Analysis of Failed Commercially Pure Titanium Dental Implants: A Scanning Electron Microscopy and Energy-Dispersive Spectrometer X-Ray Study

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Background: The failure of osseointegration in oral rehabilitation has gained importance in current literature and in clinical practice. The integration of titanium dental implants in alveolar bone has been partly ascribed to the biocompatibility of the implant surface oxide layer. The aim of this investigation was to analyze the surface topography and composition of failed titanium dental implants in order to determine possible causes of failure.

Methods: Twenty-one commercially pure titanium (cpTi) implants were retrieved from 16 patients (mean age of 50.33 ± 11.81 years). Fourteen implants were retrieved before loading (early failures), six after loading (late failures), and one because of mandibular canal damage. The failure criterion was lack of osseointegration characterized as dental implant mobility. Two unused implants were used as a control group. All implant surfaces were examined by scanning electron microscopy (SEM) and energy-dispersive spectrometer x-ray (EDS) to element analysis. Evaluations were performed on several locations of the same implant.

Results: SEM showed that the surface of all retrieved implants consisted of different degrees of organic residues, appearing mainly as dark stains. The surface topography presented as grooves and ridges along the machined surface similar to control group. Overall, foreign elements such as carbon, oxygen, sodium, calcium, silicon, and aluminum were detected in failed implants. The implants from control group presented no macroscopic contamination and clear signs of titanium.

Conclusion: These preliminary results do not suggest any material-related cause for implant failures, although different element composition was assessed between failed implants and control implants. *J Periodontol 2005;76:1092-1099.*

KEY WORDS

Comparison studies; dental implants, failure; microscopy, electron, scanning; osseointegration; peri-implant diseases; titanium; x-rays, spectrometry, energy-dispersive.

Dental implants have gained importance in daily clinical practice, even though failures have been reported.[1,2] These failures can be chronologically classified as early or late. Early failures have been attributed to surgical trauma, inadequate bone quality and quantity, lack of primary stability, and bacterial contamination of the recipient site.[3] Late dental implant failures are commonly associated with the occurrence of peri-implantitis and/or biomechanical overload. Peri-implantitis is described as a destructive inflammatory process affecting the soft and hard tissues around osseointegrated implants, leading to the formation of a peri-implant pocket and loss of supporting bone.[4,5]

The integration of titanium dental implants in alveolar bone has been partly ascribed to the biocompatibility of the surface oxide layer.[6] Several authors described the titanium oxide (TiO_2) layer as a thin layer of 2 to 6 nm of TiO_2 covered by a carbon-dominated contamination layer and trace of nitrogen (N), calcium (Ca), phosphorous (P), chloride (Cl), sulfur (S), sodium (Na), and silicon (Si).[7-10]

Recent evaluations have questioned the importance of implant surface cleanliness after peri-implantitis treatment.[11-14] It has been hypothesized that surface contaminants may be released from the contaminated implant surface, enhancing and perpetuating the inflammatory response, thus altering the healing process and

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Table 1.

Patient and Retrieved Implant Data

Patient/Implant	Age (years), Gender	Implant Position and Primary Stability*	Implant Length and Diameter (mm)	Months in Place	Loading Time (months)	Procedure
1A[†]	56 F	Maxilla anterior, good	13.0 × 3.75	60	53	Smoker, overdenture
1B		Maxilla anterior, optimal	10.0 × 3.75	60	53	Healed dental socket
2	46 M	Maxilla posterior, good	10.0 × 3.75	36	27	Sinus lift, cement
3	69 F	Mandible posterior, good	13.0 × 3.75	24	‡	No loading
4	62 M	Maxilla posterior, good	7.0 × 3.75	60	54	Cement
5	69 M	Maxilla posterior, optimal	10.0 × 3.75	72	65	Sinus lift, screw
6	41 M	Mandible posterior, optimal	13.0 × 3.75	5	‡	Healed dental socket
7	51 M	Maxilla posterior, good	10.0 × 3.75	24	17	Smoker, cement
8	37 F	Maxilla posterior, bad	10.0 × 3.75	6	‡	Sinus lift
9	45 M	Mandible posterior, optimal	10.0 × 4.0	5	‡	Onlay bone graft
10	33 M	Maxilla posterior , bad	10.0 × 3.75	4	‡	Osteotome
11	39 F	Maxilla anterior, good	15.0 × 4.0	6	‡	Onlay bone graft
12	40 F	Maxilla anterior, optimal	8.0 × 5.0	6	‡	Onlay bone graft
13A	58 F	Maxilla anterior, good	15.0 × 3.75	6	‡	Onlay bone graft
13B		Maxilla posterior, good	10.0 × 3.75	6	‡	Sinus lift
13C		Maxilla posterior, bad	13.0 × 3.75	6	‡	Sinus lift
14A	49 M	Mandible posterior, good	13.0 × 3.75	6	‡	Onlay bone graft
14B		Mandible posterior, good	10.0 × 3.75	6	‡	Onlay bone graft
15A	62 M	Mandible posterior, good	11.5 × 4.0	6	‡	Onlay bone graft
15B		Mandible posterior, bad	8.0 × 5.0	6	‡	Onlay bone graft
16	67 F	Maxilla posterior, good	13.0 × 4.0	2	‡	Sinus lift

