Nanometer-sized Beams for Soft Condensed Matter Studies

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ESRF
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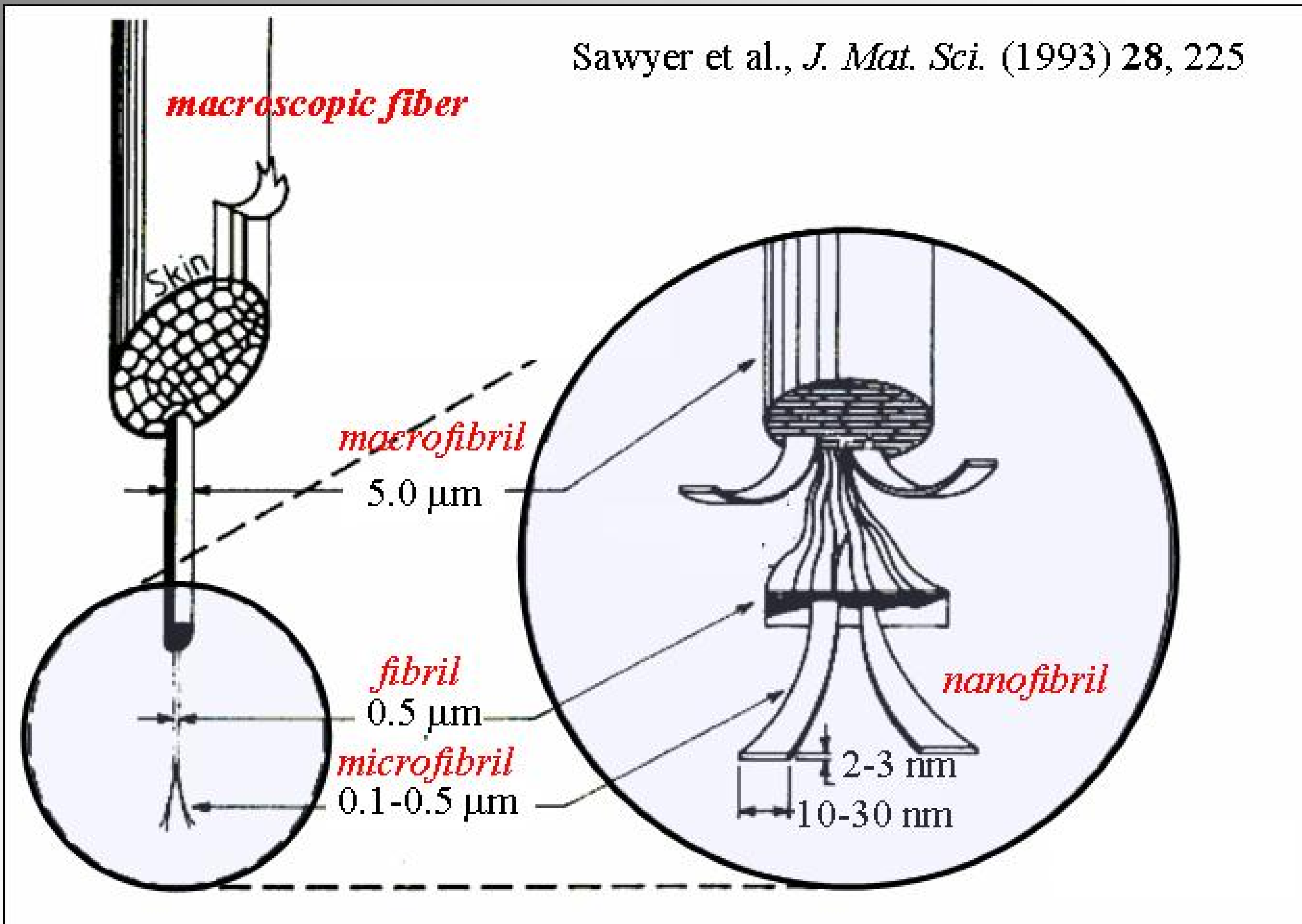
6 GeV synchrotron radiation source
4 nmRad emittance
18 European partners
36 public; 12 national beamlines

Hierarchical organization



top-down
functional units on mulitple length scales

bottom-up
crystallization and crystal structures

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Techniques used in microdiffraction

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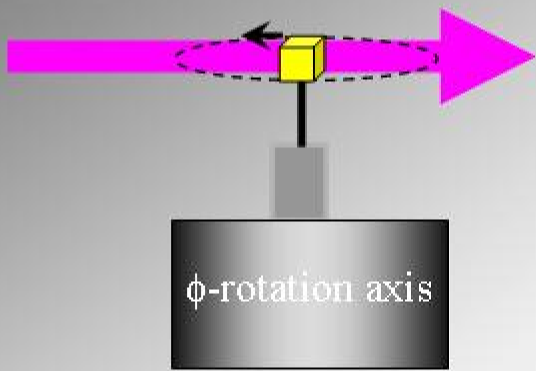
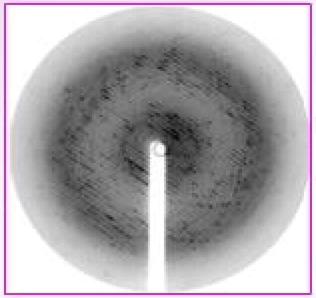
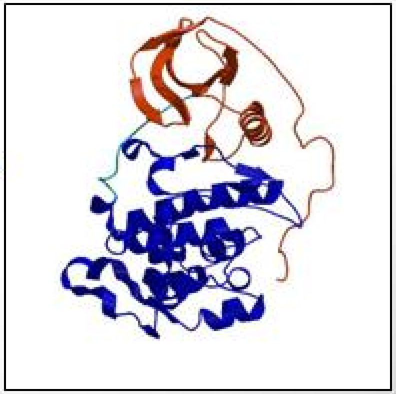
single crystal microdiffraction



ϕ-rotation

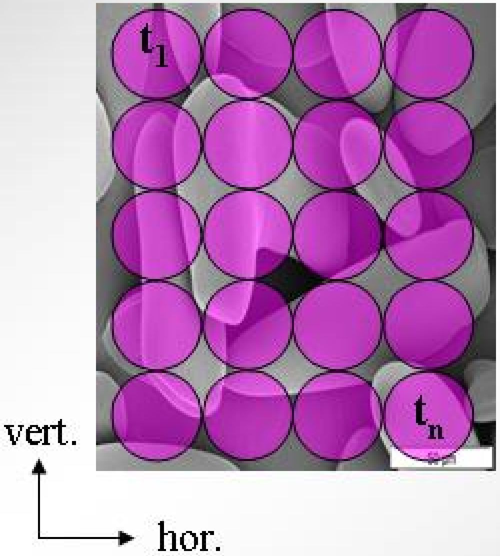


n detector-frames: $\phi_i \ldots \phi_n$



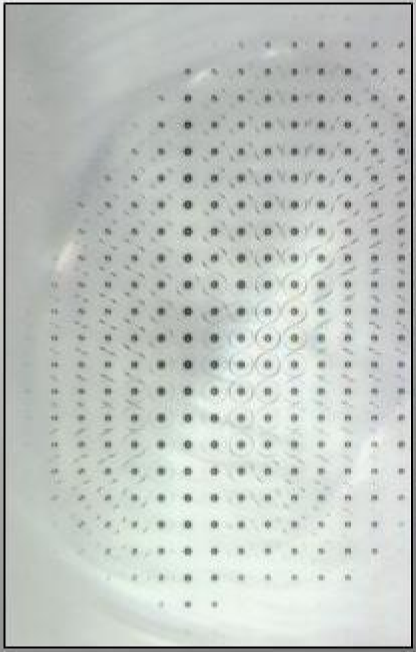
crystal structure

SAXS/WAXS microscopy



mesh-scan

n detector frames: $t_i \ldots t_n$



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scope of micron and submicron beams

status and possible evolution of microSAXS/WAXS cameras

controling and probing of small sample volumes

ESRF ID13 beamline: focal spots

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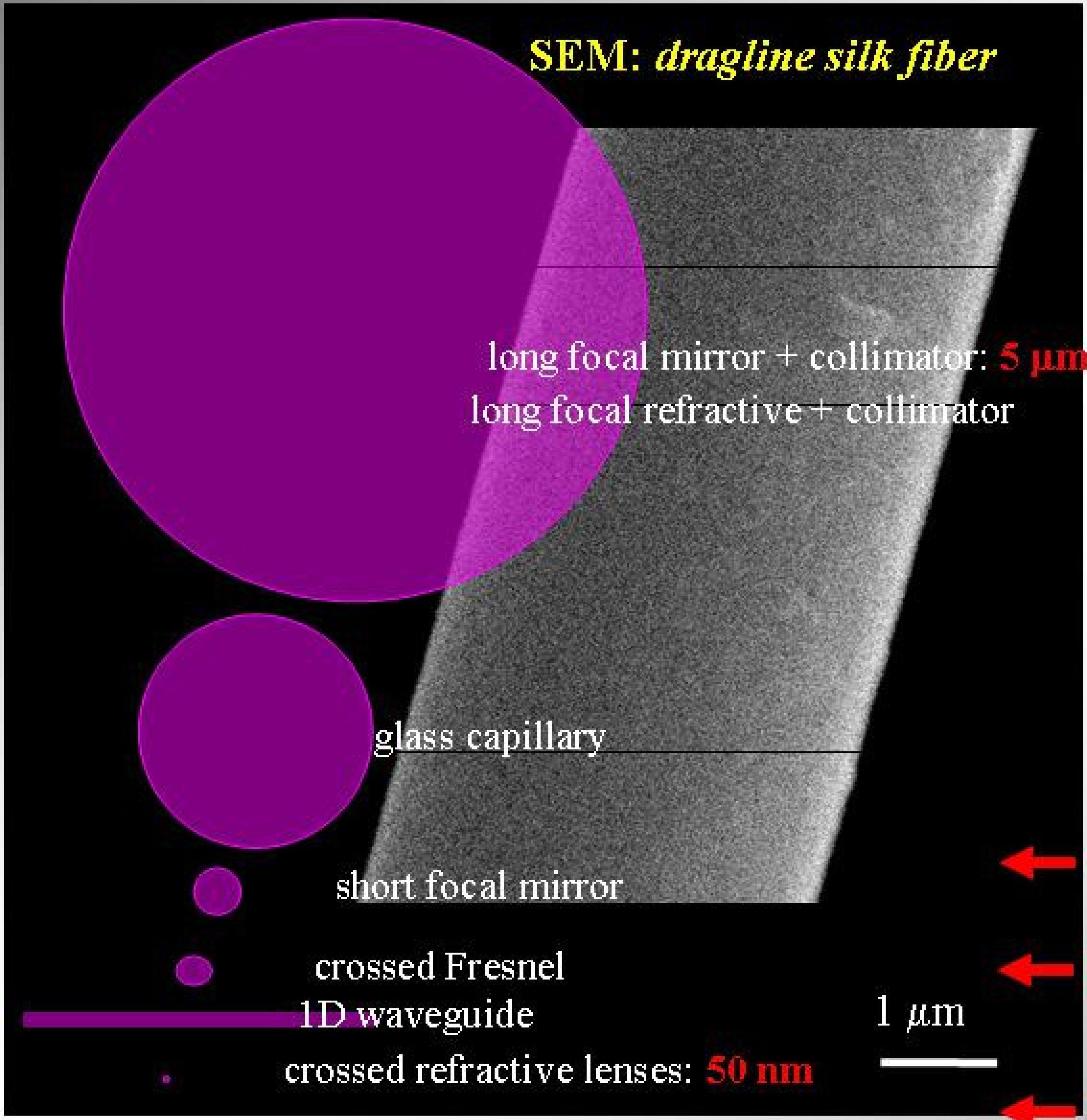

