

Thermo Scientific als Kooperationspartner zur Analytischen Tribologie

ThermoFisher
S C I E N T I F I C

Netzwerktreffen Analytische Tribologie

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(Central Region, Scandinavia)*

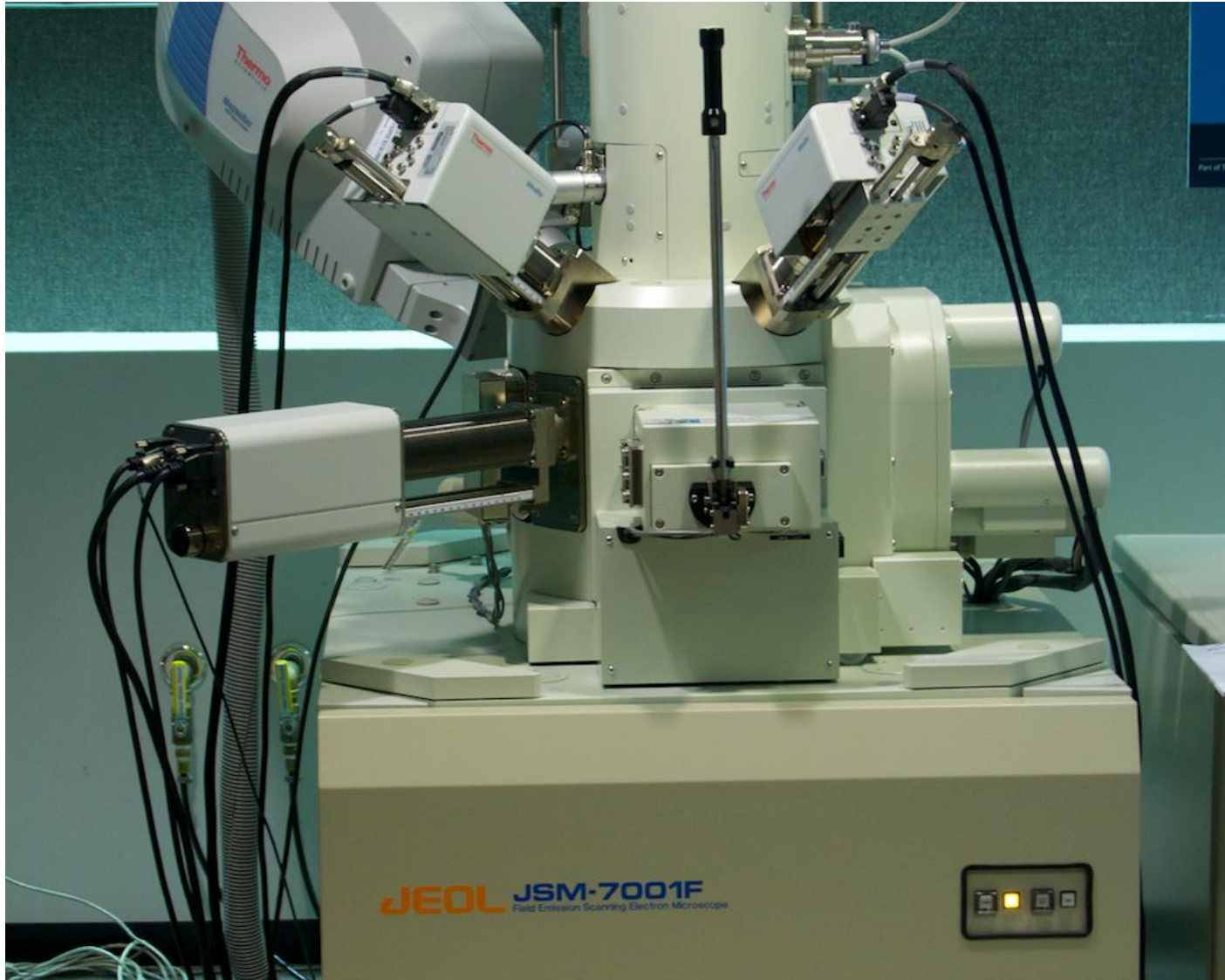
Thermo Fisher Scientific ist nicht nur ein Life Science Konzern, sondern auch ein Partner im Bereich Materialwissenschaften mit:

- Mikroanalytik mit EDS/WDS/EBSD
- Oberflächenanalytik mittels XPS
- Molekülspektroskopie mittels Raman Imaging System
- Rheometer
- Prozesstechnologie

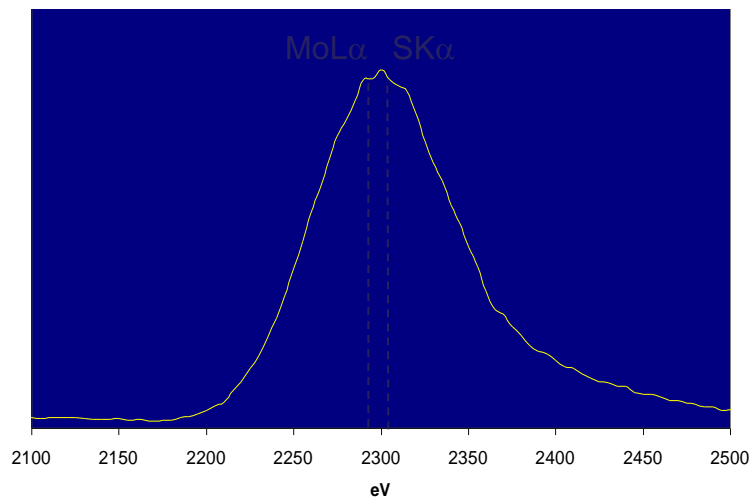
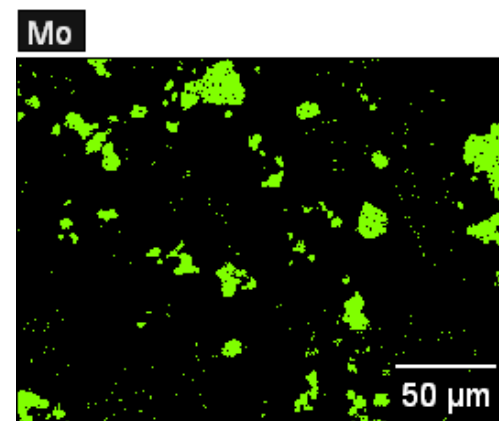
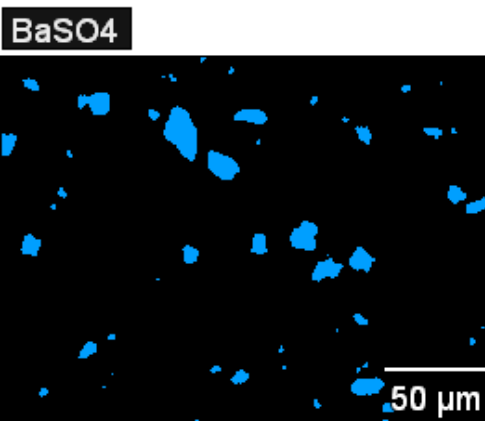
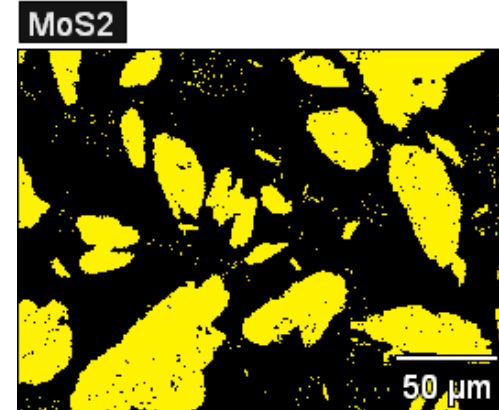
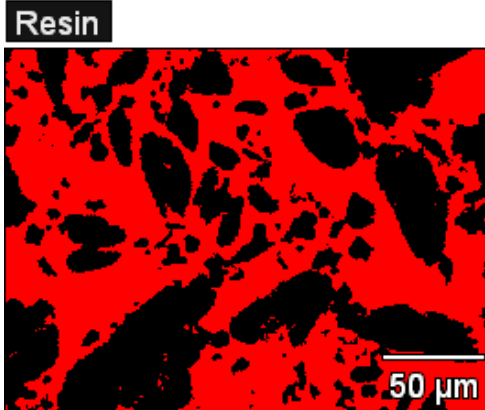
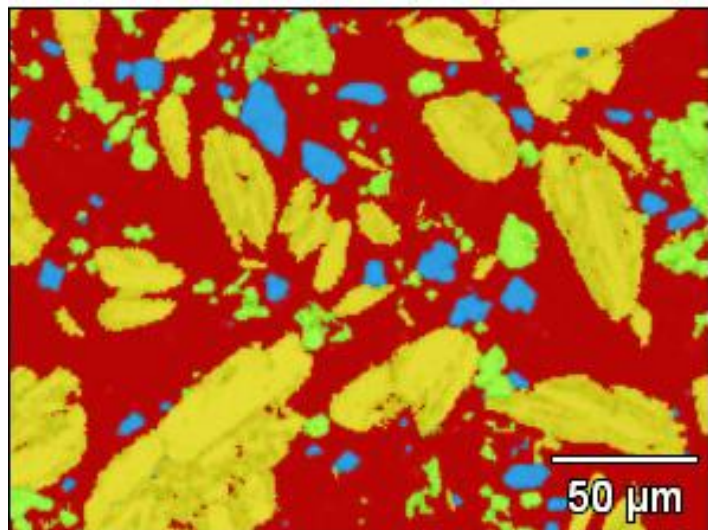
...und viele Kombinationen wie

