The purpose of this investigation was to create an acid-etched implant surface that is similar to that created by sandblasting combined with acid etching and to compare it with the surfaces of various commercially available screw-type implants. Titanium grade 5 disks were machined in preparation for acid etching. Tests were carried out using different acids and combinations of them with varying time exposures. All etched surfaces were scanned with an electron microscope, and digital images were created for visual evaluation and description of the surfaces. The etched surfaces were evaluated for surface morphology (combination of microroughness and waviness); the surface most like the sandblasted/acid-etched surface was best obtained with a combination of sulfuric and hydrochloric acids. The etched titanium disks were fixed in acrylic resin (2 were cut and polished and 2 were scored and fractured) and the surface profile was examined. In the second part of the investigation, 28 screw-shaped implants that were manufactured from commercially available titanium grade 5 were selected and divided into 2 groups: 3 implants were used as controls (machined surface), and 25 implants were processed using the preferred etching method determined in the first part of the investigation. Magnifications of 27, 200, and 2000 were used to analyze the first 2 consecutive crests of threads, flanks, and root of threads of each implant with the treated surface. A 3-dimensional optical interferometer 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). In addition, in a third experiment, the surfaces of 5 commercially available screw-type implants and the experimental ones were analyzed and compared. It was concluded that the new experimental acid-etched titanium surface had the features of a roughened titanium surface, with glossily microroughness and large waviness. In general, the experimental surface was significantly rougher than the selected commercially available implants and similar to a sandblasted/acid-etched surface (top Sa: 2.08 ± 0.36 μm, Sdr: 1.34 ± 0.3 μm, valleys: 1.16 ± 0.1 μm and 0.68 ± 0.1 μm, flanks: 2.24 ± 0.8 μm and 1.27 ± 0.1 μm, respectively).

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

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chemistry of the surface as well as cleanliness were considered to be the most important requirements for the implant material. Predecki et al. observed rapid bone growth and good mechanical adhesion with an implant that had an irregular surface. Bowers et al. confirmed these findings in a histologic study. Many researchers have been working during the last decade on the development of new surface textures in attempts to improve primary implant stability and bone healing.

Titanium implant surfaces have been modified by additive methods (eg, titanium plasma spraying) to increase the surface area and provide a more complex surface macrotopography. Subtractive methods (eg, blasting, acid etching) have also been used to increase the surface area and to alter its microtopography or texture. Buser et al. analyzed the percentage of direct bone-implant contact for different surfaces: sandblasted, hydroxyapatite coated, titanium plasma sprayed, and acid etched (and different combinations of these processes). The highest percentage of bone-implant contact was recorded at the sandblasted surface treated by acid etching (hydrochloric and sulfuric acids). Acid etching of titanium is of particular interest because it creates a microtextured surface (fine rough surface with micropits of 1 to 3 \( \mu m \) and larger pits of approximately 6 to 10 \( \mu m \)) that appears to enhance early endosseous integration and stability of the implant. It has also been shown in rabbits that implants with a macrotextured surface (fine rough surface with micropits of 1 to 3 \( \mu m \) and larger pits of approximately 6 to 10 \( \mu m \)) that appears to enhance early endosseous integration and stability of the implant. This may be related to a change in surface roughness and/or chemical composition. It has also been shown in rabbits that implants with a macrotextured surface (fine rough surface with micropits of 1 to 3 \( \mu m \) and larger pits of approximately 6 to 10 \( \mu m \)) that appears to enhance early endosseous integration and stability of the implant.

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

### MATERIAL AND METHODS

**Acid-etching procedure**

Titanium grade 5 disks (8 mm in diameter and 2 mm in height) were machined in preparation for acid etching. All disks were etched using acids, either alone or in combination (Table 1). A series of etching processes was performed, with the duration of exposure and acid combination changed. Exposure times were as follows: 12 hours initial exposure followed by 6-hour increments until 72 hours of exposure were reached.

The titanium disks were etched with 4 different pure acids or a combination of these at 11 different exposure times at \( \pm 20^\circ C \). Thus there were 44 experimental groups with 5 samples in each group, for a total of 220 disks.

**Topographic evaluation of the titanium disks**

All surfaces were examined with a scanning electron microscope (SEM) (JEOL JSM-5600, Tokyo, Japan) using \( \times 27, \times 200 \), and \( \times 2000 \) magnification. 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 designated as unidirectional when the machining grooves were still present. When the machining grooves could not be distinguished, the surface was characterized as complex.

