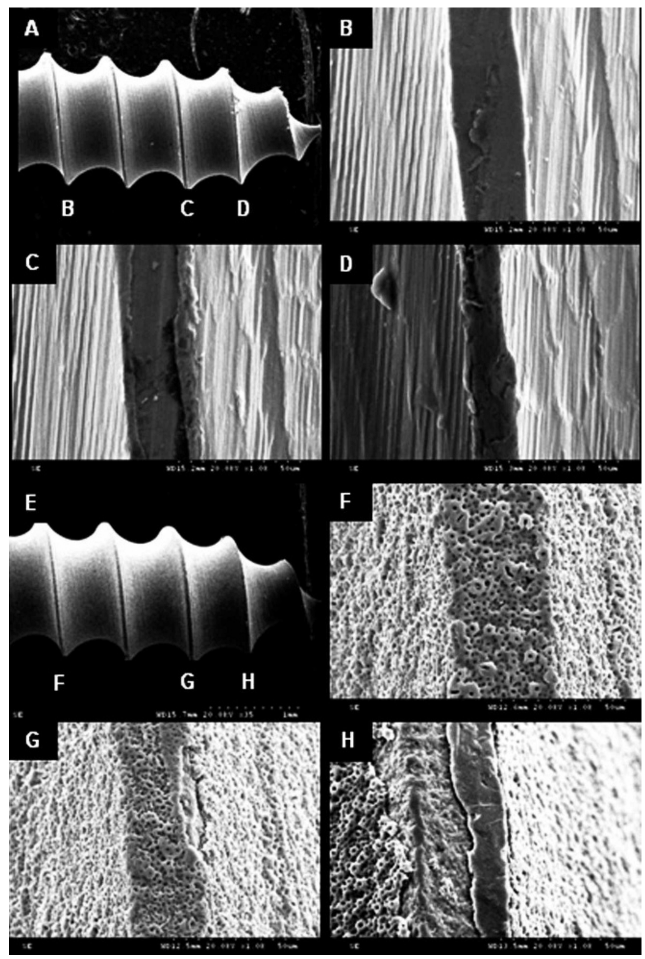

Figure 4. Scanned images of the used machined surface miniscrew (A through D) and the used anodic oxidized miniscrew (E through H). (A) Used miniscrew, ×35. (B) A thread edge close to the head, ×1000. (C) A middle thread edge, ×1000. (D) A thread edge close to the tip, ×1000. (E) Used miniscrew, ×35. (F) A thread edge close to the head, ×1000. (G) A middle thread edge, ×1000. (H) A thread edge close to the tip, ×1000.

