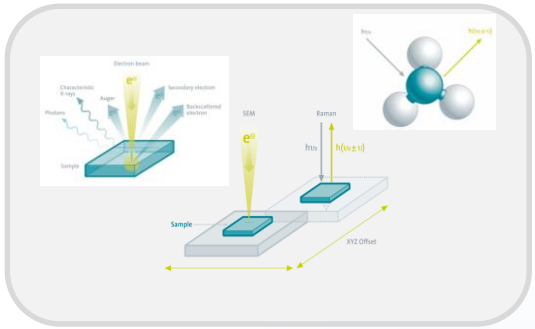
The Benefits of Correlative Scanning Electron – and Confocal Raman Microscopy

Description and Case Study

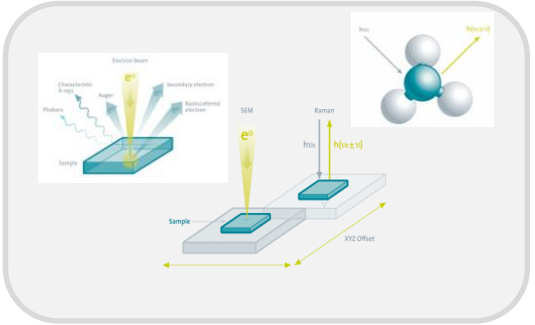
Maxime Tchaya, Ute Schmidt, Olaf Hollricher
WITec GmbH, Germany



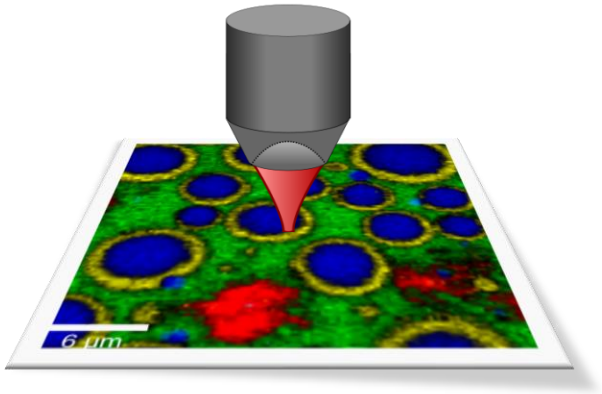
made in Germany

Outline

- The instrument and techniques:
  Raman Imaging and SEM = RISE Microscopy
- RISE applied to polymer research
- RISE applied to Li battery research
- RISE applied to graphene research
- Summary

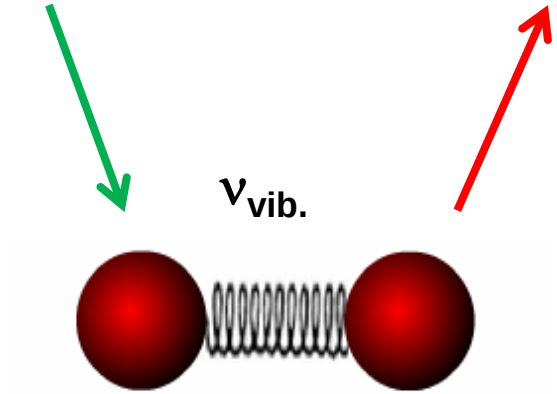


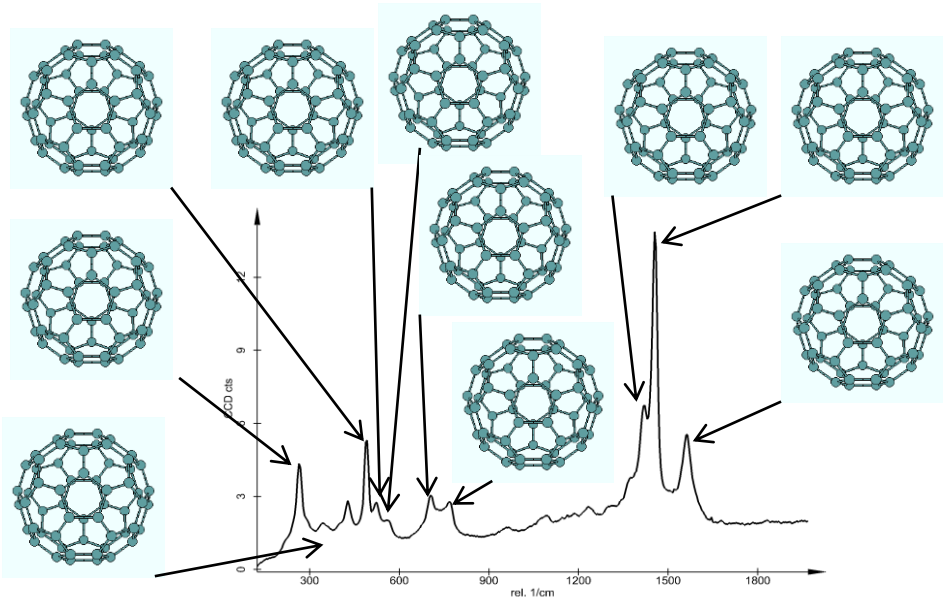
Confocal Raman Imaging



chemical/molecular imaging with diffraction limited resolution

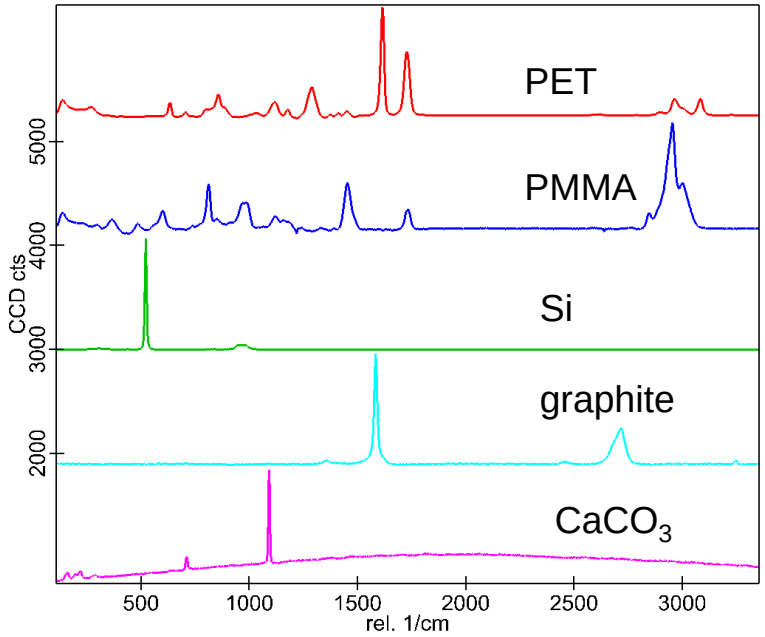
ν_{light} $\nu_{Raman} = \nu_{light} - \nu_{vib.}$

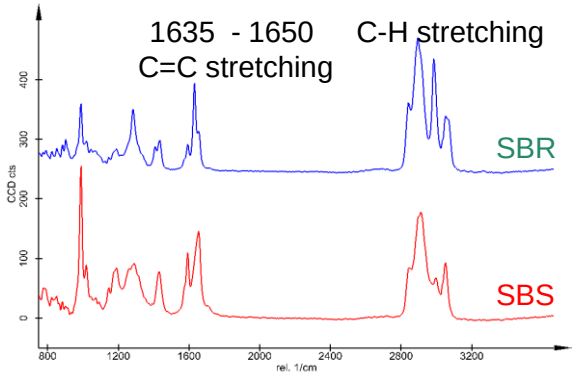


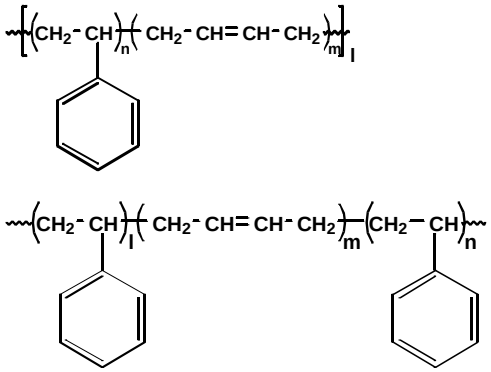


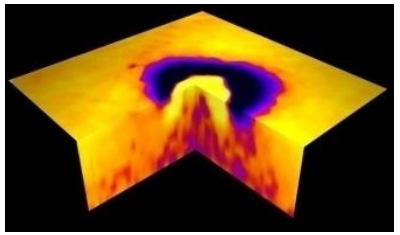
What do we learn from Raman spectra and images

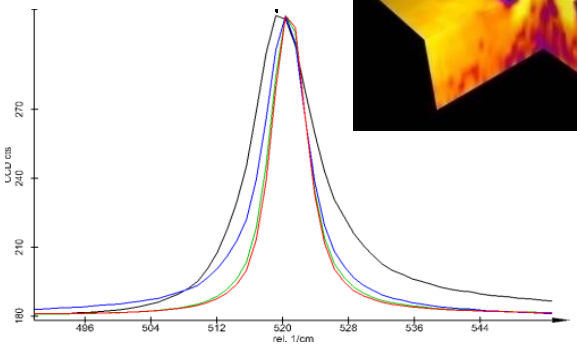
- Chemical composition
- Polymorphs
- Stress => shift of Raman band
- Crystallinity => width of Raman band



