

**Quantitative and qualitative element-analysis
of implant-surfaces by SEM and EDX**

ANALYSIS REPORT

Customer/Name of Manufacturer: TRATE AG

Analyzed Product(s) / Sample: ROOTT DENTAL IMPLANT
Type: 02.00 (non sterile)
LOT: none

Technical analyses carried out by:

dedeMED • Medical Material
Research Institute Berlin

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Project manager:

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Head of Surface Analyses
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1 Material and Method / Study Protocol

1.1 Scientific Workstation and Test Procedure

The scientific workstation is a Phenom ProX Scanning Electron Microscope, equipped with a high-sensitivity backscattered electron detector that allows compositional and topographical imaging modes. EDX analyses are performed with a thermoelectrically cooled Silicon Drift Detector (SDD)

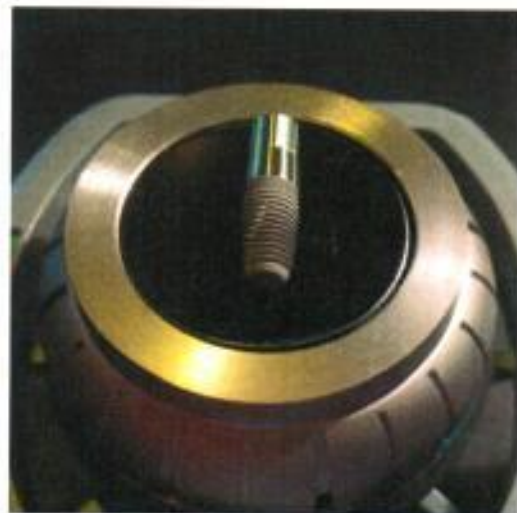


Workstation with Phenom ProX Scanning Electron Microscope

Without touching the surface, each implant will be taken out of the package with sterile forceps and will be fixed on the sample holders. After the vacuum is generated in the electron microscope imaging and EDX-analyses will be completed.



Fixed implant on the sample holder (example)



1.2 3D Roughness Reconstruction

With a specific 3D roughness reconstruction application, based on a „shape from shading“ technology, the SEM system used in this study is able to generate three-dimensional images and submicrometer roughness measurements.

3D imaging helps to interpret sample characteristics and makes images understandable. In addition the system is able to measure the average roughness (Ra) and the roughness height (Rz). Due to shape-from-shading technology Ra and Rz data in this study are to be generally understood merely as approximate values.

1.3 SEM-Examination of Implant Surface

Scanning electron microscopy (SEM) enables the topical evaluation of the implant surface. In particular at low voltages and small working distances, images with high contrast can be obtained. The high-sensitivity backscattered electron detector is generating images in compositional and topographical modes to a magnification of 10.000x.

Besides information about morphology and surface topography, the BSE detector allows to draw conclusions about the chemical nature and allocation of different remnants or contaminations on the sample material.

1.4 Qualitative and Quantitative Analysis of Implant Surfaces (EDX)

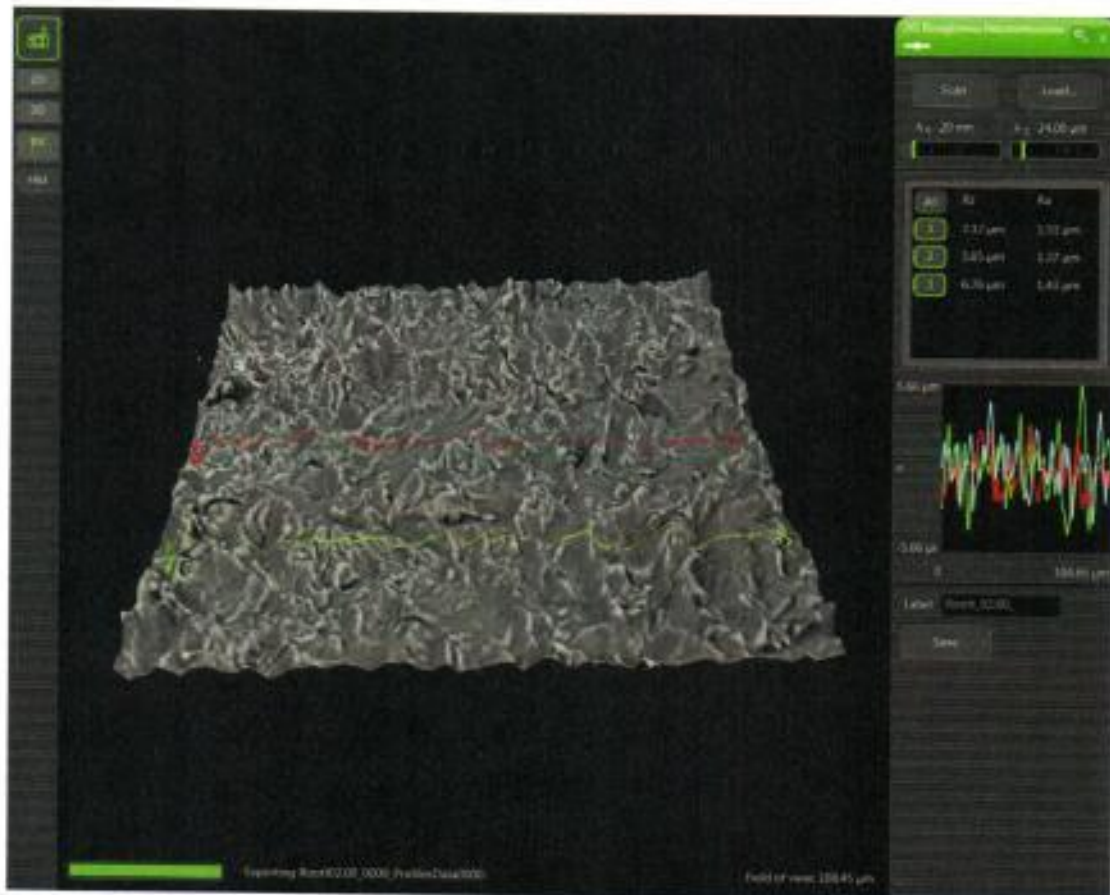
Energy Dispersive X-ray Spectroscopy (EDX) analyzes X-rays generated by the electrons of the electron beam (CeB₆ electron source) while they are interacting with the sample. Each element emits specific X-ray peaks. The element identification software allows to identify even hidden elements within the sample via the spot mode analysis. All results are verified using iterative peak stripping deconvolution. An area-analysis and one or more spot analyses are performed for each tested implant (analysis of spots and areas by EDX). An area-analysis covers the entire implant area in the focus of the microscope. For a spot analysis, the electron beam is focused on a specific area to get information about selective accumulations on the implant surface.