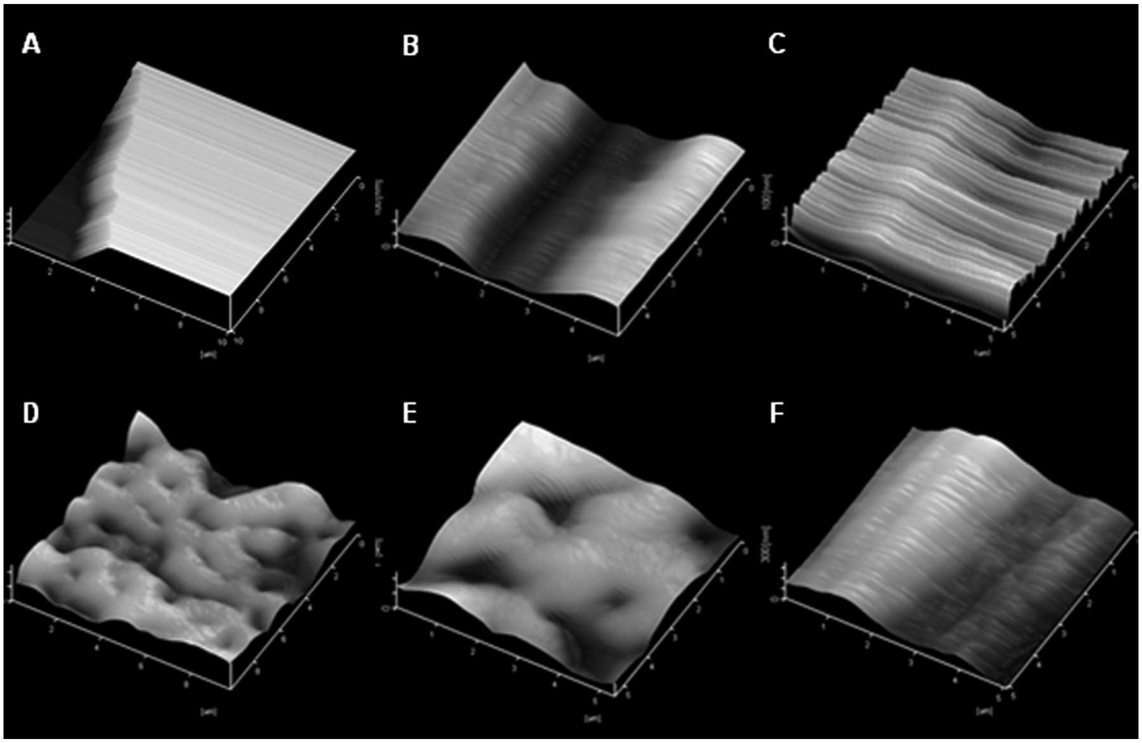

Figure 5. Representative three-dimensional modified AFM images of the miniscrews. (A) A thread valley of the unused machined surface miniscrew. (B) A middle thread edge of the unused machined surface miniscrew. (C) A middle thread edge of the used machined surface miniscrew. (D) A thread valley of the unused anodic oxidized surface miniscrew. (E) A middle thread edge of the unused anodic oxidized miniscrew. (F) A middle thread edge of the used anodic oxidized miniscrew (A, D: 10 μm $\times$ 10 μm; B, C, E, F: 5 μm $\times$ 5 μm).

Before surface changes were observed in the used miniscrews, they were removed from demineralized bone fragments without providing additional torque to preserve surface texture. If they were removed from bone with the use of torque, additional surface damage would be artificially introduced, thus masking the surface damage related to self-drilling insertion and 12 weeks placement in the bone.

SEM demonstrated that only a thread edge close to the tip of the used anodic oxidized miniscrew became smooth by smearing, compared with the unused anodic oxidized miniscrew. A thread edge close to

Table 2. Comparison of Surface Roughness Parameters of Thread Edges of the Miniscrews[a]

		Ra (nm)			Rmax (nm)			RMS (nm)		
			95% CI			95% CI			95% CI	
		Median	Min	Max	Median	Min	Max	Median	Min	Max
Anodic oxidized miniscrew										
Head	Unused	151.6	140.4	162.7	942.3	863.1	1022	185	170.9	199.2
	Used[b]	52.97	42.01	63.93	370.4	258.8	481.9	66.07	52.25	79.89
Middle	Unused	224.4	206.9	241.8	1416	1333	1499	273.4	251.9	295
	Used[b]	62.35	45.97	78.75	317.3	253.1	381.4	73.21	55.24	91.17
Tip	Unused	25.05	20.95	29.15	134.2	107.8	160.7	29.39	24.84	33.94
	Used[b]	7.34	6.22	8.45	62.01	54.86	69.16	9.22	7.94	10.5
Machined surface miniscrew										
Middle	Unused[c,d]	24.29	20.22	28.35	125.7	116.2	135.1	29.38	25.34	33.43
	Used[e,f]	35.97	26.23	45.7	260.1	193	327.2	43.97	32.49	55.45

[a] CI indicates confidence interval; Ra, roughness average; Rmax, peak-to-peak height; and RMS, root mean square.
[b] $P < .05$ (unused anodic oxidized miniscrew vs used anodic oxidized miniscrew).
[c] $P < .05$ (unused anodic oxidized miniscrew vs unused machined surface miniscrew).
[d] $P < .05$ (used anodic oxidized miniscrew vs unused machined surface miniscrew).
[e] $P < .05$ (used anodic oxidized miniscrew vs used machined surface miniscrew).
[f] $P < .05$ (unused machined surface miniscrew vs used machined surface miniscrew).

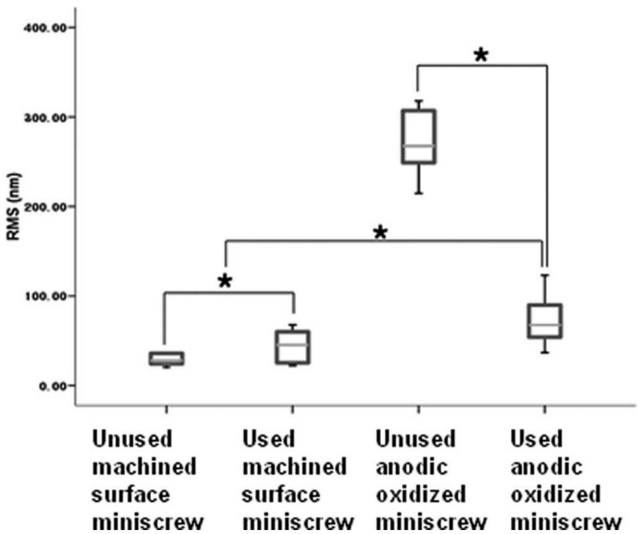

Figure 6. Box plots representing the root mean square (nm) values of a middle thread edge of unused and used miniscrews (*$P < .05$).

the tip of the used machined surface miniscrew became rough, compared with the unused machined surface miniscrew. This change in the miniscrew tip area could be due to tip design and/or stress experienced upon insertion. A self-drilling–type miniscrew tip had the form of a sharp point to increase the cutting force below the cortical tissue. Stress was concentrated in the tip area when the insertion process occurred, so the tip area was more vulnerable than other parts of the miniscrew.

