SEM examination and qualitative/quantitative elemental analysis of 65 implant systems – an intermediate report

Surface analysis of sterile-packaged implants

DR DIRK DUDDECK[1,2] AND DR JÖRG NEUGEBAUER, PHD[1,3]

For the third time in a row, the Quality and Research (Q&R) Committee of BDIZ EDI is examining sterile-packaged implants under the scanning electron microscope for the more than 5,500 members of the association. In cooperation with the University Hospital of Cologne, extensive qualitative and quantitative elemental analyses are performed on each of the implants studied. In 2008/2009, the surfaces of 23 implants were analyzed [1], a number that had grown to 54 different implants from manufacturers in nine countries by 2011/2012 [2]. Here, isolated implants showed residue from the manufacturing and/or packaging process, pecularities in the external threading or residual filings inside the implant. The halftime count in the current 2014/2015 study, which will be completed by the end of March 2015, already includes more than 60 implants. This report presents the interim results.

[1] Interdisciplinary Policlinic for Oral Surgery and Implantology and Department of Oral and Maxillofacial Plastic Surgery, University of Cologne, Germany
Director: Professor Joachim E. Zöller

[2] dedeMED – Medical Materials Research Institute
Klingsorstraße 116
12203 Berlin
Germany
www.dedemed-research.de

[3] Zahnärztliche Gemeinschaftspraxis Dres. Bayer, Kistler, Elbertzhagen und Kollegen
Von-Kühlmann-Straße 1
86899 Landsberg am Lech
Germany

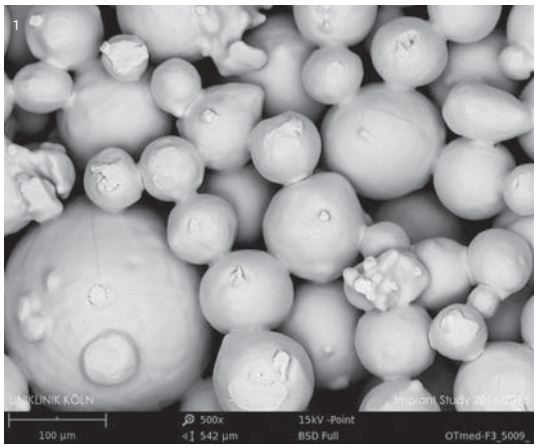
If you can believe some manufacturers, this study is completely unnecessary and BDIZ EDI could easily save itself the trouble. After all, they say, all implants investigated carry the CE mark, for which the manufacturer must have a quality management system in place for development, production and marketing under the EU declaration of conformity. Only certified proof of a performant QM system entitles implant manufacturers to affixing the CE marking to their products and placing them on the market in Europe. That the quality of a medical device is not necessarily related to the award of the CE mark by the EU Notified Bodies became evident in the 2012 scandal over substandard breast implants made of inferior industrial silicone. A research team from the British Medical Journal sought the coveted CE mark for a fictitious Chinese hip implant, which according to its (equally fictitious) documentation releases toxic metal ions and had high loss rates – and received it from five out of five Notified Bodies [3].

To be sure, dental implants have fortunately not been scandal-ridden and exhibit respectable 5-year survival rates even in the presence of organic impurities. Nevertheless, occasional implant losses occur for which there is no clinical explanation. But how can good production quality be compared? Are there any differences in quality at all?

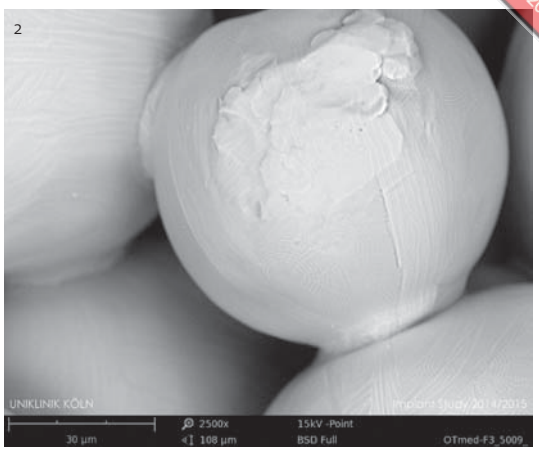
Background and objectives

Potential targets for analysis include the complex internal implant geometries (which are of primary relevance for prosthetic long-term success and will be the Q&R Committee's next study target) and the implant surfaces where bone and soft-tissue apposition will take place, which will therefore be the aspect of the implant in direct and permanent contact with the tissue. The surface of an implant determines the initial phase of the biologic response to the inserted implant and integration with the surrounding tissue [4]. Using the relatively simple tools of scanning electron microscopic (SEM) examination and qualitative and quantitative elemental analysis, the surface finish can be examined for technical precision and possible impurities.

The surface quality of implants depends on a number of different factors. We must distinguish between the actual manufacturing process, from the CNC-machined blank with product-specific surface processing, and the handling of the sterile-packaged implant. The packaging itself may also be



1 | OT-F^3 implant, sintered, OT medical (x 500).



2 | OT-F^3 implant, sintered, OT medical (x 2,500).

a source of organic contamination of the implant surface. The ever-evolving zirconia implants also undergo complex processing before they are packaged and sterilized.

The number of different implant systems is likely to be more than 300 worldwide. And this rising number of systems is associated with a rising number of technical and biological complications that practitioners have to deal with. The increasing "implant tourism" (patients travelling to other countries for purportedly cheaper implant treatment) forces more and more clinicians to address subsequent complications. Those treatments are difficult not least due to the fact that the implant system used cannot be ascertained [5].

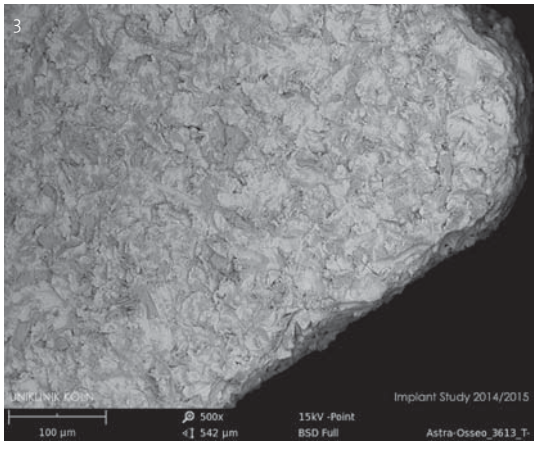
Implants differ from each other mainly in terms of different macro designs, such as differences in thread pitch or a more or less progressive thread design depending on the indication [6] as well as different surface treatments that determine their microstructure.

