Surface Topographical Changes of a Failing Acid-Etched Long-Term in Function Retrieved Dental Implant

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The aim of the present study was to report the main topographical and chemical changes of a failing 18-year in function retrieved acid-etching implant in the micro- and nanoscales. A partially edentulous 45 year old rehabilitated with a dental implant at 18 years of age exhibited mobility. After careful examination, a 3.25 × 13-mm press-fit dental implant was retrieved. Scanning electron microscope (SEM) analysis was carried out to study topographical changes of the retrieved implant compared with an unused implant with similar topographical characteristics. Moreover, X-ray photoelectron spectroscopy (XPS) analysis was used to study the surface composition of the retrieved failing implant. Clear changes related to the dual dioxide layer are present as visible in ≥×500 magnification. In addition, it was found that, for the retrieved implant, the surface composition consisted mainly of Ti2p, O1s, C1s, and Al2p. Also, a meaningful decrease of N and C was noticed, whereas the peaks of Ti2p, Al2p, and O1s increased when analyzing deeper (up to ×2000s) in the sample. It was shown that the superficial surface of a retrieved press-fit dual acid-etched implant 18 years after placement is impaired. However, the causes and consequences for these changes cannot be determined.

Key Words: dental implant, topography, chemical composition, surface, failure, case report

INTRODUCTION

Implant primary and secondary stability represents the decisive factor for long-term success of implant therapy.[1] Undoubtedly, both bone quality and quantity play an important role on osseointegration; moreover, when facing a poor bone quality scenario, implant surface topography becomes even more important, representing a factor of paramount importance due to the need of higher primary stability.[2] Albrektsson et al[3] described the most important factors for the establishment of osseointegration: the material, design, and surface of the implant; bone condition; surgical technique; and loading protocol after implant placement. Hence, modification of the micro-topography of dental implants has been widely investigated, aiming at increasing the interlocking between the implant and bone.[4] These investigations have resulted in advances increasing surface roughness that will also have an influence on cell proliferation and differentiation, extracellular matrix synthesis, activation of molecular switches of transduction pathways through focal adhesion formation and cytoskeleton rearrangements,[5] and cell shape.[6–8] Hence, implant macro- and micro-designs are determinant factors in the loading protocol and also in long-term success. Additionally, the combination of surface topography and chemistry may be crucial in the corrosion and in bacterial colonization resistance, which might lead to implant failure.[9]

Despite surface topography and chemistry of different implant designs tested in vitro to find the best performance, there are still few clinical trials showing their behavior after functioning for long time.[10–14] These studies provide valuable and real information about the fate of implant prototypes, although in some cases there is not an exhaustive report of the patient's/implant's history. In fact, analysis to study the characteristics of retrieved implants helps in understanding the way that any surface modification can bear. In this direction, a careful inspection of the surface topographic features allows for having good information about the mechanical performance of the outmost surface layers. Also, analysis of changes in chemical composition in the external layers permits confirmation of any

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reactive process on the surface and the possibility of ion diffusion from media to implant or vice versa (any ion liberation from implant to media after long periods of time). The aim of the present study was to report the main topographic and chemical changes of a failing 18-year retrieved implant in the micro- and nano-scales using scanning electron microscopy (SEM). Additionally, we aimed to analyze the chemical characteristics of the implant surface layers using X-ray photoelectron spectroscopy (XPS) analysis to assess composition changes that might have been triggered in the material by the use/function.

Materials and Methods

Case presentation

A paritally edentulous 45 year old rehabilitated subject with a dental implant exhibited mobility 18 years after placement. After careful examination, a 3.25 × 13-mm Ti6Al4V press-fit Biomet 3i Osseotite dental implant (Implant Innovations, Inc, Palm Beach, Fla) was retrieved due to excessive marginal bone loss that triggered mobilization.

Implant cleaning

Prior to the ecapture of SEM images, the implant was carefully cleaned using a solution of 2% antiseptic cleaner (pH, 6; DERQUIM DSF 11, PanreacQuímica S.A., Barcelona, Spain). The implant was gently rubbed with a smooth cotton cloth to remove any biological rests from the surface. It was rinsed repeatedly with distilled water and sonicated in distilled and deionized water (Milli-Q system, EMD Millipore, Darmstadt, Germany). Finally, it was placed in distilled water again for a few seconds and allowed to air dry at room temperature.

