Study of the Influence of Acid Etching Treatments on the Superficial Characteristics of Ti

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Modifications in texture and surface chemistry are commonly used to increase the biologic response to implants. We evaluated the influence of five different acid etching treatments on the chemical and topographical superficial characteristics of cpTi grade IV discs (test groups). One group of samples were only polished (control group). The samples were analyzed by electron microscopy (SEM, EDS), atomic force microscopy, and grazing incidence XRD. The acid etching treatments which produced higher values for the amplitude roughness parameters showed a combination of strong acids (HF and HCl/H2SO4) at high concentrations, with a relatively high temperature (≥ 60 °C) for a considerable time (≥ 60 min). Titanium and oxygen were the only elements detected by EDS on the surface in all groups, whereas titanium hydride was detected when the samples were analyzed by GIXRD. Only the group subjected to thermal treatment showed presence of rutile phase on the surface.

Keywords: titanium, surface modification, acid-etching, surface characterization

1. Introduction

Alterations in endosseous dental implant design have been revisited extensively to decrease treatment time frames by reducing the healing period for osseointegration establishment. Thomas and Cook1 examined the variables that potentially influence bone apposition to an implant surface. Of twelve parameters studied, only surface characteristics were shown to have a significant effect on bone integration of an implant. Because the implant surface is the first component to interact with the host, several surface modifications have been extensively investigated in the search for improved bone healing that would allow immediate or early loading of dental implants2. Alterations in surface texture and chemistry are modifications used commonly to increase the biological response to implants. The beneficial effect of rough implant surfaces on peri-implant bone formation is considered to be based on the changes in microtopography and subsequent alterations of surface energy that result in increased interaction with the adjacent biological environment by adsorption of proteins and blood components which in turn can enhance cell attachment and implant integration3.

Recently, commercially pure titanium (cpTi) has become widely used as a biomaterial for dental implants, orthopaedic implants, cardiovascular appliances, and implant-supported dental crowns because of outstanding characteristics that include high specific strength, high resistance to corrosion, greater biocompatibility, low modulus of elasticity, and high capacity to be osseointegrated with bone4. Furthermore, the creation of roughened titanium surfaces has long been demonstrated to be an effective way to promote the interfacial biomechanical properties of bone-anchored implants by means of increasing the interlocking capacity of surface5. The literature shows that when micromechanical anchorage is achieved, the bone-repair process switches from slower bone corticalization around the implant surface to more rapid bone trabeculization and bone ingrowth into the rough surface6. Some biomechanical studies in animal models found that the surface texture of titanium implants has a significant influence on their anchorage in bone7,8.

Bone interlocking or micromechanical anchorage at the interface is not a feature common to all surfaces; to achieve it, a certain level of roughness seems to be required. Some authors9 showed that machined surfaces do not achieve micromechanical anchorage at the interface. When torqued, bone was not found attached to the machined surface, whereas anchorage has been documented for other surfaces with physical and chemical surface modifications9. The surface topography obtained by acid attack can be modulated according to prior treatment as, for example, by sandblasting, using acid mixtures, using different temperatures, and using different etching times. Acid etching is a subtractive method, wherein pits are created in the titanium surface10. Acid etching of titanium is of particular interest because it creates a microtextured surface (fine rough surface with micro pits of 1-3 µm and larger pits of approximately 6-10 µm) that appears to enhance early endosseous integration and stability of the implant7.

Some studies support the belief that the subtractive surface modification by acid etching has a positive effect on the strength of endosseous integration. Lazzara et al.11 compared the bone response to Osseotite (an implant system that has a machined and dual acid-etched surface - MDAS) and machined surfaces (MS) placed in the human posterior maxilla. After 6 months of healing, the bone contact at the
MDAS was significantly higher when compared to MS, and the osteoconductive effect (the amount of bone apposition) of the MDAS over the MS was particularly pronounced in the softer trabecular bone. Klokkevold et al.\(^2\) compared the anchorage of MDAS and MS in the rabbit tibia model, and observed a statistically significant higher mean removal torque for the MDAS after 1, 2, and 3 months. Treating titanium by HF/HNO\(_3\) or by HCl, HF, and H\(_2\)PO\(_4\), He et al.\(^1\) and Zareidoost et al.\(^3\), respectively, showed that acid-etched titanium surfaces had higher roughness, lower cytotoxicity level and better biocompatibility than the control samples not submitted to acid etching.

