Enhancement and Characterization of Titanium Surfaces with Sandblasting and Acid Etching for Dental Implants

Busra Balli, Tuncay Dikici, Mustafa Toparli

***Abstract*—**Titanium and its alloys have been used extensively over the past 25 years as biomedical materials in orthopedic and dental applications because of their good mechanical properties, corrosion resistance, and biocompatibility. It is known that the surface properties of titanium implants can enhance the cellular response and play an important role in Osseo integration. The rate and quality of Osseo integration in titanium implants are related to their surface properties. The purpose of this investigation was to evaluate the effect of sandblasting and acid etching on surface morphology, roughness, the wettability of titanium. The surface properties will be characterized by scanning electron microscopy and contact angle and roughness measurements. The results show that surface morphology, roughness, and wettability were changed and enhanced by these treatments.

***Keywords*—**Dental implant, etching, surface modifications, surface morphology, surface roughness.

I. Introduction

MODERN dentistry has been based on restoring contour, function, aesthetic, speech and oral health. Clinical dental implant therapy includes the use of endesseous dental implants [1]. Dental implants are biocompatible metal anchors surgically positioned in the jawbones, used where original teeth are missing and considered as artificial tooth roots [2]. Implant material must be a biocompatible material because implant surface interacts with biological fluids, blood, bone, and tissues.

Titanium and its alloys have been used as implant materials because of their good corrosion resistance, biocompatibility, and mechanical properties [3]. The chemical composition of the surface of titanium implants changes with the chemical composition of implant material and surface modifications. Generally dental implants made from Grade 2 and Grade 4 cpTi materials as they are stronger than other grades [4].

Implant surface properties are important factors affecting the rate, quality, and extent of osseointegration and play a determinant role in biological interactions [5]-[6]. With surface treatments, it is possible to alter and enhance morphology, roughness, and wettability. These surface treatments can be sandblasting, acid etching, alkali treatment, anodization, and titanium plasma spraying [5].

One of the approaches for increase the surface roughness is blasting the surface with hard, chemically stable and biocompatible ceramic particles such as TiO_2, Al_2O_3 or resorbable and osteoconductive particles like calcium phosphates. Different surface roughness values can be achieved by changing the size of ceramic particles. The ceramic particles are projected through a nozzle at high velocity by compressed air [4].

Another approach for increase surface roughness and surface reactivity of the metal is acid etching [7]. This process can be made by immersing the titanium implants into strong acids like H_2SO_4, HCl, HNO_3, and HF. Or a mixture of HCl and H_2SO_4 heated above 100°C (dual-acid etching) is used to produce a homogeneous micro porous surface. On titanium surface micro pits with size ranging 0.5–2 micrometers in diameter can be produced with acid etching process and approximately 138° contact angle value can be obtained in this roughness range [4].

Sandblasting and acid etching (SLA) is most widely used the technique by implant manufacturers. SLA- treated implants have ideal surface roughness, so it provides better bioactivity and bone formation [8]. Even though the idea that a rough surface is more likely to promote the growth of the bone tissue over the implant surface and should give a better anchorage to the implant; the optimum surface roughness is still not determined [9].

The aim of this study to control effect of sandblasting and acid etching processes on the surface morphology and hydrophilicity. We also intended to show how efficient the pressure of ceramic particles that used for sandblasting, acid concentration, and etching time are.

II. Materials and Methods

A. Surface Modification

All discs were polished with SiC papers gradually from 80 to 2000 grit. Then all discs were sandblasted with Al_2O_3 particles size of 250 micrometers and substantially etched using pure acids or in a combination of hydrochloric acid and sulphuric acid. A series of etching processes were performed by changing the etching time and acid concentrations.

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All etching procedures were done by using hydrochloric acid and sulphuric acid in combination. Two sandblasting and etching procedures were described in Tables I and II.

