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New Acid Etched Titanium Dental Implant Surface

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SUMMARY

The purpose of this investigation was to create an acid etched implant surface that results in a surface similar to that gained by using sandblasting combined with acid etching and to compare it with surfaces of commercially available screw-type implants.

Titanium grade V discs were machined in preparation for acid etching. Tests were carried out using different acids and their combinations with variable time exposures. All etched surfaces were scanned with electron microscope (JEOL JSM-5600, Japan) and digital images were made for visual evaluation and description of the surfaces. The etched surfaces were evaluated for surface morphology (combination of micro roughness and waviness) and were best attained by a combination of sulphuric and hydrochloric acids. The etched titanium discs were fixed in resin (two were cut and polished and two – scored and fractured) and the surface profile was examined in.

The second part of the investigation used screw-shaped titanium implants. Twenty-eight screw-shaped implants that were manufactured from commercially available titanium grade V, were selected and divided into two groups: 3 implants as controls (machined surface) and 25 implants processed using the etching methods used in the first part of the investigation. Magnifications of 27, 200 and 2000 were used to analyse the first two consecutive crests of threads, flanks and root of threads of each implant with the treated surface. A three-dimensional optical interferometer (MicroXam, Phase-Shift, USA) was used to characterize the surface roughness of both control and test groups. Three screws were selected from each group and measured at 9 sites: 3 measurements each on the crest, root and flank of the threads. To describe the surface roughness in numbers, the following parameters were used: the average height deviation (Sa), the developed interfacial area ratio (Sdr), the fastest decay autocorrelation length (Sal) and the density of summits (Sds). Surfaces of 5 commercially available screw-type implants and the experimental ones were comparatively analyzed. It was concluded that the new experimental etched titanium surface had features of a roughened titanium surface with glossily micro-roughness and large waviness. In general, the experimental surface was significantly rougher than the selected commercially available implants and similar to SLA treated surface (top Sa 2,08μm (SD 0,36); Sdr 1,34μm (SD 0,3); valley – 1,16μm (SD 0,1) and 0,68μm (SD 0,1); flank – 2,24μm (SD 0,8) and 1,27μm (SD 0,1) respectively).

Key words: commercial titanium, titanium dental implants, endosseous integration, surface texture, acid etching.

INTRODUCTION

Brånemark et al. (1) started the new era of implantology when they published the findings about titanium dental implants in 1969. Since then this method still remains popular and reliable, with only shape and surface of the titanium implants having changed. Excellent titanium biocompatibility assures good tissue integration (2, 3). Baier et al. (4) discussed what features play the most significant role in early acceptance and immobilization of the implant in the tissue bed. Texture, charge and chemistry of the surface as well as cleanliness were considered to be the most important requirements for the implant material (5). Predecki et al (6) observed rapid bone growth and good mechanical adherence with an implant that had an irregular surface. Bowers et al. (7) confirmed these findings in a histological study. Many researchers have been working during the last decade on the development of new surface textures in order to improve initial implant stability and bone healing (8, 9, 10, 11, 12, 13, 14).

Characteristics of titanium implant surfaces have been modified by additive methods (e.g. titanium plasma spray) to increase the surface area and provide a more complex surface macro-topography. Subtractive methods (e.g. blasting, acid etching) have also been used to increase the surface area and to alter its micro-topography or texture (15). Buser et al. (16) analyzed the percentage of direct bone-implant contact for different surface modifications: sandblasted, hydroxyapatite, titanium plasma-sprayed and acid etched. The highest percentage of bone-implant contact was recorded with the sandblasted surface treated by acid etching (hydrochloric and sulphuric acids). Acid etching of titanium is of particular interest because it creates a micro-textured surface (fine rough surface with micro pits of 1-3μm and larger pits of approximately 6-10μm) that appears to enhance early endosseous integration and stability of the implant (17). This may be related to a change in surface roughness and/or chemical composition (13). It has also been shown in rabbits that implants with macro-textured surface (significant waviness with large elements of 10-30μm

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and peaks of different size) ensured better endosseous integration (15). A sandblasted and acid etched surface (SLA) provides both a microroughness and waviness that seems to enhance bone contact with the implant surface. The titanium surface was first sandblasted with large particles creating a grossly rough surface followed by acid etching, forming a micro rough surface (18).

