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Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB

Parts2clean, Stuttgart, 28.09.2023 – Dr. Jakob Barz

Surface analysis with XPS, SEM, and Raman for cleaning processes and quality control

Methods and examples

Fraunhofer in Stuttgart

5 Fraunhofer institutes at campus Stuttgart

- Fraunhofer Institute for Industrial Engineering IAO
- Fraunhofer Institute for Building Physics IBP
- **Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB**
- Fraunhofer Institute for Manufacturing Engineering and Automation IPA
- Fraunhofer Information Center for Planning and Building IRB

Figures and facts

1,800
employees

65,000 m²
infrastructure area

€117.4 million
operational budget

Status: 2021

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Range of services

We conduct research on your behalf

Process, technology and product development

From laboratory to technical and pilot scale

Design and construction of demonstration plants and prototypes

Implementation of new technologies

Analysis and testing services

Physico-chemical analysis*

Surface and particle analysis*

GLP unit: Cell-based test systems (Category 9)

Microbiological testing

Consultancy and studies

Feasibility studies

Technology analyses

Profitability studies

* accredited testing field

Cooperation models – ways of working together

One-off contracts
industrial contract research

Joint research
projects with multiple partners

Strategic partnerships
national and international

Licensing
of technologies and processes

Technology transfer through to spin-offs

Training

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Chapter 01

Surface analysis at Fraunhofer IGB

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Instrumentation

Large devices

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X-ray photoelectron spectrometer

Scanning electron microscope

Raman microscope

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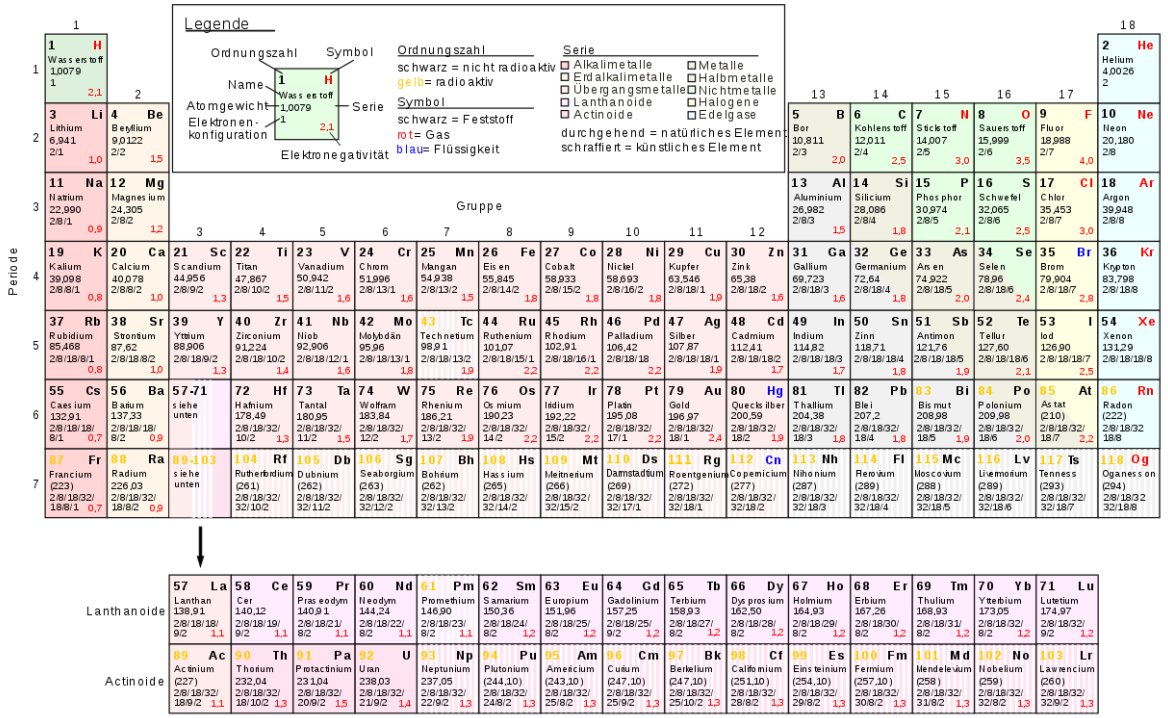
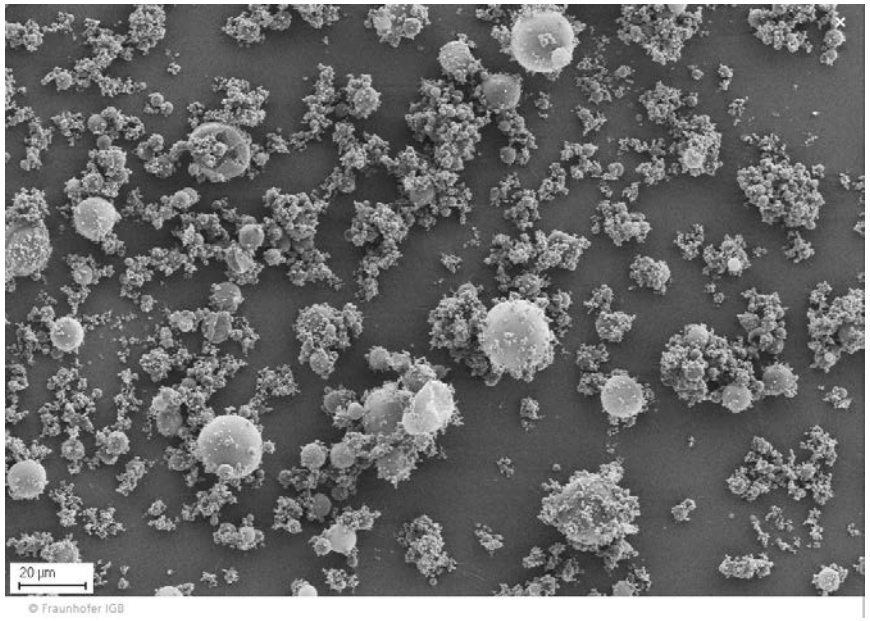
What is being analyzed