TABLE I
FIRST SANDBLASTING AND ETCHING PROCEDURE AT 60°C

	1	*2*	*3*	*4*
Sandblasting (1 minutes)	2 bars	3 bars	4 bars	5 bars
HCl/H_2SO_4	4:1	3:1	2:1	-
	5	6	7	8
Sandblasting (1.5 minutes)	2 bars	3 bars	4 bars	5 bars
HCl/H_2SO_4	1:4	1:3	1:2	1:1

In the second group, all 7 samples were sandblasted with Al_2O_3 at 4 bars pressure during 1.5 minutes. Afterwards, etching procedure was performed with different times and acid concentrations. 7 different acid concentration and etching time combinations were described in Table II.

TABLE II
SECOND ETCHING PROCEDURE WITH DIFFERENT EXPOSURE TIME AND ACID CONCENTRATIONS AT 60°C

Sample Number	Acid concentration (HCl/H_2SO_4)	Time (minutes)
1	1:2	15
2	2:1	15
3	1:1	5
4	1:1	15
5	1:1	30
6	1:1	60
7	1:1	120

B. Characterization

After sandblasting and acid etching processes, the surface roughness of titanium samples were measured by profilometry, surface roughness was determined by Ra alteration. Also by contact angle measurements hydrophilicity of surfaces were determined and surface morphologies of titanium samples were characterized by scanning electron microscopy images.

III. RESULTS AND DISCUSSION

After the first sandblasting procedure, Ra values of the first group of samples were given in Fig. 1. They all have proper surface roughness which were in 0.5-2.0 micrometers range, but the surfaces were not uniform enough for osteoblastic proliferation. They should have been made all uniform with any further treatment like acid etching.

Considering both surface roughness and morphology results from the first procedure the best results combination are belonging to sample 6. Its surface roughness was 0.88 micrometers which should be between 0.5 and 2 micrometers which are compatible with [4] and its surface morphology had very homogeneous and uniform micro pits for using as a dental implant surface. "Fig. 2" has shown the scanning electron microscopy image of surface morphology of sample 6 from first sandblasting and etching procedure which was sandblasted with 3 bars during 1.5 minutes and substantially etched with HCl/H_2SO_4 combination in 1:3 ratios in concentration.

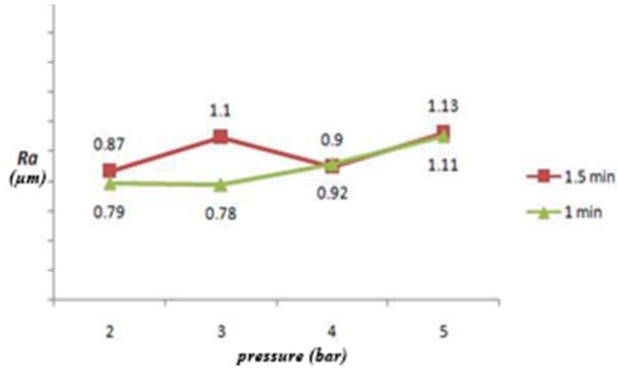

Fig. 1 Ra values depending on blasting time and pressure

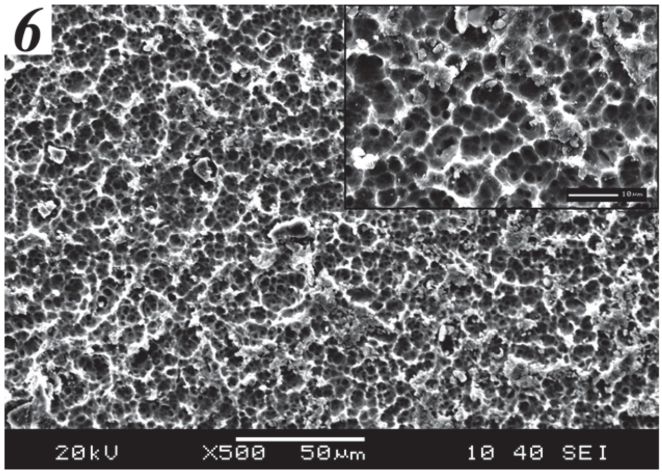

Fig. 2 Scanning electron microscope image of surface morphology of sample 6 from first sandblasting and etching procedure