However, as in a previous study,[18] only morphologic validation could be done with SEM, without quantitative and numeric analyses. Therefore, AFM was performed for quantitative analysis.

From the results of AFM measurements, all surface roughness parameters of thread edges of used anodic oxidized miniscrews were significantly reduced in comparison with those of unused miniscrews ($P < .05$). As for the reduction in surface roughness of thread edges of the used anodic oxidized miniscrew, three possible causes could be considered. First, Table 1 shows that the 21.53 Ncm insertion torque value in this study was higher than the 7 to 17 Ncm reported in previous studies; thus a large amount of friction between cortical bone and the miniscrew was expected, and the applied shearing force seemed to be large.[19] According to Motoyosi et al.,[20] proper insertion torque of 5 to 10 Ncm for self-drilling miniscrews was proposed, and an insertion torque greater than this value range could negatively affect stability. Second, because the miniscrews were subjected to tension or torsion over 12 weeks by the NiTi coil spring, surface changes might occur in the miniscrews.[21,22] Third, the deposits around the miniscrews or the materials precipitated on its surface from contact of the miniscrew with biological fluids were likely due to clogs in open pores.

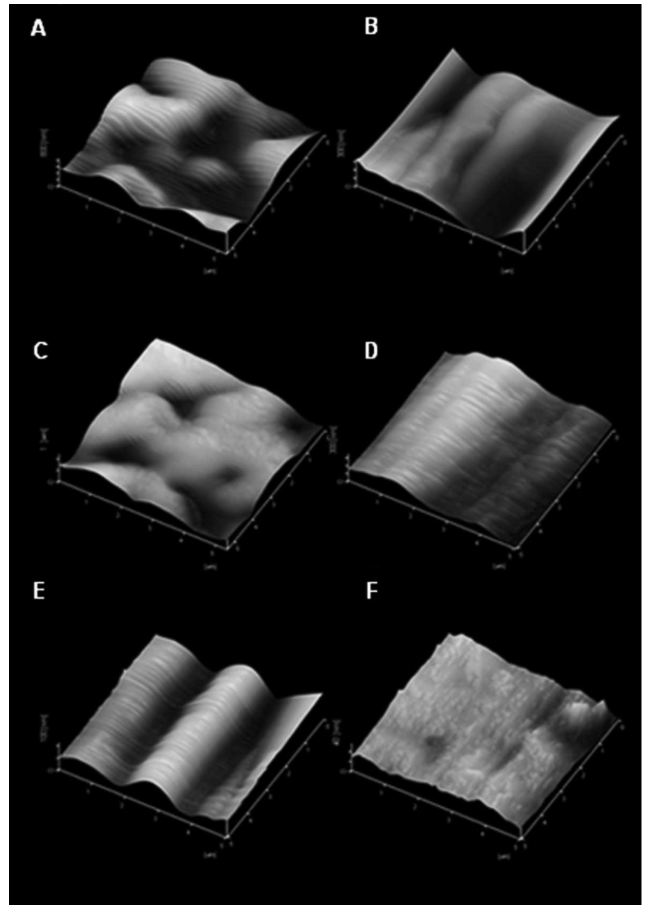

Figure 7. Representative three-dimensional modified AFM images of the anodic oxidized miniscrews. (A) A thread edge close to the head of the unused miniscrew. (B) A thread edge close to the head of the used miniscrew. (C) A middle thread edge of the unused miniscrew. (D) A middle thread edge of the used miniscrew. (E) A thread edge close to the tip of the unused miniscrew. (F) A thread edge close to the tip of the used miniscrew (5 μm $\times$ 5 μm).

The surface of a middle thread edge of unused anodic oxidized miniscrews was the roughest among the others, and their roughness parameters were lower after placement than before placement. However, their roughness parameters were greater than those of the unused or used machined surface miniscrews. Additionally, SEM showed no definite changes in the thread valleys of two groups of miniscrews after placement. The surface area of the thread valleys was greater than that of the thread edges. Anodic oxidized miniscrews would have greater surface area than machined surface miniscrews, even if the surface texture of thread edges of anodic oxidized miniscrews had been changed, and they would tend to maintain their unique surface characteristic. Further research will be needed to evaluate the impact of improved surface characteristics of the anodic oxidized miniscrew on biological stability.

CONCLUSION

- Anodic oxidized miniscrews improve surface characteristics as compared with machined surface miniscrews, even if the surface texture is altered by the self-drilling procedure and during the initial loading period.

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