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:
Bio3 Implants GmbH

Analyzed Product(s):

Implant Advanced LOT 0350760902
Implant Advanced LOT 0350760898

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

Project manager:
Dr. med. dent. Dirk U. Duddeck

Head of Surface Analyses
Mobile: +49 171 5477 91
Fax: +49 30 640 80 206
Mail: duddeck@bdizedi.org

In cooperation with the
European Association of Dental Implantologists BDIZ-EDI
Quality & Research Committee

-

Technical analyses carried out by: dedeMED ▪ Medical Materials Research Institute Berlin
Dr. Dirk Duddeck ▪ Klingsorstrasse 116 ▪ 12203 Berlin ▪ Germany

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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 Cologne[1]. 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 EDI[3], 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 an 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) Duddeck 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) Duddeck, 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.

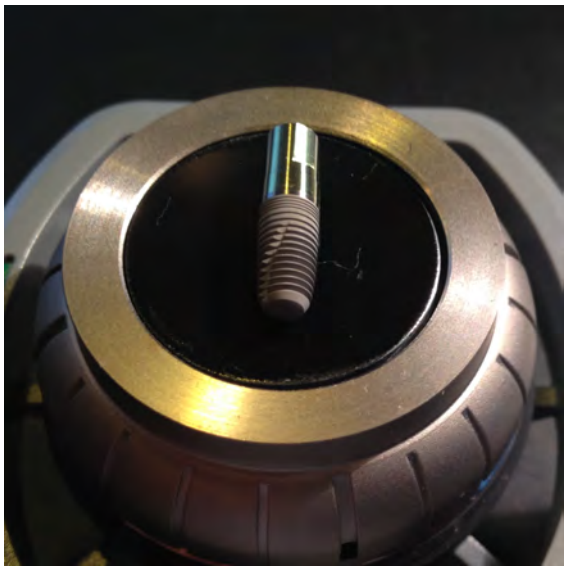
2 Material and Method / Study Protocol

2.1 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 (example) on the sample holder

2.2 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.

2.3 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 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)

Energy Dispersive X-ray Spectroscopy (EDX) analyzes X-rays generated by the electrons of the electron beam (CeB_6 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.

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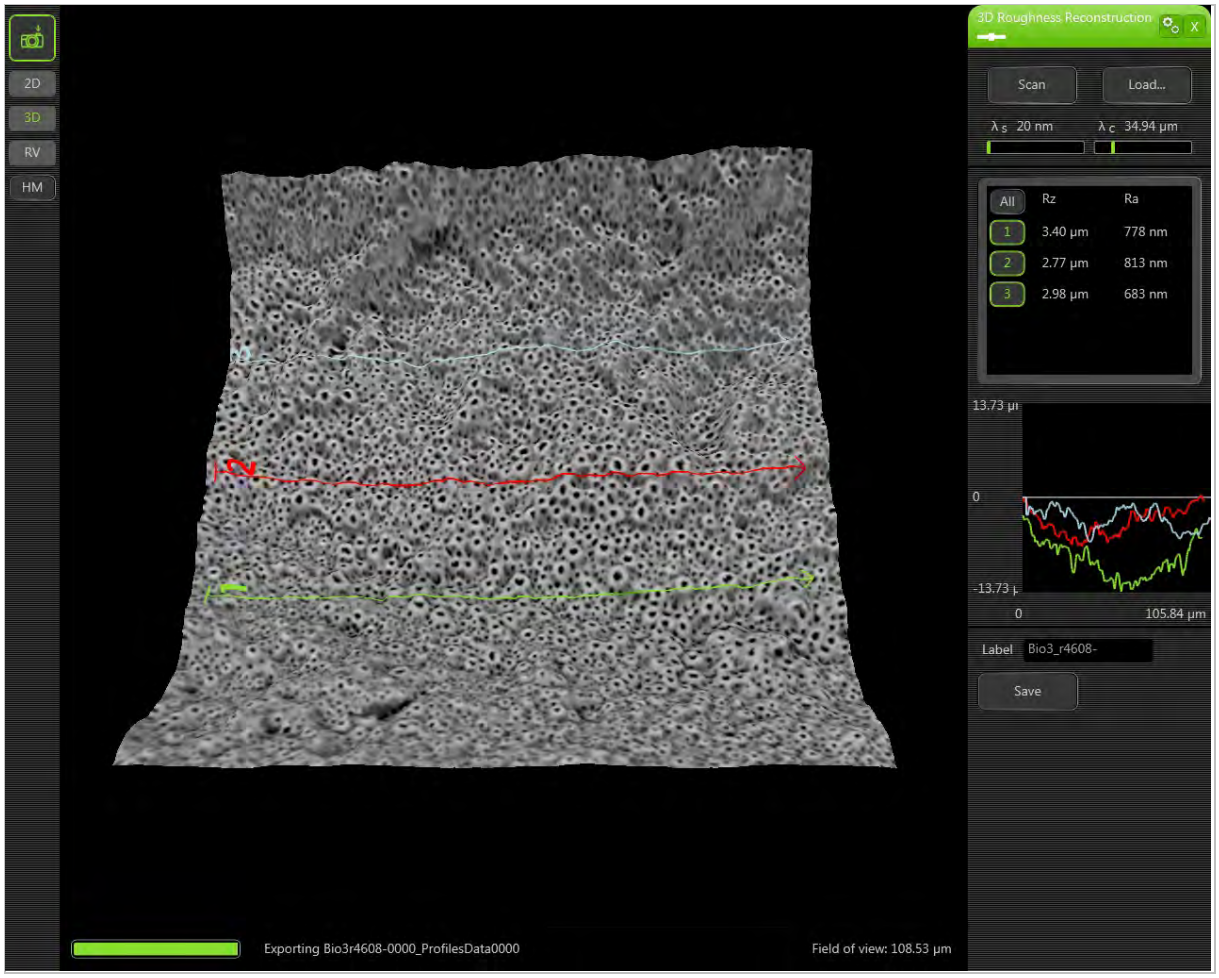
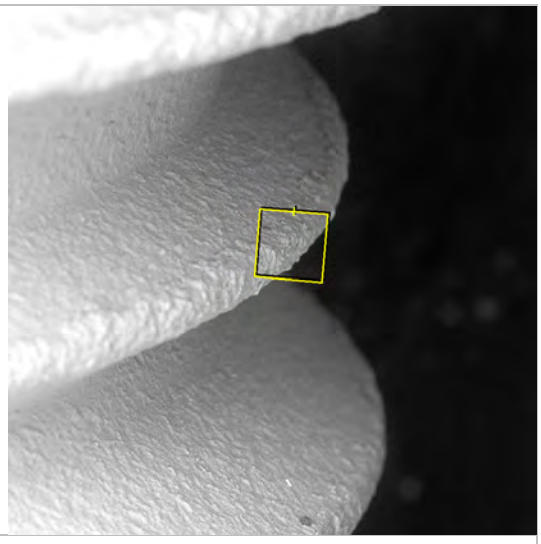
3.1 3D Roughness Reconstruction

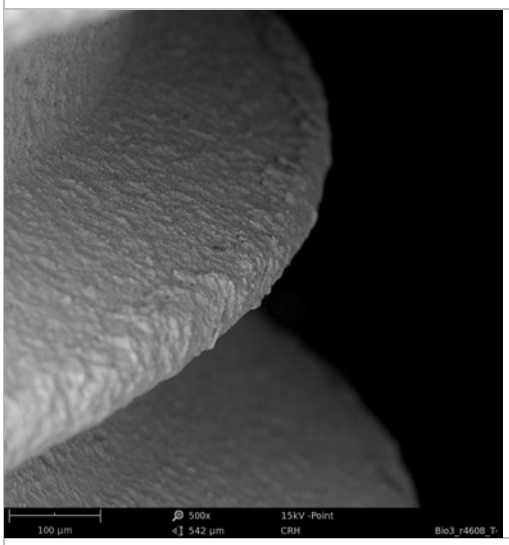


3.2 Surface-Topography - Material Contrast Images (Thread)

Phenom camera



Field of view



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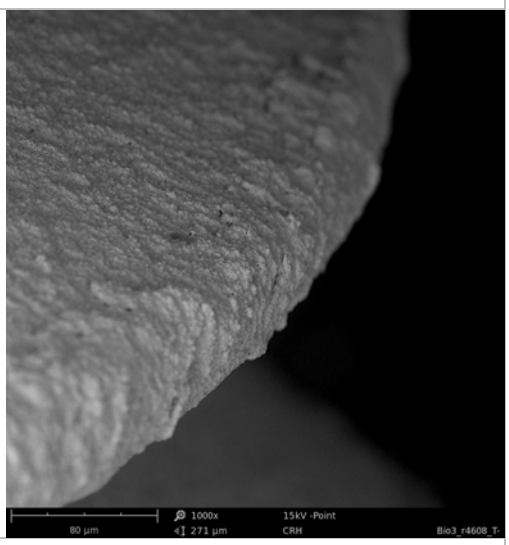