Joshua D.wondrousch, Mattlaabs - Eigenes Werk, basierend auf: File:Periodic table (German).svg, Gemeinfrei, https://commons.wikimedia.org/w/index.php?curid=19964114

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Chapter 02

XPS– x-ray photoelectron spectroscopy

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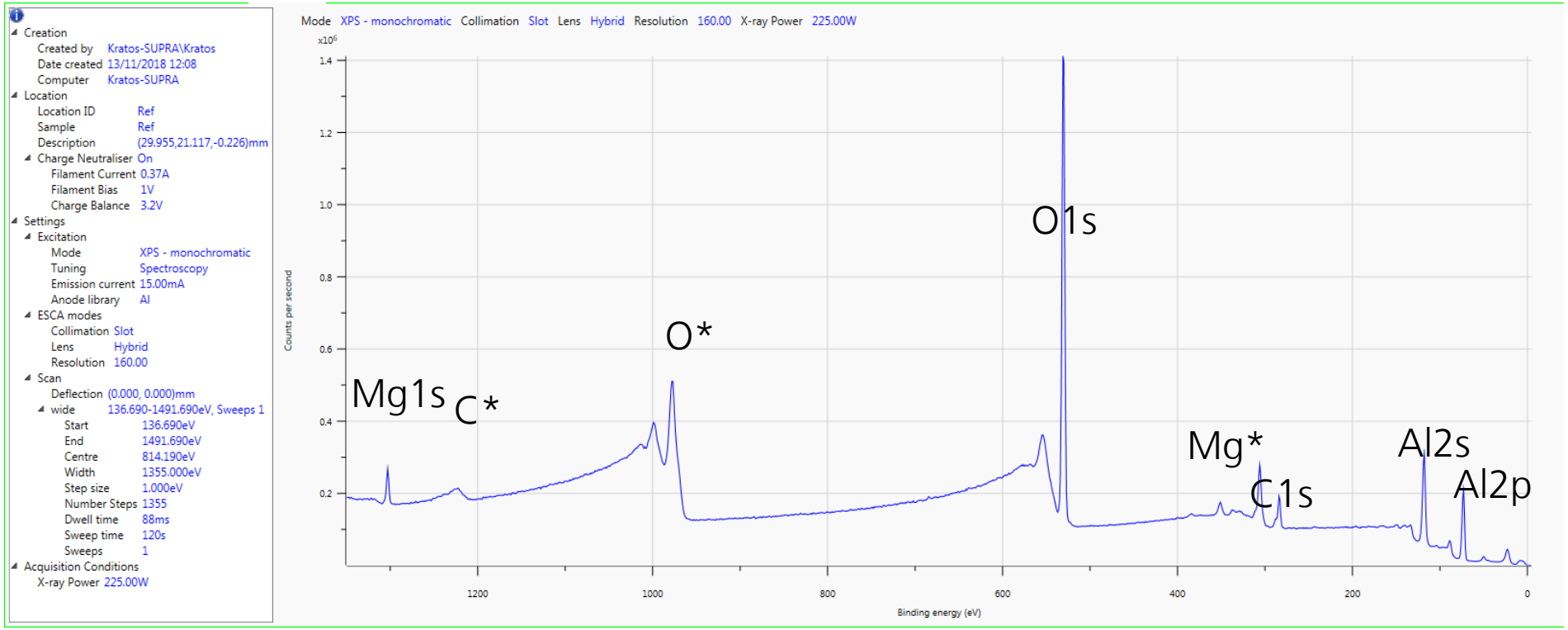
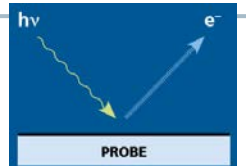
Aluminum surface

