Summer student session 18.09.2006

- **An absolute intensity calibration at BW4 by using a reference sample**

- **GISAXS Experiment – Au-nanoclusters on a Si-111 surface**

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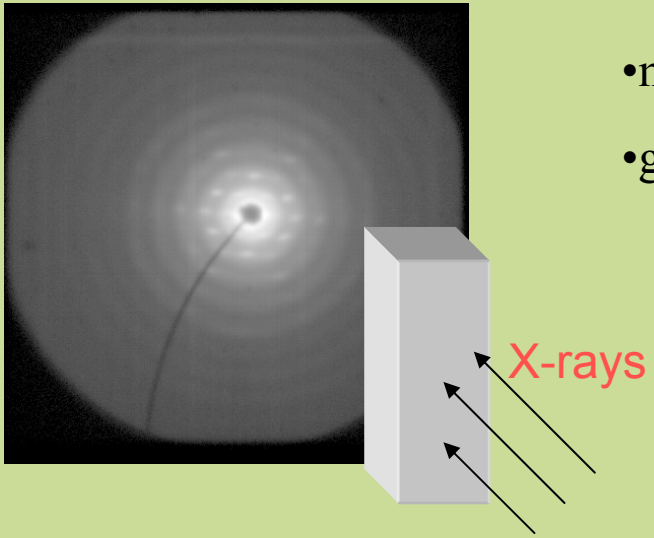
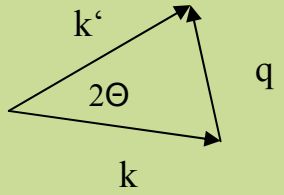
What is SAXS ?

(Small Angle X-ray Scattering)

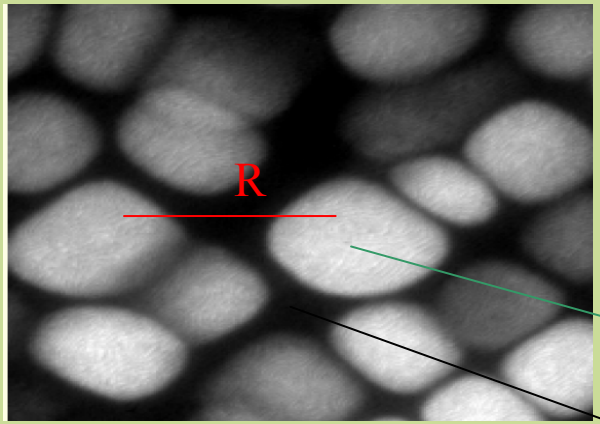


- X-rays are scattered at electrons in atom shell
- momentum transfer q, scattering angle 2Θ
- geometry:



k: incident wavevector
k‘: scattered wavevector

➢large q ($2\Theta > 1°$) ⟶
- investigation of atomic distances (Angstroem)
- crystal: Bragg‘s equation: $2d \sin\Theta = n\lambda$



TEM of a Superalloy (Ni-Al solid solution matrix and precipitates (Al,..)

➢small q ($2\Theta < 1°$) ⟶
- distances R about 10nm - 1µm
- need a contrast in density
- intensity = Shape x form factor

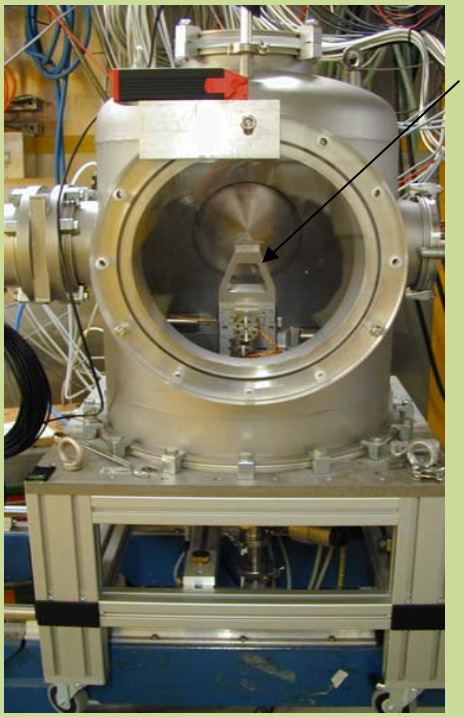
precipitate

amorphous matrix

interference due to assembly of particles

BW4: A dedicated beamline for:

- Small Angle X-Ray Scattering (SAXS), also USAXS (d_{max}>1μm) (L_{SD}=12m)
- Grazing Incidence Small Angle X-Ray scattering (GISAXS)
- focussing with Be-lenses: minium beam size of **30 x17 μm²**
  →μGISAXS (small step-scanning along the sample)



evacuated sample environment

Sample and detector

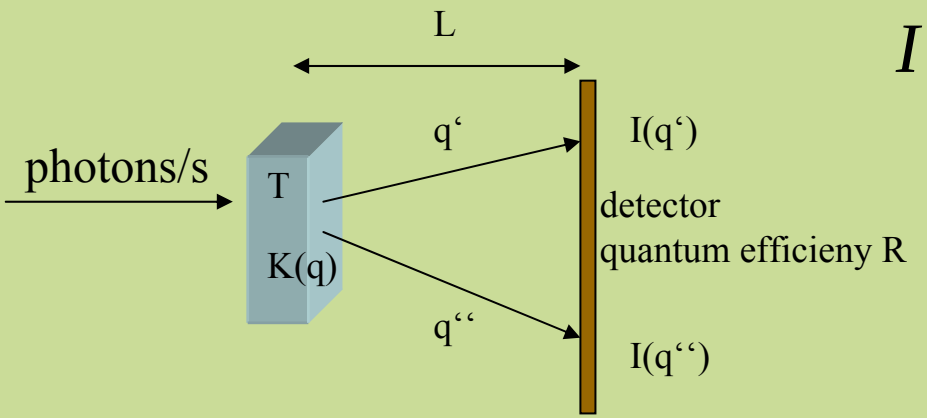
evacuated beam pipe (around 10^{-6} mbar)

•sample to detector distances between 2m and 12m

Absolute intensity - what, why and how?

- ratio between scattered intensity I(q) [photons /(s mm^2)] to power P [photons/s] of incident beam
- by knowing the absolute intensity the electron density and relative atomic mass can be obtained

- the intensity at a certain scattering vector q‘ can be obtained by:



$$I(q') = P \cdot \frac{T}{L^2} \cdot R \cdot K(q')$$

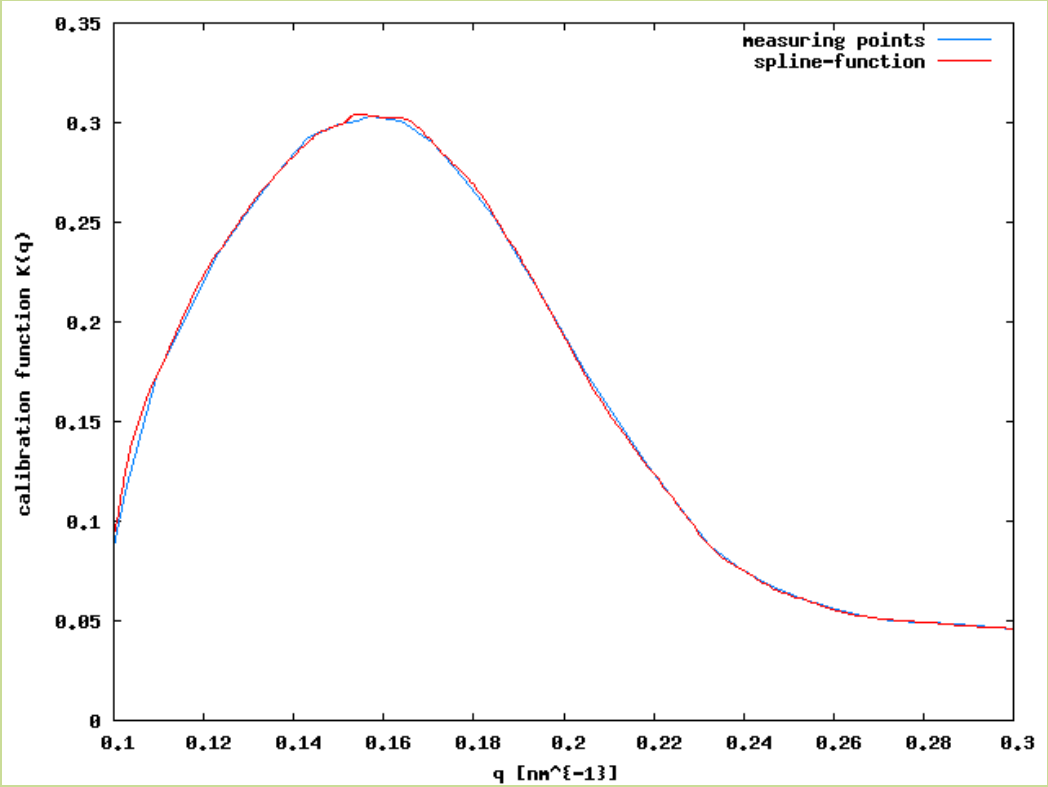
L: sample to detector distance
T: transmission
R: detector efficiency
K(q): calibration function,
contains the cross section

