

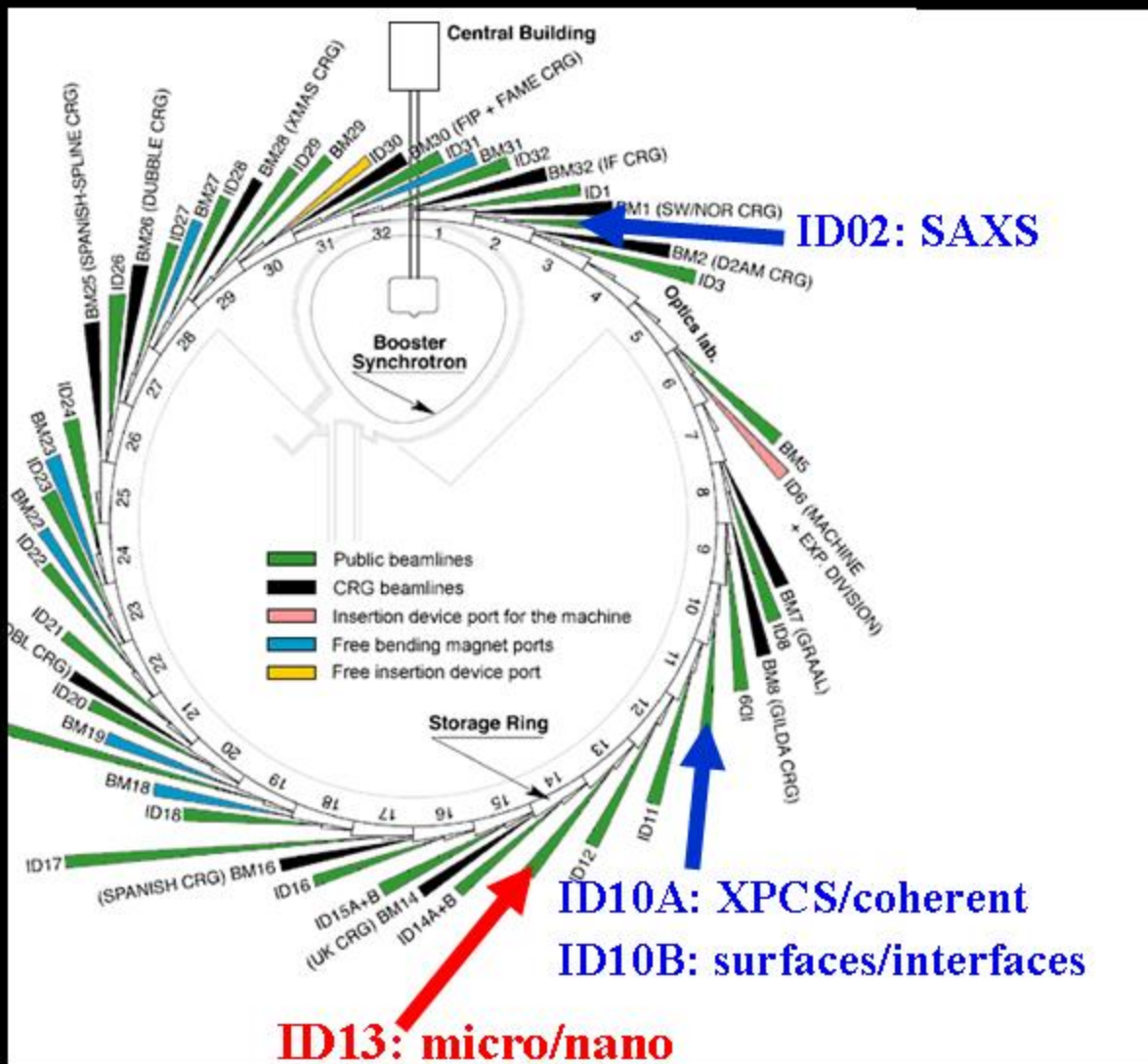
# *Soft Condensed Matter Diffraction with Microfocus Techniques*

*C. Riekkel*

*European Synchrotron Radiation Facility*



# Soft Condensed Matter Group beamlines



## Topics

1. ID13 beamline layout
2. fiber diffraction
3. large data sets
4. microRaman
5. optical manipulation
6. European SAXS funding

# Microfocus beamline

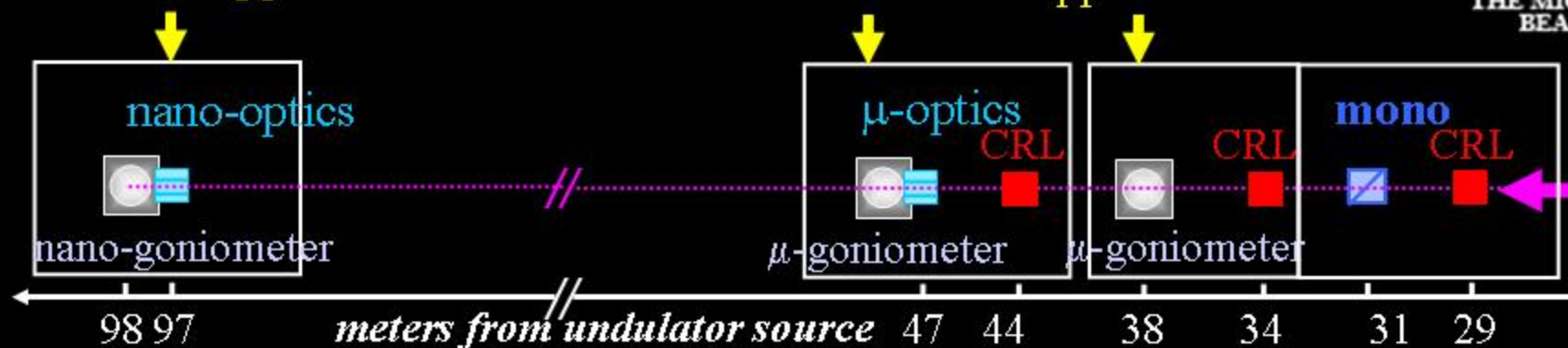


# ID13 beamline configuration



nanofocus applications

microfocus applications



**2007: Nanofocus extension**



*parabolic CRL+collimator* ● **5  $\mu$ m**

*KB mirrors* ● **1  $\mu$ m**

*linear Fresnel lenses* ● **300 nm**

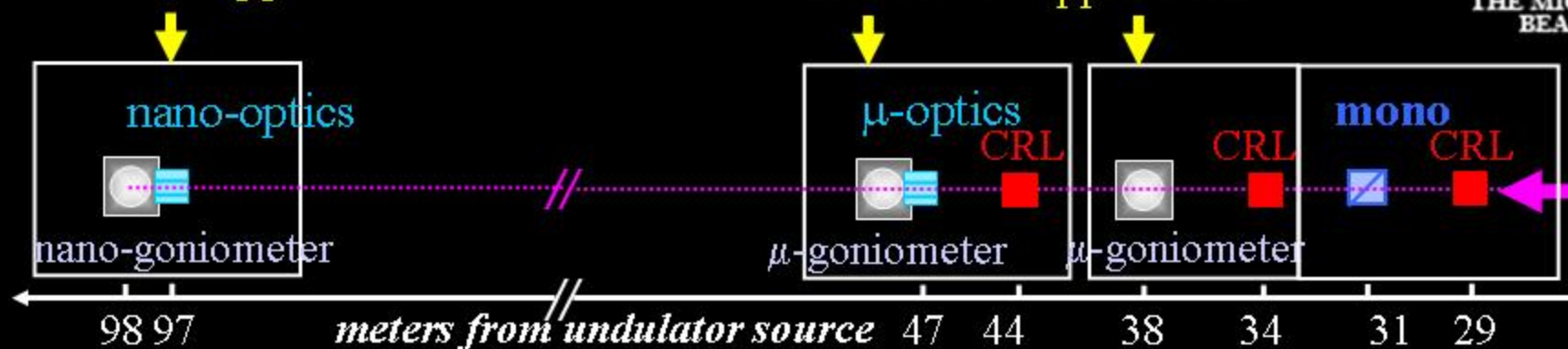
*linear CRL* ●  **$\approx$ 50 nm**

# ID13 beamline configuration



nano-focus applications

microfocus applications

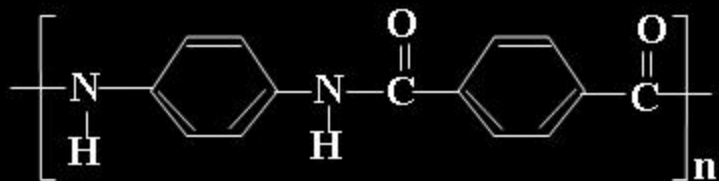


**proposed Upgrade Program Beamline 8**

**separate “micro” and “nano” branches by canted undulators**

# Fiber diffraction

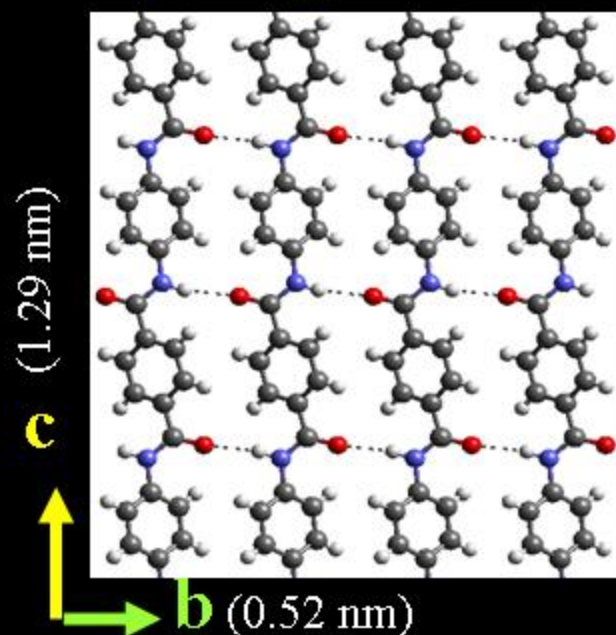
# *Poly (p-phenylene terephthalamide) structure*