The purpose of this investigation was to create implant surface using acid etching only, which would result in a surface similar to that gained by using sandblasting combined with acid etching. The experimental surface would then be compared with surfaces of commercially available screw-type implants.

MATERIALAND METHODS

Acid etching procedure

Titanium grade V discs (8mm in diameter and 2mm in height) were machined in preparation for acid etching. All discs were etched using pure acids or in combination (Table 1). A series of etching processes were performed changing the exposure duration and acid combination. Exposure times were as follows: 12 hours initial exposure followed by 6 h increments until 72h of exposure was reached.

The titanium discs were etched with 4 different pure acids or their combination at 11 different exposure times at +20^{o} C, thus forming 44 experimental groups with 5 samples in each group for a total of 220 discs.

Topographical evaluation of the titanium discs

All surfaces were scanned with scanning electron microscope (SEM) (JEOL JSM-5600, Japan) using 27, 200 and 2000 magnification and digital images were made for visual examination of the surfaces.

The machined implant surfaces were first characterized using the SEM. The surfaces were oriented in the direction of the machine grooves and the surface was rated on the degree of etching. Surface orientation was unidirectional where the machining grooves were still present. When the machining grooves could not be distinguished, the surface was characterized as complex one.

Another important indicator was regularity of etching. If the surface was etched unequally and had intact areas, it was characterized as irregular surface. Equally etched surface was characterized as regular or uniformly etched surface.

Digital photos were evaluated on the principle that darker spots represented pits and lighter ones – peaks. Pits were measured in diameter without estimating their depth. Micro pits of 1-3µm and bigger elements of approximately 6-10µm formed the microtexture of the surface. Microtexture characterized the roughness of titanium surface while macrotexture consisted of large elements of 10-30µm – it's and was characterized as waviness.

From these results, the etching method that achieved a surface most similar to a SLA surface was selected as the most acceptable. The profile of the selected surface was additionally evaluated visually. Prepared titanium discs were fixed in resin. Two were cut and polished and two – scored and fractured without polishing and a detailed examination of the surface profile was performed. Both profiles appeared to have significant roughness.

Table 1. *Application of different pure acids / their combination in the test groups.*

Group	Acids
I	HCl
II	HCl and H_2SO_4
III	H_2SO_4/HCl and H_3PO_4
IV	H_2SO_4 and HCl

Screw shaped titanium implants

Thereafter we investigated the screw-shaped titanium implants. Twenty-eight screw-shaped implants with 4 threads were manufactured from commercially available titanium grade V (3,5mm in diameter and 6mm in length). Three of them were chosen as control (machined surface) and 25 implants were etched using the method, which was created by the authors on the titanium discs. Five series of surface etching process were performed on 5 implants in each series. Exposition and temperature of etching process were controlled for creation of new standardized implant surface.

Topographical evaluation of the screw shaped titanium implants

Implants were ultrasonically cleaned prior to examination. Implants with the experimental surface were examined with electron microscope (JEOL JSM-5600, Japan) using 27, 200 and 2000 magnification and digital images were made for visual evaluation according to the previously stated principles. The first two consecutive crest of threads, flanks and roots of threads of each implant were analyzed.

A three-dimensional optical interferometer Micro-Xam, Phase-Shift, USA was used to characterize the surface roughness of both control and test group implants. The surfaces of three implants were analyzed topographically according to the method proposed by Wennerberg (19). Three screws were selected from each group and each screw was measured at 9 sites: 3 times on the thread crest, root and flank. Each measured area was 200X200µm. Gaussian filter of 50X50µm size was used to distinguish between roughness and form or undulations in accordance of the requirements of the standard (SS-ISO 11562:1996). To describe the surface roughness in numbers, the following parameters were used: the average height deviation (Sa), the developed interfacial area ratio (Sdr), the fastest decay autocorrelation length (Sal) and the density of summits (Sds).