Scanning Electron Microscopy

The SEM images of the implant were taken with a scanning electron microscope (Quanta 3D FEG, FEI, Hillsboro, Oreg) operated at different voltages (indicated at the bottom of each of the images) with secondary electrons. Images with magnifications that ranged from 150 to 50 000 augmentations were taken randomly in different sections of the implants.

X-ray photoelectron spectroscopy

The XPS measurements were performed in a K-Alpha (Thermo Scientific, Waltham, Mass) with a monochromatic AlK_{α} (1486.68 eV) X-ray source, a spot size of 300 μm, and 50.4 W (12 kV × 4.2 mA) power. Survey XPS spectra were acquired using a pass energy of 200 eV. All measures were conducted inside an ultrahigh vacuum chamber with a pressure of 10^{-8} mbar. The XPS spectra were acquired before and after sputtering with an Ar^{+} ion etching of 3 keV. The binding energies were measured in reference to the C1s peak at 285 eV. The XPS spectra were background subtracted using the Shirley method.

Results

SEM analysis

When analyzing the SEM images, no topographic changes could be identified up to ×350. At this point, we can appreciate the crystallographically oriented boundaries caused by the acid etching chemical process. However, from ×500 is clear evidence of changes in the surface topography when comparing the retrieved implant and a similar unused dental implant. First, we noticed the presence of scratching in the retrieved implant, which did not manifest in the unused implant. These findings were more clearly observed at ×1000 (Figure 1). It is noticeable that in the unused implant we found a superficial layer that was not present in the retrieved implant. In fact, this is highlighted by the presence of "orbed-shape" structures that, in the unused implant, we believe must be beneath the superficial thin layer (Figure 2). At this point, the scratches could be observed in the retrieved implant, whereas in the unused implant the superficial layer was pristine. As assumed, we found that this outer oxide layer is covering the orbed-shape structure in the unused implant, which is evidence of the wear caused while placing. In addition, "crater-like" structures in the unused implant were seen at ×2500. On the contrary, these were not found in the retrieved implant, most likely because the oxide layer that was partially missing in the retrieved implant would have produced these structures. Again, if we keep increasing the magnification up to ×3500 (Figure 3), we can appreciate the oxide layer around the orbed-shape structure in the unused implant, whereas for the retrieved implant, the structure is well defined and displays the partial absence of the oxide layer.