**Kevlar® 29,49,149**

## ATOMIC STRUCTURE

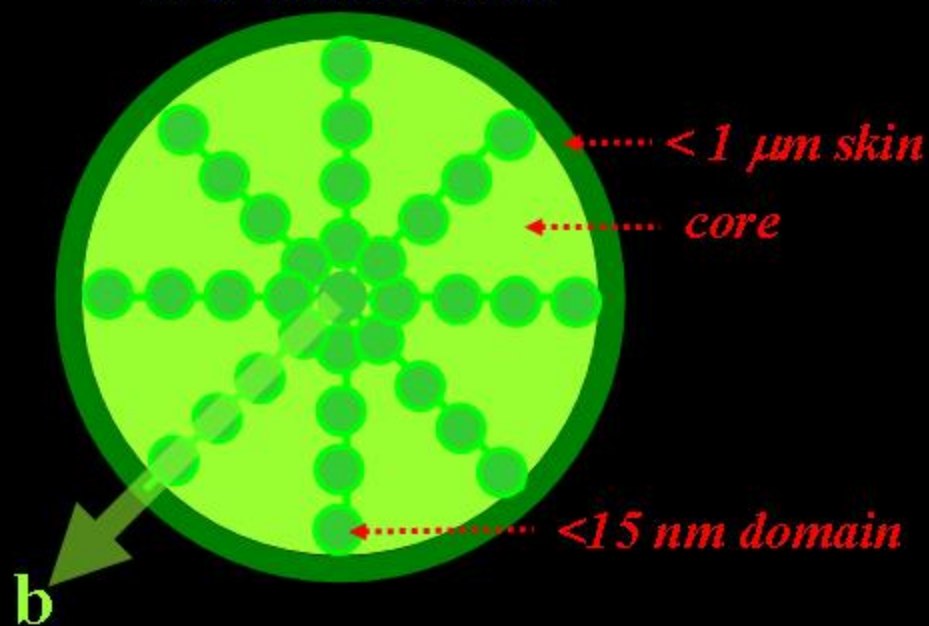
*fiber diffraction*



Northolt, *Eur. Polym. J.* (1974) **10**,799

## MORPHOLOGY

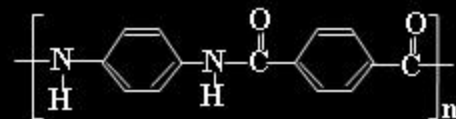
*TEM on thin slices*



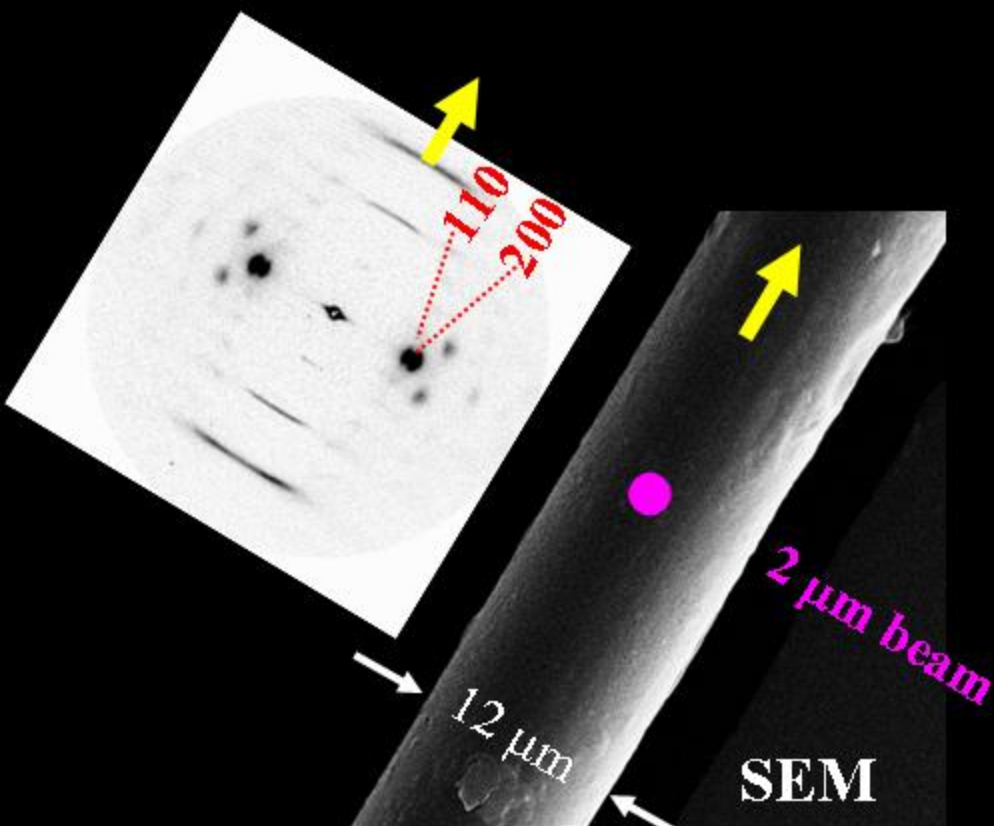
Dobb et al., *J. Polym. Sc.*(1977) **58**, 237



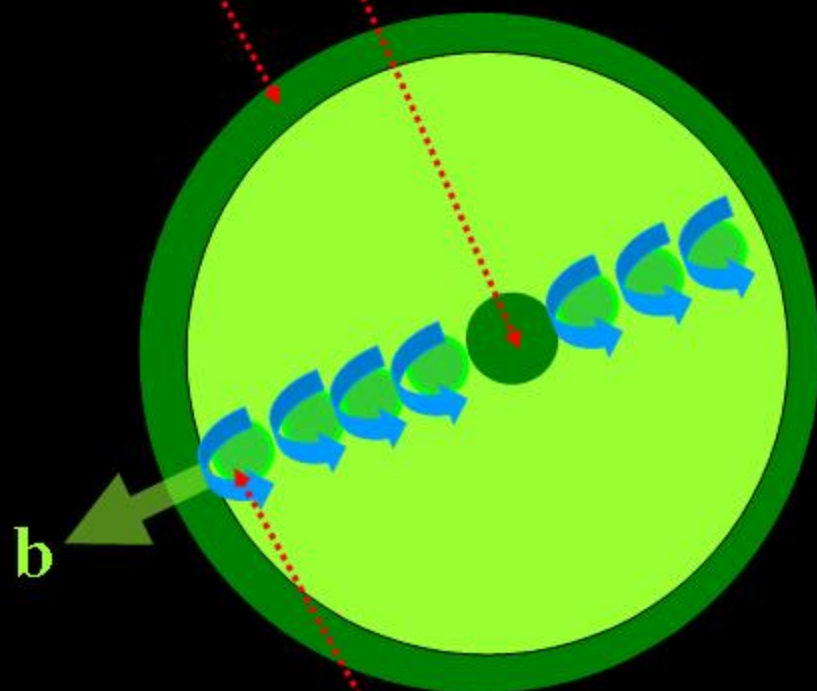
# Kevlar: micro-WAXS



THE MICRO/NANO  
BEAMLINE



*rotationally disordered  
skin & core domains*

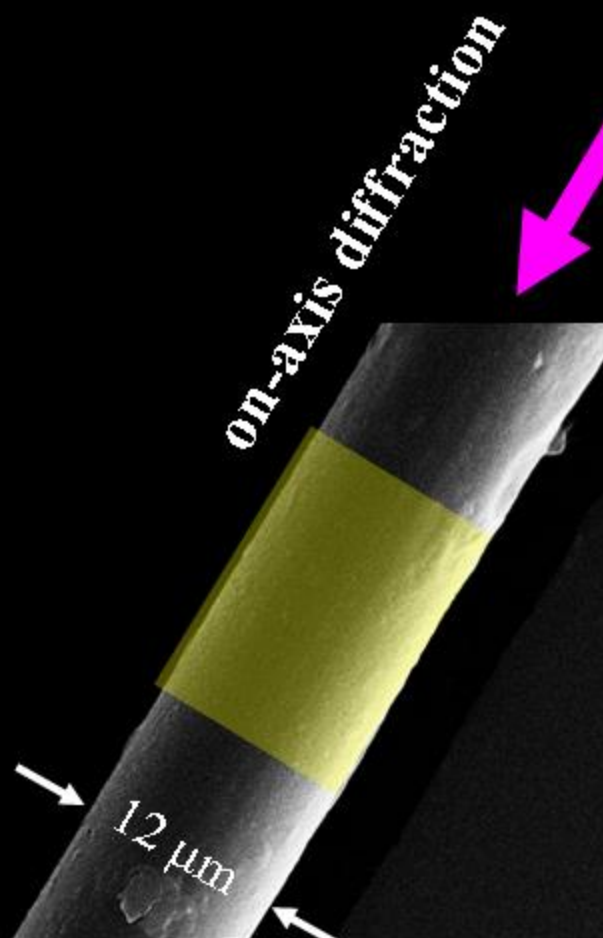
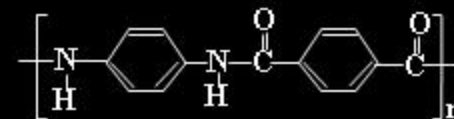