Calibration function

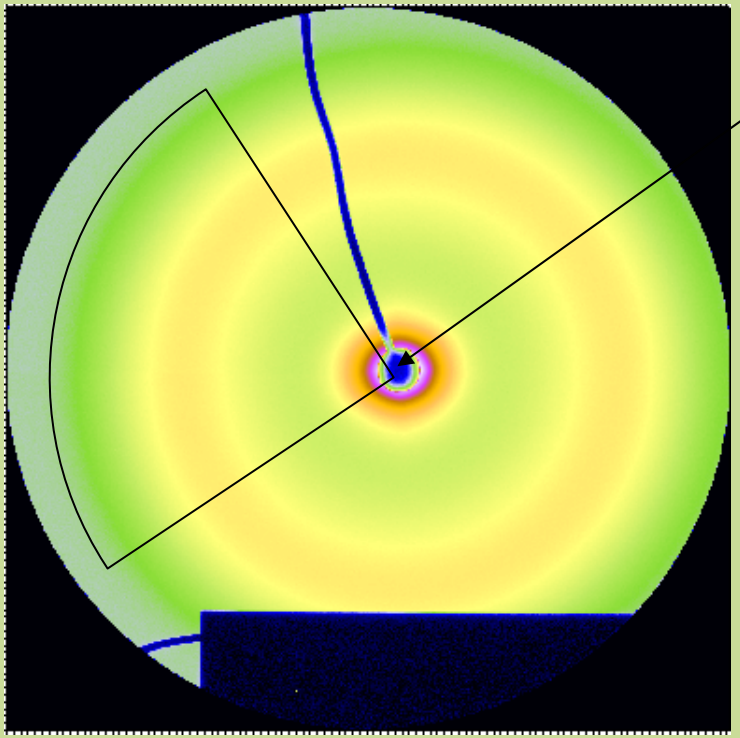
- calibration function K(q) for a Lupolen sample was measured 10 years ago at BW4 using a Kratky camera
- beamline was upgraded, so a new calibration is needed

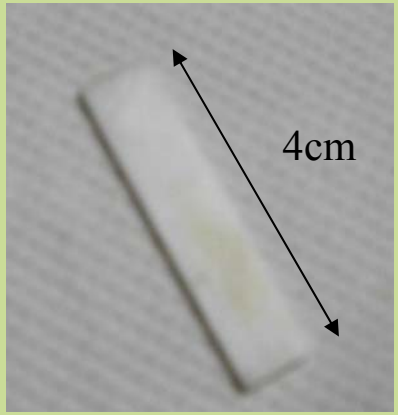
$$P = \frac{I(q')}{K(q')} \cdot \frac{L^2}{R \cdot T}$$

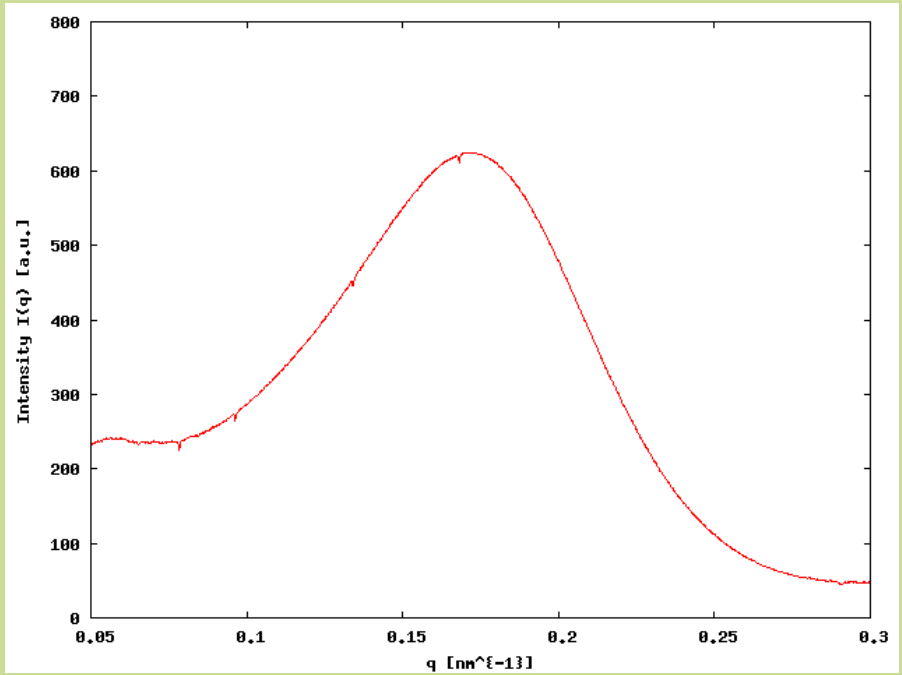
- obtaining P by measuring the scattering pattern of the Lupolen