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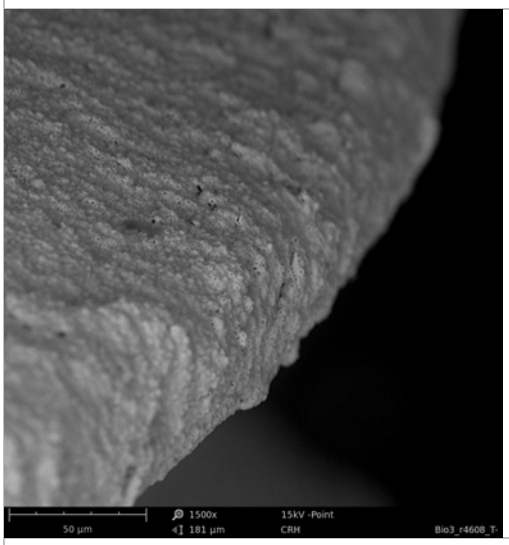

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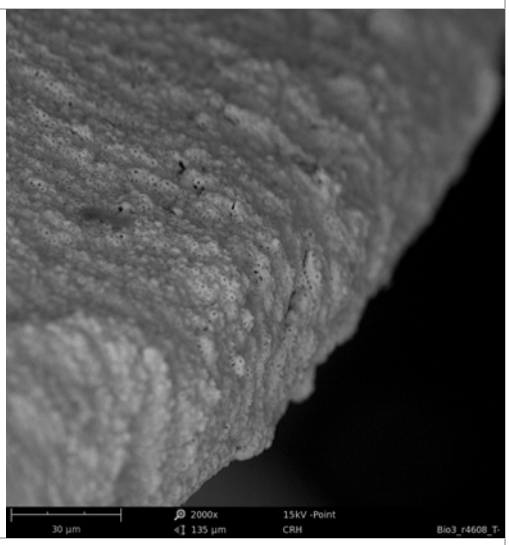

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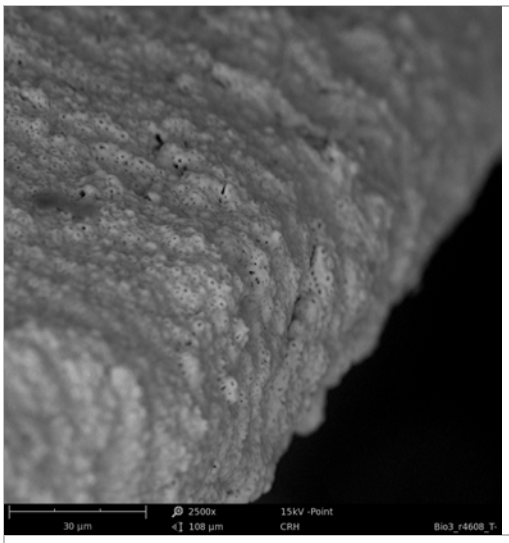
Surface-Topography - Material Contrast Images (Thread)



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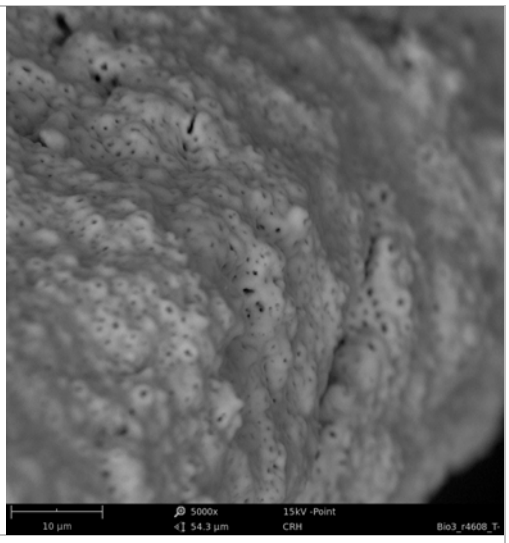

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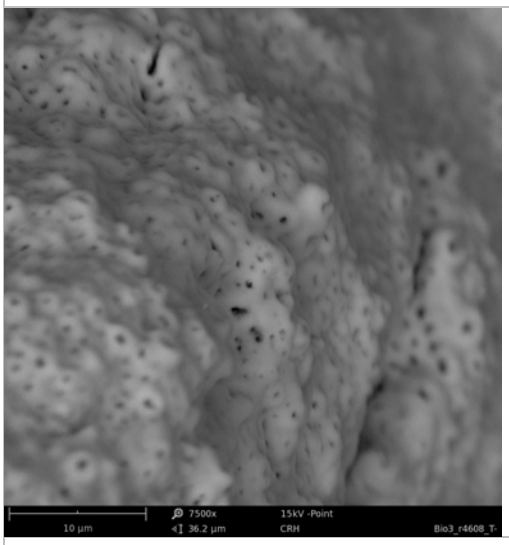

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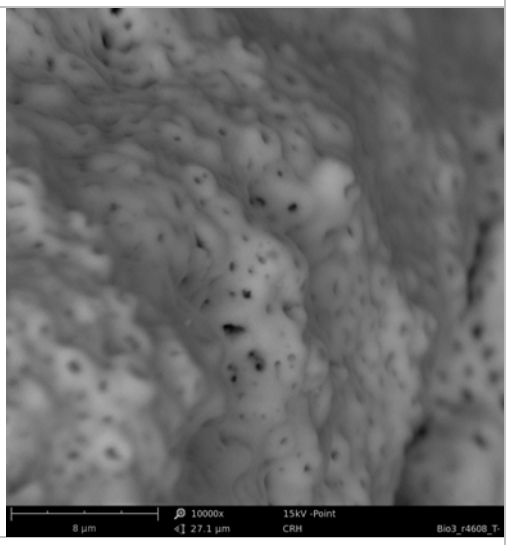

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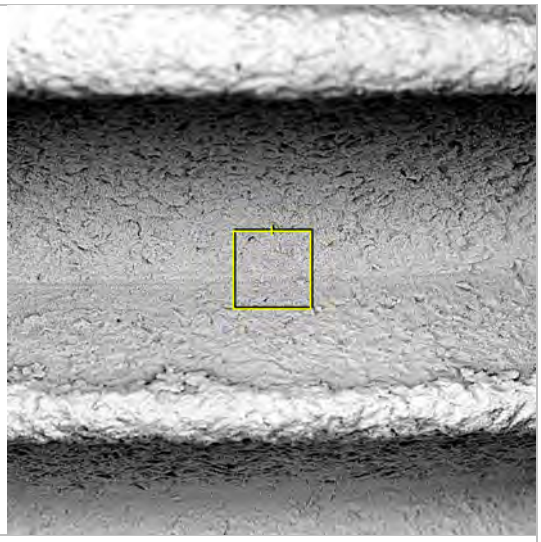
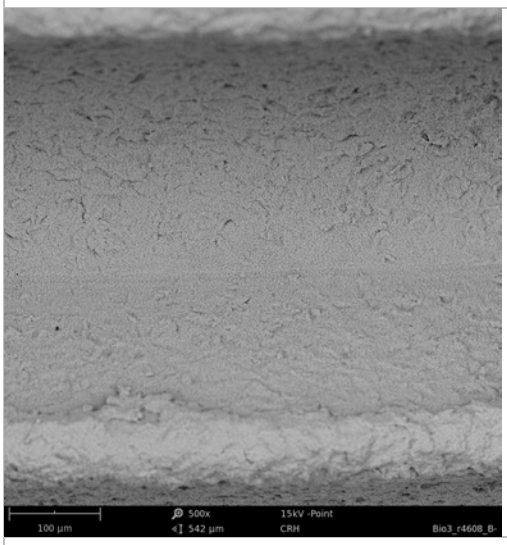
3.3 Surface-Topography - Material Contrast Images (Body)

Phenom camera



Field of view



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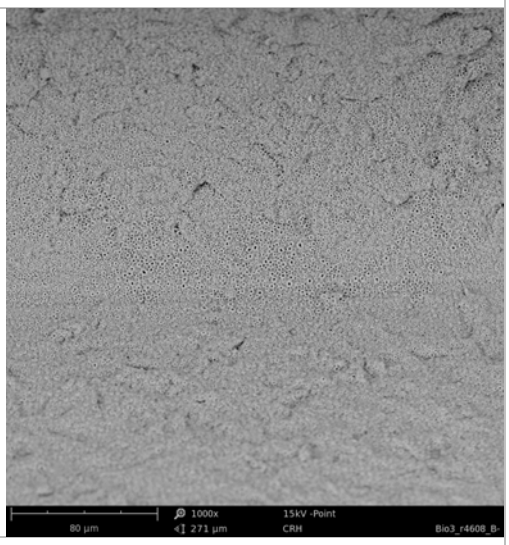

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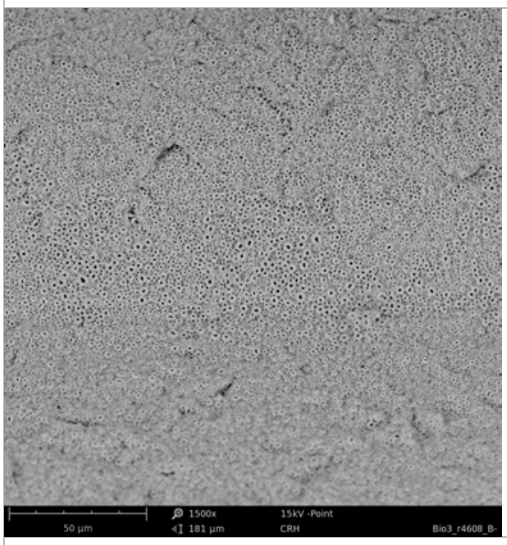

