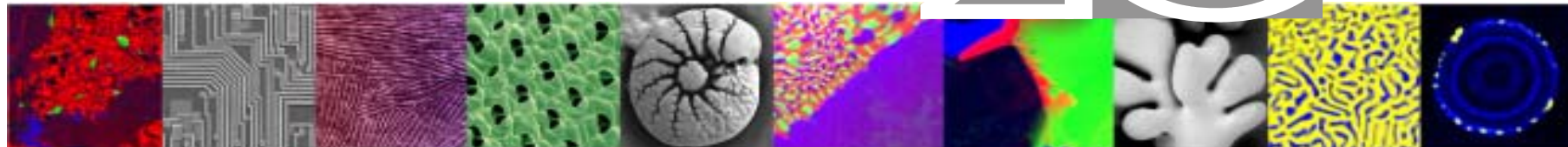


# Imaging Methods Applied to Polymer Blends

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## Equipment at FELMI-ZfE Graz:

### *Spectrometer:*

Bruker Equinox 55 (rapid and step scan)

MIRacle ATR Accessory (diamond and Ge)

### *Microscope:*

Bruker Hyperion 3000 with MCT spot detector (middle band) and 64\*64 elements FPA detector for imaging, motorized xyz stage, polarizers

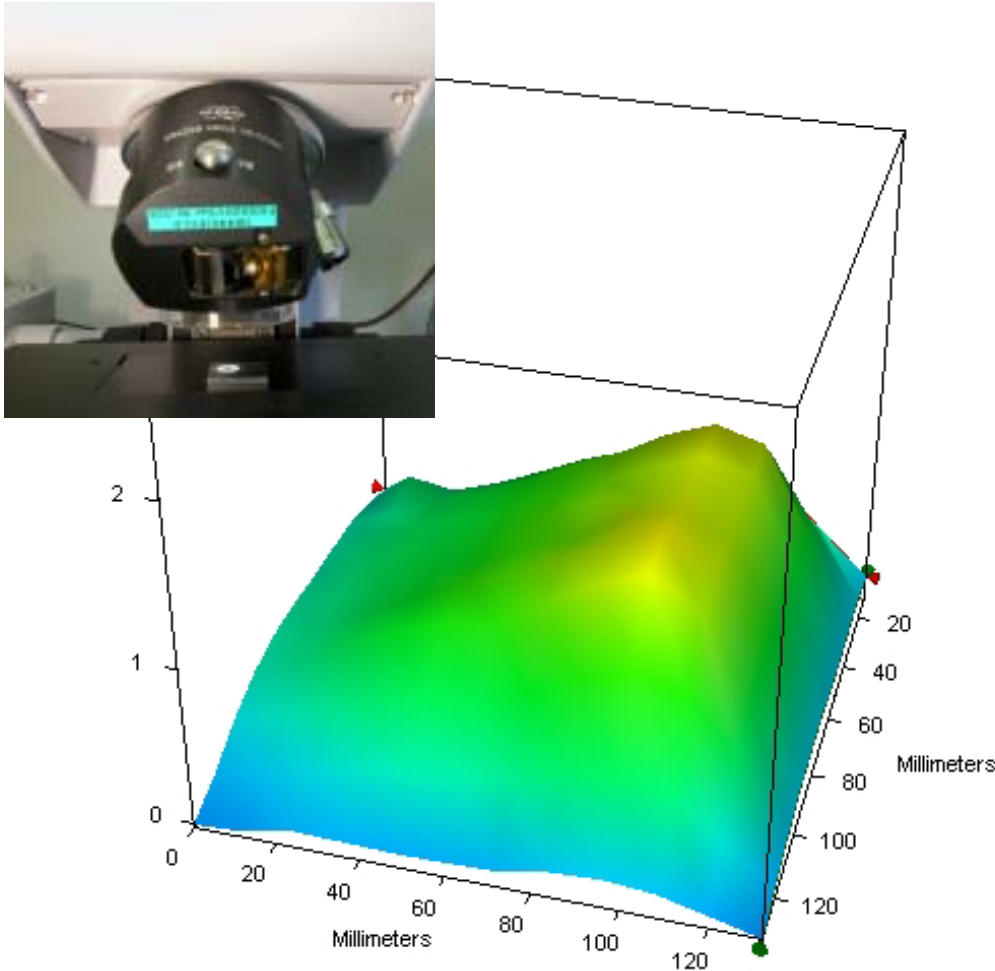
### *Objectives:*

reflection and transmission, ATR (Ge), Grazing Angle

**FPA detector:** 4\*4 mm<sup>2</sup>; 4096 elements.

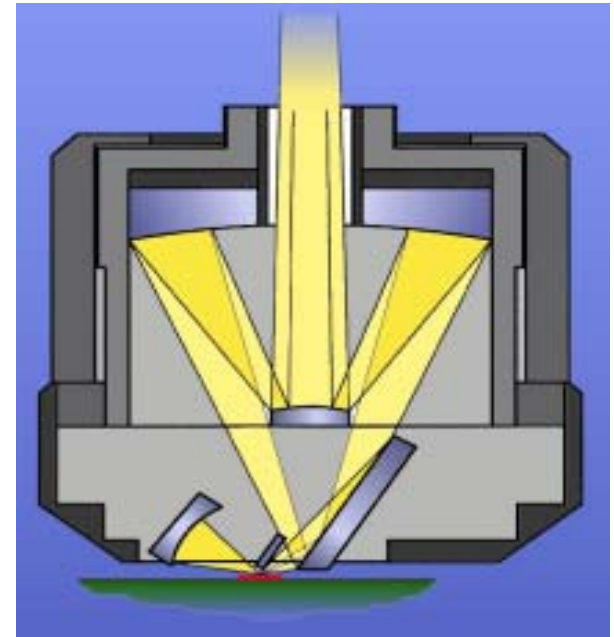
**Imaging area on sample:** 266\*266 μm<sup>2</sup> in transmission mode (15x objective) and 50\*50 μm<sup>2</sup> for ATR mode (20x objective, Ge crystal).



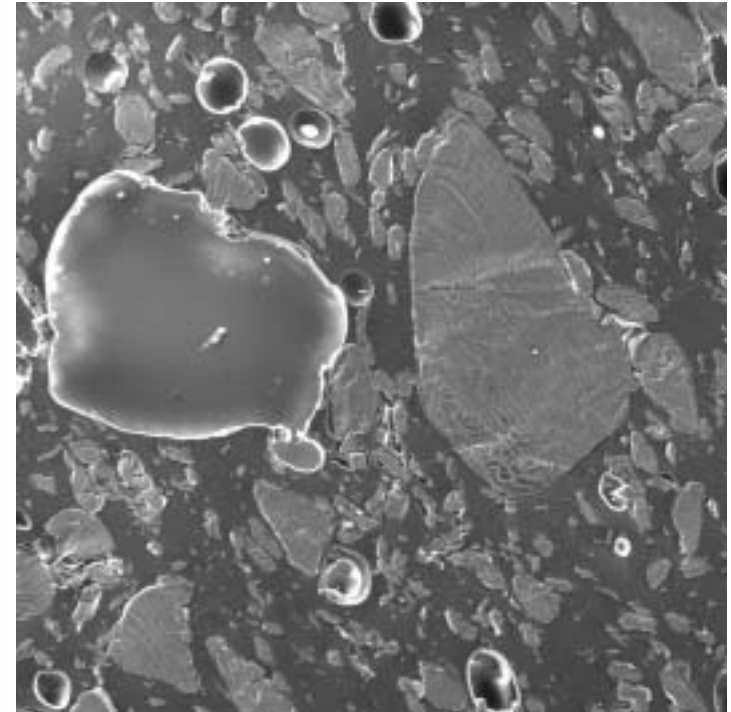
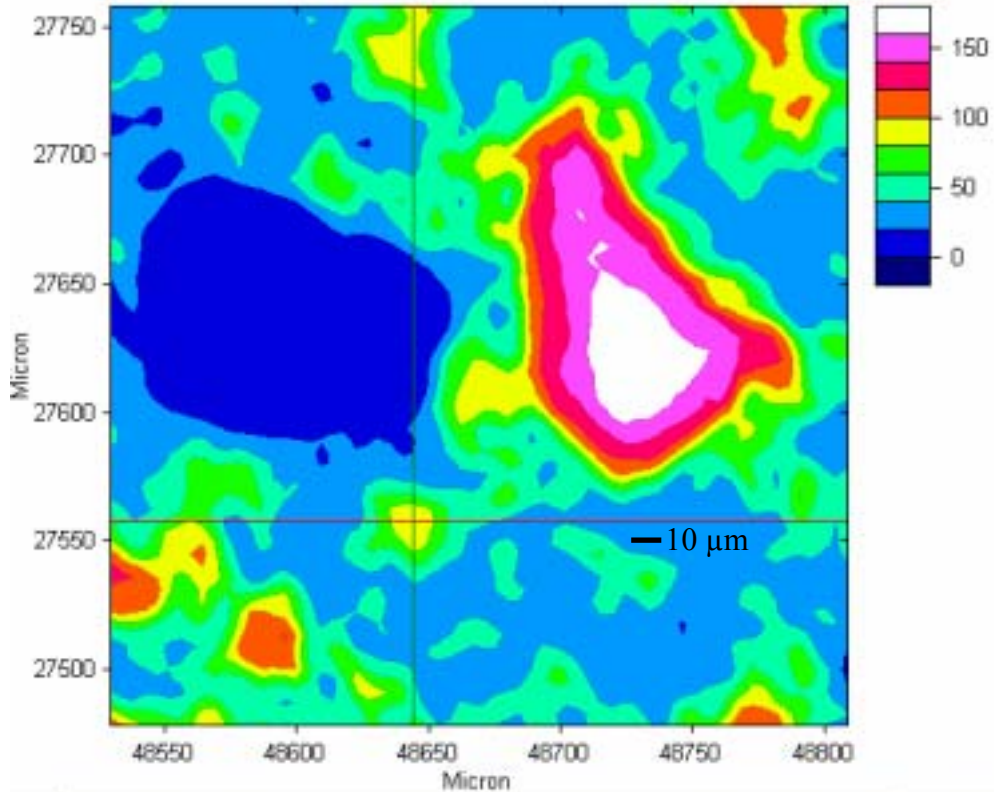