Measurement of Lupolen







$$P = \frac{I(q')}{K(q')} \cdot \frac{L^2}{R \cdot T}$$

further parameters:
L=12855mm
R=0.8

•transmission was obtained by an ionization chamber and beamstop diode

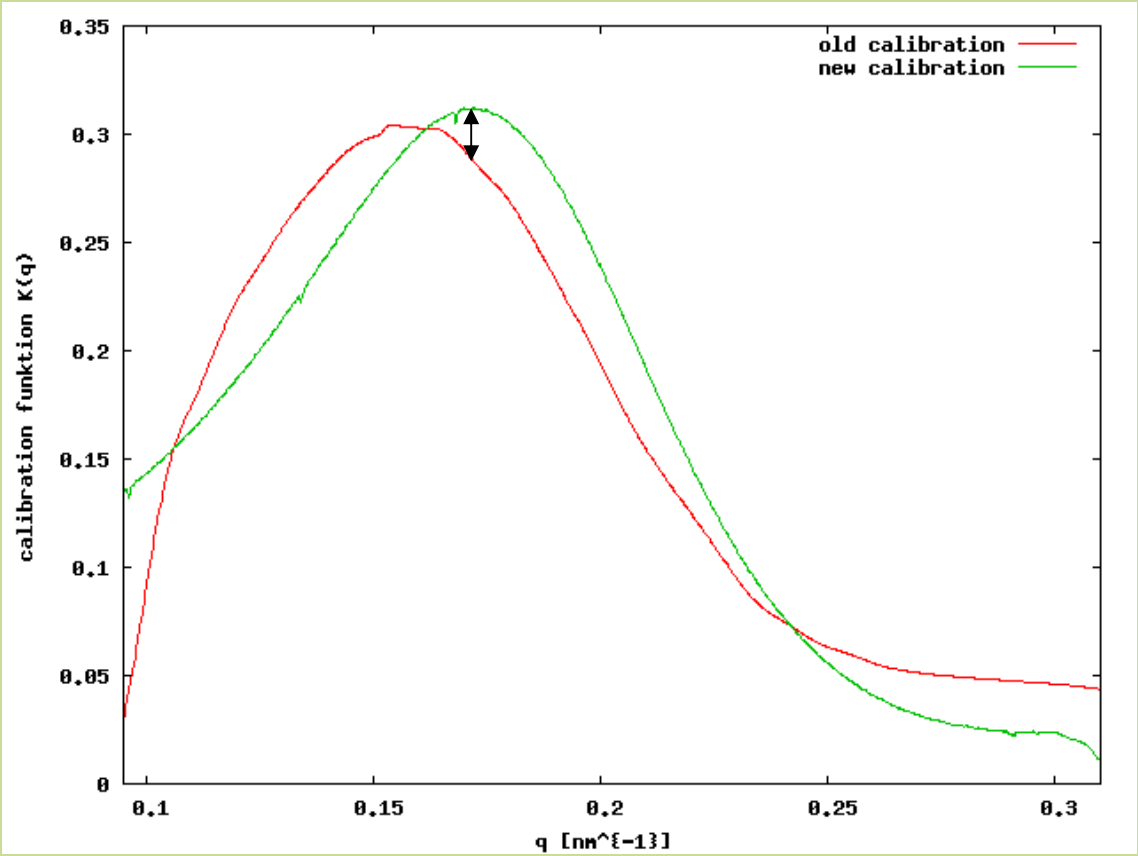
Results

$P = 6.67 \cdot 10^{11}$ photons/s at 100mA Doris current

(after systmatic error correction, systematic error is around 7%)