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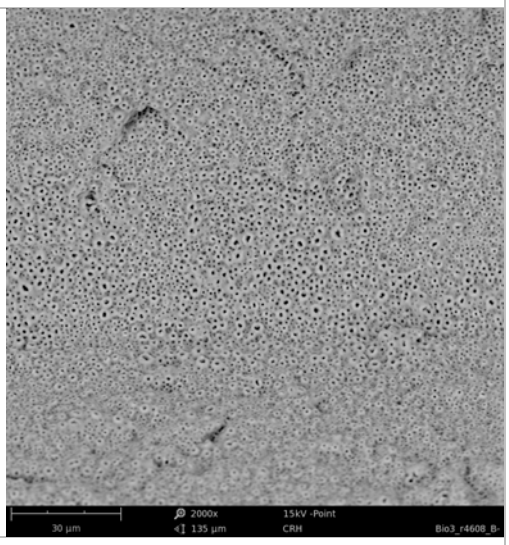

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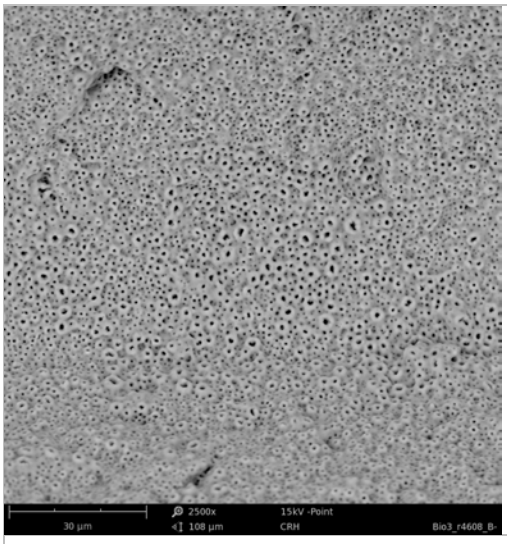
Surface-Topography - Material Contrast Images (Body)



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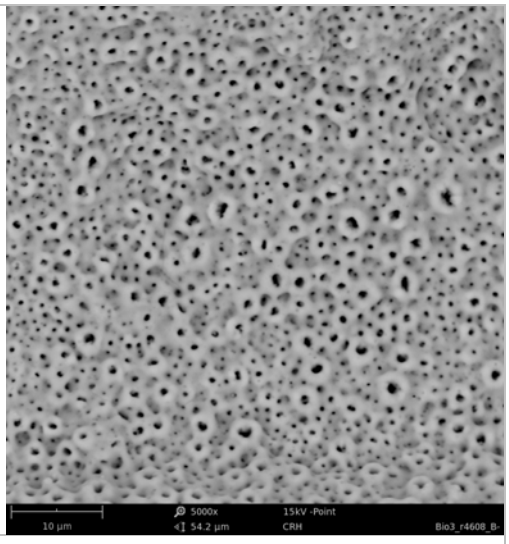

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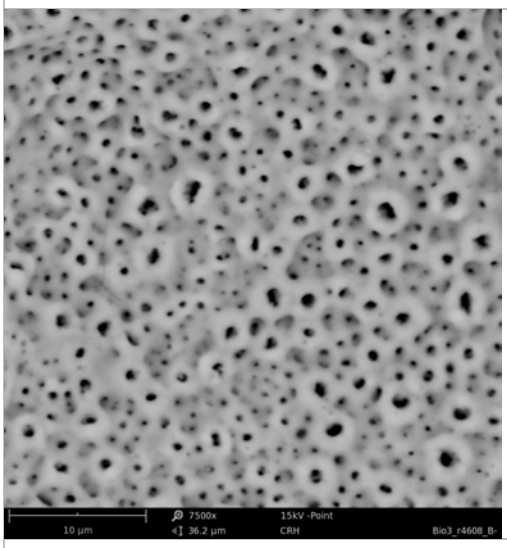

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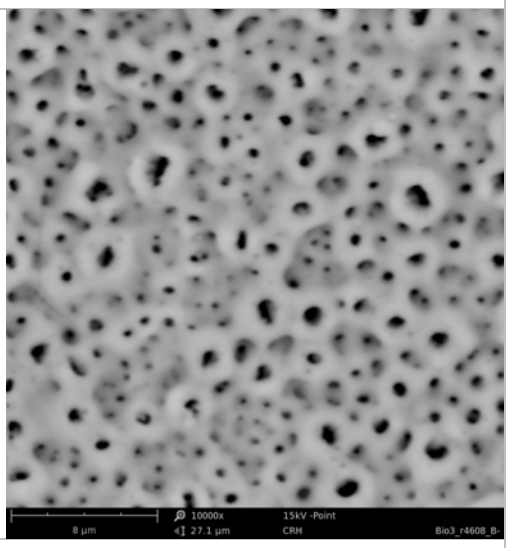

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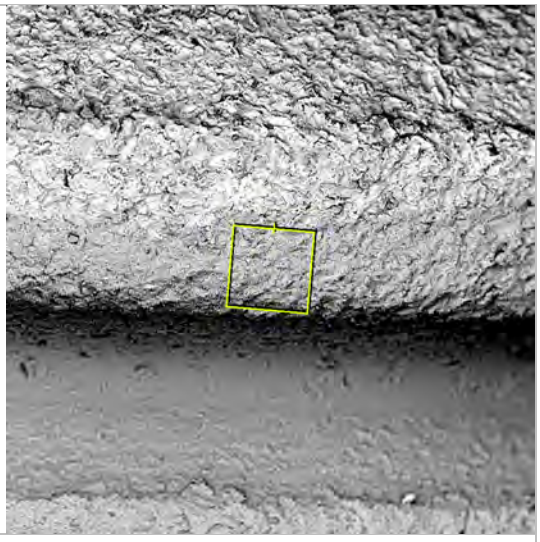
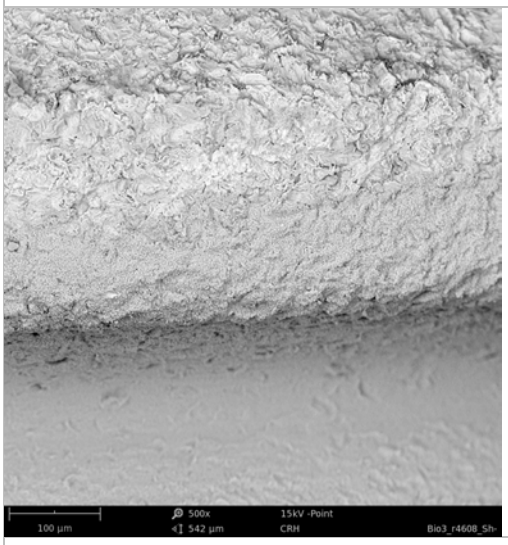
3.4 Surface-Topography - Material Contrast Images (Shoulder)

Phenom camera



Field of view



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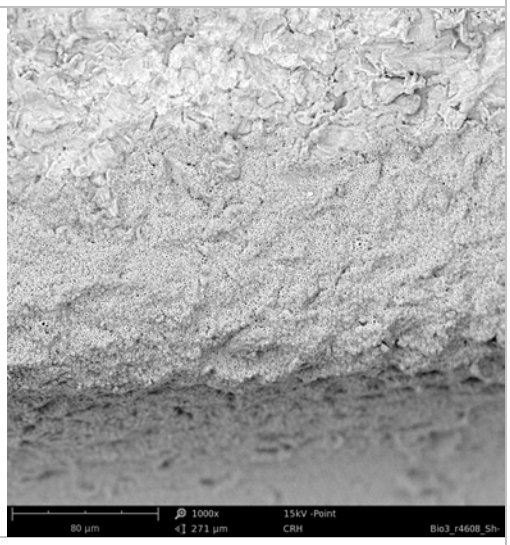

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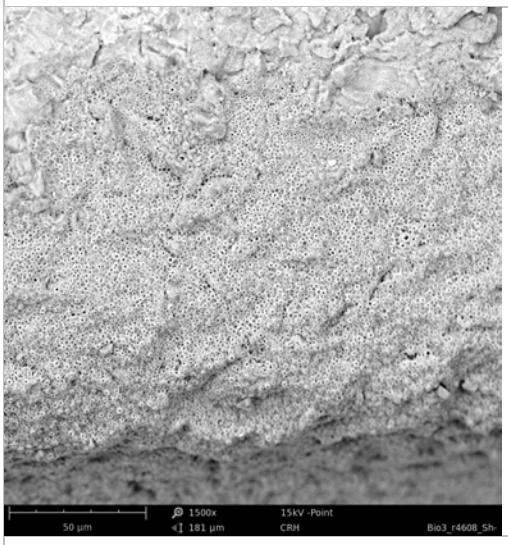