Statistics

Statistical analyses were performed using the SPSS/PC+ version 10.0.1 program (SPSS Inc., Chicago, Illinois, USA). Standard deviations of the means were calculated.

RESULTS

Topographical evaluation of the titanium discs

The test titanium discs were examined visually is described as follows:

The control group (machined surface) had regular unidirectional grooves with some irregular shallow roughness (Fig.1a);

Group I (etched with HCl) had a microtexture that was poor without evidence of micropits (Fig.1b);

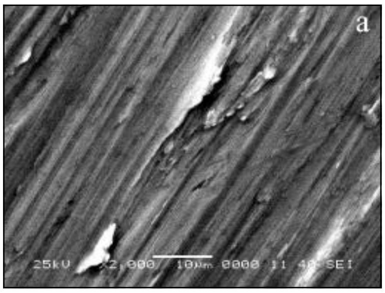
Group II (etched with HCl and H2SO4) yielding a rather rough surface but the microtexture was poor with few micro pits and smooth waviness (Fig.1c). The length of time that Group I and II were subjected to their respective acids did not change the surface texture.

Group III (etched with H2SO4/ HCl and H3PO4) yielded an interesting surface hsowing distinct waviness without microtexture (Fig.1d);

Group IV (etched with H2SO4 for 72h and HCl for 30h) showed significant surface roughness with micro pits of 1-10µm and large valleys of 20-30µm with peaks of different sizes (Fig.1e). The waviness and roughness of the surface was regular and without intact areas.

This acid etching method was selected for further investigation with screw shaped titanium implants.

Evaluation of the surface profile in the cut and polished (Fig.2a) and scored and broken (Fig 2b) groups showed the surface to be rough with small depressions and prominences of 1 to 10µm in size, visible in the profile. Wide trenches of 30µm could be seen (Fig. 2).



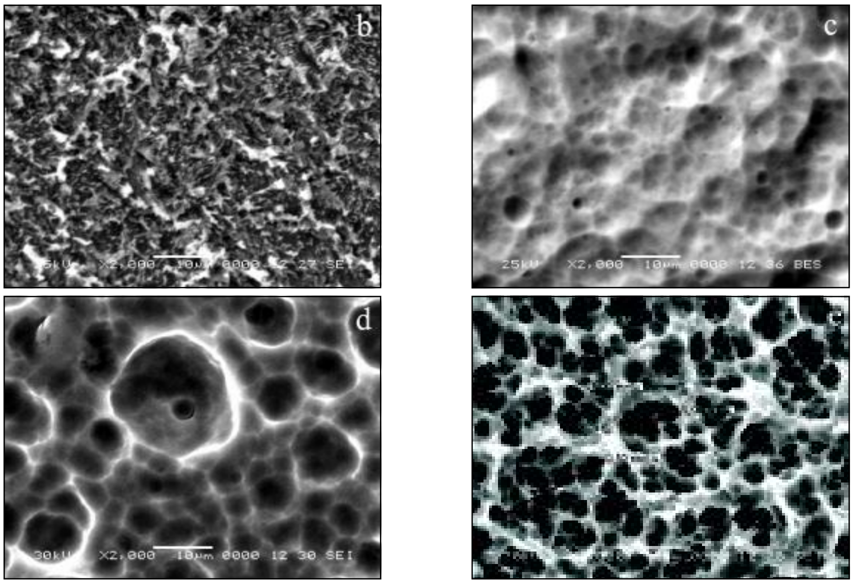


__Fig. 1.__ Scanning electron microscopy images of titanium discs. (a) Disc with machined surface. Regular machining grows apparent on the surface. X2000 mag. (b) Disc with acid etched (HCl) surface with poor microtexture without micropits. (c) Disc with acid etched (HCl and H_2SO_4) surface. Poor microtexture with few micropits and smooth waveness (d) Disc with acid etched (H_2SO_4/HCl and H_3PO_4) surface. Clear expressed surface waveness without microtexture. (e) Disk with acid etched (H_2SO_4 and HCl) surface with 1-10 µm micropits, large 20-30 µm valleys and different size peaks.