Mapping of a thin polymer film wedge (20 – 60 nm) with the GIR objective.

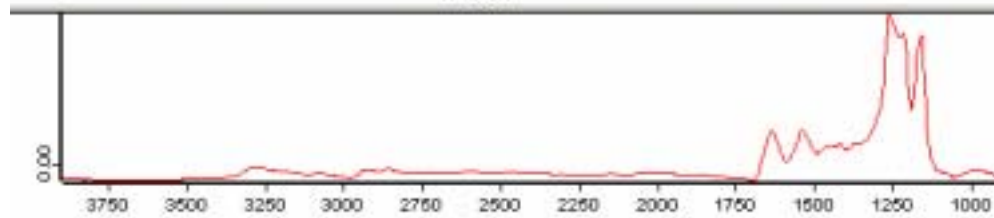
144 points measured (area 2\*2 cm<sup>2</sup>) with 200 scans each; ca. 4 hours total measuring time.



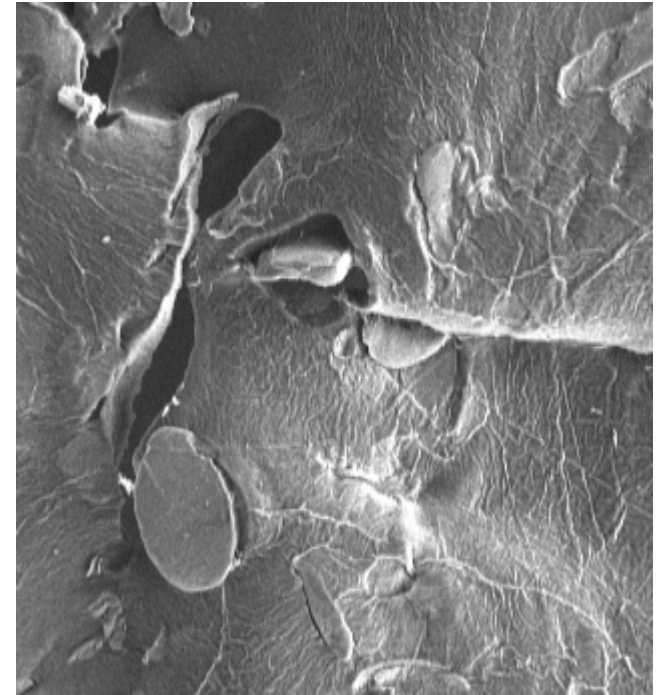
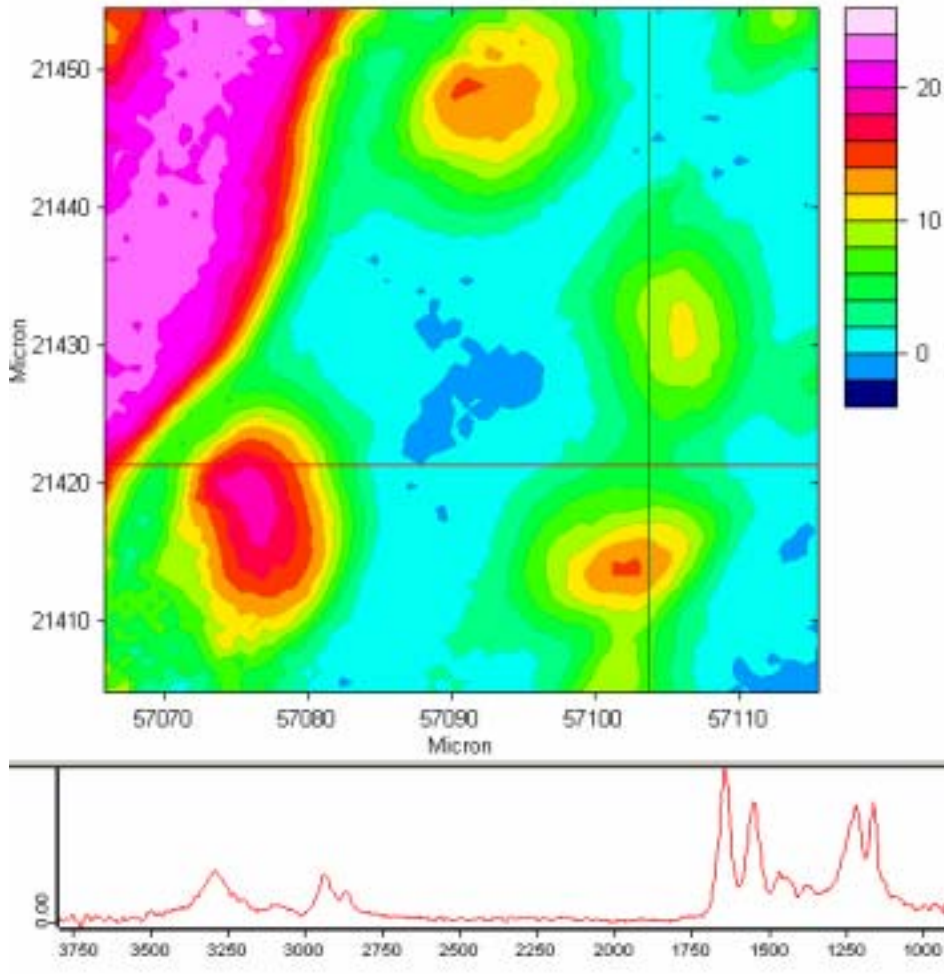
- Mapping is time consuming (higher costs, loss of stability)
- Spatial resolution is limited by diffraction at apertures
- Better reproducibility (no stage/sample movement)
- Kinetic experiments with FPA detector possible
- Sample area: depending on detector array size (more flexible with mapping)
- High computing power needed for imaging (fast readout and processing, data storage); but now measurements can be performed in rapid scan mode (rather than in step scan mode)



IR image (PTFE bands integrated) and SEM image (same details); PTFE clusters of 8 - 10  $\mu\text{m}$  can be detected.



Measuring conditions: 8  $\text{cm}^{-1}$  spectral resolution, 10 scans (continuous scan), scan time ca. 3 minutes.

