

Leistungsstarke Analysemethoden: Konfokale Raman-Spektroskopie und Röntgenbeugung

15 Februar 2024 – Edith Perret



Empa

Swiss Federal Laboratories for Materials Science and Technology



3 sites



End of 2022:

1021 employees
577 scientists
401 tech/admin staff
43 trainees

869 ISI publications
226 PhD studies running

Departments

- Advanced materials & surfaces
- Engineering sciences
- Energy, mobility & environment
- **Materials meet life**

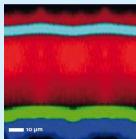
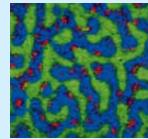
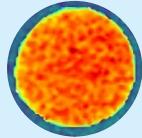
Laboratory of Advanced Fibers, Center for X-ray Analytics

New high-resolution confocal Raman imaging instrument



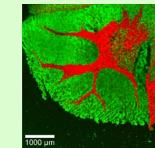
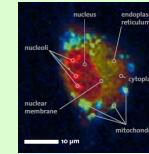
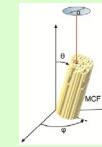
Polymer Science:

Fibers, Thin Films, Coatings...



Life Science:

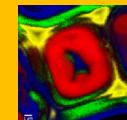
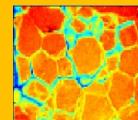
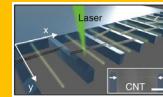
Bone, Cells, Tissues...



MICROSTRUCTURE

Materials Science:

CNTs, Ceramics, Wood...

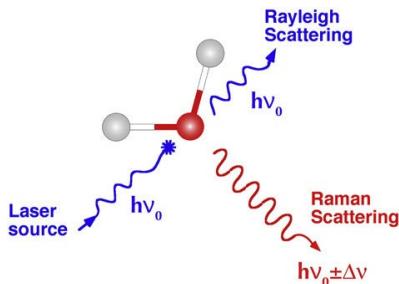




How does it work ?

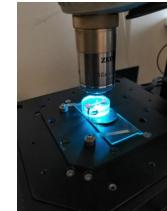
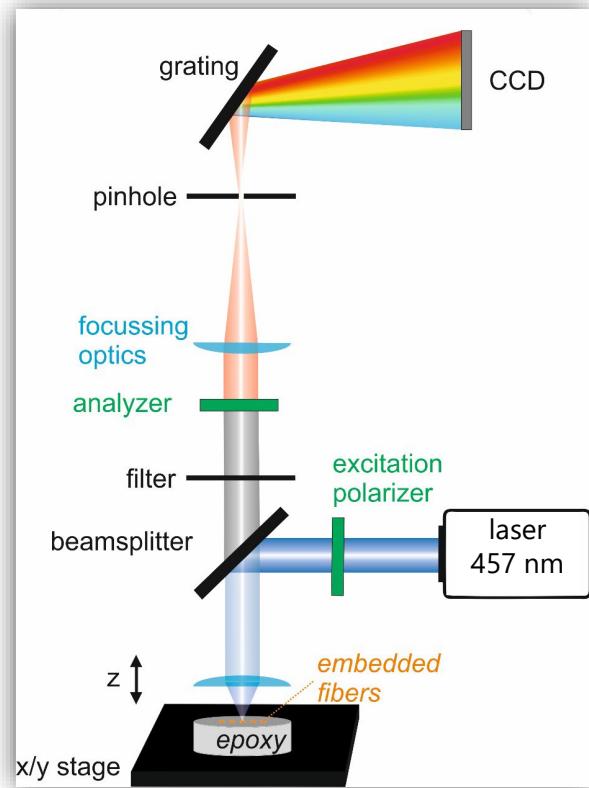
Raman effect:

Inelastic scattering
of photons by matter.