From the surface roughness measurements of both first and second sample groups, the highest Ra value (3.72 micrometers) was observed at Titanium surface which has etched with 1:1 HCl/H_2SO_4 ratio during 60 minutes. From the contact angle measurements, it was observed that the most hydrophilic surface (4°) is the same surface with the surface having the highest surface roughness. Scanning electron microscopy images of the surfaces were examined, observed cracks formed on the surface due to the length of the etching time (60 minutes). Scanning electron microscopy images of first, second and third samples of second etching procedure were shown in Fig. 3. From the examination of images, it could be seen that the most proper surface morphology and homogeneous surface roughness could be obtained with 5 and 15 minutes etching time and 1:1, 1:2, 2:1 HCl/H_2SO_4 acid ratios at first, second and third samples. However, as given in Table III, these surfaces have lower Ra and higher contact angle values. Their contact angle measurement images can be seen in Fig. 4. Fig. 4 has shown the contact angle measurement images of first, second and third samples of second etching procedure. It was indicated in Fig. 4 that contact angles are too high to be used for dental implants because of advance of lower contact angle in bone-to-implant contact and increase in removal torque [10].

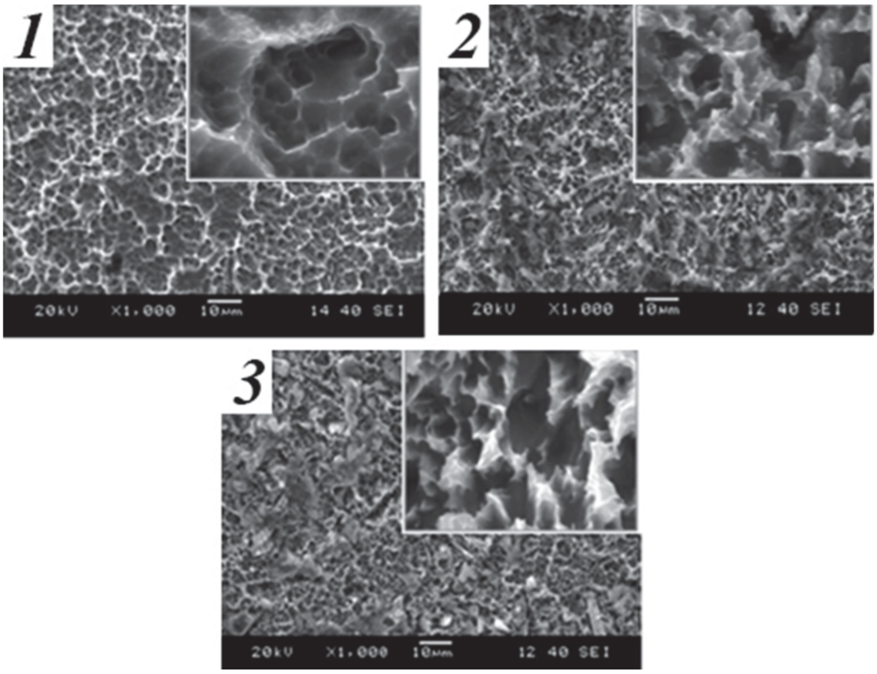

Fig. 3 Scanning electron microscopy images of surface morphologies of first, second and third surfaces from second sandblasting and etching procedure

When both surface morphologies and roughness and contact angles were examined the best results were obtained at the surface which was etched in 1:1 HCl/H_2SO_4 ratio with 15 minutes etching time. This is sample 5 from second etching procedure. This treatment gave us 8.4±1.1° contact angle value and 1.62 micrometers Ra value which is compatible with [4] and a homogeneous surface morphology. It could be clearly seen from Fig. 5 and Fig. 6 that it has a rough and uniform surface and lower contact angle and hence higher hydrophilicity that is needed in dental implant surfaces [4]-[10].