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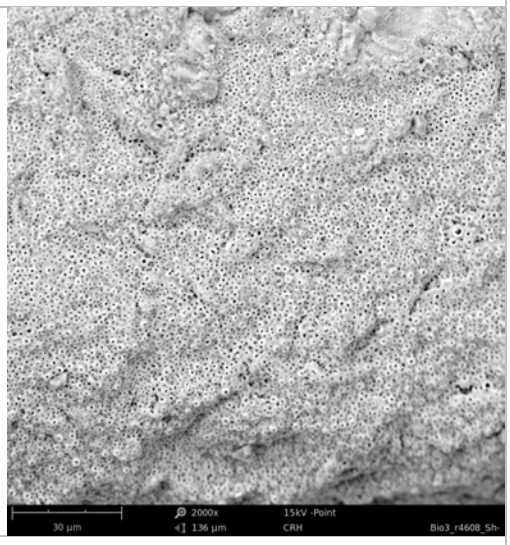

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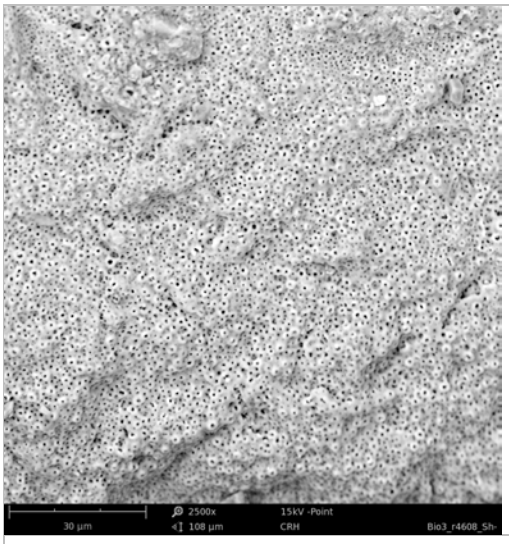
Surface-Topography - Material Contrast Images (Shoulder)



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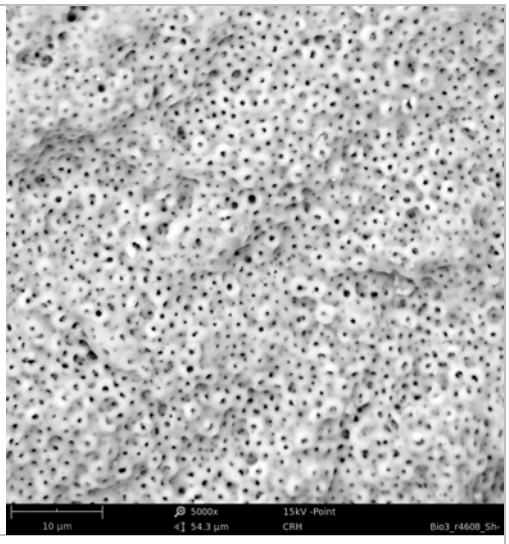

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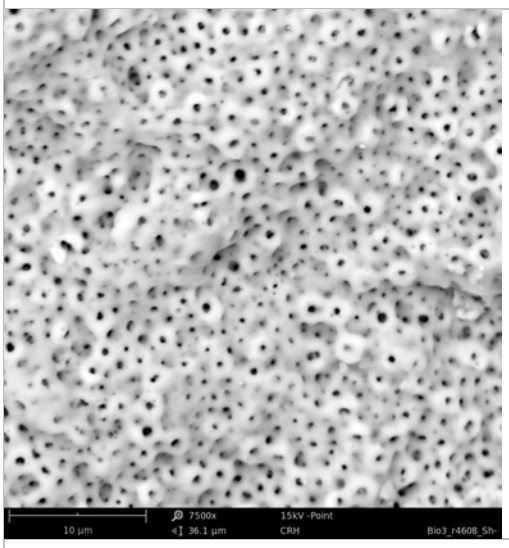

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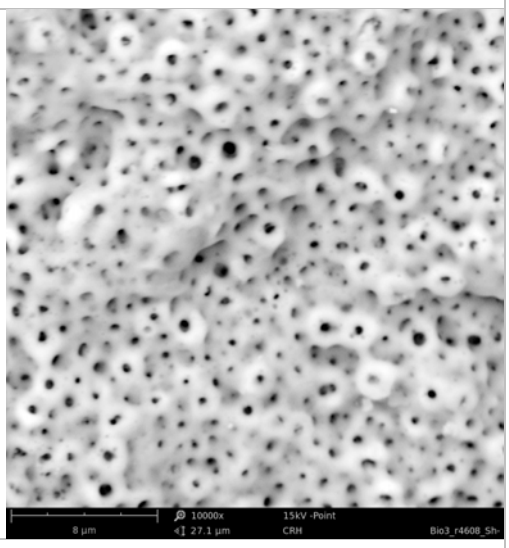

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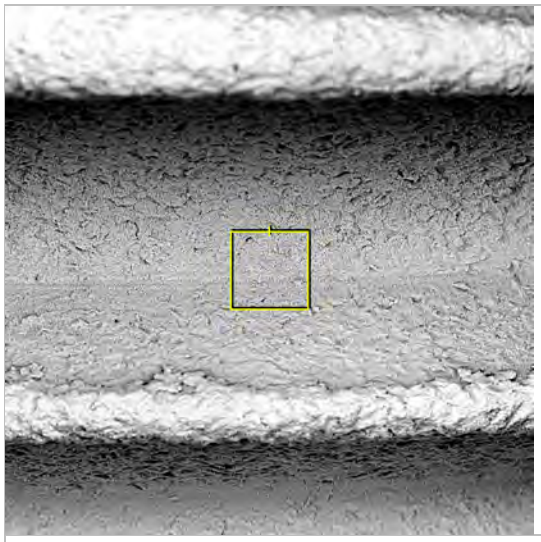
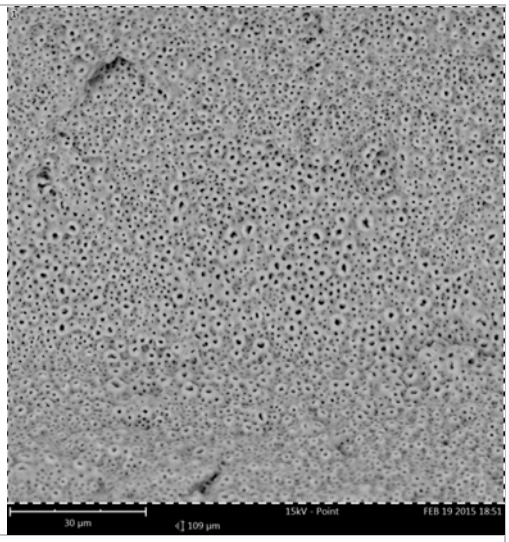
3.6 EDX Area Analysis