* At time of insertion.
† Letters indicate that the samples were removed from the same patient.
‡ No loading.

possibly provoking the dissolution of titanium.[7,15-17] In addition, alterations of this oxide layer surface may compromise reosseointegration.[14,18]

Therefore, the aim of this study was to evaluate, using scanning electron microscopy (SEM) and energy dispersive x-ray spectrometer (EDS), the surface properties of dental implants retrieved from humans in order to determine the possible role of surface contamination in the failure of osseointegration.

MATERIALS AND METHODS

Patients and Implants

Twenty-one dental implants were retrieved from 16 patients (seven female; mean age 50.33 ± 11.81 years) who did not show any medical or dental contraindication for implant placement. All dental implants (except implant #3) were inserted at the Department of Periodontology, Dental School at Araraquara, Brazil, to support fixed prostheses in partially and totally edentulous patients. The dental implants evaluated in this study were from four different manufacturers[¶#**††] and included a variety of lengths and diameters (Table 1).

Nine dental implants were placed in posterior maxillae (bone type 4, poor density),[19] five in resorbed maxillae, and seven in the posterior mandible. Bone grafting procedures were performed when available bone height

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and volume were insufficient. Fifteen dental implants were retrieved from grafted areas (seven maxillary sinus lift and eight onlay grafts). The mean loading time of the failed implants because of overload/peri-implantitis was 44.83 ± 18.53 months. Overall, the mean implant time was 18.43 ± 22.48 months. No complications or infection were noted for any patient immediately after insertion (except for implant 3 which presented paresthesia).

Dental Implant Retrieval and Processing

The criterion for dental implant removal was lack and/or loss of osseointegration, except for implant 3 (unloaded) that was retrieved by a reverse torque because of mandibular canal damage. Some aspects in the apical portion of this specimen were osseointegrated. The lack of osseointegration was recorded as the slightest mobility tested by rotating and moving the implant back and forward. All the retrieved dental implants were mobile and were surrounded by a radiolucent line on radiographs using a standardized method with the parallel, long cone technique (Fig. 1). The failed dental implants were retrieved after an insertion time ranging from 2 months up to 5 years.

The implants were retrieved under local anesthesia by gently unscrewing them with stainless steel forceps, which were carefully positioned on the healing abutment or on the abutment/prosthetic restoration in order to avoid any possible contamination of the implant surface. The implants were rinsed with saline solution and immersed in 4% formaline[20] and stored in sterile plastic or glass vials (Table 2).

The implants were inspected for macroscopic soft tissue remnants, which were removed using titanium tweezers.

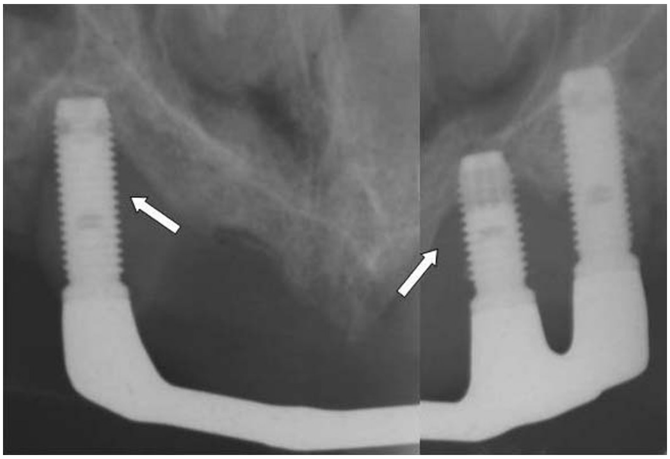

Figure 1.
Radiographic view of the failed implants in patient 1. Note the peri-implant bone lesions (arrows).