TABLE III
RESULTS OF 4 SAMPLES FROM SECOND ETCHING PROCEDURE

Sample number	Ra value	Contact angle value
1	0.94 μm	52.6 ± 2.2°
2	0.97 μm	49.7 ± 3.8°
3	0.85 μm	128.6 ± 5.8°
5	*1.62 μm*	*8.4 ± 1.1°*

Fig. 4 Contact angles of first, second and third samples from second sandblasting and etching procedure

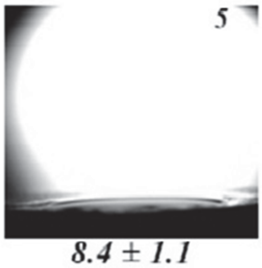

Fig. 5 Contact angle of the fifth sample from second sandblasting and etching procedure

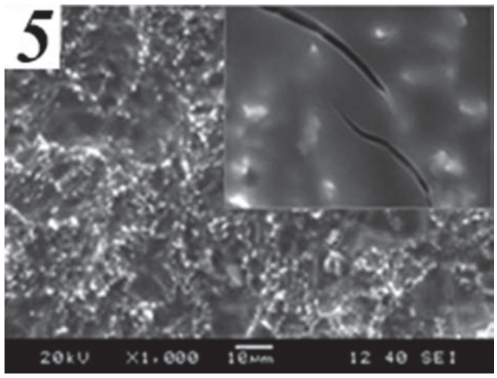

Fig. 6 Scanning electron microscopy image of fifth surface morphology from second sandblasting and etching procedure

Fig. 6 has shown the scanning electron microscopy image and Fig. 5 has shown the contact angle measurement image of fifth surface morphology from second procedures which was sandblasted with Al_2O_3 at 4 bars pressure during 1.5 minutes.

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REFERENCES

[1] C. N. Elias, "Titanium dental implant surfaces," *Revista Matéria,* vol. 15, pp. 138–142, June, 2010.
[2] Y. Oshida, B. T. Tuna, O. Aktoren, K. Gencay, "Dental implant systems," *International Journal of Molecular Sciences,* vol. 11, pp. 1580-1568, April 2010.
[3] H. P. Hyeoung, P. Song, S. K. Kyeong, Y. J. Woo, K. P. Byeung, S. K. Hyung, S. B. Tae, H. L Min, "Bioactive and electrochemical characterization of TiO_2 nanotubes on titanium via anodic oxidation," *Electrochimica Acta,* vol. 55, pp. 6109-6114, August 2010.
[4] L. L. Gu´ehennec, A. Soueidan, P. Layrolle, Y. Amouriq, "Surface treatments of titanium dental implants for rapid osseointegration," *Dental Materials*, vol. 23, pp. 844-854, July 2007.
[5] S. Lavenus, G. Louarn, P. Layrolle, "Nanotechnology and Dental Implants," *International Journal of Biomaterials,* vol. 2010, pp 1-9, December 2010.
[6] G. Mendonca, D. B. S. Mendonca, F. J. L. Aragao, L. F. Cooper, "Advancing dental implant surface technology from micron- to nanotopography," *Biomaterials*, vol. 29, pp. 3822-3825, October 2008.
[7] H. Garg, G. Bedi, A. Garg, "Implant Surface Modifications: A Review" *Journal of Clinical and Diagnostic Research*, vol. 6, pp. 319-324, April 2012.
[8] Z. Lin, Y. Wang, D. N. Wang, B. Zhao, J. Chang, "Porous structure preparation and wettability control on titanium implant," *Surface & Coatings Technology,* vol. 228, pp. 131-136, July 2013.
[9] R. V. Bathomarco, G. Solorzano, C. N. Elias, R. Prioli, "Atomic force microscopy analysis of different surface treatments of Ti dental implant surfaces," *Applied Surface Science,* vol. 233, pp. 29-34, May 2004.
[10] C. N. Elias, Y. Oshida, J. H. C. Lima, C. A. Muller, "Relationship between surface properties (roughness, wettability, and morphology) of titanium and dental implant removal torque," *Journal of the Mechanical Behavior of Biomedical Materials,* vol. 1, pp. 234-242, July 2008.