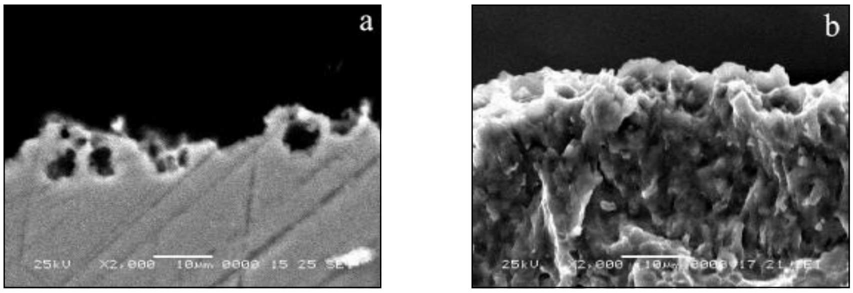


__Fig. 2.__ Scanning electron microscopy images of titanium discs profile. (a) Cut incised-polished disc surface. (b) Cut-fractured disc surface. Both surface profiles exhibited expressed roughness.

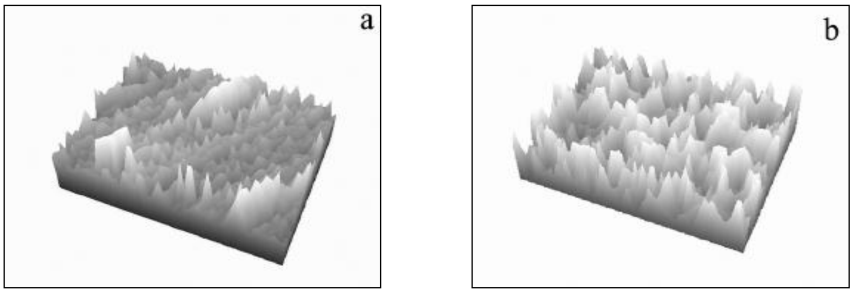


__Fig. 3.__ Digital images from the topographical measurement. (a) Machined surface with clear direction of the surface topography. (b) Acid etched (H_2SO_4 and HCl) surface with the regular distribution expressed peaks and valleys.

Topographical evaluation of the screw shaped titanium implants.

Digital topographical images (Fig. 3) were performed in order to compare machined and acid etched surfaces on screw shaped titanium surfaces. The surfaces of the machined titanium implants were examined and shown that these implants had mainly unidirectional machining grooves and ridges (Fig.3a). The acid treated titanium implants showed roughness and waviness that was evenly spread over the entire surface (Fig.3b). Surface texture was characterized by regularly distributed on the peaks and valleys.

Electron microscopy scans of the machined surface of a control implant had grooves, which were more expressed on thread crests than at the root or flanks of the thread (Fig. 4 a1). The unidirection of deep grooves and ridges remained from the machining process. The implant surface treated with sulphuric and hydrochloric acids was found to have a very complex surface without any intact areas. But the rough-

Table 2. *Surface roughness measured with optical interferometer at different sites of thread on machined and experimental etched thread*

Title	Sa (SD) (µm)		Sal (SD) (µm)		Sds (SD) (/µm2)		Sdr (SD) (%)	
etched								
flank	2,24	(0,81)*	5,44	(1,19)	0,06	(0,00)	127,06	(10,80)*
top	2,08	(0,36)**	7,85	(2,14)	0,07	(0,00)	134,45	(29,10)**
valley	1,16	(0,08)+	6,37	(0,90)	0,07	(0,00)	67,78	(5,31)+
machined								
flank	1,29	(0,98)*	8,01	(8,99)	0,12	(0,01)	29,75	(10,88)*
top	1,28	(0,70)**	10,12	(6,67)	0,11	(0,01)	31,76	(16,85)**
valley	0,62	(0,09)+	6,66	(1,91)	0,09	(0,01)	20,83	(3,79) +
	,,+p<0,04*							**,**,+p<0,001*

Sa - the average height deviation, Sal - the fastest decay autocorrelation length, Sds - the density of summits, Sdr - the developed interfacial area ratio, SD - the standard deviation.