The most extensive long-term studies have probably been performed on almost smoothly machined implants with no additional surface treatment after machining, an implant type that has been in use since the early 1960s [7]. They are often used as a reference in materials science studies to document the effects of additional surface treatments. The machined SurfLink dental implant (Nano Bridging Molecules), which is structurally identical with the MK III (Nobel Biocare), shows a smooth surface (see SurfLink infobox, p. 62).

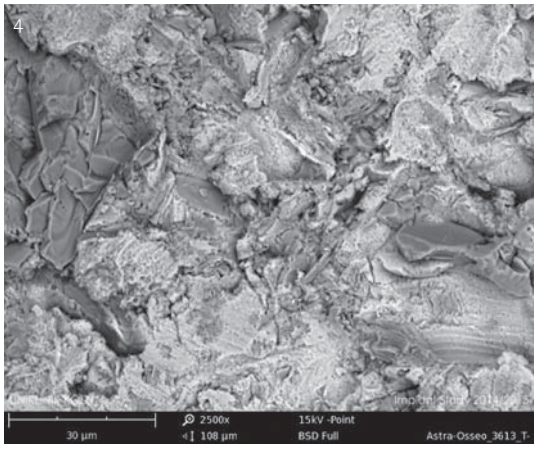
The microstructure of an implant has a major influence on osteoblast proliferation and differentiation. Numerous research groups and implant manufacturers have developed techniques for structuring the surface, leading to faster, optimized osseointegration and facilitating higher success rates and/or earlier loading of the inserted implants [8-12].

Surfaces can be shaped by additive or ablative procedures. Additive procedures such as titanium plasma coating are no longer in widespread use. Sintered implant surfaces (Figs. 1 and 2) such as that of the OT-F^3 implant (OT medical), where spherical particles are applied to the surface, have the advantage of providing a relatively large surface area. Due to its construction, its design includes no thread.

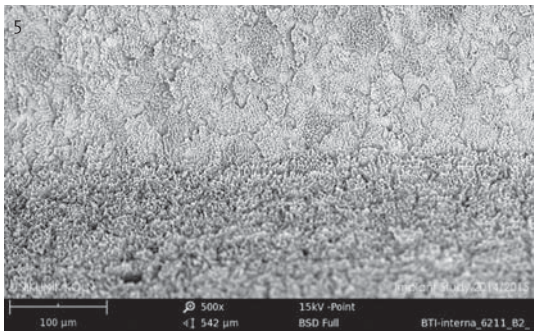
In ablative or subtractive procedures, implants are sometimes only blasted with hydroxylapatite (Zimmer) or titanium oxide (Astra, Dentsply Implants) (Figs. 3 and 4). Alternatively, they are merely etched,



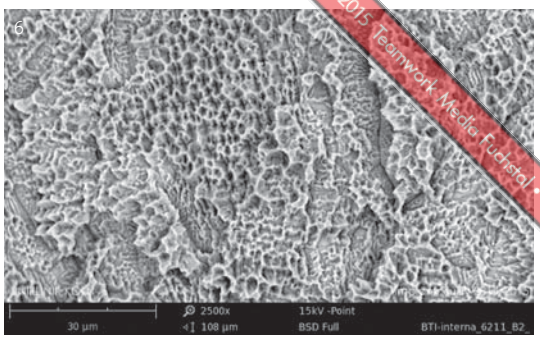
3 | Astra implant, blasted with titanium oxide, Dentsply Implants (x 500).



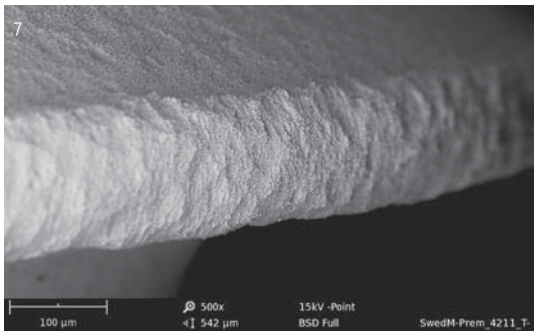
4 | Astra implant, blasted with titanium oxide, Dentsply Implants (x 2,500).



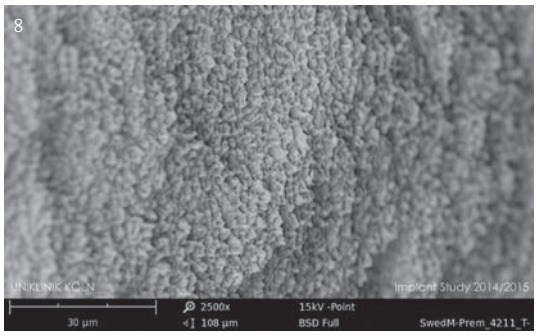
5 I Interna implant, etched, BTI (x 500).



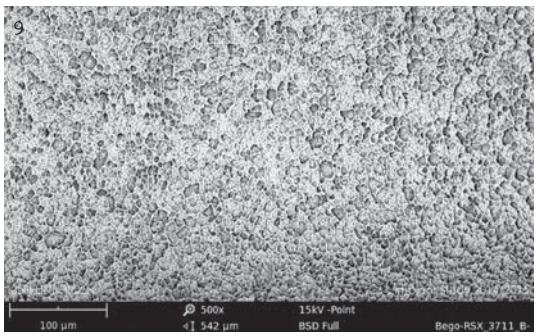
6 I Interna implant, etched, BTI (x 2,500).