“clean” surface





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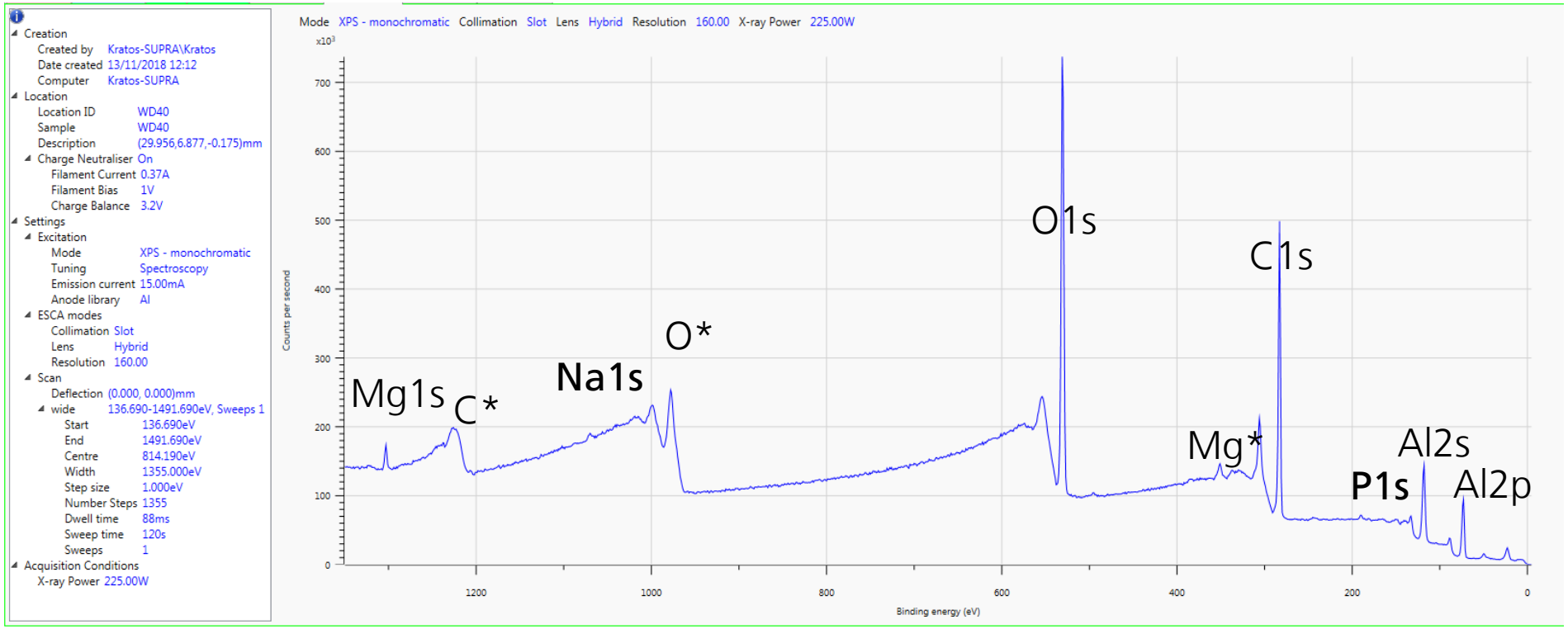
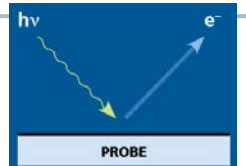
Aluminum

WD40





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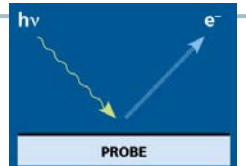
Aluminum

WD40

Element E_B (eV)	C ges	O2 532,2 - 534,3	O1 530,8	Al ox	Al met	Mg 50,6	Si	Na	P
	Elementzusammensetzung in Atom%								
S135 Referenz Aluminium matte Seite	11,7	-	53,4	21,0	11,6	2,5	-	-	
S136 Aluminium mit WD40	64,9	-	22,1	7,4	4,1	1,0	-	0,1	0,4
S136 Aluminium mit WD40 Tropfenbereich	78,7	-	13,1	4,6	2,2	0,6	-	0,2	0,4

- With increasing thickness of the contamination, the substrate signal is reduced. The signal of the contamination increases.
- The oxygen signal contains both contributions of the metal oxide and carbon-oxygen compounds.



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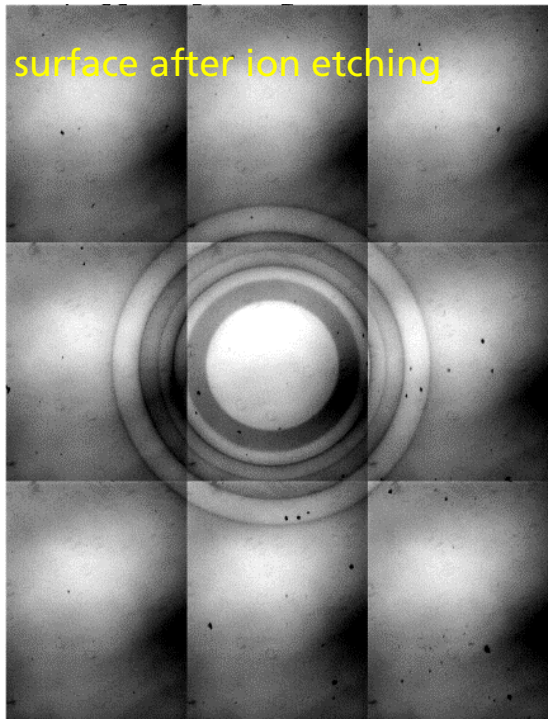
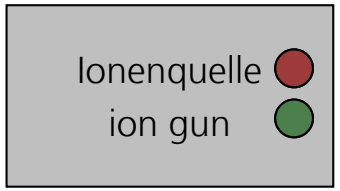
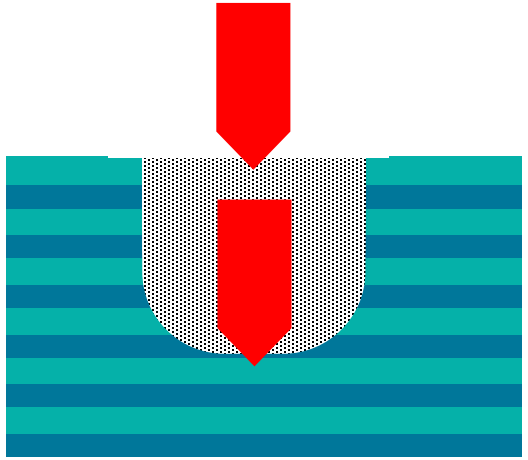
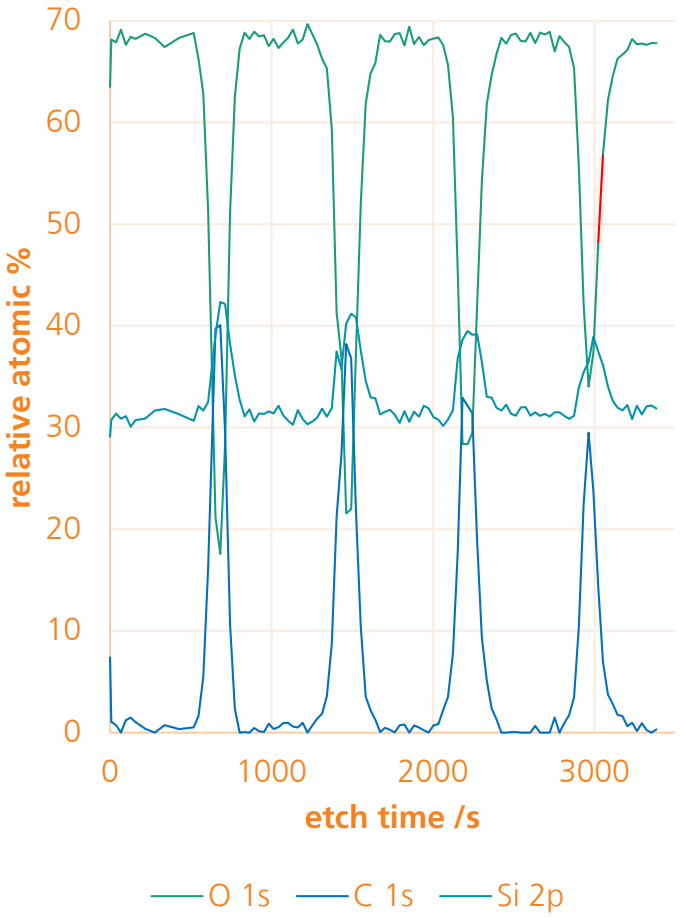
XPS: Depth profiling