1.118 µm

118 nm

Smilgies report

12 nm

ESRF ID13 beamline layout

micro/nano-SAXS/WAXS *single crystal microdiffraction*

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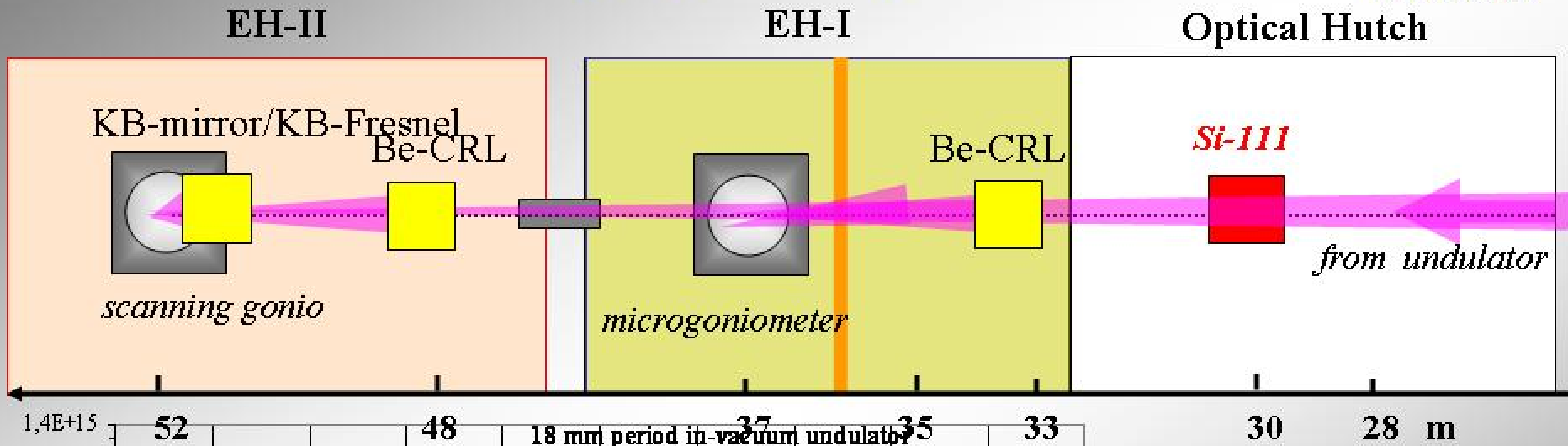

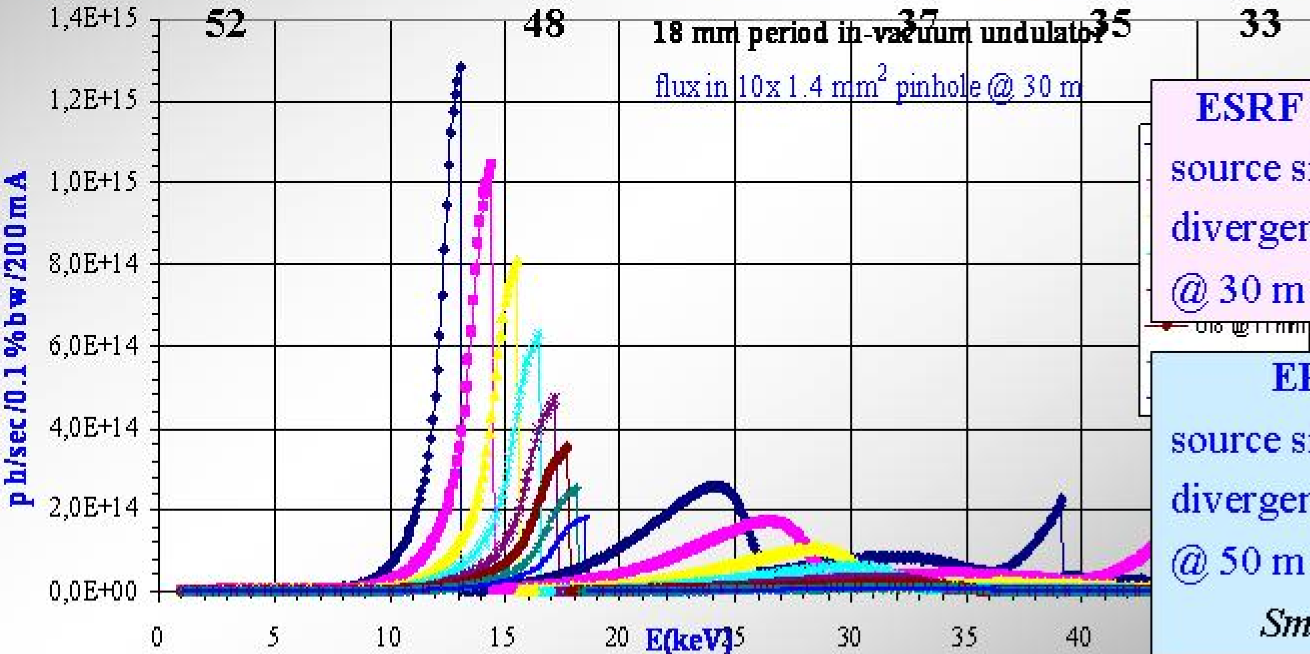

ESRF (13 keV) 1.2 m low-β undulator

source size	$134_h * 25_v$	μm^2
divergence	$0.2_h * 0.02_v$	$mrad^2$
@ 30 m	$6.2_h * 0.6_v$	mm^2

ERL (15 keV) 5 m undulator

source size	$12.2_h * 12.2_v$	μm^2
divergence	$0.014_h * 0.014_v$	$mrad^2$
@ 50 m	$0.5_h * 0.5_v$	mm^2

Smilgies report: **fwhm values**

Micro-SAXS/WAXS

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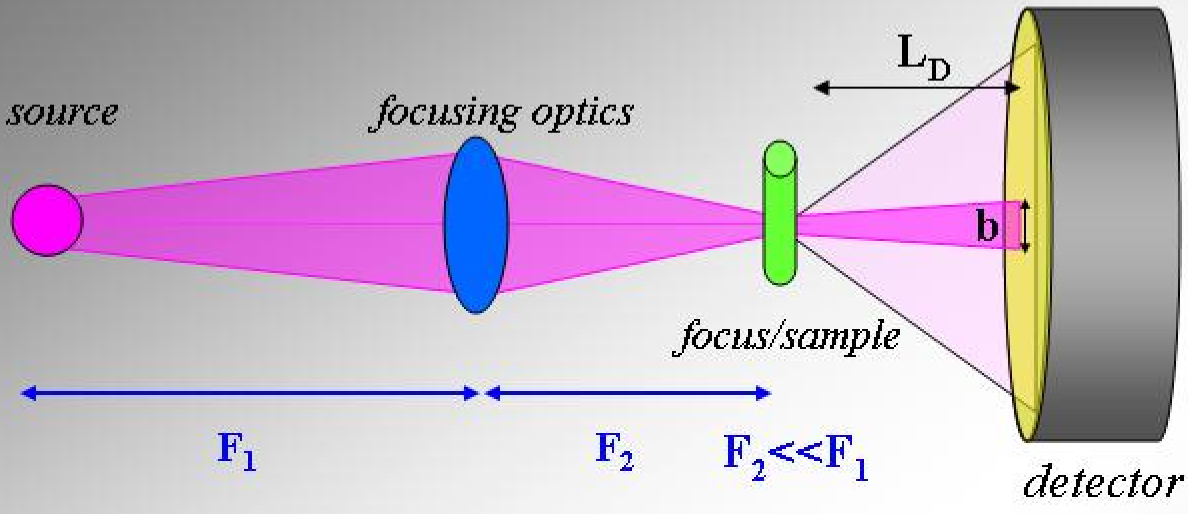
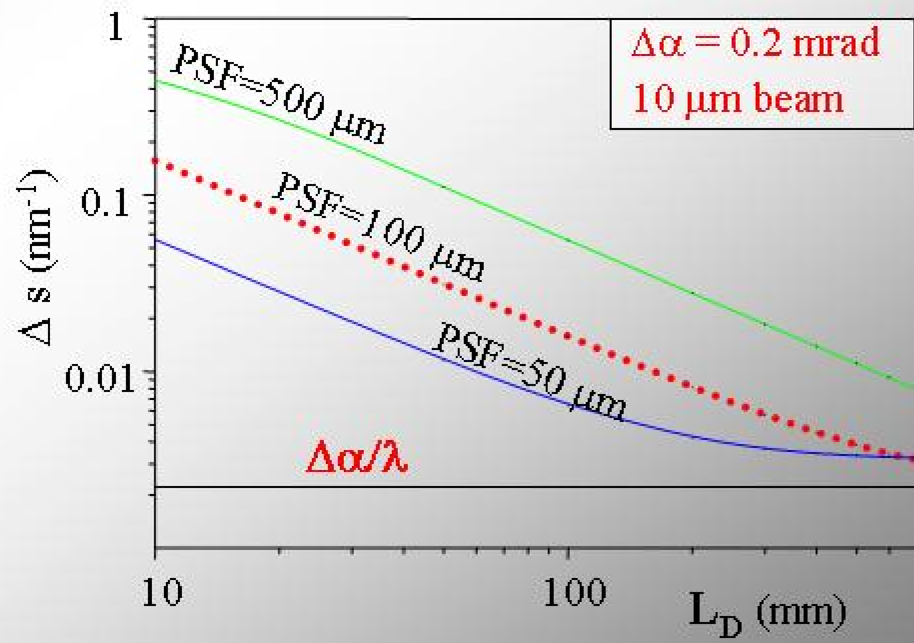