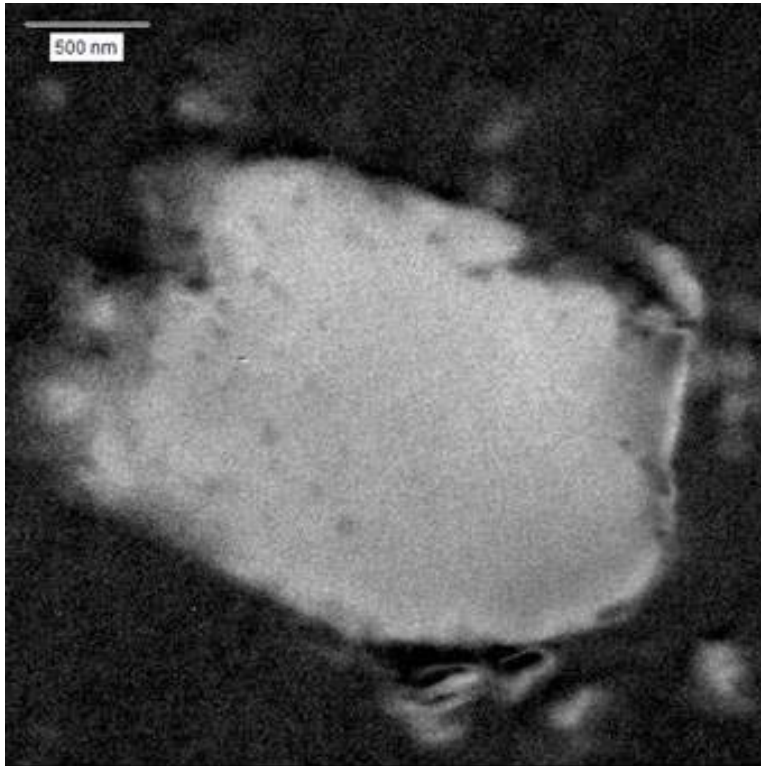


Comparison of IR-ATR and SEM images (same area). IR spectrum of a region between two adjacent clusters.

Sample was partly destroyed during ATR measurement (PTFE clusters torn out).

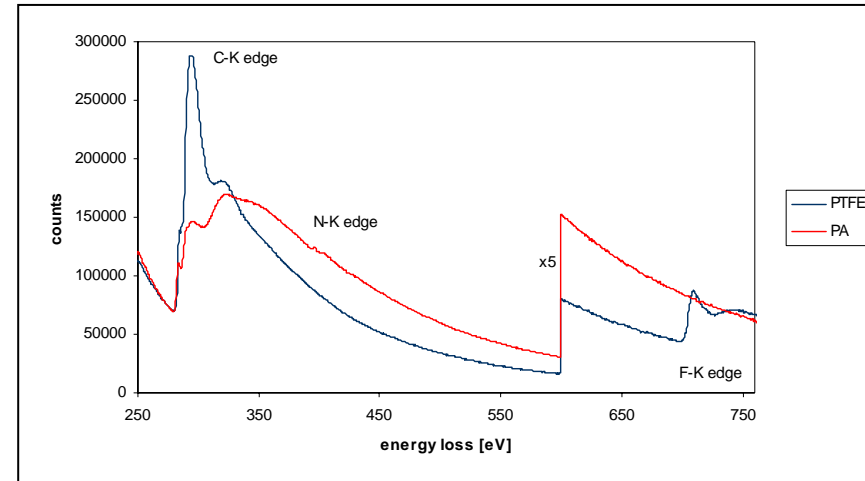
Measuring conditions:  $8 \text{ cm}^{-1}$  spectral resolution, 20 scans (continuous scan), scan time ca. 6 minutes.

# PA-PTFE Blend: TEM/EELS



**EELS** (Electron Energy Loss) Spectroscopy:

1. elemental distribution
2. binding information (ELNES).