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

Digital photos were evaluated on the principle that darker spots represented pits and lighter ones represented peaks. The diameter but not depth of the pits was measured. Micropits of 1 to 3 \( \mu m \) and larger elements of approximately 6 to 10 \( \mu m \) formed the microtexture of the surface. The term “microtexture” was used to characterize the roughness of titanium surfaces, whereas “macrotexture” consisted of large elements of 10 to 30 \( \mu m \) and was characterized as waviness.

### Table 1

<table>
<thead>
<tr>
<th>Group</th>
<th>Acids*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>HCl</td>
</tr>
<tr>
<td>2</td>
<td>HCl and H(_2)SO(_4)</td>
</tr>
<tr>
<td>3</td>
<td>H(_2)SO(_4)/HCl and H(_3)PO(_4)</td>
</tr>
<tr>
<td>4</td>
<td>H(_2)SO(_4) and HCl</td>
</tr>
</tbody>
</table>

*HCl indicates hydrochloric acid; H\(_2\)SO\(_4\), sulfuric acid; H\(_3\)PO\(_4\), phosphoric acid.
From these results, the etching method that achieved a surface most similar to an SAE surface was selected as the most acceptable. The profile of the selected surface was additionally evaluated visually with the SEM. Prepared titanium disks were fixed in acrylic resin. Two were cut and polished, and 2 were scored and fractured without polishing; a detailed examination of the surface profiles was then performed. Both profiles appeared to have significant roughness.

**Screw-shaped titanium implants**

The second portion of the study investigated screw-shaped titanium implants. Twenty-eight screw-shaped implants with 4 threads each were manufactured from commercially available titanium grade 5 (3.5 mm in diameter and 6 mm in length). Three were chosen as controls (machined surface), and 25 implants were etched using the method created earlier on the titanium disks. Five series of surface-etching process were performed on 5 implants in each series. Exposition and temperature of the etching process were controlled for creation of new standardized implant surfaces.

**Topographic evaluation of the screw-shaped titanium implants**

Implants were ultrasonically cleaned prior to examination. Implants with the experimental surface were examined with the SEM using ×27, ×200, and ×2000 magnification, and digital images were made for visual evaluation according to the previously stated principles. The first 2 consecutive crests of threads, flanks, and roots of threads of each implant were analyzed. A 3-dimensional optical interferometer (Micro-Xam, Phase-Shift/ADE, Tucson, AZ) was used to characterize the surface roughness of both control and test group implants. The surfaces of 3 implants in each group were analyzed topographically according to the method proposed by Wennerberg and Albrektsson.24 Three screws were selected from each group, and each screw was measured at 9 sites (3 times each on the thread crest, root, and flank). Each measured area was 200 × 200 μm. A Gaussian filter of 50 × 50 μm was used to distinguish between roughness and form or undulations in accordance of the requirements of the ISO standard (SS-ISO 11562:1996). To describe the surface roughness in numbers, the following parameters were used: average height deviation (Sa), developed interfacial area ratio (Sdr), fastest decay autocorrelation length (Sal), and density of summits (Sds). The surface of 5 different commercially produced screw-shaped implants23 and the implant with the experimental surface were comparatively analyzed by taking measurements at the same sites.

**Statistics**

Statistical analyses were performed using the SPSS/PC+ version 10.0.1 program (SPSS Inc, Chicago, Ill). Means and standard deviations were calculated.

**RESULTS**

**Topographic evaluation of the titanium disks**

The titanium test disks were examined visually and described as follows.

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

Group 1 (etched with hydrochloric acid [HCl]) had a microtexture that was poor, without evidence of micropits (Figure 1b).

Group 2 (etched with HCl and sulfuric acid [H2SO4]) yielded a rather rough surface, but the microtexture was poor, with few micropits and smooth waviness (Figure 1c). The length of time that groups I and II were subjected to their respective acids did not change the surface texture.

Group 3 (etched with H2SO4/HCl and phosphoric acid [H3PO4]) yielded an interesting surface that showed distinct waviness without microtexture (Figure 1d).

Group 4 (etched with H2SO4 for 72 hours and HCl for 30 hours) showed significant surface roughness, with micropits of 1 to 10 μm and large valleys of 20 to 30 μm with peaks of different sizes (Figure 1e). The waviness and roughness of the surface were regular and without intact areas.

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

**Topographic evaluation of the screw-shaped titanium implants**

The group 4 acid-etching method (etched with H2SO4 for 72 hours and HCl for 30 hours) was selected for further investigation with screw-shaped titanium implants. Digital topographic images (Figure 3) were created so that the machined and acid-etched surfaces on screw-shaped titanium implants could be compared. The surfaces of the machined titanium implants were examined; these implants had mainly unidirec-
Electron microscopic scans of the machined surface of a control implant showed grooves, which were more pronounced on thread crests than at the roots or flanks of the threads (Figure 4a). The unidirectionality of deep grooves and ridges remained.