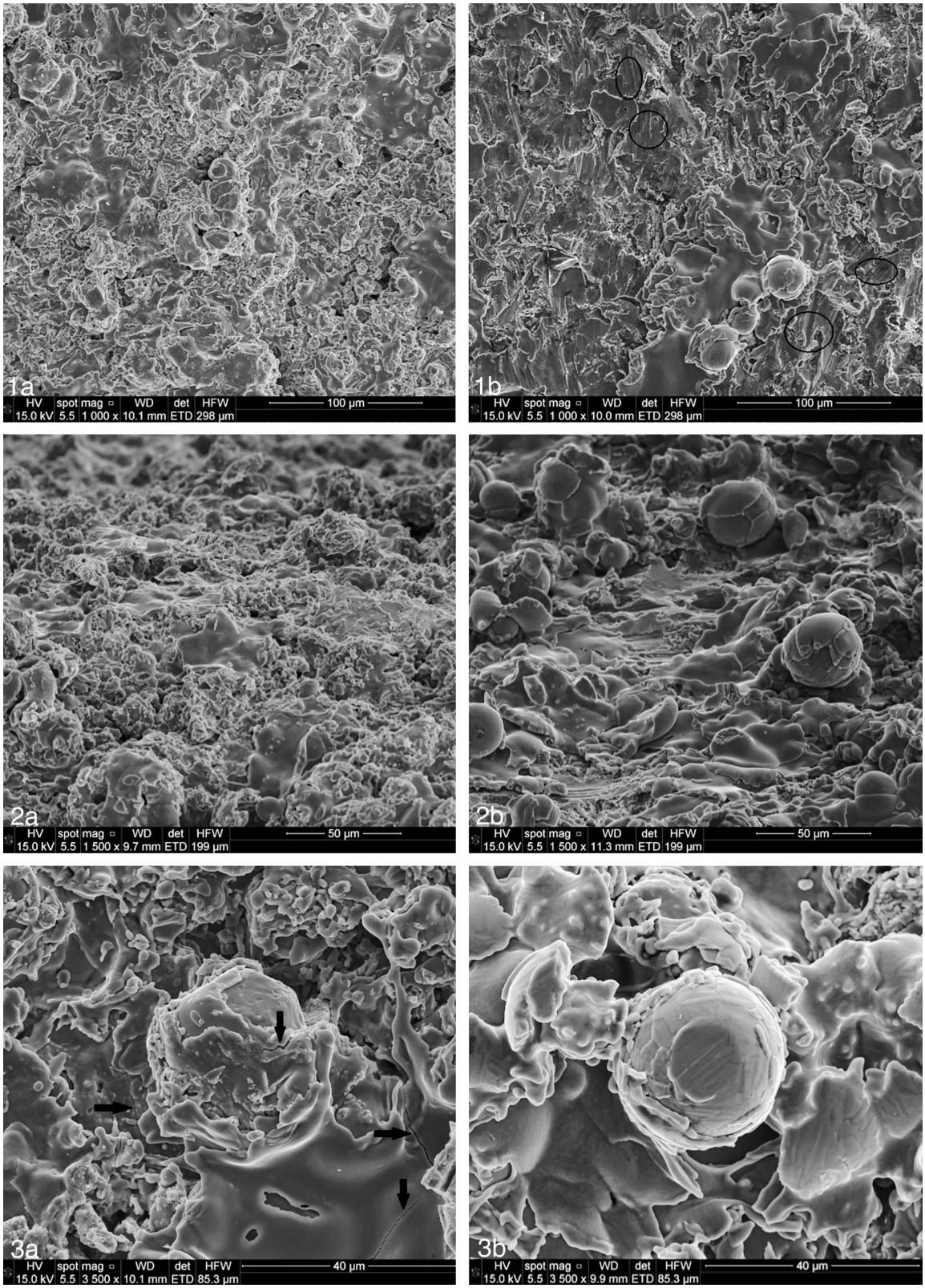
XPS analysis of the retrieved implant

The XPS analyses were carried out at the superficial layer and at five depth levels (20, 40, 60, 1000 and 2000). The spectrogram for the retrieved implant is illustrated in Figure 4 and is described in the Table. It was found out that at 20s the major peaks were concentrated at Ti2p (13.64%), O1s (39.9%), and C1s (26.43%). Furthermore, it was evident that Al increased significantly (from 5.20% at the superficial layer to 14.37% at the 20s). When analyzed in depth, it was noted that the S and Zn peaks disappeared at 20s and F peaks disappeared at 40s. Moreover, a meaningful decrease of the N and C peaks was seen (4.17% at 20s to 3.54% at 60s and 26.43% at 20s to 21.13% at 60s, respectively), whereas the Ti2p, O1s, and Al2p peaks increased as the depth increased (13.64% at 20s to 17.06% at 60s, 39.90% at 20s to 40.15% at 60s and 14.37% at 20s to 17.68% at 60s, respectively). These results are shown in the Table. Additionally, the increasing/decreasing patterns were maintained in the deeper depths (up to 2000s) as demonstrated in the Table.

Discussion

Implant surface chemical modifications play one of the major roles in osseointegration inasmuch as it is a determinant factor in the direct bone-to-implant contact created between the bone and the titanium oxide layer.[1] Surface roughness seems to influence the osteoblast differentiation and the creation of a matrix around the implant, which will anchor the implant to the bone substratum.[15]

The Osseotite dental implant presents a rough surface achieved through dual thermo-etching. It is immersed in 15% hydrofluoric acid, etched in a mixture of H_2SO_4/HCl acids, and



Figures 1–3. Figure 1. Magnification of (a) unused and (b) retrieved implant at ×1000. Note: marked areas display the scratches of the retrieved implant. **Figure 2.** Perspective view magnification of (a) unused and (b) retrieved implant at ×1000. **Figure 3.** Magnification of (a) unused and (b) retrieved implant at ×3500. Note: arrows indicate the presence of superficial layer that is not displayed in the retrieved implant.