If necessary, elemental mapping reveals the distribution of elements within the sample. Selected elements can be mapped. Compiling these elements with the backscattered image gives a clear insight into the distribution of elements within the sample. Line scan allows analysis to be performed over a selected line. A line profile of every selected element is displayed on the screen.

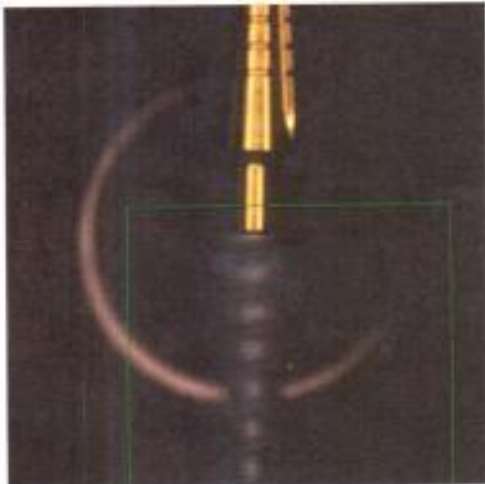
2 ROTT Dental Implant Surface 02.00



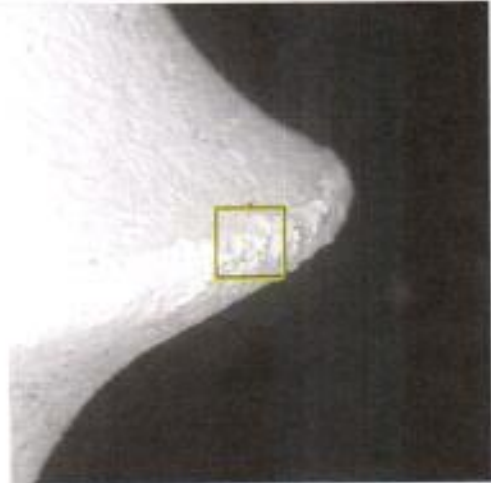
2.1 3D Roughness Reconstruction



2.2 Surface-Topography - Material Contrast Images (Thread)



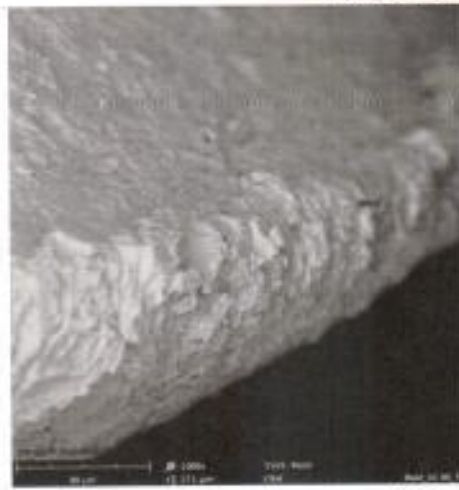
Phenom camera



Field of view



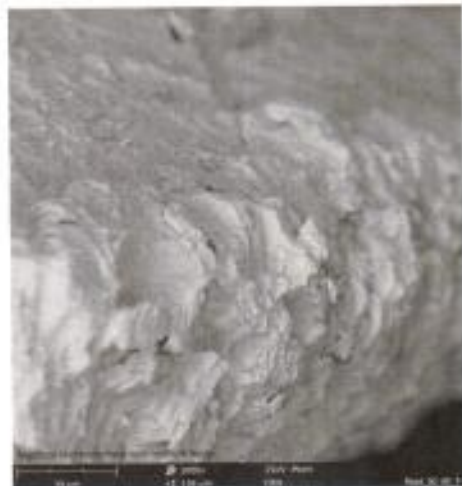
500x



1.000x



1.500x



2.000x

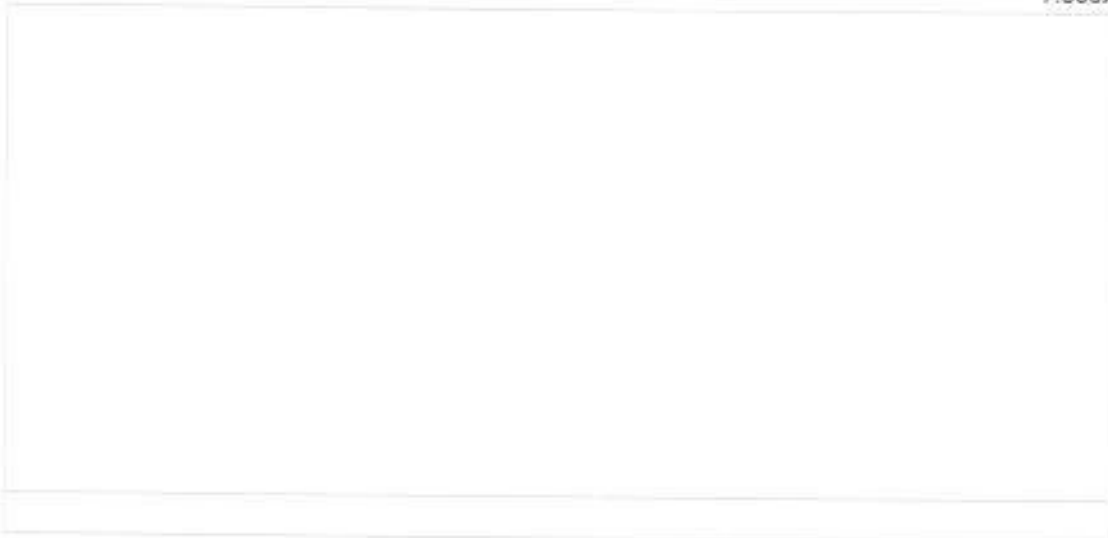
Surface-Topography - Material Contrast Images (Thread)