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

After dehydration in a graded series of alcohol, the implants were dried and mounted on metallic stubs using double side tape. In addition, two unused dental implants from different manufacturers[¶#] were used as controls (Table 3). These control implants were mounted with no pretreatment. All samples were placed in the vacuum chamber of an SEM.[‡‡] Thereafter, the implant surfaces observed by SEM underwent an element analysis. The regions of interest[7] (Fig. 2) and element detection were done simultaneously by electron beam-induced x-ray radiation at two different regions. An EDS x-ray[§§] was coupled to the SEM.

RESULTS

More than 83% of the evaluated dental implants were retrieved from grafted areas. Different amounts of organic residues were detected on most of the retrieved samples (Fig. 3). These residues appeared mainly as dark areas or proteinaceous material (Fig. 4). Some implants presented visible residual bone in the apical portion of the implant (Fig. 5) or residual proteinaceous material. The dental implant surface was dominated by grooves and ridges along the machining direction and appeared essentially unchanged on the retrieved samples, compared to control (Fig. 6).

The EDS analysis showed that all failed dental implant surfaces consisted of TiO_2, with varying amounts of contaminants. The control dental implants showed signs of Ti (Table 3), while in the failed implants only weak Ti signs were detected at the outermost surface. In most cases, carbon was the dominant element detected; O, N, Na, Ca, Al, and P were also detected. Si was detected in the organic overlayers of some samples (Table 2). The exception was specimen 10, which displayed unusual bismuth (Bi).

DISCUSSION

The present study evaluated the surface composition and presence of contaminants in retrieved dental implant surfaces. Although the dental implants evaluated in this study were from different manufacturers, the surface composition appeared to be the same. However, from the material surface science point of view, the implants probably differ significantly, because the companies apply proprietary preparation procedures and/or sterilization. Among the possible differences are origin and purity of titanium, oxide film formation and oxide film crystallographic structure, surface roughness, and oxide film thickness.[10]

EDS surface analysis showed the incorporation of some contaminants into both retrieved and control

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[‡‡] JSM-T330A, JEOL Ltd, Tokyo, Japan.
[§§] Noran Instruments, Inc., Middleton, WI.

implant surfaces. There are many events that may possibly trigger dental implant incompatibility. Inorganic contaminants such as C, Ca, Na, and P in the retrieved implants were probably relevant to the absorption mechanism of solvated ions that naturally occur in body fluids. In addition, the control of the surface characteristics of cpTi dental implants when they are manufactured is regarded as an important factor to achieving an optimal tissue response during healing of bone and soft tissues.[21] The degree of titanium surface contamination can determine its mechanical stability and osseoinduction/osseointegration qualities.[22] In addition, more than 83% of the evaluated implants were retrieved from grafted areas, suggesting that the grafted jawbone site can compromise successful osseointegation more than the oxide layer composition.

Kasemo and Lausmaa[23] noted that there is usually a large C signal, a smaller N signal, and traces of Cl, S, and Ca present in dental implants. They attribute the C, N, Si, and Cl containing molecules to adsorption during preparation procedures. They also reported that the Ca usually persisted throughout the oxide layer and may have been the result of surface segregation of minute Ca quantities in the commercially pure Ti stock. The Standard Specification for Unalloyed Titanium for Surgical Implant Applications (ASTM-F-67)[24] specifies that nitrogen content should be less than 0.05 wt% in unalloyed material. However, only the Ca and Na were found in control implants used in this study. The Bi detection in both samples from patient 10 may be the result of patient use of a gastric protector reported in anamneses; however, it is difficult to determine its origin.

In the oxide layer part of

Table 2.