Thus, considering the importance of this subtractive method of surface modification, the aim of the present study was to evaluate the influence of five different acid etching treatments described in the literature on the chemical and topographical superficial characteristics of cpTi grade IV.

### 2. Material and Methods

Eighteen machined cpTi grade IV discs (12.7 × 2.0 mm) were used as the substrate material for the experiment. All discs were cut from a rod using a IsoMet\textsuperscript{®} Low Speed Saw (Buehler\textsuperscript{®}, Lake Bluff, USA) with a Diamond Wafering Blade No. 11-4244 (diameter 4"-102mm / thickness 0.012"-0.3mm) from the same manufacturer. The samples were embedded in polymethyl methacrylate, in order to be polished in a polishing machine until a #2000 silicon carbide paper.

The discs were separated into a control group with 3 discs and 5 test groups with 3 discs each. Each test group was acid-etched by a different acid treatment (defined as groups AT1 to AT5) previously described in the literature\(^1\)\(^4\)\(^8\)\(^9\), Table 1 presents a detailed description of these 5 acid-etching treatments. The firing in vacuum was performed in a VITA Vacumat 40T vacuum furnace (VITA Zahnfabrik H. Rauter GmbH & Co.KG, Bad Säckingen, Germany).

The surface morphology of treated samples was examined by scanning electron microscopy (SEM - JEOL, model JXA-8900RJ, Tokyo, Japan). The secondary electron (SE) detection mode with an acceleration voltage of 25 kV was selected for SEM analysis and the vacuum pressure was maintained below 1 × 10\(^{-5}\) Torr. The load current (LC) was approximately 85 µA. For a direct comparison of the surface morphology, the same magnification of 1000× was selected for all samples.

In order to obtain quantitative analysis of the surface roughness, atomic force microscopy (AFM - NTegra Aura, NT-MDT, Moscow, Russia) of the samples was performed. AFM images were acquired in air using semicontact mode with a sharpened gold-coated silicon tip (nominal spring constant of 2.5-10 N/m and nominal resonance frequency of 110-200 kHz, NSG01 probe series, NT-MDT, Moscow, Russia). The scanning area for the measurements was 50 × 50 µm\(^2\). The images obtained by AFM were characterized by 2\(^{nd}\) order extraction filter, using the software Image Analysis 2.1.2 (NT - MDT, Moscow, Russia). The seven amplitude surface roughness parameters calculated by the software were evaluated (\(S_d, S_s, S_h, S_z, S_{av}, S_{sk}\) and \(S_{rz}\)). The mean value and standard deviation of these parameters was obtained from 15 satisfactory scans of each group (5 from each of the 3 samples), from random sites on the surface. A statistical analysis of the mean values of the surface roughness parameters was compared with One-Way Analysis of Variance (ANOVA), since a normal distribution of the variables was observed by the Kolmogorov-Smirnov test. The Tukey’s HSD test was also performed, in order to find means that are significantly different from each other. The degree of statistical significance was considered \(P < 0.05\). The Statistical Package for the Social Sciences (SPSS) version 20 software (SPSS Inc., Chicago, USA) was used to perform the statistical analysis.

The surface chemical composition was analyzed by energy dispersive x-ray spectroscopy (EDS - JEOL, model JXA-8900RJ, Tokyo, Japan). The most central area of the samples was chosen, and the analysis was made with a magnification of 200x. The elemental chemical composition was calculated by the mean value and standard deviation from the 3 samples of each group.

Moreover, grazing incidence X-ray diffraction (GIXRD) measurements were carried out in a Ultima IV X-ray diffractometer (Rigaku, Tokyo, Japan), using Cu-K\(_{a1}\) radiation at 30 kV and tube current of 20 mA, without any filter or monochromator, in the angle range of 10°−90° (2\(\theta\)) with a grazing incidence of 3\(^{\circ}\), making the diffraction sensitive to the surface. The step of measurement was set to 0.05\(^{\circ}\) with a scan rate of 0.5\(^{\circ}\) per minute. The divergence slit was set to 1 mm, with a Div H.L. Slit of 2 mm. The results were analyzed in Search-Match software (Crystallographica, Oxford, United Kingdom). GIXRD experiments were carried out in order to distinguish chemical compounds at the sample surface.