- Rheometer mit IR Kopplung
- XPS mit EDS und Raman Einheit
- Strahlungsmessung, Gas Analysatoren, Handheld

All Analytical Techniques with double EDS System

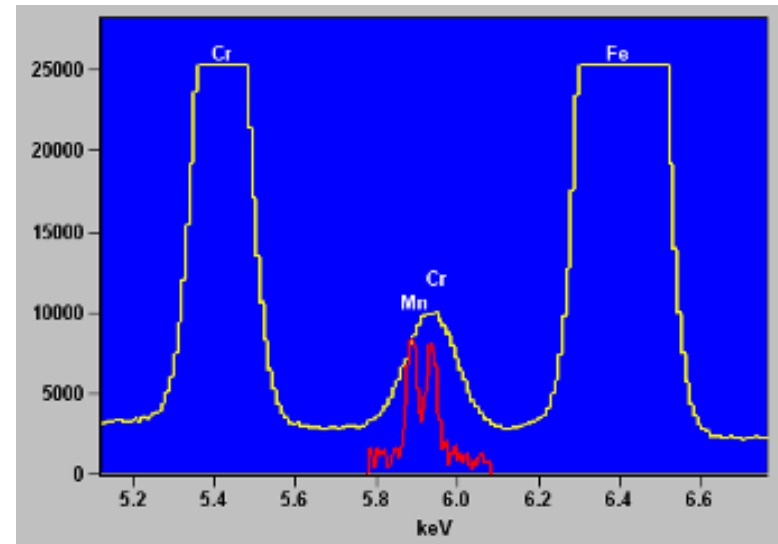


Example – Mo, S, Ba – COMPASS, Xphase, DTP



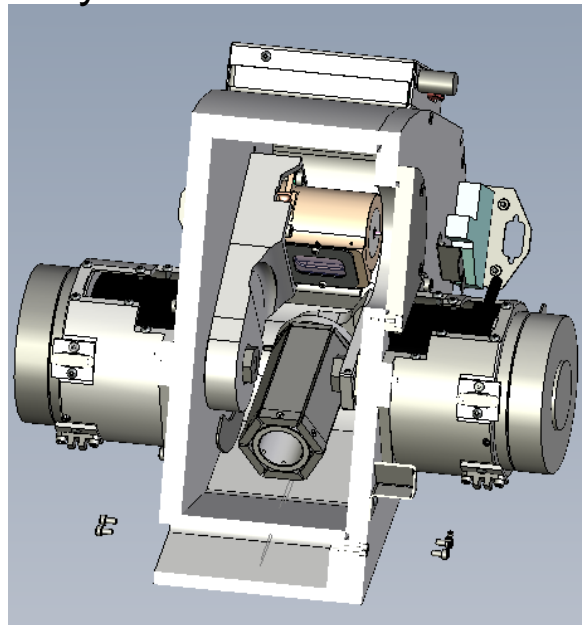
- Distinguishing the three main phases is not possible without robust peak deconvolution

WDS spectrometer „MagnaRay“ integrated in NORAN System 7 (Wavelength Dispersive X-ray Spectroscopy)

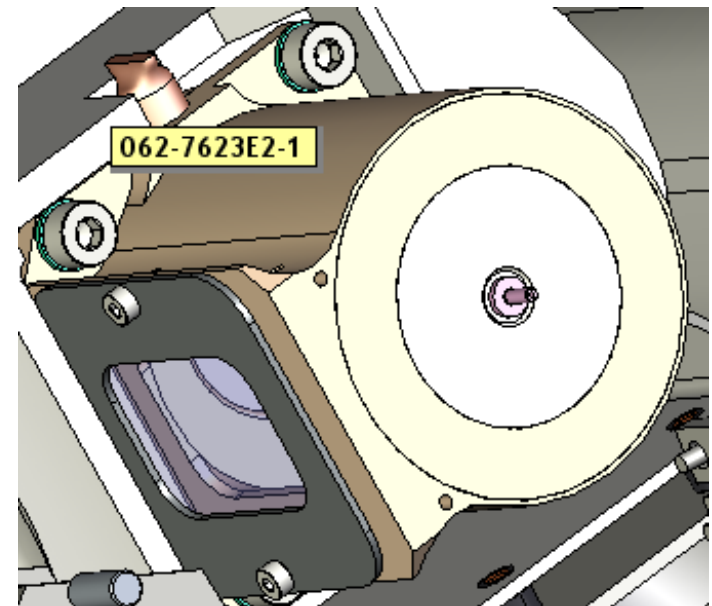


New developments for MagnaRay

very small volume

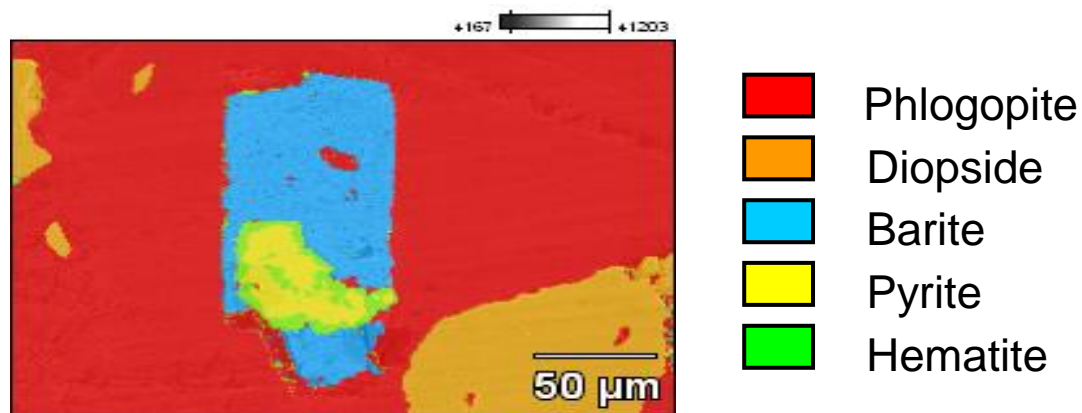


Combined X-ray lens to convert divergent X-rays into parallel beam - provides very good efficiency