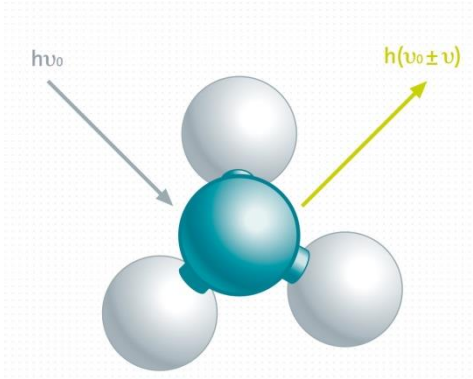




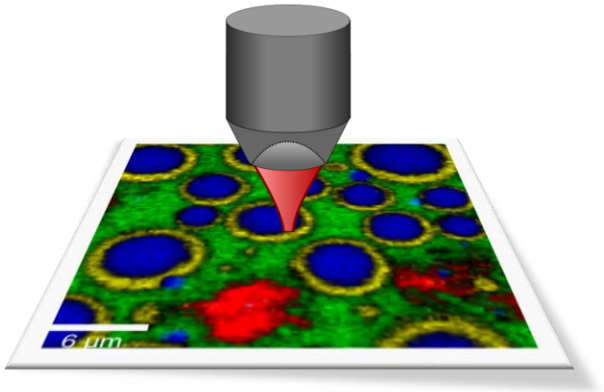




Correlative Microscopy: RISE microscopy



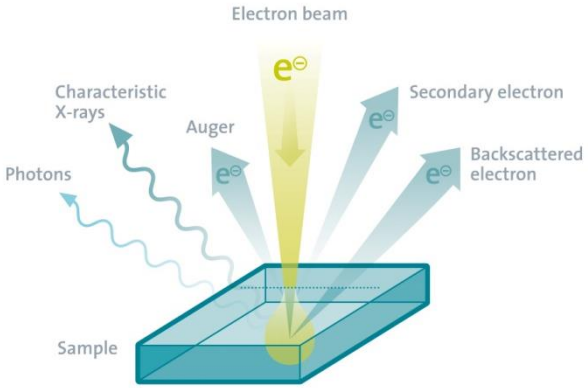
Confocal Raman Imaging

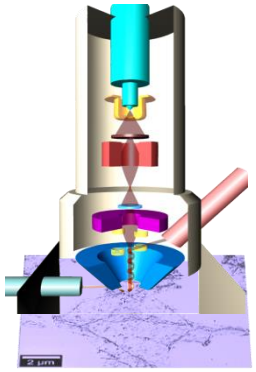


chemical/molecular imaging with diffraction limited resolution

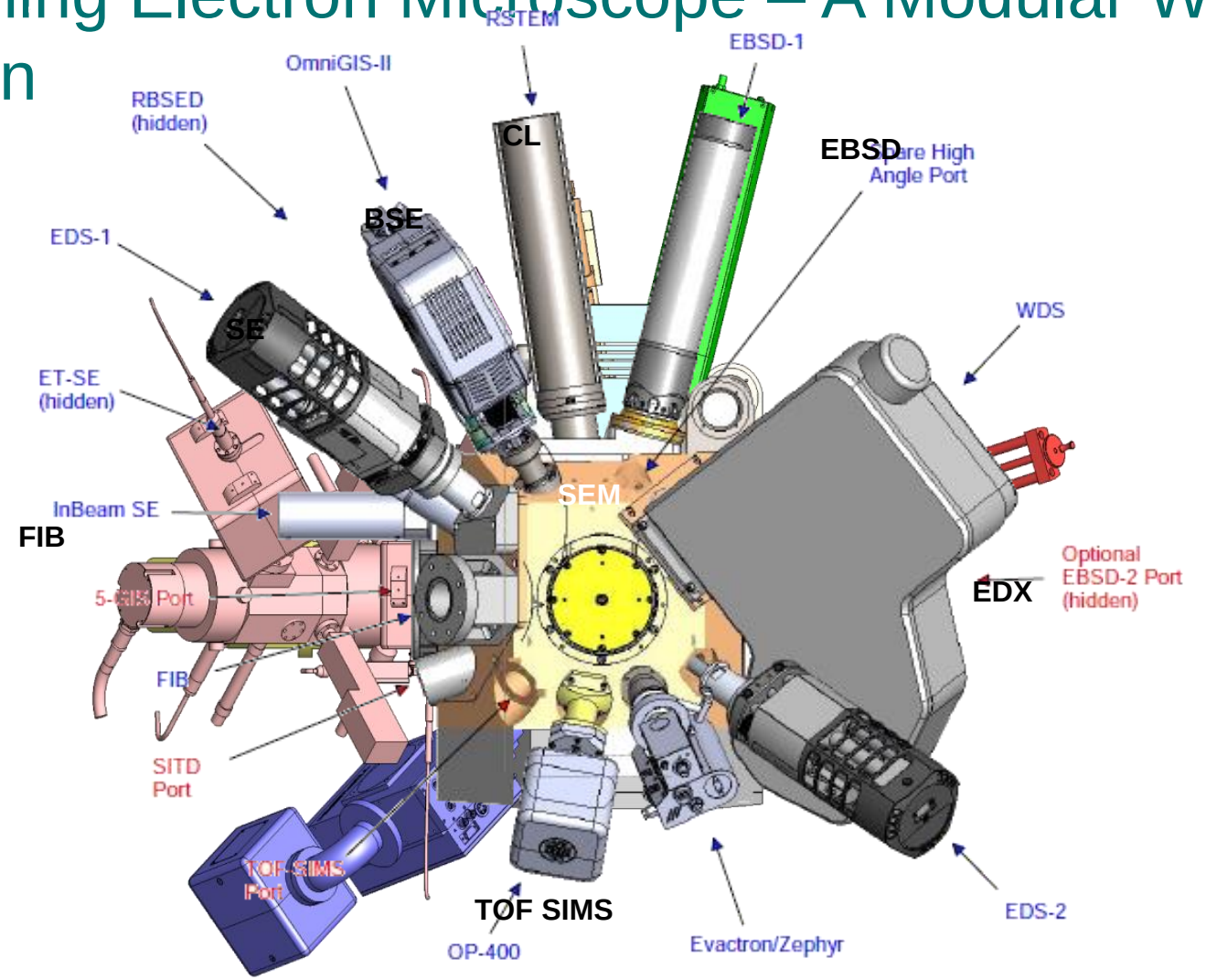
SEM

High resolution 2D imaging EDS: information about atomic composition



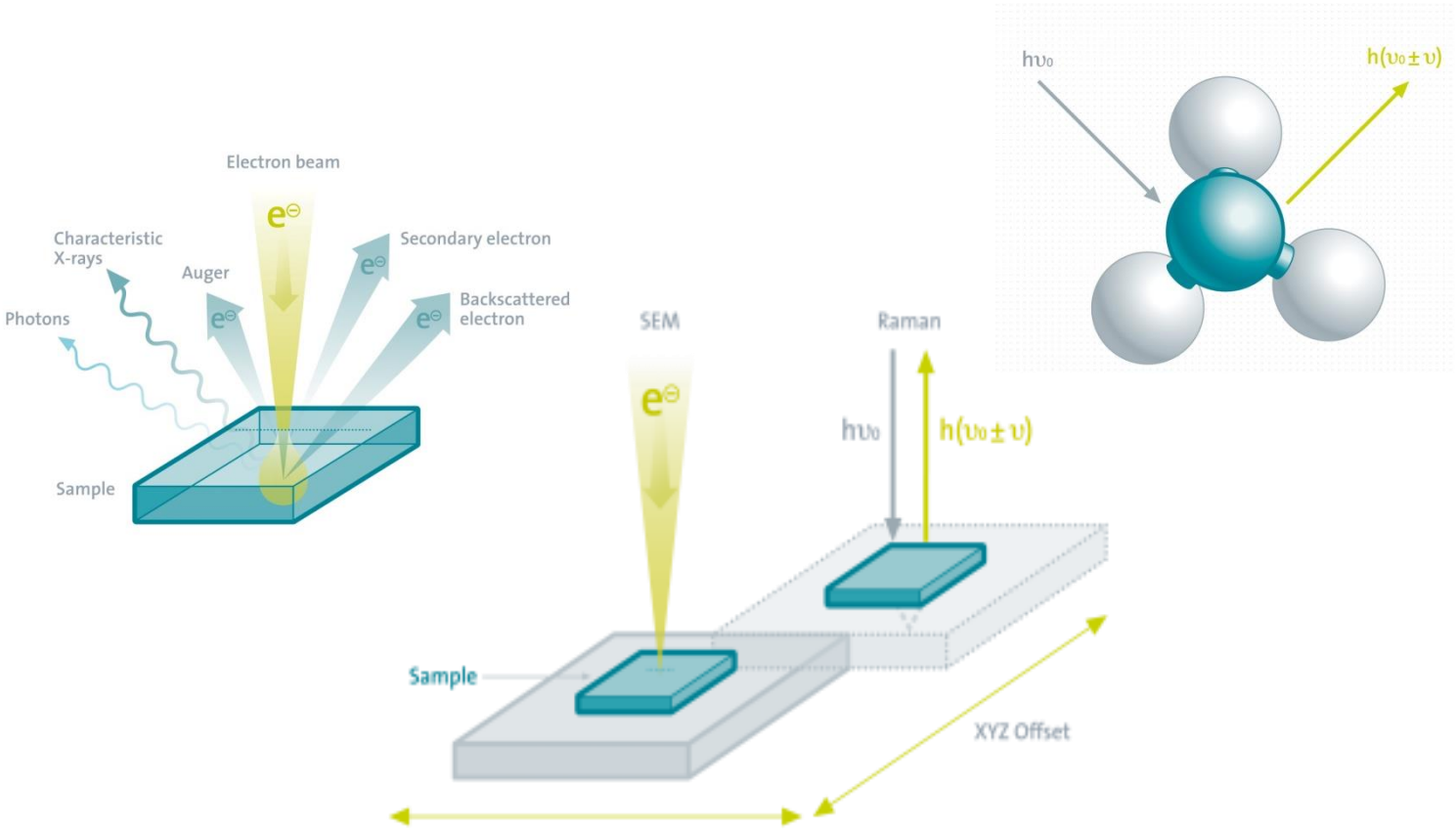


Scanning Electron Microscope – A Modular Work Station

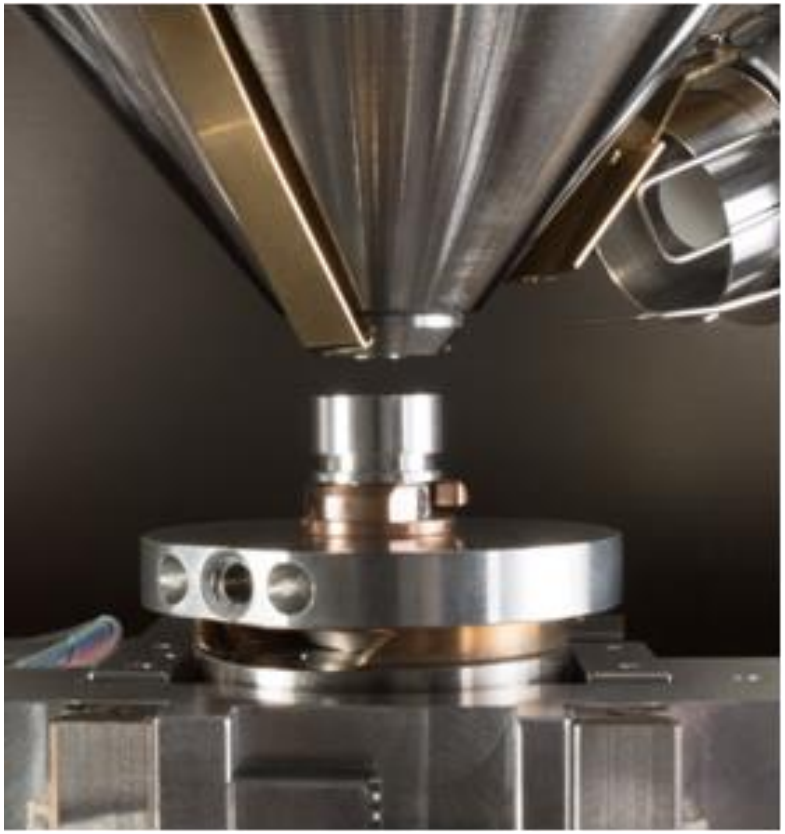


Coincidence Point at Analytical WD = 9 mm

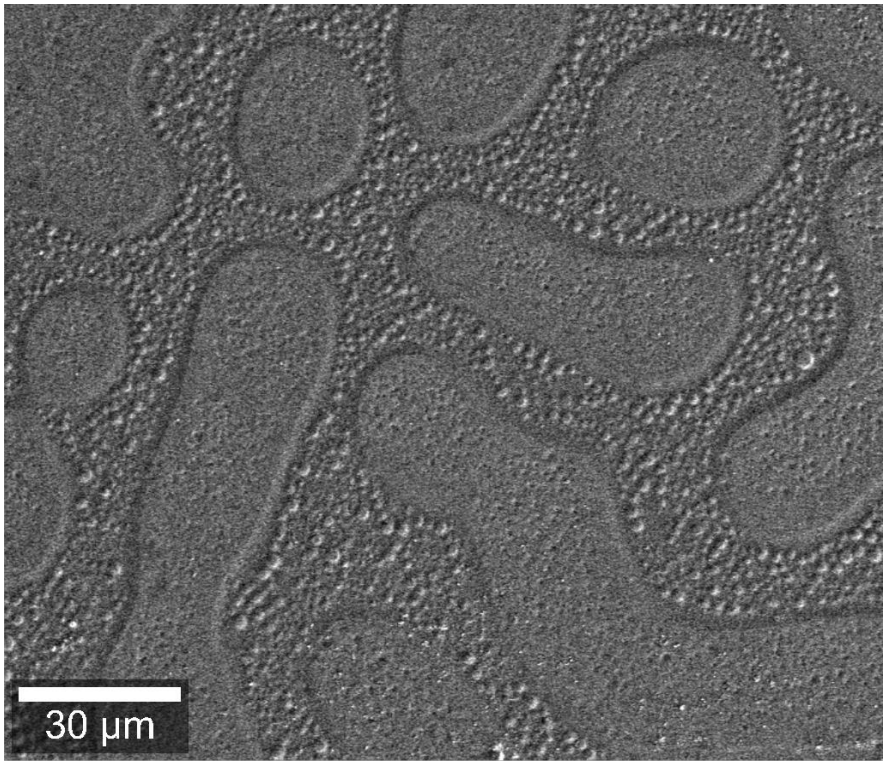
RISE Schematics and Principles of Operation