### Table 1. Details of the acid-etching groups.

<table>
<thead>
<tr>
<th>Acid-etching Group</th>
<th>Acid solution</th>
<th>Temperature (°C)</th>
<th>Etching time (min)</th>
<th>Additional treatment</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>AT1</td>
<td>1% HF / 30% HNO(_3)</td>
<td>RT</td>
<td>60</td>
<td>-</td>
<td>Orsini et al.(^14)</td>
</tr>
<tr>
<td>AT2</td>
<td>12% HF</td>
<td>RT</td>
<td>2</td>
<td>-</td>
<td>Cho and Park(^15)</td>
</tr>
<tr>
<td></td>
<td>70% HCl / H(_2)SO(_4)</td>
<td>80</td>
<td>5</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>AT3</td>
<td>70% HCl / 60% H(_2)SO(_4)</td>
<td>60</td>
<td>60</td>
<td>-</td>
<td>Carvalho et al.(^18)</td>
</tr>
<tr>
<td>AT4</td>
<td>0.11 mol/L HF + 0.09 mol/L HNO(_3)</td>
<td>RT</td>
<td>10</td>
<td>dried in an oven at 50 °C for 24 h</td>
<td>Yang et al.(^17)</td>
</tr>
<tr>
<td></td>
<td>5.80 mol/L HCl + 8.96 mol/L H(_2)SO(_4)</td>
<td>80</td>
<td>30</td>
<td>dried in an oven at 50 °C for 24 h</td>
<td></td>
</tr>
<tr>
<td>AT5</td>
<td>48% H(_2)SO(_4)</td>
<td>60</td>
<td>60</td>
<td>firing in vacuum at 600 °C for 10 min</td>
<td>Iwaya et al.(^16)</td>
</tr>
</tbody>
</table>
3. Results

3.1. SEM analysis

Figures 1a-f revealed characteristic differences at the microscopic level according to the surface modification methods used for the samples as measured by SEM. Differences of the surfaces were obvious due to differences of the etching processes. The control group samples were mainly characterized by multidirectional grooves as result of the polishing process (Figure 1a). Samples submitted to acid treatment classified as AT1 showed a homogenous distribution of irregularities throughout the surface (Figure 1b). AT2 (Figure 1c) showed the presence of ridges and valleys with bigger dimensions as shown in AT1 samples, in many regions resembling grooves with a width varying from 1 to 2 μm. AT3 samples showed a surface with a great number of micro pits of 1-3 μm and some larger pits (Figure 1d). AT4 showed an almost homogeneous distribution of valleys and peaks with sharp edges (Figure 1e). Acid etching treatment AT5 did not eliminate the multidirectional grooves (Figure 1f), suggesting that the acid treatment was not effective to change surface morphology.

3.2. AFM analysis

The qualitative and quantitative surface topography demonstrated different degrees of roughness. Table 2 shows the mean values of tridimensional roughness parameters for the control group and acid-etched samples (groups AT1 to AT5) as determined by AFM. It can be observed that the surfaces of the samples from control group showed smaller mean values of the amplitude roughness parameters than the test groups. AT1 showed closest mean values of $S_y$, $S_z$, and Average when compared to the control group, and the same mean value of $S_y$ and $S_z$ as the control group. The samples from AT2, AT3, and AT4 groups showed higher mean values. The topographic maps obtained by AFM showed, qualitatively, the difference in roughness between the six surfaces: control group (Figure 2a) and test groups AT1 to AT5 (Figures 2b-f, respectively).