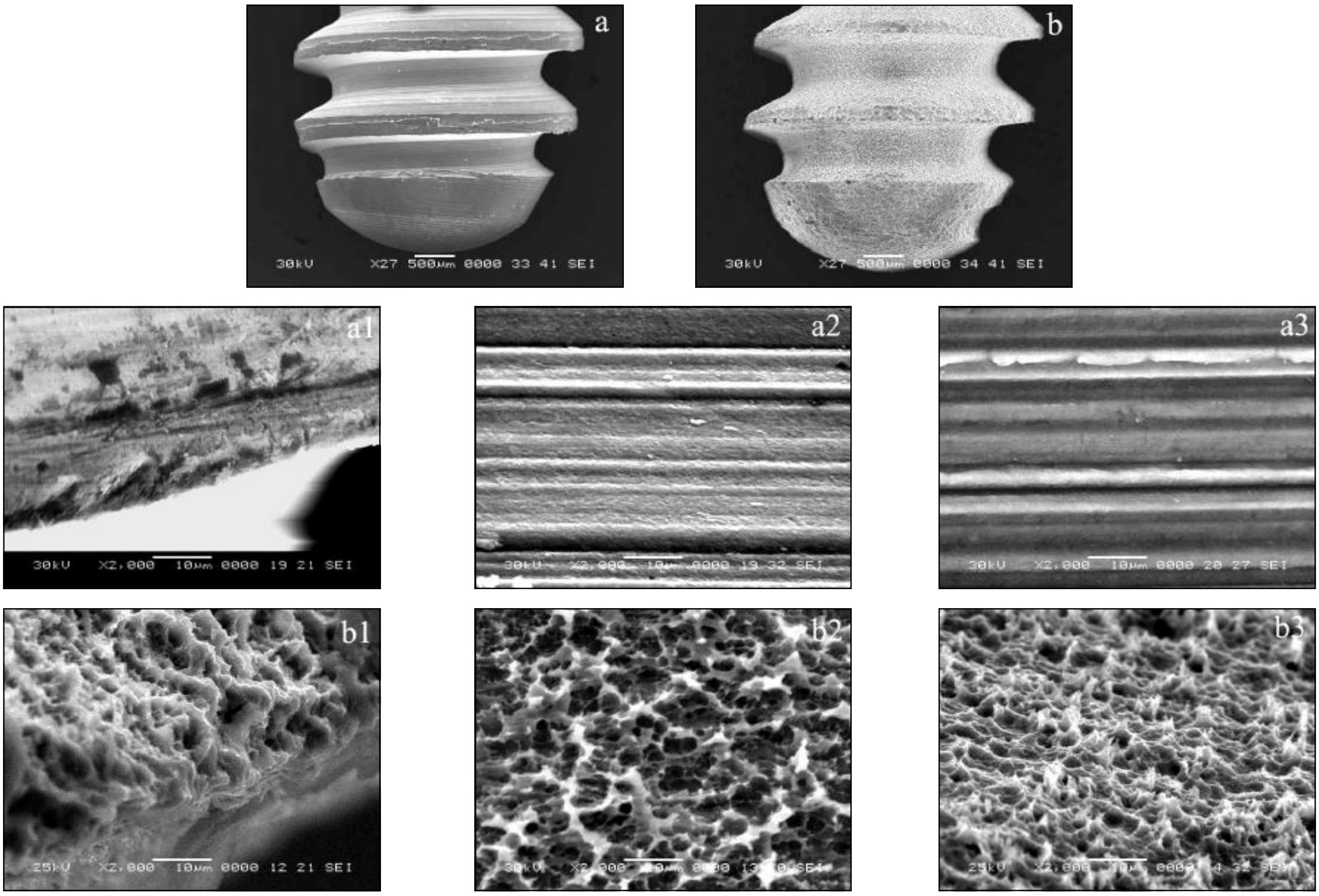


Fig. 4. Scanning electron microscopy images of experimental titanium implants. (a) Implant with machined surface. X27 mag. Clear direction of grooves and ridges as remnants from the machining process. (a1) Machined thread top with deep irregular grooves and ridges. X2000 mag. (a2) A machined thread valley with less distinct ridges and grooves. X2000 mag. (a3) A machined thread flank with distinct ridges and grooves. X2000 mag. (b) implant with acid etched surface X27 mag. Regular distribution of surface texture. (b1) Acid etched thread top with 1-10 µm micropits and large features of approximately 30 µm and different size peaks. X2000 mag. (b2) Acid etched valley with 1-20 µm micropits and small peaks. X2000 mag. (b3) Acid etched flank with clear expressed 1-10 µm micropits, large features of approximately 30 µm and different size peaks. X2000 mag.

ness of the surface was more expressed at the crests and flanks (Fig. 4 b1 and b3) than in the root areas (Fig. 4 b2).

Measurements with an optical interferometer established that the following parameters - height deviation of acid etched surface (Sa), the surface enlargement (Sdr), the fastest decay autocorrelation length (Sal) and the density of summits (Sds), – were significantly bigger than of machined surfaces (Table 2). It is worth noting that common roughness of surface was significant but the roots were again smoother than the crests or the flanks.

The surfaces of 5 commercially produced screw-shaped implants (18) and the implant with the experimental surface were comparatively analyzed taking measurements at the same sites (Table 3). Results showed that all commercially available implants had the smoothest surface at the flanks, while flanks of the experimental implants were the roughest – Sa 2.24µm (SD 0.8). It is worth noting that the experimental implant screws were generally rougher when compared to other commercially available implants. However the surface enlargement (Sdr) was rather similar to the implant with SLA surface.