Surface Composition and Contaminants of Retrieved Implants at Different Areas

Implant	Storage Material	Area Evaluated	Elements Detected	SEM Observations
1A	Plastic	Cylinder Flank	C, Na, O, N, P, Ti C, Na, O, N, P, Ti	Organic film
1B	Plastic	Bottom of thread	C, N, O, P, Ti C, Na, O, Al, Ti	Dark area No contamination
2	Glass	Neck Neck	C, Na, O, Al, Ti, Si C, Ca, O, Ti	
3	Plastic	Tip of thread Bottom of thread	C, Ca, P, O, Ti C, Ca, Al, Ti	Bone debris
4	Plastic	Cone Neck	C, Ca, Al, Ti C, Ca, Al, P, Si	Soft tissue
5	Glass	Flank Tip of thread	C, Na, Ca, Al, P, Si C, Ca, O, P, Ti	Soft tissue
6	Plastic	Cylinder Cylinder	C, Ca, O, P, Ti C, Na, Al, O, N, P, Ti, Si	Dark area Soft tissue and dark area
7	Glass	Bottom of thread Cylinder	C, Na, Al, O, N, Ti, Si C, Na, O, P, Ti	No contamination
8	Plastic	Cone Tip of thread	C, O, P, Ti C, Na, Ca, P, Ti	 No contamination
9	Plastic	Cone Neck	C, Na, Ca, O, P, Ti C, Na, Ca, P, Ti, Bi	 Organic film
10	Plastic	Flank Neck	C, Na, Ca, O, Ti, Bi C, Na, Ca, P, Ti	 Organic film
11	Plastic	Cone Neck	C, Na, Ca, P, Ti C, O, Na, Al, Ti	
12	Plastic	Flank Neck	C, Na, O, Al, Ti C, Na, P, Al, Ti	Soft tissue
13A	Plastic	Bottom of thread Flank	C, O, Na, Al, Ti C, Na, Ca, P, Ti	Soft tissue
13B	Plastic	Cone Cylinder	C, Ca, P, Ti C, Na, Al, Ti	Soft tissue
13C	Plastic	Cone Tip of thread	C, Na, Al, Ti C, Na, P, Ti	Soft tissue
14A	Plastic	Flank Bottom of thread	C, Na, Al, Ti C, Na, P, Ti	No contamination
14B	Plastic	Tip of thread Cylinder	C, Na, Ti C, O, Al, Ti	
15A	Plastic	Bottom of thread Flank	C, O, Al, Ti C, O, Al, Ti	Soft tissue
15B	Plastic	Tip of thread Flank	C, O, Al, Ti C, Ca, P, Ti	Soft tissue and dark area
16	Plastic	Cone	C, Ca, P, Ti	Soft tissue and some debris

Table 3.

Surface Composition of Control Implants

Implant	Area Evaluated	Elements Detected	SEM Observations
A	Cylinder Flank Bottom of thread	O, Ti, Ca Ti, O Ti, Na, O	No contamination
B	Flank	Ti, O	No contamination

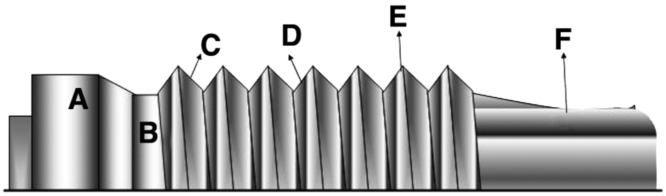

Figure 2.
Schematic drawing showing the different analysis locations on a dental implant. A: cylinder; B: neck; C: bottom of thread; D: tip of thread; E: flank; F: cone (adapted from Esposito et al.[7]).