The amplitude parameters characterize the surface based on the vertical deviations of the roughness profile from the mean line. The roughness parameter $S_a$ represents the degree of symmetry of the surface heights about the mean plane. The sign of $S_a$ indicates the predominance of peaks (i.e. $S_a > 0$) or valley structures ($S_a < 0$) comprising the surface. Thus, it can be observed that all groups but AT4 have a predominance of peaks. $S_a$ indicates the presence of inordinately high peaks/deep valleys ($S_a > 3.00$) or lack thereof ($S_a < 3.00$) making up the texture. In this case, groups AT1 and AT5 were the only ones with a presence of inordinately high peaks/deep valleys.

Comparison of the mean values of the surface roughness parameters with ANOVA showed that the difference of the values between the groups revealed to be statistically significant for all parameters. Groups AT2 and AT3 showed statistically significant difference for the mean value of the roughness parameters when compared to the other groups, but not between themselves. The difference of the mean values of the control, AT1 and AT5 groups were also statistically significant for most of the parameters when compared to the other study groups, but not between themselves.

![Figure 1. SEM pictures of control group (a), AT1 (b), AT2 (c), AT3 (d), AT4 (e), and AT5 groups (f) (original magnification 1000x – scale bar 25 μm).](image-url)
Table 2. Mean values (± SD) of tridimensional roughness parameters as determined by AFM (scanning area of 50 × 50 µm²), and P-values for one-way ANOVA comparisons.

<table>
<thead>
<tr>
<th>Group</th>
<th>S_y (µm)</th>
<th>S_z (µm)</th>
<th>Average-mean height (µm)</th>
<th>S_q (µm)</th>
<th>S_k</th>
<th>S_{ka}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>1.30 ± 0.37&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.65 ± 0.19&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.54 ± 0.17&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.11 ± 0.02&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.14 ± 0.03&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.62 ± 0.62&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>AT1</td>
<td>1.54 ± 0.41&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.76 ± 0.20&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.69 ± 0.24&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.11 ± 0.02&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.14 ± 0.03&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.29 ± 0.82&lt;sup&gt;abc&lt;/sup&gt;</td>
</tr>
<tr>
<td>AT2</td>
<td>6.41 ± 0.61&lt;sup&gt;b&lt;/sup&gt;</td>
<td>3.21 ± 0.31&lt;sup&gt;b&lt;/sup&gt;</td>
<td>3.11 ± 0.39&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.78 ± 0.13&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.97 ± 0.16&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.15 ± 0.21&lt;sup&gt;bc&lt;/sup&gt;</td>
</tr>
<tr>
<td>AT3</td>
<td>6.83 ± 0.41&lt;sup&gt;b&lt;/sup&gt;</td>
<td>3.42 ± 0.23&lt;sup&gt;b&lt;/sup&gt;</td>
<td>3.25 ± 0.41&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.85 ± 0.15&lt;sup&gt;b&lt;/sup&gt;</td>
<td>1.06 ± 0.17&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.16 ± 0.21&lt;sup&gt;bc&lt;/sup&gt;</td>
</tr>
<tr>
<td>AT4</td>
<td>5.22 ± 0.95&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.55 ± 0.44&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.52 ± 0.55&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.57 ± 0.16&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.73 ± 0.19&lt;sup&gt;a&lt;/sup&gt;</td>
<td>-0.16 ± 0.30&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>AT5</td>
<td>2.97 ± 1.09&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.48 ± 0.56&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1.34 ± 0.51&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.16 ± 0.05&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.22 ± 0.08&lt;sup&gt;a&lt;/sup&gt;</td>
<td>0.45 ± 0.58&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

P-value  0.000   0.000   0.000   0.000   0.000   0.002   0.000

<sup>abc</sup> Groups denoted with the same superscripted letter do not indicate statistically significant difference in surface roughness (P ≥ 0.05) between the groups, according to the Tukey’s HSD test.

Figure 2. AFM topography maps for the control group (a), AT1 (b), AT2 (c), AT3 (d), AT4 (e), and AT5 groups (f) (scanning area: 50 × 50 µm²).
3.3. EDS analysis

EDS analysis of the surfaces showed titanium and oxygen to be the only elements in all groups, with a similar average % atomic concentration of 60% and 40% (± 5%), respectively. Figure 3 shows a typical EDS spectrum of the group AT2.

