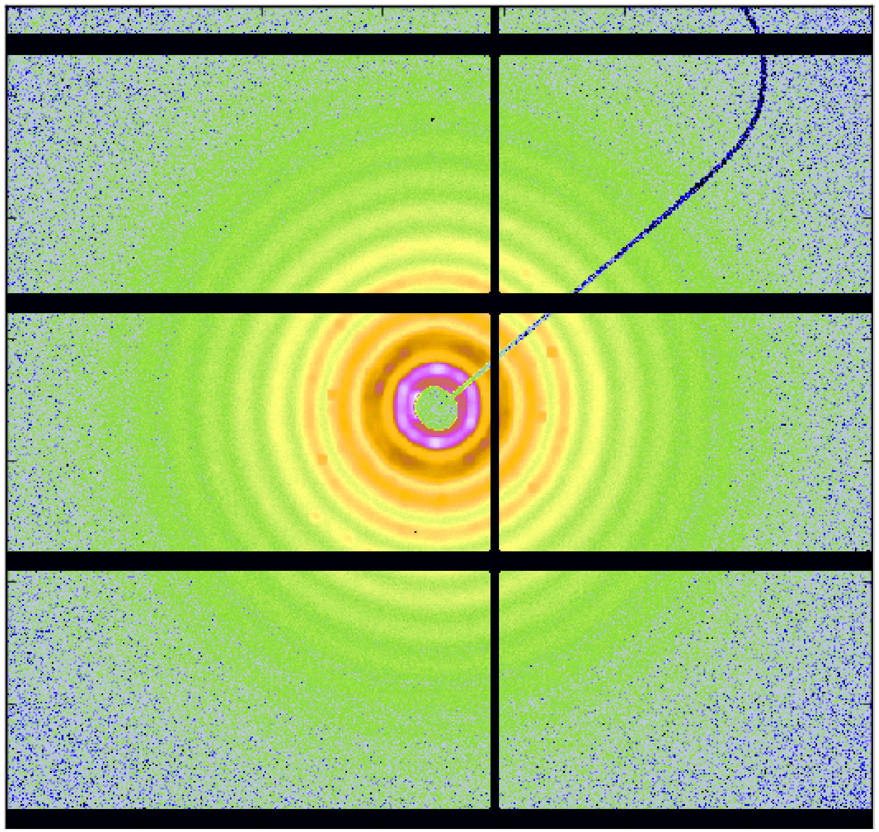
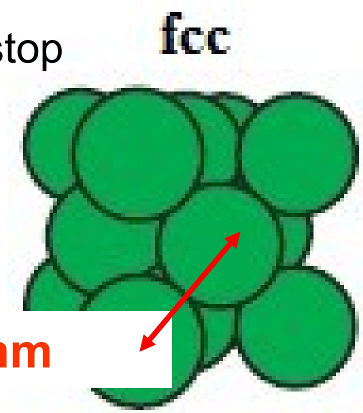
SAXS continued

Illustration

- USAXS at photonic crystals
- USAXS in highly concentrated colloidal suspensions



Beamstop



270 nm

http://ab-initio.mit.edu/book

http://lamp.tu-graz.ac.at/~hadley/ss1/emfield/photonic_crystals/photonic_table.html

Courtesy: V. Boyko (BASF)

Outline

- SAXS – Introduction
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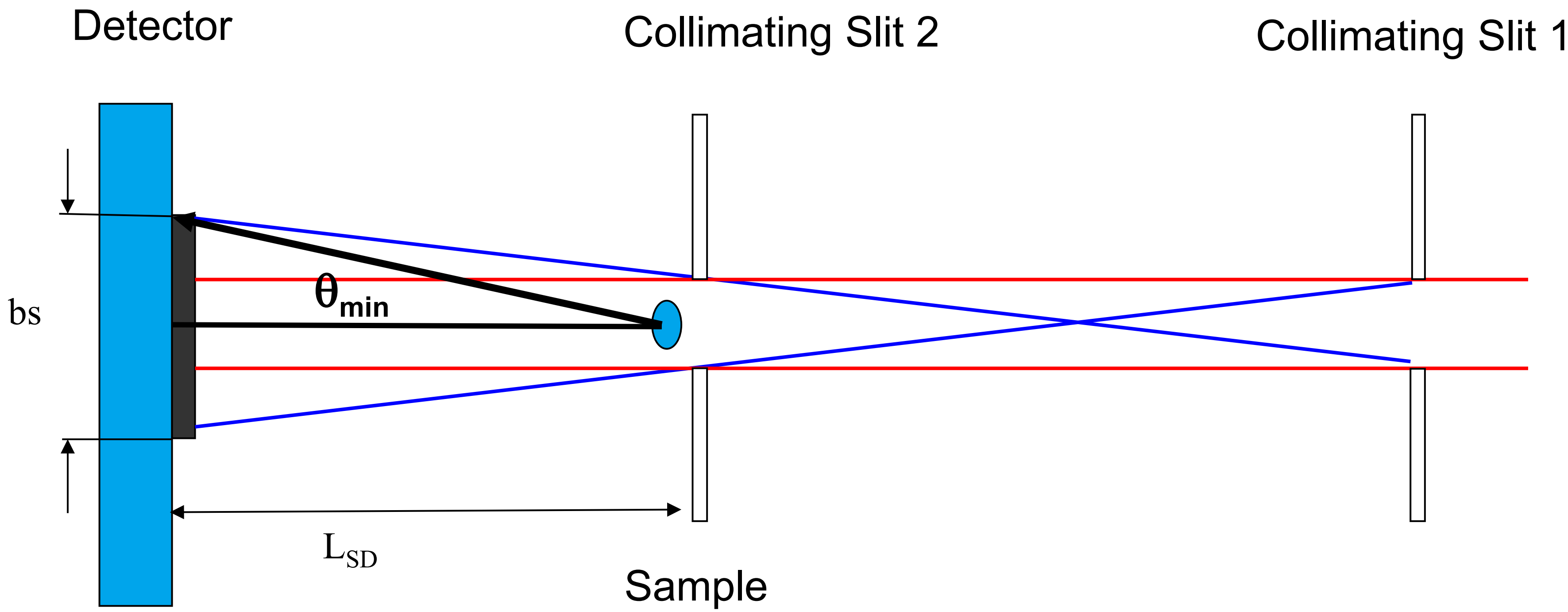
SAXS collimation and scattering geometry



L_{SD} determines resolution

$\theta_{min} = bs / (2\,L_{SD})$

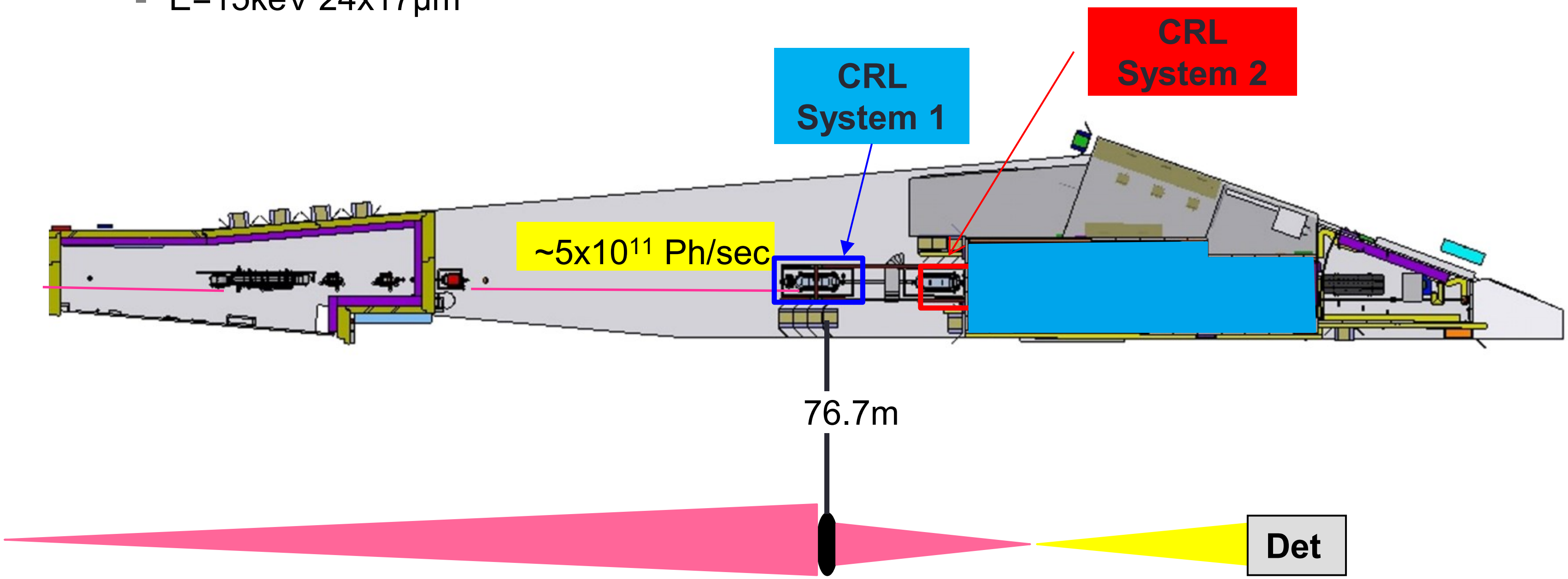
Use Bragg's law:

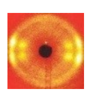
$$d_{max} = \frac{\lambda}{\theta_{min}}$$

Layout – Different μfocussing schemes

- Flexible choice of beam size and divergence
- Fixed focal spot position and size
  - E=13keV 22x12μm²
  - E=15keV 24x17μm²

- Full user operation within design values!