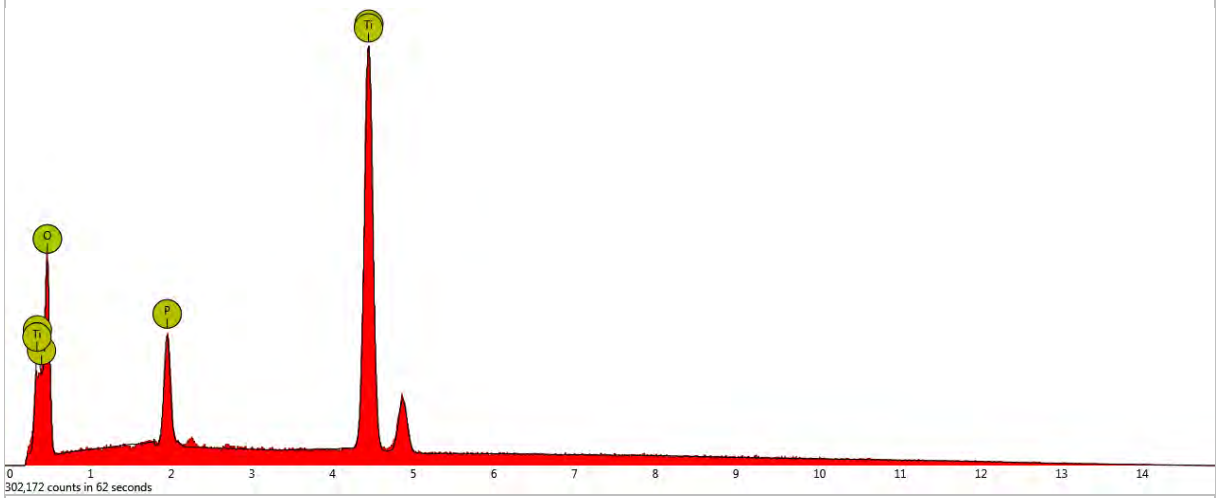
Field of view



Area

	Atomic percentage	Certainty
O	76.6 %	0.99
Ti	20.1 %	1.00
P	3.4 %	0.99

Quantitative elemental analysis



Qualitative elemental analysis

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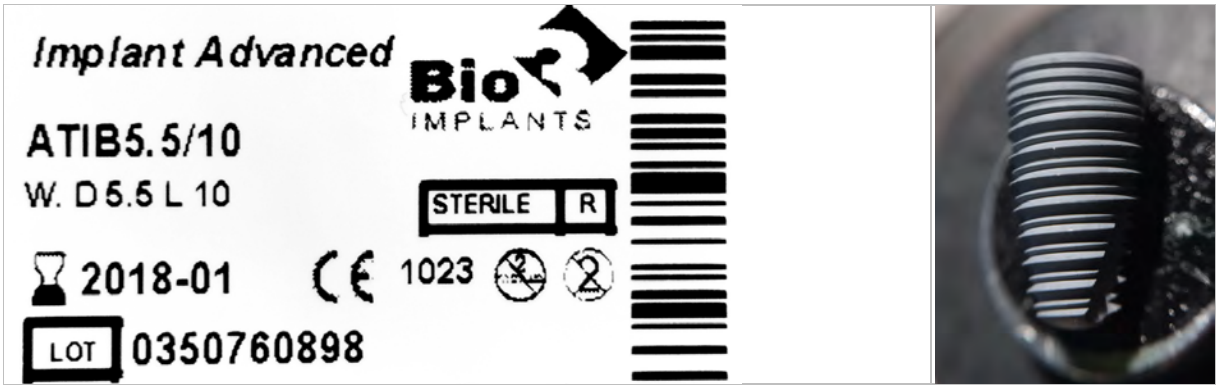
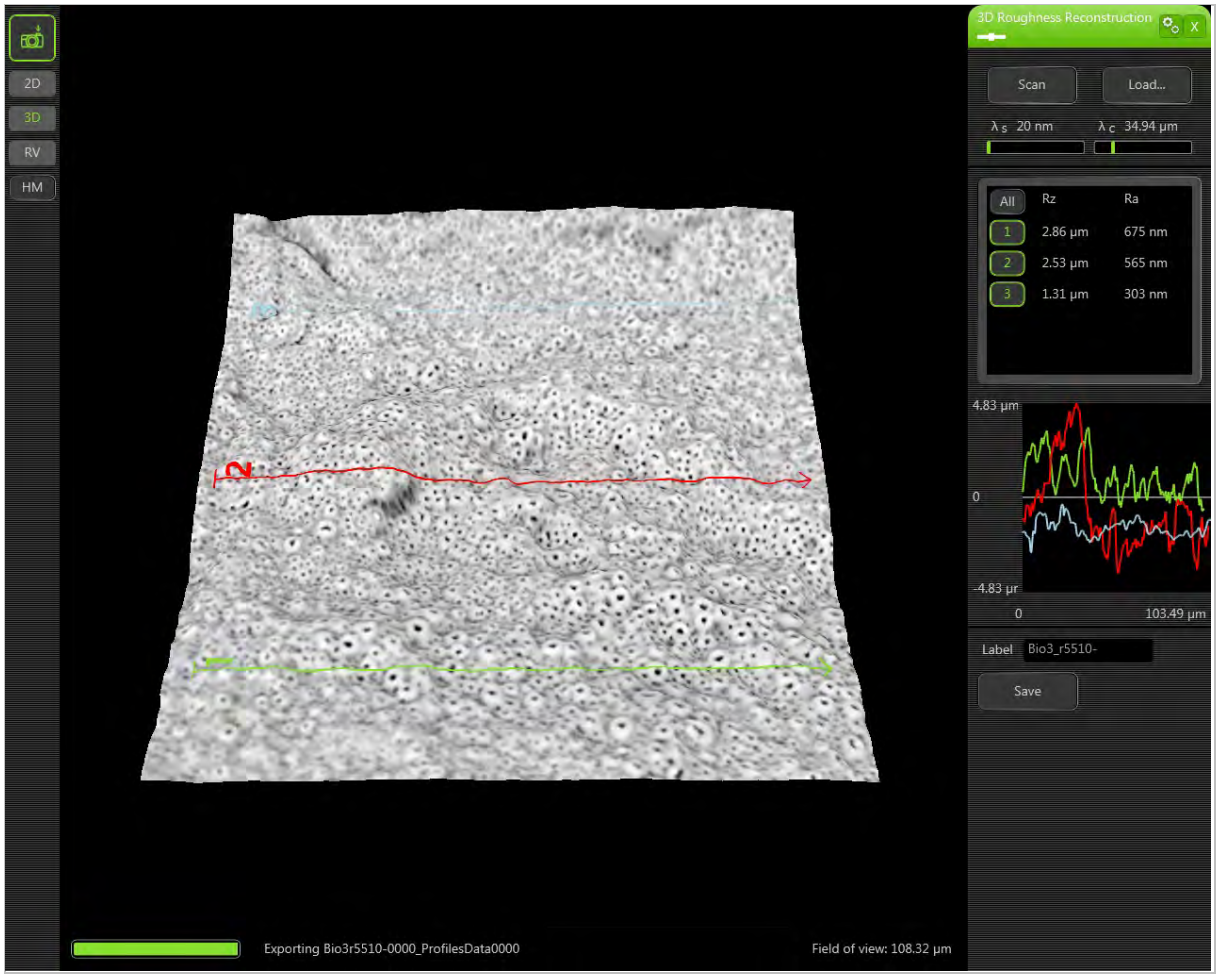
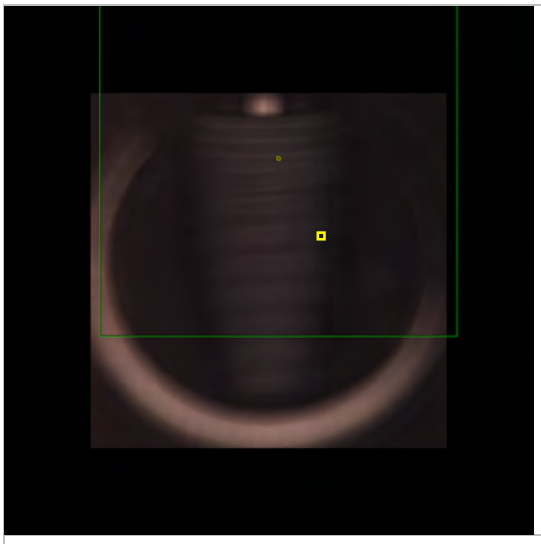


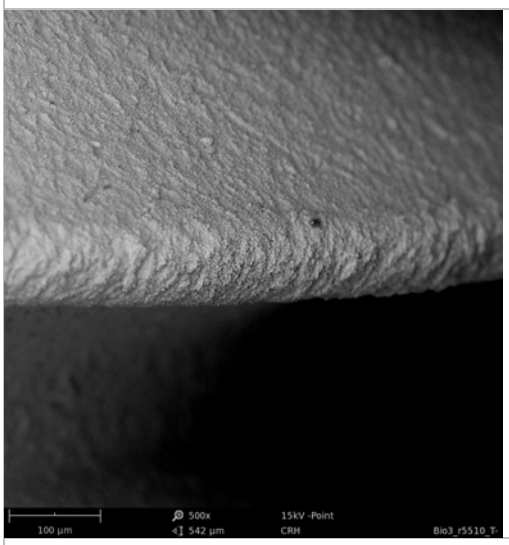
4.1 3D Roughness Reconstruction



4.2 Surface-Topography - Material Contrast Images (Thread)



Phenom camera



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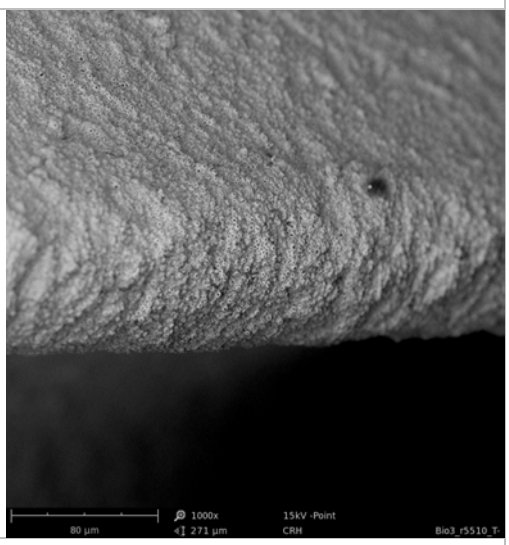

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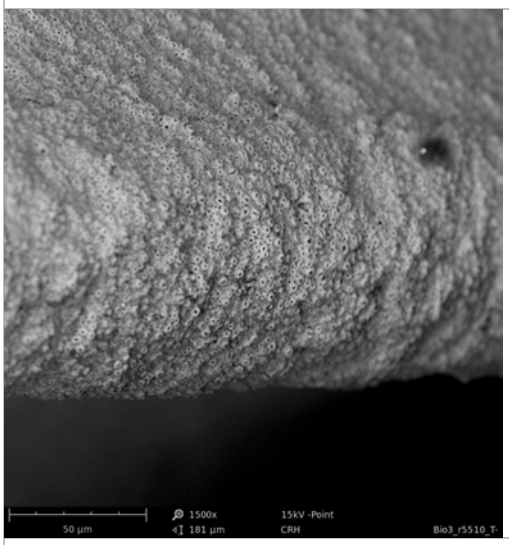

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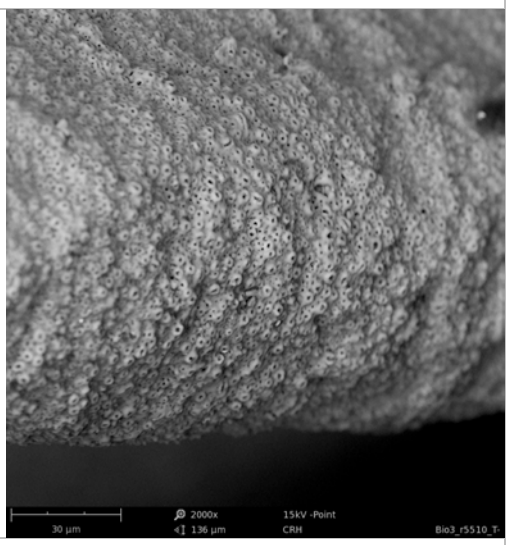