3.4. XRD analysis

Figure 4 presents a typical GIXRD diffractogram for a sample from the control group, where diffraction peaks were labeled according to Miller indices, as described elsewhere. The diffractogram analysis confirmed only the presence of titanium (Ti). The GIXRD results obtained for the samples from groups submitted to acid etching showed the presence of Ti and titanium hydride (TiH$_2$) in groups AT1 to AT4 (Figures 5a-d, respectively), and Ti, TiH$_2$, and rutile (TiO$_2$) in group AT5 (Figure 5e).

4. Discussion

Chemical texturing is a method of surface roughening through acid etching. This method has advantages compared with current methods of roughening such as grit blasting, plasma spraying, and beads. As the production process does not stress the adjacent material, there is no risk of flaking or delamination and the process does not leave particles of grit. All test groups have been acid attacked; nevertheless, due to the distinct treatment parameters, every implant displayed distinct surface roughness characteristics and a proper surface aspect that could not be mistaken. Seven distinct roughness parameters have been reported for each surface. However, their significance in terms of predicting an enhanced bone response or an increased implant fixation remains questionable. Some studies have demonstrated that increases in the numerical surface roughness of an implant enhance biomechanical anchorage in bone, as determined by torque removal tests and resistance to push-out forces. These studies showed a tendency toward an increase in bone contact, and resistance to removal, with an increasing numerical implant surface roughness. In the groups AT2, AT3, and AT4 it can be seen that the etching created micropits, when the SEM image is compared with the control group, which also reflected in the higher values of the amplitude roughness parameters in these samples. Groups AT2 and AT3 also showed statistically significant difference for the value of the roughness parameters when compared to the other groups. This was also true for the group AT4 for some parameters, but not all. Specific to dental implants, studies have shown that histologic and biomechanical characteristics were improved due to increase in the as-machined surface texture by varied methods resulting in average implant surface roughness ($S_a$) ranging from 0.5 to 2 $\mu$m. Thus, the values found in the present study for the roughness parameter $S_a$ seems to be appropriate in these groups. Although biological tests were not conducted in the present study, it can be suggested that these surfaces with the highest values of roughness parameters (AT2, AT3 and AT4) could also present a greater osseous anchorage, if implants had been treated with these acid etchings, and if could only consider the surface topographic characteristics, based on results found in the literature. Several studies reported a good correlation between increased $R_a$ (bi-dimensional correlate of the three-dimensional $S_a$) and stronger anchorage, although the $R_a$ is obviously insufficient by its own to characterize a given surface. Wong et al. evaluated pushed-out titanium cylinders placed in the knee trabecular bone of mini-pigs after 3 months, comparing four surfaces. The surface with the highest $R_a$ showed the highest anchorage; a strong linear correlation, $r^2 = 0.90$, was found between the $R_a$ and the torque test. The highest torque values were observed at each time point for the roughest surface, as determined by the $R_a$ and $R_v$. At all time points, these authors reported a high correlation of $r = 0.83$ between the $R_v$ and implant anchorage. Klokkevold et al. compared the torque resistance to removal of screw-shaped titanium implants having a MDAS with implants having either a MS, or a titanium plasma spray surface (TPS), in a rabbit model. Their results showed an enhanced bony anchorage to MDAS implants as compared to MS implants (also showing a much higher value of $R_a$ for the MDAS implants), and...
that dual acid etching of titanium enhances early endosseous integration to a level which is comparable to that achieved by the topographically more complex TPS surfaces. However, although the height-descriptive parameters of the surfaces of the groups AT2, AT3, and AT4 were significantly higher than the AT1 and AT5 surfaces, one cannot speculate on their biological performance until their bone interlocking capacity is demonstrated. It turns out that clinical implications cannot be drawn by relying on roughness-descriptive parameters alone\(^{10}\).

It has been reported that the etching process modifies the Ti surface composition of SLA-treated implants, and XRD and metallographic microscopy analysis indicated the presence of 20 to 40\% of titanium hydride (TiH\(_x\), \(x \leq 2\)) in addition to Ti\(^{26}\). Before attacking the metallic titanium, the acids must first dissolve the protective titanium oxide layer. During the course of the corrosion process of titanium, native hydrogen ions (H\(^+\)) are released. These small ions diffuse rapidly into the metal because the latter is left without its dense protective oxide layer; the sub-surface is therefore enriched with hydrogen\(^{27}\). When saturation in hydrogen is reached, titanium hydride is formed. Titanium hydride may be biologically important because a hydride layer is much better suited as a template for binding biomolecules.