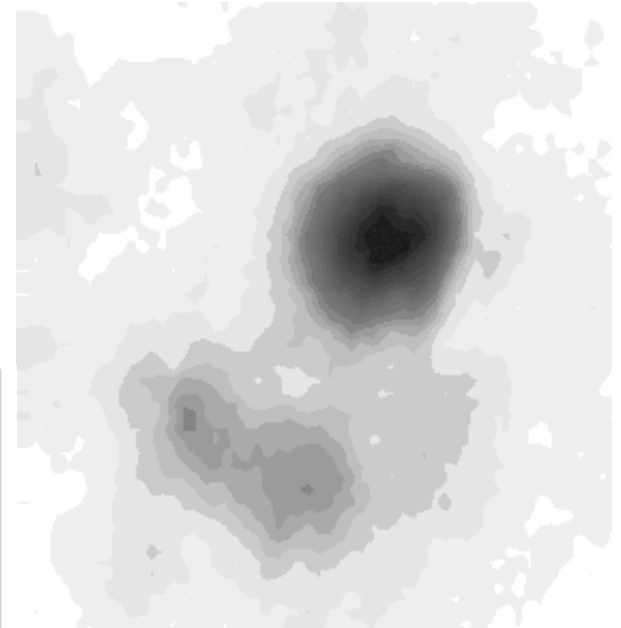
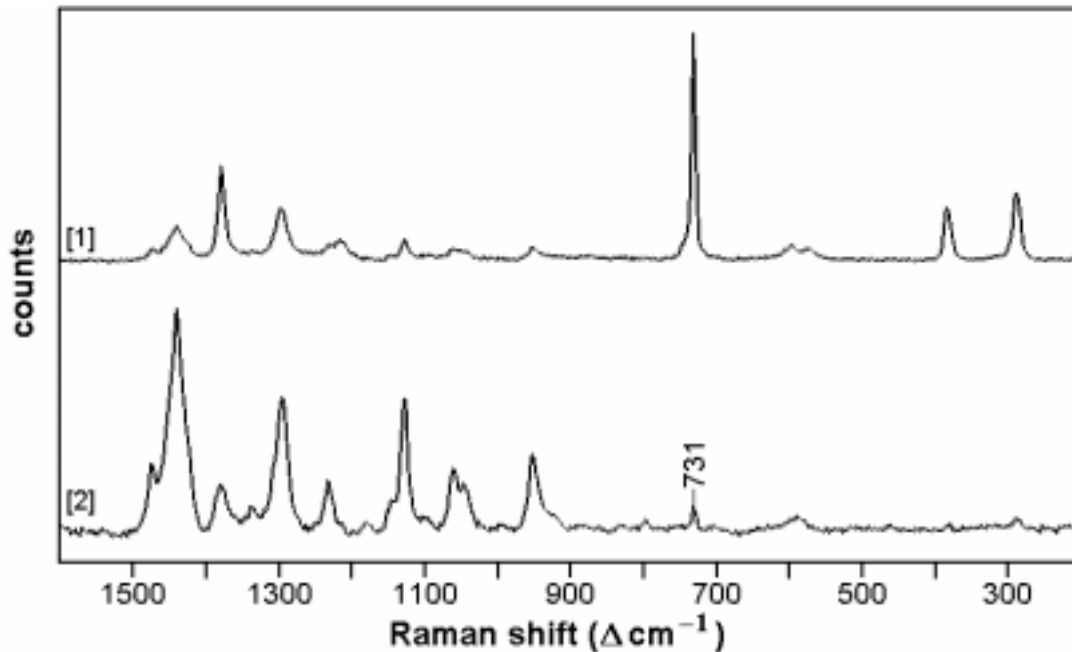


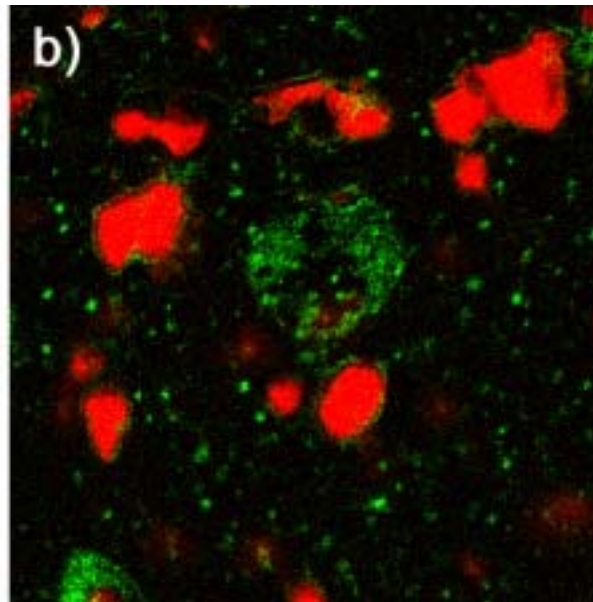