sealed Xenon counter – no P10 gas needed

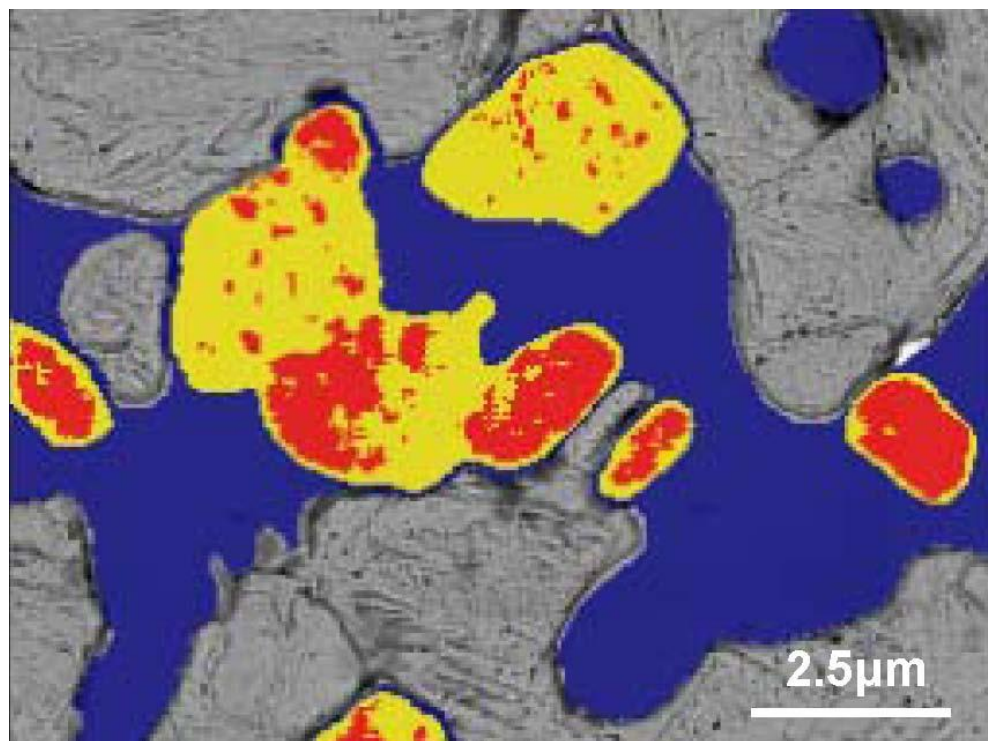
Complex multi-element geological sample



Wt%	Diopside	Diopside	Diopside	Diopside	Pyrite	Pyrite	Barite	Barite	Hematite
O	45.89	45.39	45.72	45.35	0.00	0.00	27.46	27.52	32.39
Mg	9.02	9.17	9.12	8.93	0.00	0.00	0.00	0.00	0.01
Al	3.46	3.68	3.33	3.73	0.00	0.00	0.00	0.00	0.01
Si	22.91	22.81	22.12	22.64	0.00	0.00	0.00	0.00	0
S	0.00	0.02	0.01	0.00	53.19	52.57	13.67	13.11	0.1
K	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01
Ca	18.25	18.31	17.78	18.37	0.00	0.00	0.00	0.00	0
Fe	0.93	1.01	0.88	1.36	48.91	49.28	0.00	0.00	66.58
Ba	0.15	0.11	0.14	0.22	0.00	0.00	58.75	58.23	0.01
Total	100.61	100.50	99.10	100.60	102.10	101.85	99.88	98.86	99.11

Microprobe Accuracy!

WDS is used mostly at low energies where most peak overlaps occur

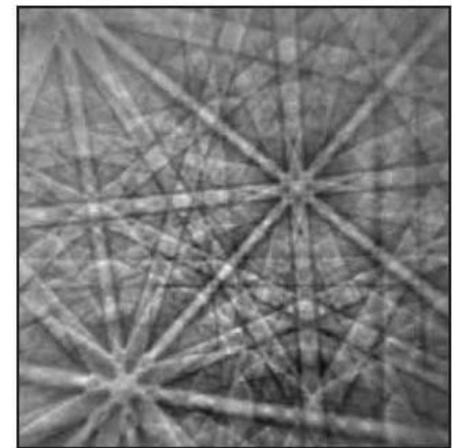
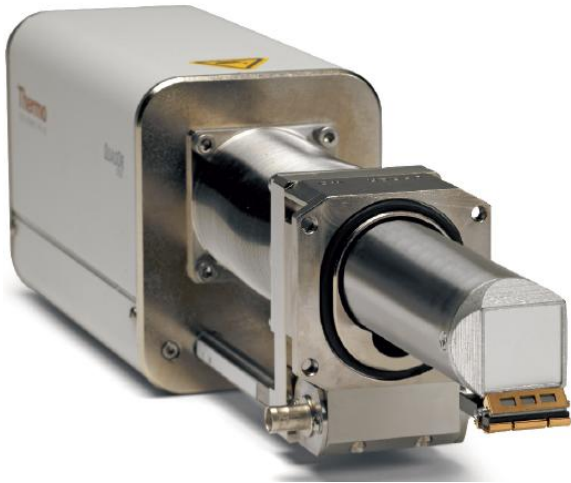


WDS mapping of **Boron** in a Fe-Cr-B alloy at 5kV and 3nA.

- Blue phase 0.5wt% B
 - Yellow phase 0.9wt% B
 - Red phase 1.4wt% B
- Low concentrations of light element in a heavy matrix

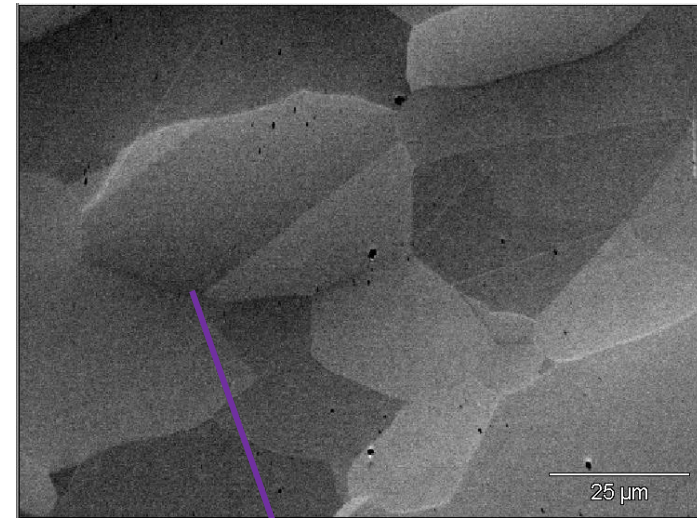
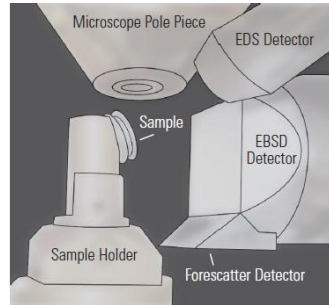
Low energy quantitative analysis

EBSD camera QuasOr in NORAN System 7 (*Electron Backscatter Diffraction*)

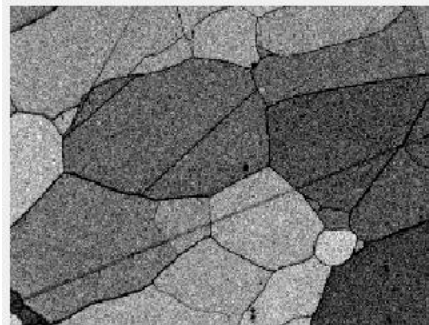


Kikuchi Pattern

QuasOr EBSD camera



Pattern Quality



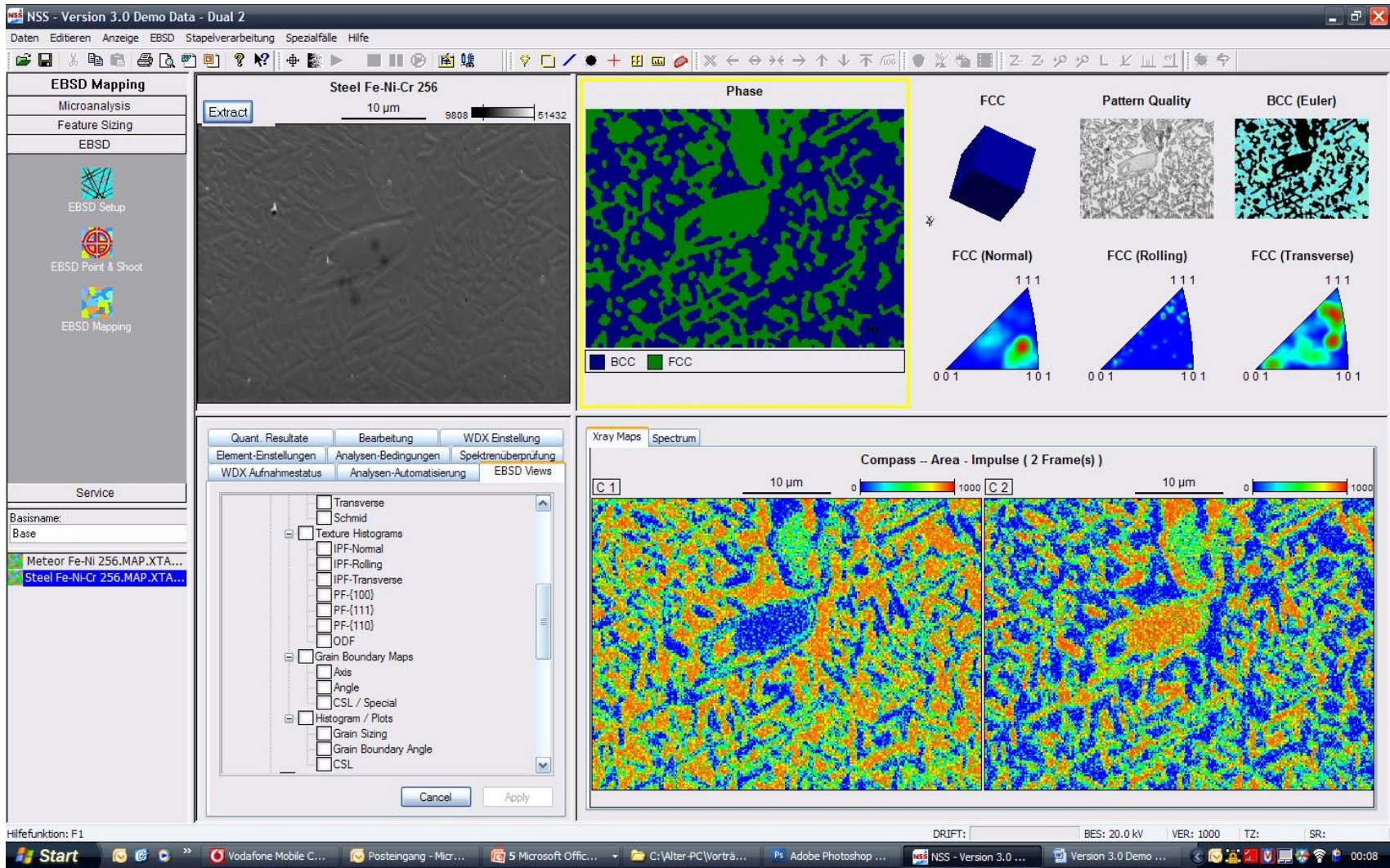
BCC (Euler)