low-β undulator
$134_h * 25_v$ µm²
$0.2_h * 0.02_v$ mrad²

source/optics detector

$$\Delta s = \sqrt{(\Delta\alpha/\lambda)^2 + (\Delta\alpha_d/\lambda)^2}$$

$$\Delta\alpha_d = \sqrt{(b^2 + D_{res}^2)/L_D^2}$$



Micro-SAXS/WAXS

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dry collagen

combined SAXS/WAXS on single detector

requires microbeam and high resolution CCD

Riekel et al., *J. Appl. Cryst.* (2000) **33**, 421

Scanning set-up and KB-mirror

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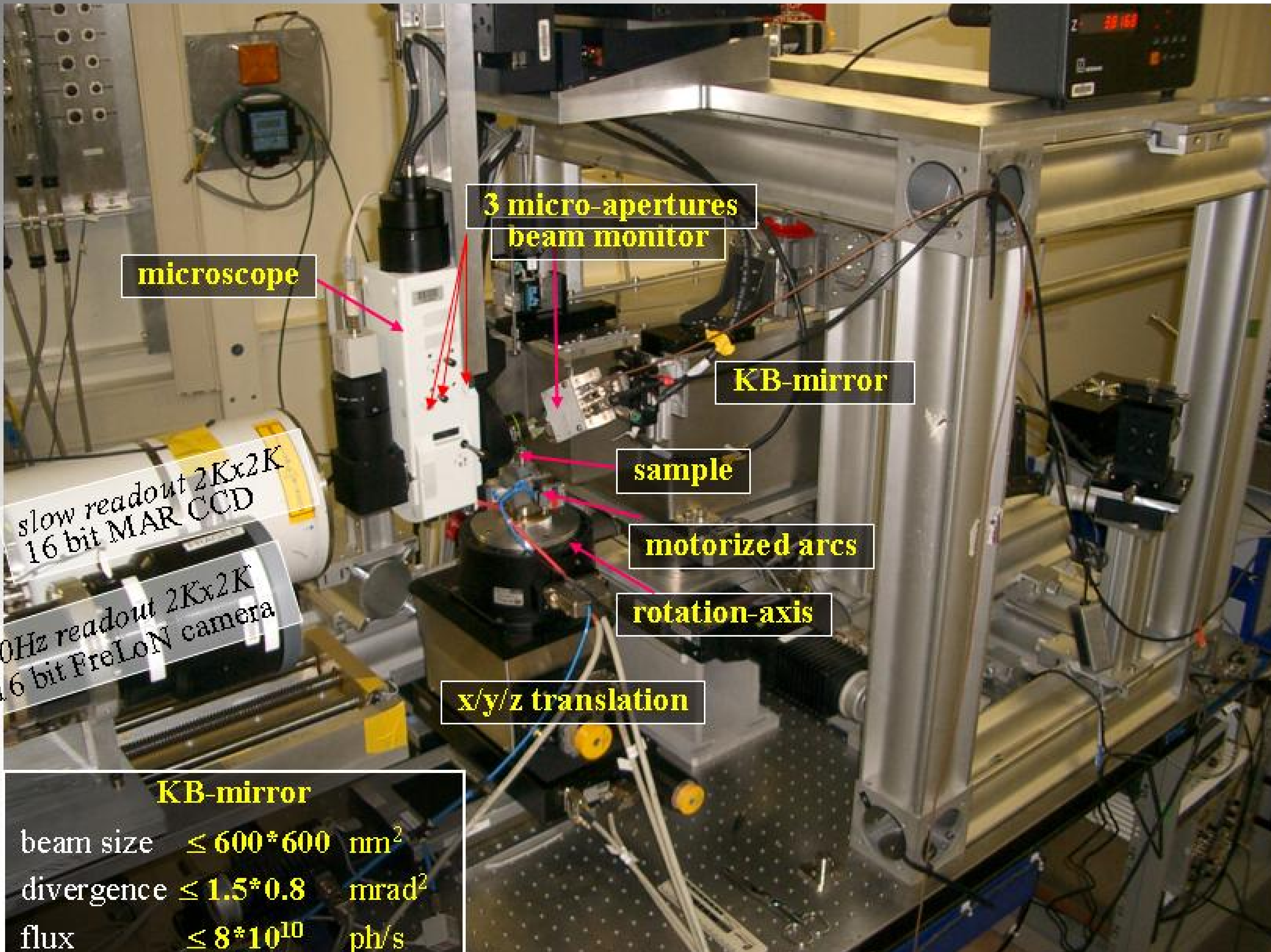
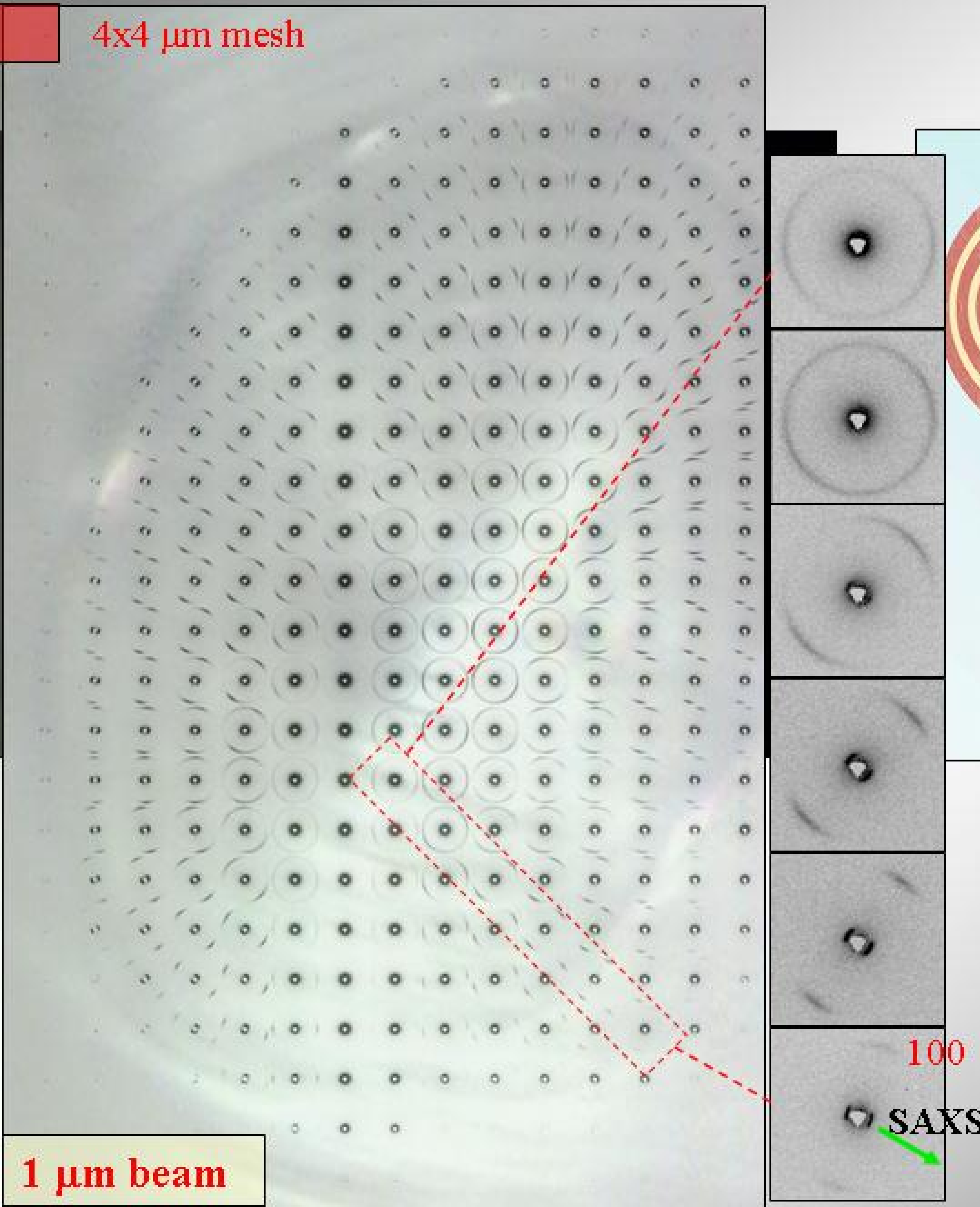
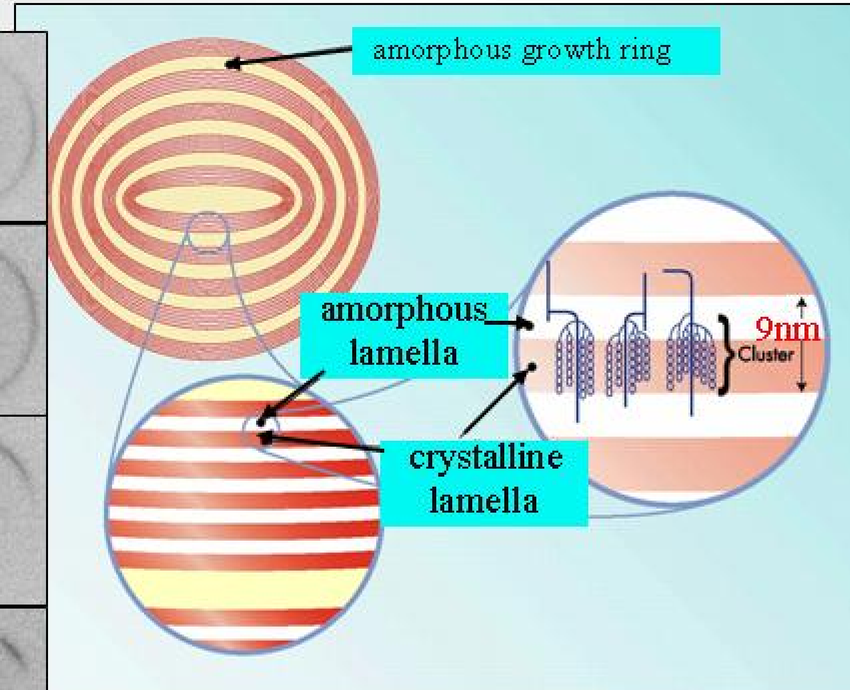


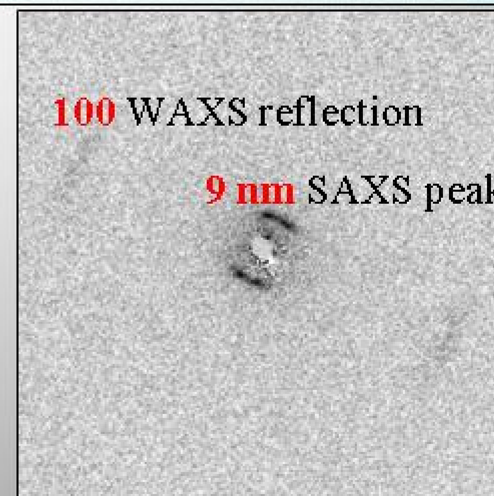
KB-mirror

beam size	≤ 600*600	nm^2
divergence	≤ 1.5*0.8	$mrad^2$
flux	≤ $8*10^{10}$	ph/s

SAXS/WAXS microscopy: potato starch granule







SAXS camera comparison

ESRF-ID13 5 µm beam (13 keV/Si-111)
1:2.3 refractive lens focus + collimator

divergence	**≈ 50**	µrad
Q_{min}	**≈ 0.045**	nm^{-1}
flux	**≈$5*10^9$**	ph/s

$ERL_{1\mu m}$ $0.9*1.2$ μm^2 beam (15 keV/Si-111)
1:10 KB-mirror focus

divergence	**181_h*107_v**	µrad
Q_{min}	**≈0.012**	nm^{-1}
flux	**≈$1.1*10^{11}$**	ph/s

$ERL_{5\mu m}$ 5 µm beam (15 keV/Si-220)
unfocused + collimator

divergence	**$17.6_h*14.1_v$**	μrad^2
flux	**≈ $2.5*10^{12}$**	ph/s

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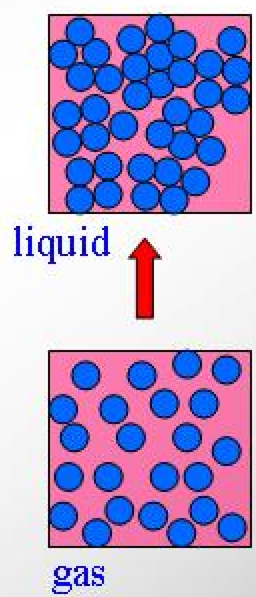
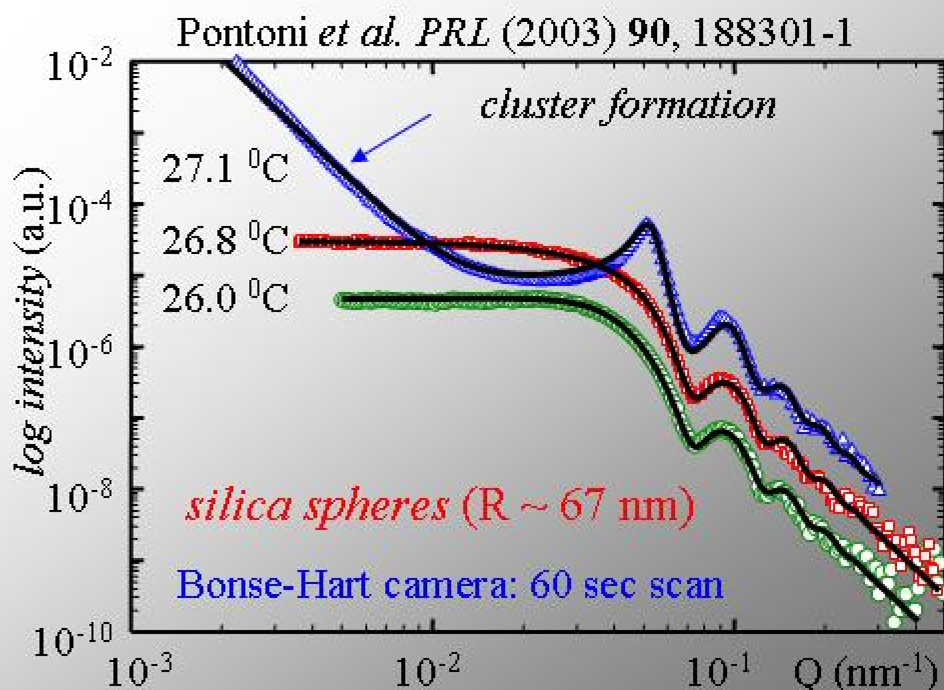
many applications in complex fluids, colloidal phase transformations, onset of crystallization processes…