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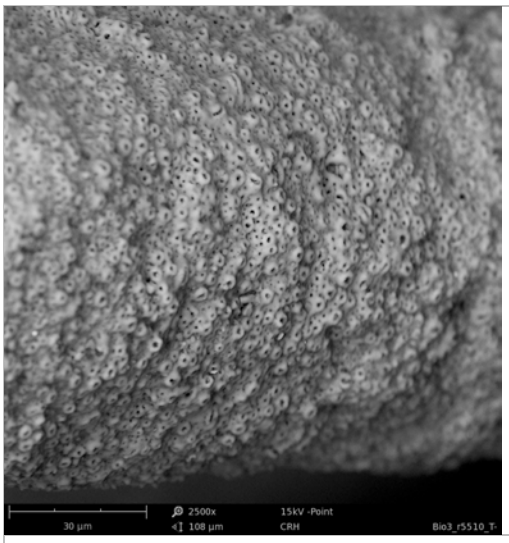
Surface-Topography - Material Contrast Images (Thread)



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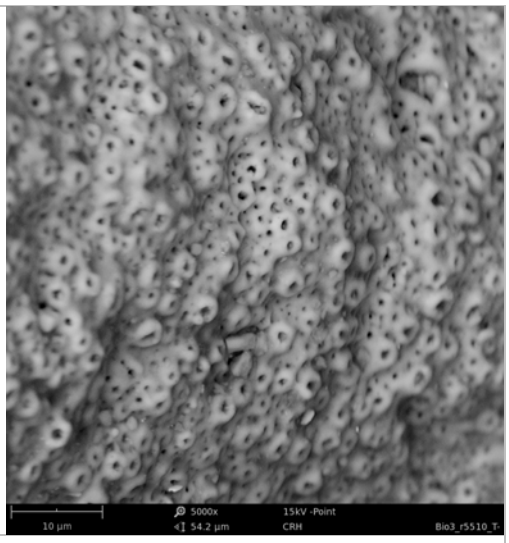

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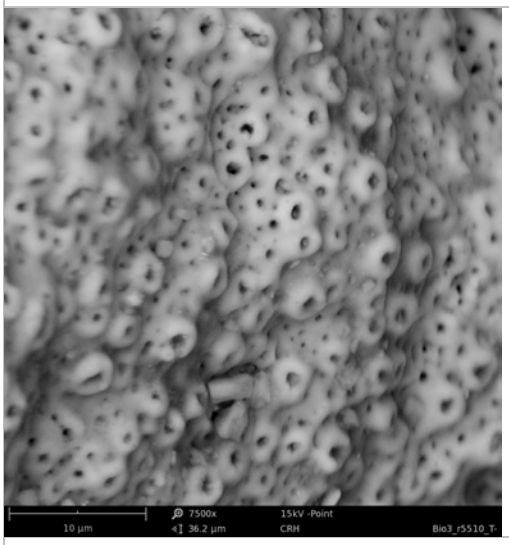

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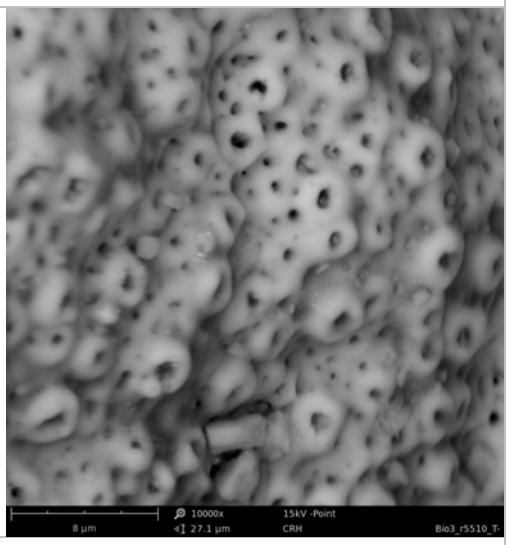

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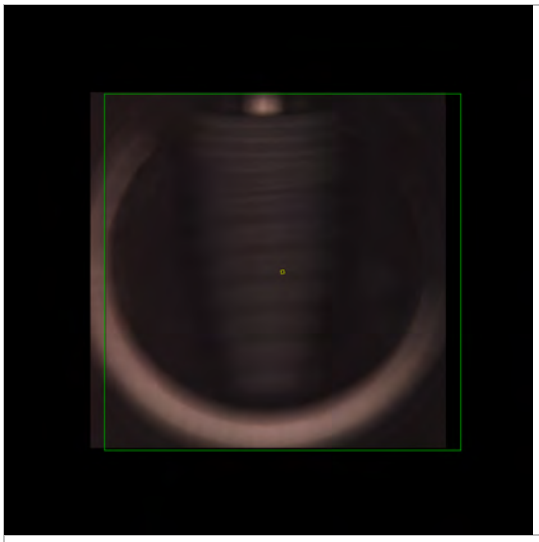
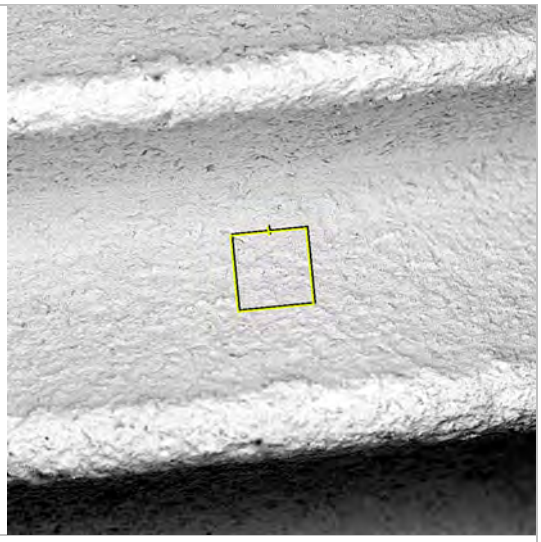
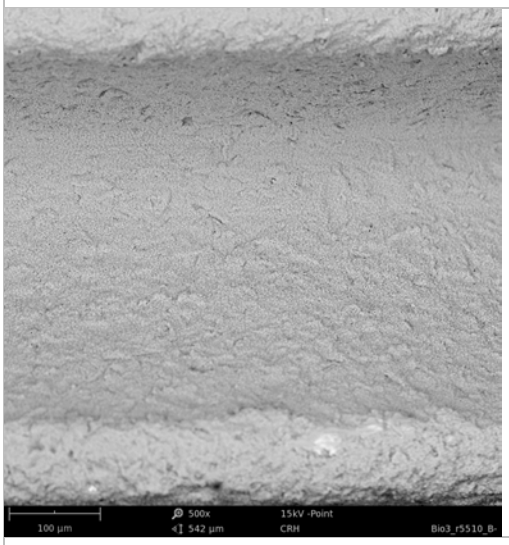
4.3 Surface-Topography - Material Contrast Images (Body)



Phenom camera



Field of view



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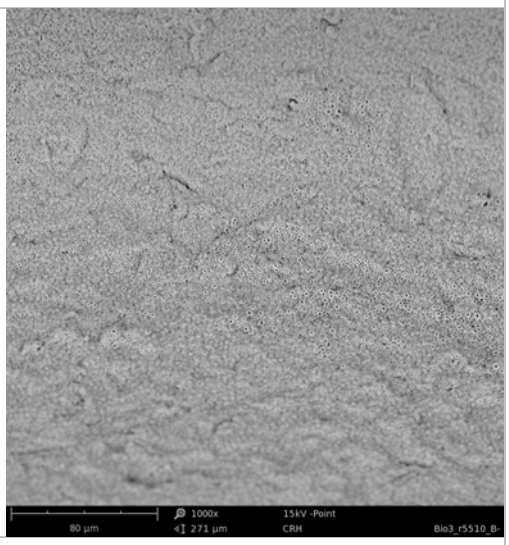

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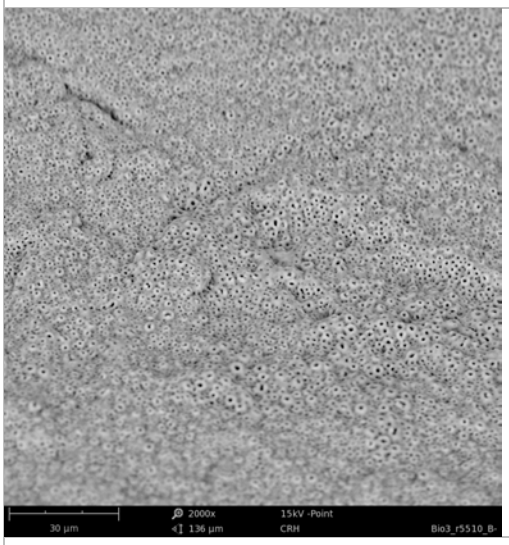