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TITANIUM IMPLANT SURFACE OPTIMIZATION

Figure 1. Images of electron microscopic scans of titanium disks. (a) Disk with machined surface. Regular machining grooves are apparent on the surface (magnification ×2000). (b) Disk with hydrochloric acid–etched surface shows a poor microtexture without micropits. (c) Disk with hydrochloric/sulfuric acid–etched surface displays a poor microtexture with few micropits and smooth waviness. (d) Disk with surface etched by sulfuric/hydrochloric acids and phosphoric acid. Surface waviness is clearly expressed without microtexture. (e) Disk with surface etched by sulfuric and hydrochloric acid shows micropits of 1 to 10 μm, large valleys of 20 to 30 μm, and peaks of different size.

Functional machining grooves and ridges (Figure 3a). The acid-treated titanium implants showed roughness and waviness that were evenly spread over the entire surface (Figure 3b). Surface texture was characterized by regularly distributed peaks and valleys.
from the machining process. The implant surface treated with sulfuric and hydrochloric acids was found to have a very complex surface without any intact areas, but the roughness of the surface was more pronounced at the crests and flanks (Figure 4b and 4d) than in the root areas (Figure 4c).

Measurements with an optical interferometer established that $S_a$, $S_{dr}$, $S_{al}$, and $S_{ds}$ were significantly greater on acid-etched surfaces than on machined surfaces (Table 2). It is worth noting that the roughness of the acid-treated surface was significant, but, again, the roots were smoother than the crests or the flanks.

The surfaces of 5 commercially produced screw-shaped implants and the implant with the experimental surface were comparatively analyzed by taking measurements at the same sites (Table 3). The results showed that all commercially available implants had the smoothest surface at the flanks, whereas the flanks of the experimental implants were the roughest ($S_a 2.24 \pm 0.8 \mu m$). It is worth noting that the experimental implant screws were generally rougher than other commercially available implants. However, the surface enlargement ($S_{dr}$) was rather similar to that of the SLA implant (sandblasted/acid-etched surface, Institut Straumann, Waldenburg, Switzerland).

**DISCUSSION**

Commercially available implants are present 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 (micropits of 1 to 3 $\mu$m and larger elements...
approximately 6 to 10 μm) surfaces result in early
enhancement of bone-implant integration.20

Studies by Wennerberg et al12,23–26 demonstrated
that an optimal surface roughness (75-μm particles)
made surfaces more resistant to torque and resulted in
greater bone-to-metal contact than small (25-μm) or
course (250-μm) particles. The optimal surface had an
average height deviation of about 1.5 μm, resulting in
a surface enlargement of 50%.

Implants with macrotextured surfaces (eg, plasma
sprayed or hydroxyapatite coated) have shown
enhanced bone-to-implant contact during the late
osseointegration period.25 Some authors have report-
ed erosion of the hydroxyapatite layer27 and peri-
implant bone loss, resulting in a higher failure rate12,28
for implants with hydroxyapatite-coated surfaces. On
the other hand, Buser et al21 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
the presumption that a significantly roughened
surface (titanium plasma–sprayed surface) does not
by itself stimulate early bone and implant integration.

The titanium surface was first sandblasted with large
particles, creating a significantly rough surface that
was then acid etched, forming a finely rough surface.
This surface texture improved primary implant stability
in bone of low density and improved the quality of the
bone-to-implant interface.25 It should be emphasized
that this titanium surface was gained using 2 methods
of processing: sandblasting and acid etching. The
probability of surface contamination and of micropar-
ticle dissemination into the surrounding tissues is
extremely low.29 The study of Diniz et al30 showed that
characterization of the titanium surface is essential in
the evaluation of the material manufacturing process,
because the presence of residual aluminum particles
may have deleterious effects on the formation of the
osseous peri-implant tissue. Furthermore, Mueller et
al31 proved that metal-bone contact showed a
tendency for more bone when bioceramics, rather
than aluminum oxide, were used as blasting materials.

The present study used a single method—acid
etching—to create a new titanium surface that
included all the aforementioned surface texture
features. The present study showed that precise
selection of acids and of the sequence of the etching
process played primary roles in preparation of the
rough titanium surface. The surface was less rough if it
was etched with HCl and then H2SO4. Very similar
results were demonstrated when processing implants
with HCl only or with H2SO4/HCl and then H3PO4.