Acquisition modes:

- Single spectra
- 2D scans
- 3D mapping
- Polarized



WITec alpha 300 R

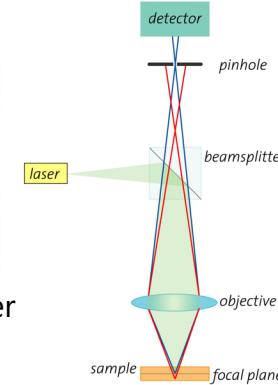
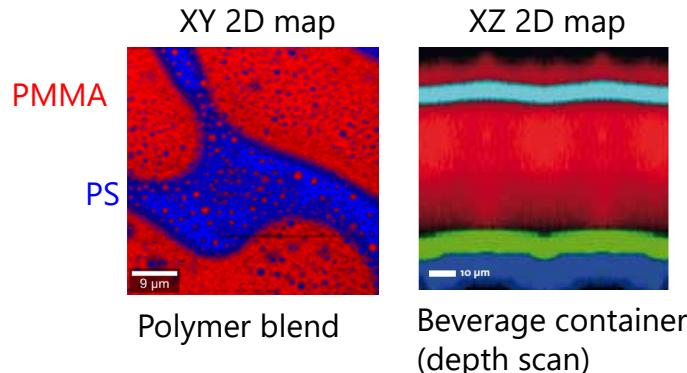
Lateral resolution: ~ 300 nm
Depth resolution: ~ 1 μm



What can it be used for?