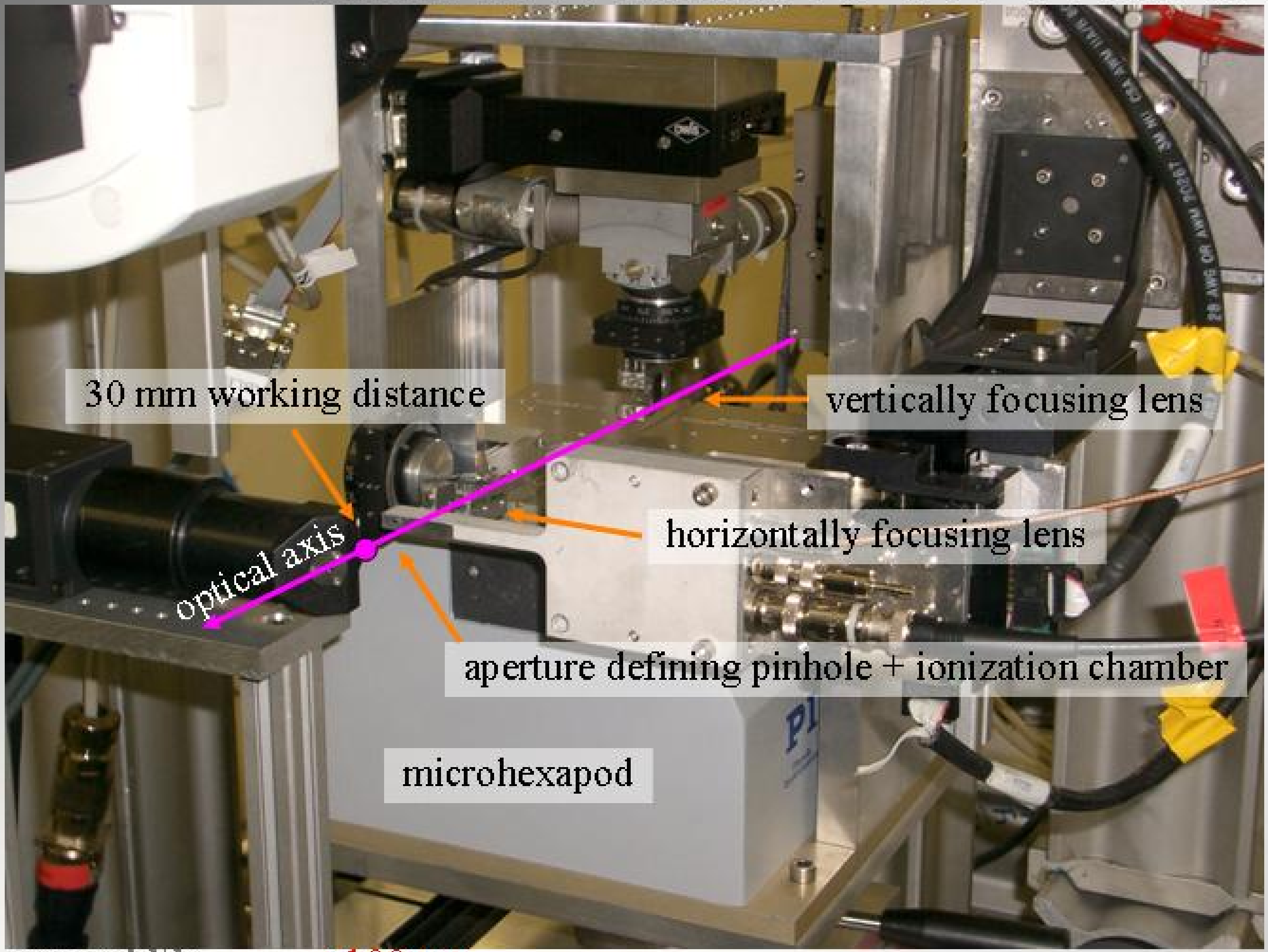
consider focusing camera with pinhole geometry covering range of Bonse-Hart camera

- *reduce divergence*
- *relax on beam size*
- *increase band width*





Coherence matched KB-Fresnel system



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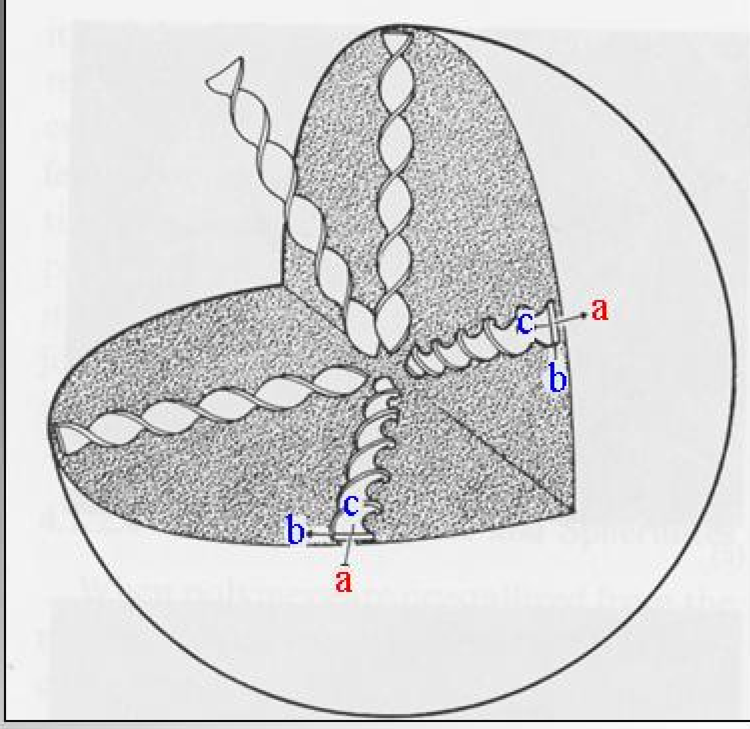
David et al., PSI

zone width:	≥100 nm	
lens diameter:	200_h(140)* 50_v (25) µm	Nöhammer et al., *APL* (2005) **86**, 163104
focal spot:	**300*300**	nm^2 routinely used; **140 nm** demons
divergence:	**1**	mrad
Q_{min}	**≥0.1**	nm^{-1}
flux:	**$1*10^{10}$**	ph/s (Si-111; 12.7 keV)

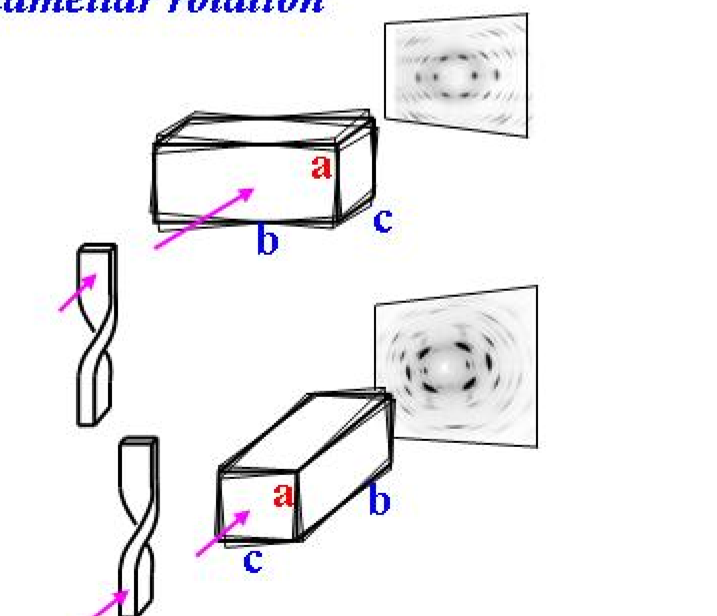
ERL - 100:1 ⟶ **118 nm** beam

divergence	**≈ 1.4**	mrad
Q_{min}	**≈ 0.12**	nm^{-1}
flux	**$\approx 10^{11}$**	ph/s (Si-111; 15 keV)

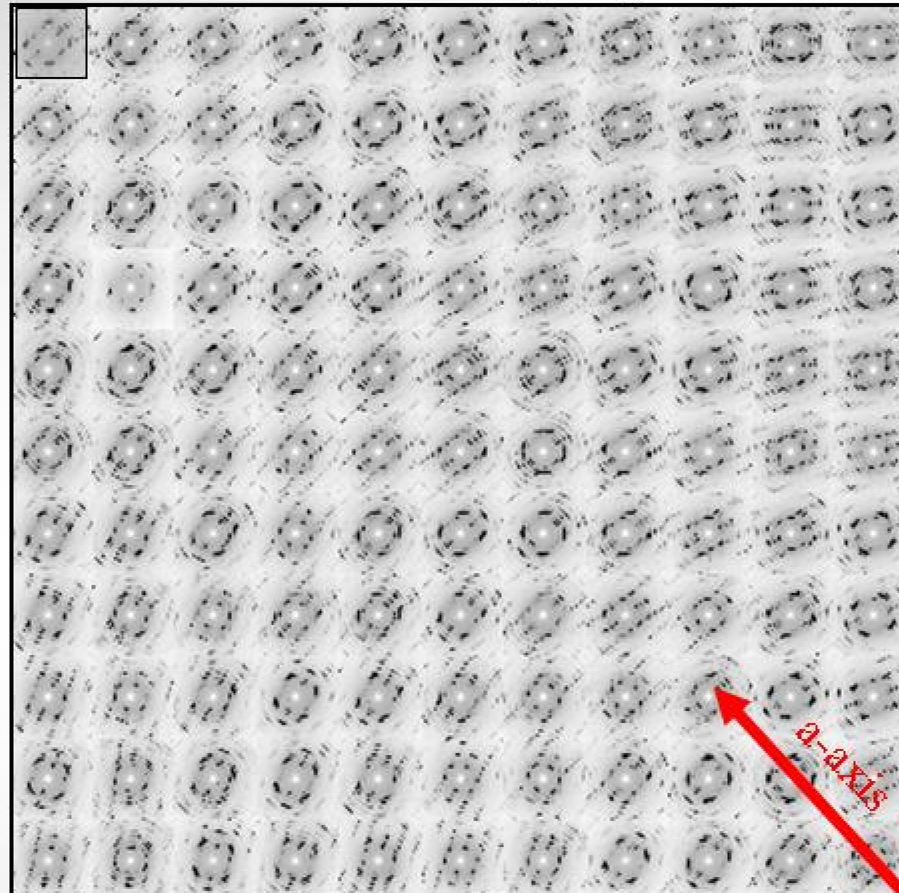
Spherulites



lamellar rotation



*mesh-scan: 3*3 μm² grid*



3 μm beam: 10 sec/frame

Gazzano et al., *Macrom. Chem. Phys.* (2001) **202**, 1405

SAXS/WAXS microscopy prospects

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current framing time: **1.1 sec** → 36000 frames in ≈11 hours

future framing time: **e.g. 1 msec** → 36000 frames in ≈36 sec

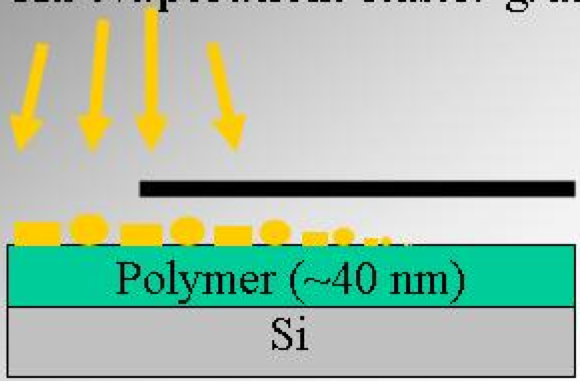
allows multiple composite image collection: texture analysis, slow crystallization, biocomposite materials, local deformation...

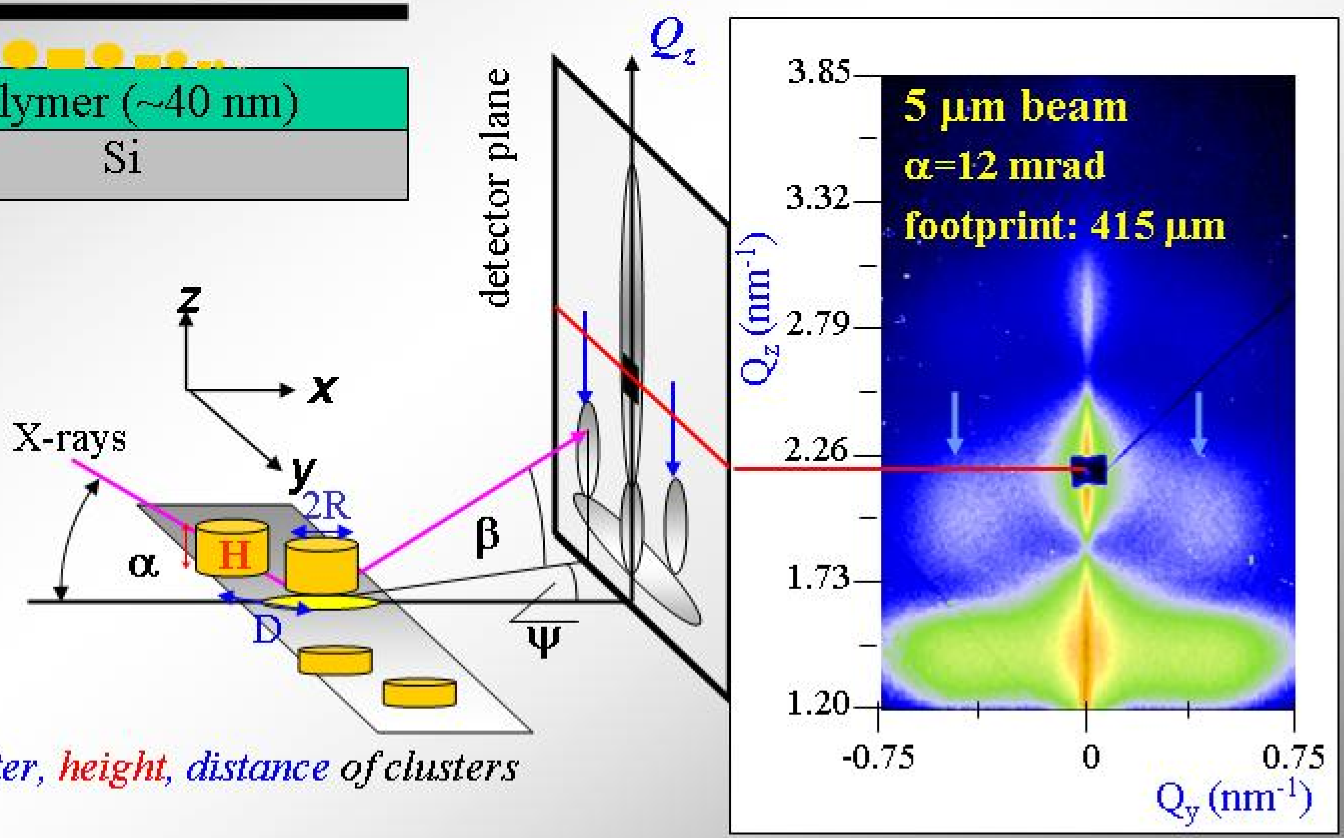
REQUIREMENTS

- fast scanning mode: *scan sample or focal spot?*
- detector: *single photon counting detector with fast readout; preferably pixel detector*
- software: *on-line data analysis and display*