*bulk domains with cooperative  
rotational disorder: 52° fwhm*

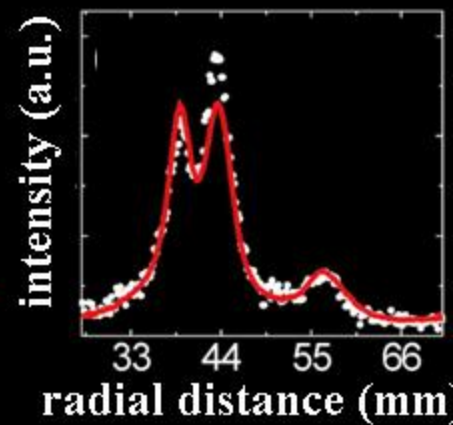
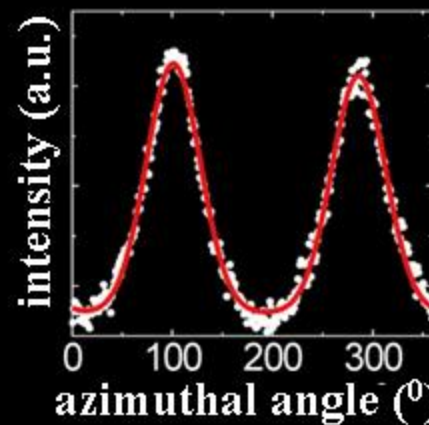
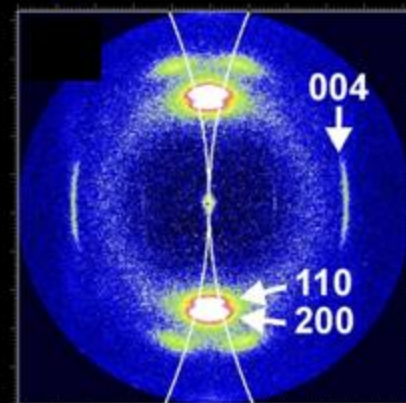
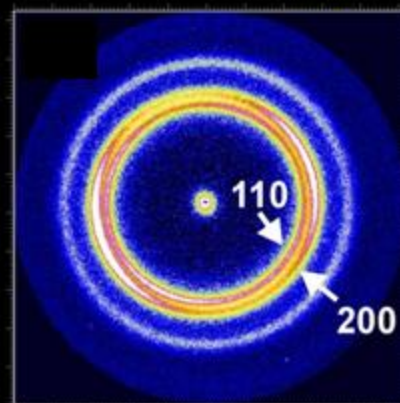
**150 nm beam:** Roth et al., *Macromolecules* (2003) 36, 1585



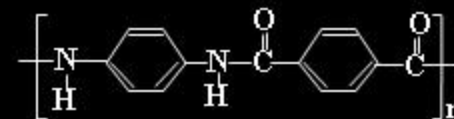
# Kevlar: on-axis WAXS



on-axis geometry “normal”



# Kevlar: micro-dissection



The screenshot displays a software interface for micro-dissection. The main window shows a vertical Kevlar fiber with a 30 μm scale bar. A green circle highlights a point on the fiber. The interface includes a toolbar with various tools, a color palette, and a control panel on the right with sliders and buttons. A trial version watermark is visible at the top right of the software window.

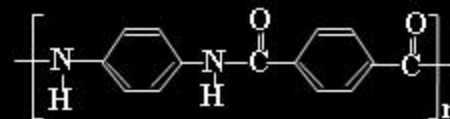
Copyright (c) ESRF

Element List

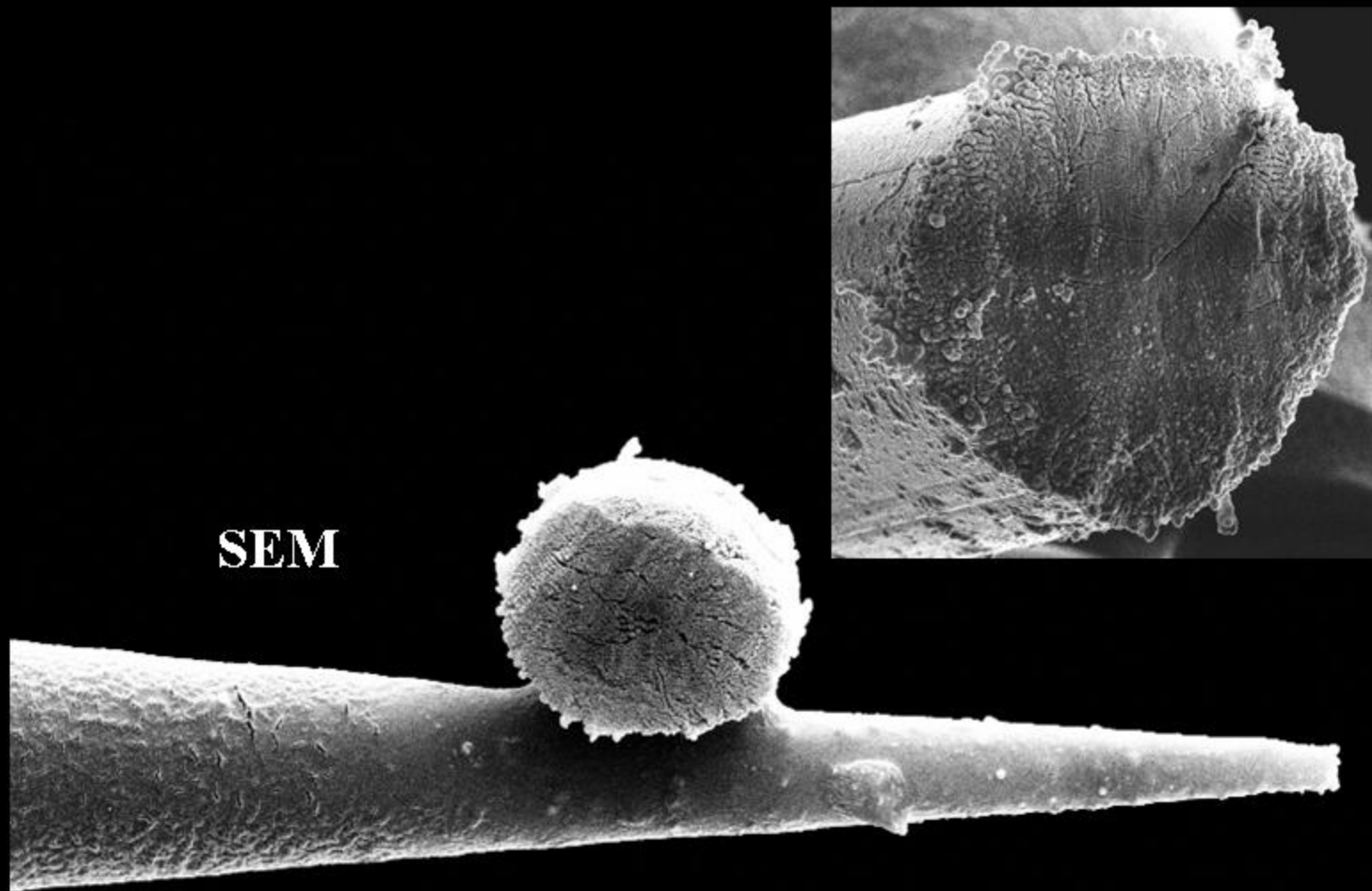
| Color | Nr | Name | Type | cut_shot | Area (μm <sup>2</sup> ) | Grp | Comment | H x W           | Position           |
|-------|----|------|------|----------|-------------------------|-----|---------|-----------------|--------------------|
|       | 2  |      | Line | 1, -     | 0                       |     |         | 32.9 x 0.0 (μm) | (70034.5, 19075.1) |