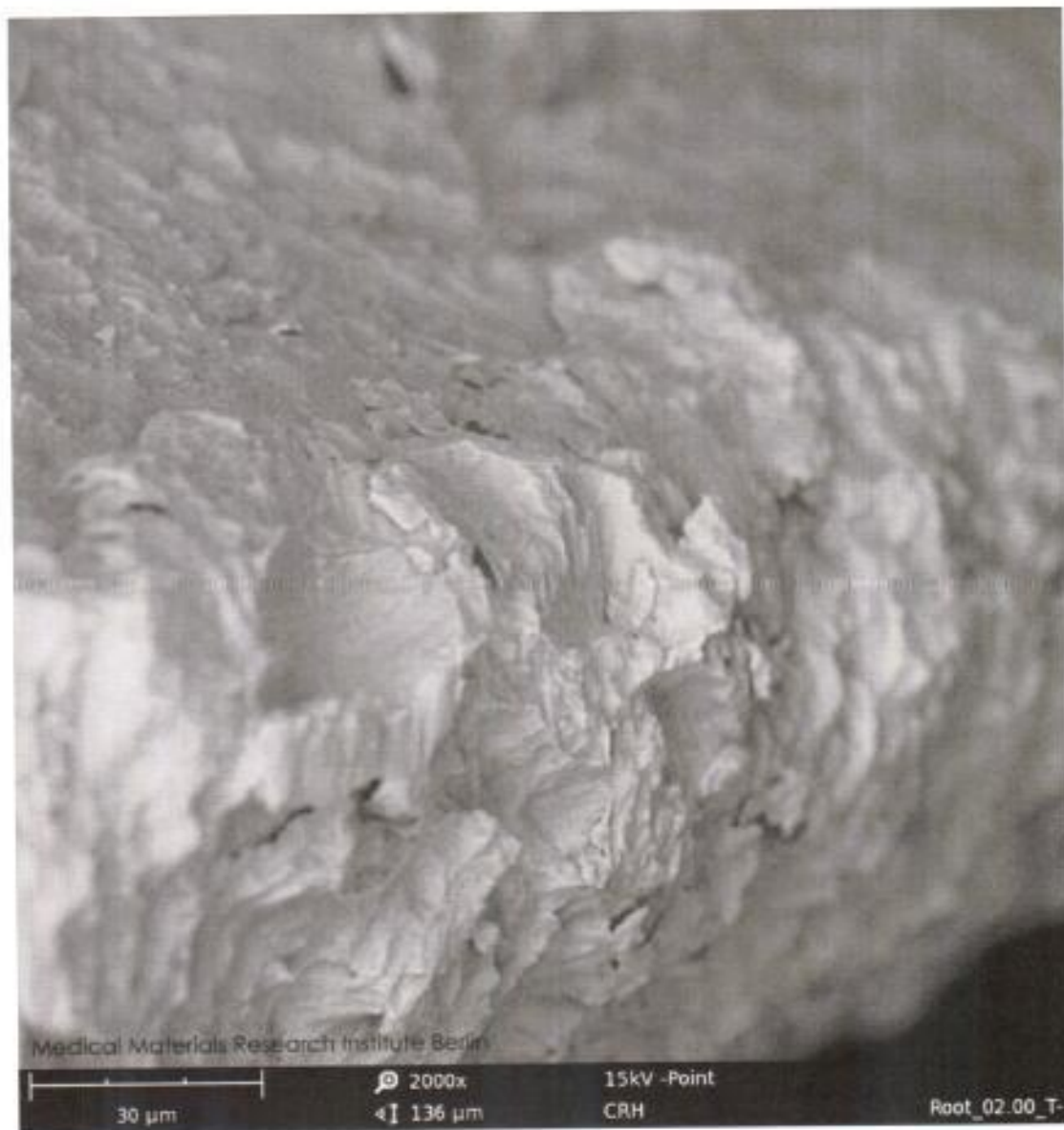
5,000x



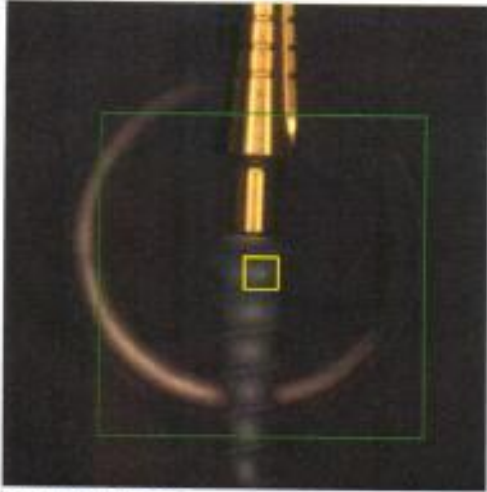
7,500x



Surface-Topography - Material Contrast Images (Thread 2.000x)



2.3 Surface-Topography - Material Contrast Images (Body_1)



Phenom camera



Field of view



500x



1.000x



2.000x

Surface-Topography - Material Contrast Images (Body_1)



2.500x



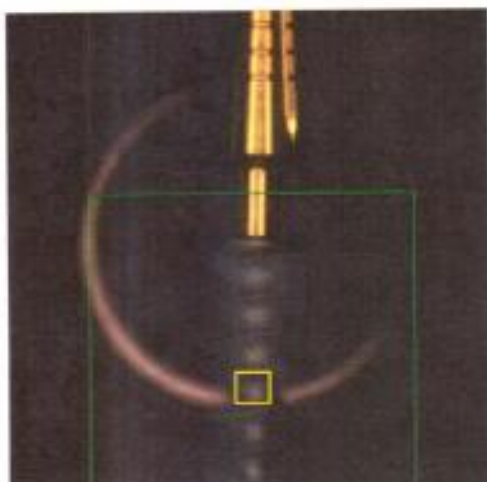
5.000x



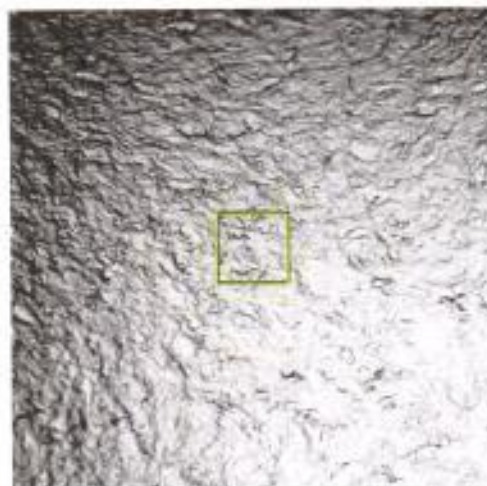
7.500x

Surface-Topography - Material Contrast Image (Body_1 2.500x)