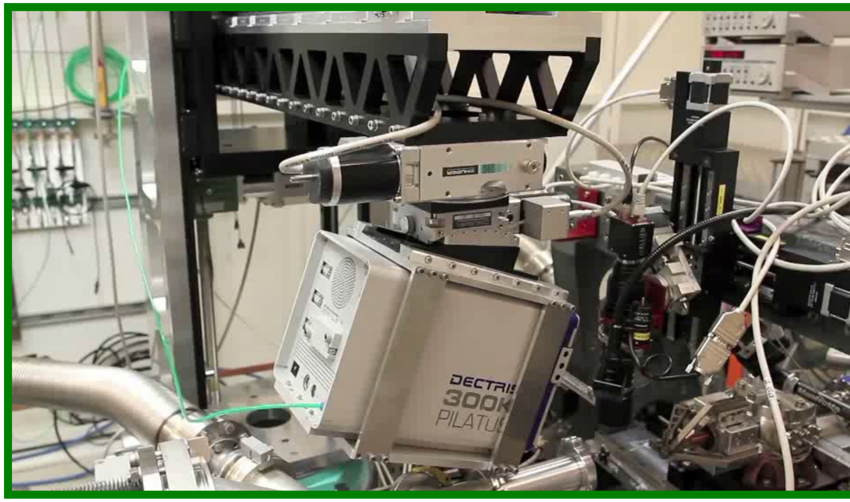


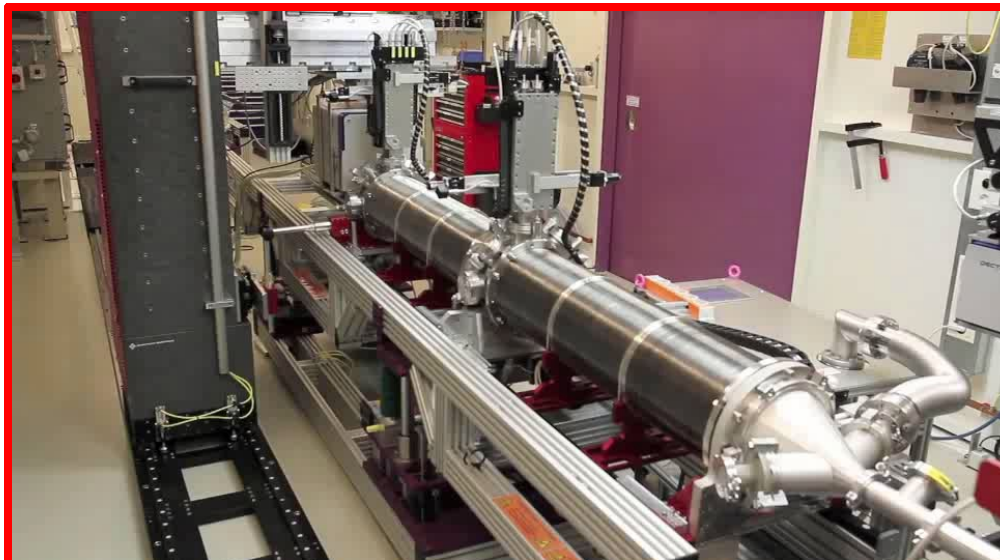
- Nanofocus end station: 
  - <1.5x1.5μm²
  - 500x500nm²

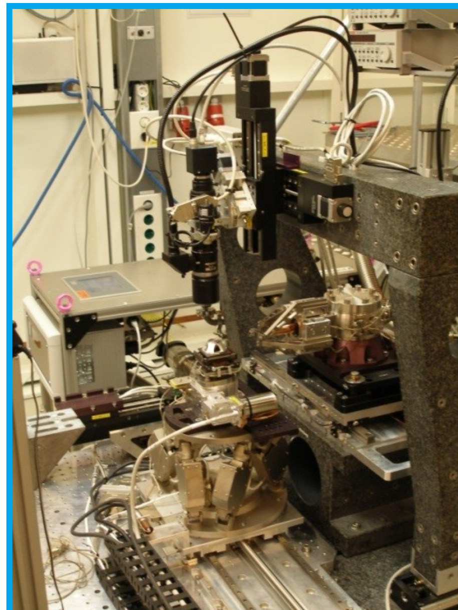
To be finished

Rapid change (GI)SAXS / (GI) WAXS - 2012

- Adjust scattering angles
  ↔ dΩ
  ↔q-ranges
- 5cm<D_{SD}<8.6m
- Highly flexible
- Separate WAXS device

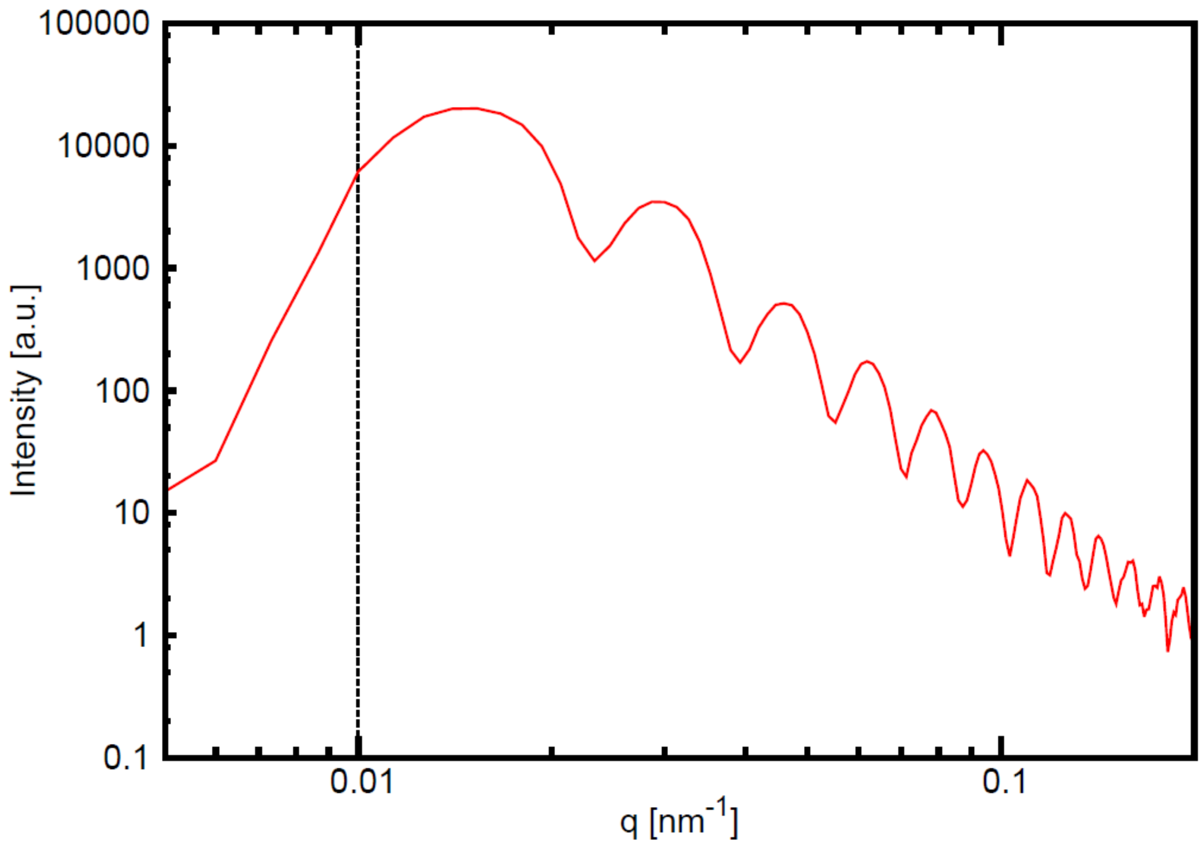






μUSAXS focus

- Beam size: 32x23μm²
- SDD=8470mm
- N_2=12
- PS particles:
  - 400nm
  - Dried on glass slide
  - t_{acq}=1s
  - background corrected