Profiling with ion beam: Ar^+, Ar_n^+ (n = 500..3000)









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Chapter 03

Scanning electron microscopy (SEM)

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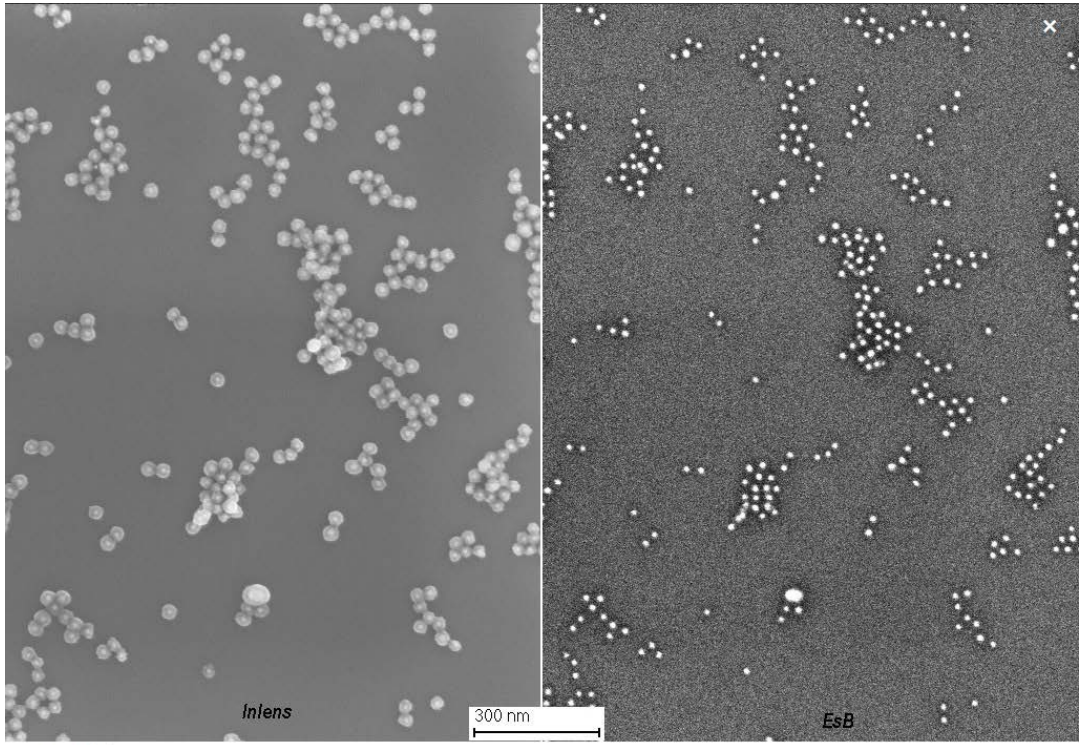
SEM imaging