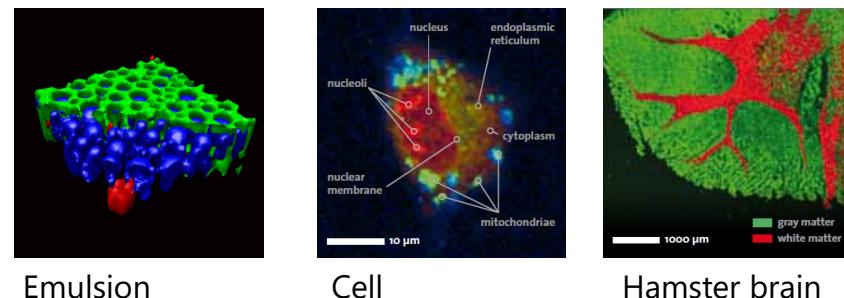
Polymer science:

- Types of polymers
- Crystallinity
- Molecular orientation
- Existence of specific bonds



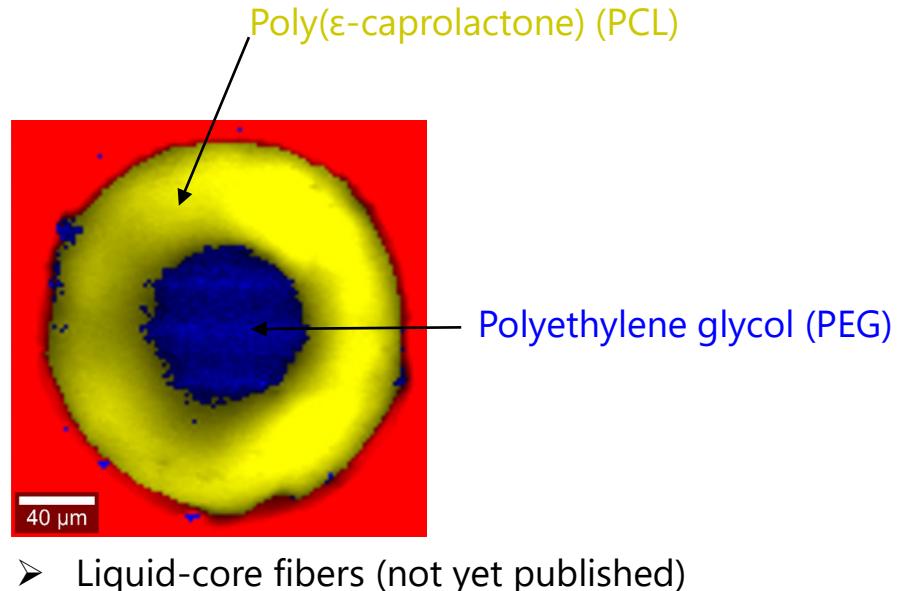
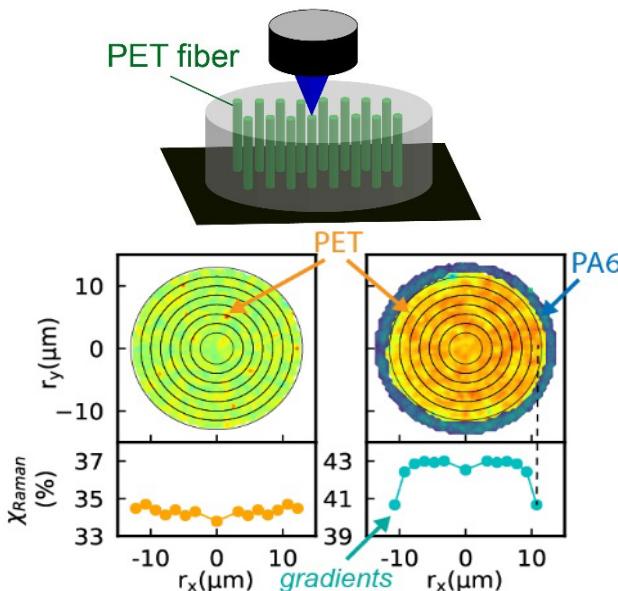
Material and Life Science:

- Presence of chemical structures,
- Orientation (e.g. collagen in bones)





Highlights: Fiber cross-sections



Perret, E. et al. Polymer 2021, 229, 124011.

Perret, E. et al. Polymer 2022, 238, 124422.

- 2D Raman maps: Radial gradients in crystallinity and molecular orientation.

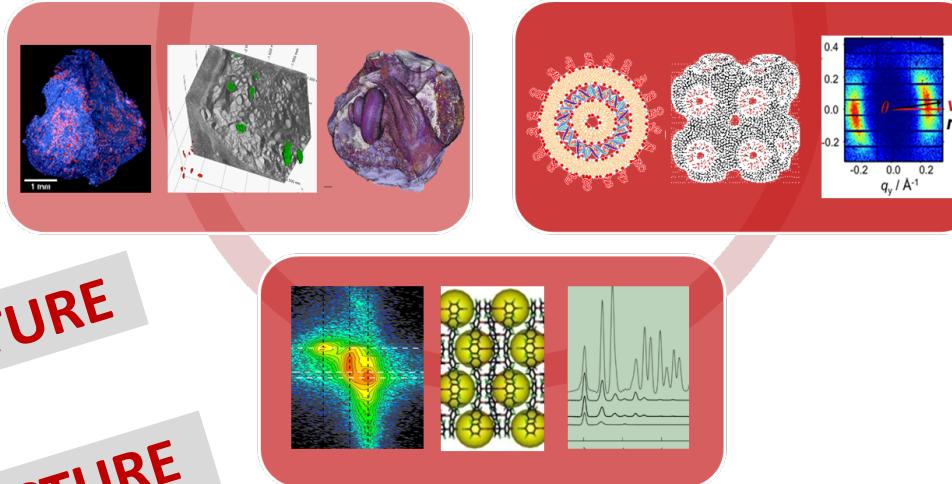


Center of X-ray analytics



Dr. Robert Zboray
Dübendorf

MICROSTRUCTURE
NANOSTRUCTURE



Head of Center of X-ray analytics
Prof. Dr. Antonia Neels

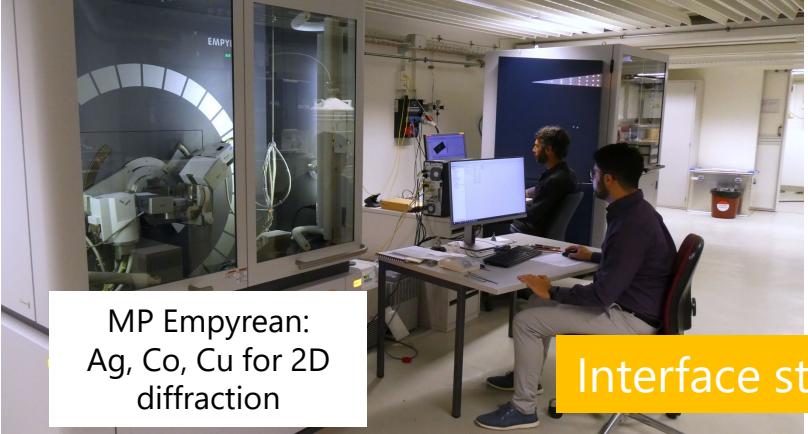


Dr. Bruno Silva
St. Gallen





X-ray lab equipment



MP Empyrean:
Ag, Co, Cu for 2D
diffraction



Stoe-IPDS: 2D Diffraction

Bruker D8:
HRXRD

Interface studies for Medtech



RX Solutions:
X-ray nano-CT



BRUKER Nanostar:
WAXD/SAXS

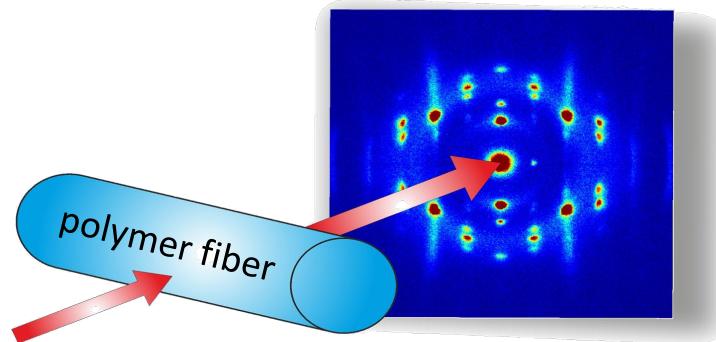
Nano-science for Biomed



How can we analyze nanostructures?

Wide-angle x-ray diffraction (WAXD):

- Orientation of crystallites
- Crystallinity
- Lattice constants
- Plane spacings
- Crystallite sizes along specific directions



Small-angle x-ray scattering (SAXS):

- Distances between lamellae
- Crystallite sizes

In-situ measurements
(temperature, tensile stress...)