FCC (Euler)

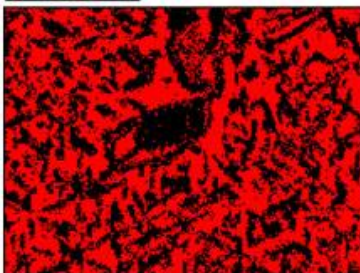


Steel sample

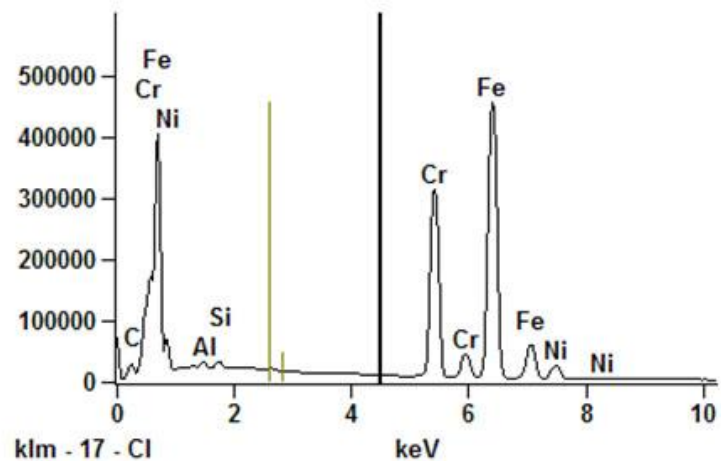


Steel sample – two phases

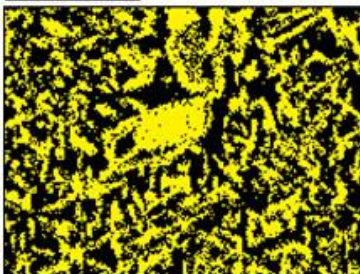
Phase1



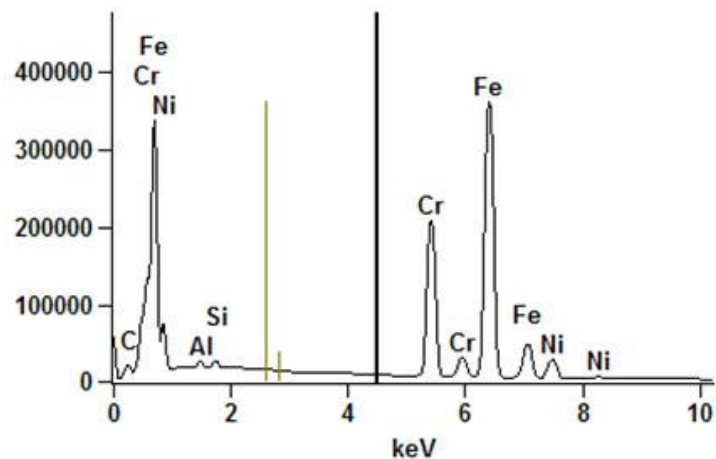
Steel Fe-Ni-Cr 256 Phase1



Phase2



Steel Fe-Ni-Cr 256 Phase2

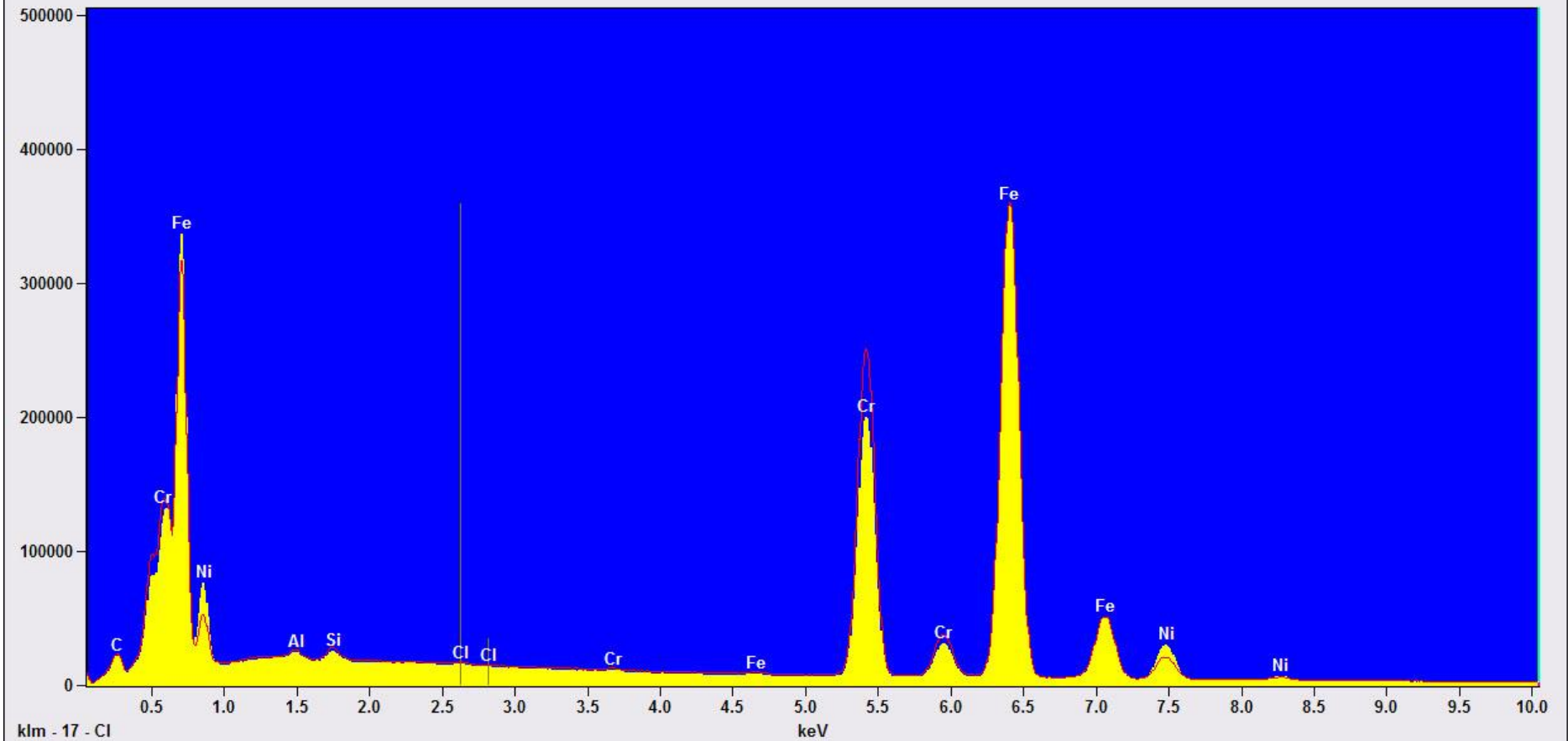


Steel sample – compare spectra

Skalierung, Counts: 474834

Steel Fe-Ni-Cr 256 CP2
steel fe-ni-cr 256 cp1

Cursor: 10.045 keV
2714 Counts



Surface Analysis with XPS

(X-ray Photoelectron Spectroscopy)