Core-shell particles



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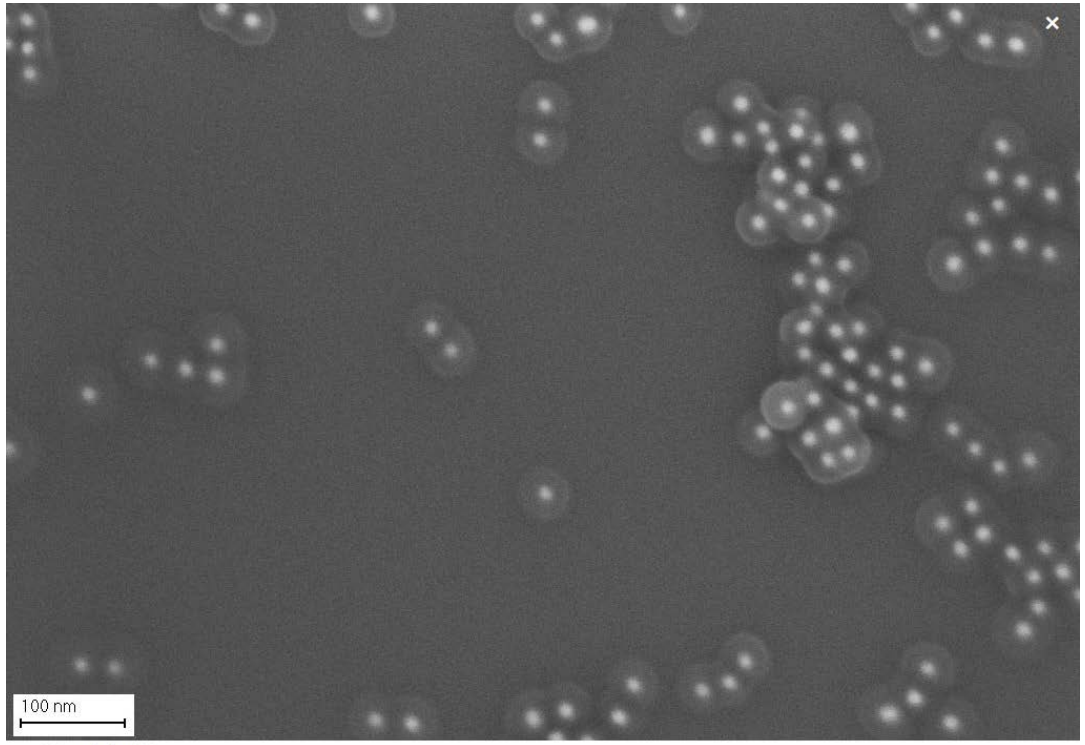
Linke Bildhälfte: InLens SE1-Signal. | Rechte Bildhälfte: EsB-Detektor, niederenergetische rückgestreute Elektronen (BSE).



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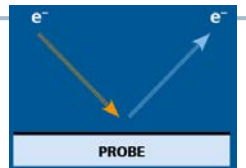
Mixed Signalerfassung, Abbildung mit SE1 und BSE, die Grenze zwischen Hülle und Kern ist deutlich erkennbar.



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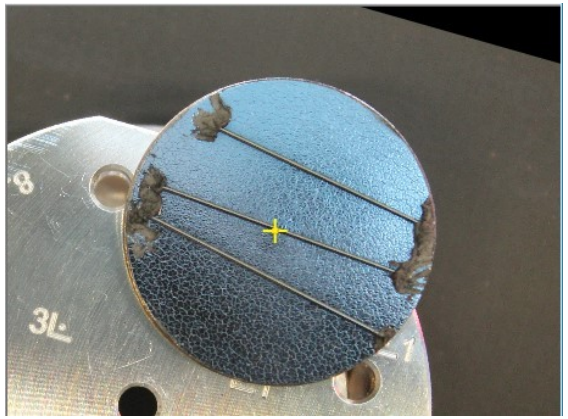
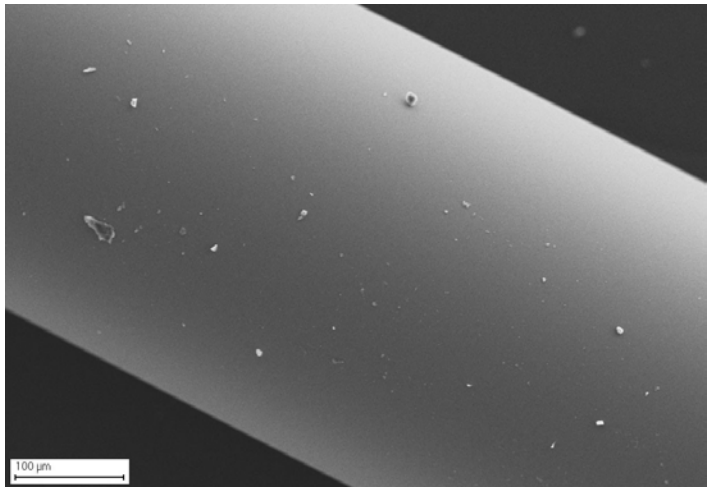
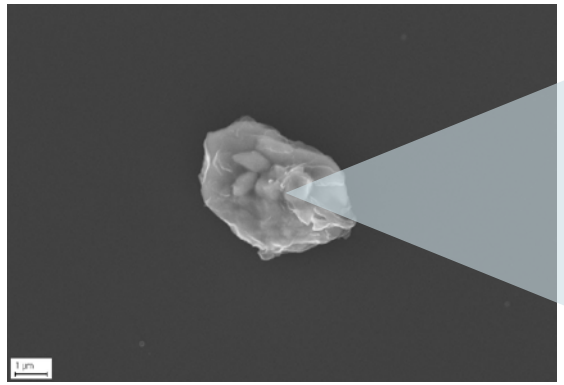
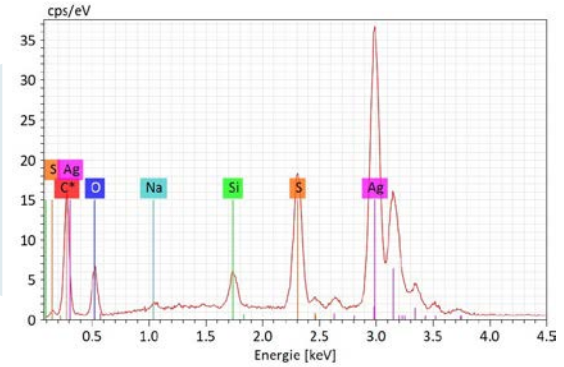
SEM-EDX