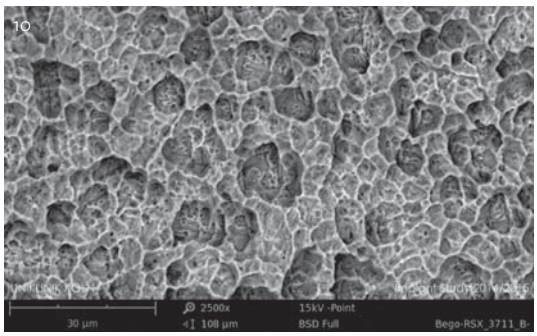
7 I Premium implant, blasted with zirconia and etched, Sweden Martina (x 500).



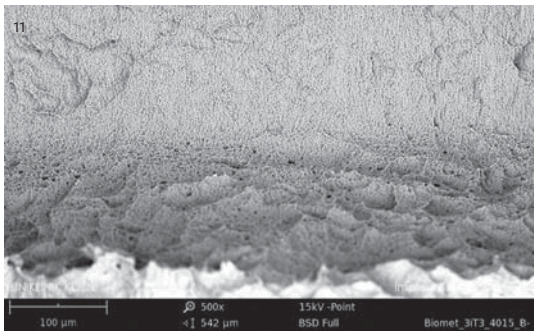
8 I Premium implant, blasted with zirconia and etched, Sweden Martina (x 2,500).



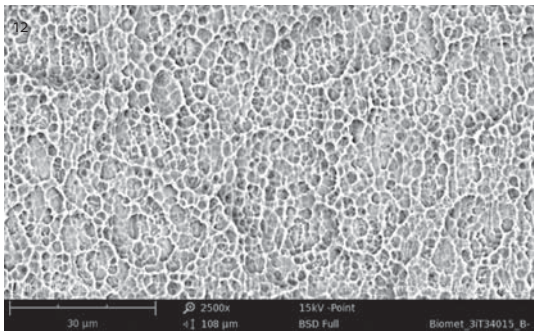
9 I RSX implant, blasted with aluminium oxide and etched, Bego (x 500).



10 I RSX implant, blasted with aluminium oxide and etched, Bego (x 2,500).



11 I T3 implant with CaP nanoparticles, blasted with CaP and etched, Biomet 3i (x 500).

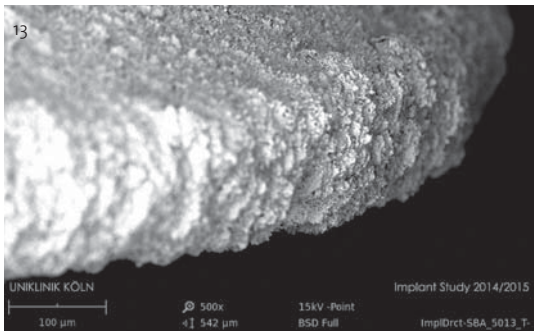


12 I T3 implant with CaP nanoparticles, blasted with CaP and etched, Biomet 3i (x 2,500).

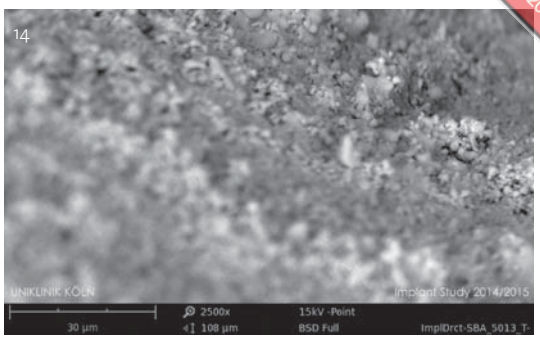
as in the Interna implant (BTI) (Figs. 5 and 6). Different abrasive agents are used for the blasted and etched implants that produce microroughness of 2 to 10 µm. Subsequent acid-etching not only removes the grit from the implant but also generates surface roughness of less than 2 µm. Abrasives include titanium, as in the ZirTi surface by Sweden Martina (Figs. 7 and 8), and aluminium oxide (Al_2O_3), as in the RSX implant by Bego. The typical structure is clearly visible, and in this case it shows no residue of the blasting material (Figs. 9 and 10). The T3 implant by Biomet 3i is first blasted with calcium phosphate, then etched twice and then coated with calcium phosphate nanoparticles (Figs. 11 and 12).

Implant Direct takes a similar route with its SBActive surface. This surface is blasted with hydroxyapatite, etched and then coated with highly crystalline hydroxyapatite. This coating, which is approximately 10 µm thick, is easy to see in a lateral image of a thread flange (Figs. 13 and 14).

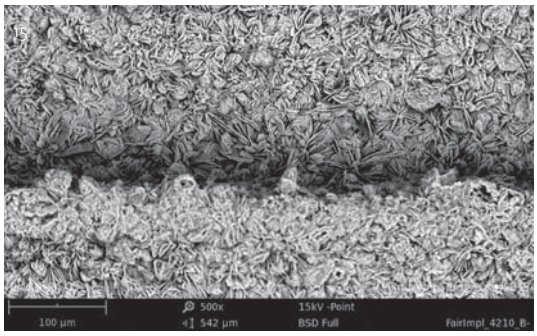
Resorbable calcium phosphate coatings, as in the Bonitex surface of the Alphatech implant (Henry



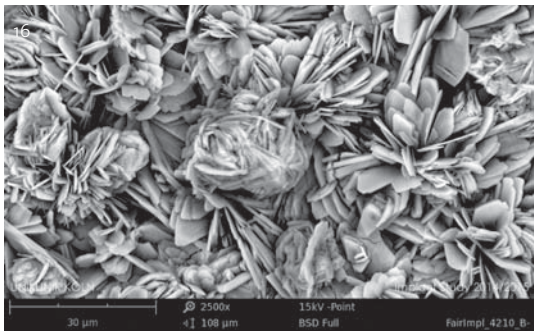
13 | HA-blasted and etched (SBActive) surface with HA coating, Implant Direct (x 500).