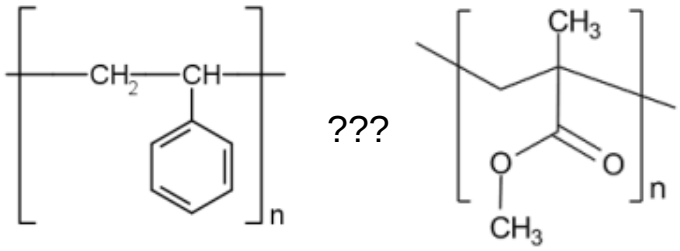


Workflow

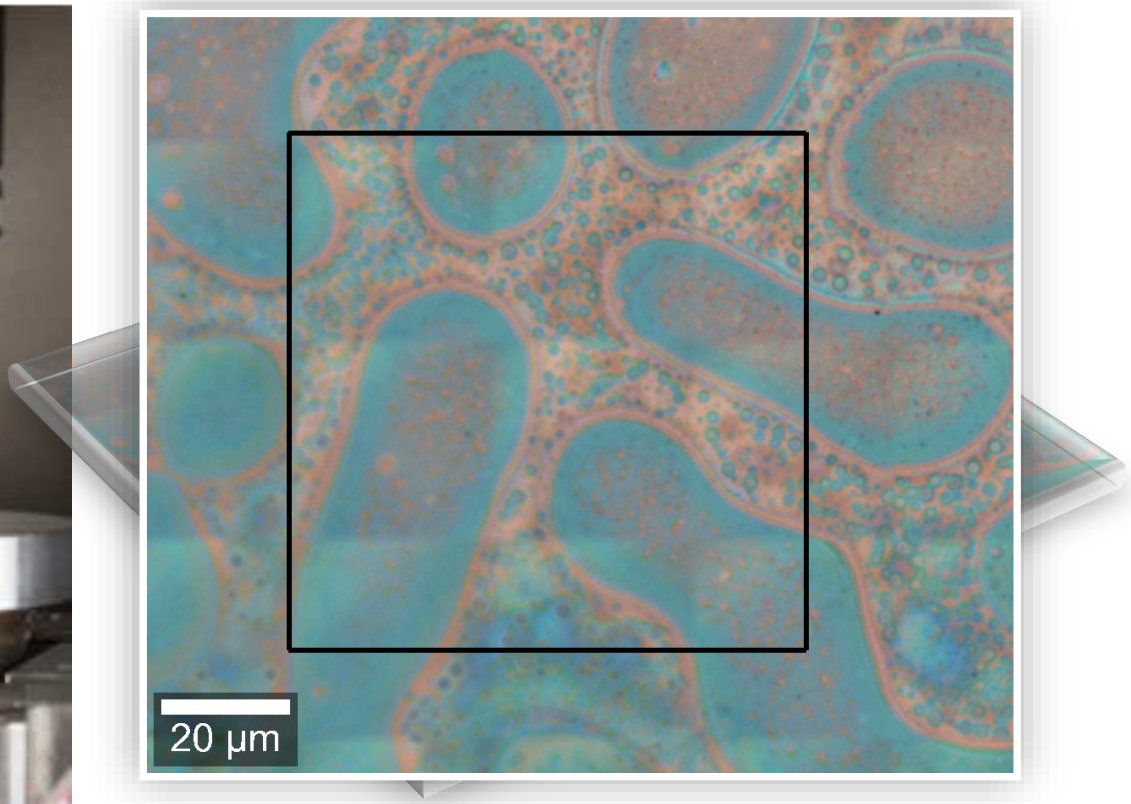


SEM image of a thin polymer film of PS-PMMA

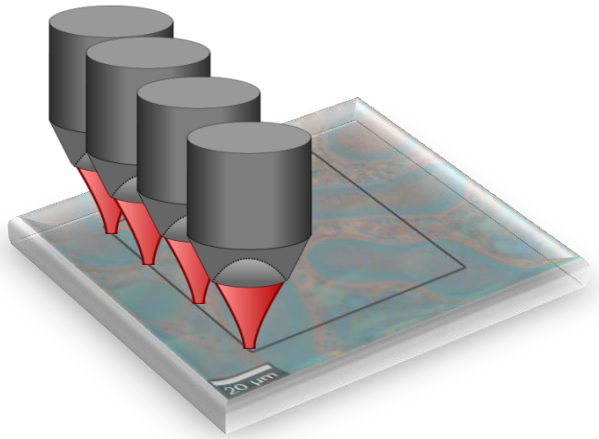


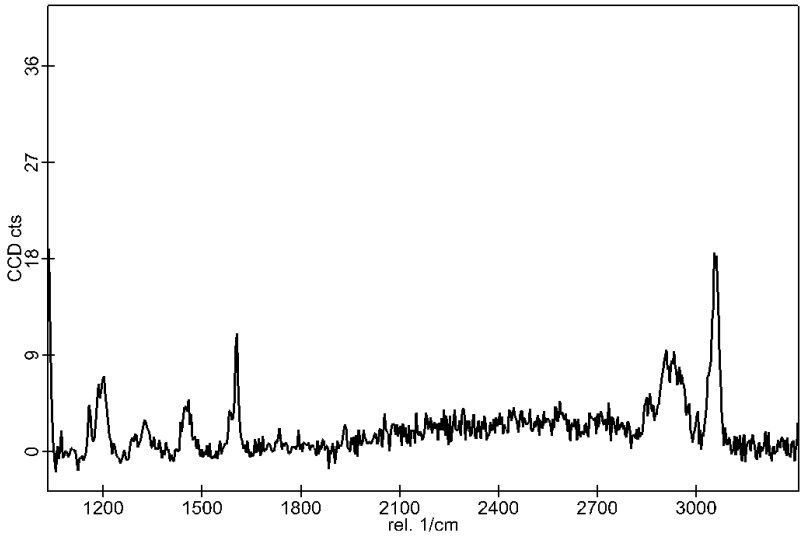


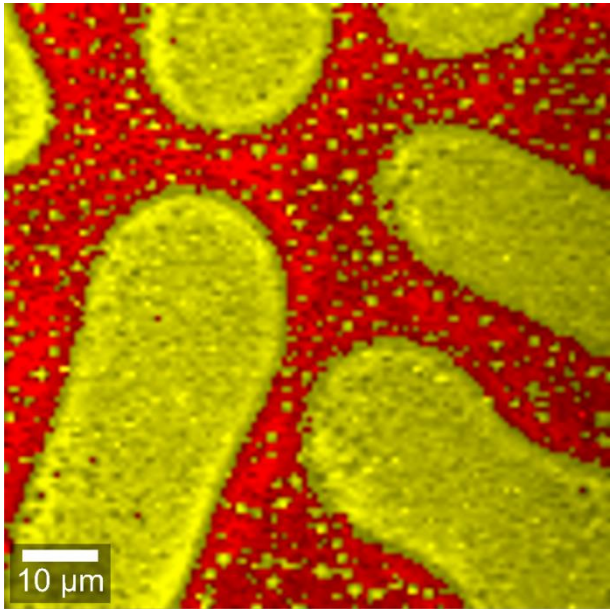
Workflow

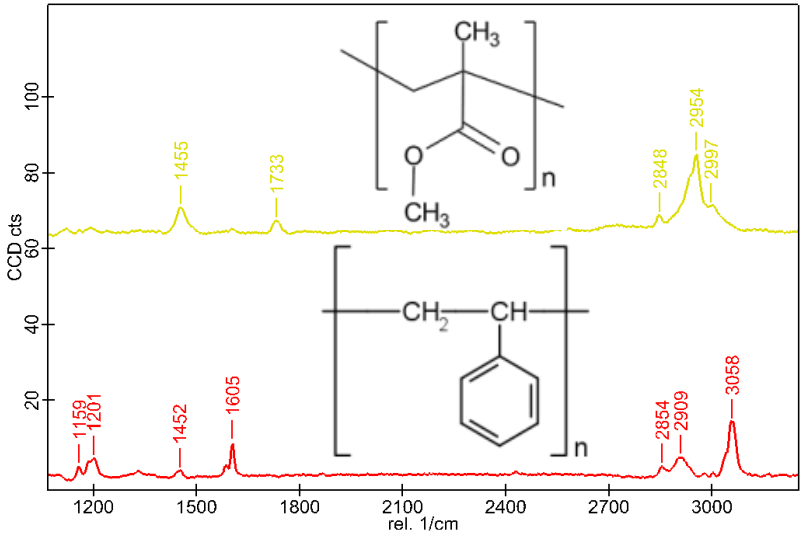


Raman Imaging

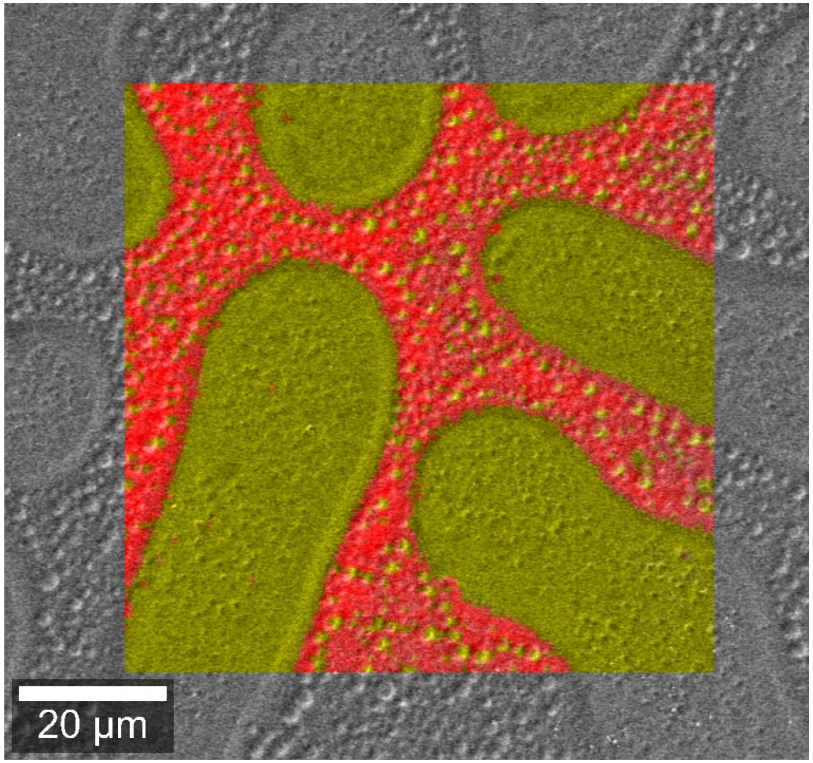




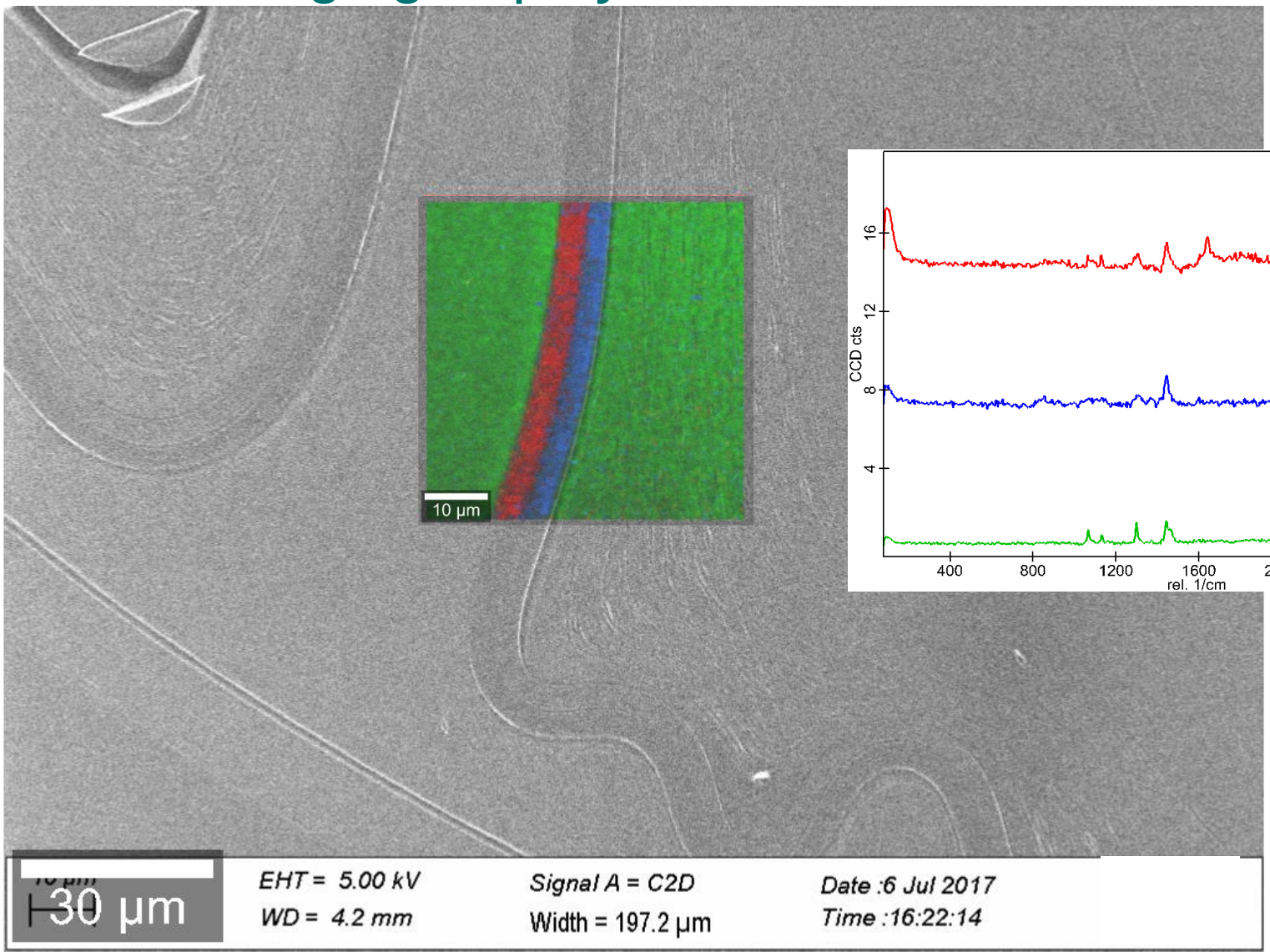




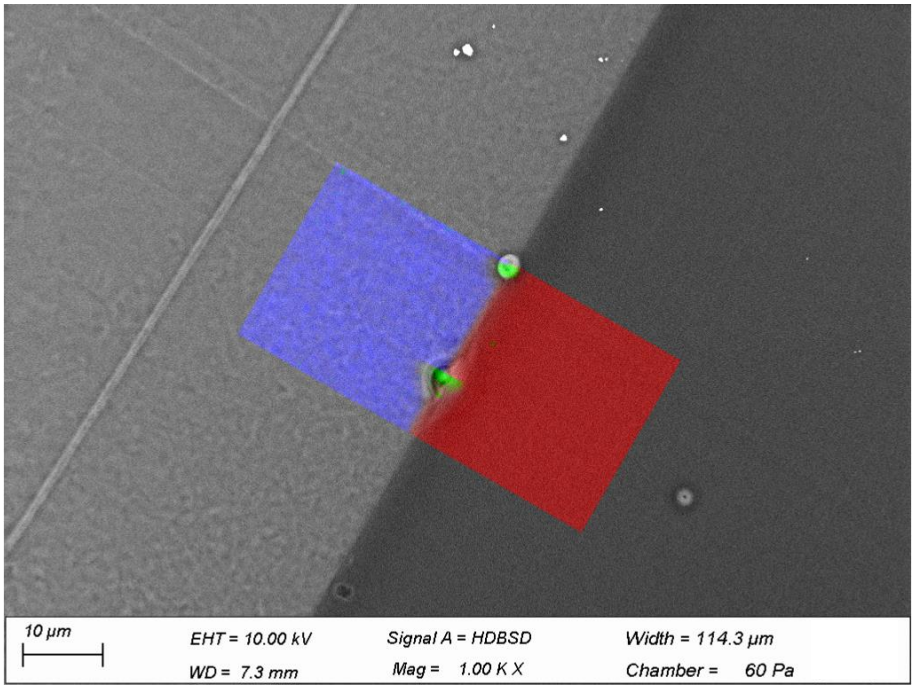
Overlay Raman and SEM images => RISE image