Phenom camera



Field of view



500x



1,000x



1,500x



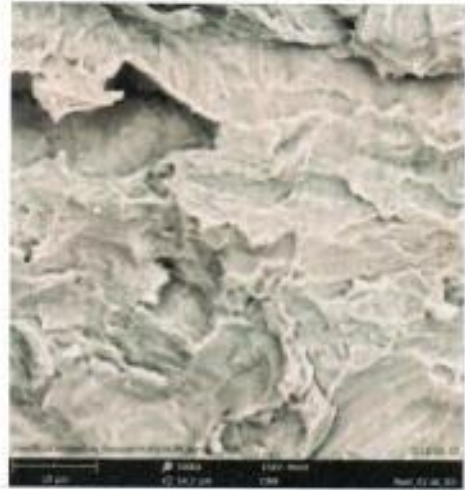
2,000x

2.4 Surface-Topography - Material Contrast Images (Body_2)

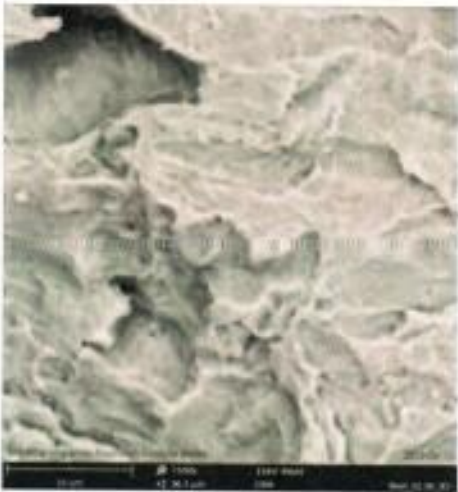
Surface-Topography - Material Contrast Images (Body_2)



2.500x



5.000x



7.500x

Surface-Topography - Material Contrast Images (Body_2 2.500x)



2.5 EDX Area Analysis (Body_1)



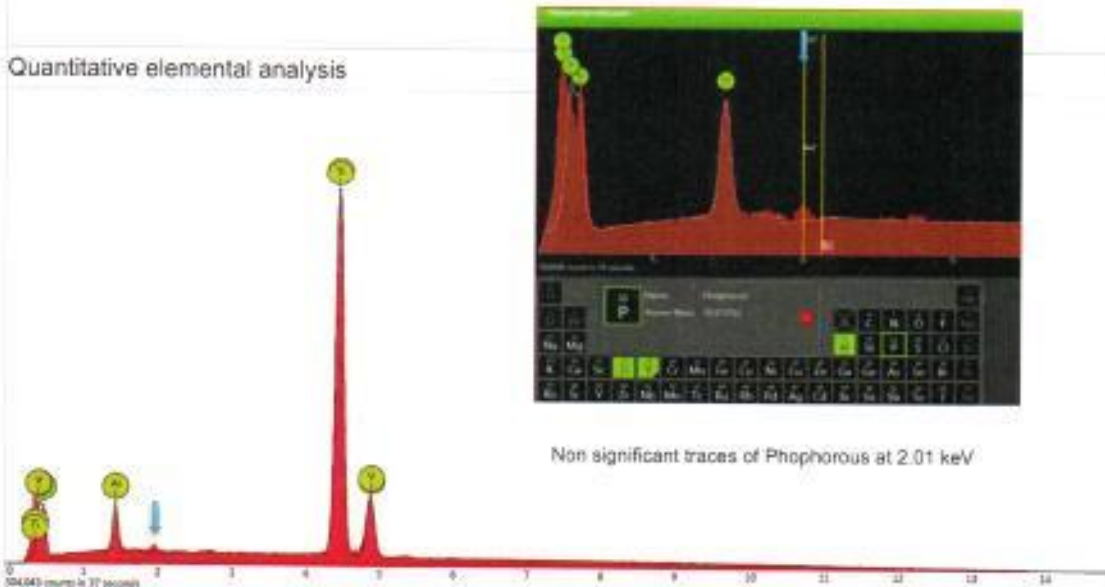
Field of view



Area

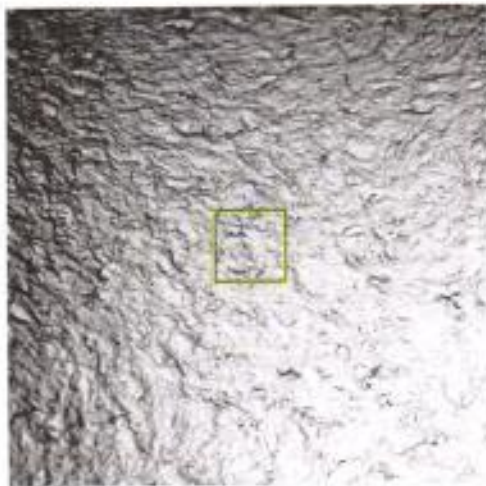
	Atomic percentage	Certainty
Ti	86.7 %	1.00
Al	10.2 %	0.98
V	3.2 %	0.95

Quantitative elemental analysis



Qualitative elemental analysis

2.6 EDX Area Analysis (Body_2)



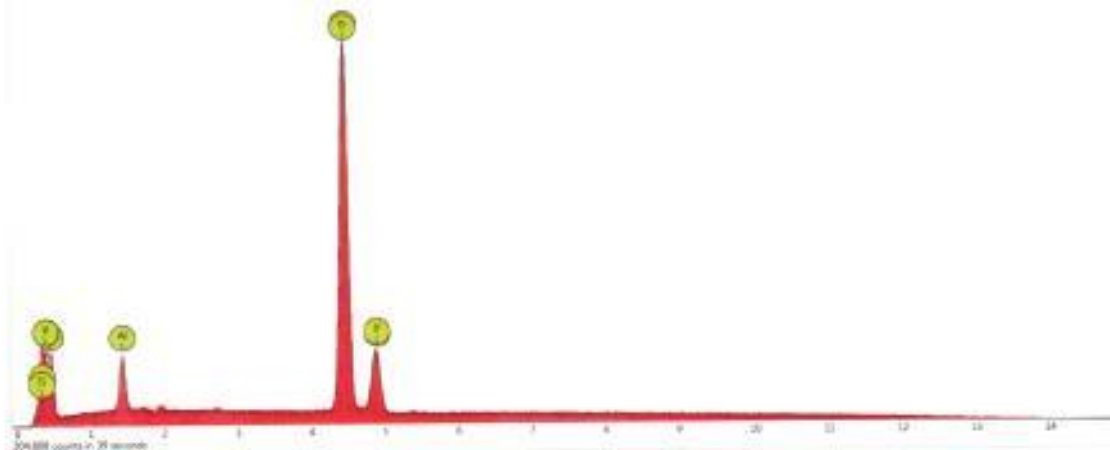
Field of view



Spot #3 (proximity reading)