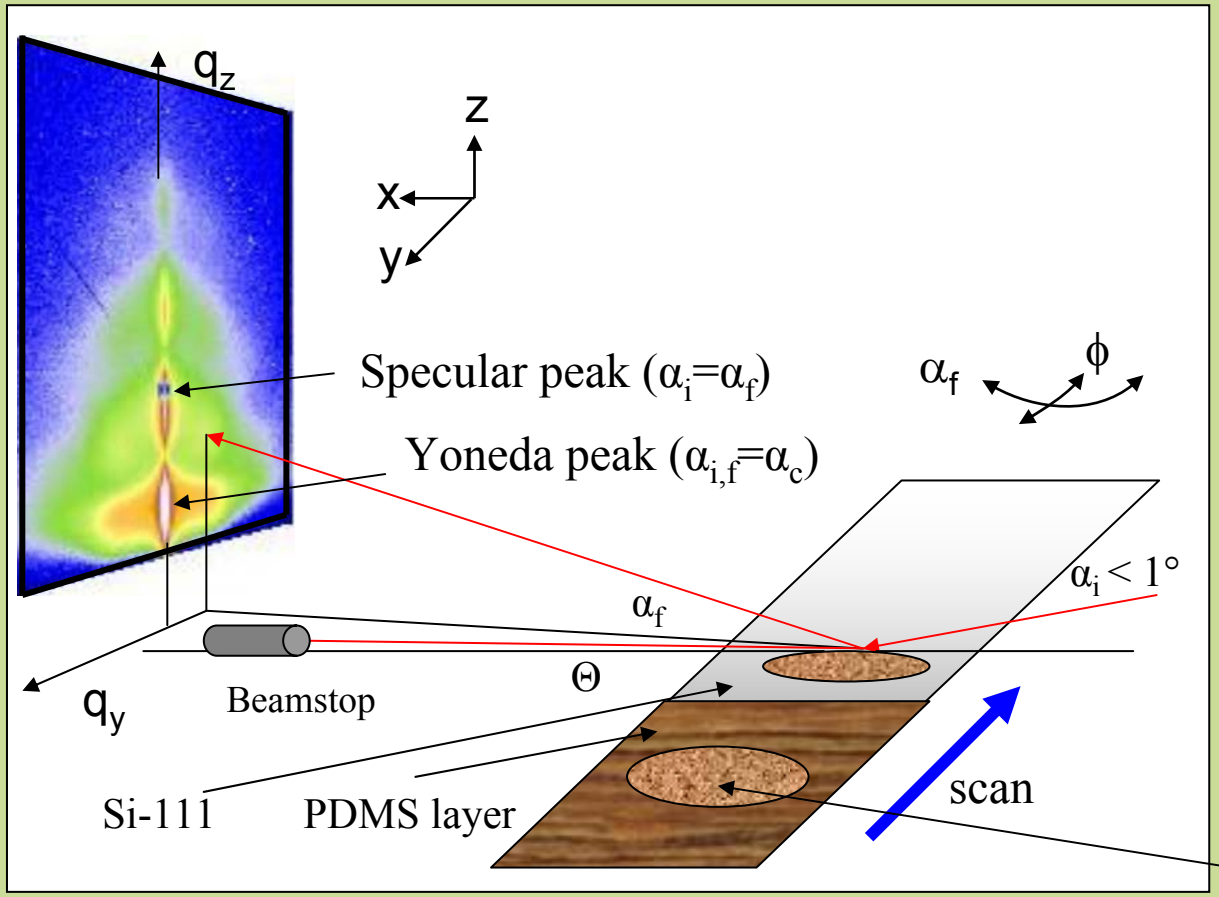
- structure of Lupolen has changed

- but whole scattered intensity is conserved

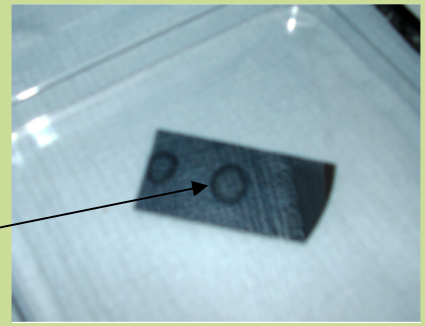


Grazing Incidence Small Angle X-ray Scattering

- surface sensitive method: penetration depth depends on the incident angle
- Yoneda peak: interference of incident and reflected beam at the critical angle
- Specular peak: Regular reflected beam