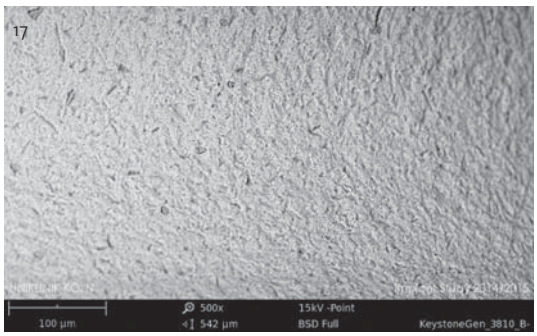
14 | HA-blasted and etched (SBActive) surface with HA coating, Implant Direct (x 2,500).



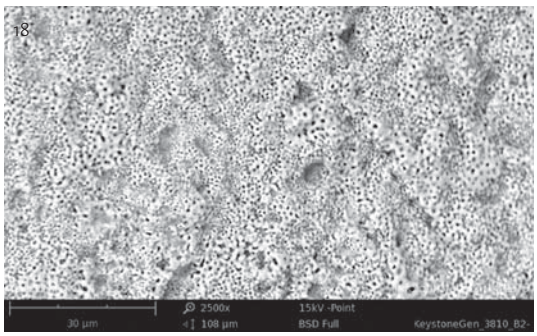
15 | CaP-coated (Bonitex) surface of the FairTwo, Fair Implant (x 500).



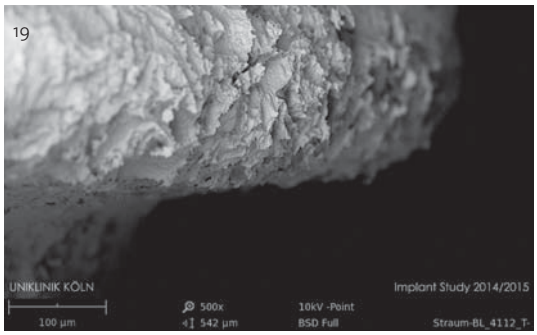
16 | CaP-coated (Bonitex) surface of the FairTwo, Fair Implant (x 2,500).



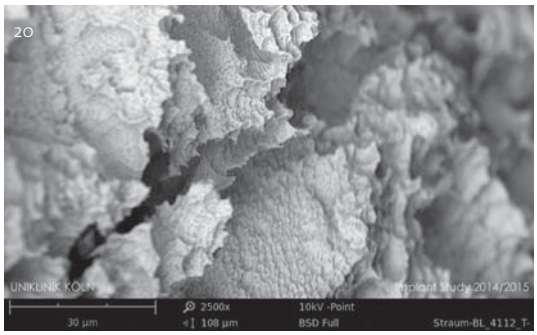
17 | Anodically oxidized (BioSpark) Genesis implant, Keystone (x 500).



18 | Anodically oxidized (BioSpark) Genesis implant, Keystone (x 2,500).



19 | Bone Level implant made of Roxolid with a SLA surface, Straumann (x 500).



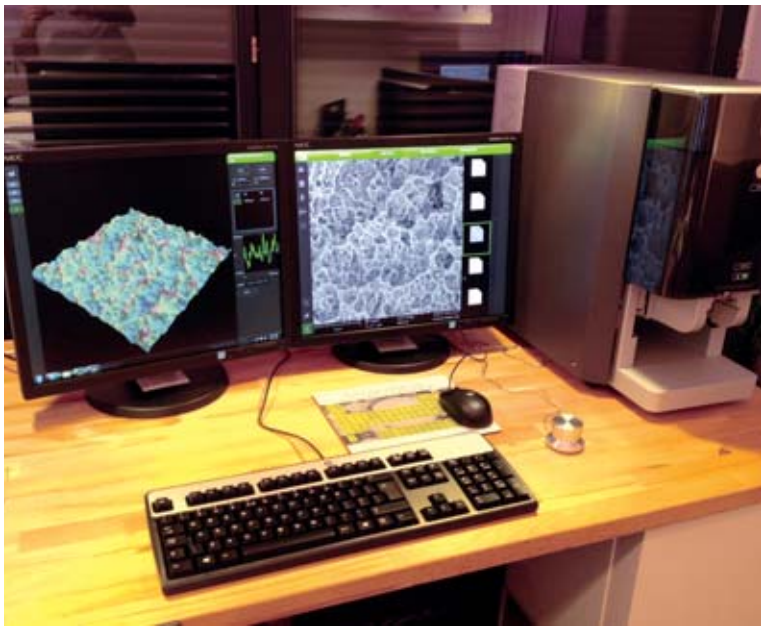
20 | Bone Level implant made of Roxolid with a SLA surface, Straumann (x 2,500).

Schein), in the CP version of the Integra implant (bicon), the Swiss Implant System (SGS Dental) or the FairOne and FairTwo implants (Fair Implant) (Figs. 15 and 16), intend to increase the osteoconductivity of the implants [13].

Anodically oxidized surfaces such as the TiUnite surface by Nobel Biocare or the BioSpark surface of the Keystone Genesis implant (Figs. 17 and 18) exhibit the typical micropores. An additional anodizing layer gives the polished Genesis implant shoulder its characteristic pink shade.

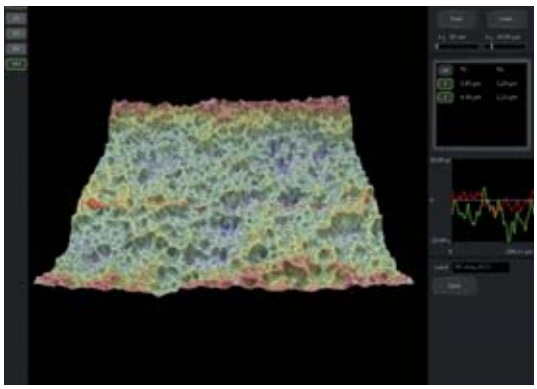
Roxolid (Straumann) is an alloy of titanium and zirconium whose biomechanical properties are favourable, especially for small-diameter implants (Figs. 19 and 20).

Other implant materials such as the various zirconia implants – which have improved considerably in recent years, especially in terms of surface roughness – and tantalum-titanium hybrid implants, as well as the first dental implants made of polyether ether ketone (PEEK), will be addressed in detail in the second part of this study report.