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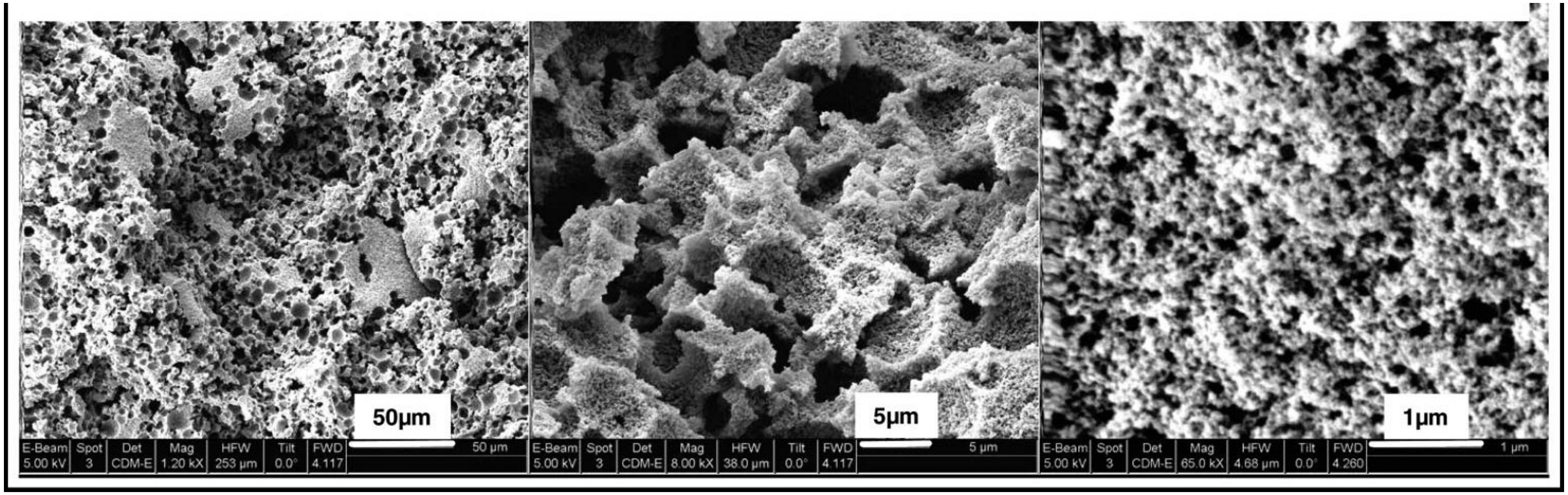
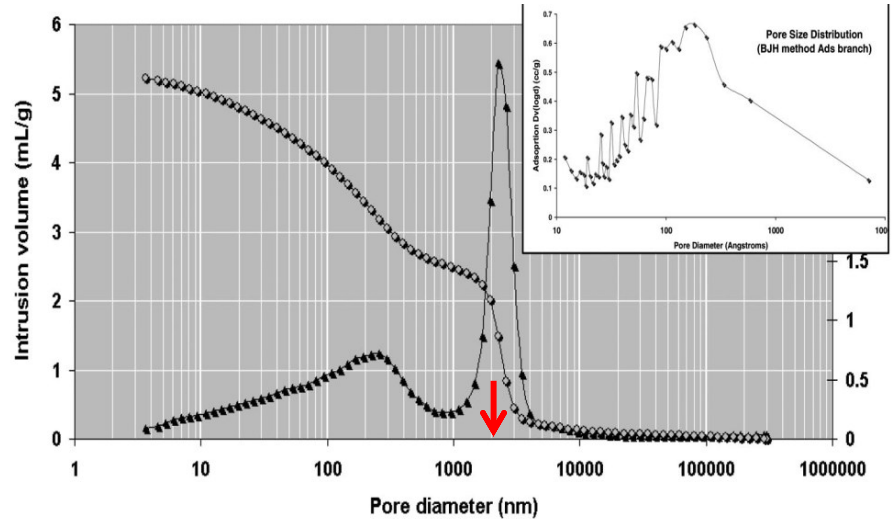
Aerogels

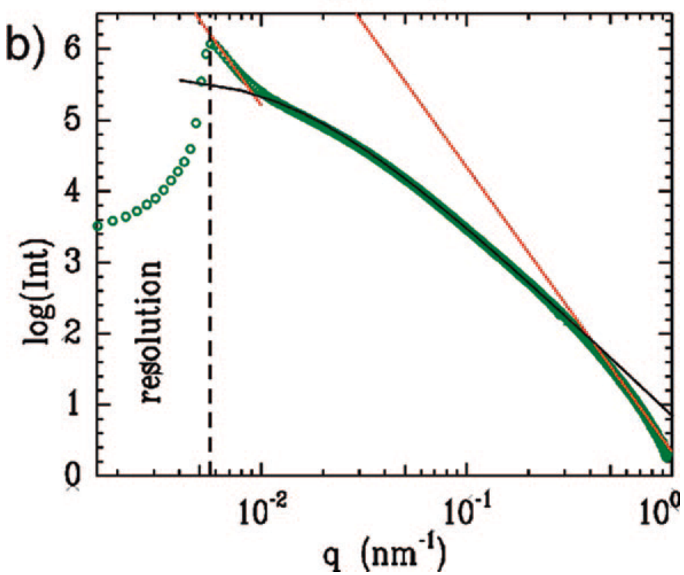
- Highly porous materials:
  - OLED: matching of refractive indices
  - molecular sieves
  - sensors
- Challenges
  - **Generation of** pores with dimensions greater than 100 nm, yet submicron
  - **Characterization of size**

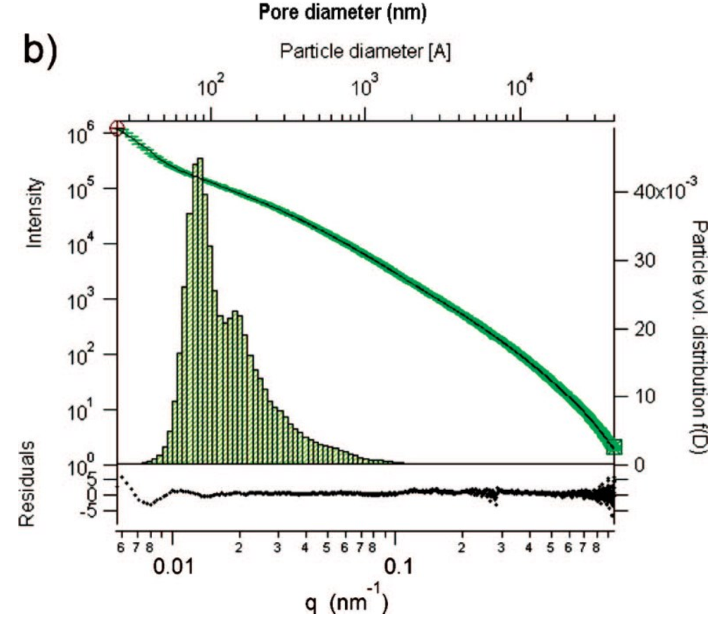


Quantitative analysis

- Bimodal distribution particles
- $I(Q) =$
  $\Delta\varrho^2 N \int_0^\infty V_P^2\,(R) P(R) * D(R) dR$
- Porod law:
  - Particle size ~ 30nm
  - Pore size estimate >1300nm







Eggers, SVR et al., Langmuir **24**, 5887 (2008)