Element	Atomic percentage	Certainty
Ti	85.6 %	1.00
Al	10.9 %	0.98
V	3.4 %	0.95

Quantitative elemental analysis



Qualitative elemental analysis

3 Synopsis

Name of Manufacturer:	Trade AG
Analyzed Product(s):	ROOTT DENTAL IMPLANT Type: "Version 02.00" LOT: none Exp. Date: not sterile
Investigator(s):	Dr. Dirk U. Duddeck
Study carried out by:	dedeMED Medical Material Research Institute, Berlin
Studied period:	June 2015
Methodology:	Phenom proX Scanning Electron Microscope, equipped with high-sensitivity backscattered electron detector (compositional & topographical modes) EDX Analysis: Detector type: Silicon Drift Detector (SDD) Thermoelectrically cooled (LN ₂ free) Detector active Area: 25 mm ² X-ray window: Ultra-thin Silicon Nitride (Si ₃ N ₄) window allowing detection of elements C to Am Energy resolution Mn K α \leq 140 eV Max. Input count rate: 300,000 cps
Summary/Conclusions:	The new surface type shows a medium rough surface and high precision of the outer geometry. No significant traces of organic residues were not found. No remnants of the blasting process (HA/TCP) were found.

4 COORDINATING INVESTIGATOR(S) SIGNATURE(S)

I have read this report and confirm that to the best of my knowledge it accurately describes the conduct and results of the analysis.

INVESTIGATOR: Dr. Dirk U. Dudeck

DATE: June 11, 2015

D. Dudeck



**UNIKLINIK
KÖLN**

Zentrum für
**Zahn-, Mund- und
Kieferheilkunde**

Klinik und Poliklinik für Mund-, Kiefer-, und
Plastische Gesichtschirurgie und Interdisziplinäre
Poliklinik für Orale Chirurgie und Implantologie

(BDIZ EDI) Implant-Study 2014/2015

Quantitative and qualitative element-analysis of implant-surfaces by SEM and EDX

PRELIMINARY STUDY REPORT

Name of Manufacturer:

TRATE AG

Analyzed Product(s):

ROOTT Dental Implant C - LOT E0916KL02C3010

Head:

Prof. Dr. Dr. Joachim E. Zöller

Interdisciplinary Policlinic for
Oral Surgery and Implantology
Dep. for Craniomaxillofacial and
Plastic Surgery; E 01 Trakt D
University of Cologne
Kerpener Str.62
50937 Cologne, Germany

In cooperation with the

European Association of Dental Implantologists BDIZ-EDI
Quality & Research Committee

Project manager:

Dr. med. dent. Dirk U. Duddeck

Head of Surface Analyses

Mobile: +49 171 54 77 991

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1 Background and Aim

Implant surfaces are modified by microstructures and surface extension to improve osseointegration. Numerous studies showed an increased adhesion and osteoblastic matrix-production on retentive titanium surfaces.

In 2008 the University of Cologne, Germany and the BDIZ EDI (European Association of Dental Implantology) with its Quality and Research (Q&R) Committee (www.bdizedi.org) performed a scanning electron microscopic study and analyzed the surfaces of 23 enossal titanium implants of several manufacturers at the Interdisciplinary Policlinic for Oral Surgery and Implantology, Department for Craniomaxillofacial and Plastic Surgery, University Cologne¹. The tested implants showed isolated and/or extensive deposits. Depending on manufacturing process, accumulations of organic material (carbon) or inorganic material like aluminum, silicon, phosphor, sulfur, chlorine, potassium and calcium were found.

In 2011-2012 we performed the same protocol on 57 dental implants from different manufacturers. However, the manufacturing of implants requires an adequate system of quality controls. Although some manufacturers have made substantial improvements since our first survey in 2008, the study in 2011-2012 again singled out a few implants with larger areas of surface blasting residue and selective organic impurities.²

The BDIZ EDI³, representing more than 5,500 active implantologists in Europe, was asked in its general meeting to continue these analyses periodically and to publish the results in the European EDI Journal.

While using the same material and methods regarding the technical setup this study allows comparisons to the results of previous studies.

As a continuance of the two studies cited above the aim of this study is to verify improvements of manufacturing and quality management as well as to demonstrate the high quality level of the participating manufacturers and implant companies.

1) Duddleck DU: Comparative investigation of various implant surfaces by SEM-Analysis. (Posterpresentation) 18th Annual Scientific Meeting of the European Association of osseointegration, 30 September - 3 October 2009, Monaco, France

2) Duddleck, DU et. al. Surface characteristics and quality of implants in sterile packaging, EDI Journal 2013-1

3) The BDIZ EDI has set the preliminary standards for a qualifying procedure of all implant systems and maintains structured continuing education. The primary tasks of BDIZ EDI are to provide members with support and advice and to improve the quality of implant materials.