21 I Phenom proX scanning electron microscope.

22 I SEM sample unit with mounted implant.



23 I 3D roughness reconstruction of the implant surface (SICmax, SIC).

Manufacturer	Country
3M Espe	Germany
Alpha Dent	United Kingdom
Alphatech (Henry Schein)	Germany
Argon Dental	Germany
Bego	Germany
bicon	USA
Bio 3	Germany
Biomet 3i	USA
Biotec BTK	Italy
bredent	Germany
BTI	Spain
C-Tech	Italy
Camlog	Germany/ Switzerland
Champions	Germany
Dentaurum	Germany
Dentsply Implants Astra/ Xive/Ankylos	Germany/ Sweden
Fair Implant	Germany
Implant Direct	USA
Keystone	USA
medentis	Germany
Medical Instinct	Germany
MIS	Israel
Nano Bridging Molecules	Switzerland
Natural Dental Implants	Germany
Neoss	United Kingdom
NucleOSS	Turkey
Osstem	South Korea
OT medical	Germany
Schütz	Germany
SIC	Switzerland
SGS	Hungary
Southern	South Africa
Straumann	Switzerland
Sweden Martina	Italy
Trinon	Germany
TRI	Switzerland
Z-Systems	Switzerland

Table 1 List of implant manufacturers in the study (interim status, as of January 2015).

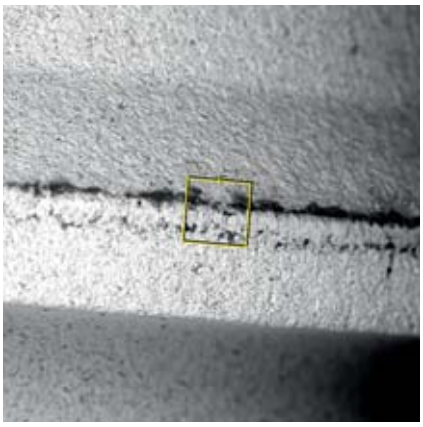
Materials and methods

So far in this study, 65 different implant systems from 37 manufacturers and ten countries have been examined by scanning electron microscopy (Table 1). The SEM instrument used (proX; Phenom, Netherlands) (Fig. 21) facilitates an exact representation of the surface topography and features a highly sensitive detector for backscattered electrons (BSE). This provides a first impression of the composition of the material examined already during the imaging phase (material contrast image), since elements with low atomic numbers (and fewer electrons) such as carbon or aluminium are shown as dark, while elements with higher atomic numbers, such as titanium or zirconium, appear as relatively bright.

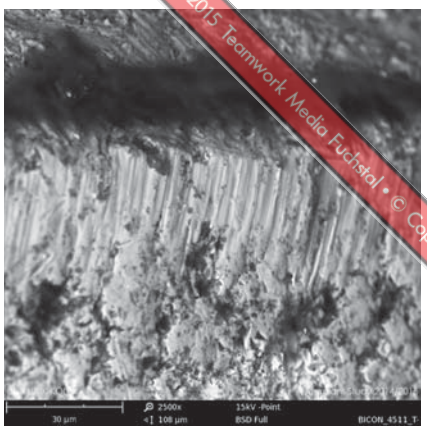
For the examinations, the implants were taken out of their packaging using a sterile forceps and attached to the sample holder (Fig. 22) before being introduced into the vacuum chamber.

In addition to detailed images, the instrument provides qualitative and quantitative elemental analyses of the various implants, using energy-dis-

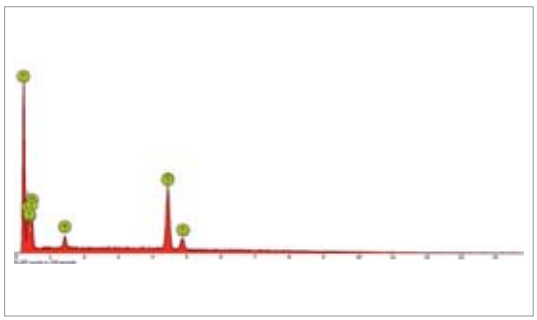
24 I Simple sterile packaging (LDPE zip lock bag) in a blister.



25 I Conspicuous organic residue on the external surface (Field of View).



26 I Organic residue, Integra, bicon (x 2,500).



27 I Qualitative elemental analysis.

	Atomic percentage	Certainty
C	65.7 %	0.99
O	27.9 %	0.97
Ti	5.4 %	0.98
Al	0.7 %	0.95
V	0.3 %	0.89

Table 2 Quantitative elemental analysis.

persive X-ray spectroscopy (EDX). Here, the electron ray causes the primary electrons emitted and the atoms of the sample surface to interact and to release electrons of the inner shell as a "secondary electron". The resulting gaps are immediately filled by an electron from a higher orbital. The resulting difference in energy is emitted as an X-ray quantum and detected by a thermoelectrically cooled detector, measuring both the elemental composition and their concentrations. An areal analysis and one or more spot analyses (in case of irregularities) were performed for each implant.

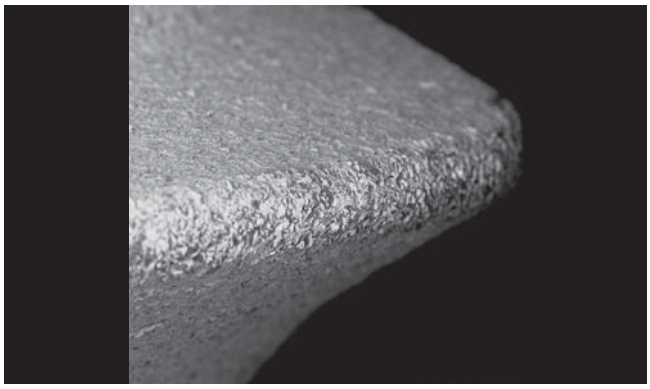
To document the surface roughness of each of the investigated implant systems, a so-called 3D roughness reconstruction was additionally performed that allows a visual comparison of the respective surface structures. Here, the three-dimensional shape of the object is calculated from the brightness distribution in the grid of the four quadrants of the backscattered electron detector. Using this shape-from-shading technology, implant-typical surface geometries can be represented spatially (Fig. 23).