DISCUSSION

Commercially available implants are available with several surface texture types. This study resulted in the development of a surface texture using acid etching technology. It has been shown that finely pitted (micro pits of 1-3µm and larger elements of approximately 6-10µm in size) surfaces had an early enhancement implant and bone integration (15).

Studies by Wenneberg et al. (11, 19, 20, 21) demon-

Table 3. *Comparison of surface roughness measured at 5 different sites on commercially produced screw-type implants and the experimental implant with optical profilometry.*

Implant /manufacturer	Sa	(SD)	Sdr	(SD)
Nobel Biocare (Goteborg, Sweden)				
top	0,99	(0,5)	1,29	(0,2)
valley	0,60	(0,3)	1,17	(0,1)
flank	0,65	(0,1)	1,26	(0,1)
TiOblast (Astra Tech AB, Molndal,Sweden)				
top	1,27	(0,2)	1,32	(0,1)
valley	1,15	(0,2)	1,31	(0,1)
flank	0,84	(0,1)	1,25	(0,0)
Osseotite (3i, Palm Beach Gardens, FL)				
top	1,97	(1,0)	1,42	(0,2)
valley	0,69	(0,1)	1,12	(0,0)
flank	0,54	(0,1)	1,13	(0,0)
SLA (Institut Straumann AG, Waldenburg, Switzerland)				
top	1,79	(0,2)	1,42	(0,1)
valley	1,32	(0,1)	1,27	(0,0)
flank	1,23	(0,1)	1,30	(0,1)
Bonefit (Institut Straumann AG)				
top	2,08	(0,1)	1,79	(0,0)
valley	2,12	(0,7)	1,91	(0,4)
flank	2,09	(0,2)	1,88	(1,1)
Experimental implant				
top	2,08	(0,4)	1,34	(0,3)
valley	1,16	(0,1)	0,68	(0,1)
flank	2,24	(0,8)	1,27	(0,1)

Sa - the average height deviation, Sdr - the developed interfacial area ratio, SD - the standard deviation.

strated optimal surface roughness of particles of 75µm made surface more resistant to torque and greater bone-to-metal contact than small (25µm) or coarse (250µm) particles. The optimal surface had an average height deviation of about 1.5µm resulting in a surface enlargement of 50%.