Surface Analysis

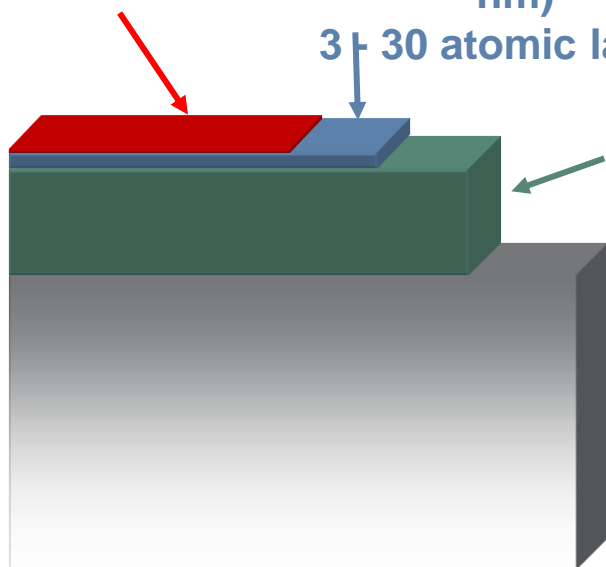
**Surface (1 nm)
3 atomic layers**

**Ultra-thin film (1 to 10
nm)**

3 - 30 atomic layers

**Thin Film (10 nm to 1 μ m)
30 - 300 atomic layers**

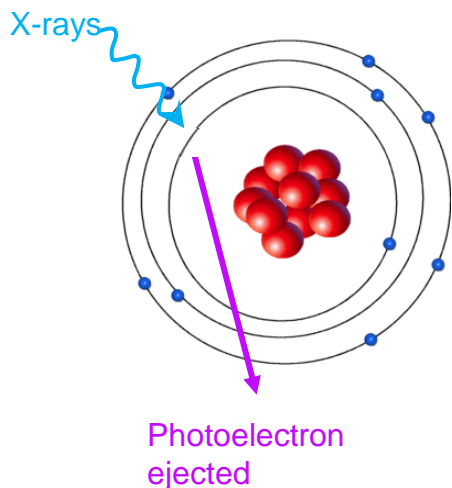
Bulk



Note: Approximate figures only. Actual values depend upon materials

- XPS measures
 - Surface, Ultra-thin film using XPS
 - Ultra-thin film using ARXPS
 - Thin film using sputter profiling

XPS – the data

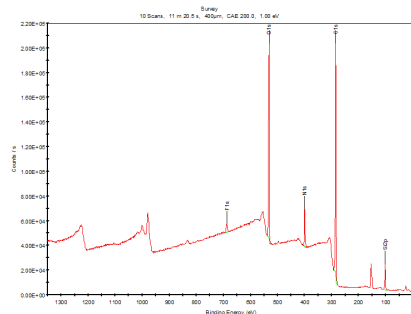


Binding energy tells

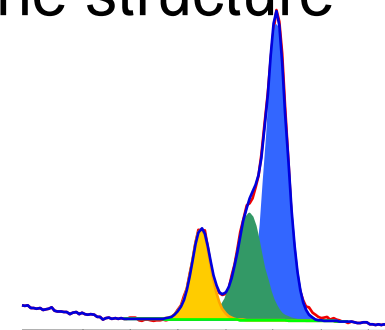
Element

Transition

Wide scan



Fine structure

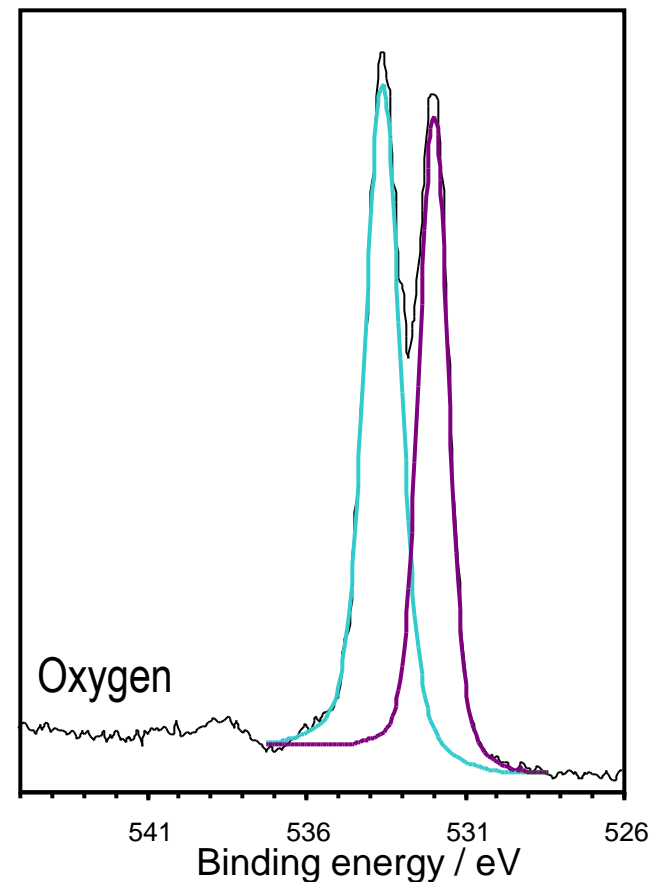
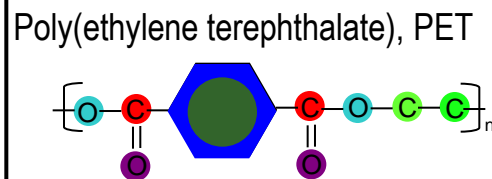
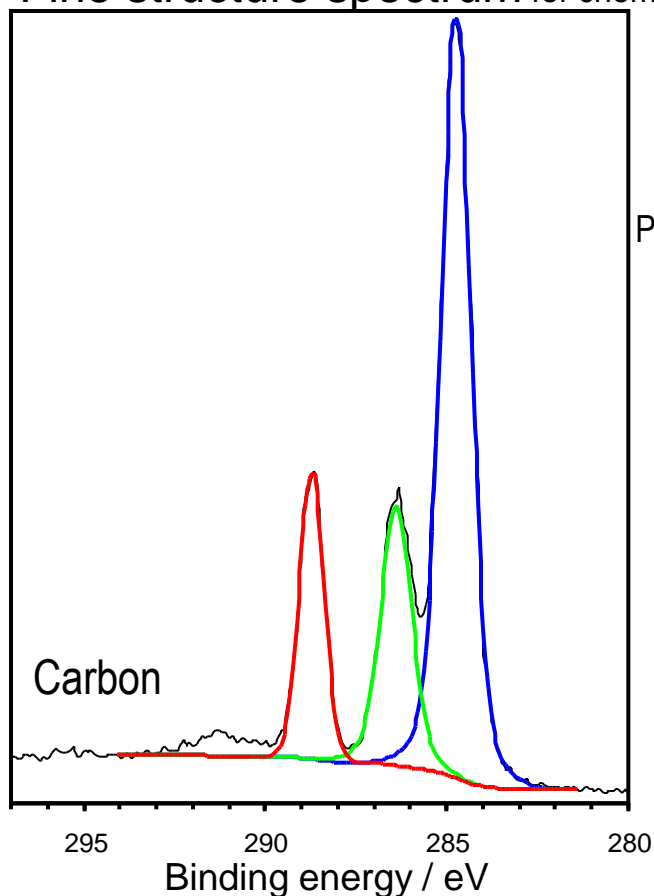


Elemental identification
and quantification

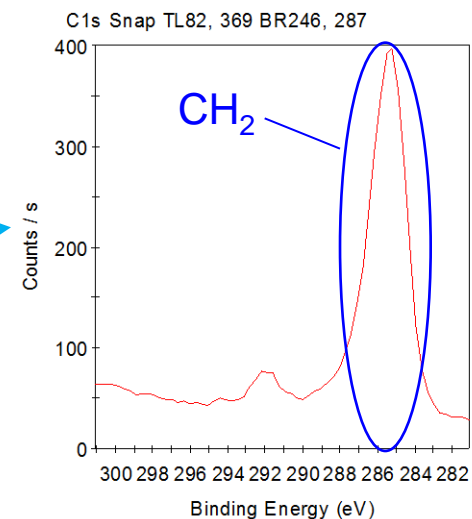
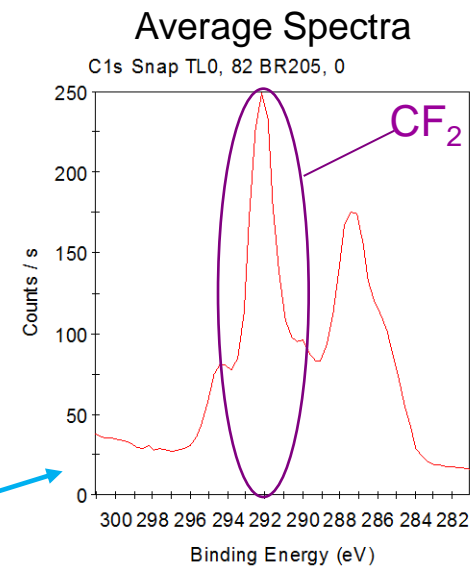
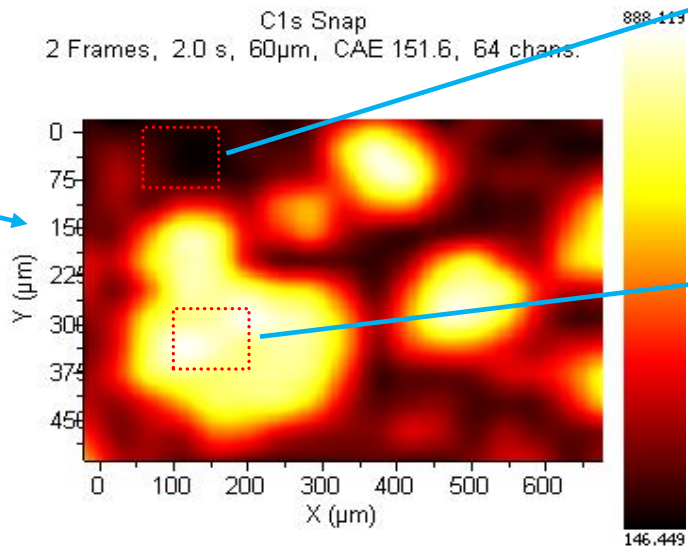
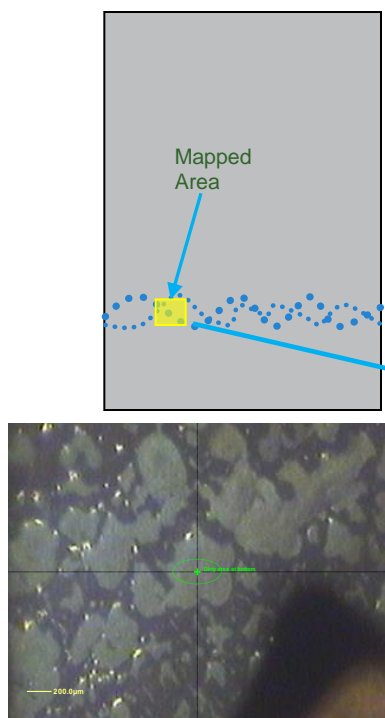
Chemical state identification
and quantification