Implant systems from the manufacturers listed in Table 1 have been studied for this interim report.

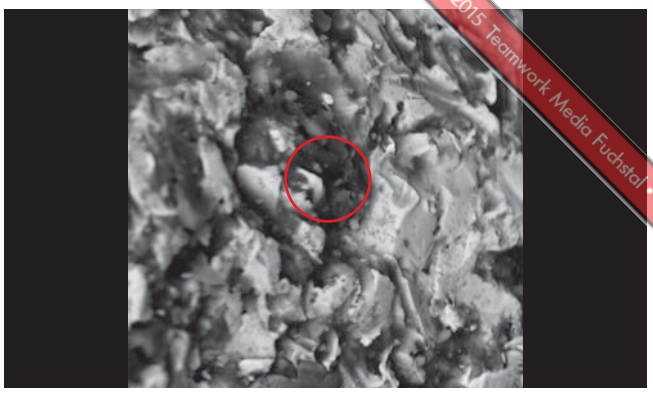
Results

As in the 2008/2009 study, the Integra implant (bicon), whose inner sterile packaging still consists of a simple zip lock bag made of soft polyethylen (LDPE) (Fig. 24), showed again organic residue. This systematic residue, which is not limited to isolated spots, was predominantly found near the outer edges of the parallel threads (Figs. 25 and 26) and may originate from direct contact with the packaging.

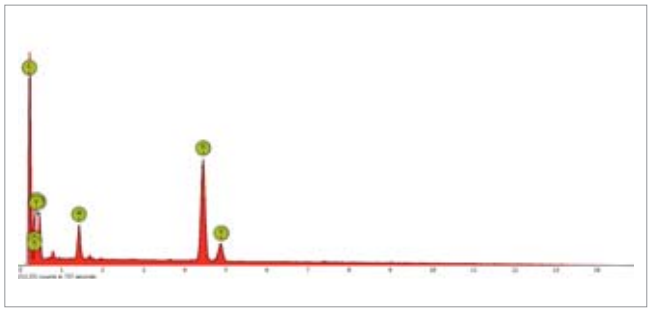
The qualitative elemental analysis shows not only the peaks typical of grade 5 titanium (Ti-6Al-4V) for titanium, aluminium and vanadium but also a clear peak for carbon (Fig. 27), which was confirmed by the quantitative analysis (Table 2). The calcium phosphate-coated version of this same implant had not exhibited organic contamination in the 2011/2012 study despite using the same packaging, possibly because of the lower surface roughness of the implant.



28 I Lower thread structure, QK implant, Trinon (x 500).



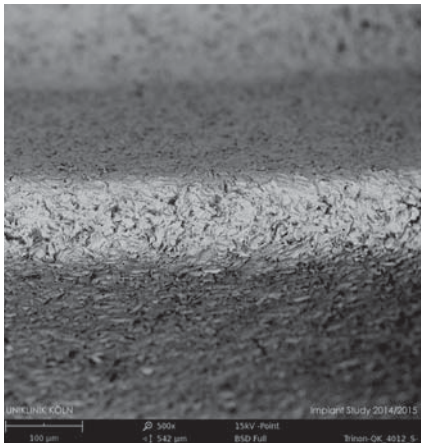
29 I Thread structure, QK implant, Trinon (x 5,000).



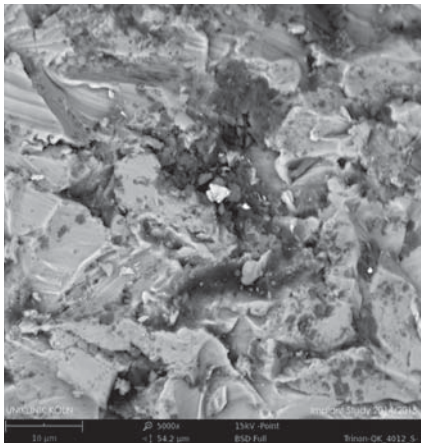
30 I EDX spectrum (qualitative elemental analysis), marked area, QK implant, Trinon.

	Atomic percentage	Certainty
C	86,6 %	0.99
Ti	10.4 %	0.99
Al	2.4 %	0.98
V	0.6 %	0.96

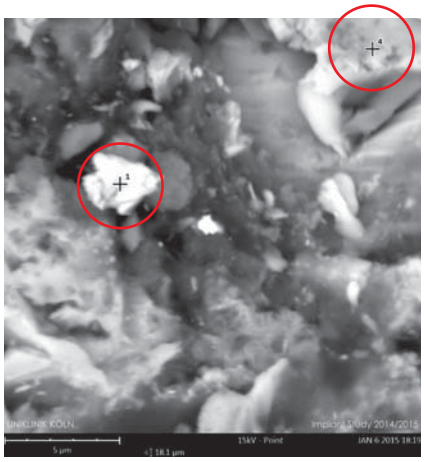
Table 3 Quantitative elemental analysis of the same area.



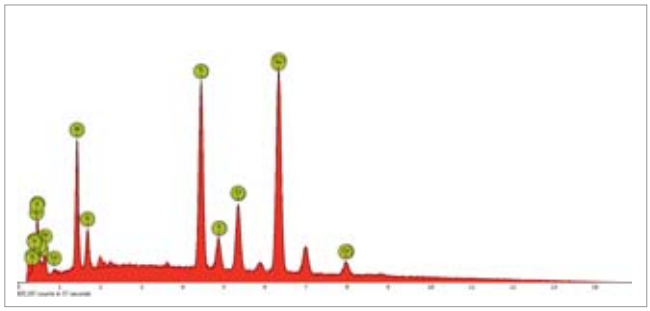
31 I Upper thread, QK implant, Trinon (x 500).



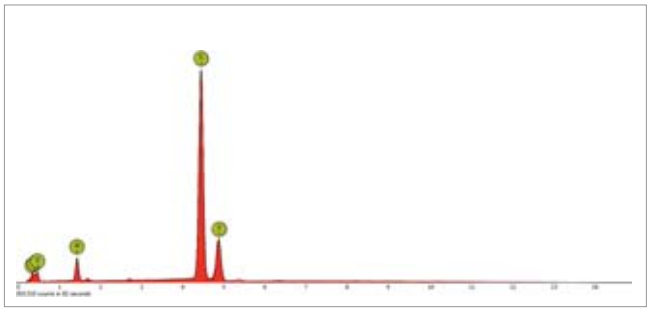
32 I Upper thread, QK implant, Trinon (x 5,000).