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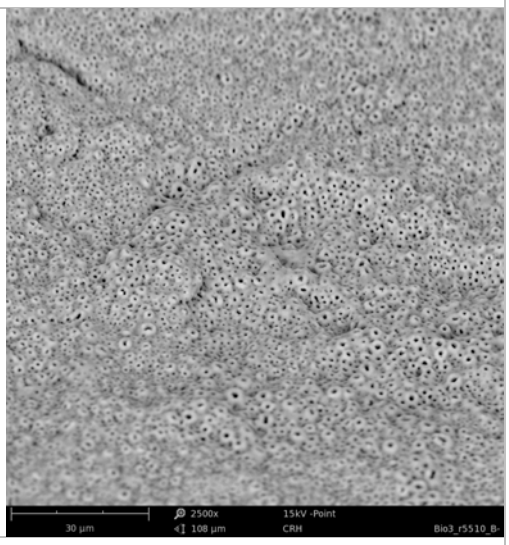

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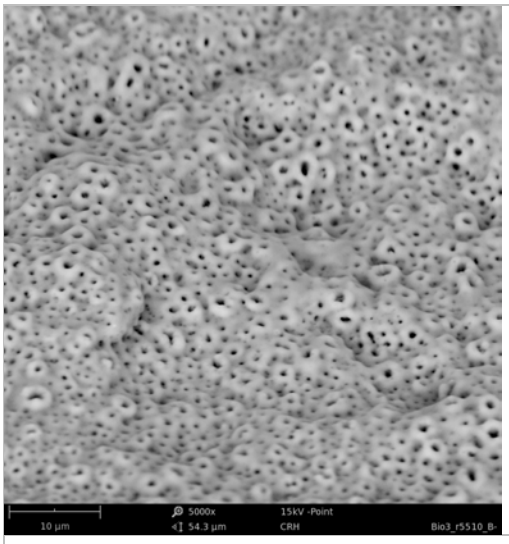
Surface-Topography - Material Contrast Images (Body)



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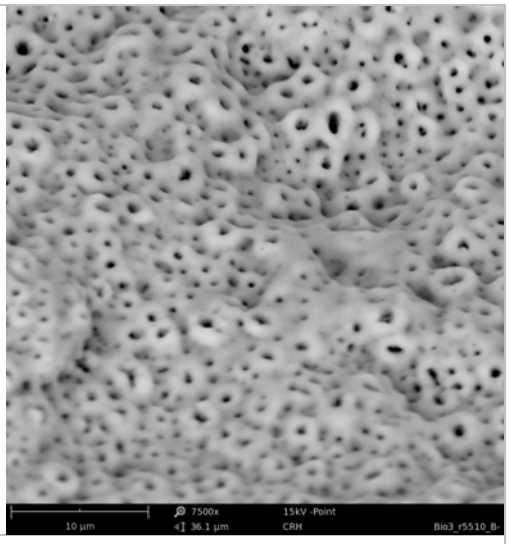

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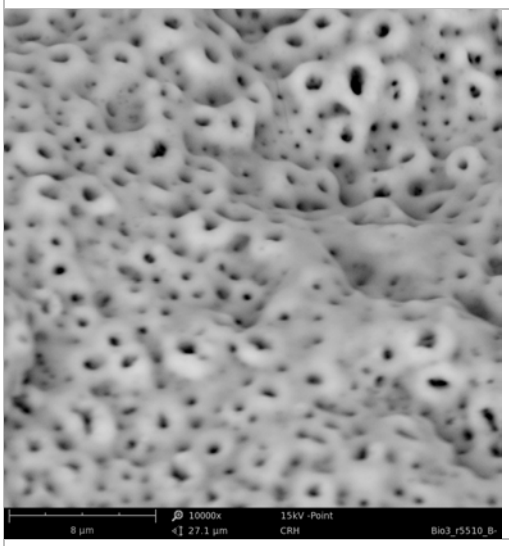

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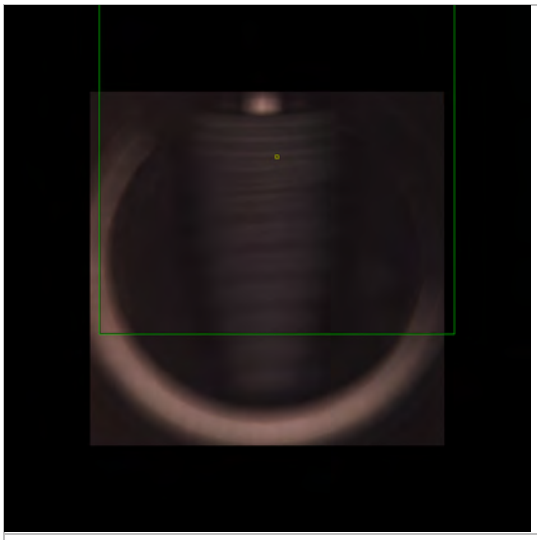
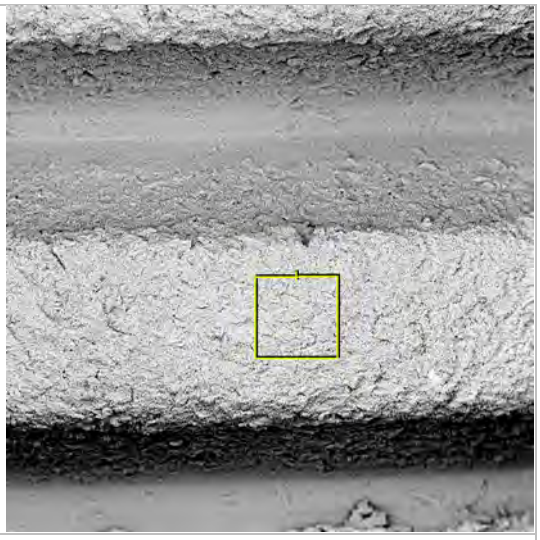
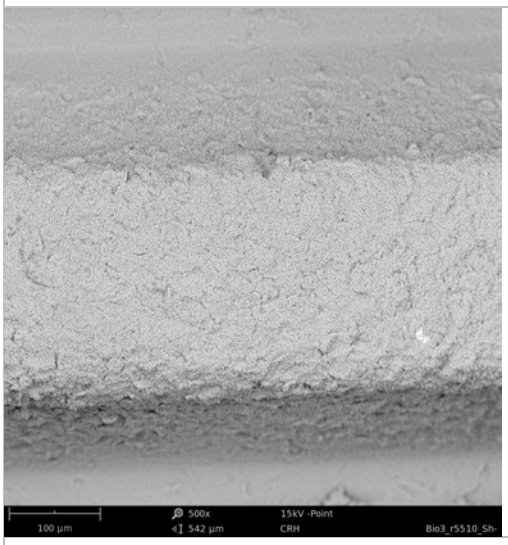
4.4 Surface-Topography - Material Contrast Images (Shoulder)



Phenom camera



Field of view



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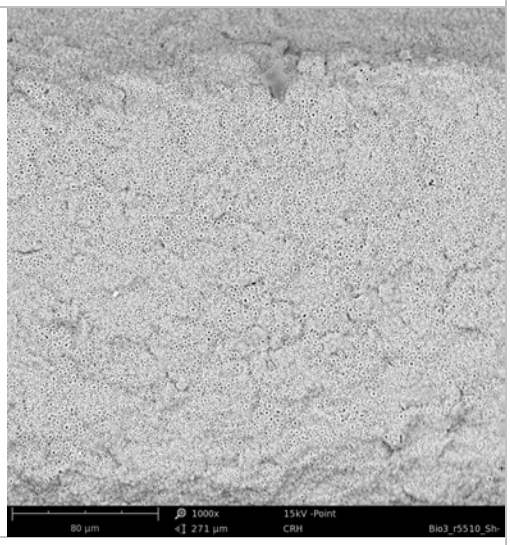

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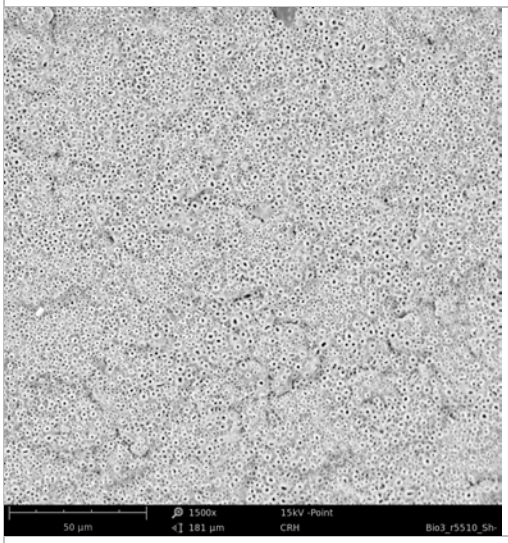

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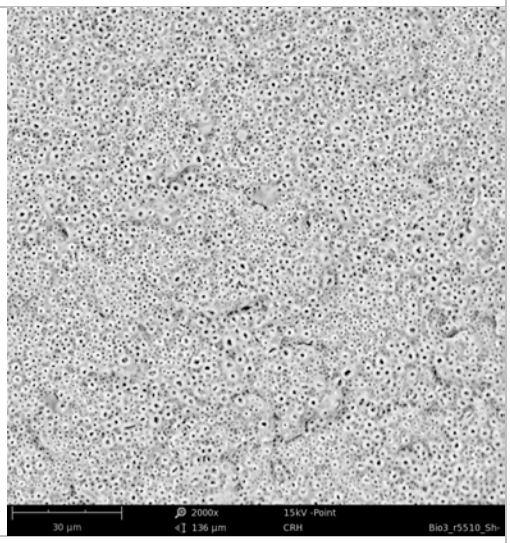