Fine structure spectrum comprises the chemical state information which makes XPS unique in surface analysis

Fine structure spectrum for chemical state information:



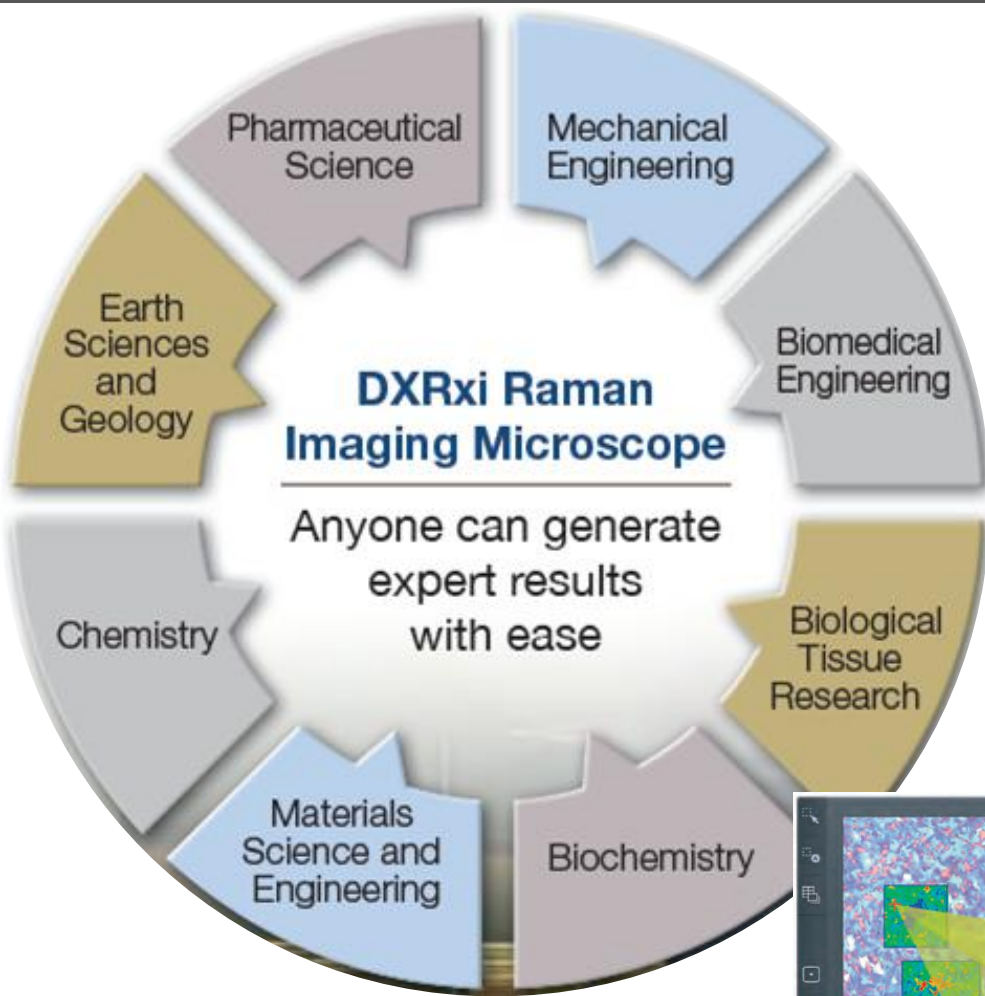
By moving the sample underneath the X-ray beam, and acquiring spectra at many points on the sample chemical maps can be formed. Mapping and imaging techniques allow determination the spatial distribution of elemental and chemical states at the sample surface.



Raman Imaging System



The Raman Imaging Value Proposition

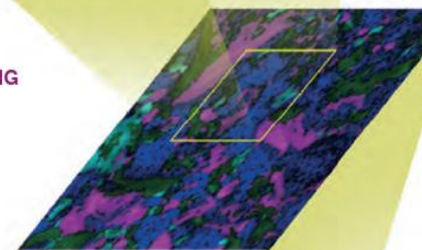


Advanced materials analysis benefits from an image-driven approach

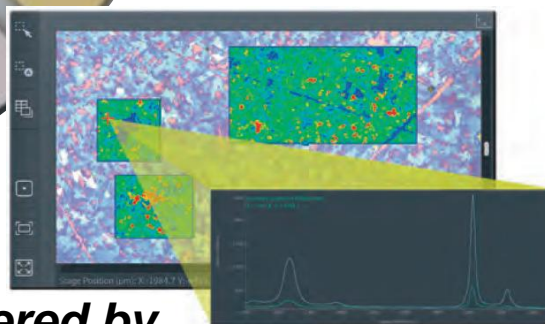
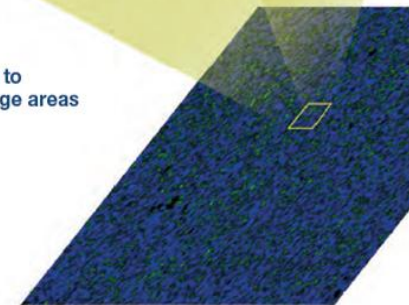
The performance advantage of **FINE DETAIL**