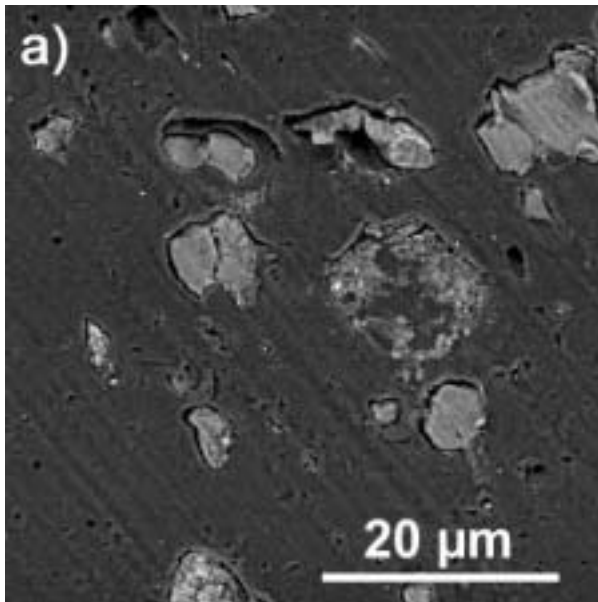
Elemental distribution map of fluorine in the PA / PTFE compound obtained by the 3 window method