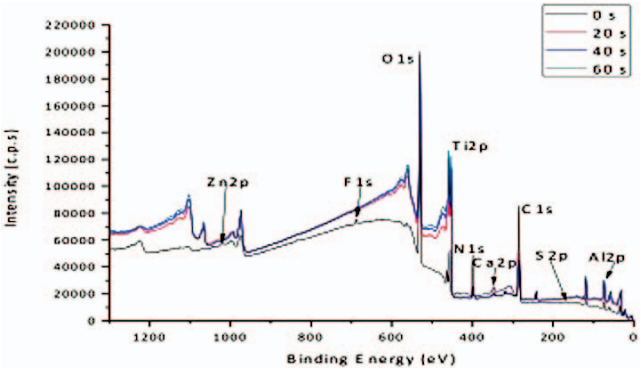


Figure 4. Spectrogram by XPS of the 18-year-old retrieved dental implant.

then heated at 60–80°C for 3–10 minutes.[16] The chemical composition of this surface is mainly based on TiO_2, and <34% C, <18% Na, and <4.8% N.[14] Regarding the crystal structure, it has been reported to be amorphous, with a porous film of 0.003–0.014 μm.[14] All these processes create a "wrinkled-like" landscape visible at low magnification. It is speculated that the benefit of this surface treatment is the increased contact osteogenesis, especially with poor bone quality, by increasing platelet activation and red blood cell agglomeration.[17,18] The present study showed the topographic changes of a failing press-fit acid etching retrieved implant after a long-term period in function. As displayed by SEM, it was found that changes in surface topography characteristics occurred. One possible hypothesis is the fact that since the implant was press fitted, scratches were produced as well as the partial removal of the superficial layer. This explanation is supported by the idea that, for the placement of press-fit implants, it was advised to drill a lower-diameter osteotomy to achieve primary stability of the implant, inducing a relevant strain in the peri-implant bone.[19] On the other hand, this fact might be explainable by the effects triggered from the combination of inflammatory cells released in the presence of pathogenic bacteria invasion after peri-implant marginal bone loss and by the pathogens' products (such as enzymes). Therefore, although in this report, the cause cannot be clarified, it is clear that, for some reason, failing (not failed) implants may have topographic changes that somehow jeopardize implant stability at some time point. Further in vitro studies should be conducted to demonstrate the real reason and its consequence.

Moreover, our findings showed that the deeper the Osseotite surface is analyzed by XPS, the more concentrations of Ti2p O1s and Al2p are present. In addition, it was shown that the element composition at the most superficial level after sputtering (20s) consisted of Ti, O, Al, and C. Therefore, this study is in agreement with the results shown by Kang et al,[12] Morra et al,[13] and Sul et al,[14] who found the highest peaks of Ti, O, and C after sputtering the implant surface. Nevertheless, some contradictions in terms of the amounts of each element were remarkable. Our study is in concordance with Morra et al,[13] who revealed a reduced amount of Ti compared with O or C. Furthermore, the differences in binding energy between these elements were consistent with previous studies.[12,20–22] Also, a relatively high amount of N (4.17%) at 20s was noticed. The binding energy of trace N might be associated with organic amines.[12] Additionally, as expected, this element significantly decreases as the analysis proceeds deeper. Larger-size human retrospective and animal in vivo trials should be conducted to further verify these findings and to study the effects of implant placement on topographic and chemical surface changes.

Conclusion

Within the limitations, the present study showed that the superficial surface of a retrieved press-fit dual acid-etched implant 18 years after placement is impaired. However, the causes and consequences for these changes cannot be determined. Furthermore, as expected, we found an increase of O, Ti, and Al when analyzing further depths. Nonetheless, further in vitro and in vivo studies are needed to determine the influence of surface roughness on chemical and topographic changes.

Abbreviation

XPS: X-ray photoelectron spectroscopy

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Table

Relative atomic concentrations (At. %) from the XPS measurements of the retrieved implant at the surface and at different depths after 5 consecutive etchings of 20s, 40s, 60s, 1000s, and 2000s

Ion	At. % Superficial	At. % 20s	At. % 40s	At. % 60s	At. % 1000s	At. % 2000s
O1s	27.48	39.90	40.11	40.15	36.86	37.76
C1s	52.72	26.43	23.70	21.13	—	—
Ti2p	2.89	13.64	15.58	17.06	29.47	30.26
N1s	9.37	4.17	3.75	3.54	1.30	0.97
Al2p	5.20	14.37	16.41	17.68	22.94	24.19
F1s	0.72	0.92	0.00	0.00	—	—
Ca2p	0.85	0.56	0.45	0.34	—	—
Zn2p	0.13	0.00	0.00	0.00	—	—
S2p	0.65	0.00	0.00	0.00	—	—

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