The agility of **RAPID IMAGING**



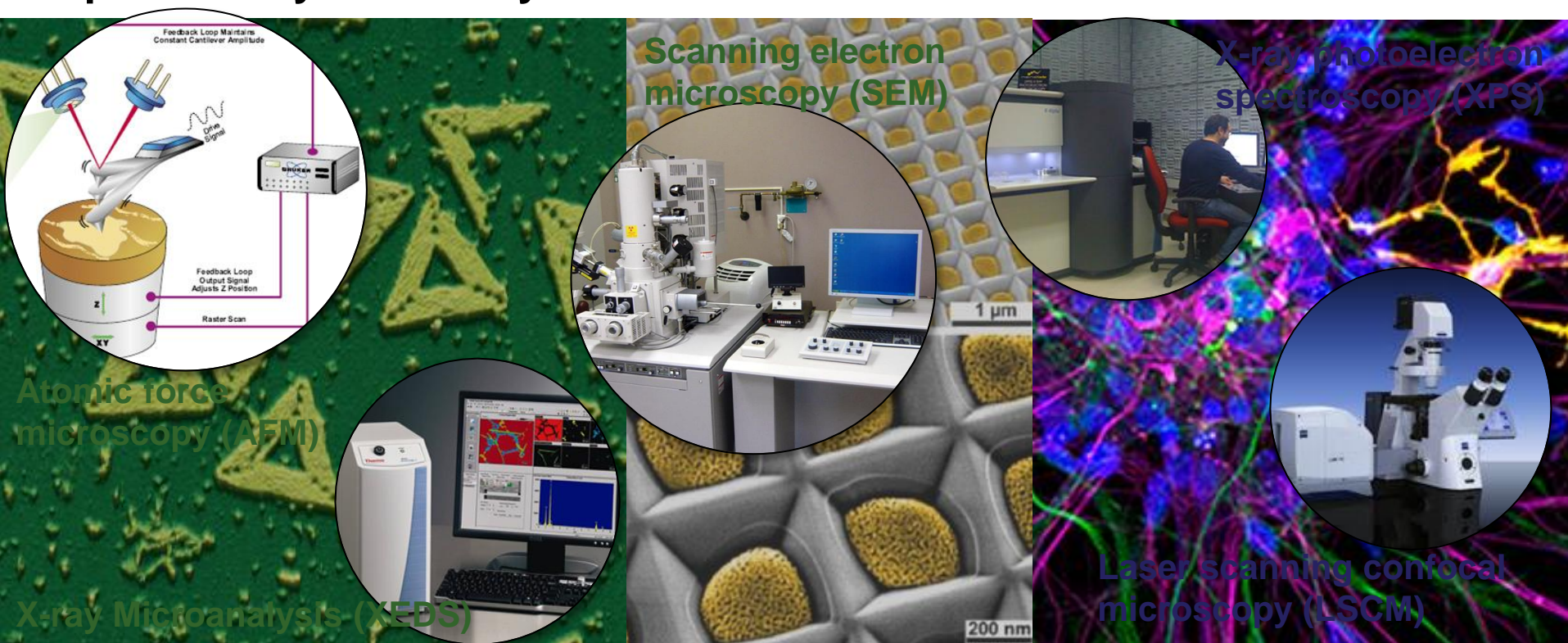
The **POWER** to look over large areas



Emphasis on microscopy, powered by spectroscopy keeps the answer in focus

The Multiuser Laboratory Toolkit

- **Multiple techniques** are increasingly used with the same sample
- Strong emphasis on **image data** and **data visualization**
- ~70% of those polled use **SEM/TEM**, ~50% use **AFM**
- Work continues to become more **interdisciplinary**, requiring broad **proficiency with many different instruments**



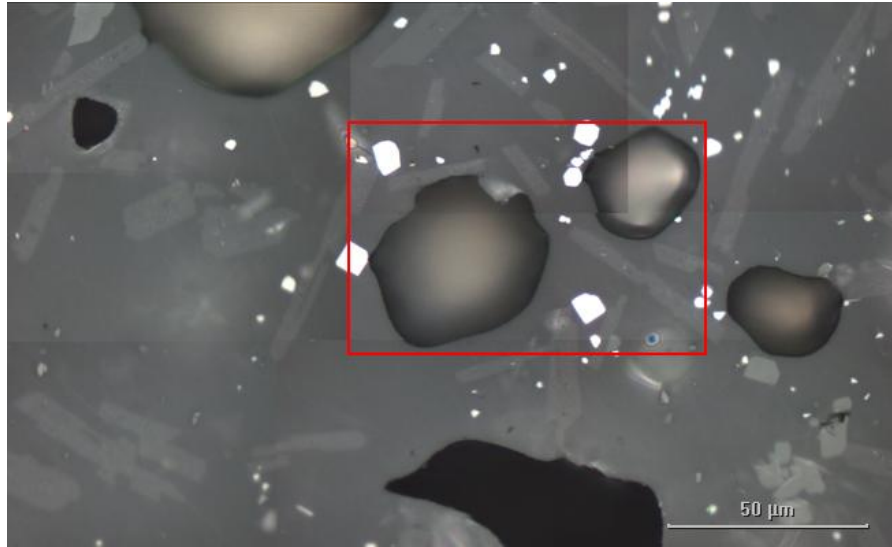
Introducing the DXRxi Raman Imaging Microscope

A total imaging system: hardware and software integration combines **powerful performance** with **image-centric** analysis and **ease of use**



A completely new approach to Raman imaging!

Inclusion in Minerals



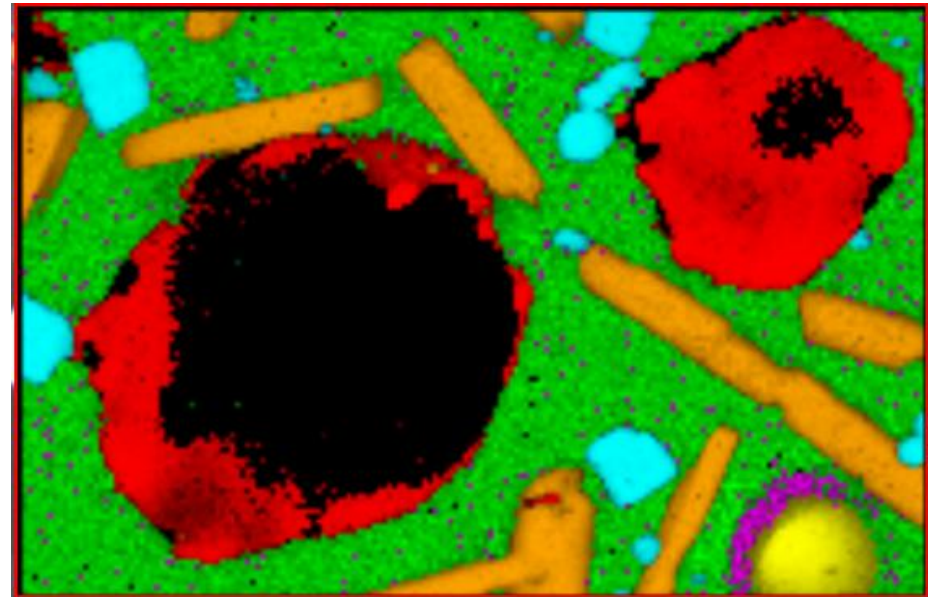
The DXRxi maintains the same confocal capability as the DXR

Quantifying the volatiles in the sample will provide clues about the nature of volcanoes.

455 nm laser, 5.9 mW, 100X objective

21,000 spectra

Acquisition parameters: 40 Hz (25 ms/spectrum),
100 scans, 0.5 µm pixel size

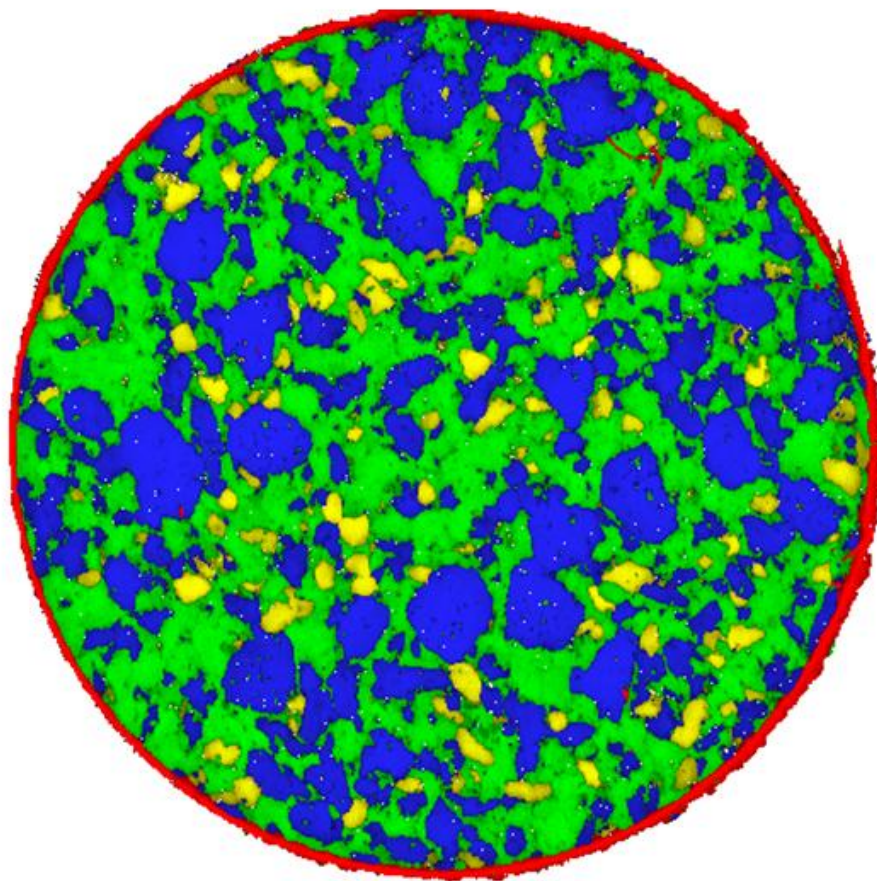


■ Glass ■ CO₂ ■ Carbon ■ OH
■ Chalcocite? ■ Labradorite?