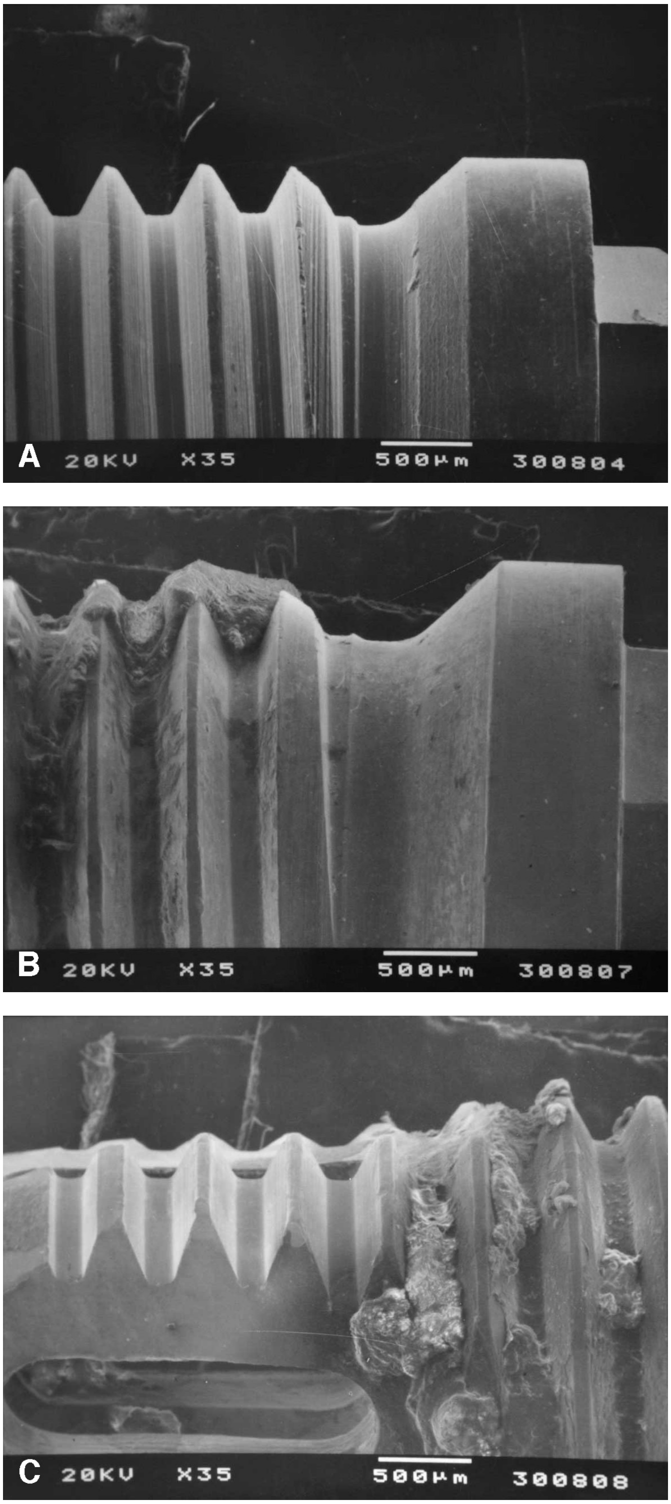

Figure 3.
A) *SEM of control dental implant.* ***B)*** *SEM of retrieved sample showing residues as proteinaceous material.* ***C)*** *SEM of retrieved dental implant from patient 1 showing organic debris on cone area.*

the implant, the intensity of the oxygen signal should not be considered as representative of the true composition of the oxide layer. Indeed, Ti is highly reactive metal, even in an ultra-high vacuum environment, because of the technical procedure used in SEM. The detection of this unrelated element is not related to impurities in cpTi. Olefjord and Hansson[17] suggest that inorganic contaminants should be avoided because they might result in dissolution of the titanium. The presence of Si and P as well as Ca and Na probably come from the finishing process in the Ti implant preparation and body fluids, respectively. In contrast, Si is a major constituent of glass, and the observed Si contamination was mainly attributed to ion dissolution from the glass storage vials, which agrees with Esposito et al.[7] In addition, but probably less so, sources of the contamination by Si and C could also be the residues left by rubber gloves.[25] Other contamination may have originated from several sources, including the fabrication process, cleaning and sterilization procedures such as Na detection, the environment during handling and storage (glass vials), insertion, or retrieval and analysis preparation procedures.[7,8,10,16,25]

The influence of contaminants on the dissolution rate of Ti in body fluids has not been evaluated. It is suggested that in principle, organic compounds lower the dissolution rate of titanium because they block the sites for the oxygen cathodic reaction. Foreign ions on the titanium oxide surface may catalyze the oxygen reaction and thereby promote the dissolution of titanium. For example, iron, chromium, and molybdenum ions are added during implant surface preparation to reducing