Grazing incidence microSAXS (microGISAXS)

Au evaporation: *cluster gradient generation*





accessible: diameter, height, distance of clusters

Roth et al., *APL* (2003) **82**,1935

NanoGISAXS

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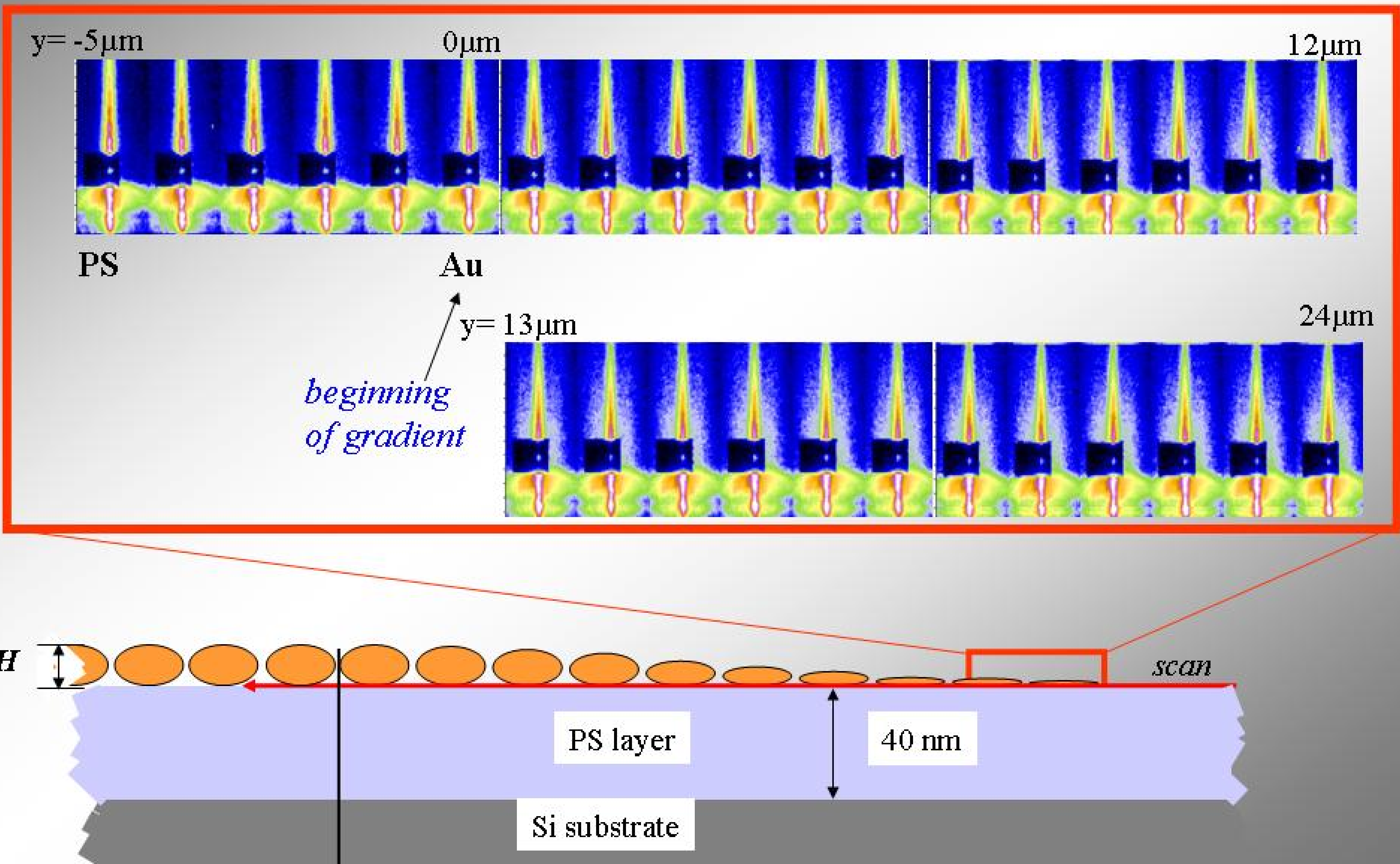
300nm KB-Fresnel beam – footprint @ 12 mrad: **≈25 µm**

λ=0.097 nm



Potential nanoGISAXS applications

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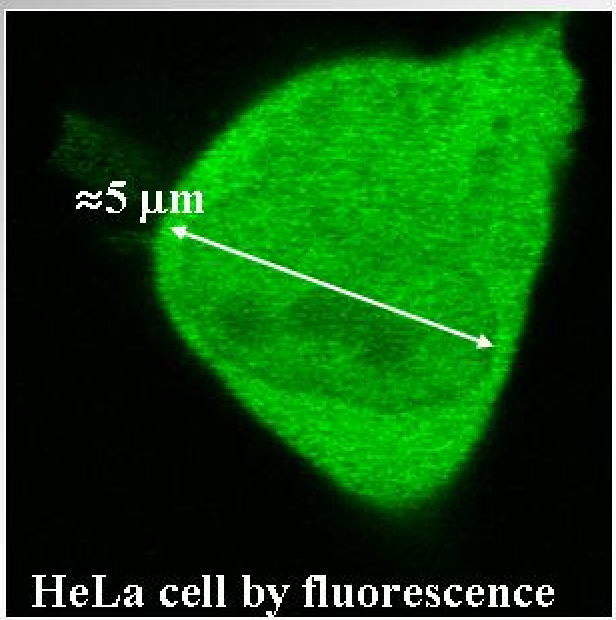
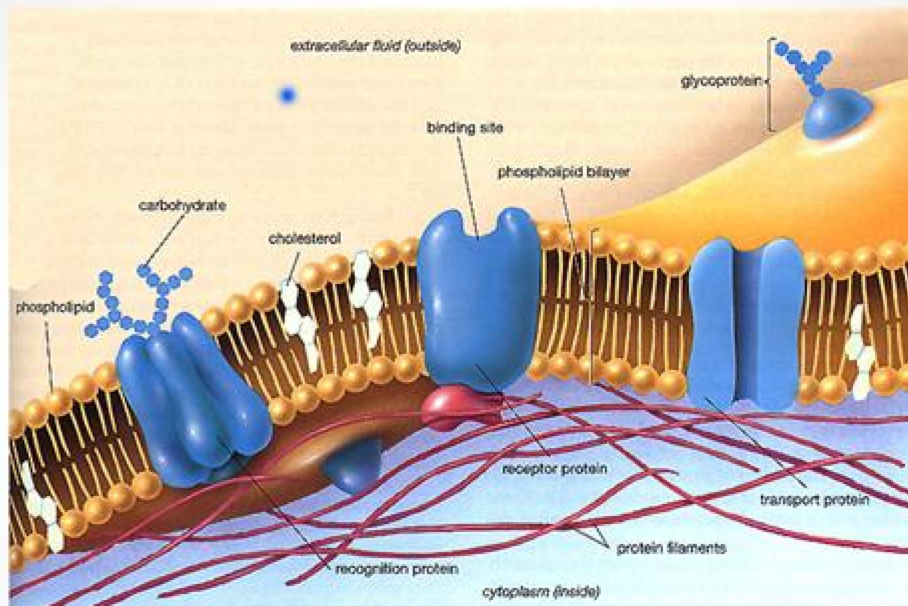
beam	≈ 100	nm
divergence	≈1	mrad
footprint	<20	μm

lighter atom surface layers (e.g. proteins), surface layers on fibers, confined environments





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Summary of possible ERL SAXS/WAXS optics

* **spot size and divergence:** *adaptable to sample requirements*
* **modular optics**: *on axis focusing, possibly by refractive lenses*
* **complimentary add-on optics:** *for special (e.g. coherency) applications*
* **beam size**: *from µm to 10th of nm*
* **beam divergence**: *from mrad to a few µrad*
* **camera length**: *≥100 m in order to obtain working distance for the smallest spots*

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scope of micron and submicron beams