Analytical TEM: Philips CM 20, 200 kV, Gatan imaging filter (HRGIF)

**Raman image** created with 731  $\text{cm}^{-1}$  line of PTFE (image size: 20  $\mu\text{m}$  \* 20  $\mu\text{m}$ ).

Raman microscope: Renishaw System 2000; HeNe laser (633 nm); measuring time: 10 minutes.

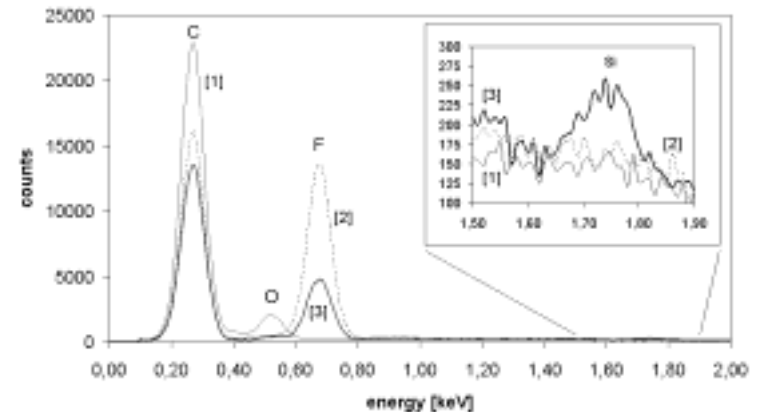




SEM images (a) and EDXS elemental mapping (b):

**fluorine:** red

**silicon:** green



- Samples:
  - IPF Dresden (Klaus-Jürgen Eichhorn, Gisela Pompe, Dieter Lehmann)
  - IB Steiner, Spielberg
- Preparation, measurements:
  - Bruker Optics Germany (Nikola Johner, Alexander Piry)
  - ZfE Graz (Elisabeth Ingolić, Mario Schmied, Peter Pölt, Sanja Šimic, Gerald Kothleitner)
- Financing:
  - FFF and RFTE Vienna



# References

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- Morphology of a PA / PTFE blend studied by Raman imaging; Andreas Gupper, Peter Wilhelm, Mario Schmied and Elisabeth Ingolic; Macromol. Symp. 184, 275-285 (2002).
- Degradation of Poly (vinyl chloride) Studied by Micro Raman Spectroscopy; A. Gupper, P. Wilhelm, M. Schiller; Addcon World 2002, Budapest, Conference Proceedings, pp. 227-235 (2002).
- Raman Microscopic Studies on the Orientation and Crystallinity of Polypropylene; A. Gupper, P. Wilhelm, P. Zipper, D. Gregor-Svetec, M. Gahleitner; Proc. XVIIIth International Conference on Raman Spectroscopy, Budapest, J. Mink et al. (eds.), Wiley, pp.697-698 (2002).
- Imaging Methods Applied to Polymer Blends, P. Wilhelm, A. Gupper, M. Schmied, G. Kothleitner; FACSS 2003, 239 (2003).
- Investigations of Thin Polymer Films with Vibrational Spectroscopic Techniques; FACSS 2003, 549 (2003).

- Scanning electron microscopy, micro scale analysis, X-ray spectrometry (EDXS, WDXS), ESEM
- High resolution transmission electron microscopy, nano scale analysis, EELS, EFTEM, electron diffraction
- Focused ion beam equipment (nano structures, preparation)
- FT-IR and Raman micro spectroscopy, light microscopy



**Graz:** Cultural Capital of Europe 2003; beautiful ancient and modern architecture; nice landscape; charming people; prominent scientists, artists ...



*Gustav Adolf Smekal  
predicted the Raman  
effect in Graz (1923)*