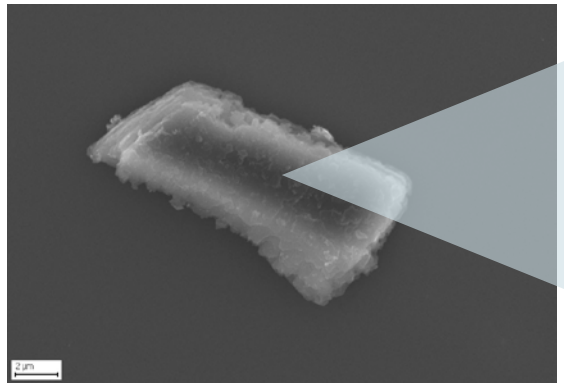
analysis of particles on a fiber

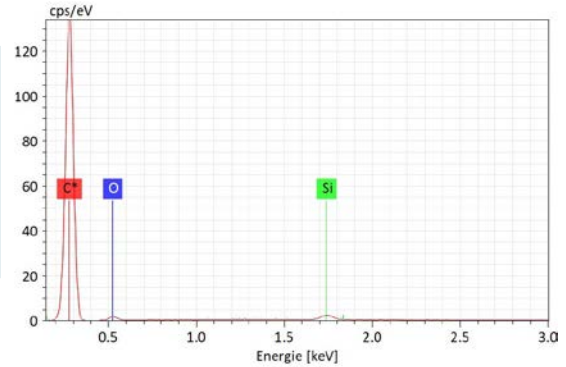


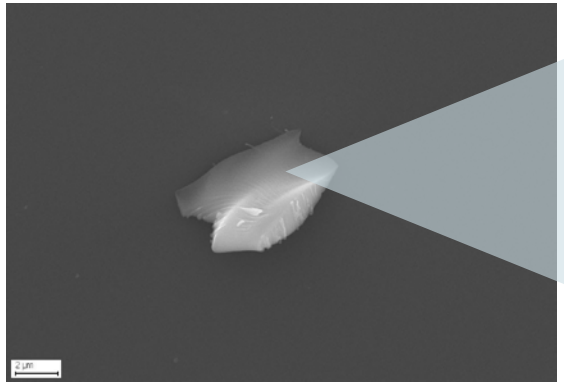


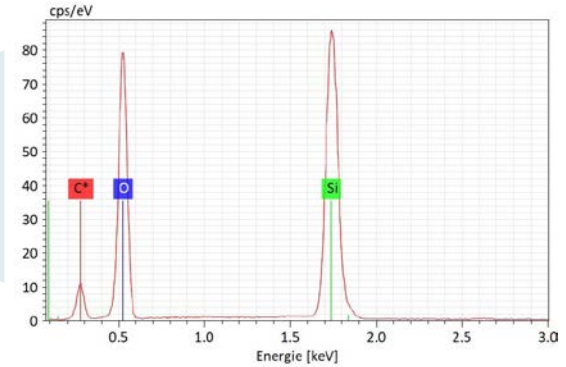


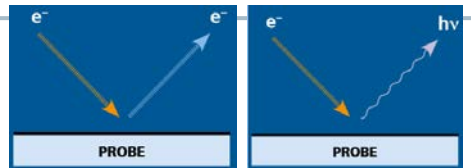












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Chapter 04

Confocal Raman microscopy

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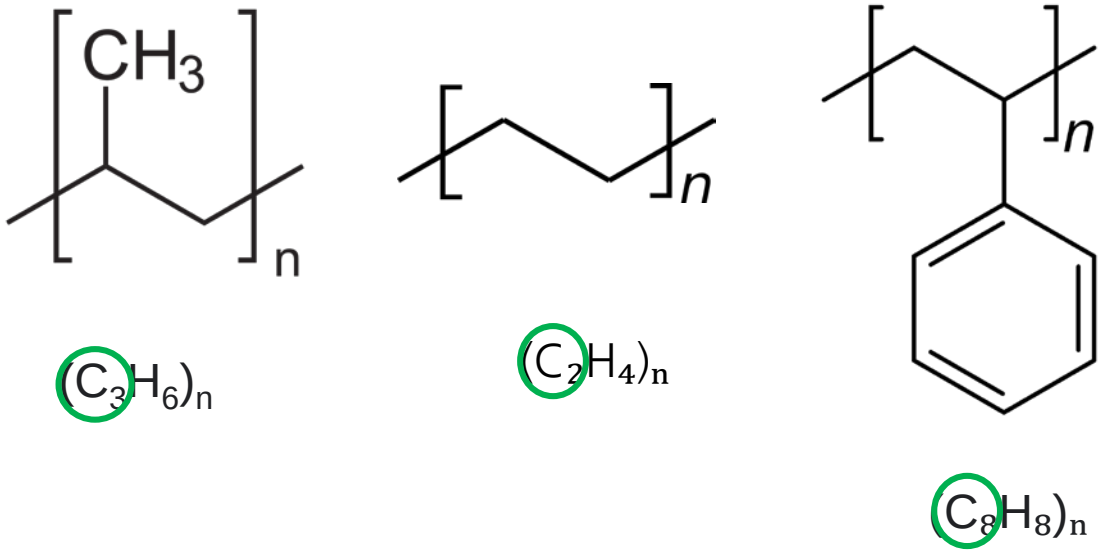
Raman spectroscopy

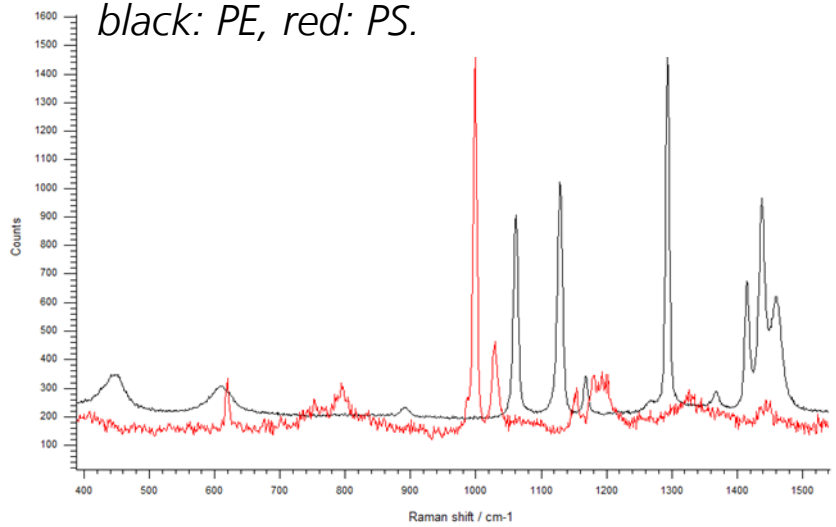
Complementary to XPS / SEM-EDX



$(C_3H_6)_n$ $(C_2H_4)_n$ $(C_8H_8)_n$

: only carbon is detectable by **SEM-EDX** and **XPS**

Raman spectra of micro plastic.
black: PE, red: PS.