C. Koenig, ESRF-ID13

# *Kevlar: micro-dissection*



THE MICRO/NANO  
BEAMLINE



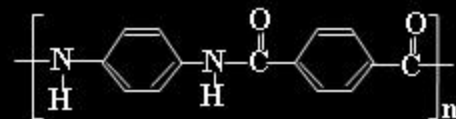
SEM

10 $\mu$ m

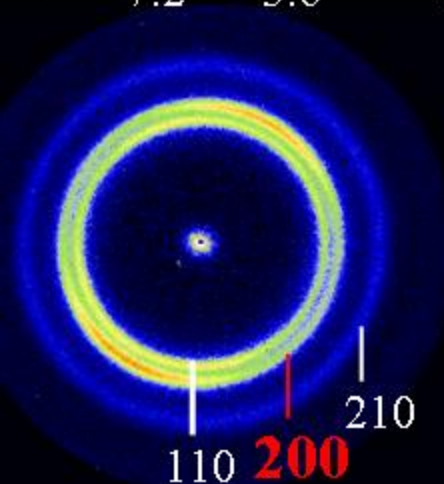
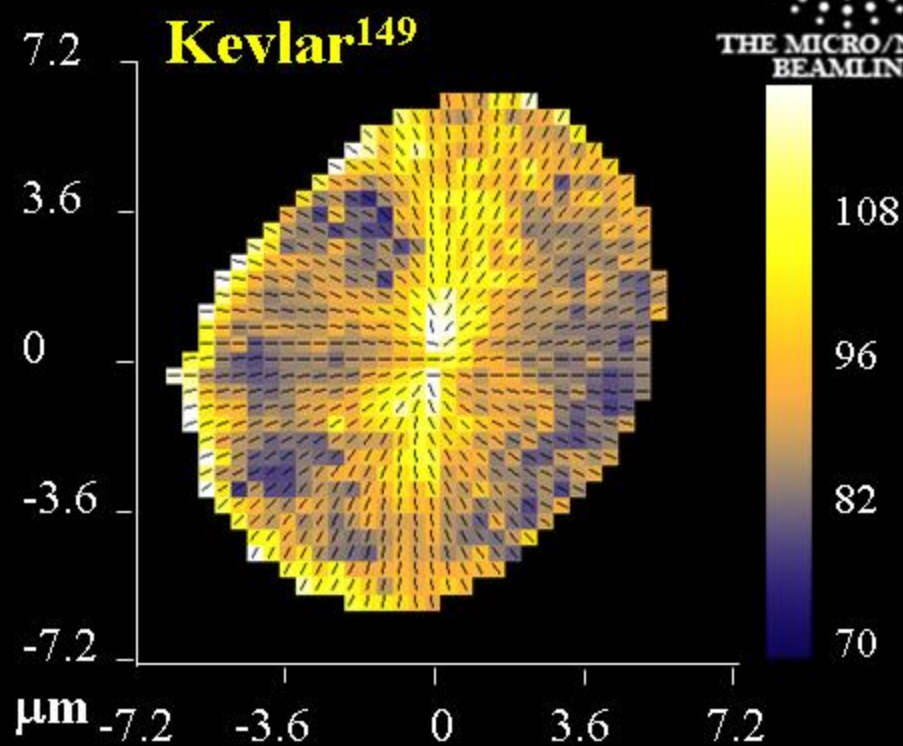
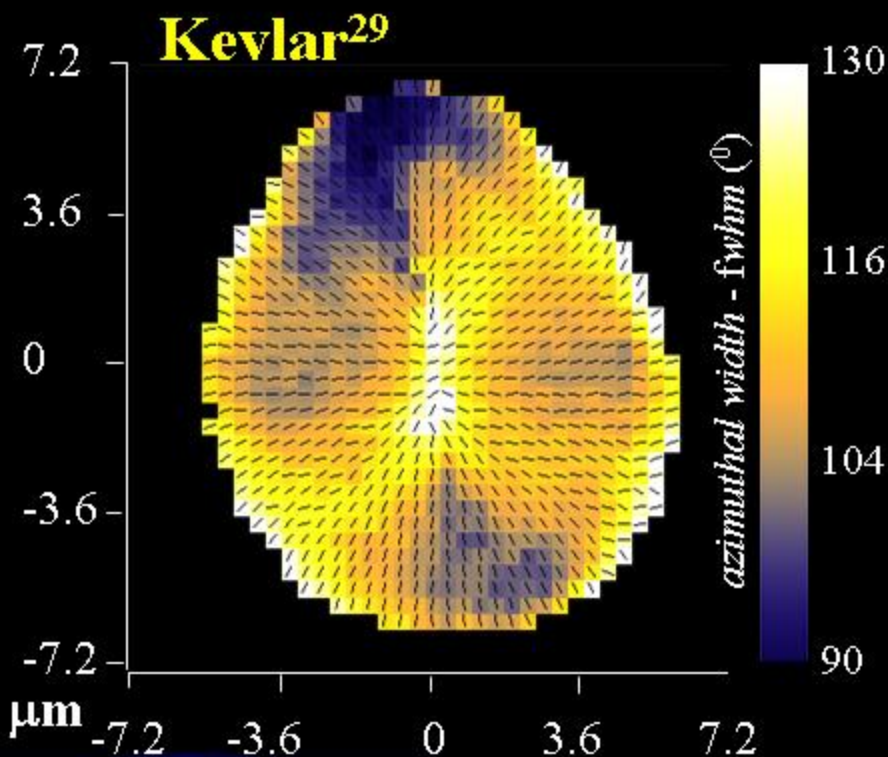




# Kevlar: on-axis WAXS



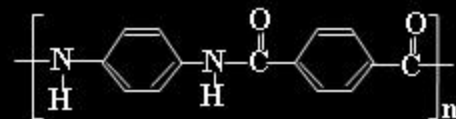
THE MICRO/NANO  
BEAMLINE



on-axis pattern - 1 μm beam  
recursive fit of **200 reflection**

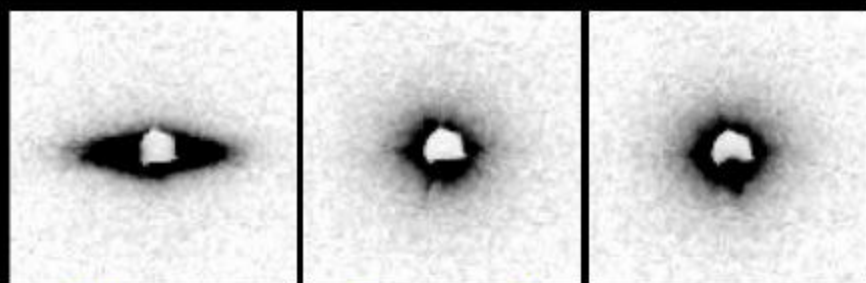
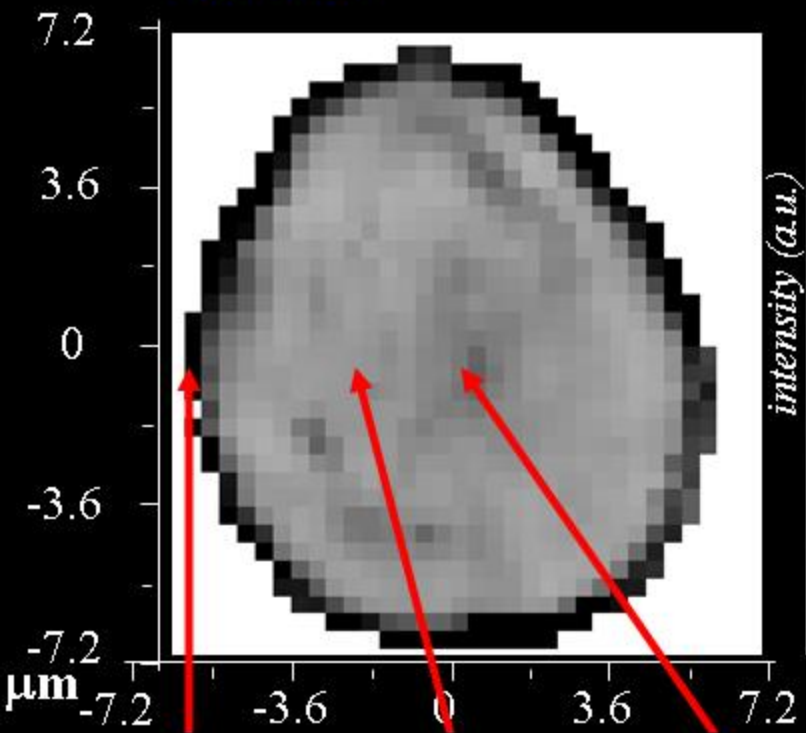


# Kevlar: on-axis SAXS



THE MICRO/NANO  
BEAMLINE

## Kevlar<sup>29</sup>

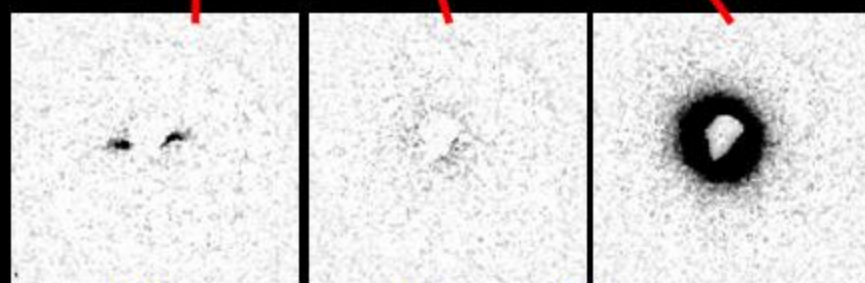
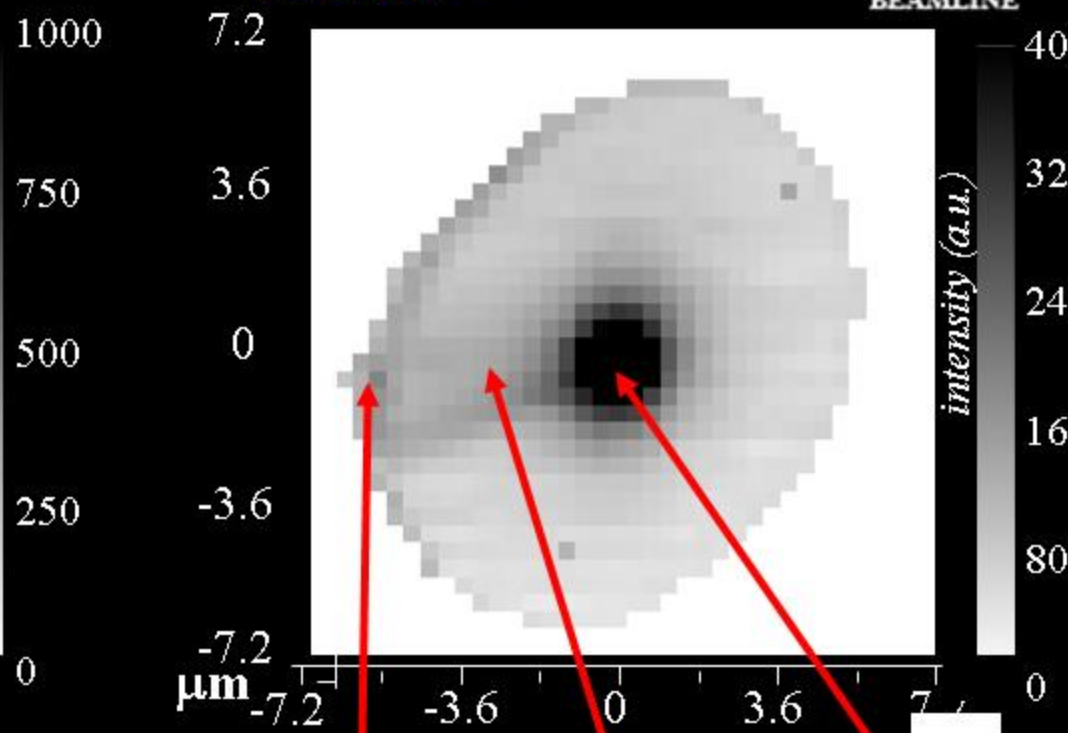