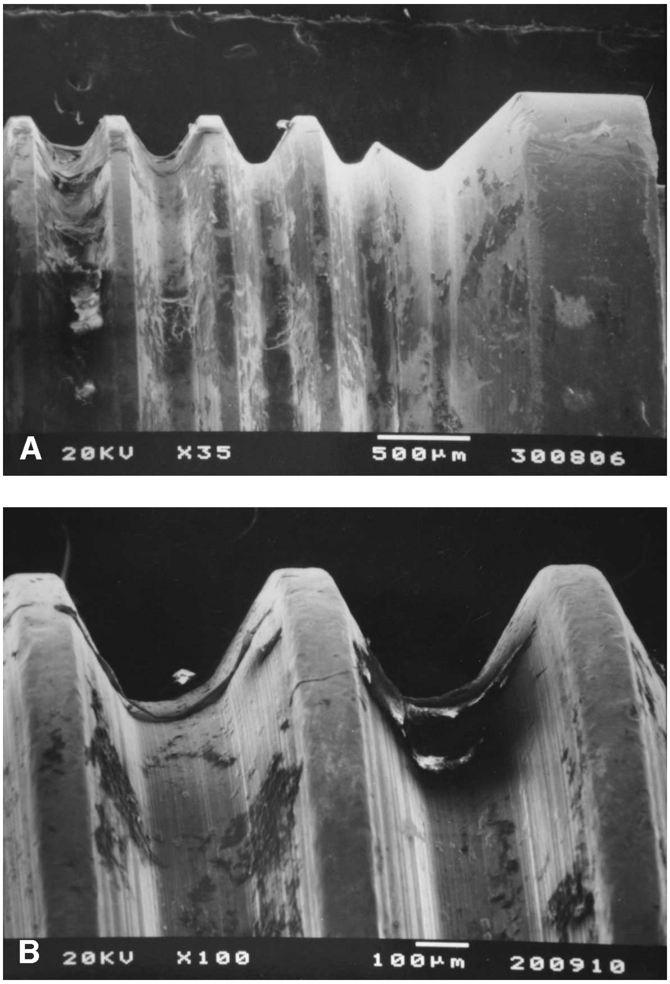

Figure 4.
A) *SEM of retrieved dental implant residues as dark areas.* ***B)*** *SEM of retrieved dental implant showing residues as dark area.*

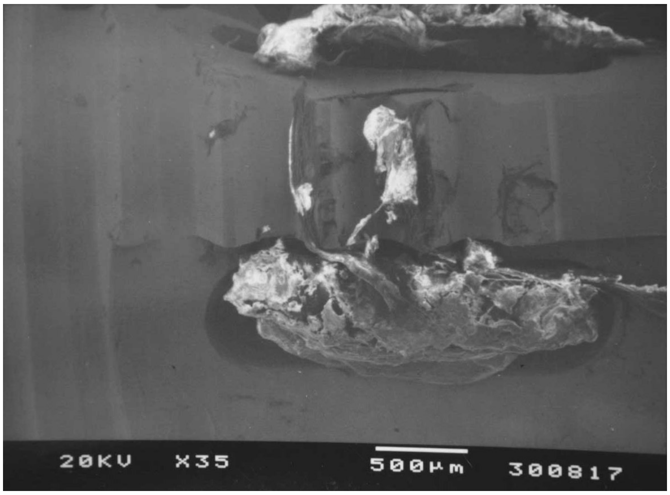

Figure 5.
SEM of retrieved implant showing residual bone in apical area.