Sample provided by Jenny Riker, University of Bristol

Tablet analysis

MCR Analysis



Determine:

- Size of each domain
- Distribution of domains
- Overall composition of tablet

11 x 11 mm surface area
532 nm laser, 10X objective

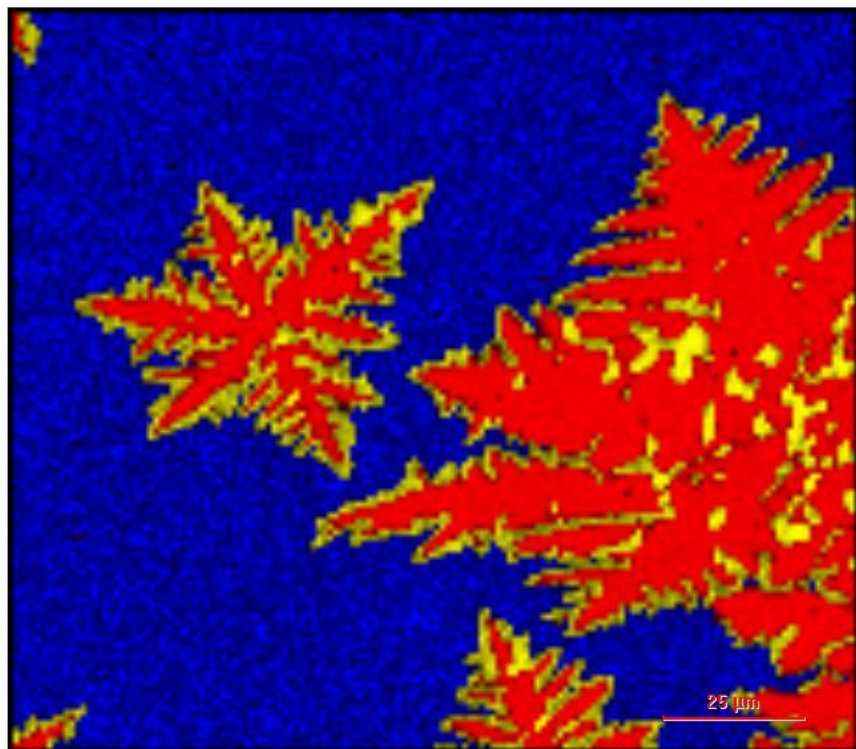
226,000 spectra, 25 μm pixel size

Acquisition parameters: 550 Hz (1.8
ms/spectrum)

8 minute collect time!!

■ Aspirin ■ Acetaminophen ■ Caffeine ■ Titanium Dioxide

Graphene on Copper



Graphene grown on copper and analyzed *on the copper* with the 455nm laser

$$I_{scatter} \propto \frac{1}{\lambda_{ex}^4}$$

455nm laser

150 x 150 μm surface area

90,000 spectra!!!

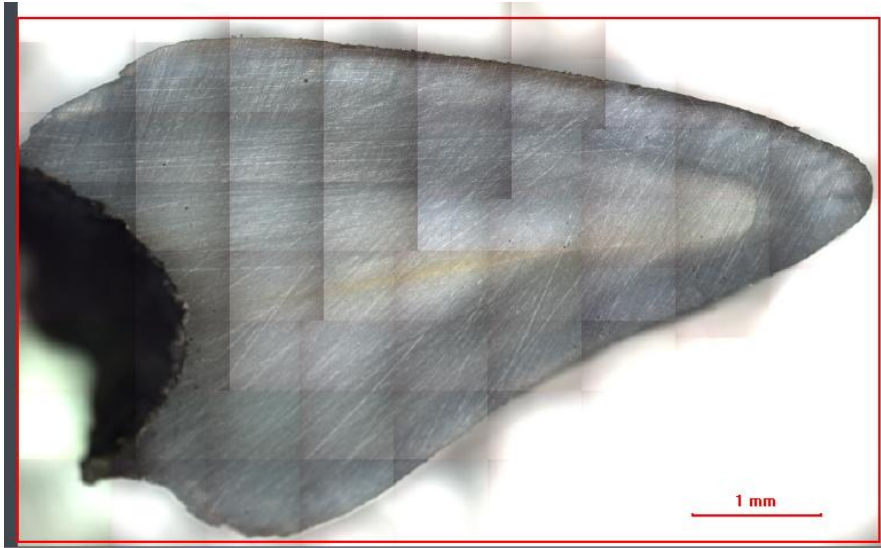
2.0 μm pixel size

Acquisition parameters: 100 Hz (10 ms/spectrum),

4 scans

■ Copper Substrate ■ Oxidized Copper ■ Single-layer graphene

Tooth analysis



780nm laser, 5.9 mW, 10X objective

29,000 spectra!!!

Acquisition parameters 40 Hz (25 ms/spectrum),
100 scans, 30 μ m pixel size

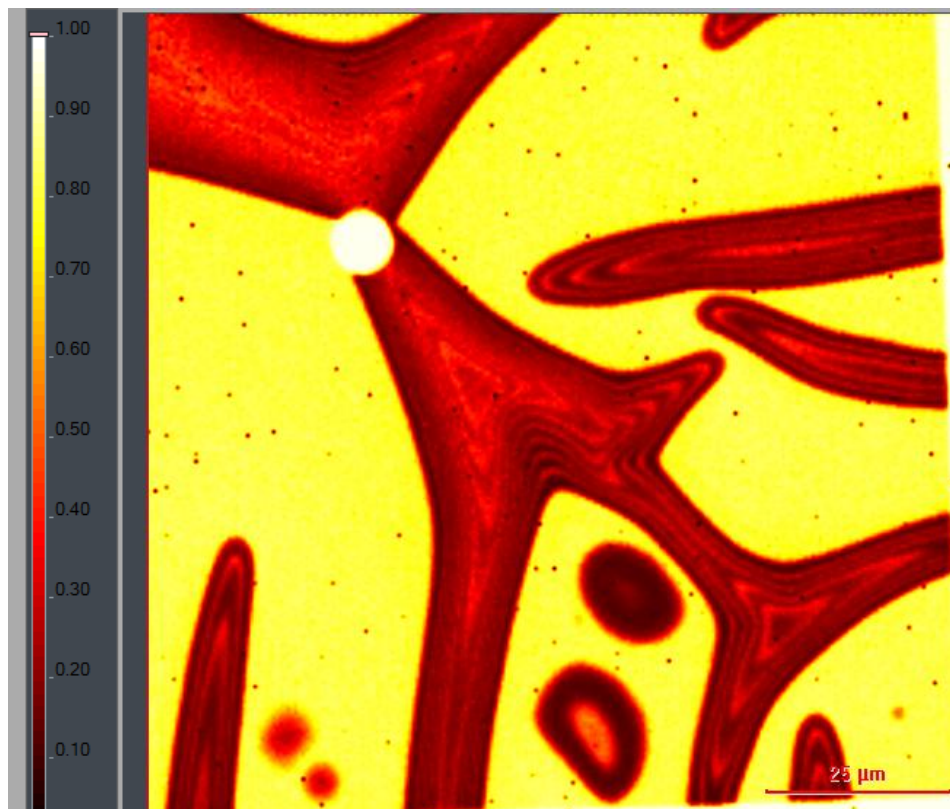


■ Pulp ■ Circumpulpal Dentin ■ Dentin ■ Enamel

~22 hour collect time

***Impossible on a traditional
mapping instrument!***

Silicon Stress



455 nm laser, 1.0 mW, 100X objective

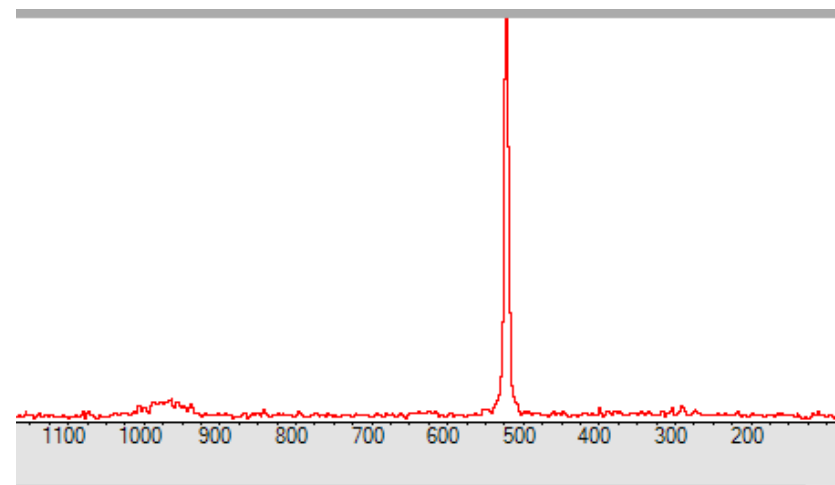
50,000 spectra

Acquisition parameters: 100 Hz (10 ms/spectrum),
25 scans, 0.5 μm pixel size

~4 hour collect time!!

Sample is a Si substrate with a layer of Si/Ge deposited followed by an additional layer of Si

The presence of the Ge causes stress in the second layer of Si, which is imaged



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