33 I Detail image (x 10,000) for EDX spot analysis (left: metal particles, right: control).



34 I EDX spectrum, spot #1 (metal particles).



35 I EDX spectrum, spot #4 (control).

	Atomic percentage	Certainty
Fe	35.2 %	0.99
O	20.9 %	0.98
Ti	15.1 %	0.99
Al	12.5 %	0.99
Cr	7.7 %	0.99
Cu	4.8 %	0.97
Si	3.2 %	0.98
V	0.7 %	0.94

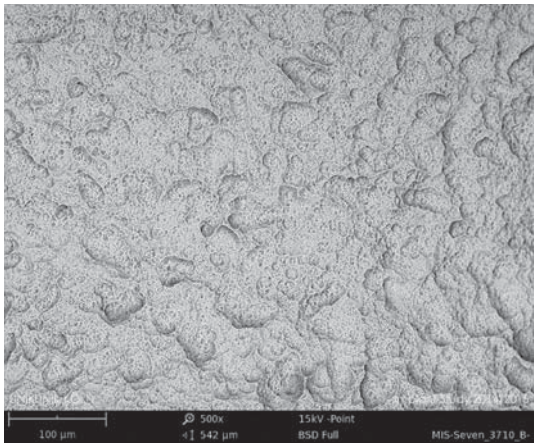
Table 4 Elemental distribution, spot #1 (metal particles).

	Atomic percentage	Certainty
Ti	53.9 %	1.00
O	36.0 %	0.98
Al	5.6 %	0.99
V	4.4 %	0.98

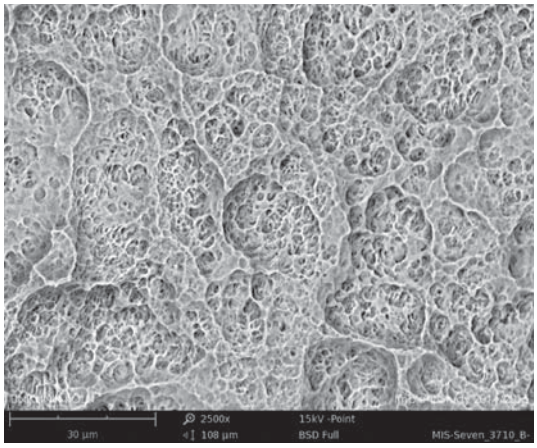
Table 5 Elemental distribution, spot #4 (control).

A different factor must have been responsible for the organic residue on the QK implant (Trinon, Germany) (Figs. 28 to 30, Table 3), as there is no contact with the packaging and the organic residue is not limited to the outer edges of the thread. The same implant shows smaller particles that already stand out in the material contrast image due to their bright grey tone. The elemental analysis detected iron, copper and chromium (Figs. 31 to 35, Tables 4 and 5). No similar clusters of these metal particles, approximately 3 µm in size, were found in any of the implants investigated so far in this study. As for the organic contaminants, one can only speculate about the clinical relevance of this finding, since the literature offers no conclusive evidence.

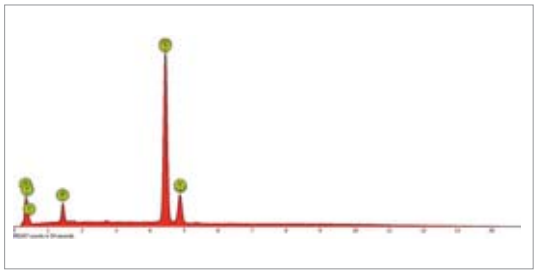
The C1 implant and the Seven implant (both MIS) stood out positively in the current study. Whereas during the 2011/2012 study, the Seven implant still exhibited blasting material on up to seven per cent of the surface, the current study did not even find isolated spots with residue on the two MIS implant types of grade 23 titanium (Ti 6Al-4V ELI) (Figs. 36 to 38, Table 6). Another positive surprise was the TRI-Vent Implant (TRI), which in the current study had a very precise external geometry (Fig. 39).



36 I Residue-free surface, MIS Seven implant (x 500).



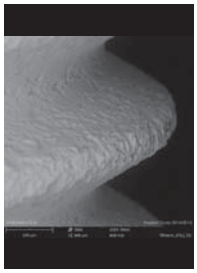
37 I MIS Seven implant surface with micro-nano-structure (x 2,500).



38 I Inconspicuous EDX spectrum of the MIS Seven surface (areal analysis).

	Atomic percentage	Certainty
Ti	87.3 %	1.00
Al	8.6 %	0.98
V	4.0 %	0.96

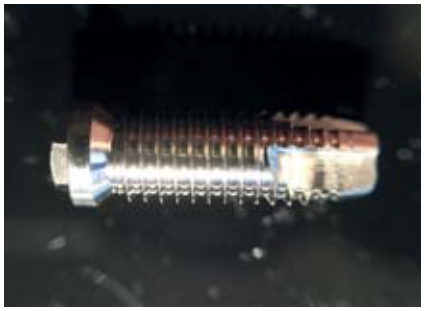
Table 6 Elemental composition resulting from the implant material (Ti 6Al-4V ELI).



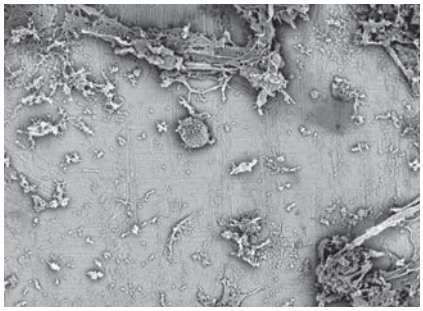
39 I Precise outer geometry, TRI-Vent, TRI (x 340).