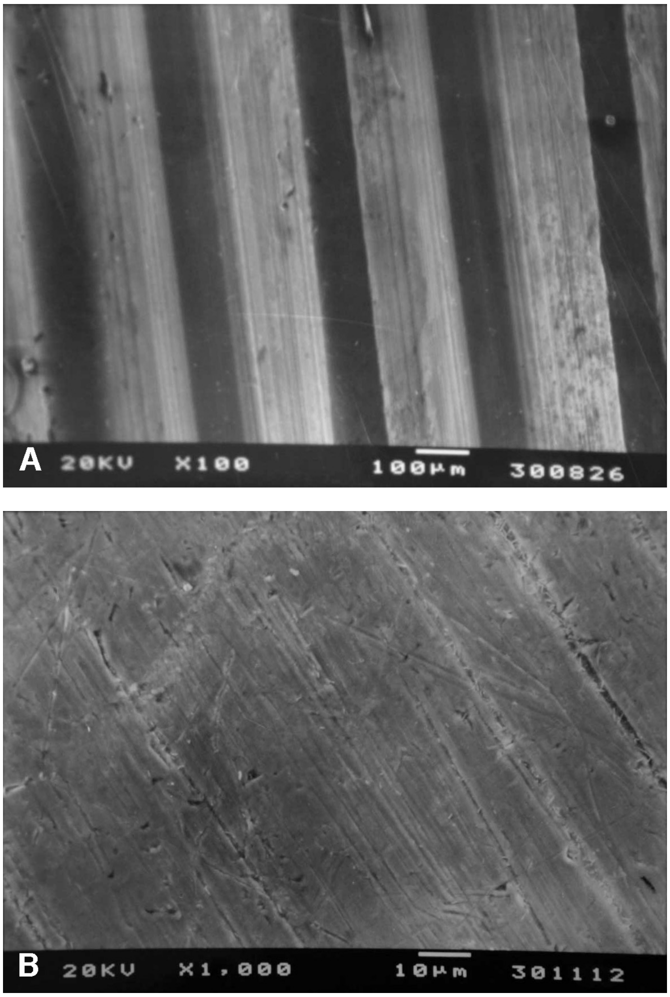

Figure 6.
A) *SEM showing grooves and ridge along the machined direction on retrieved implant.* ***B)*** *SEM showing grooves and ridge along the machined direction on a control implant.*

acids, such as sulphuric acid, to intensify the electrochemical reactions so that passivation can occur. It has been reported[26] that the corrosion products formed on the surface of Ti during its exposure to $NaNO_3$ and NaCl containing aqueous solution catalyze the reduction process of NO^{3-} and thereby raise the dissolution rate of Ti. Zinc in both solid and liquid form may cause embittlement of Ti.[24] It is not known whether the Ca^{2+} and Si^{4+} found on the surface in this study have the effect of promoting dissolution of Ti and thus "poison" the tissue or cause stress-corrosion cracking of the implant surface. In addition, the products released from microorganisms such as periodontal pathogens may increase this reaction.

The contact time of the dental implant with air prior to EDS analysis could be critical for the titanium levels which seemed to be the result of absorption of carbon.[27] The bioactivity of a material is associated with the surface energy of this material. This characteristic is an important criterion for determining acceptability and is influenced by purity of the material. Contamination of the material surface with hydrocarbons and other molecules and elements can change the surface energy and thereby also the potential bioacceptability of the dental implant surface.[28]

Some studies[7,10,29,30] evaluated the failed implant surface using x-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES). This equipment represents a much higher surface sensitivity than EDS used in our study, which is usually available on scanning electron microscopes. AES is able to determine the chemical composition of the uppermost 5 to 30 Å of the implant surface, corresponding to 2 to 10 atomic layers. In EDS equipment, the element composition evaluation was derived from analysis of the characteristic x-ray emission caused by excitation of atoms in the dental implants by the impinging electrons. The EDS equipment has a relatively high energy and penetrates relatively deep into the sample. The compositional information is, therefore, averaged over a depth of 1 µm. In XPS and AES equipment, the important information is carried out from the implants by electrons whose energies are typically in the range of 0.1 to 1 keV, and whose mean of escape depth is approximately 1 nm. These characteristics and differences among equipment may explain the different elements detected in our sample data.

There is evidence that oxide continues to grow in vivo. Implants retrieved after 6 and 8 years of function have demonstrated oxide layers of 200 nm.[31,32] These oxide layers contained P, Ca, and Si as well as Ti, O, and C. Titanium compounds have been identified in the tissues next to implants.[32,33] Above-normal compound levels have also been documented in rabbit spleen and lung tissue following implant insertion.[34] It is, therefore, prudent to focus careful attention on any oxide contaminants since these could be leached out over time.

In conclusion, data from the present study suggest that the EDS analysis did not show any material-related cause in either early or late dental implant failure. However, these results should be considered with caution due to the methodology utilized in this study and further investigations must be conducted.

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