Implants with macrotextured surfaces, e.g., plasma sprayed or hydroxyapatite coated, had more enhanced bone to implant contact at late osseointegration period (15). Some authors have reported erosion of the hydroxyapatite layer (22) and peri-implant bone loss resulting in a higher failure rate (12, 23) for implants with hydroxyapatite surface. On the other hand, Buser et al. (16) showed that implants with sandblasted and acid etched surfaces had higher bone to implant contact percentages than implants with titanium plasma sprayed surfaces. This confirms presumption that significantly roughened surface itself (titanium plasma sprayed surface) does not stimulate early bone and implant integration. The titanium surface was sandblasted at first with large particles creating a significantly rough surface, which afterwards was acid etched, forming a finely rough surface. This surface texture improved the initial implant stability in bone of low-density and increased the quality of the bone to implant interface (18). It should be emphasized that this titanium surface was gained using two methods of processing – sandblasting and acid etching. Probability of surface contamination and of micro-particle dissemination into the surrounding tissues is extremely low (24).

In our study we used one method – acid etching – to create a new titanium surface combining all the aforementioned surface texture features. The present study showed that precise acid selection and the sequence of processing played the main role in preparation of the rough titanium surface. The surface was poorer if it was etched with hydrochloric acid and then with sulphuric one. The very similar results were demonstrated processing implants only with hydrochloric acid or with sulphuric/hydrochloric acids and phosphoric one. Sulphuric and hydrochloric acids in sequence and time showed the best results. The newly created titanium surface according its topography was very much alike to a SLA surface. It combines the main properties of roughened titanium surface: glossily micro rough and large waviness. In general, the experimental surface was rougher when compared to commercially available implants.

Although the implant surface created using specific acid etching methods resembles an SLA created surface, further research is necessary to study the biological response to it.