status and possible evolution of microSAXS/WAXS cameras

controlling and probing of small sample volumes

Hydration of potato starch granule

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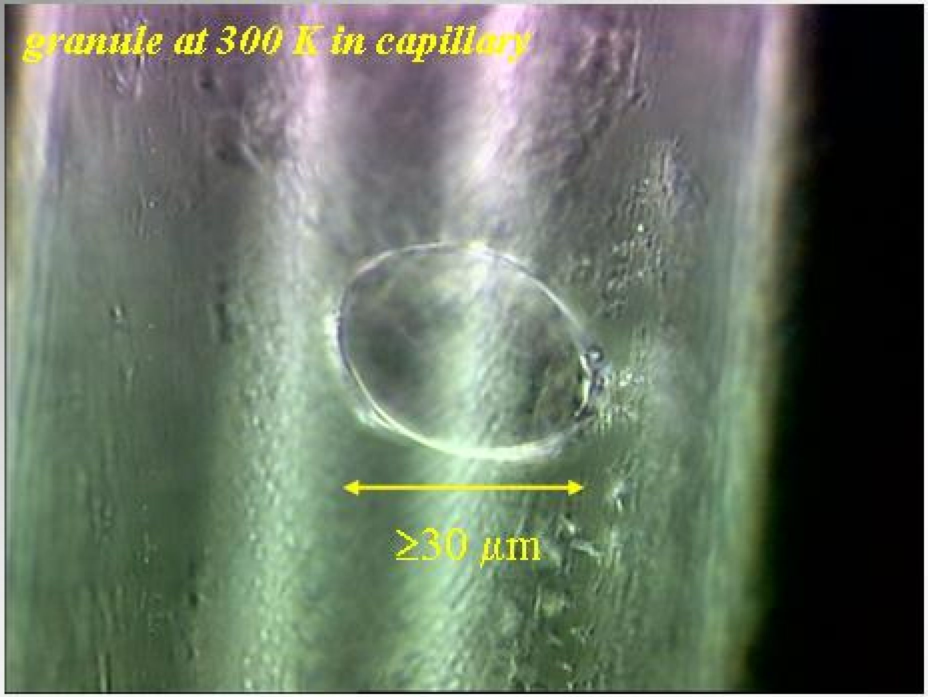
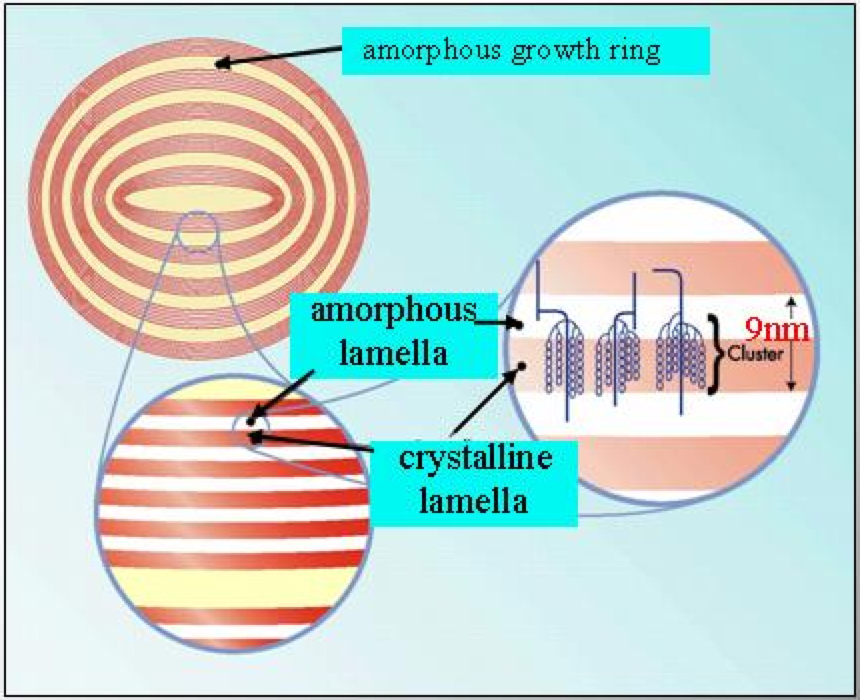
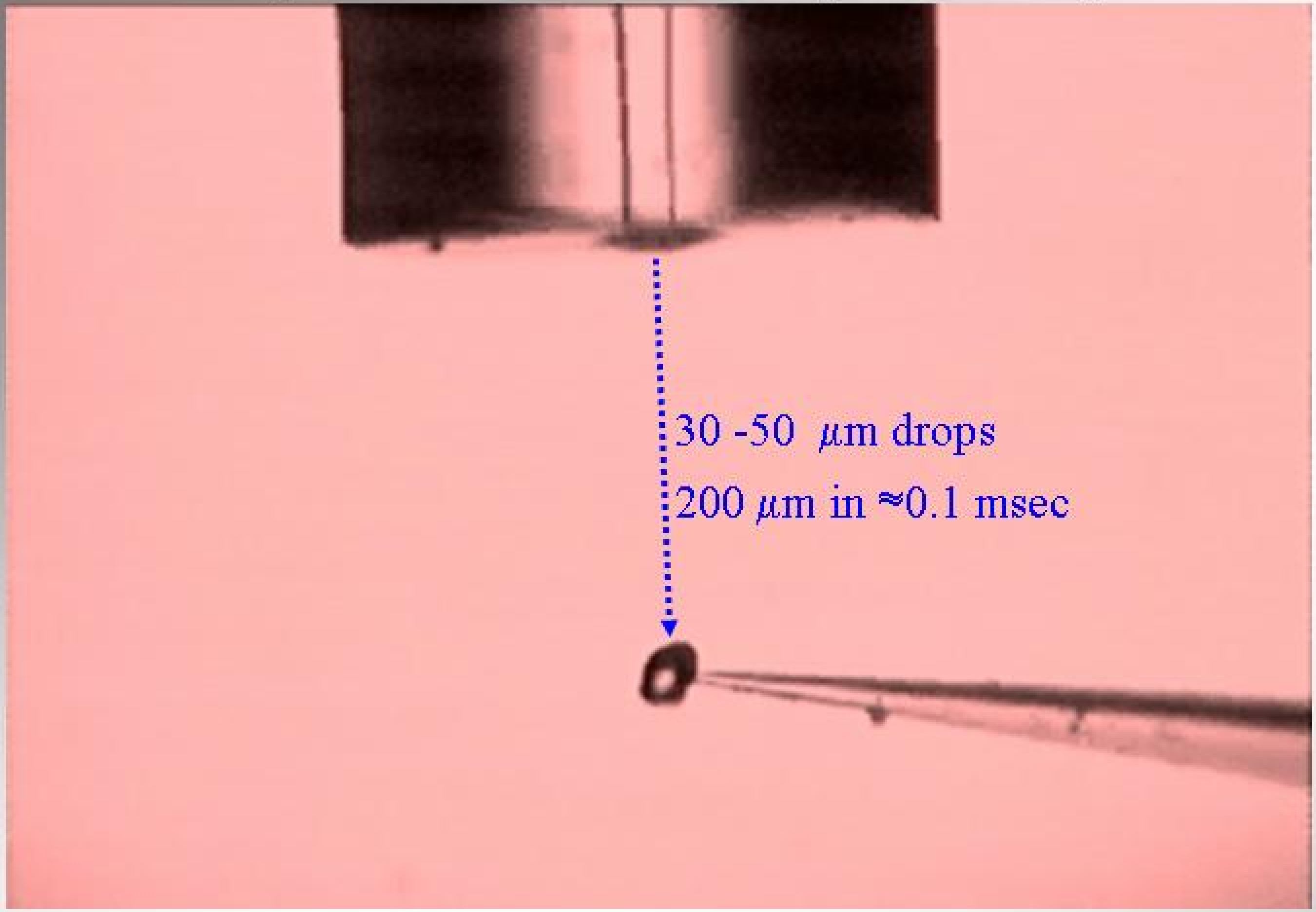
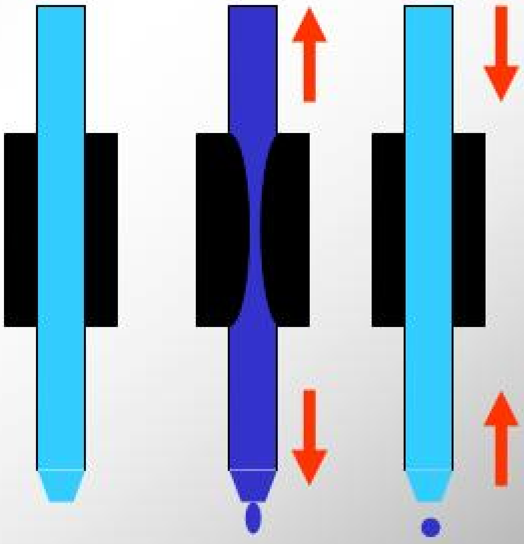
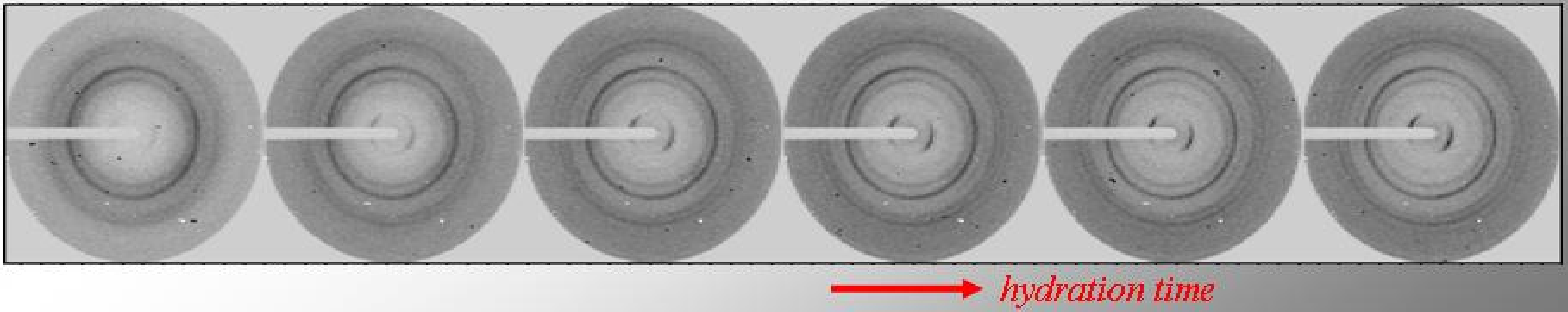




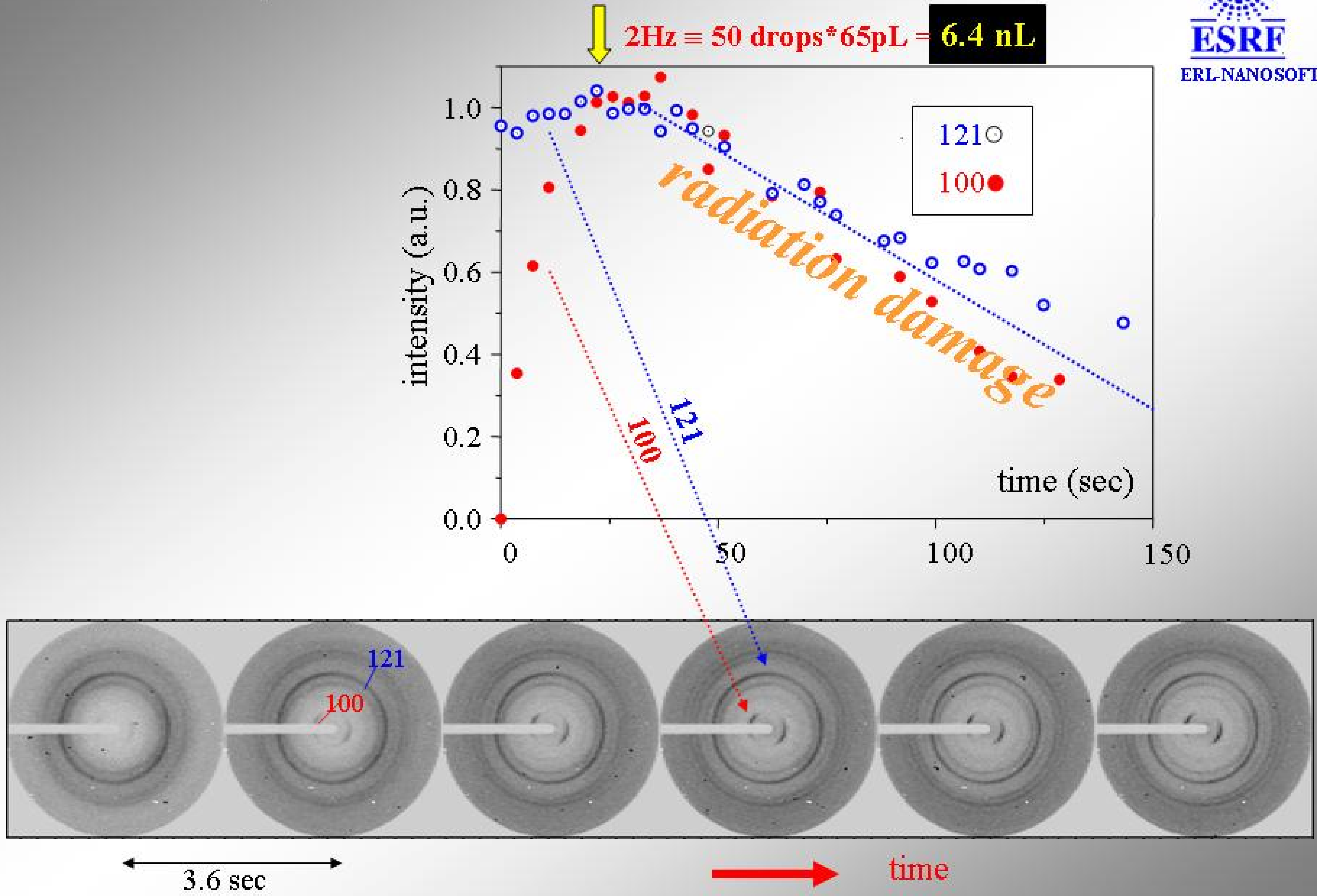
Microfluidics: microdrop starch granule hydration