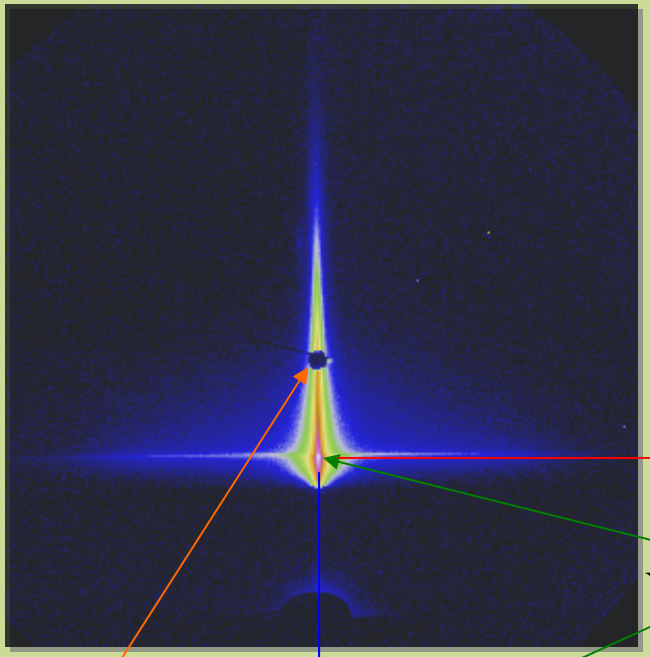


- **Diffuse scattering:**
  - q_z-direction: mainly correlation perpendicular to surface (height of clusters, roughness, layer thickness)
  - q_y-direction: In-plane structures (distances, radius)

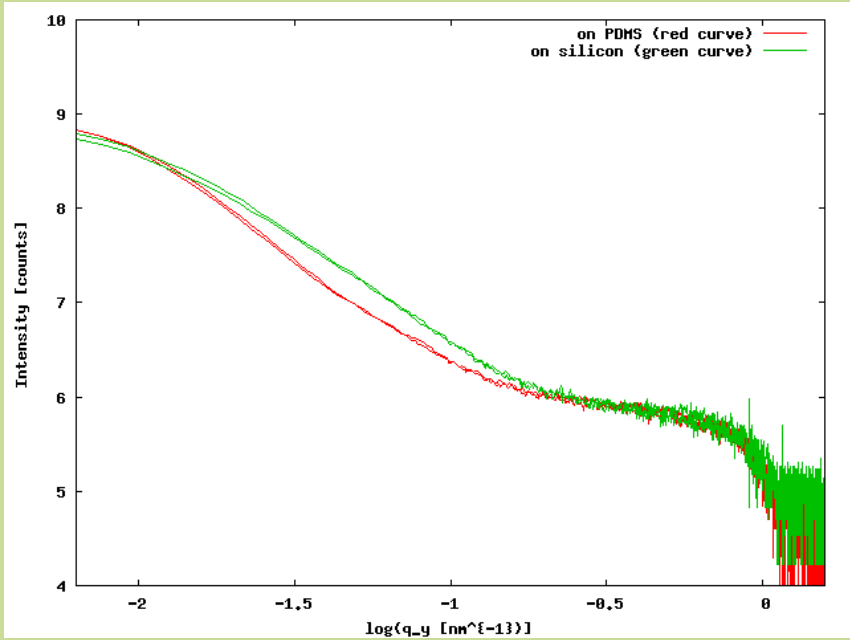


nanocluster in aqueous solution

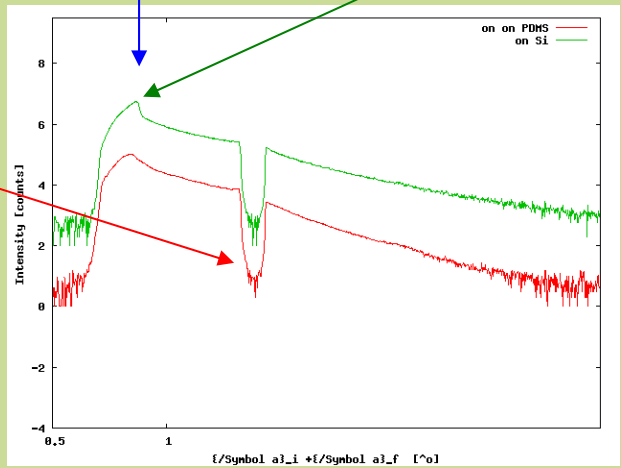
µGISAXS



Yoneda peak



Specular peak



evaluation so far:

- small length scales → no difference Si/PDMS
- larger length scales→ agglomeration of clusters on Si, distance about 120nm

➢no agglomeration on PDMS

Thanks for your attention!!