skin

intermediate

core

## Kevlar<sup>149</sup>



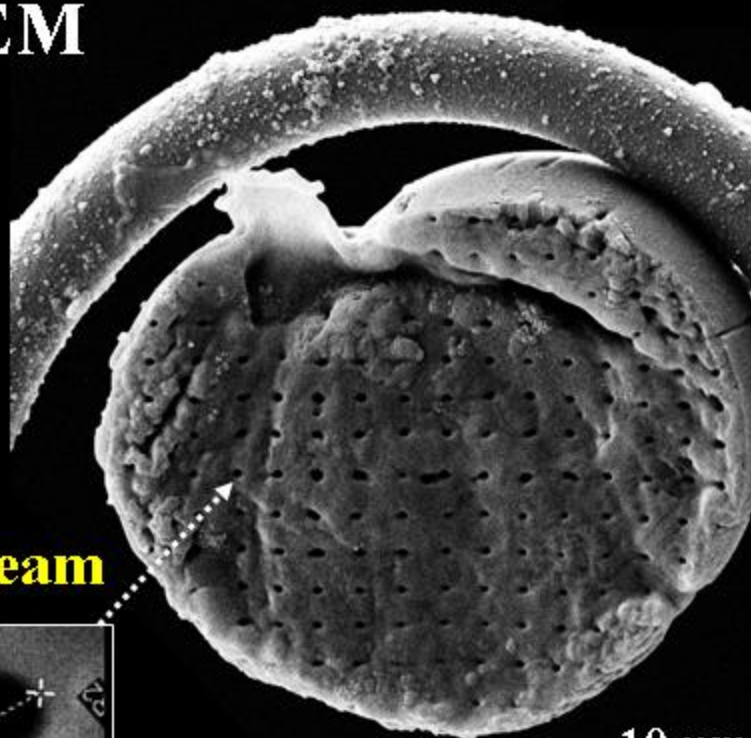
skin

intermediate

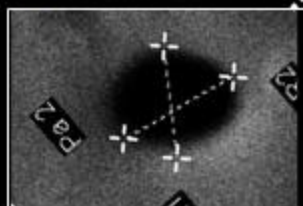
core

# Starch granule section irradiated at 100 K

SEM



1 μm beam



ZOOM

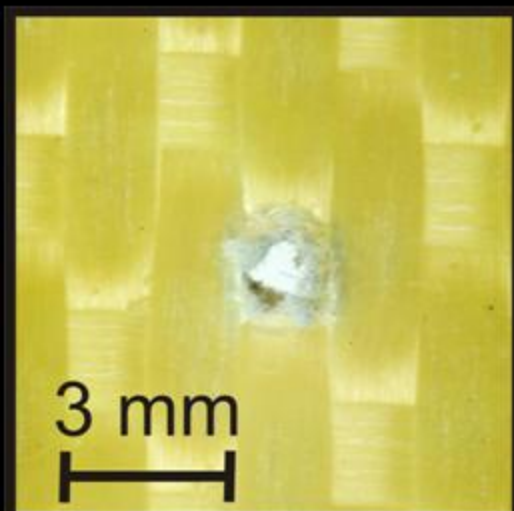
10 μm

Koenig et al., *submitted*

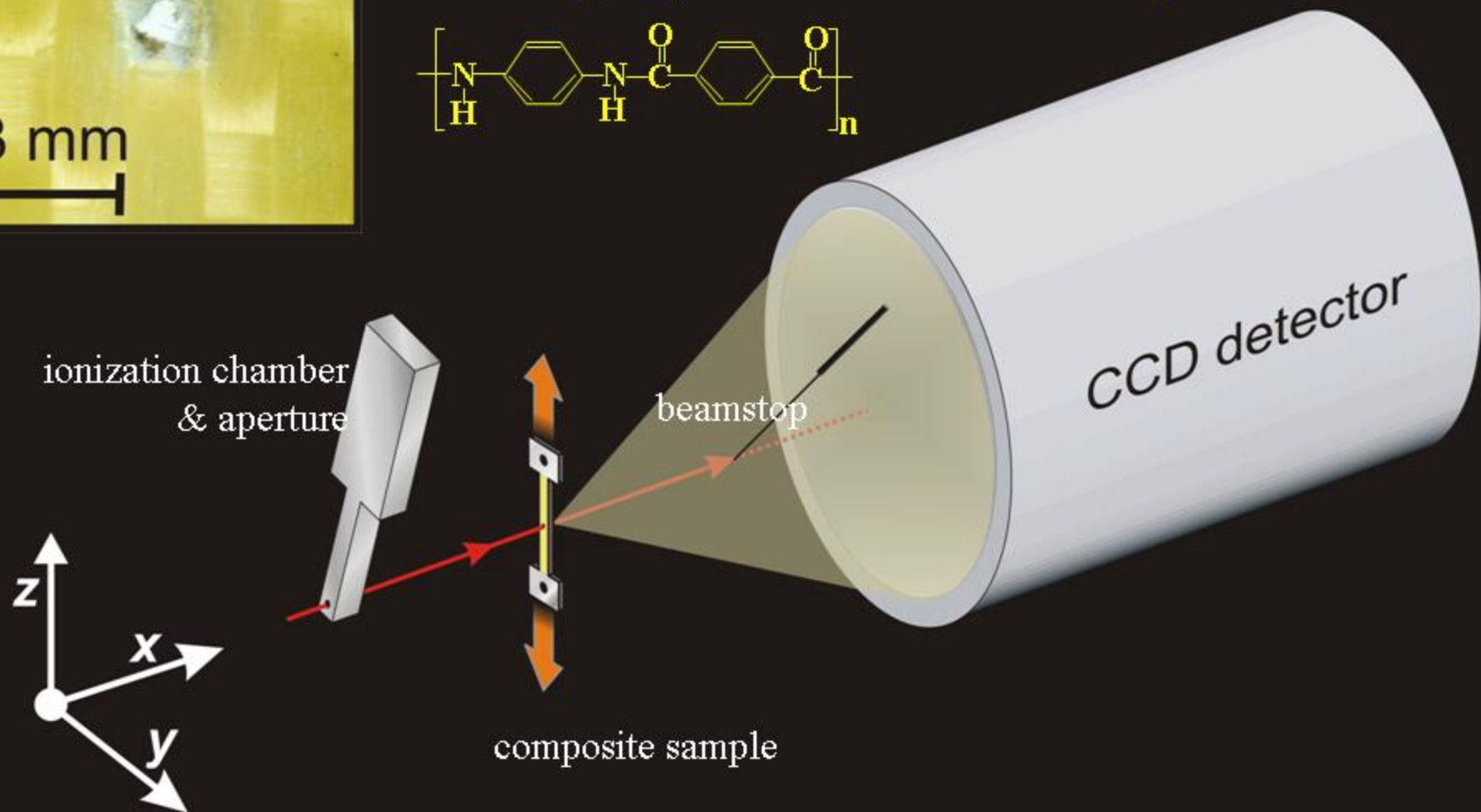
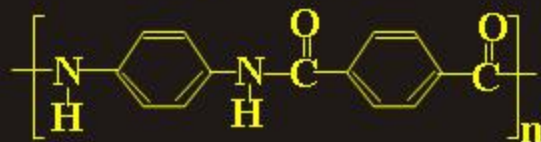
*Cricket sensor hair* Seidel et al., *Micron* (2008) 39, 198

# Large data sets

# Woven composites

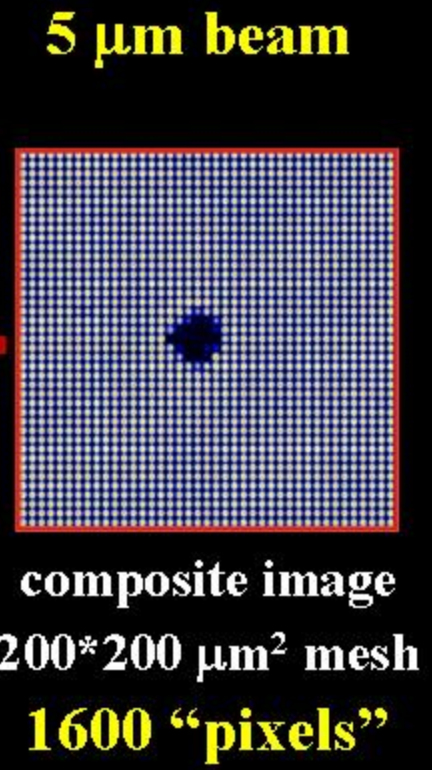
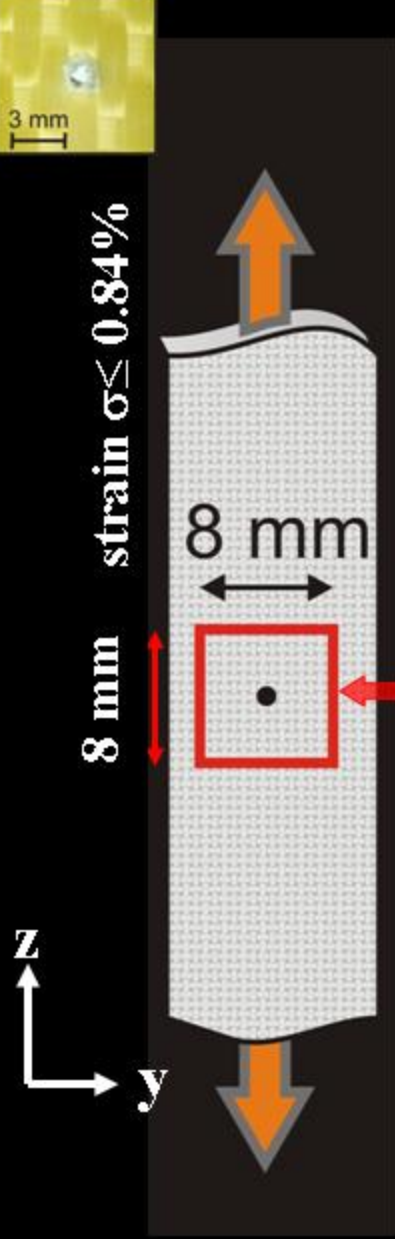