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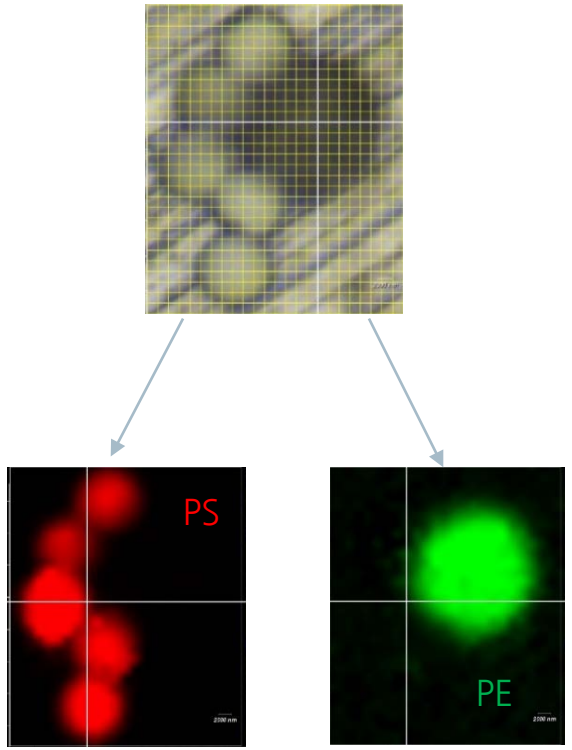
Surface analysis with confocal Raman microscopy



- Spectra are acquired on a predefined grid.
- Positioning is precisely done via an automated stage.
- Evaluation of each local spectrum is automatically carried out via
  - different algorithms,
  - PCA,
  - Comparison to reference spectra,
  - cluster analysis,
  - ….

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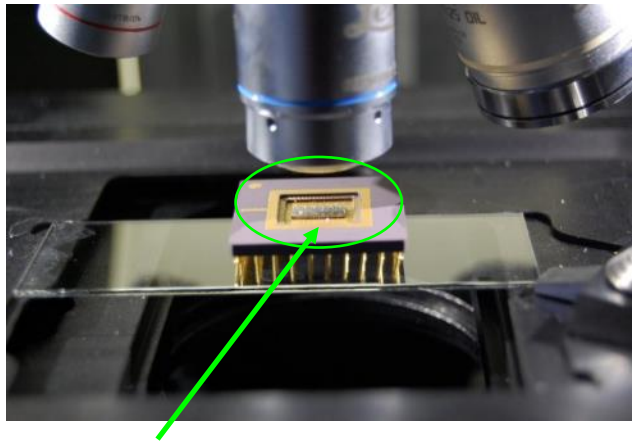
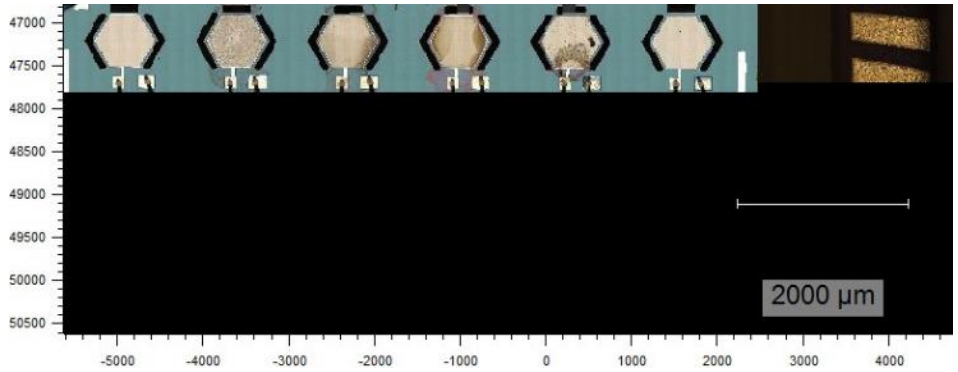
Raman analysis