2 Material and Method / Study Protocol

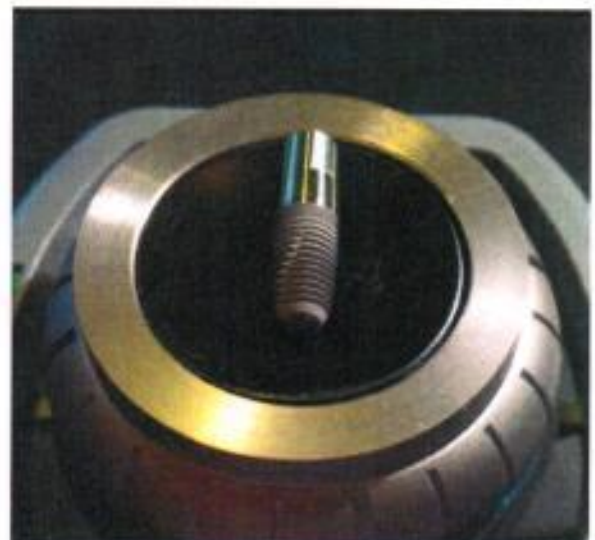
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Scanning electron microscopy (SEM) enables the topical evaluation of the implant surface. In particular at low voltages and small working distances, images with high contrast can be obtained. The high-sensitivity backscattered electron detector is generating images in compositional and topographical modes to a magnification of 20.000x.

Besides information about morphology and surface topography, the BSE detector allows to draw conclusions about the chemical nature and allocation of different remnants or contaminations on the sample material.

2.4 Qualitative and Quantitative Analysis of Implant Surfaces (EDX)

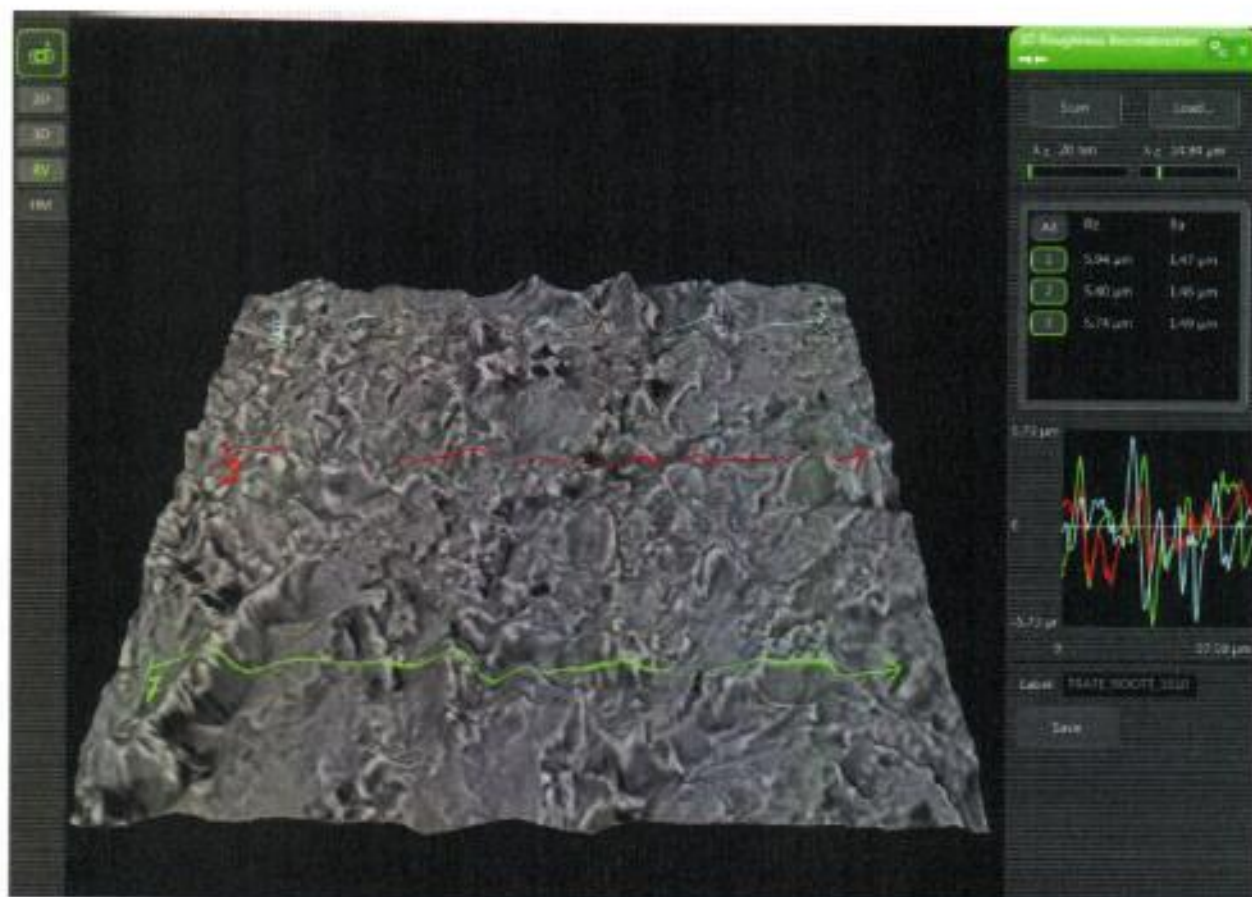
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If necessary, elemental mapping reveals the distribution of elements within the sample. Selected elements can be mapped. Compiling these elements with the backscattered image gives a clear insight into the distribution of elements within the sample. Line scan allows analysis to be performed over a selected line. A line profile of every selected element is displayed on the screen.

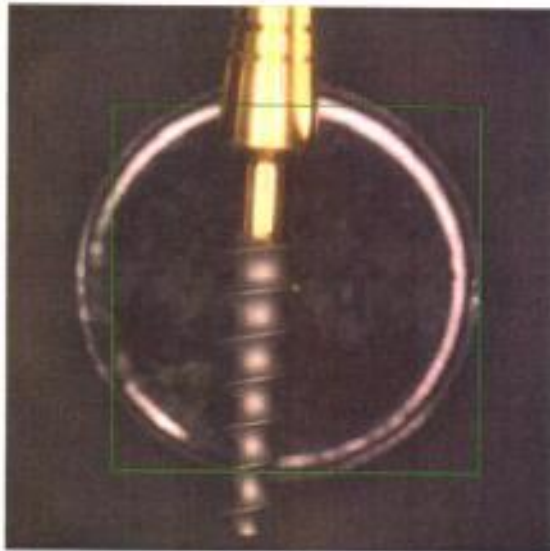
3 TRATE – ROOTT Dental Implant C - Sample #1 LOT E0916KL02C3010