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was etched with HCl and then H2SO4. Very similar
results were demonstrated when processing implants
with HCl only or with H2SO4/HCl and then H3PO4.
H2SO4 and HCl applied in sequence and time showed
the best results. The topography of the newly created
titanium surface was very much like that of a
sandblasted and acid-etched surface. It combined

| Table 2
| Surface roughness as measured with optical interferometer at different locations of threads on machined and experimental acid-etched implants |
| Implant Type | 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 (0.02)*** | 6.66 (1.91) | 0.02 (0.01) | 20.83 (3.79)*** |

* P < .05 for Sa; ** P < .01 for Sdr.
†Sa indicates average height deviation; Sdr, developed interfacial area ratio; Sal, fastest decay autocorrelation length; and Sds, density of
summits.

FIGURES 4 AND 5. Images of electron microscopic scans of experimental titanium implants. FIGURE 4. (a) Implant with machined surface
(magnification ×27). A clear direction of grooves and ridges remains from the machining process. (b) Top of machined thread with
irregular deep grooves and ridges (magnification ×2000). (c) Machined thread with less distinct ridges and grooves (magnification ×2000).
(d) Valley and flank of machined thread with distinctive ridges and grooves (magnification ×2000). FIGURE 5. (a) Implant with acid-etched
surface (magnification ×27). Regular distribution of surface texture. (b) Top of acid-etched thread with micropits of 1 to 10 μm, large
elements of approximately 30 μm, and peaks of different size (magnification ×2000). (c) Acid-etched valley with micropits of 1 to 20 μm
and small peaks (magnification ×2000). (d) Acid-etched flank with clearly expressed micropits of 1 to 10 μm, large elements of
approximately 30 μm, and peaks of different size (magnification ×2000).
the main properties of a roughened titanium surface: glossy micromoughness and pronounced waviness. In general, the experimental surface was rougher than of than commercially available implants.

Although the implant surface created using specific acid-etching methods resembles a surface created by both sandblasting and acid etching, further research is necessary to study the biologic response to it.

**REFERENCES**


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**TABLE 3**

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

<table>
<thead>
<tr>
<th>Implant and Manufacturer</th>
<th>Sa</th>
<th>SD</th>
<th>Sdr</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nobel Biocare (Goteborg, Sweden)</td>
<td>Top 0.99</td>
<td>0.5</td>
<td>1.29</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>Valley 0.60</td>
<td>0.3</td>
<td>1.17</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>Flank 0.65</td>
<td>0.1</td>
<td>1.26</td>
<td>0.1</td>
</tr>
<tr>
<td>TiOblast (Astra Tech, Molndal, Sweden)</td>
<td>Top 1.27</td>
<td>0.2</td>
<td>1.32</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>Valley 1.15</td>
<td>0.2</td>
<td>1.31</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>Flank 0.84</td>
<td>0.1</td>
<td>1.25</td>
<td>0.0</td>
</tr>
<tr>
<td>Osseotite (3i, Palm Beach Gardens, FL)</td>
<td>Top 1.97</td>
<td>1.0</td>
<td>1.42</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>Valley 0.69</td>
<td>0.1</td>
<td>1.12</td>
<td>0.0</td>
</tr>
<tr>
<td></td>
<td>Flank 0.54</td>
<td>0.1</td>
<td>1.13</td>
<td>0.0</td>
</tr>
<tr>
<td>SLA (Institut Straumann, Waldenbourg, Switzerland)</td>
<td>Top 1.79</td>
<td>0.9</td>
<td>1.42</td>
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</tr>
<tr>
<td></td>
<td>Valley 1.32</td>
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<td>1.27</td>
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</tr>
<tr>
<td></td>
<td>Flank 1.23</td>
<td>0.1</td>
<td>1.30</td>
<td>0.1</td>
</tr>
<tr>
<td>Bonefit (Institut Straumann)</td>
<td>Top 2.08</td>
<td>0.1</td>
<td>1.79</td>
<td>0.0</td>
</tr>
<tr>
<td></td>
<td>Valley 2.12</td>
<td>0.7</td>
<td>1.91</td>
<td>0.4</td>
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<tr>
<td></td>
<td>Flank 2.09</td>
<td>0.2</td>
<td>1.88</td>
<td>1.1</td>
</tr>
<tr>
<td>Experimental implant</td>
<td>Top 2.08</td>
<td>0.4</td>
<td>1.34</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>Valley 1.16</td>
<td>0.1</td>
<td>0.68</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>Flank 2.24</td>
<td>0.8</td>
<td>1.27</td>
<td>0.1</td>
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</table>

*Sa indicates average height deviation in microns; and Sdr, the developed interfacial area ratio in microns.