Kevlar/epoxy fiber reinforced composite

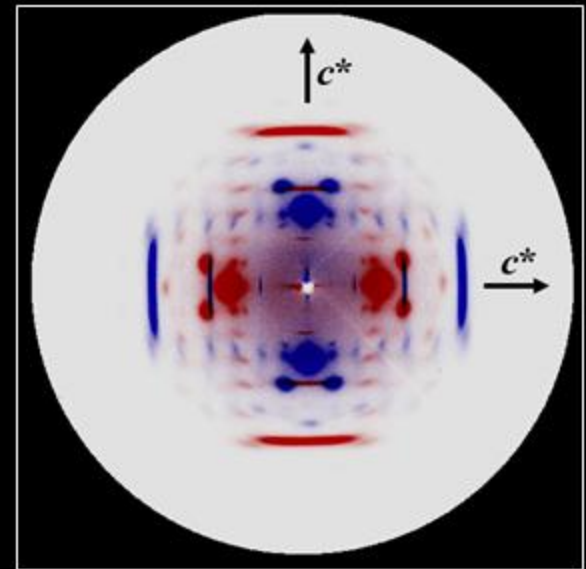
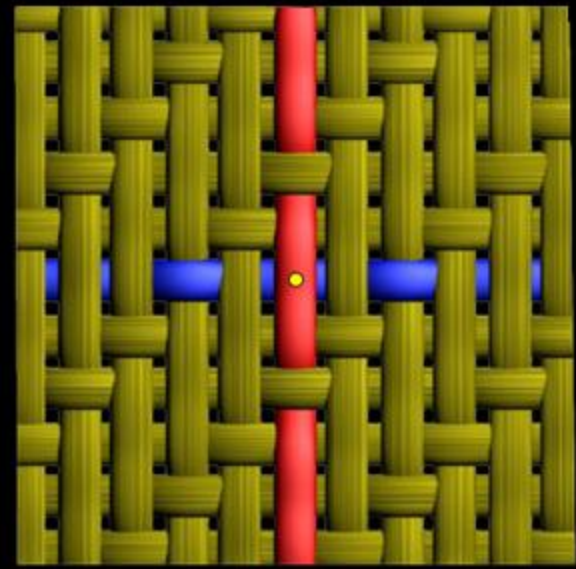




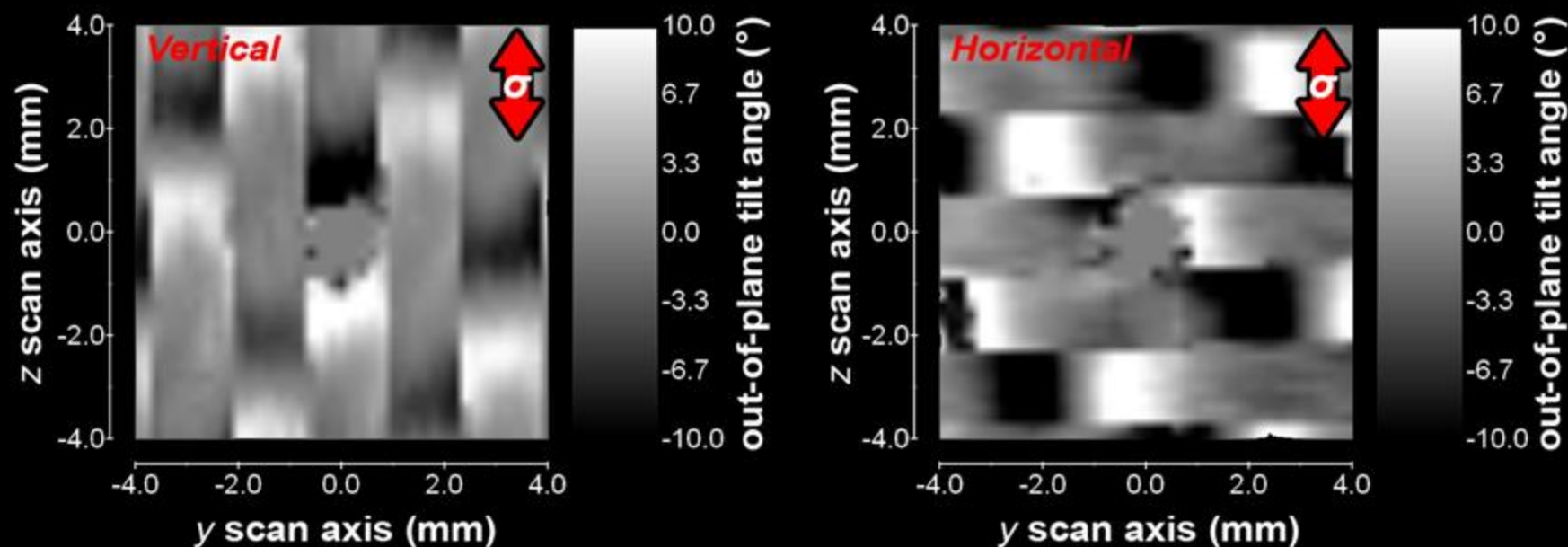
# Composite "diffraction imaging"



single "pixel"