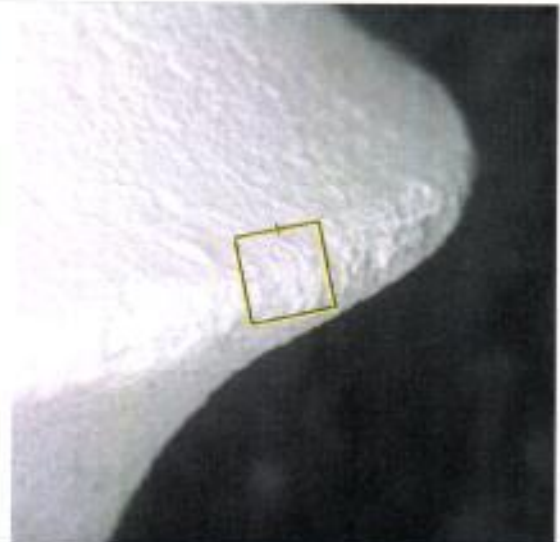
3.1 3D Roughness Reconstruction



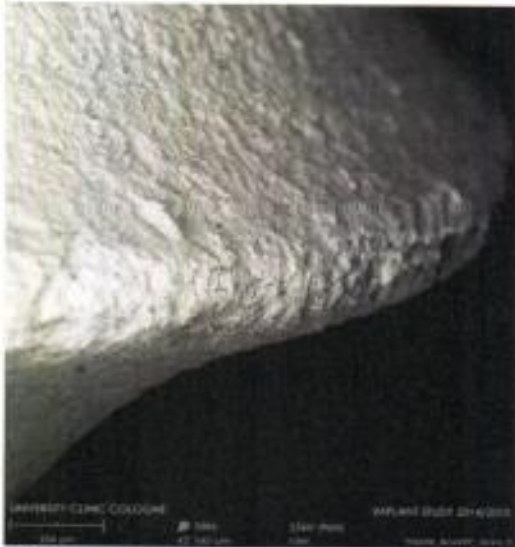
3.2 Surface-Topography - Material Contrast Images (Thread)



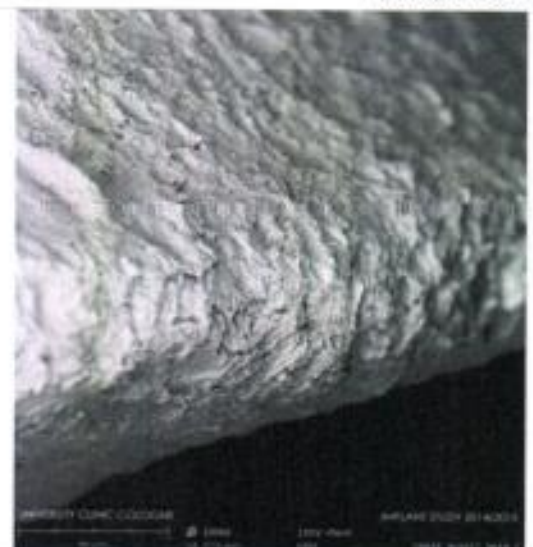
Phenom camera



Field of view



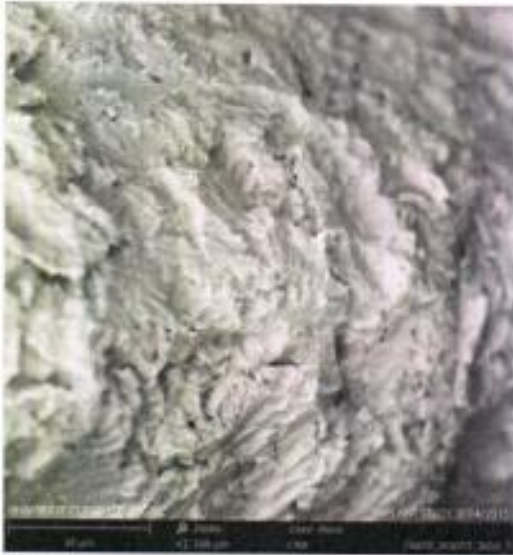
500x



1.000x



Surface-Topography - Material Contrast Images (Thread)



2.500x



5.000x

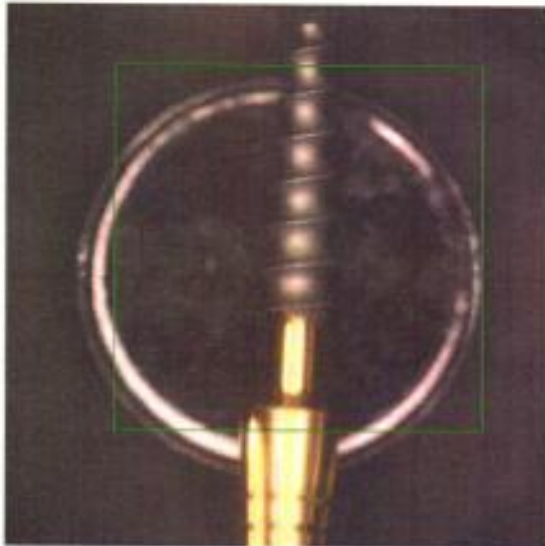


7.500x

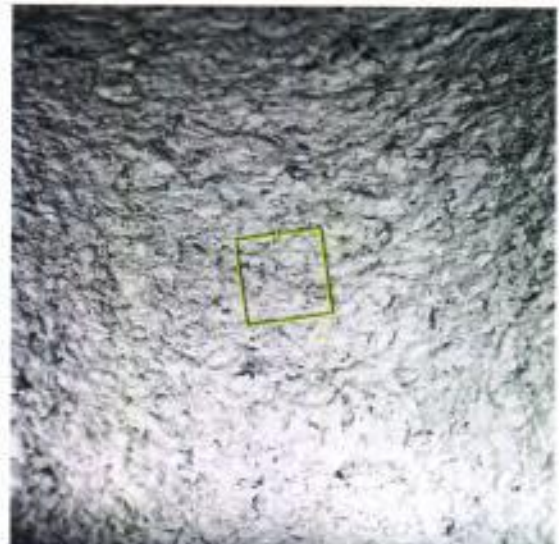


10.000x

3.3 Surface-Topography - Material Contrast Images (Body)



Phenom camera



Field of view



500x



1.000x



Surface-Topography - Material Contrast Images (Body)