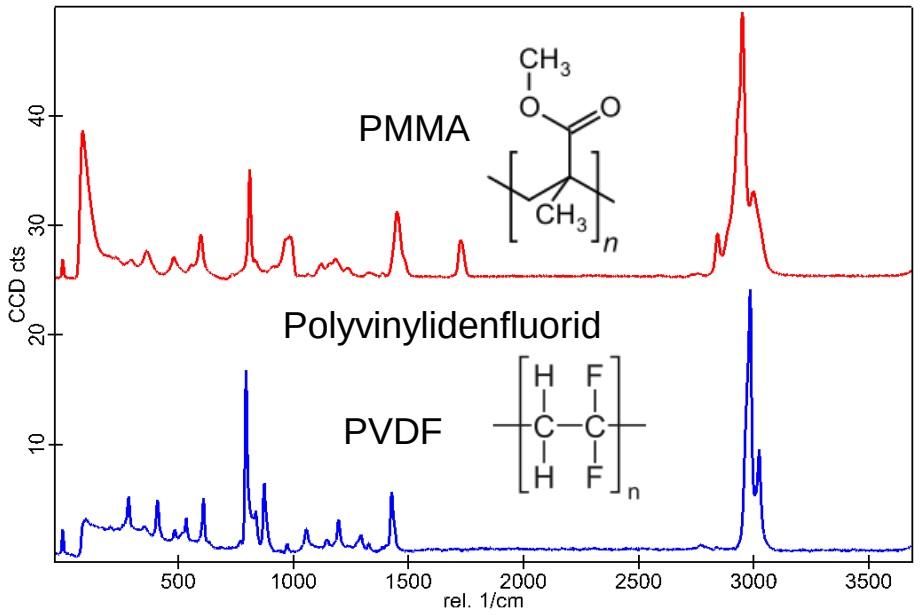


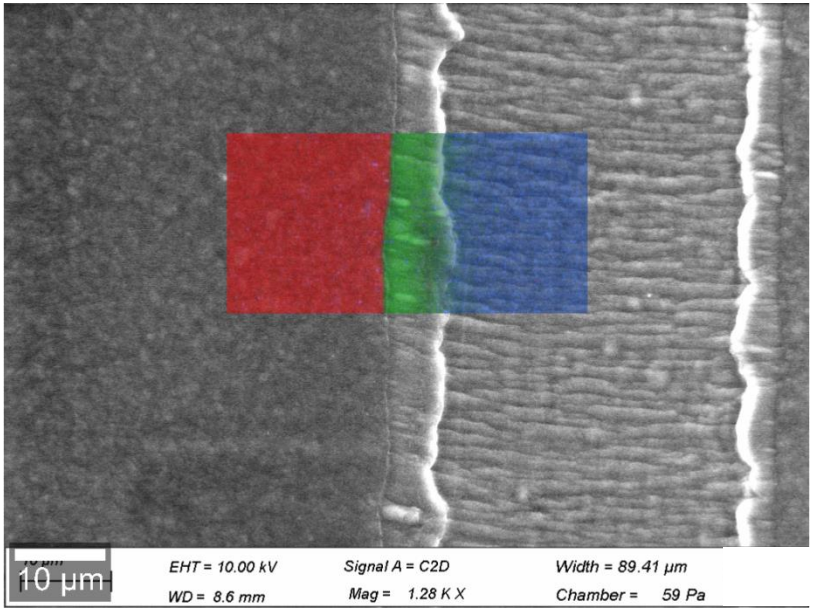
RISE imaging of polymer foil

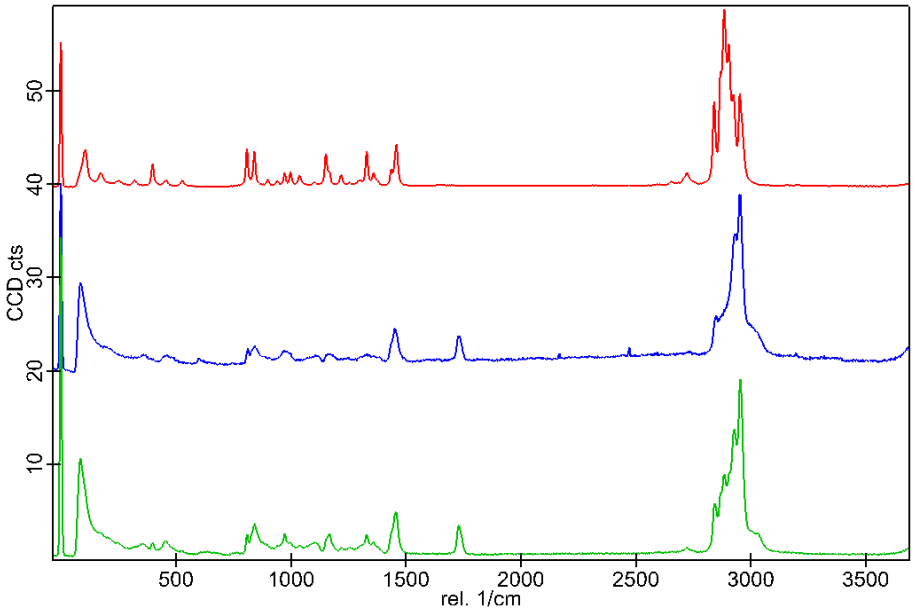


RISE imaging of Polymers

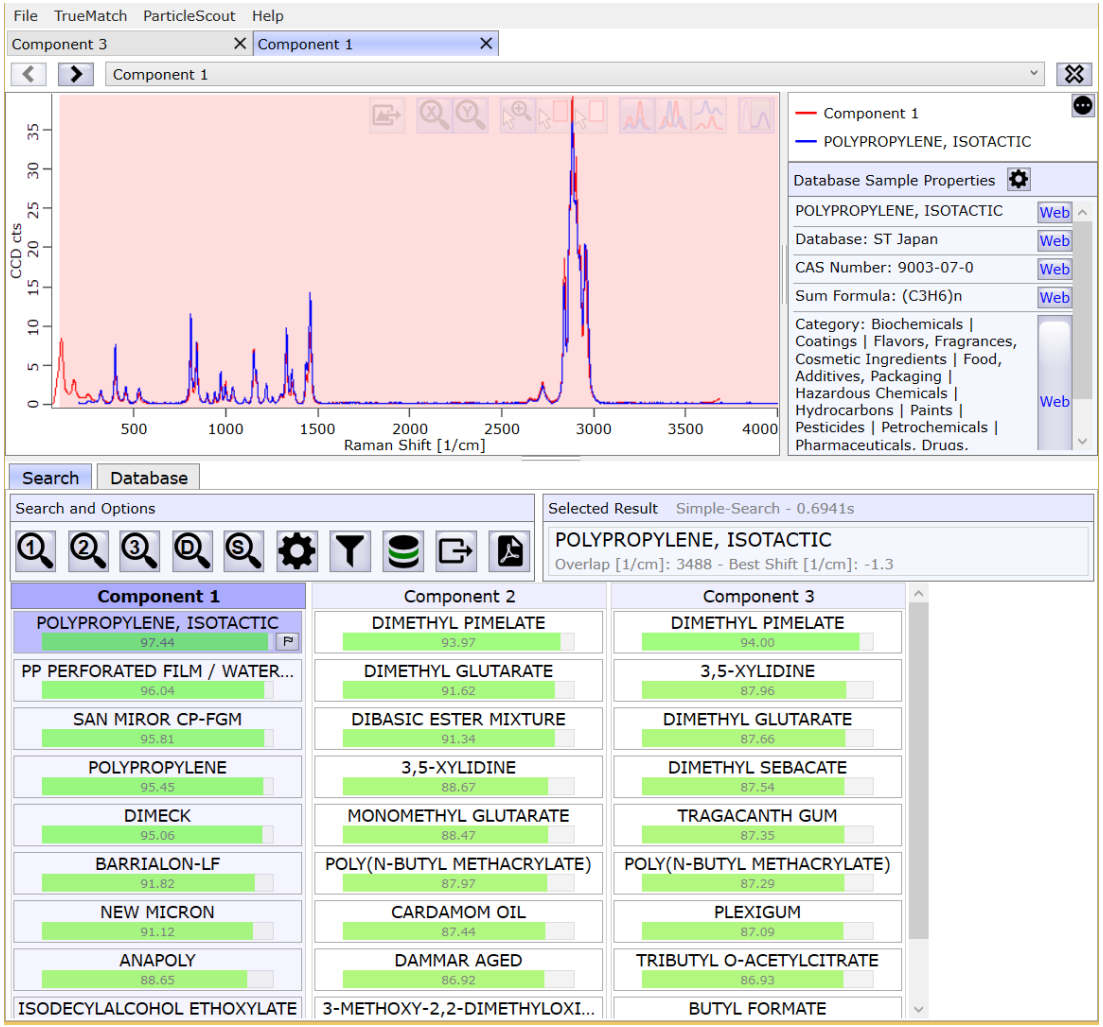




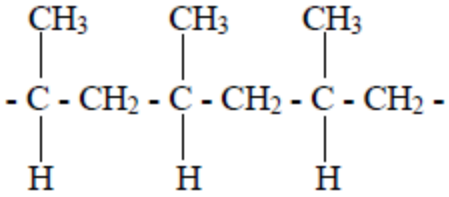

10 µm
EHT = 10.00 kV
WD = 8.6 mm
Signal A = C2D
Mag = 1.28 K X
Width = 89.41 µm
Chamber = 59 Pa

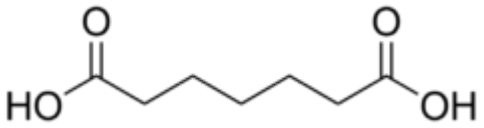

CCD cts
10
20
30
40
50
500
1000
1500
2000
2500
3000
3500
rel. 1/cm

Identification of polymers with True Match



isotactic PP

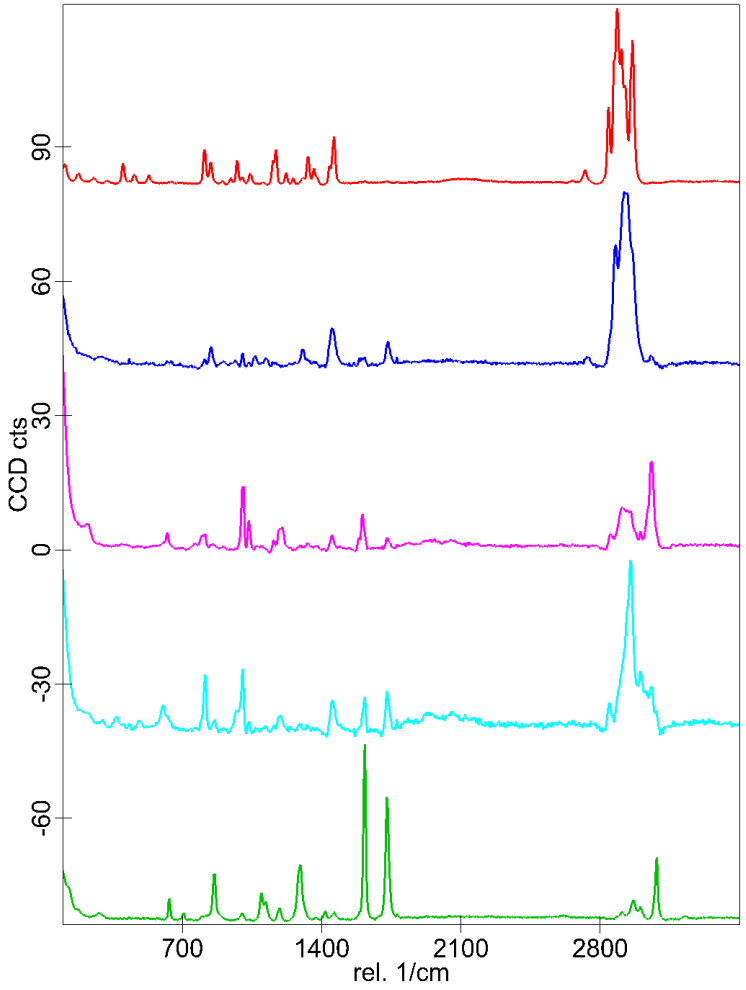


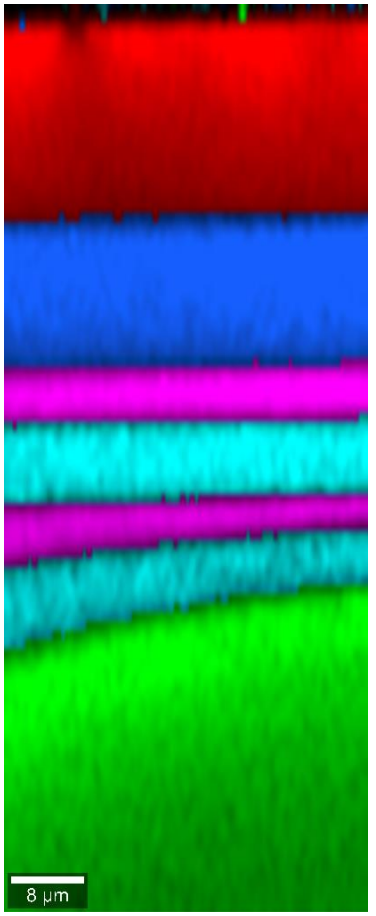


Nondestructive analysis of polymeric foils

Image Depth Scan:

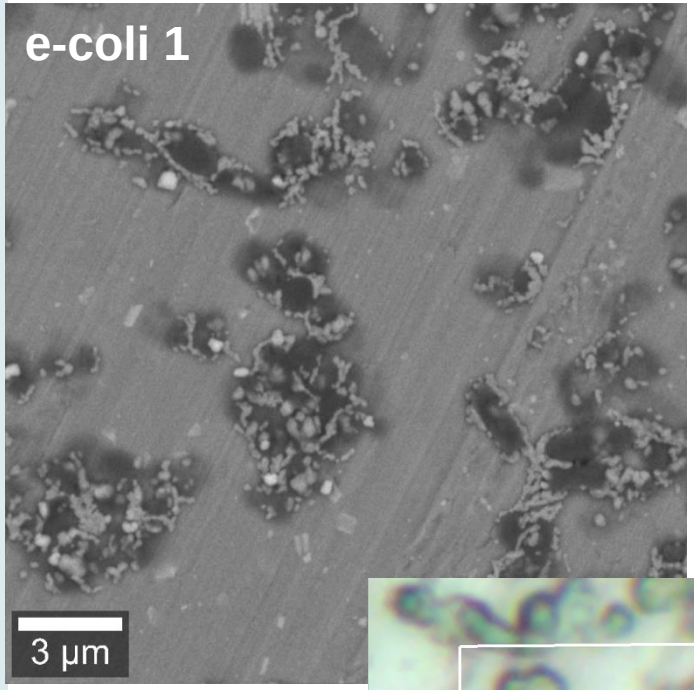
Points per Line:	120
Lines per Image:	100
Scan Width [µm]:	40
Scan Depth [µm]:	100
Integration Time [s]:	0.1

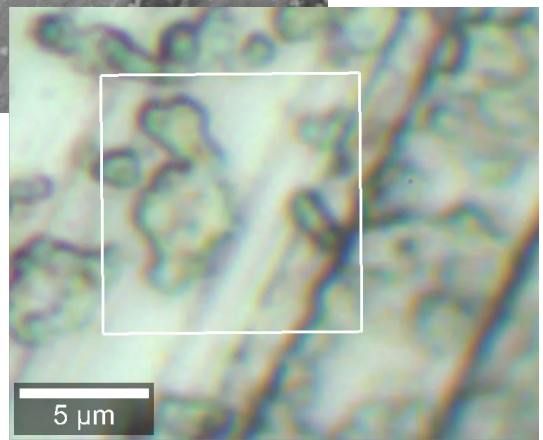


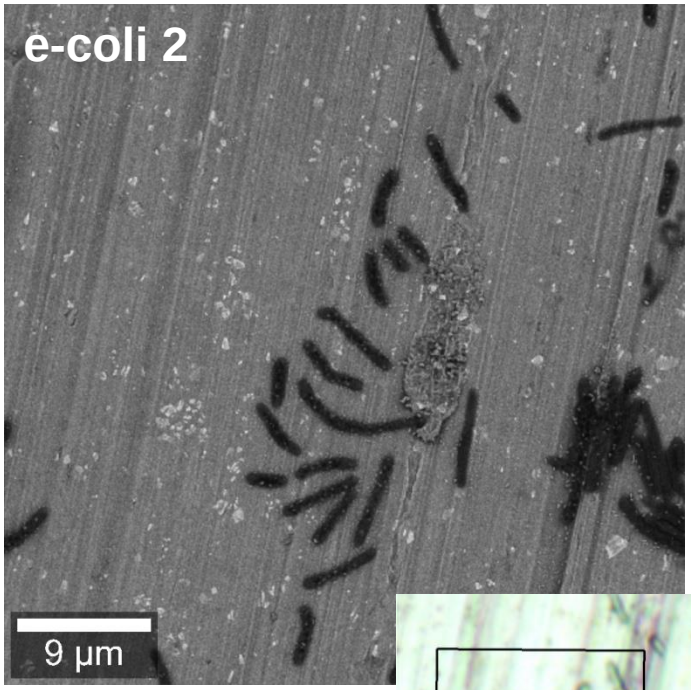


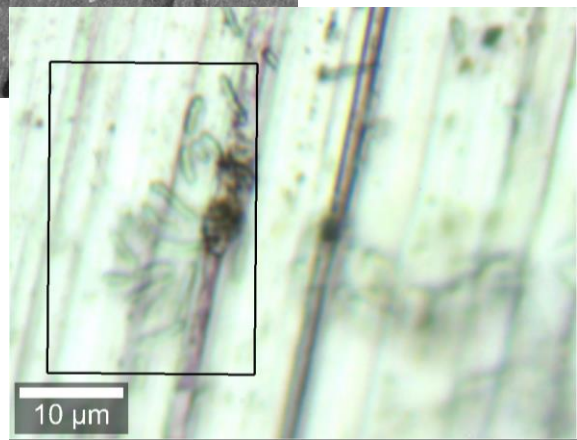
RISE imaging of coli bacteria

Differentiation of e-coli and s-coli bacteria

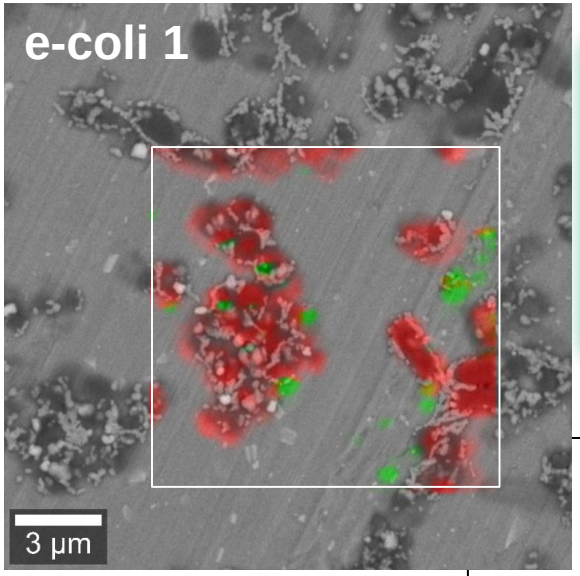






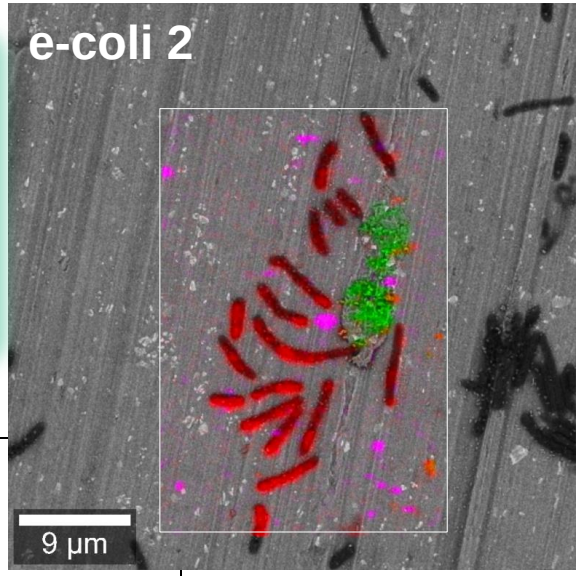


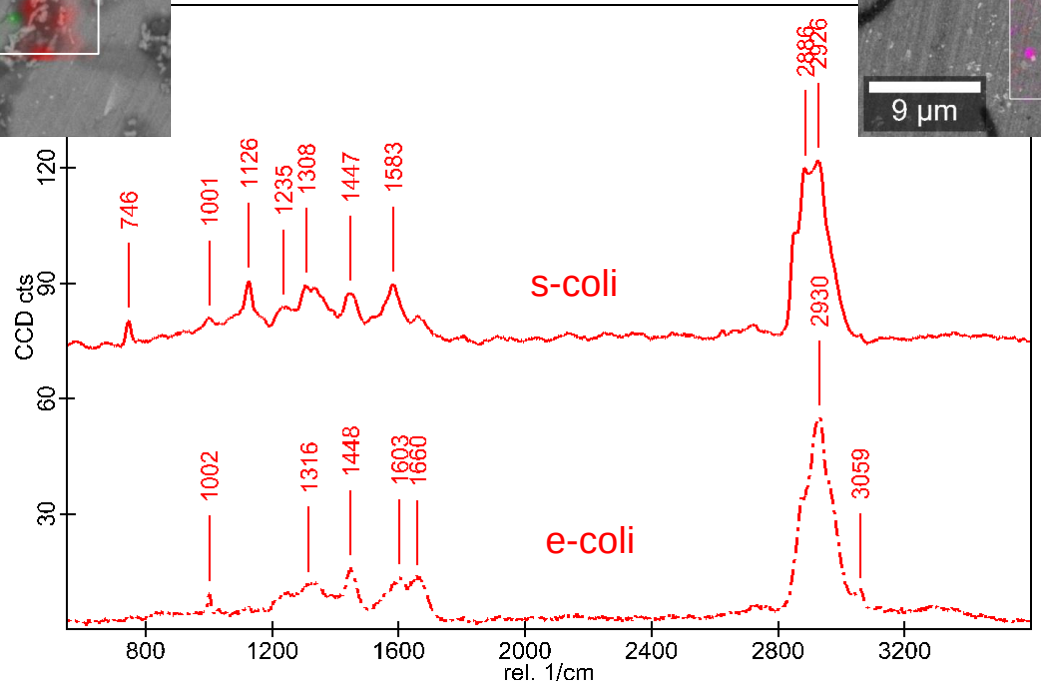
RISE imaging of coli bacteria



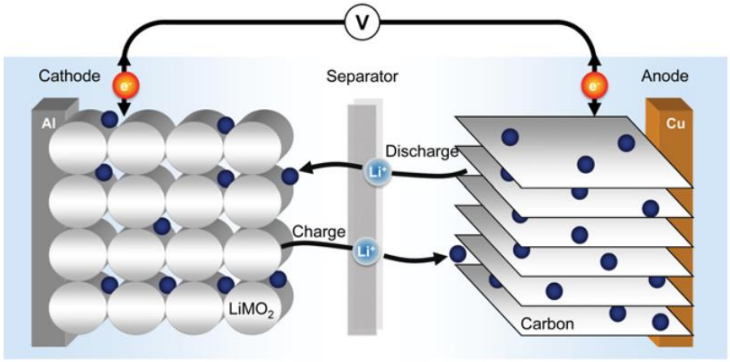
Differentiation of bacteria by:

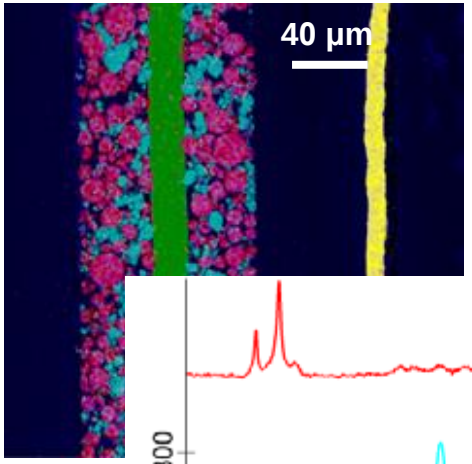
1. Microscopic shape
2. Raman spectra

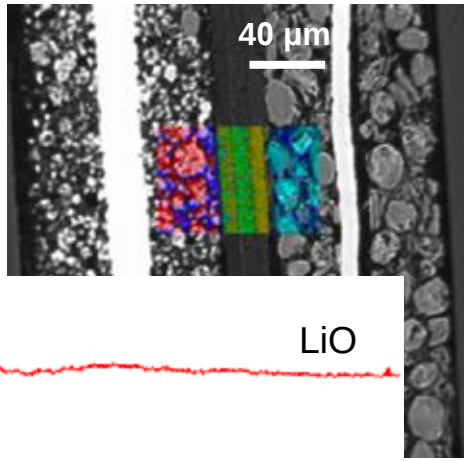


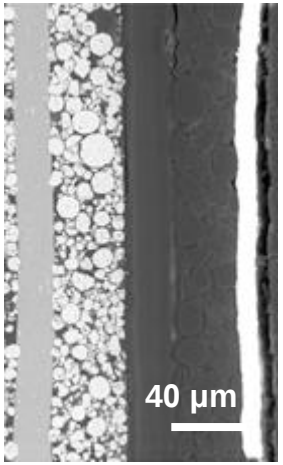


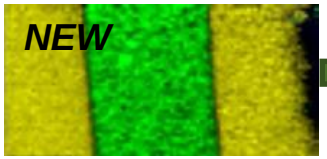
RISE imaging of Li batteries

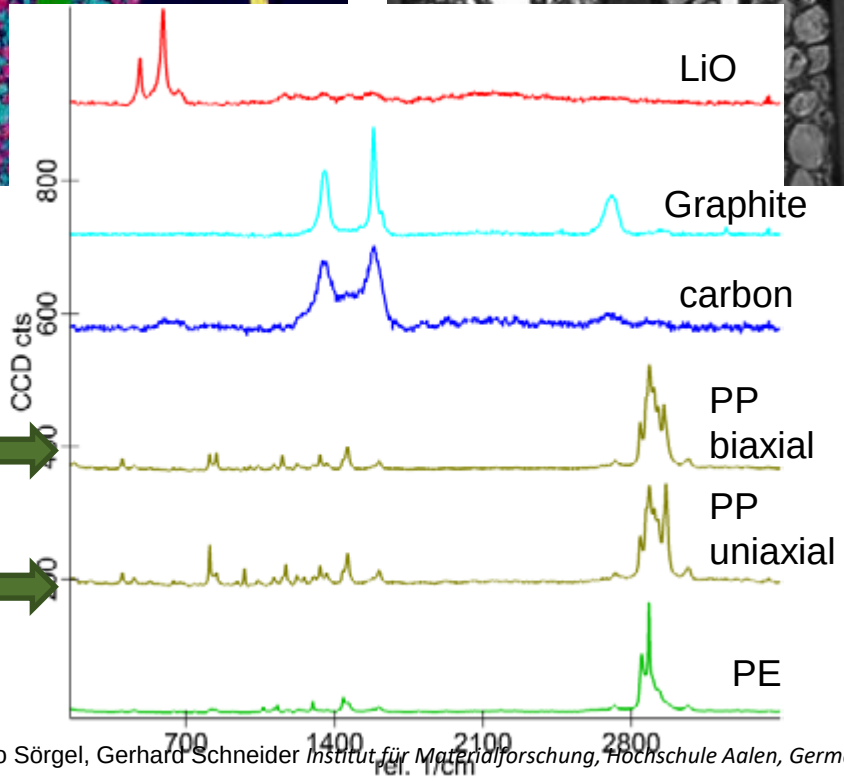






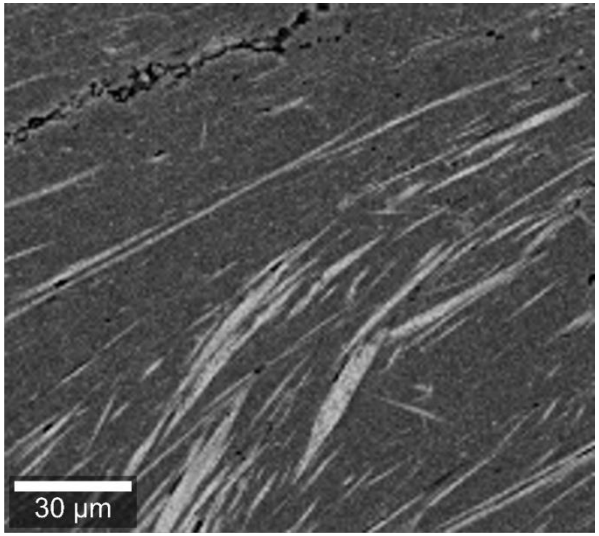




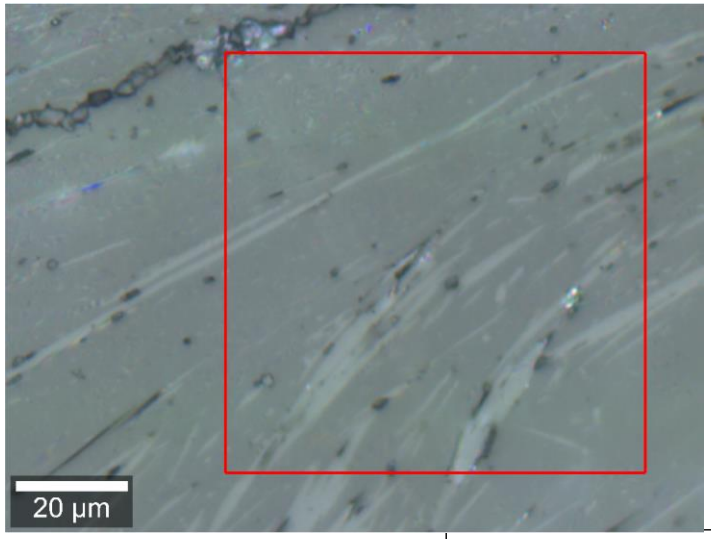


Sample courtesy: Timo Sörgel, Gerhard Schneider *Institut für Materialforschung, Hochschule Aalen, Germany*

RISE imaging of nanometric fibers in quartz (SiO_2)



SEM overview image



white light image

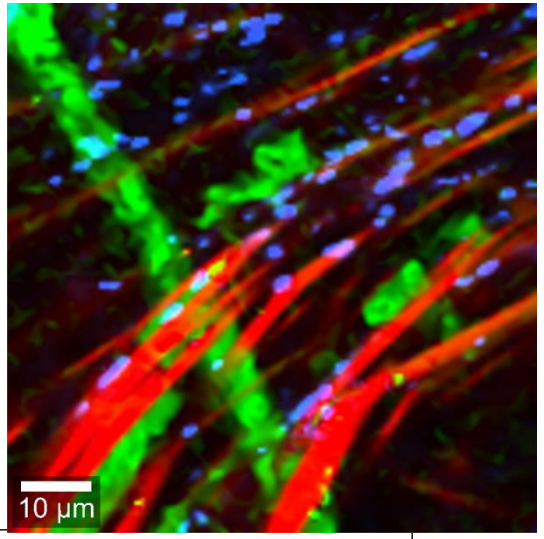
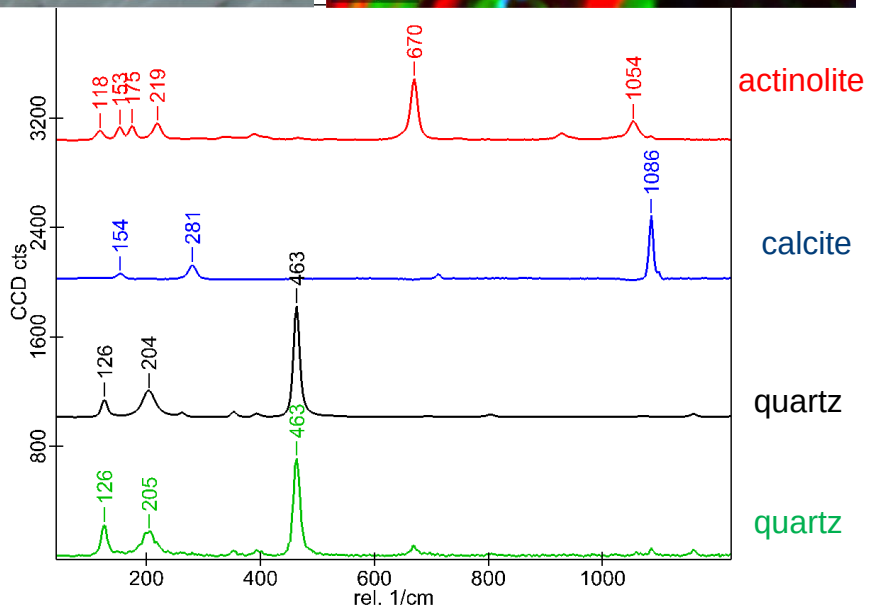


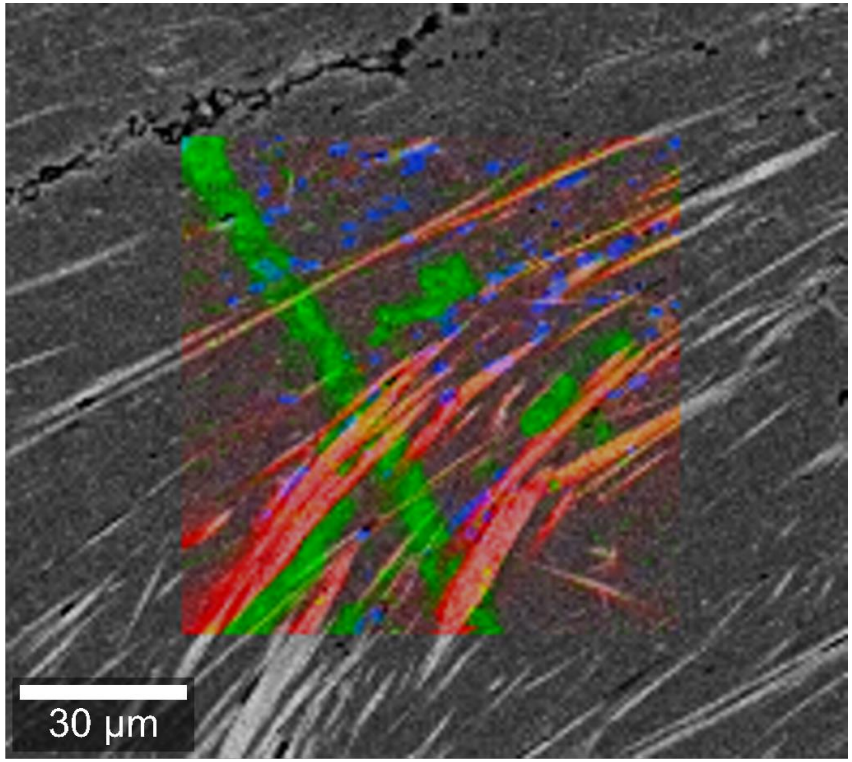
Image Scan:

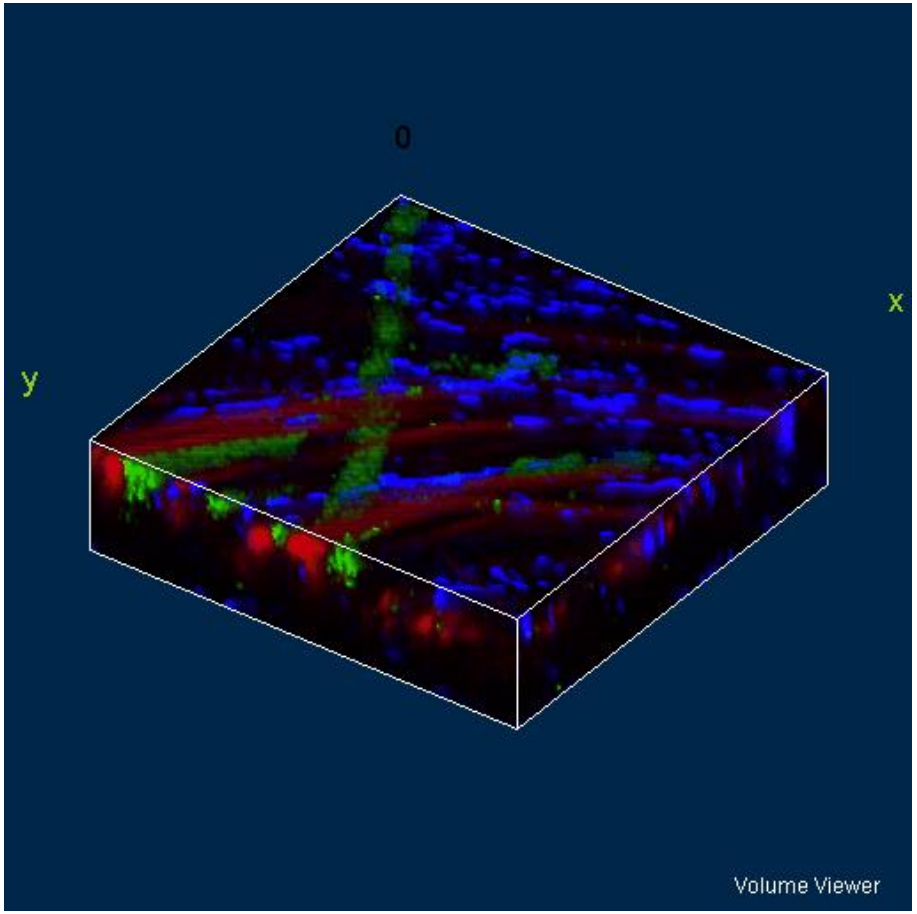
Points per Line:	150
Lines per Image:	150
Scan Width [µm]:	75
Scan Height [µm]:	75
Integration Time [s]:	0.1