# Embedded yarn geometries



*out-of-plane yarn-tilts from meridional reflections*

Davies et al., *APL* (2007) **91**, 044102

**batch processing software:** Davies, *J. Appl. Cryst.* (2006) **39**,267

**current fitting record:** *about  $4 \cdot 10^4$  CCD-patterns for iPP spherulite*

# MicroRaman



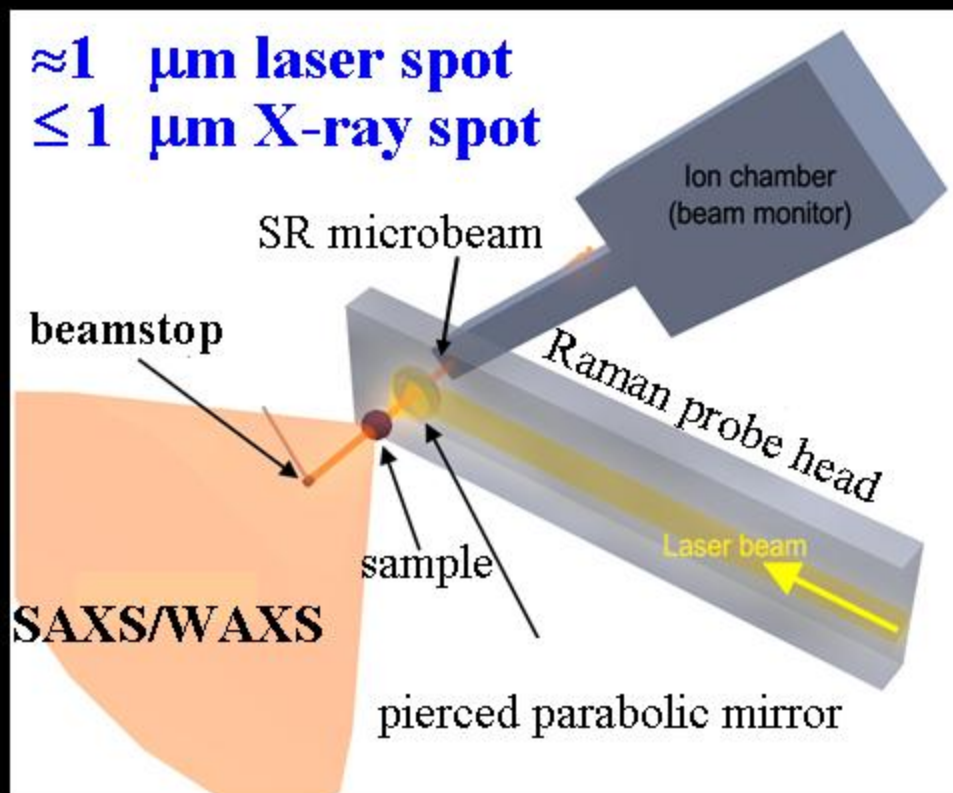
# Combined SAXS/WAXS/microRaman

**SAXS/WAXS**  $\rightarrow$  *long-range order*

**RAMAN**  $\rightarrow$  *molecular information*

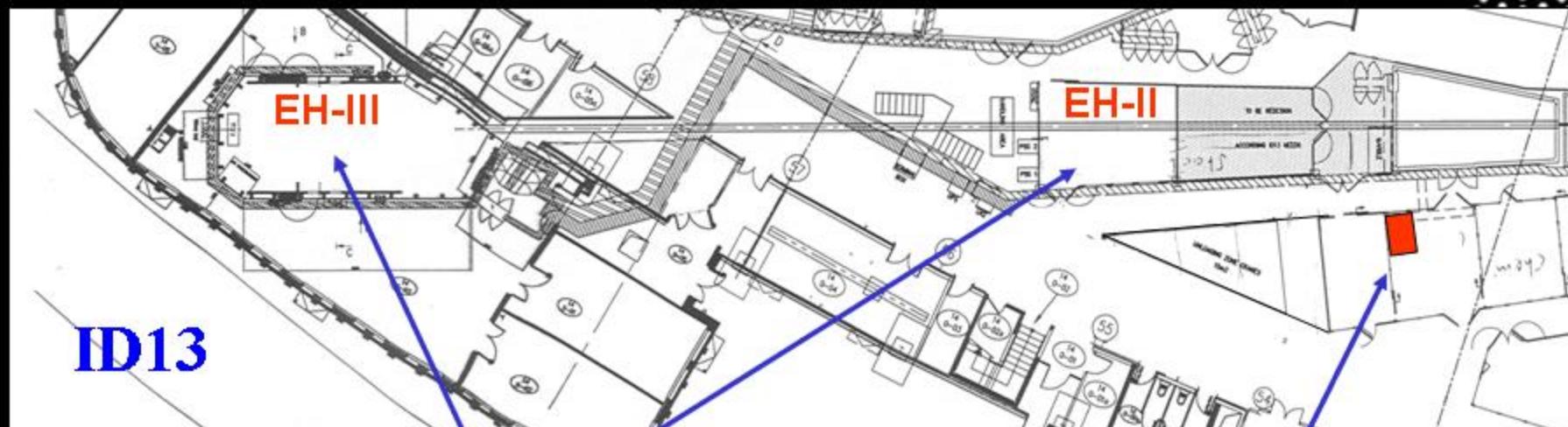
**KB-mirror**

$\approx 1 \mu\text{m}$  laser spot  
 $\leq 1 \mu\text{m}$  X-ray spot

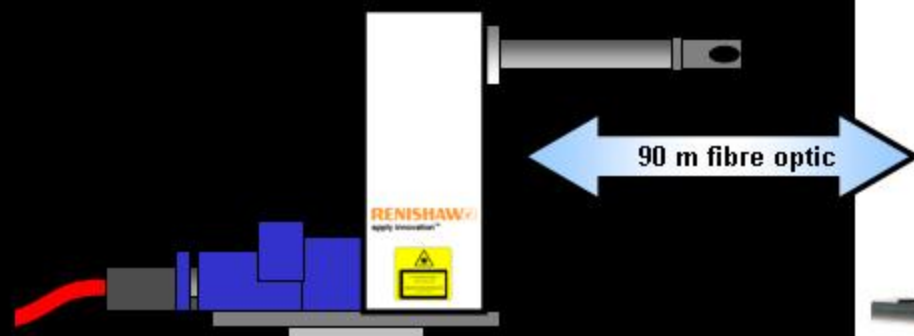




# Combined SAXS/WAXS/microRaman



**ID13**

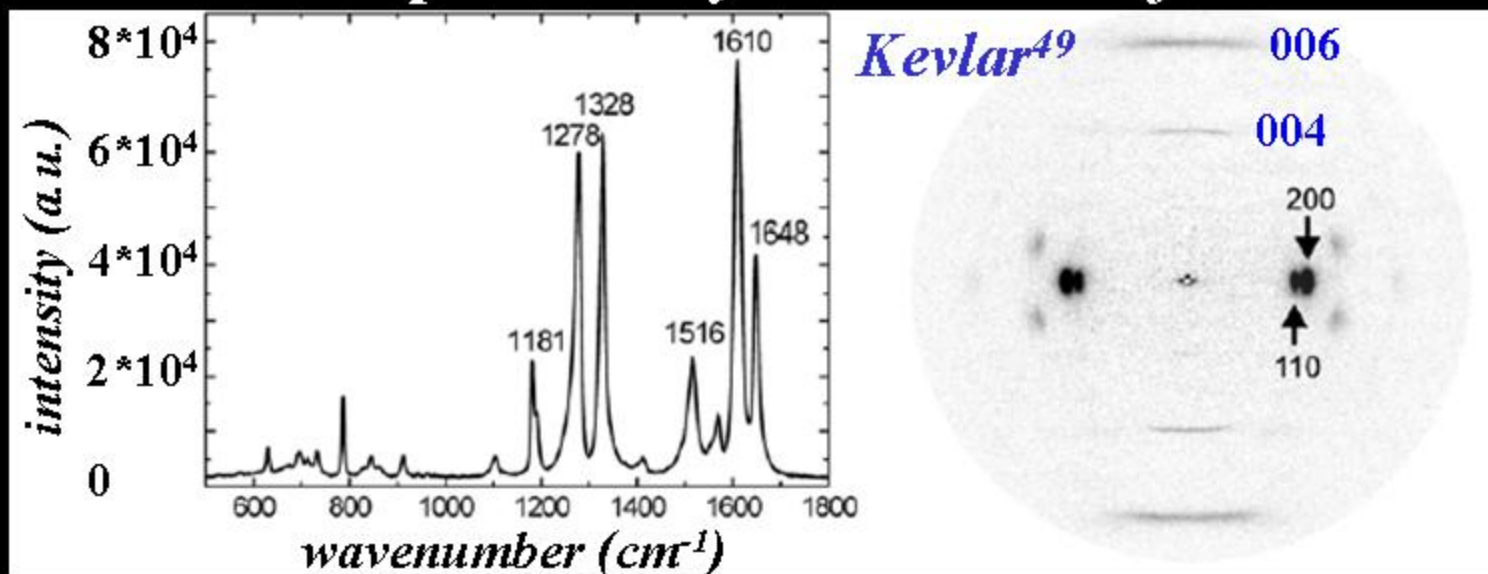


*remote probe with  
interchangeable optics and  
occasional relocation option*

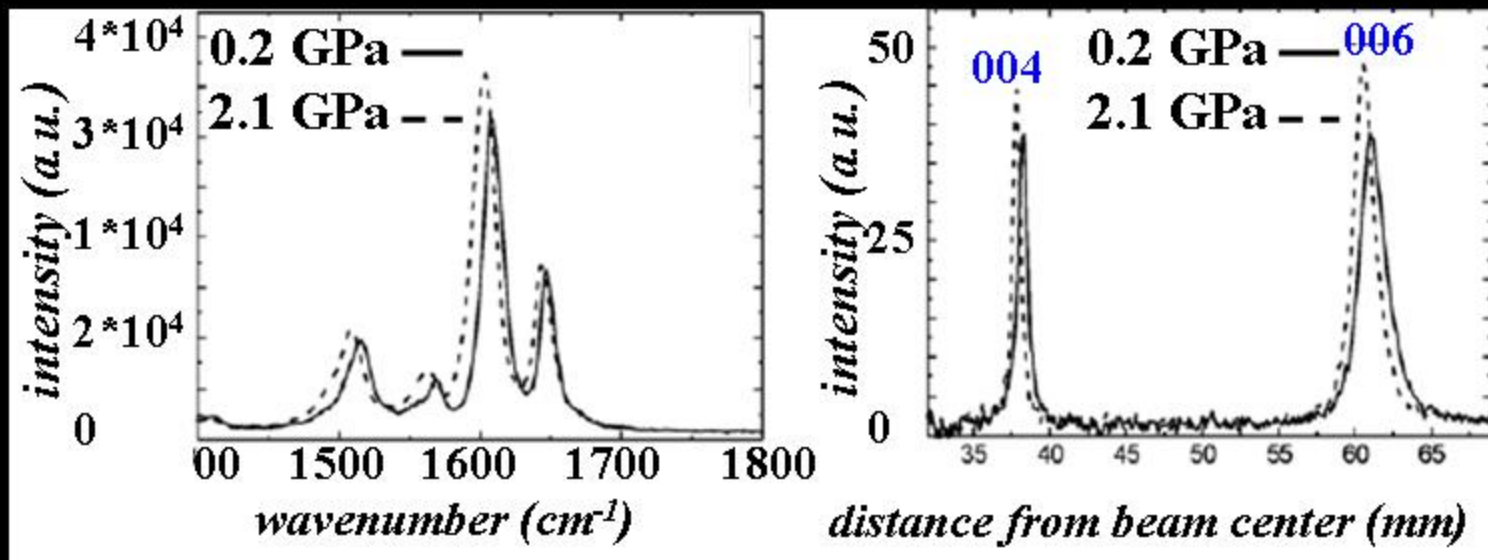


*stand-alone laboratory-based  
microscope and spectrometer*

# Kevlar: complimentary molecular information



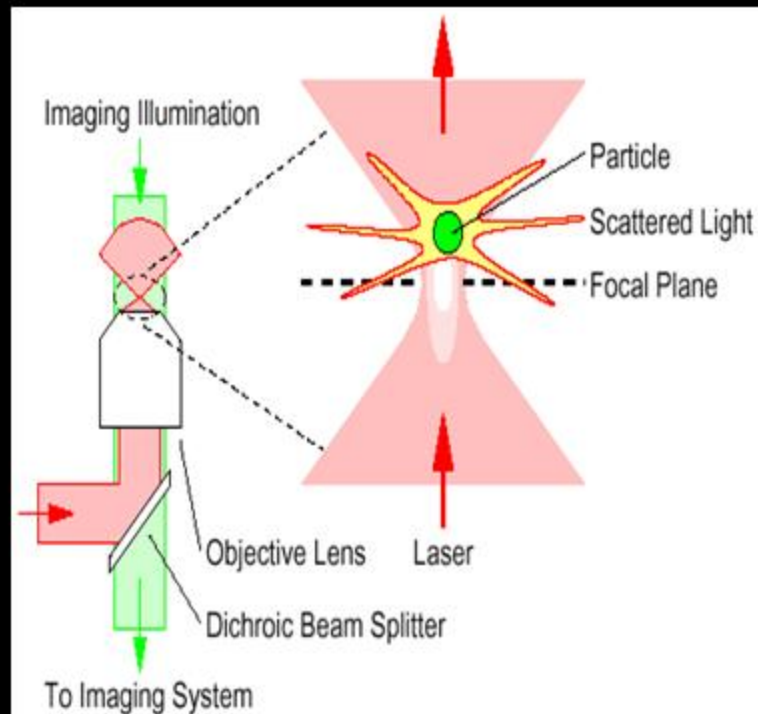
**WAXS**



**Raman**

# Optical manipulation

# Optical tweezers



*Gradient force trap (tens of pN)  
obtained by tightly focusing a CW laser  
beam through a high NA objective*