Picoliter hydration kinetics



Lemke et al., *Biomacromolecules* (2004) **5**, 1316

Microfluidics: mixing by microdrop generators

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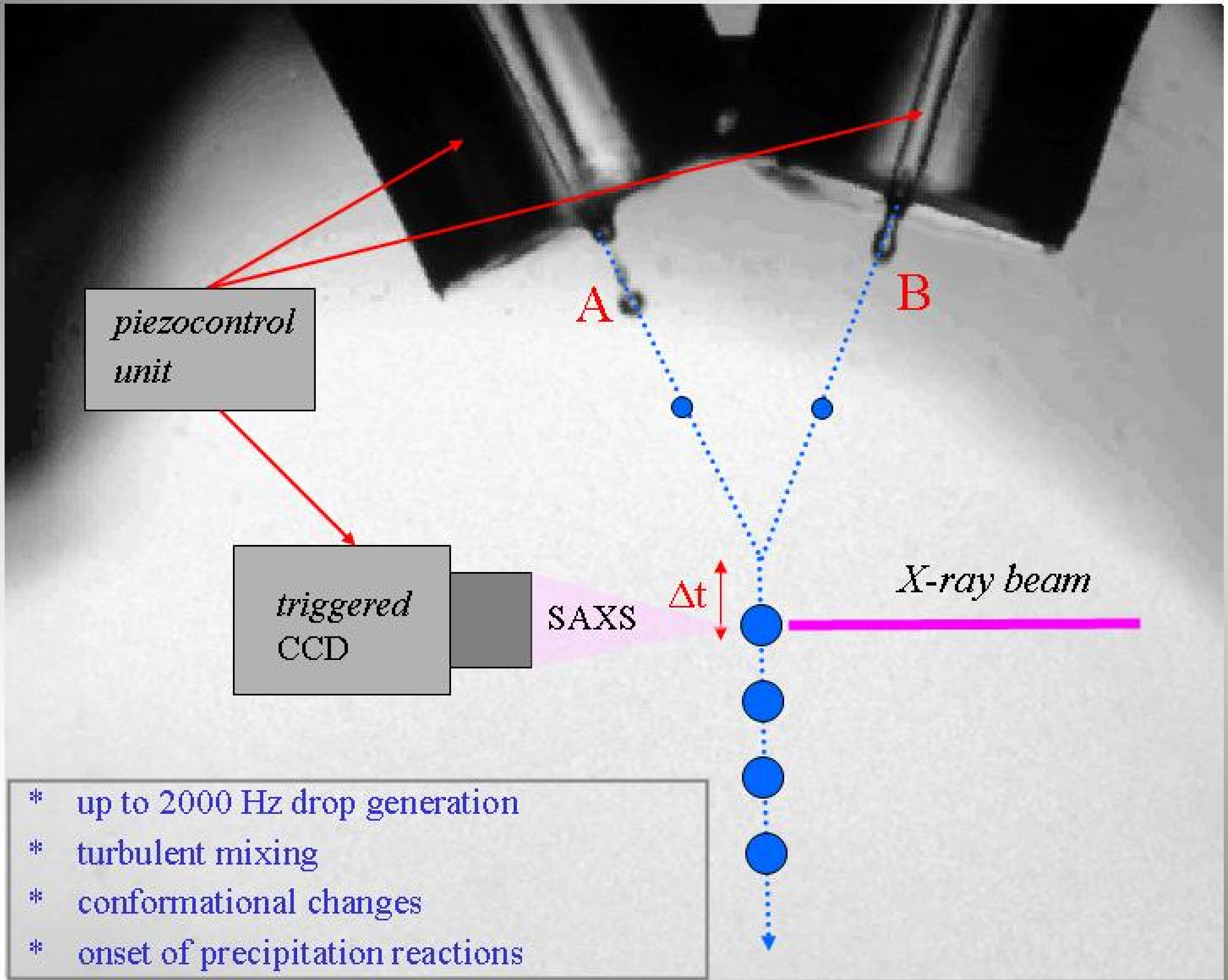
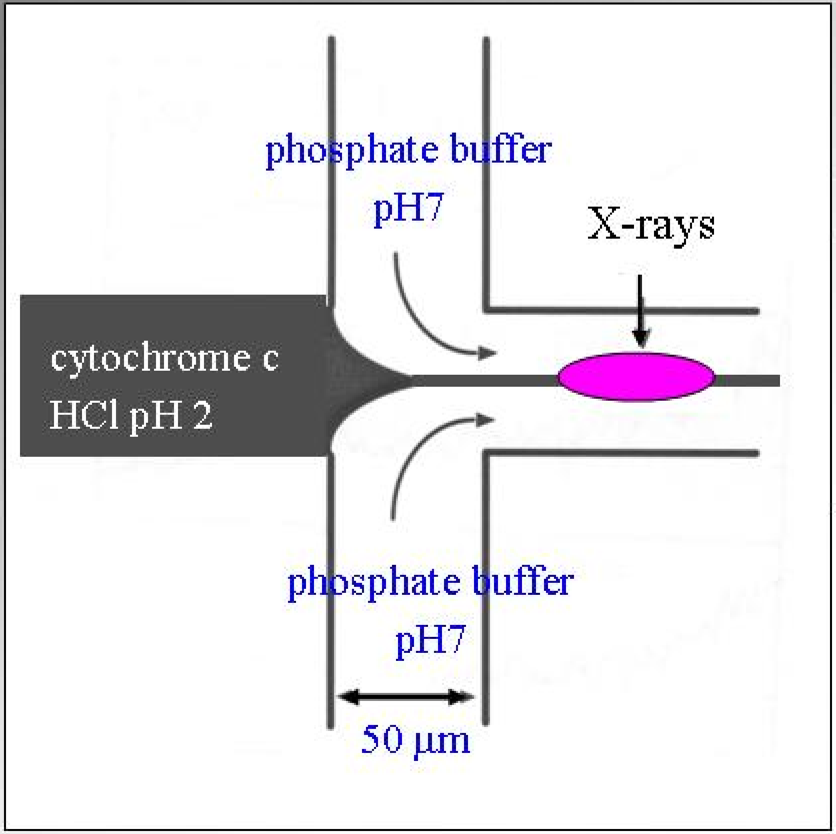
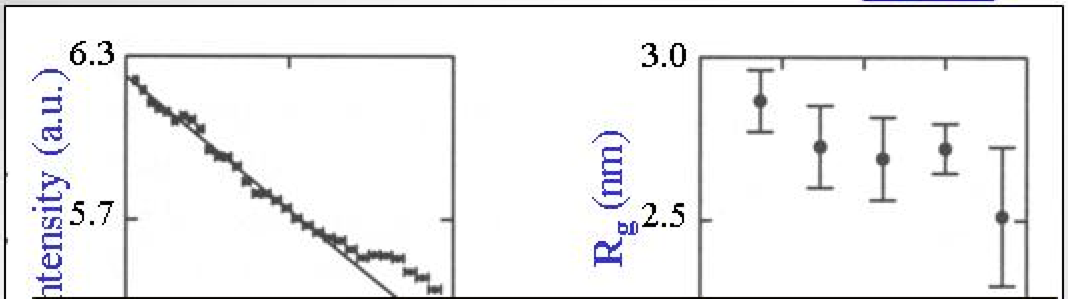


* up to 2000 Hz drop generation
* turbulent mixing
* conformational changes
* onset of precipitation reactions

Lamellar mixing by microfluidic device





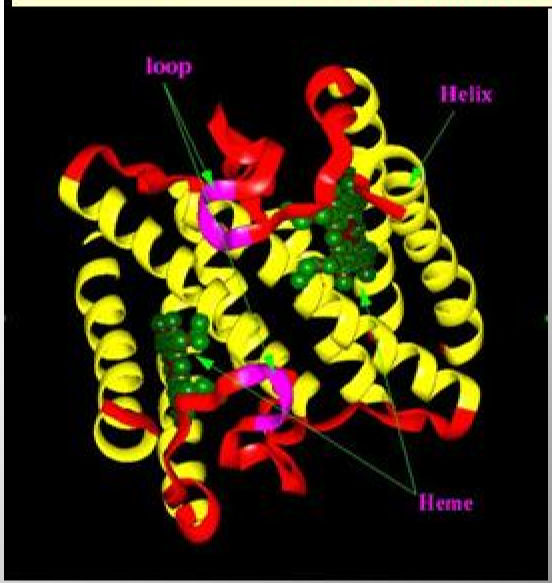
SCOPE and POTENTIAL

* ***phase transformations, micro-rheology***

* ***online biology & chemistry***

* higher brilliance beams: ***lateral scans, interfaces***

* integration of ***micro-sensors*** and ***micro-analysis*** tools



compactation of protein by SAXS

Pollack et al., *PNAS* (1999) **96**,10115

Time resolved SAXS: stopped flow mixing

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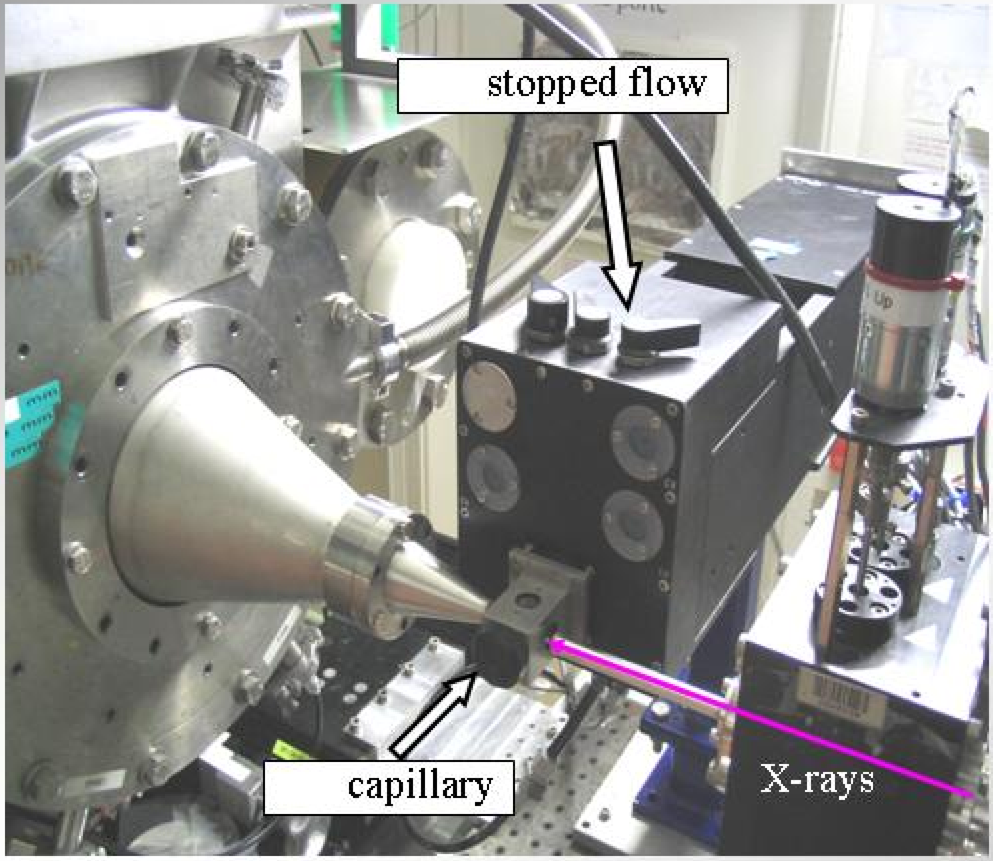
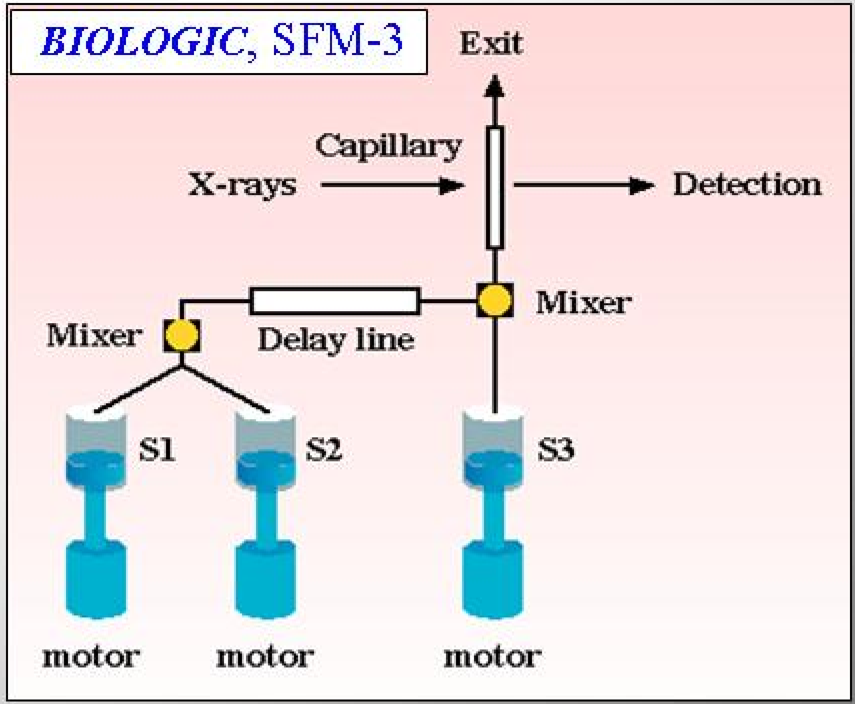
* rapid mixing of reactants in turbulent flow through mixers
* quartz capillary (wall thickness 10 μm, diameter 1.5 mm)
* dead time ≈4ms