Spectra can be found in RUFF database

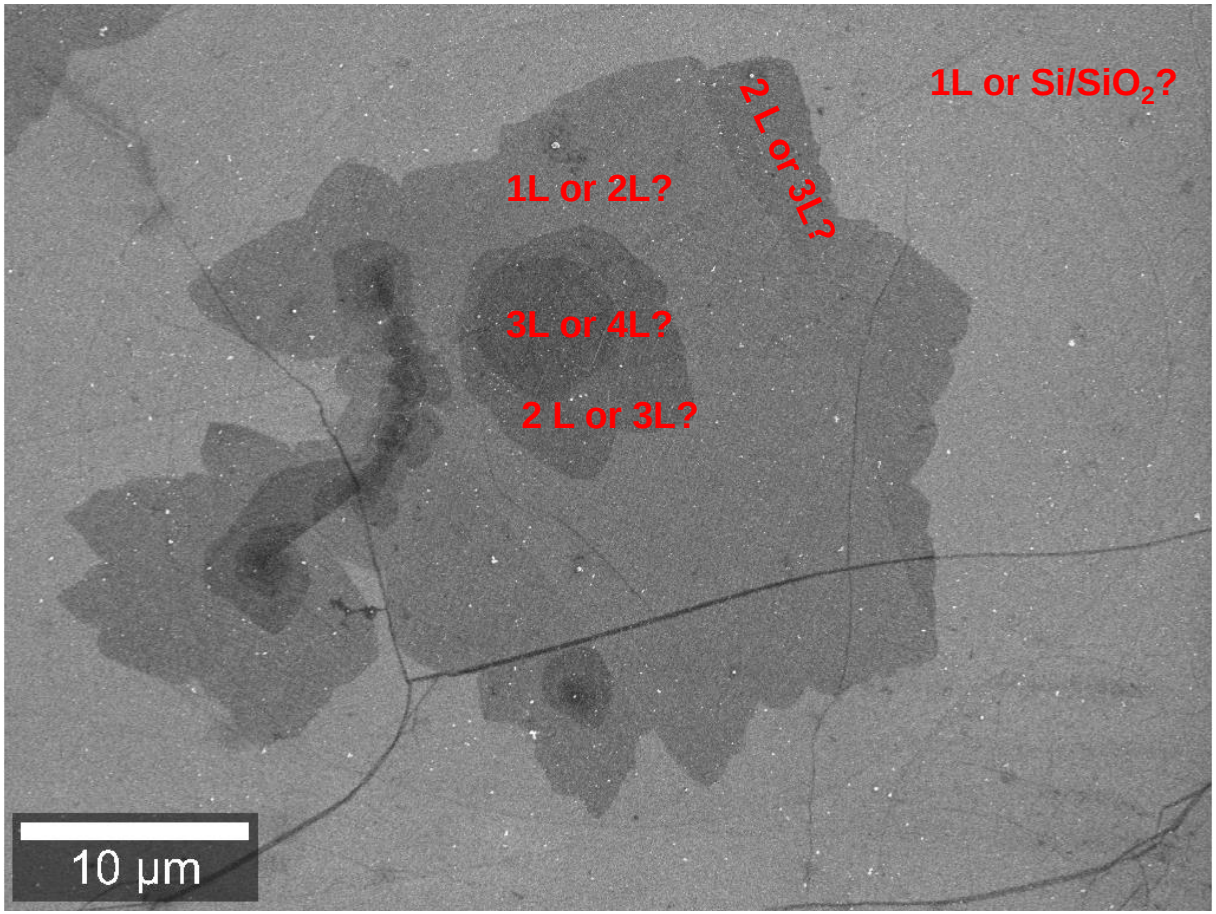
RISE imaging of nanometric fibers in quartz (SiO_2)





RISE imaging of folded CVD graphene on Si/SiO_2

SEM image



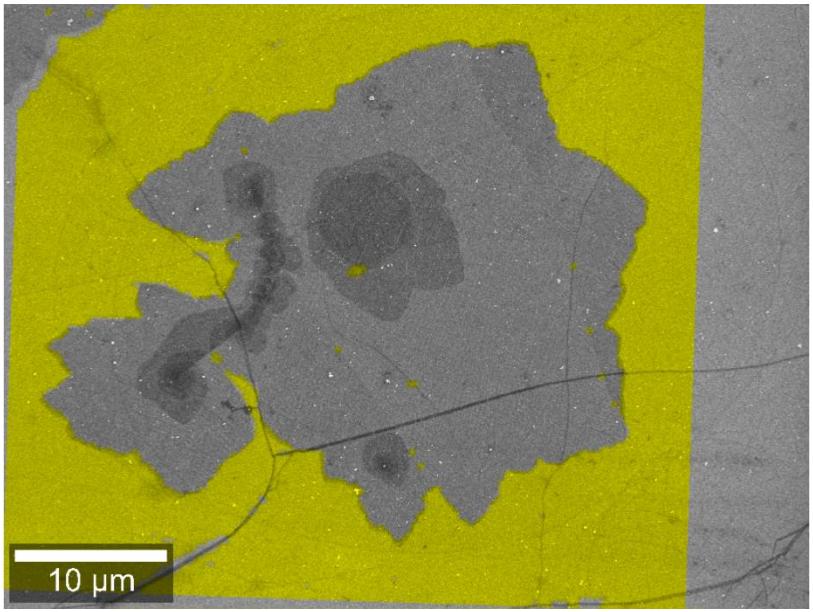
Raman Imaging Parameter:

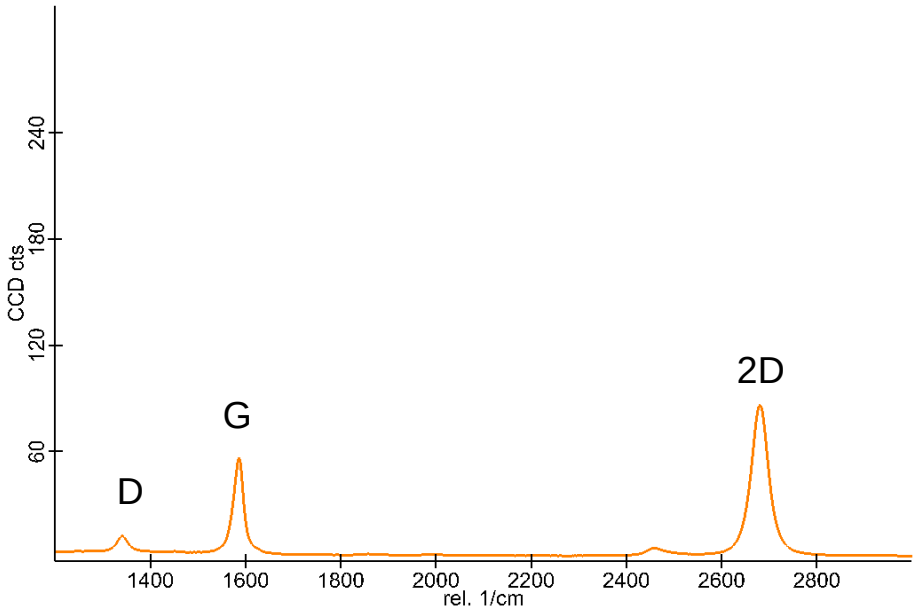
Excitation Wavelength: 532nm
Laser Power : 5mW

Points per Line: 150
Lines per Image: 150
Scan Width [µm]: 45
Scan Height [µm]: 45
Integration Time [s]: 0.05

UHTS600S_VIS:
Grating: 300 g/mm
BLZ=500nm

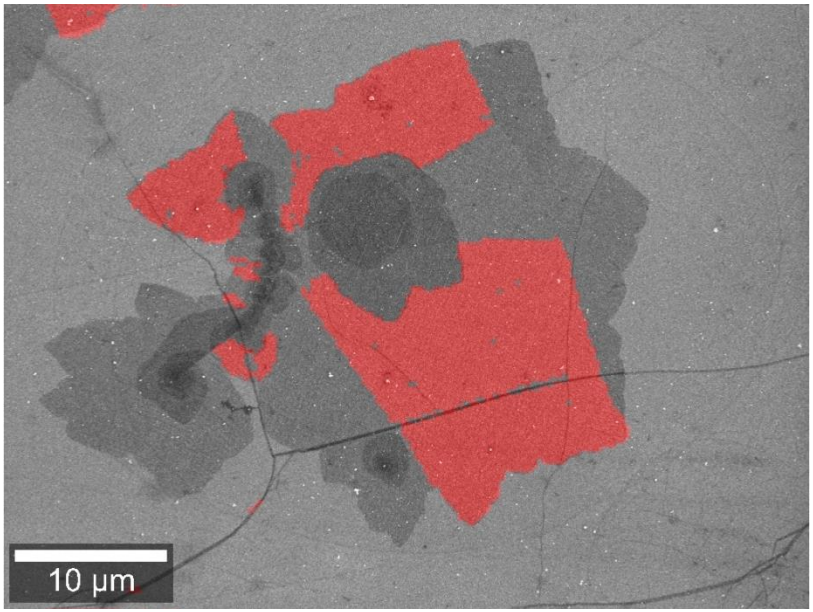
RISE imaging of folded CVD graphene on Si/SiO_2

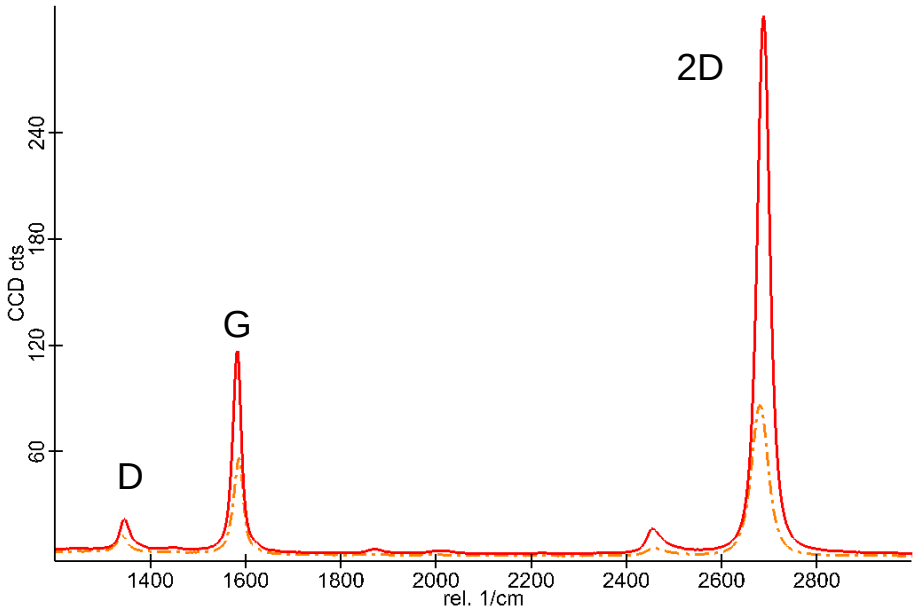




=> 1 layer of graphene

RISE imaging of folded CVD graphene on Si/SiO_2

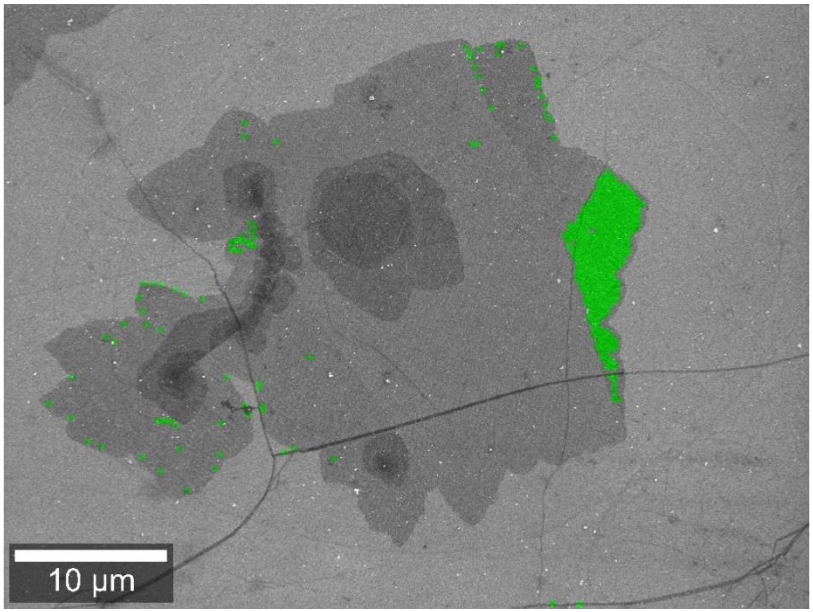


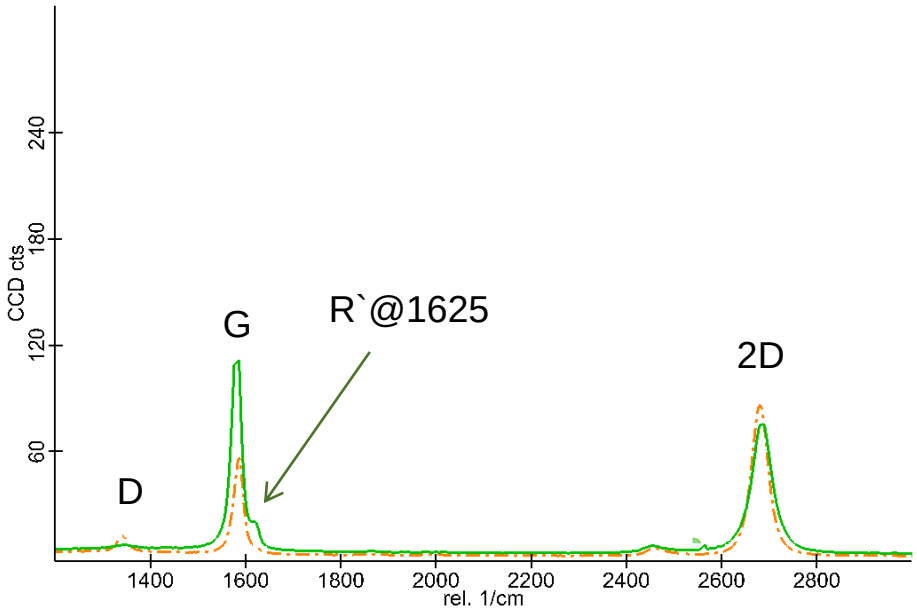


I_G and $FWHM_{2D}$ provide information about number of layers and layer mismatch (twisting)