Ashkin et al., *APL* (1971) **19**, 283

Ashkin, et al *Optics Letters* (1986) **11**, 288

$$\mathbf{F} = Q \frac{n_m W}{c}$$

**F** trapping force

**Q** dimensionless efficiency coefficient

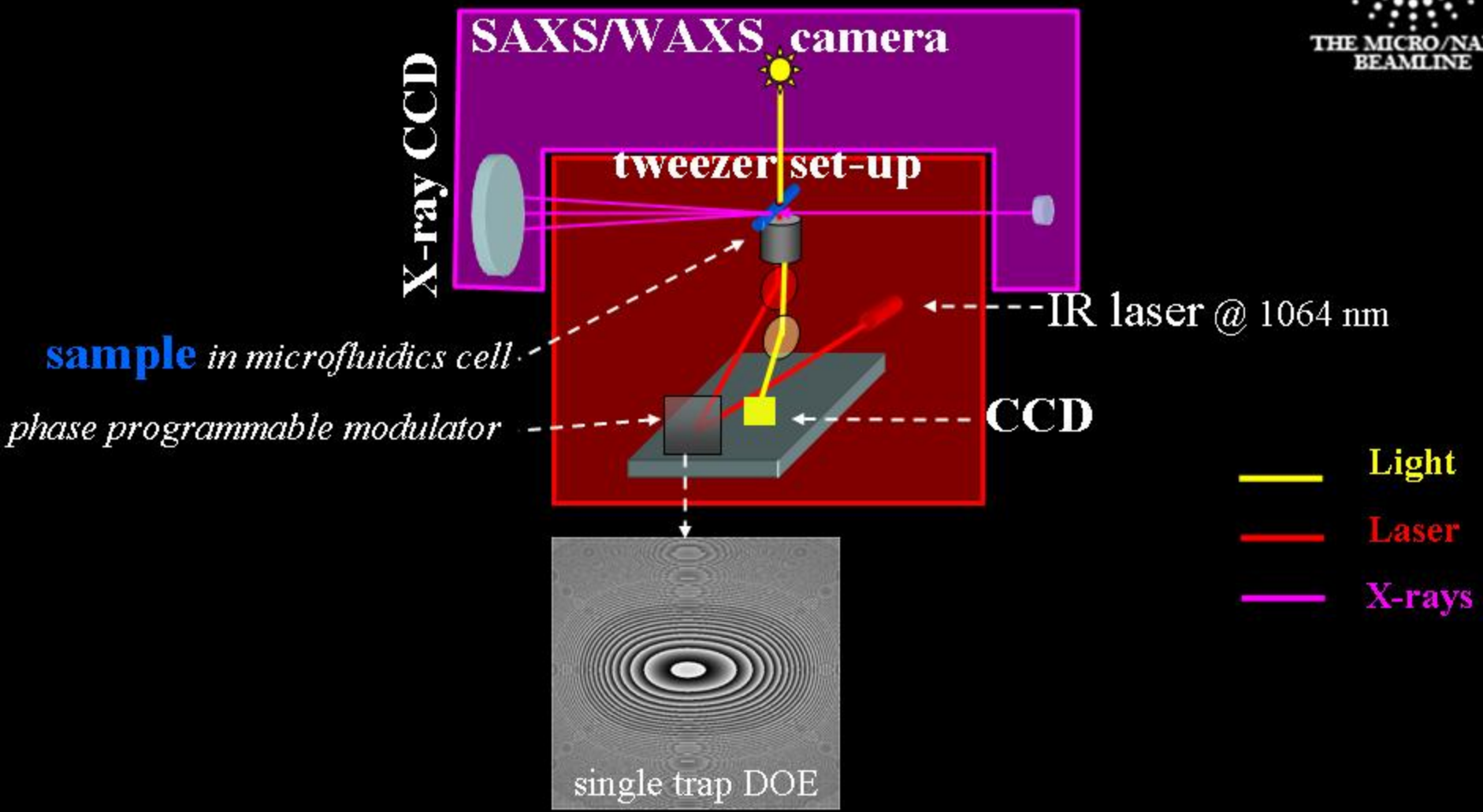
**W** power of the laser beam

**$n_m$**  refractive index of the medium

**c** light speed



# Schematic optical tweezers set-up



DOE: *diffractive optical element*

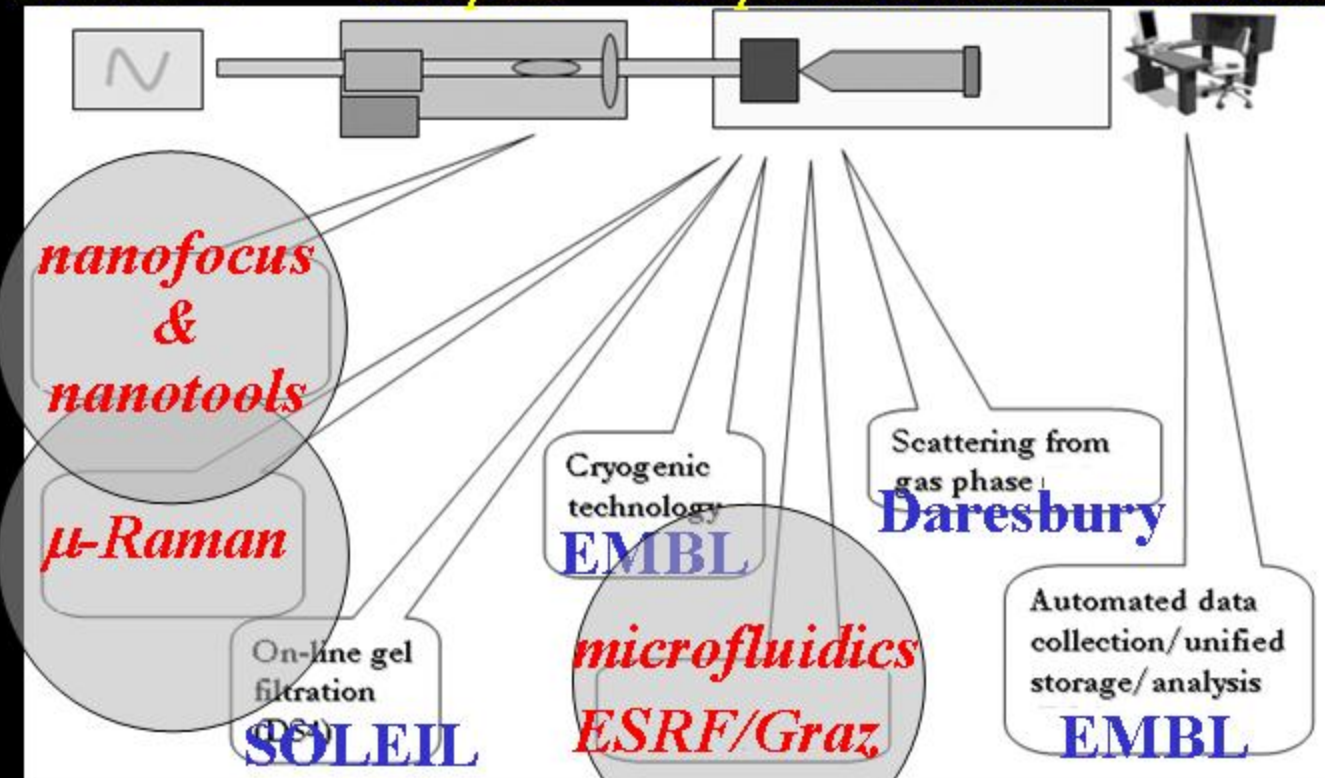
**liposomes** Cojoc et al., *APL* (2007) **91**, 234107-1

# European SAXS funding

# *SAXS at high brilliance European synchrotrons for bio- and nano-technology*

**EEC FP6 grant; spokesman: D. Svergun, EMBL-Hamburg**

*SR source      BL optics      sample environment      data analysis*



# Acknowledgements



## ID13 beamline team

**M. Burghammer**

*instrumentation, nanobeams ...*

**R. Davies**

*polymers,  $\mu$ Raman, data analysis software*

**D. Popov**

*single crystal diffraction*

**R. Gebhardt**

*biological GISAXS (SAXIER)*

**L. Lardiere**

*technician*

**L. Eybert**

*engineering*

**M. Perez**

*software*

**S. Petitdemange**

*software (HFSP)*