SurfLink: Biomimetic monolayer for accelerated osseointegration

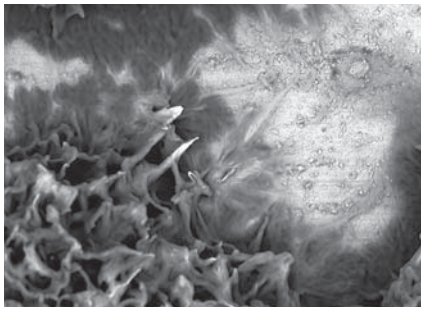
The machined SurfLink dental implant appeared inconspicuous at first (left). Unlike the original machined implant (MK III, Nobel Biocare), now available from the manufacturer only on special request, the surface of this implant has been treated with a covalently bound biomimetic monolayer, which due to its thickness of only about 1 nm cannot be detected by conventional SEM or EDX. Thus, this surface treatment is fundamentally different from the much thicker calcium phosphate coating. The monolayer presents osteoblasts with phosphorous-rich groups bound to the implant, mimicking natural hydroxyapatite [14]. The difference can be seen in machined implants retrieved from animals in an experimental study of the University of Zürich 52 weeks after insertion [15]. The SEM showed little adherent bone in the control group at the same magnification after removal of the implant (removal torque test) (centre), whereas the treated implants exhibited broad bone apposition on the smooth implant surface (right). The SurfLink treatment (Nano Bridging Molecules, Switzerland) can be applied at chairside to virtually all titanium and zirconia implant surfaces.



Machined SurfLink dental implant.



Bone growth on untreated (control) implant (x 2,500).

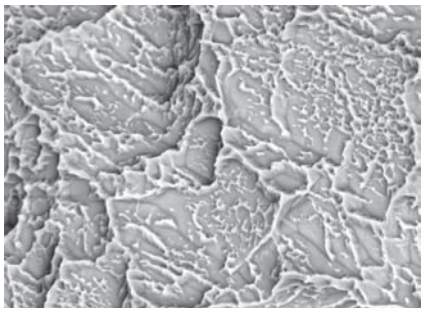


Bone growth on implant with a biomimetic monolayer (x 2,500).

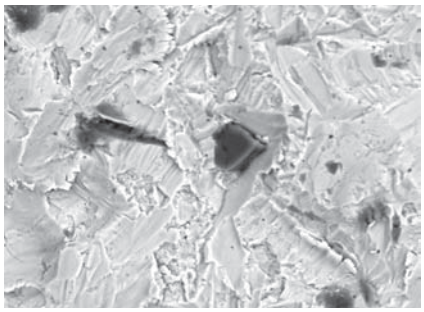
Replicate: Digitally reconstructed root-analogue titanium implant with ceramic abutment

The custom-made root-analogue Replicate implant (Natural Dental Implants, Berlin) plays a special role in this study. Unlike rotationally symmetric implants, it is fabricated individually based on digital reconstruction data acquired before extracting a hopeless tooth. After taking a CBCT, standard impressions are taken of both jaws and a bite registration is made. The impression is digitized in the micro-CT, synchronized with the CBCT data, and the tooth is reconstructed digitally from apex to crown. The digital model is segmented exactly at the site of contact with an appropriately shaped zirconia abutment. The root is milled from grade 4 titanium according to the recorded data, and its surface is sandblasted and acid-etched. The zirconia abutment and the root-shaped titanium implant are connected with solder glass (left). The SEM images show the different materials, titanium (centre) and zirconia (right).

Custom-made root-analogue Replicate implant, Natural Dental Implants.



Root aspect made of titanium without processing residues (x 5,000).



Aspect of a zirconia abutment roughened prior to inserting the crown (x 5,000).

Discussion

The manufacturers and users of implants which in this study exhibited some organic contaminants report clinical success rates that do not differ from those of implants by other manufacturers. Statements such as "Our success rates are high, so what is the problem?" may be fully justified when discussing statistical average. But the question remains: What happens to those organic contaminants once in the bone? It is hard to imagine that those organic contaminants should have a positive influence on osseointegration. At best, there will be areas with a lack of osseointegration, i.e. smaller areas or entire outer thread edges with less bone contact, because the osteoblasts will have better things to do than to settle on polyethylene residue. Or macrophages cause phagocytosis of these materials during the first remodelling phase, which would amount to biological purification of industrially manufactured surfaces. What then becomes of the phagocytosed materials is another question.

It is probably that residue on implants is tolerated in healthy patients. But can we be sure that this is also the case in immunocompromised high-risk patients? How about extended augmentation sites? And might not increased failure rates be due to processing residue, after all? These questions, however, should actually not arise in the first place, because impurities are preventable, as this study clearly shows.

Elaborate sterile packaging that prevents the implants from contact with the outer packaging are the rule in this study, not the exception. We owe it to our patients to eliminate avoidable risks and should not wait until the public, sensitized by scandals surrounding other medical devices, starts asking questions.

The publication of the results of the previous study just prior to IDS 2013 met with much praise, but was not equally popular with all manufacturers and practitioners. There was also – occasionally strong – criticism, in isolated cases even resulting in advertising contracts being cancelled. The aim of this study is and will remain the documentation of the manufacturing quality of dental implants. So it is all the more gratifying that, as in previous years, development efforts at many manufacturers have paid off in the form of a further increase in product quality, such as the elimination of organic contaminants, more precise threads or more user-friendly sterile packaging. The final study report with numerous examples and a list of all implant systems investigated will appear in the next issue of EDI Journal.

To find the list of references visit the web (www.teamwork-media.de).
Follow the link "Literaturverzeichnis" in the left sidebar.

A final report of this study will be published in the next issue. Readers will be able to request a comprehensive list of up to 100 analyzed implant systems starting in April 2015. Please request via e-mail from the BDIZ EDI office (office@bdizedi.org) or download from www.bdizedi.org.

Contact address

Dr Dirk Duddeck
Interdisciplinary Policlinic for Oral Surgery and Implantology
Department of Oral and Maxillofacial Plastic Surgery
University of Cologne
Director: Professor Joachim E. Zöller
Kerpener Straße 62 · 50937 Köln · Germany
dirk.duddeck@gmx.de