⇒2 layers of graphene
⇒Twist angle > 20^0

RISE imaging of folded CVD graphene on Si/SiO_2

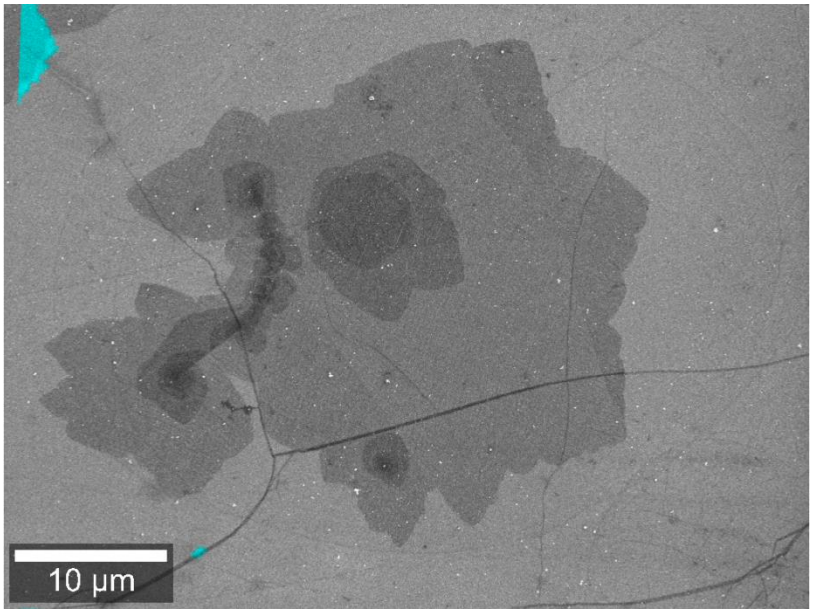


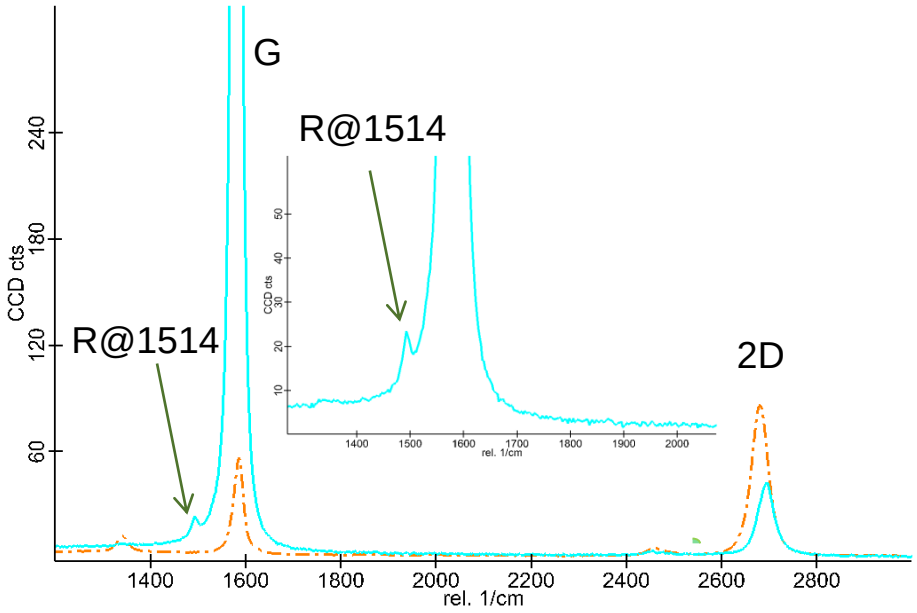


I_G and $FWHM_{2D}$ provide information about number of layers and layer mismatch (twisting)

⇒2 layers of graphene
⇒Twist angle 3-8^0

RISE imaging of folded CVD graphene on Si/SiO_2



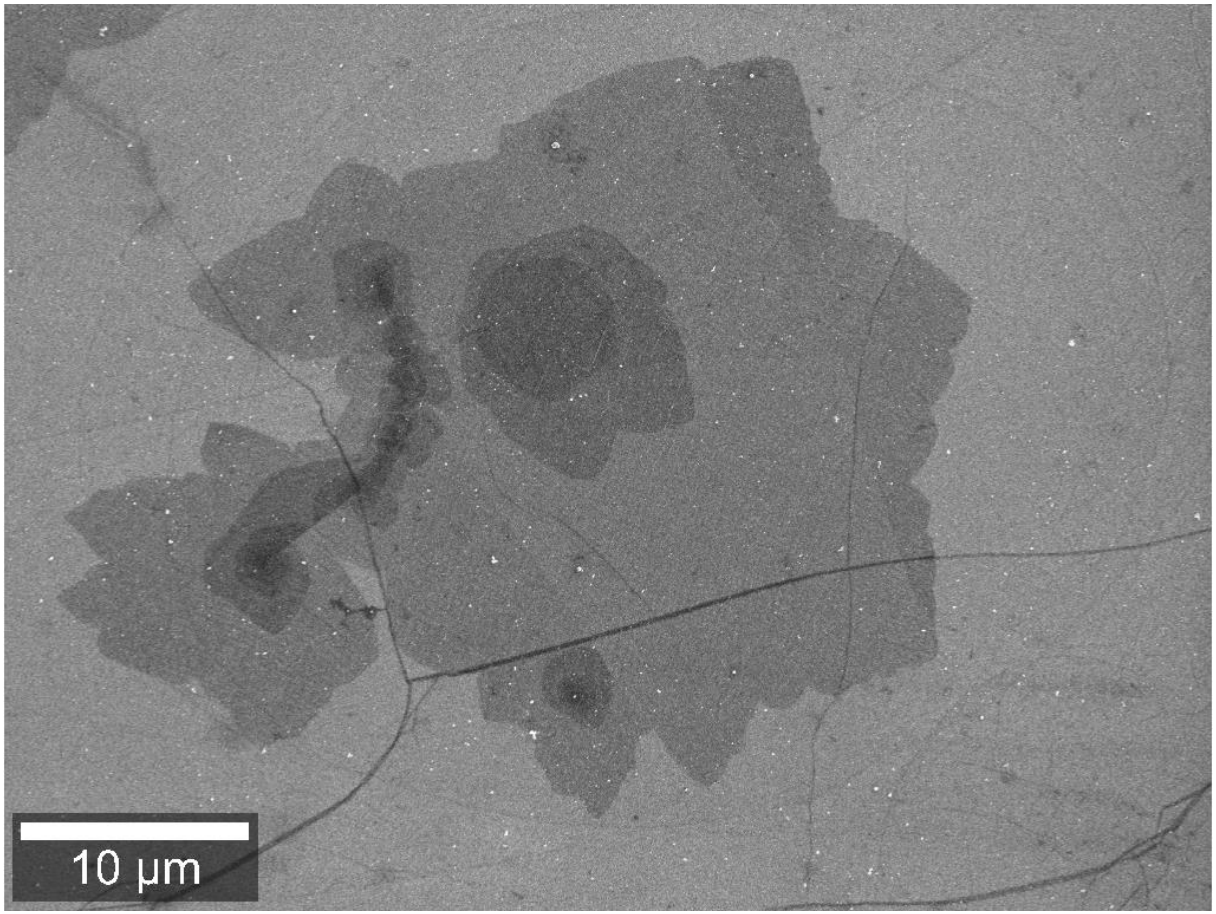


I_G and $FWHM_{2D}$ provide information about number of layers and layer mismatch (twisting)

⇒2 layers of graphene
⇒Twist angle = 12^0

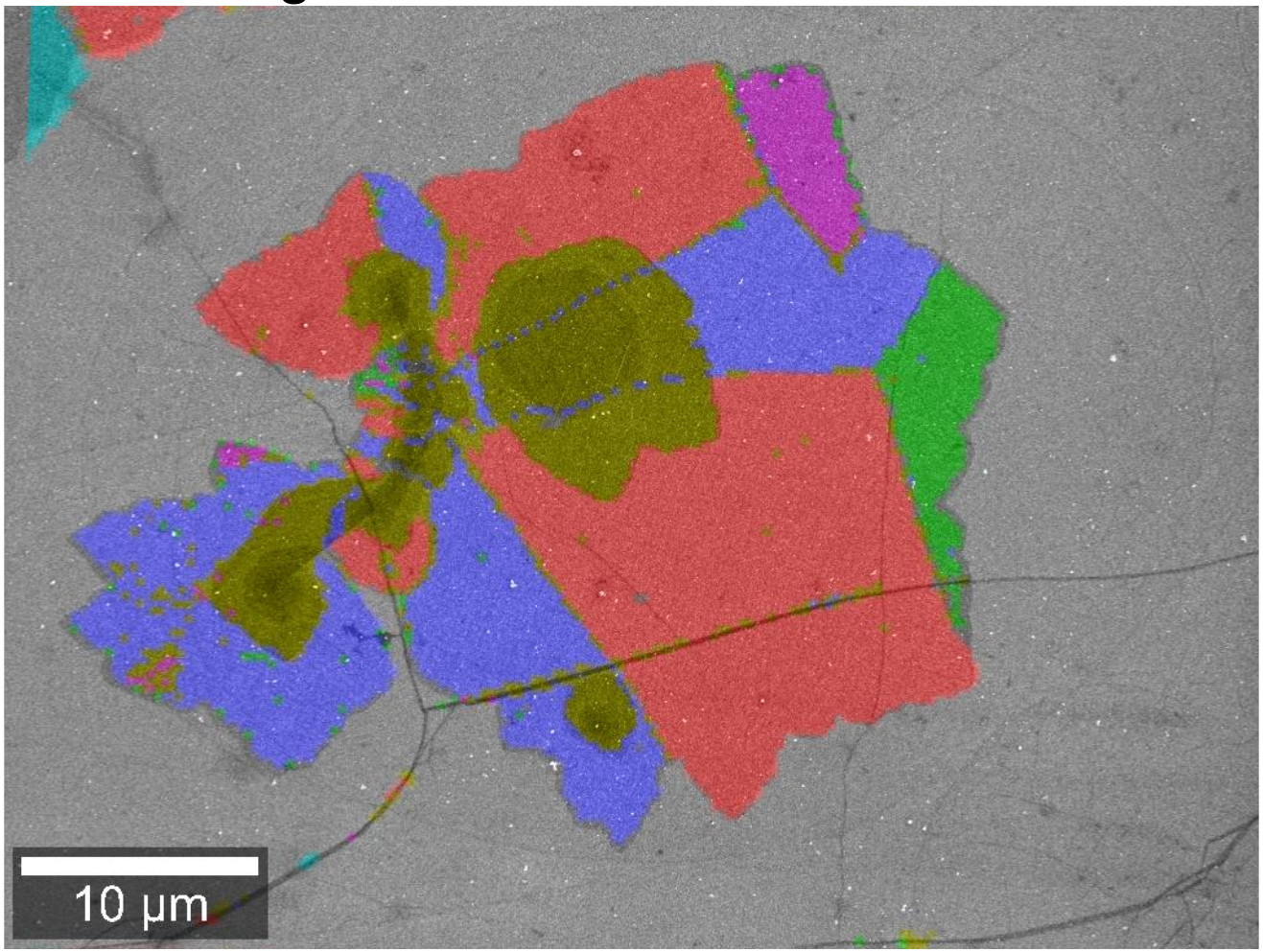
RISE imaging of folded CVD graphene on Si/SiO_2

SEM image



RISE imaging of folded CVD graphene on Si/SiO_2

RISE image



Monolayer

2L:~AB

2L: Rotation >20

2L: Rotation 3-8

2L: Rotation 0-3

2L:Rotation 12

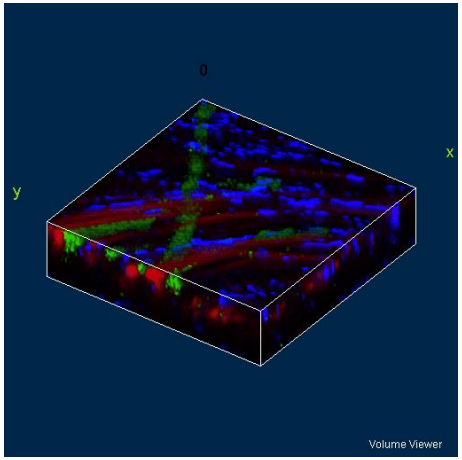
More than 2L

Y. Chen et all, Phys. Chem. Chem. Phys., **16**, 21682 (2014)

Summary

- The instrument and techniques:

  Raman Imaging and SEM = RISE Microscopy



- RISE applied to polymer research

- RISE applied to Li-oxide particles

- RISE applied to graphene research