Example

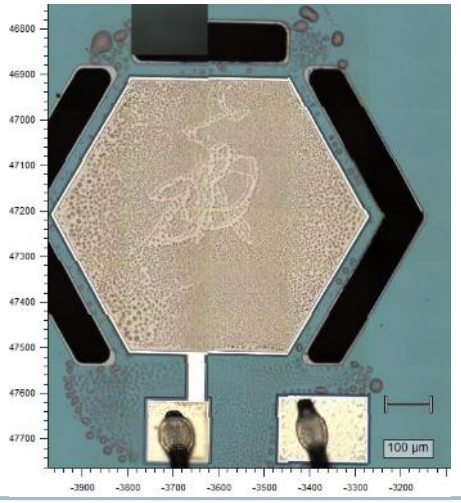


Challenge:
Surface of interest lies in a 2-3 mm cavity

=> Usage of LWD 50x objective with WD 8.2 mm



Assembled microscope images



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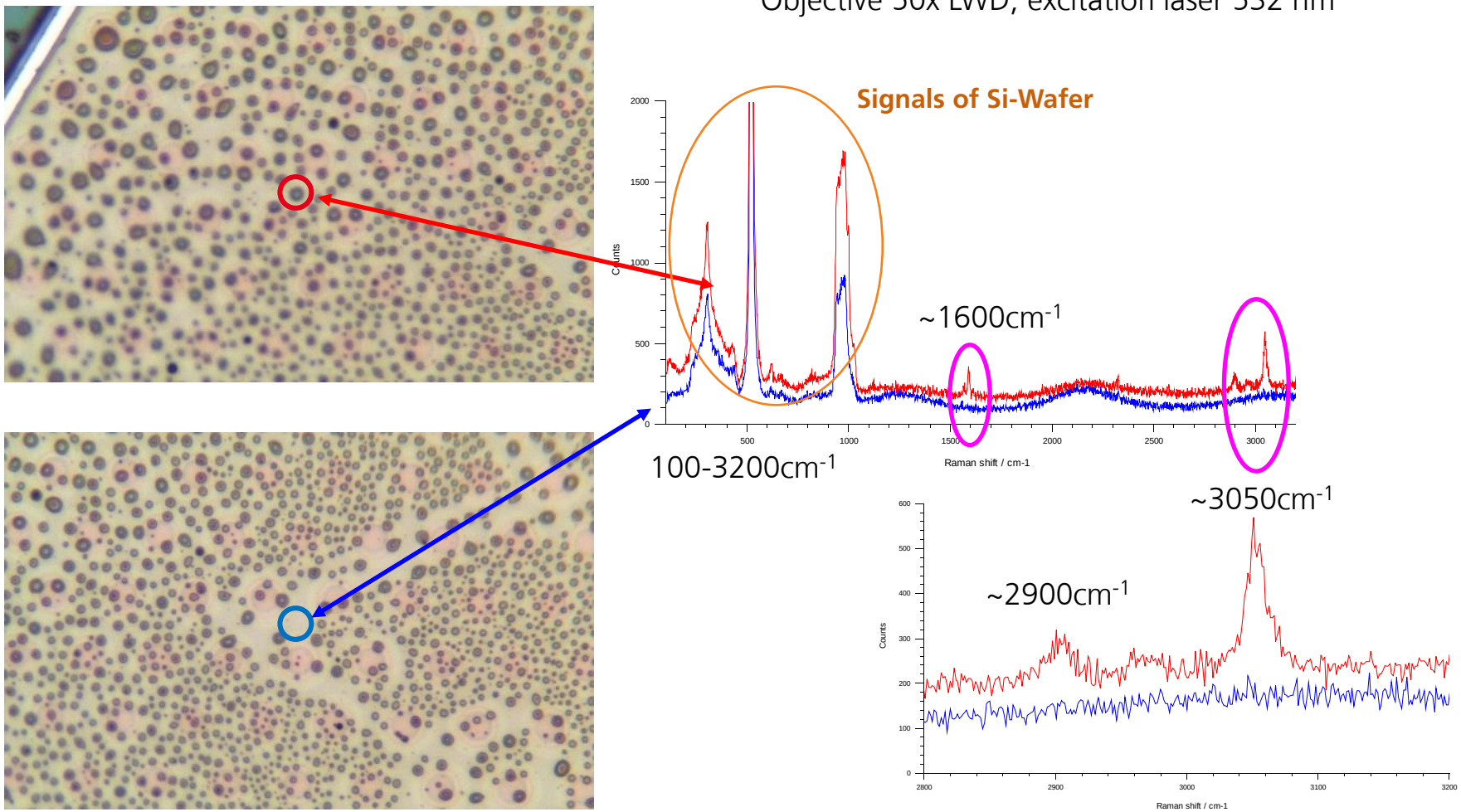
Raman analysis

Example

Objective 50x LWD, excitation laser 532 nm



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Chapter 05

Summary

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Summary / additional information

- By choosing the analytical method, the surface sensitivity of the chemical analysis can be selected. The information depth increases in the following order: Auger < XPS < EDX. In such a way, the range of few nanometers to some microns is covered.
- Auger, XPS and EDX allow for element mapping the surface. In addition, XPS allows to analyze the chemical bonds in more detail, whereas Auger and EDX offer a much higher lateral resolution.
- SEM yields additional topographical information of the surface and allows to visualize changes in conductivity and other material contrasts. In such a way, it may support the analyst to identify interesting regions for a subsequent analysis with other analytical methods.
- Confocal Raman microscopy is a complementary method. Raman spectroscopy allows to identify materials via their characteristic spectra, for example polymer particles with sizes starting already below 1 µm.

Oberflächenanalytik / Surface Analytics:
https://www.igb.fraunhofer.de/oberflaechenanalytik
https://www.igb.fraunhofer.de/surface-analytics

Für die Methoden XPS und ARXPS sind wir akkreditiert:

DAkkS Deutsche Akkreditierungsstelle D-PL-11140-23-00

Durch die DAkkS nach DIN EN ISO/IEC 17025:2018 akkreditierter Prüfbereich. Die Akkreditierung gilt nur für den in der Urkundenanlage D-PL-11140-23-00 aufgeführten Akkreditierungsumfang.

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Contact

Dr. Jakob Barz
Study Director Surface Analytics
Innovation Field Functional Surfaces and Materials
Phone +49 711 970-4114
jakob.barz@igb.fraunhofer.de

Fraunhofer Institute for Interfacial Engineering and Biotechnology IGB
Nobelstr. 12
70569 Stuttgart
www.igb.fraunhofer.de

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Thank you for your attention