ESRF-ID02 beamline



Time resolved SAXS: stopped flow mixing

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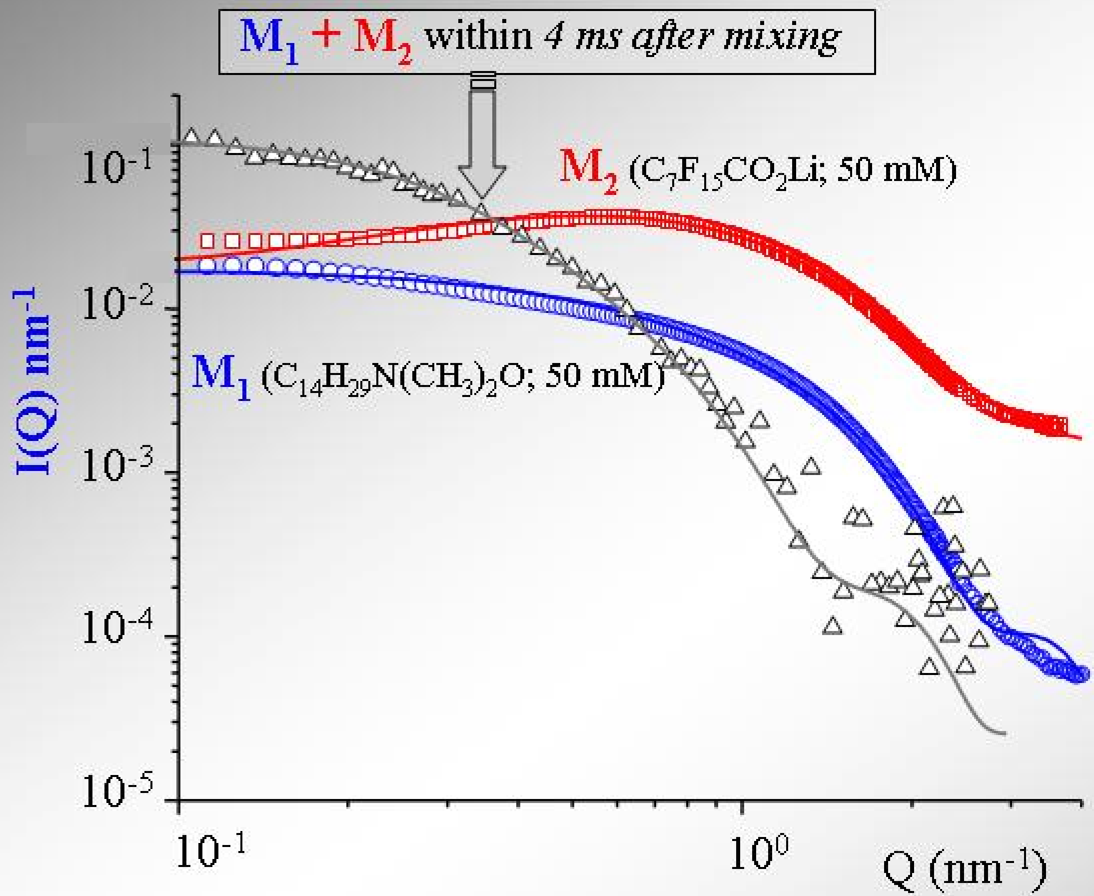

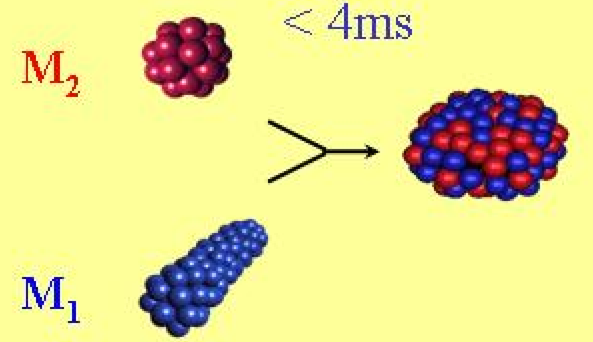

disk-like micelles with:
radius=7.5 nm; height=4.8 nm
are formed within the
mixing time (< 4 ms)

Weiss *et al.*, *PRL* (2005) **94**, 38303

ESRF ID02: ≈100 μm beam

Sample manipulation with optical tweezers

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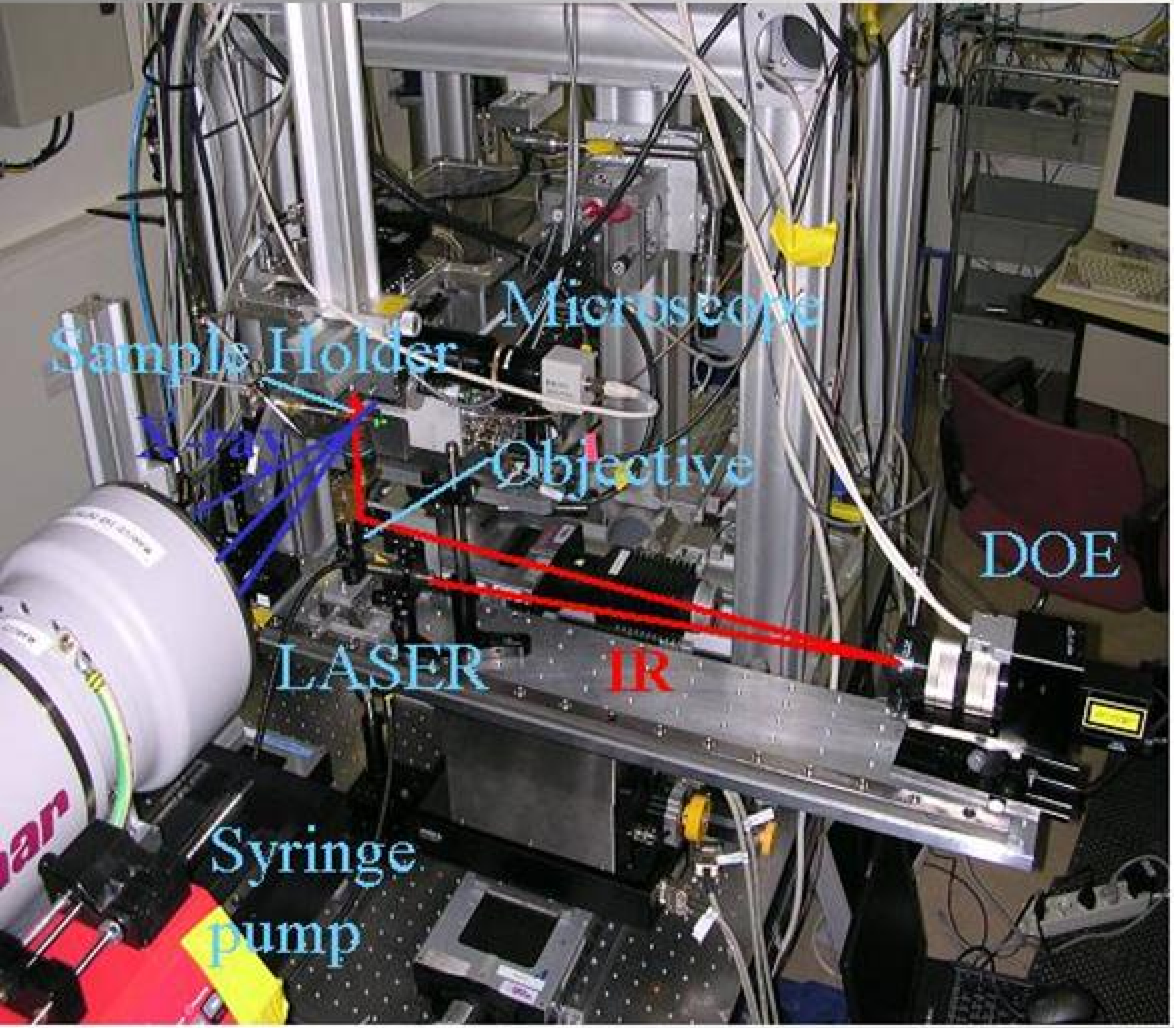

Amenitsch et al. Graz

advanced technology and nanoscience
TASC national laboratory
tecnologie avanzate e nanoscienza

Cojoc et al.
Trieste

optical tweezer set-up at the ID13 beamline including capillary holder, syringe pump and top microscope.

Amenitsch et al., SRI-2006 procedings

Optical tweezers

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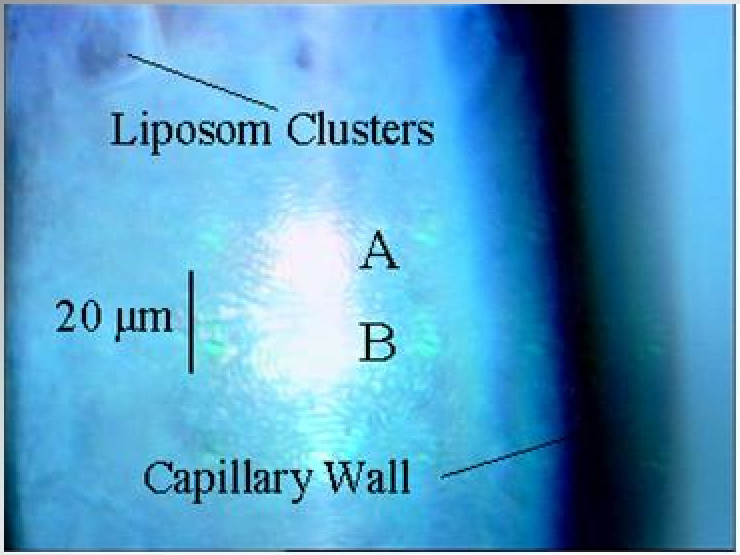
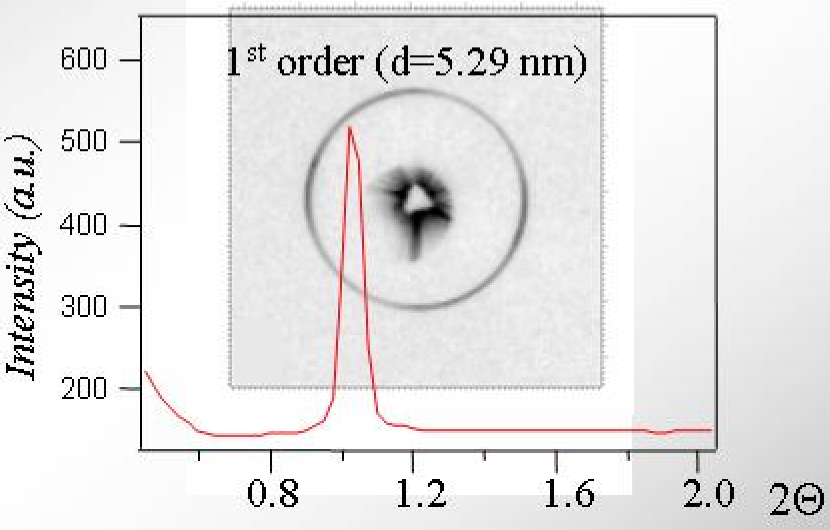


image of the ***two trapping spots*** *A, B in the capillary, under which two clusters of* ***POPE liposoms*** *have been trapped (not seen due to the intense IR light). A non trapped cluster is also indicated*



diffraction pattern obtained from a 10 µm large cluster of liposomes with an about ***1 µm beam****.*

Palmitoyl-Oleyl-Phosphatidylethanolamine

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Summary on sample environments

* radiation damage effects can be limited by active scanning of sample through beam or microfluidic systems

* mixing of nano/picoliter volumes by microfluidic systems can give access to subms timescales

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M. Roessle	(EMBL-Hamburg)	microdrop system
S. Roth	(Hasylab)	micro-GISAXS, refractive lens SAXS
H. Lemke	(Kopenhagen)	microdrop system, starch hydration
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R. Davies	(ESRF-ID13)	batch processing software, microRaman