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Scanning electron microscopy and energy dispersive X-ray study of a recovered dental implant

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Abstract A recovered dental implant has been studied for surface contamination using SEM and EDS microanalysis. The implant had been in place for 4 years in an adult male (age: 56 years), who had poor oral hygiene and was a smoker. Loosening had occurred, and the implant was removed accidentally during the taking of an impression. Using SEM, three distinct regions were identified, a clear one where the metal appeared shiny and unaffected, a discoloured one, where the surface appeared smooth and uncoated, and a region where there was a distinct deposit. All three regions gave elemental compositions of approximately 85% Ti, 12% Al, 3% V, which is a little richer in aluminium than the nominal overall composition of the usual alloy employed in implants. All three regions showed the presence of carbon, with the highest levels being associated with the surface deposit, and the lowest with the clear region. Oxygen was also present in substantial amounts, with most being found in the discoloured region. No nitrogen was detected, which suggests that the organic surface contamination is not due to interaction with proteins, despite their presence in saliva and crevicular fluid within the mouth.

Introduction

Dental implants based on titanium alloys are finding increasing use in clinical dentistry [1]. These materials exploit the excellent biocompatibility of titanium and its alloys in contact with bone. This allows the implant to become fully osseointegrated with the bone [2], *ie* to make intimate contact between the bone and the implant surface without an intervening layer of fibrous capsule [3, 4]. Devices are typically based on a screw thread, which increases the surface area against which the bone can grow, and which also distributes the forces evenly [5, 6]. This allows the implant to become securely anchored in the minimum time, and also prevents micro-movement, which otherwise promotes formation of fibrous capsule.

Clinical requirements for dental implants are severe, because their design requires them to protrude from the bone and through the gingival soft tissue. This means that there has to be adequate bone to support the device. In addition, patients must practise good oral hygiene and be non-smokers. Also, they should not be on anticoagulant therapy. Both of these latter requirements arise because of the need for good blood supply to the prepared socket, since deposition of blood at the implant surface provides the initial step in the overall process of osseointegration [7, 8].

Careful handling of implants prior to placement is important [3, 9], and typically involves the use of titanium-tipped forceps. However, despite these precautions, titanium surfaces have been shown by X-ray Photoelectron Spectroscopy, XPS, [10] and time-of-flight secondary ion mass spectrometry, ToF SIMS [11], to be contaminated with organic matter prior to use. This organic matter is rich in C-O functional groups and contains a variety of types of compound, including fatty acids and amides [11].

Despite the well-established indications for use of implants, there can still be failures, both of devices and of patient selection. In the current case, a titanium alloy implant was placed in a male patient (aged 56) who had poor oral hygiene and was a smoker. The device failed to integrate properly, and

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had become so loose that it was removed accidentally (after 4 years) during the process of taking a full-arch impression of the lower jaw. It proved to have patches of discolouration and some other obvious deposits when examined with the naked eye. A full investigation of the nature of these surface changes was carried out using SEM and EDS microanalysis and is the subject of this paper.

Materials and methods

A male patient, aged 56 years, presented for prosthodontic treatment (to JO) because of loosening of the extensive metal-ceramic bridgework he had in the lower jaw. The bridgework was based on seven abutments (4 teeth and 3 implants), and had been worn for four years following implantation. The patient was a smoker and had an abundance of dental plaque and debris in his oral cavity and also had gingivitis and had developed gingival resorption around the implants and the remaining natural teeth. X-rays revealed that there was also considerable bone resorption around the implants within the ridge. Finally, the upper jaw was edentulous and the patient wore a full denture.

The bridgework was removed, and evidence was found for mobility of three implants and two of the teeth. A preliminary impression was taken of the full arch of the lower jaw using alginate as the impression material. During the removal of impression, one of the implants (corresponding to the lower left 6 tooth) was accidentally pulled out by the impression material. The specimen (Fig. 1) was retained, and later examined using a scanning electron microscope (Princeton Gamma Tech, USA), with "windowless" EDS microanalysis for determination of the composition of the surface regions. Three distinct regions were identified, as follows (i) a clean part of the surface, (ii) a discoloured region and (iii) a zone on which material had been deposited. Elemental analysis was carried out at all three regions, and for each, the carbon:oxygen ratio was determined.

Results and discussion

An SEM image of the surface, showing the three distinct regions, is shown as Fig. 1. The regions were (a) clear (Fig 1, upper left zone), (b) discoloured (Fig. 1, upper right zone) and (c) coated (Fig. 1, lower zone). The results from EDS microanalysis of these regions are shown in Table 1. The clear surface showed a strong signal for titanium (79.31 wt%, corresponding to 57.76 atom%). There were also strong signals for aluminium and vanadium, suggesting that the alloy used to fabricate the implant was the widely used Ti-6Al-4V. However, the composition of this alloy is 90% Ti, 6% Al and 4% V, whereas the values we have obtained for relevant metals in the surface give proportions of Ti: 84.8%, Al: 12.1% and V:3.1%. In addition, there is a strong carbon signal corresponding to 29.61 atom%. This suggests that there is contamination of the surface by an organic material.