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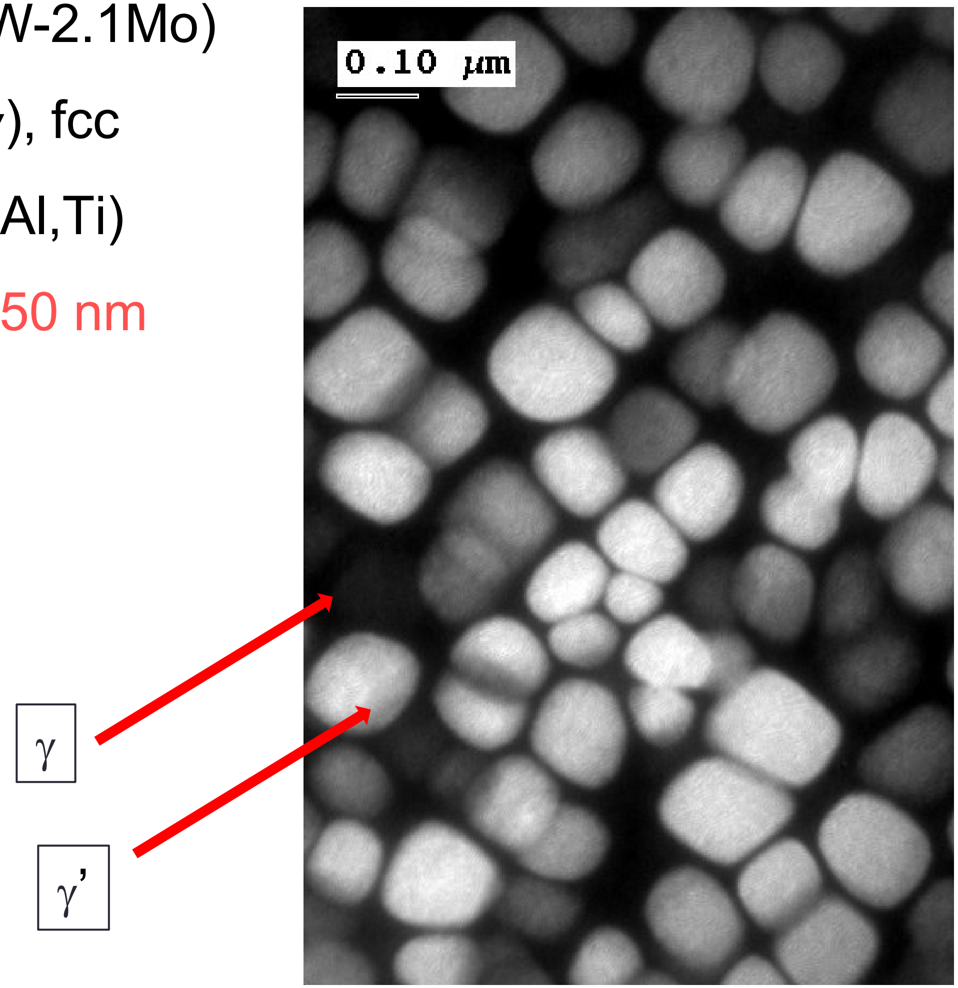
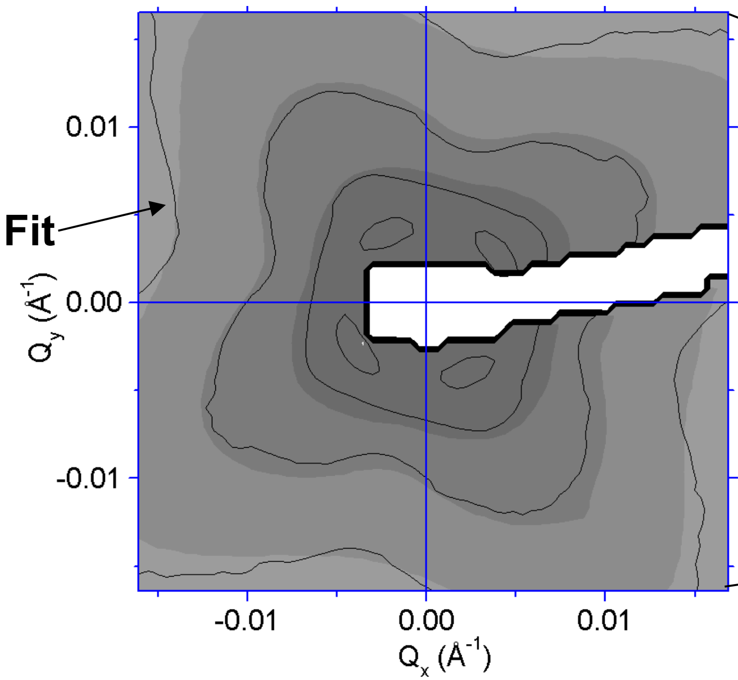
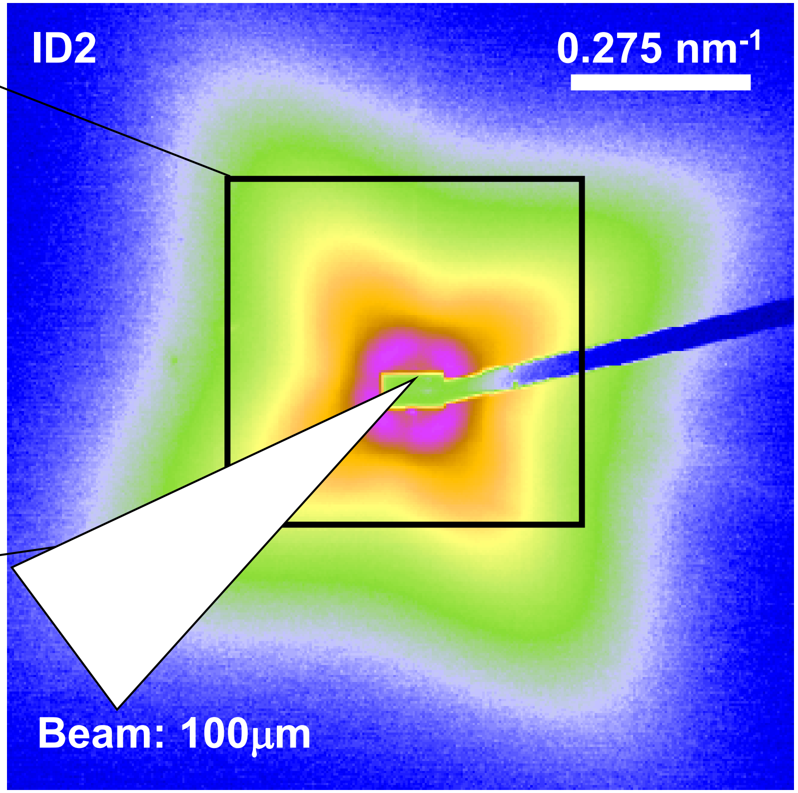
Ni-base superalloys

- Ni-base W-rich experimental single crystal superalloy (Ni-4.6Al-6.4Ta-5.7Cr-10.8W-2.1Mo)
- Ni-Al solid solution *Matrix* (γ), fcc
- *Precipitates* ($\gamma' \rightarrow$ Al,…), $Ni_3(Al,Ti)$
- TEM: γ'-precipitates   R > 50 nm
- D > 100 nm

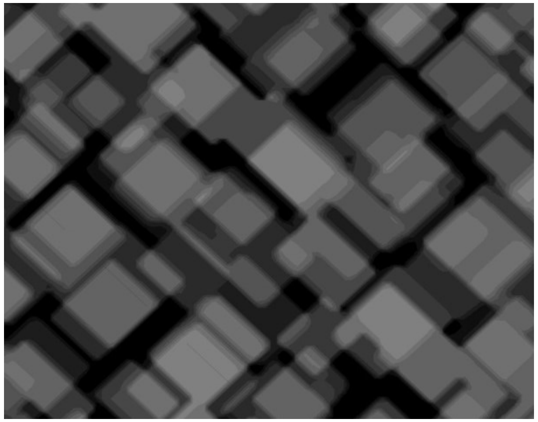


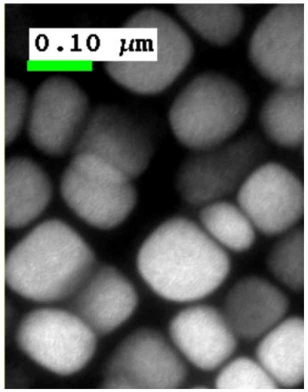




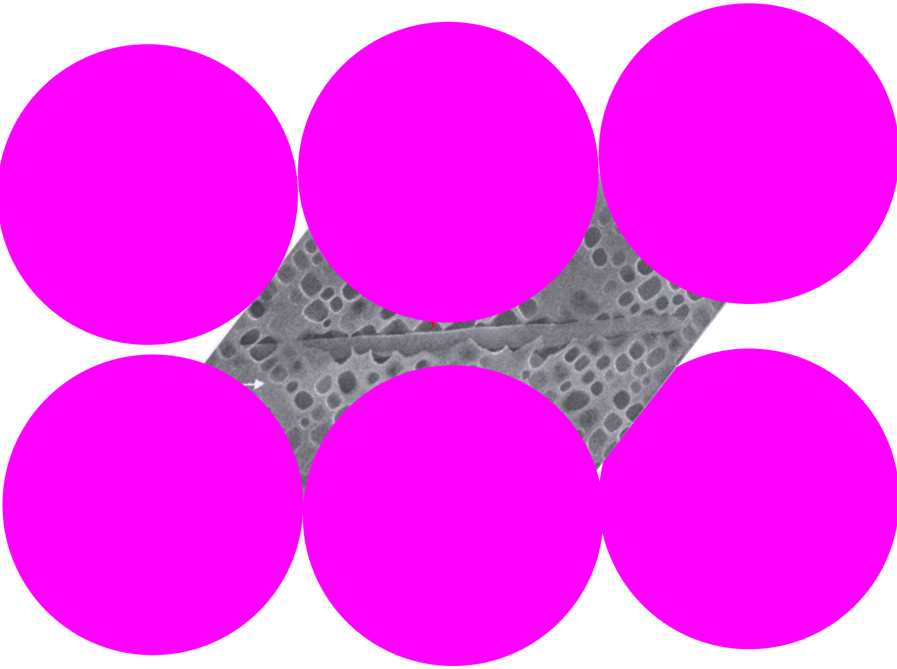
noc_4: P. Strunz et al., J. Appl. Cryst. **36**, 854 (2003)