2.500x



5.000x



7.500x



10.000x

3.4 EDX Analysis Area

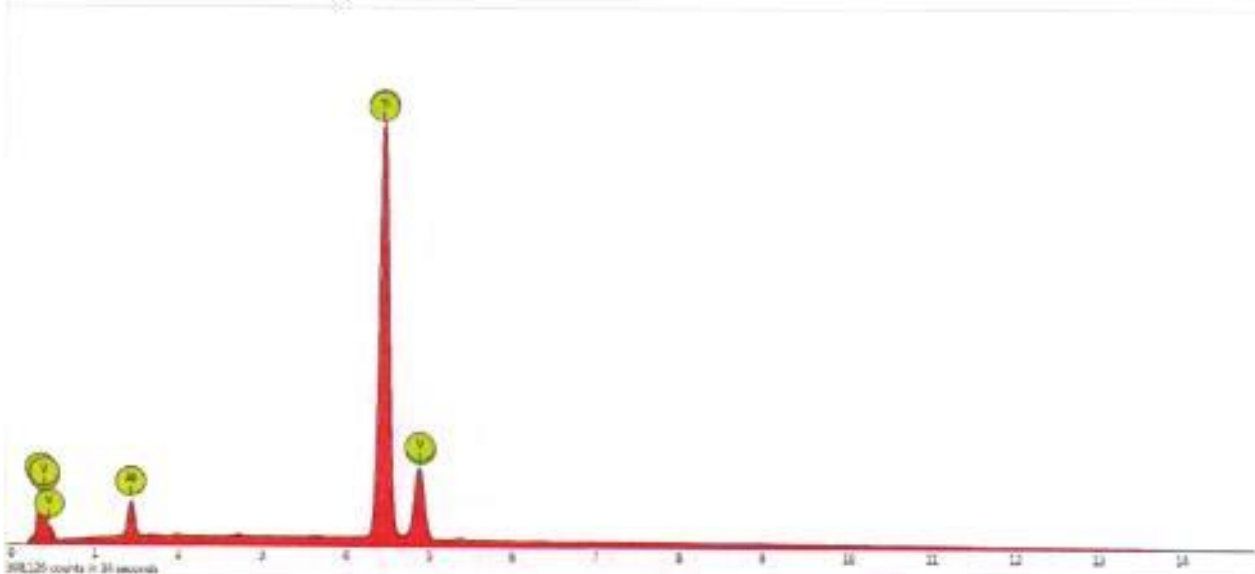


Field of view

Area

	Atomic percentage	Certainty
Ti	88.3 %	1.00
Al	6.9 %	0.98
V	4.8 %	0.97

Quantitative elemental analysis



4 Synopsis

Name of Manufacturer:	TRATE AG
Analyzed Product(s):	ROOTT Dental Implant C - LOT E0916KL02C3010 Exp. Date: 2020-11
Title of Study:	Implant-Study 2014/2015 Quantitative and qualitative element-analysis of implant-surfaces by SEM and EDX
Investigators:	Dr. Dirk U. Duddleck
Study centre:	Interdisciplinary Outpatient Dep. For Oral Surgery and Implantology, Dep. for Craniomaxillofacial and Plastic Surgery, University of Cologne
Study carried out by:	mmri.berlin - medical materials research institute berlin
Studied period:	November 2014 – December 2015
Methodology:	Phenom proX Scanning Electron Microscope, equipped with high-sensitivity backscattered electron detector (compositional & topographical modes) EDX Analysis: Detector type: Silicon Drift Detector (SDD) Thermoelectrically cooled (LN ₂ free) Detector active Area: 25 mm ² X-ray window: Ultra-thin Silicon Nitride (Si ₃ N ₄) window allowing detection of elements C to Am Energy resolution Mn K α \leq 140 eV Max. Input count rate: 300,000 cps
Summary/Conclusions:	The implant "ROOTT Dental Implant C" provided by TRATE AG for this analysis showed showed a high precision of the outer geometry. No significant inorganic remnants or organic particles could be found on this implant.

5 COORDINATING INVESTIGATOR(S) SIGNATURE(S)

STUDY TITLE: Implant-Study 2014/2015 Quantitative and qualitative element-analysis of implant-surfaces by SEM/EDX

STUDY AUTHOR (S): Dr. Dirk U. Dudeck

Interdisciplinary Polyclinic - Dep. for Oral Surgery and Implantology,
Dep. for Craniomaxillofacial and Plastic Surgery, University of Cologne,
Head: Prof. Dr. Dr. Joachim E. Zoller,
Address: Kerpener Str. 62, D 50937 Köln, Germany,
eMail: dudeck@mmri.berlin

I have read this report and confirm that to the best of my knowledge it accurately describes the conduct and results of the study.

INVESTIGATOR: Dr. Dirk U. Dudeck

DATE: December 30, 2015

D. Dudeck



Certificate of Study Result

BDIZ EDI Implant-Study 2014/2015
Quantitative and qualitative element-analysis
of 120 implant-surfaces by SEM/EDX

Name of Manufacturer: **Trate AG, Switzerland**

Analyzed Product(s): ROOTT Dental implant, compressive type
LOT: E0916KL02C3010 Exp. Date: 2020-11

Study centre: Interdisciplinary Policlinic - Dep. For Oral Surgery and
Implantology, Dep. for Craniomaxillofacial and Plastic
Surgery, University of Cologne

Study carried out by: mmri.berlin - Medical Materials Research Institute Berlin

Studied period: November 2014 – December 2015

Methodology: Phenom proX Scanning Electron Microscope,
EDX Analysis: Detector type: Silicon Drift Detector (SDD)
Max. Input count rate: 300,000 cps

Study author(s): Dr. Dirk U. Duddeck, PD Dr. Jörg Neugebauer
Interdisciplinary Policlinic - Dep. for Oral Surgery and
Implantology, Dep. for Craniomaxillofacial and Plastic
Surgery, University of Cologne,
Head: Prof. Dr. Dr. Joachim E. Zöller,
Address: Kerpener Str. 62, D 50937 Köln, Germany

Summary/Conclusions: **The implant ROOTT compressive type provided by
Trate AG for this analysis showed a high precision
of the outer geometry and no significant traces
of inorganic or organic residues.**

*I confirm that the report to the best of my knowledge accurately describes the
conduct and results of the study.*

Principal investigator: Dr. Dirk U. Duddeck
University of Cologne

City/Date: Cologne this December 13, 2015

D. Duddeck