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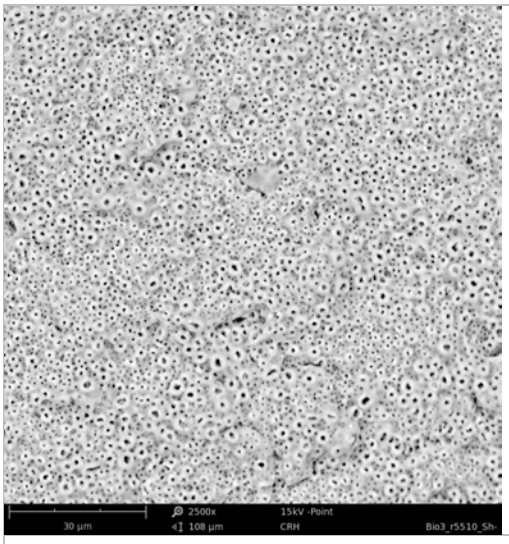
Surface-Topography - Material Contrast Images (Shoulder)



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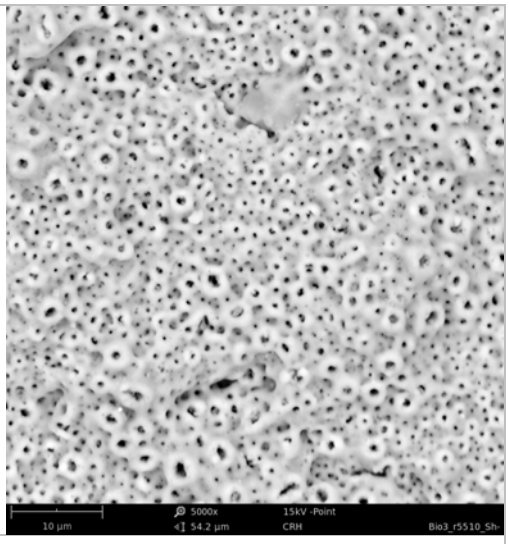

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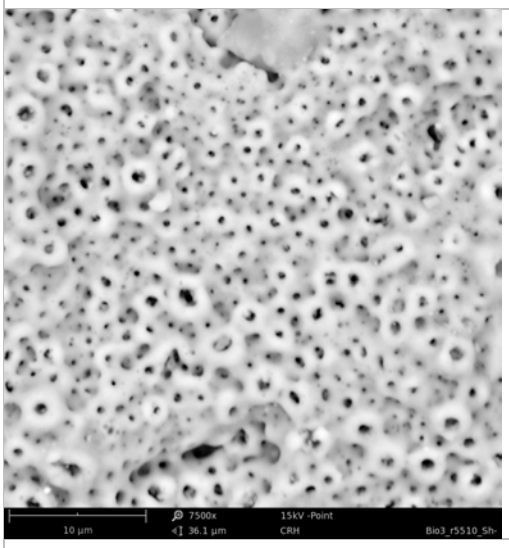

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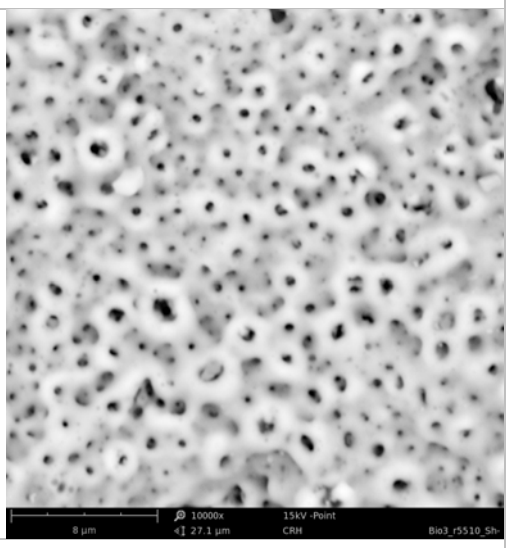

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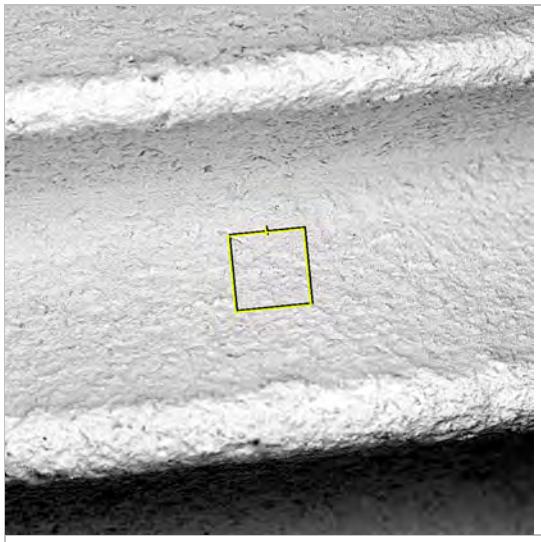
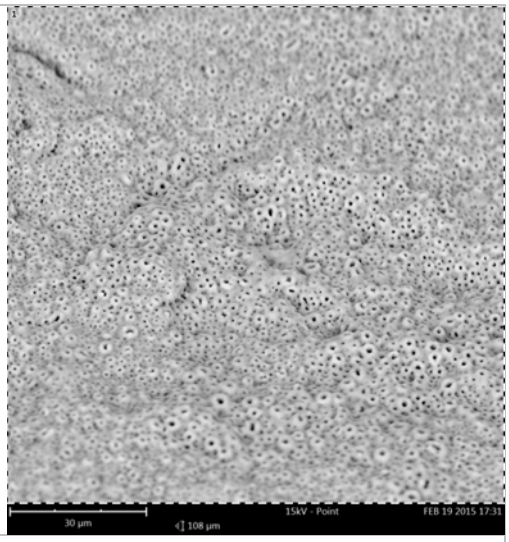
4.6 EDX Area Analysis



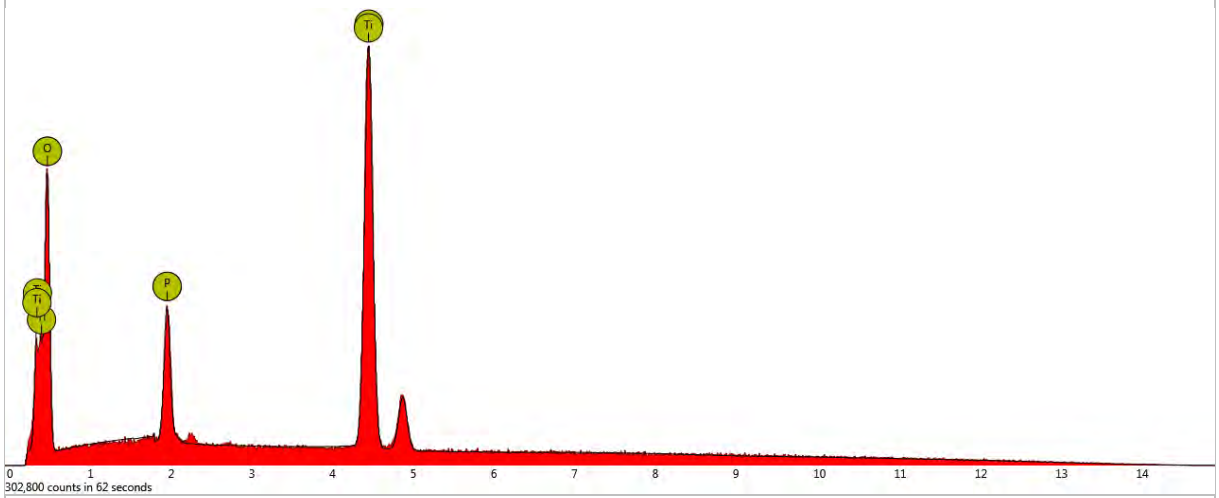
Field of view



Area

	Atomic percentage	Certainty
O	80.0 %	0.99
Ti	16.6 %	1.00
P	3.4 %	0.99

Quantitative elemental analysis



Qualitative elemental analysis

5 Synopsis

Name of Manufacturer:	**Bio3 Implants GmbH**
Analyzed Product(s):	Implant Advanced LOT 0350760902 Exp. Date: 2018-01 Implant Advanced LOT 0350760898 Exp. Date: 2018-01
Title of Study:	Implant-Study 2014/2015 Quantitative and qualitative element-analysis of implant-surfaces by SEM and EDX
Investigators:	Dr. Dirk U. Duddeck
Study centre:	Interdisciplinary Outpatient Dep. For Oral Surgery and Implantology, Dep. for Craniomaxillofacial and Plastic Surgery, University of Cologne
Study carried out by:	dedeMED - Medical Materials Research Institute Berlin
Studied period:	September 2014 – March 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_3N_4) window allowing detection of elements C to Am Energy resolution Mn Kα ≤ 140 eV Max. Input count rate: 300,000 cps
Summary/Conclusions:	The implant “Implant Advanced” provided by Bio3 for this analysis showed a high precision of the outer geometry. Significant traces of inorganic or organic residues were not found.

6 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. Duddeck

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,
eMail: duddeck@dedemed.de

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. Duddeck

DATE: Jan. 11, 2015