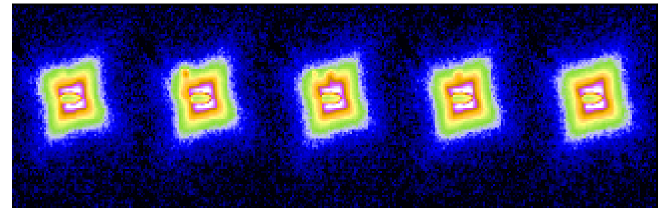
Courtesy:
Gilles
Strunz





Local precipate morphology - µSAXS





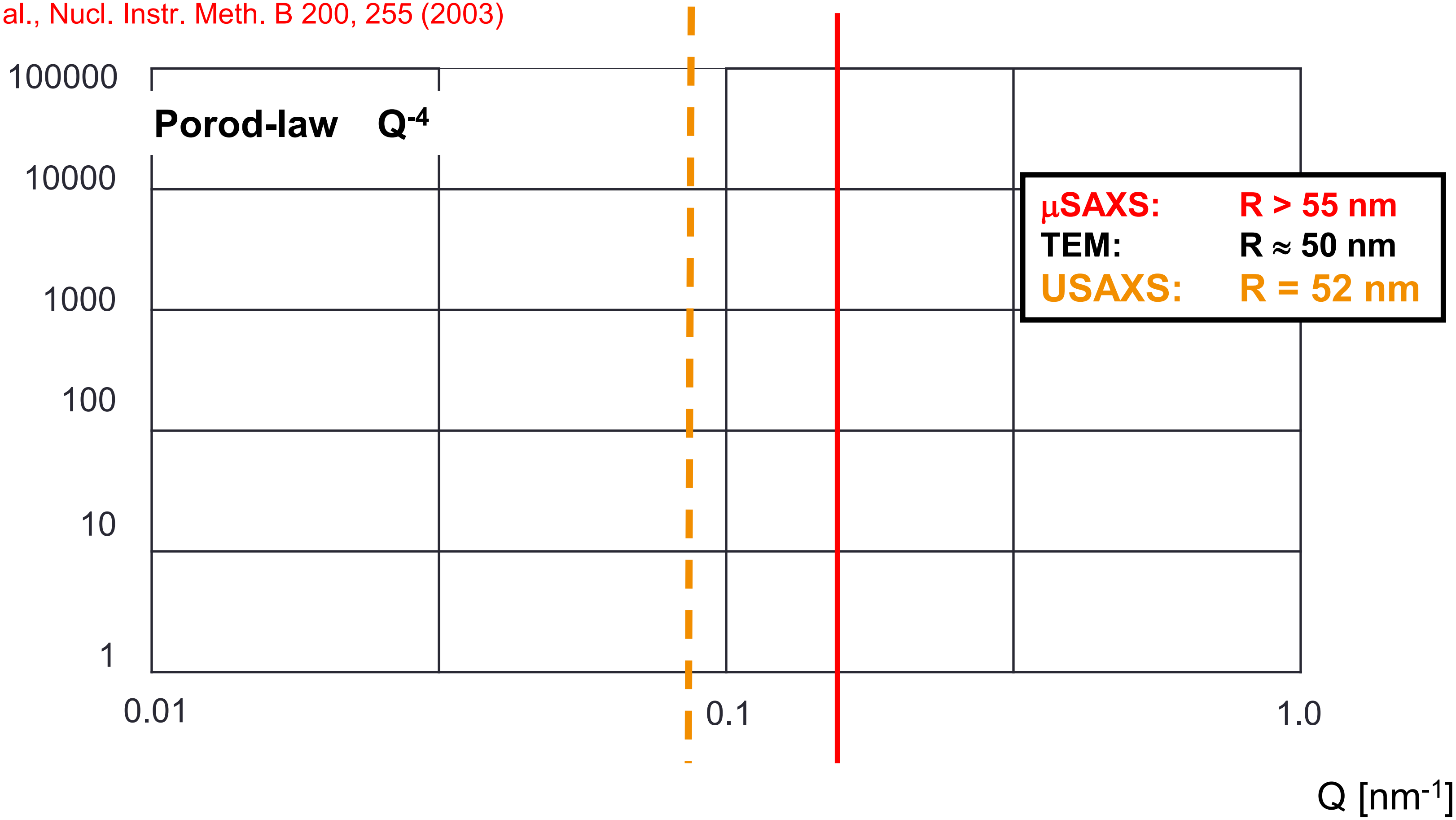
R. Gilles et al.
Scripta Mat. **39**, 715 (1998)

- σ phase precipitate:
  embrittlement of alloy
  crack formation and propagation

- streaking:	correct orientation	
- σ phase:	stack - distance	5 - 15 µm
	diameter 2R <	10 µm
	thickness t >	O(100 nm)

Microfocus: local γ' - particle size distribution

Roth et al., Nucl. Instr. Meth. B 200, 255 (2003)



Lower minimum of particle size distribution

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The drying droplet

- Self-organisation: attractive capillary forces
- correlated nano-structures
- industrial processes
  - spray drying (see also GISAXS part)
  - food processing, pharmaceuticals
  - Paintings/coatings

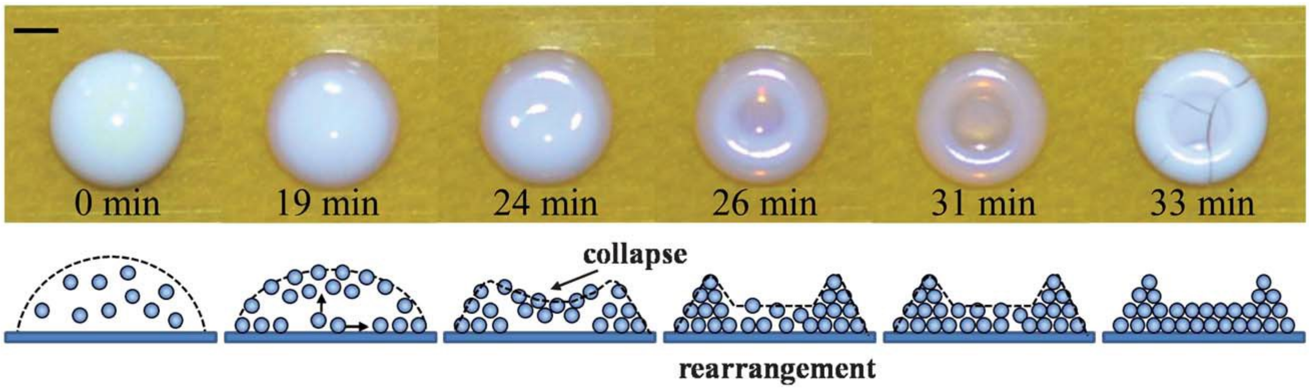
http://www.spray.com/markets_and_applications/food.aspx

Wir machen ein Experiment

- Der trocknende Tropfen
- Wandfarbe!
- Haftung!
- pPS Glassubtrate

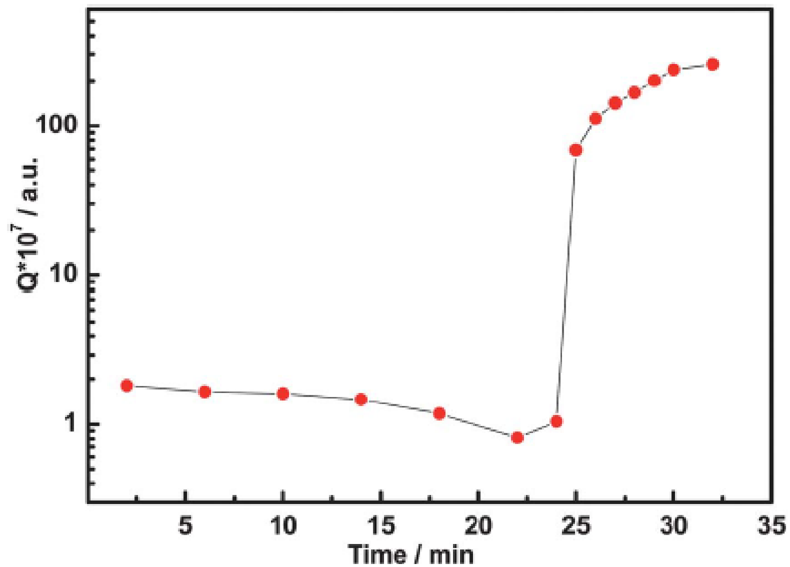
Porod invariant – practical application

> Colloidal solution: drying thick droplet



> Evaporation of water:

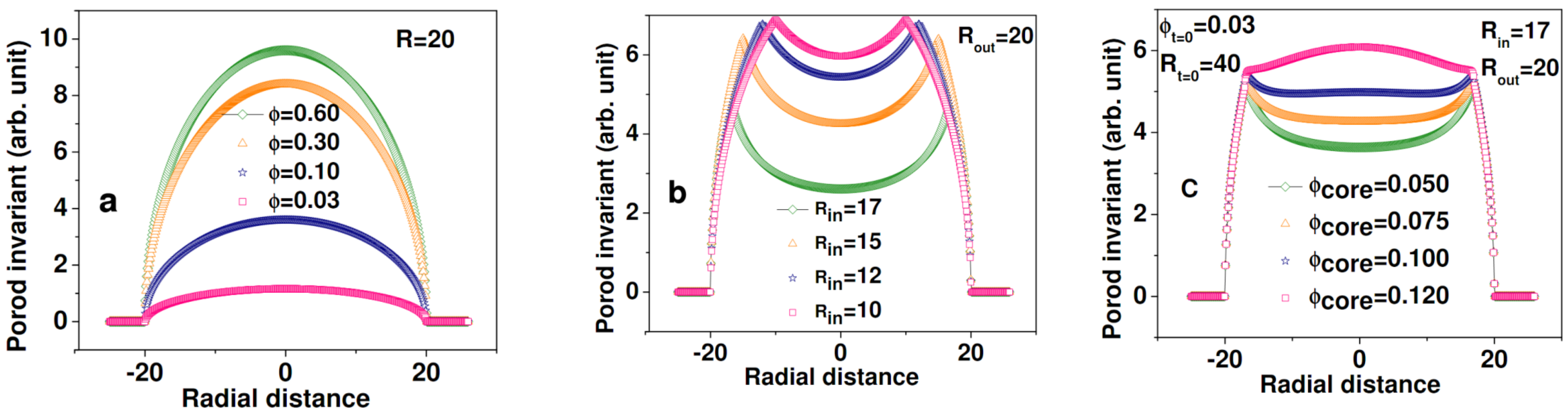
- Irradiated volume becomes smaller: shrinking
- Distance of colloidal partices decreases, $\Phi \rightarrow 1$
- $\Delta\rho$ increases (air!), as water removed from interstitial sites



Chen et al., Soft Matter 8, 12093 (2012)

The drying droplet

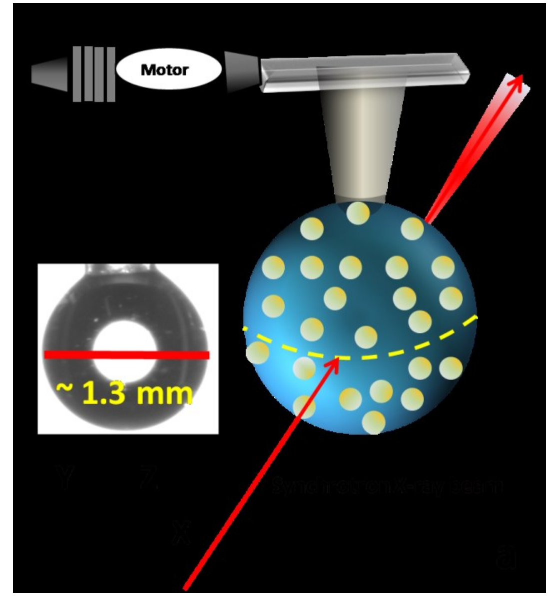
- Microbeam: local concentration
- Q~ $\phi(1-\phi)(R^2 - x^2)^{0.5}$
- Dried droplet: different gradient in ϕ
- Homogenous distribution
- Agglomeration in shell
- Continuous gradient

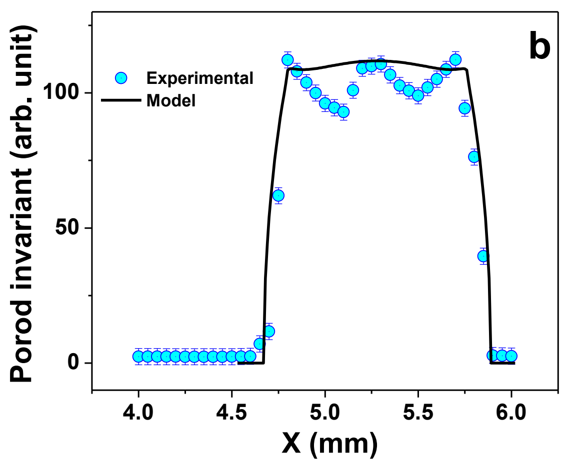


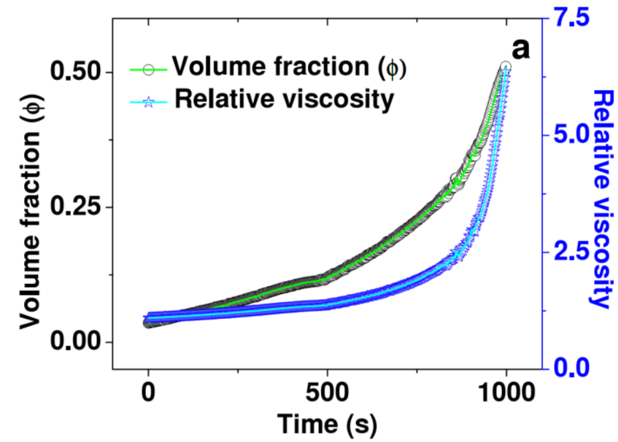
Sen, SVR et al., Soft Matter **10**, 1621 (2014)

The drying droplet – part 2

- Slow / fast drying
- Concentration of colloids:
  - Arresting of colloids
  - Homogenous
  - Core shell effect (‚coffee ring‘)
- Follow concentration profile in-situ







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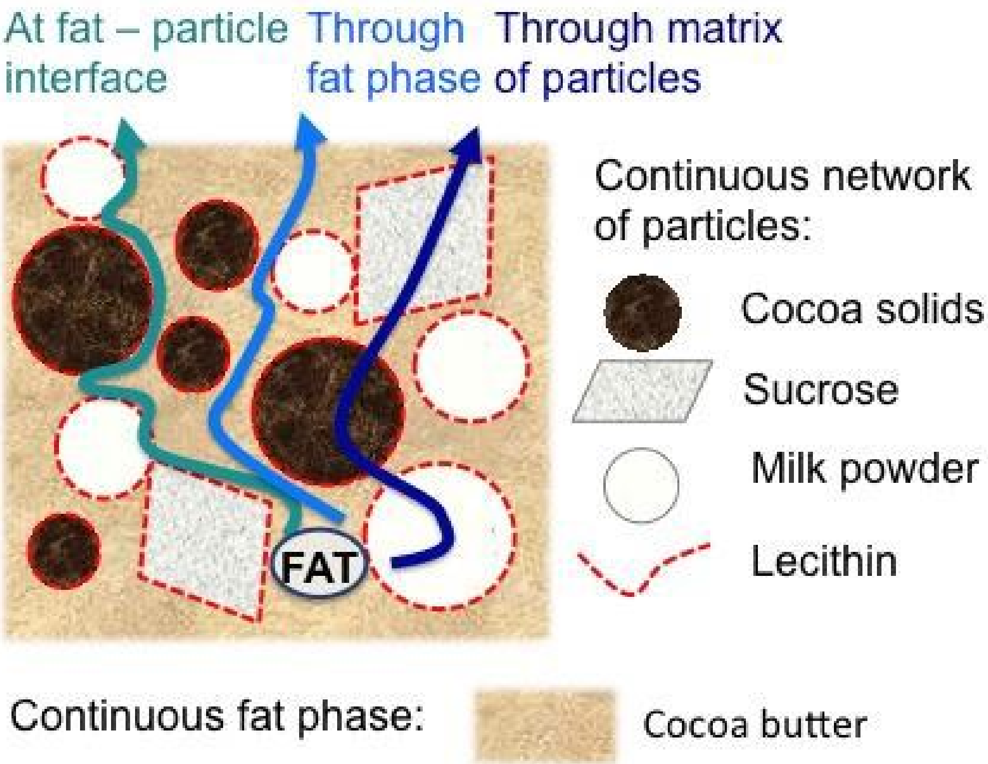
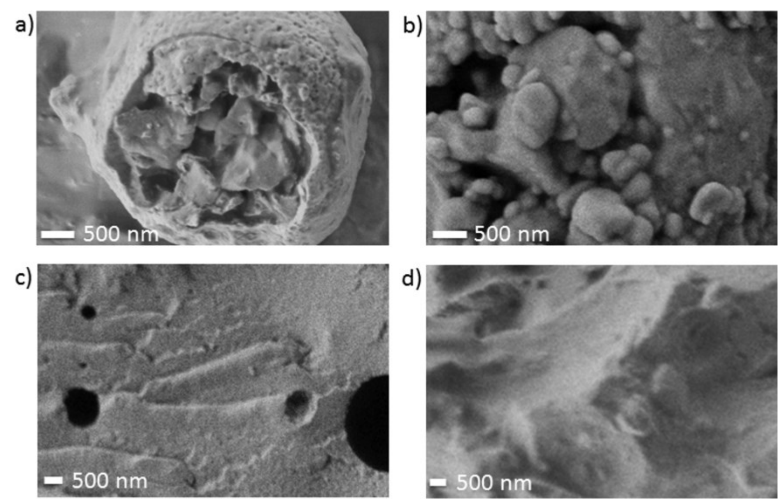
Chocolate