Table 1 Elemental composition of surface regions (Wt%)

Region	O	Ti	V	Al	S	P	Si	Zn	C	Ca
Clear	1.04	79.31	3.06	6.38	0.02	-	-	-	10.19	-
Discoloured	9.86	40.81	1.58	3.72	0.52	1.18	-	2.05	40.00	0.28
Deposit	6.84	9.10	0.29	0.75	0.40	0.75	0.32	0.68	80.57	0.30

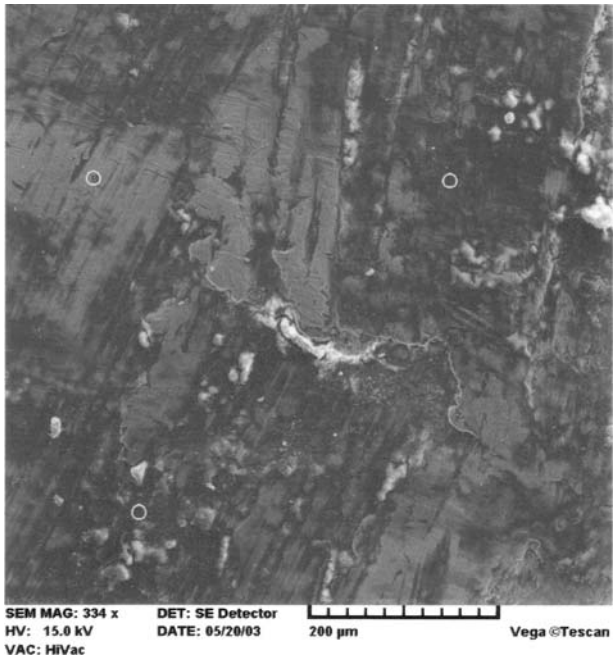

Fig. 1 SEM image of implant surface showing (a) clear region (upper left zone), (b) discoloured region (upper right zone) and (c) deposit (lower zone).

Previous studies of "as received" dental implants show that the surface may contain a variety of organic substances as potential contaminants, including fatty acids and other organic compounds [11]. Using XPS, for example, carbon contents of up to 71.9 atom% have been detected on implant surfaces, with correspondingly high concentrations of oxygen [12]. This was attributed to the tendency of titanium surfaces to adsorb carbon compounds, which they do readily either during fabrication or during storage [12]. This affinity for organic molecules clearly extends to those compounds occurring *in vivo,* as shown by the surface composition of the implant in the present case.

The discoloured surface shows much stronger signals for carbon and oxygen, and reduced signals for titanium, aluminium and vanadium. The elemental ratios are Ti: 83.5%, Al: 13.5%, V: 3.0%, which do not differ very much from those observed in the clear surface. However, the amounts of carbon and oxygen on the surface are very much greater.

Table 2 Carbon: Oxygen atomic ratio in different surface regions

Region	C:O atomic ratio
Clear	13.10:1
Discoloured	5.40:1
Deposit	15.68:1

There is also evidence for calcium, zinc, phosphorus and sulphur. The former may arise from small amounts of metallic corrosion, the latter as minor components of organic compounds involved in the discolouration process.

The region covered by an obvious deposit shows even greater amounts of carbon but less oxygen than the discoloured region, and a correspondingly lower carbon:oxygen ratio. The elemental ratio of the main alloy components is Ti: 84.8%, Al: 12.1%, V: 3.1%, which also does not differ much from the clear region.

The nature of the deposit is interesting. It is high in carbon and oxygen (Table 2), yet contains no nitrogen. In the mouth, it would have been exposed to a wide variety of naturally occurring molecules. For example, saliva contains numerous enzymes, such as esterase, cholinesterase and lipase [13]. Crevicular fluid, which is likely to have been found very close to the implant, also contains a range of enzymes, including collagenase, elastase [14] and other proteins [15]. Where gingivitis or periodontitis occur, the crevicular fluid is known to become a true inflammatory exudate [16], and contains an even wider variety of biomolecules. The occurrence of gingivitis in the patient in the current case means that this shift in the composition of the adjacent crevicular fluid would have occurred. However, despite the relative abundance of nitrogen-containing compounds, no nitrogen appeared to be deposited at the surface of the implant. Indeed, the deposit was found to consist mainly of carbon, with a ratio of carbon to oxygen to 15.68:1 (Table 2), which is surprising, given the widely reported affinity of titanium for nitrogen [17]. On the other hand, the deposit was found to contain minor amounts of phosphorus and sulphur, which indicates the complexity of the surface chemistry that led to the formation of the deposit.

Titanium and its alloys are known to have good properties for their applications dentistry [1], especially their biocompatibility when implanted into bone. However, as this study has demonstrated, they also have a high affinity for dental plaque, and are capable of developing strongly adherent deposits under clinical conditions. This emphasises the need for patients to practice good oral hygiene if these devices are to give satisfactory outcomes.

Conclusions

Our study of the surface of a dental implant recovered after 4 years in the mouth of a 56-year old male patient who was a smoker with poor oral hygiene enables the following conclusions to be drawn:

(1) The surface had become composed of three readily identified regions namely (a) a clear one where the metal appeared shiny and unaffected, (b) a discoloured one, where the surface appeared smooth but stained, and (c) a coated one with a distinct deposit of foreign matter.
(2) EDS microanalysis demonstrated that all three regions had elemental compositions of approximately 85% Ti, 12% Al, 3% V for the alloy.
(3 All three regions showed the presence of carbon, with the highest levels being associated with the surface deposit, and the lowest with the clear region.
(4) Oxygen was also present in substantial amounts, with most being found in the discoloured region.
(5) Nitrogen was not present, though it occurs in reasonable quantities in the proteins present in saliva and crevicular fluid. Despite this, none was detected in any surface region of the implant.
(6) Though the changes to the surface were the well characterised, the extent to which they contributed to the failure of the device is not clear

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