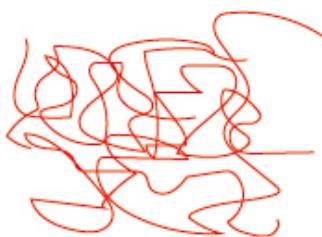


Bruker Nanostar @Empa, St. Gallen

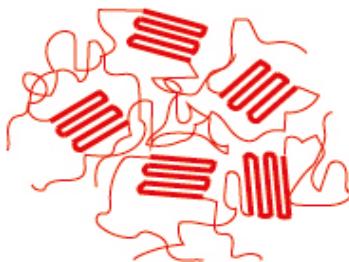


How to determine the orientation of crystallites

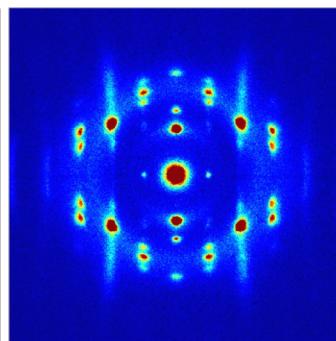
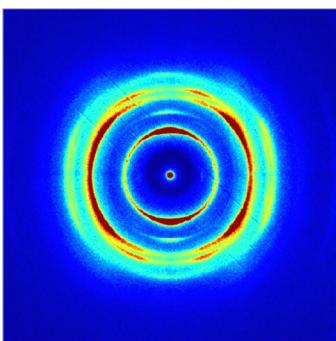
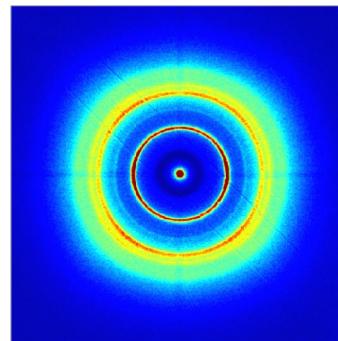
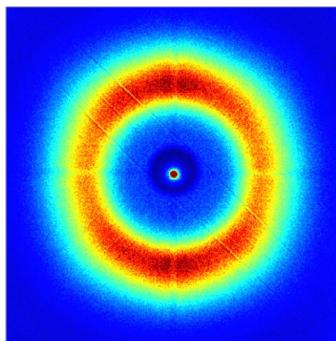
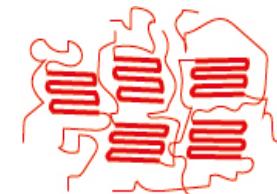
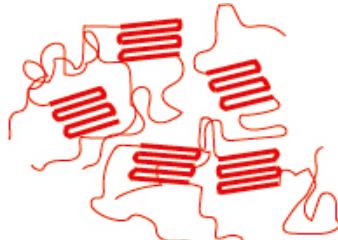
Amorphous



unoriented



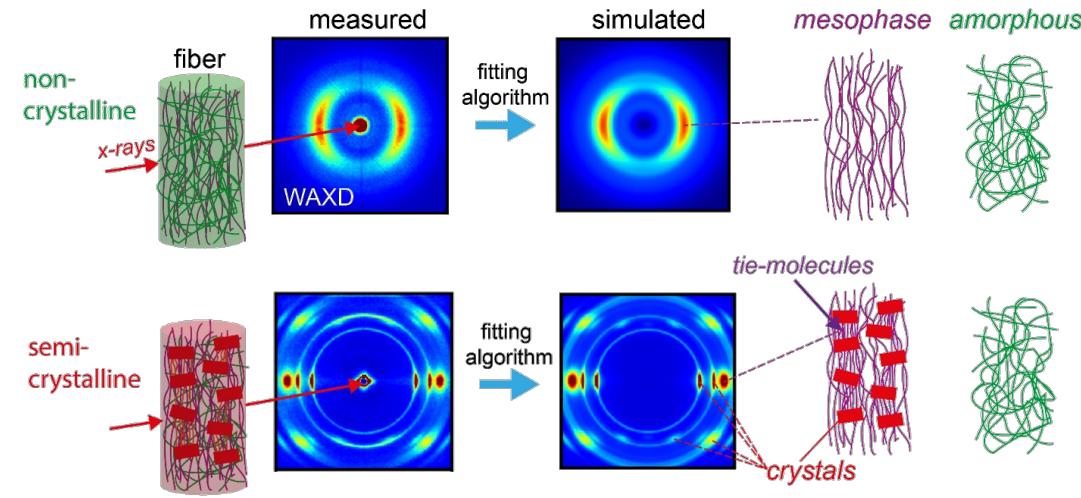
Semi-crystalline
partially oriented highly oriented





Highlights: Mesophases in melt-spun fibers

Mesophase: Non-crystalline stretched and oriented macromolecules along the fiber axis



- Mesophases affect the mechanical properties of a fiber.

Overview

E. Perret et al., Polymer, 229:124010, 2021

poly(ϵ -caprolactone) (PCL)

F. Selli et al., Polymer, 206:122870, 2020

poly-3-hydroxybutyrate (P3HB)

E. Perret et al., Polymer, 180:121668, 2019

E. Perret et al., Polymer, 197:122503, 2020

E. Perret et al., Polymer, 231:124141, 2021

Empa – The Place where Innovation Starts

+41 58 765 75 12

edith.perret@empa.ch

empa.ch



Materials Science and Technology

Acknowledgments

Laboratory 402



Center of X-ray analytics