REFERENCES

1. **Brånemark P.-I., Breine B.E., Adell R et al.** Intraosseous anchorage of dental prostheses. I. Experimental studies // Scand. J. Plastic Reconstr. Surg.– 1969, vol. 3, p. 81-100.
2. **Keller J.C., Stanfort C.M., Wightman J.P. et al.** Characterizations of titanium implant surfaces. III // J. Biomed. Mater. Res. – 1994, vol. 18, p. 939-946.
3. **Quirynen M., Bollen C.M., Papaioannou W. et al.** The influence of titanium abutmentsurface roughness on plaque accumulation and gingivitis: short-term observation // Int. J. Oral Maxillofac. Implants – 1996, vol. 11, p. 169-178.
4. **Barier R. F., Natiella J.R., Meyer A. E., Carter J.M.** Importance of implantsurface preparation for biomaterials with different intrinsic properties // Tissue integration in oral and maxillofacial reconstruction / Ed. D. Steenberghe, T. Albrektsson, P.I.Brånemark.- Amsterdam: Excerpta Medica, 1985, p. 13-40.
5. **Lucchini J.-P., Aurelle J.-L., Therin M. et al.** A pilot study comparing screw-shaped implants. Surface analysis and histologic evaluation of bone healing // Clin. Oral Implants Res. – 1996, vol. 7, p. 397-404.
6. **Predecki P., Auslaender B.A.& Stephan J.E.** Attachment of bone to threated implants by ingrowth and mechanical interlocking // J. Biomed. Mater. Res. – 1972, vol. 6, p. 401-412.
7. **Bowers K.T., Keller J.C., Randolph B.A. et al.** Optimization of surface micromorphology for enhanced osteoblast responses in vitro // Int. J. Oral Maxillofac. Implants – 1992, vol. 7, p. 302-310.
8. **Hayashi K., Inadome T., Tsumura H. et al.** Effect of surface roughness of hydroxyapatite-coated titanium on the bone-implant interface shear stregth // Biomaterials – 1994, vol. 15, p. 1187-119
9. **Wong M., Eulenberger J., Schenk R. , Hunzinker E.** Effect of surface topology on the osseointegration of implant materials in trabecular bone // J. Biomed. Mater. Res. - 1995, vol. 29, p. 1567-1575.
10. **Chehroudi B.M., McDonnell D., Brunette D.M.** The effects of micromachined surfaces on formation of bonelike tissue on subcutaneous implants as assessed by radiography and computer image processing // J. Biomed. Mater. Res. – 1997, vol. 34, p. 279-290.
11. **Wennerberg A., Hallgren C., Johansson C. , Danelli S.** A histomorphometric evaluation of screw-shaped implants each prepared with two surfaces roughnesses // Clin. Oral Implants Res. – 1998, vol. 9, p. 11-19.
12. **Piatelli A., Cosci F., Scarano A. , Trisi P**. Localized chronic suppurative bone infection as a sequel of peri-implantitis in a hydroxyapatite-coated dental implant // Biomaterials – 1995, vol. 16, p. 917-920.
13. **Lazzara R., Testori T., Trisi P. , Porter S**. A human histologic analysis of Osseotite and machined surface using implants with 2 opposing surfaces // Int. J. Periodontics Restorative Dent. – 1999, vol. 19, p. 117-129.
14. **Martinez H., Davarpanah M., Missika P., Celleti R., Lazzara R.** Optimal implant stabilization in low density bone / / Clin. Oral Implants Res. – 2001, vol. 12, p. 423-432.
15. **Klokkevold P., Johnson P., Dadgostari S et al.** Early andosseous integration enhanced by dual acid etching of titanium: a torque removal study in the rabbit // Clin. Oral Implants Res.- 2001, vol. 12, p. 350-357.
16. **Buser D., Schenk R.K., Steinemann S. et al.** Influence of surface characteristics on bone integration of titanium implants. A histomorphometric study in miniature pigs (see comments) // J. Biomed. Mater. Res. – 1991, vol. 25, p. 889-902.
17. **Davies J.E.** Mechanisms of endosseous integration // Int. J. Prosthodont. – 1998, vol. 11, p. 391-401.
18. **Wennerberg A., Albrektsson T.** Suggested Guidelines for the Topographic Evaluation of implant surfaces // Int. J. Oral Maxillofac. Implants – 2000, vol. 15, p. 331-344.
19. **Wennerberg A., Albrektsson T. Andersson B. ,Krol J.J.** A histomorphometric and removal torque study of screw-shaped titanium implants with three different surface topographies // Clin. Oral Implants Res. – 1985, vol. 6, p. 24-30.
20. **Wennerberg A., Albrektsson T. Andersson B.** Bone tissue response to commercially pure titanium implants blasted with fine and coarse particles of aluminium oxide // Int. J. Oral Maxillofac. Implants .– 1996, vol. 11, p. 38-45.
21. **Cheang P. , Khor K.A.** Addressing processing problems associated with plasma spraying of hydrxyapatite coatings // Biomaterials – 1996, vol. 17, p. 537-544.
22. **Weehler S.L.** Eight-year clinical retrospective study of titanium plasma-sprayed and hydroxyapatite-coated cylinder implants // Int. J. Oral Maxillofac. Implants – 1996, vol. 11, p. 340-350.
23. **Wilke H.J., Claes L. , Steinemann S.** The influence of various titanium surfaces on the interface shear strength between implants and bone. Clinical Implant Materials // Advances in Biomaterials / Ed. M. Heimke , U. Solstesz , A.J.C.Lee .- Amsterdam: Elsvier Sci. Publ.,1990, p. 309-314.
24. **Lacefield W.R.** Abrasion of dental implant surfaces inserted into bone: Test Results / University of Alabama at Birmingham, Biomaterials Testing Lab., 1997.

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