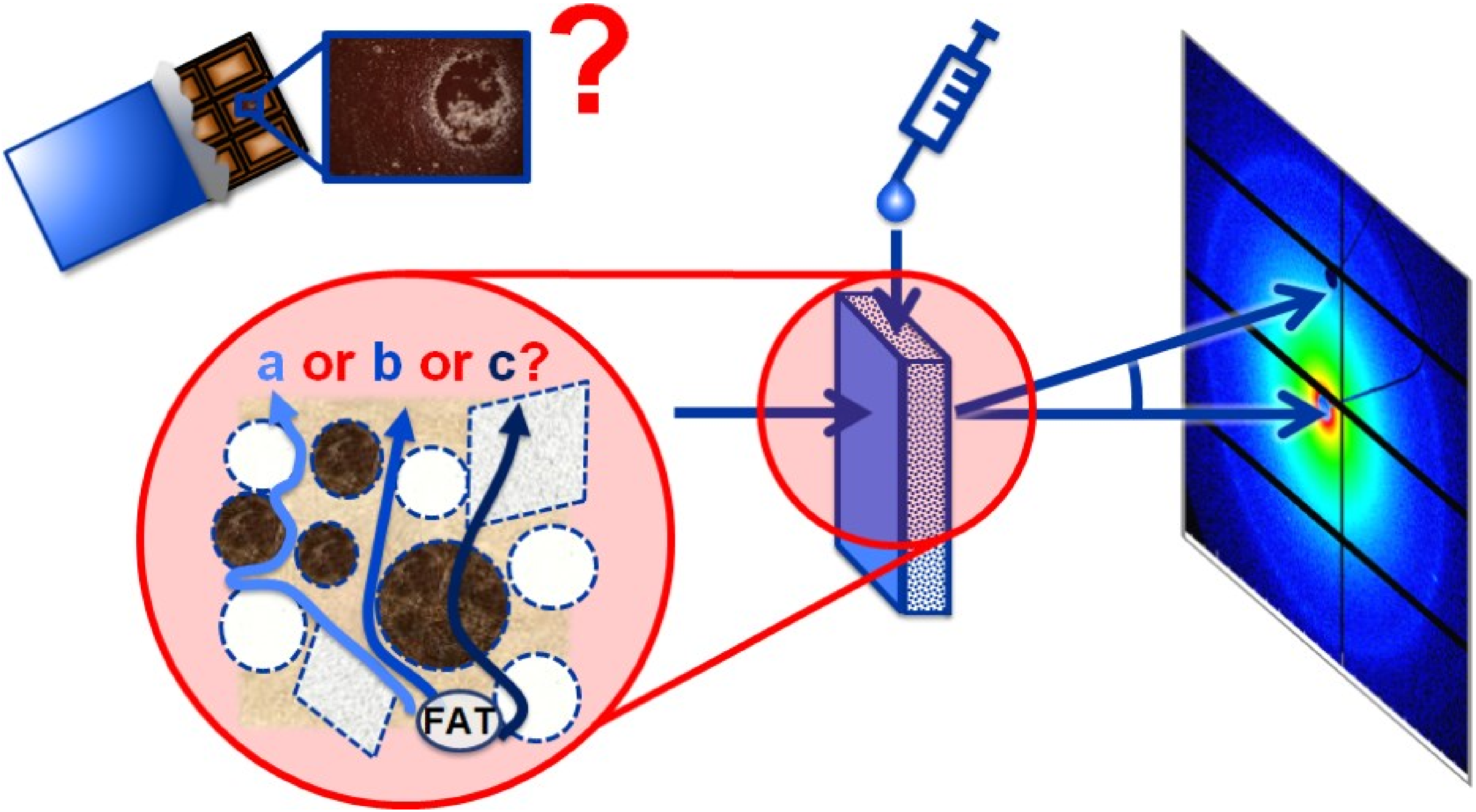
- A real multicomponent system
- Nestlé, TU HH, DESY
- Fat Blooming - pathways





Reinke, SVR et al., ACS Appl. Mater. Interfaces, DOI: 10.1021/acsami.5b02092 (2015)

Chocolate

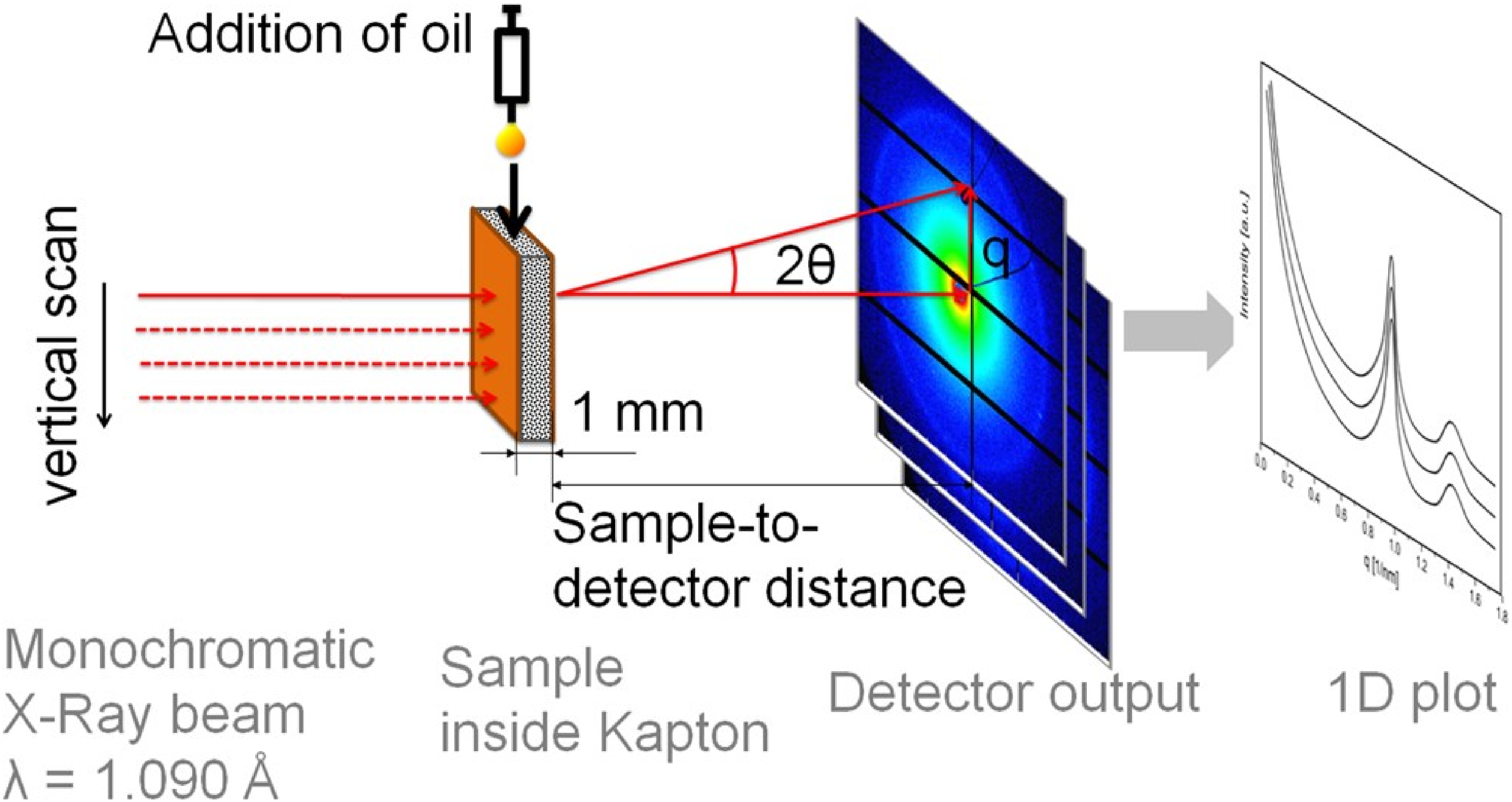


Continuous fat phase:

Reinke, SVR et al., ACS Appl. Mater. Interfaces, DOI: 10.1021/acsami.5b02092 (2015)

Chocolate

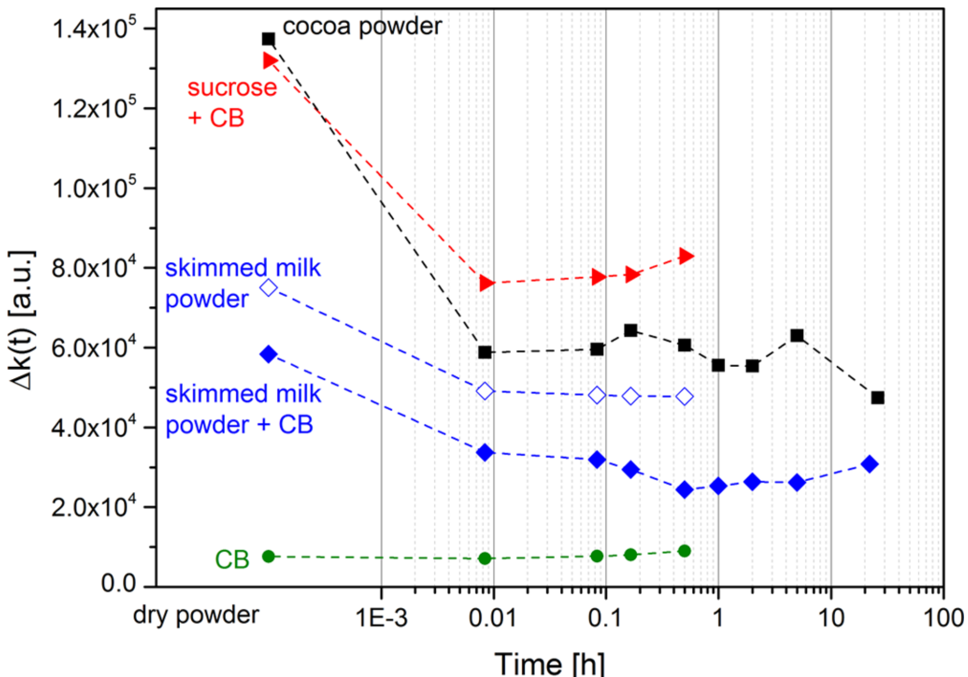
> A real multicomponent systems

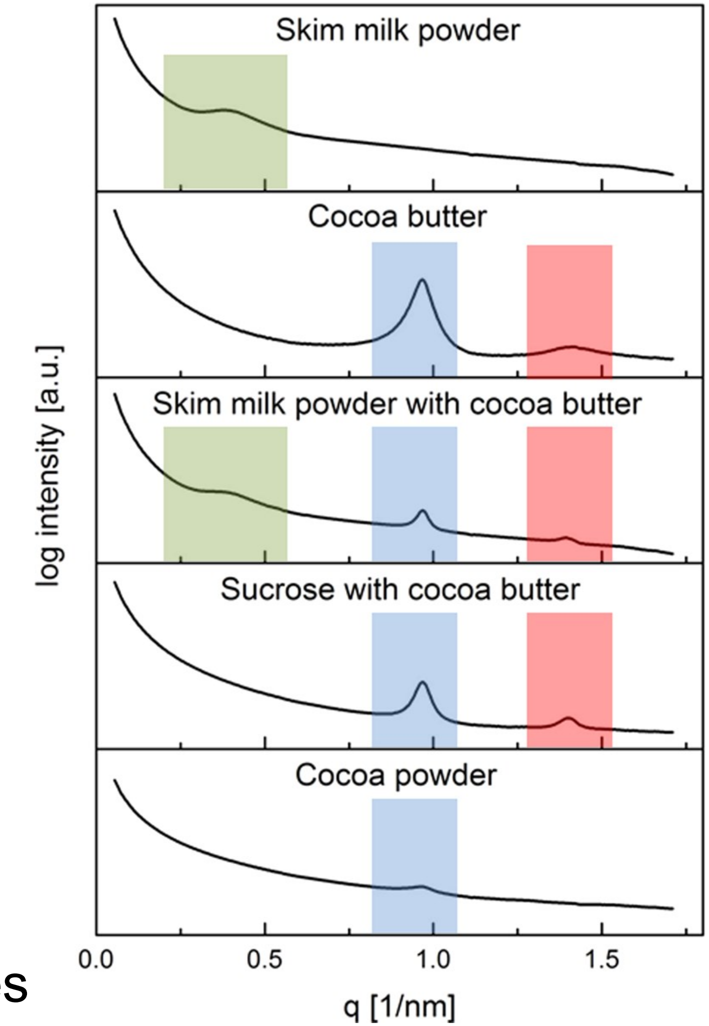


Reinke, SVR et al., ACS Appl. Mater. Interfaces, DOI: 10.1021/acsami.5b02092 (2015)

Chocolate

- A real multicomponent system
- Superposition of SAXS contributions



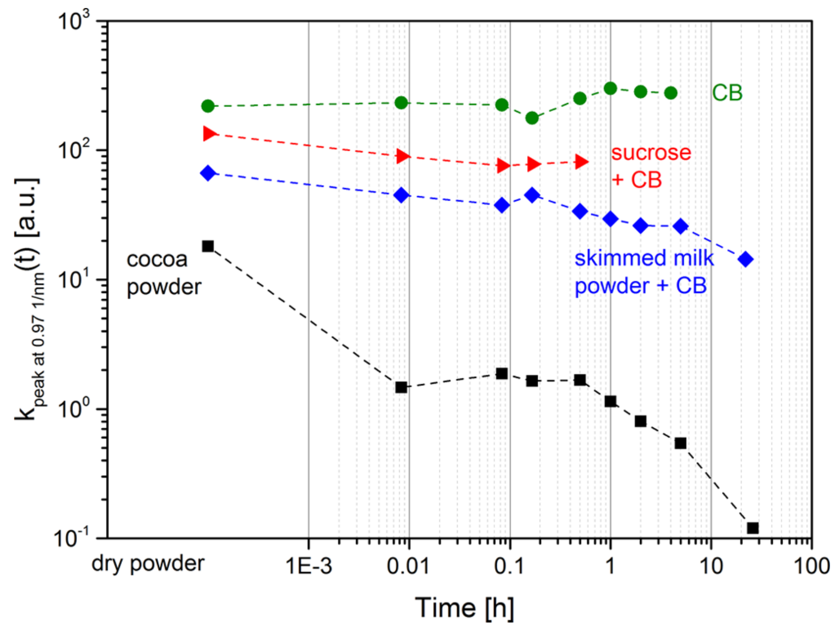


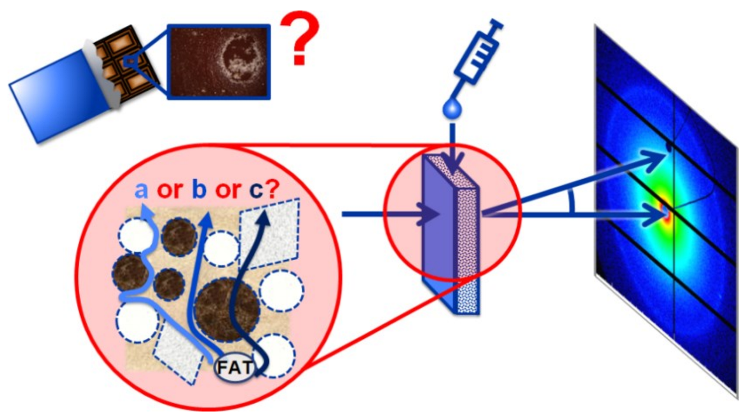
- Different density difference
- Migration: filling of voids by oil: Q decreases

Reinke, SVR et al., ACS Appl. Mater. Interfaces, DOI: 10.1021/acsami.5b02092 (2015)

Chocolate

- Peak intensities
- Pores, cracks: capillary effect
- Then: “chemical migration through the fat phase by softening and partial dissolution of the crystalline cocoa butter.“
- reduction of porosity and a minimization of defects
- a reduced content of noncrystallized liquid cocoa butter
- b or c





Reinke, SVR et al., ACS Appl. Mater. Interfaces, DOI: 10.1021/acsami.5b02092 (2015)