Figure 5. Typical GIXRD diffractograms of samples from groups AT1 to AT5. Peak identification according to crystal structures: Ti (circle), TiH\(_2\) (diamond), rutile (X).
chemically onto a titanium surface\textsuperscript{29}. In the present study, as the surface of the samples were also modified by acid etching, it was not surprising that titanium hydride was found in all test groups (from AT1 to AT5) by GIXRD analysis.

EDS analysis of the surfaces showed titanium and oxygen to be the only elements in all groups, most probably due to the natural formation of a titanium oxide passivation layer (mainly TiO\textsubscript{2}) just after sample surface preparation. The result (about 60%/40%) agrees with the mass percentage of the TiO\textsubscript{2}, the main component of a typical titanium oxide passivation layer. It was previously observed, by using XPS analysis, that dental implants surfaces treated only by sandblasting and acid-etching consists of oxidized titanium\textsuperscript{29}. Moreover, TiO\textsubscript{2} can exist in three crystalline forms called anatase, rutile, and brookite, all with different physical properties. The quasi-amorphous oxides can be crystallized by heating to temperatures of 400-500 °C or higher, which leads to transformation to more crystallized phases\textsuperscript{29}. Thus, the presence of rutile phase in group AT5 can be explained by the thermal treatment by which the samples of this group were subjected (firing in vacuum at 600 °C for 10 min).

The results showed that the acid etching treatments used here produced a surface with different topographical and chemical features compared to the polished one (control), except for AT5. The present results demonstrated that the acid etching can remove the grooves produced by the polishing process. The etching can create a surface with randomly distributed pits in a micron-scale (as seen with AT3 – Figure 1d), or homogeneously create ridges and valleys throughout the surface (as seen with AT1, AT2, and AT4), enlarging the surface area. Although the ultimate aim of acid attack is to create pits to allow for bone ingrowth, etching varies from weak to strong. The AT1 produced very small pits. AT5 did not change the surface morphology when compared with the control group. The surface aspect is probably caused by the use of either a weak acid mixture, a low etching temperature, or a short etching time\textsuperscript{10}. In the case of AT1, the room temperature might have been the cause, whereas in the case of AT5, the cause was probably a weak acid solution. The formation of great number of micropits in the group AT3 probably resulted from a combination of strong acids (HCl/H\textsubscript{2}SO\textsubscript{4}) at high concentrations, a relatively high temperature (≥60 °C) for a considerable time (≥60 min). EDS analysis of the surfaces showed titanium and oxygen to be the only elements in all groups. Only the group subjected to thermal treatment (AT5) showed presence of rutile phase on the surface. All test group samples showed the presence of titanium hydride on the surface after the acid etching treatment. Since the present study has used discs as the substrate material, the observed results cannot be directly extrapolated to complex surfaces like those in cylindrical threaded titanium implants.

5. Conclusions

Treating titanium surfaces with acid does not create a standard topography. The latter varies according to several parameters, such as prior treatment, acid mixture composition, temperature, and time of acid treatment. The acid etching treatments here classified as AT2, AT3, and AT4 produced higher values for the amplitude roughness parameters in comparison with the other groups (control, AT1, and AT5). These favorable acid treatments showed a combination of strong acids (HF and HCl/H\textsubscript{2}SO\textsubscript{4}) at high concentrations, with a relatively high temperature (≥60 °C) for a considerable time (≥60 min). EDS analysis of the surfaces showed titanium and oxygen to be the only elements in all groups. Only the group subjected to thermal treatment (AT5) showed presence of rutile phase on the surface. All test group samples showed the presence of titanium hydride on the surface after the acid etching treatment. Since the present study has used discs as the substrate material, the observed results cannot be directly extrapolated to complex surfaces like those in cylindrical threaded titanium implants.

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