2nd SEMAT/UM Workshop on Advanced Characterization of Materials

Wide- and Small Angle X-ray Measurements with Bruker AXS NanoStar Testing Station

Zlatan Denchev, DEP- UM

Topics:

1. Basic concepts of the X-ray analysis.

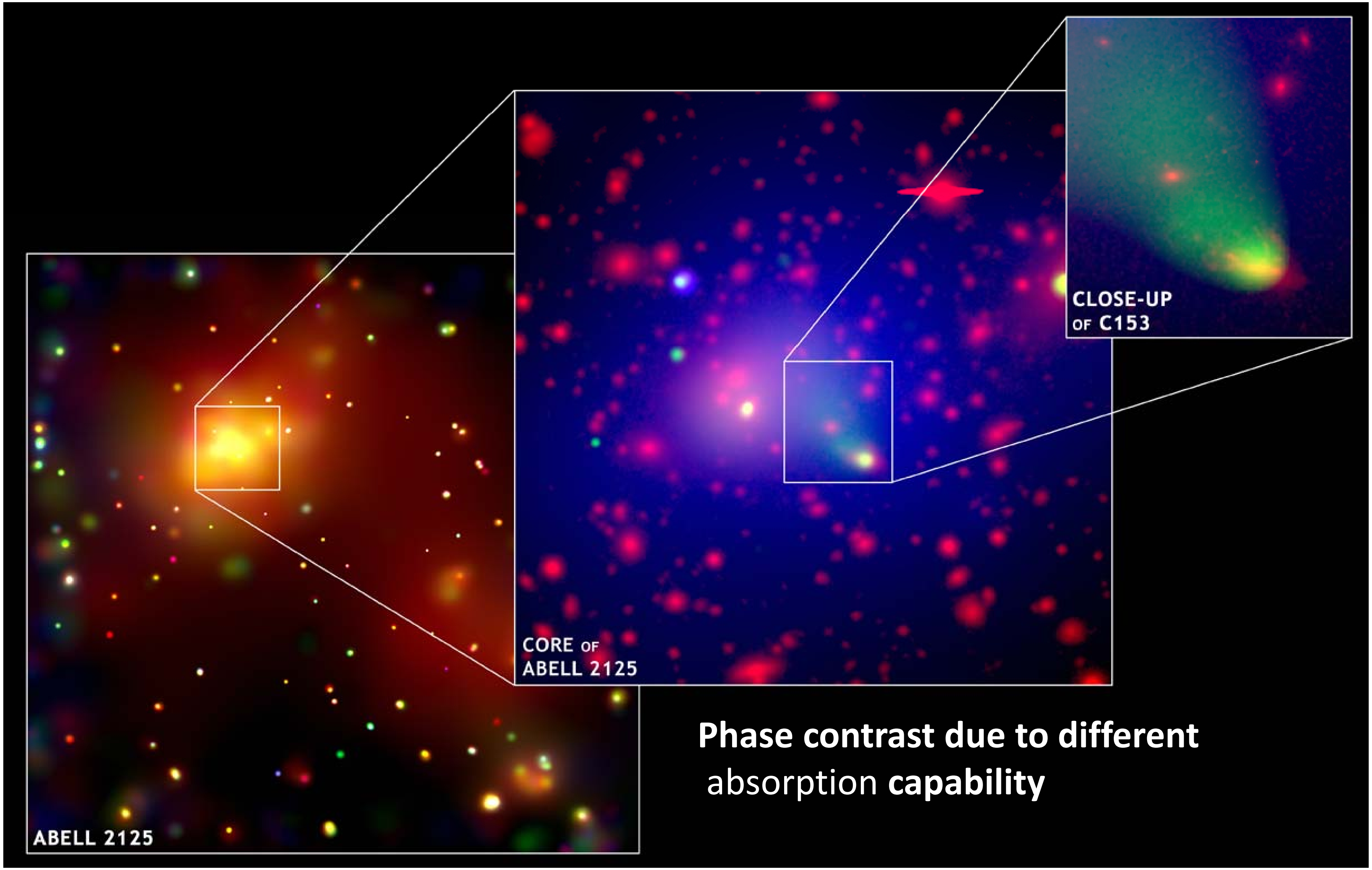
2. Bruker AXS NanoStar with HiStar 2D detector – construction, advantages, analytical possibilities.

3. The use of 2D WAXS and SAXS in various areas of materials science- examples.

4. The use of Bruker NanoStar in SEMAT – organization issues.

5. Discussion

Absorption of X-rays: visualizing the big invisible

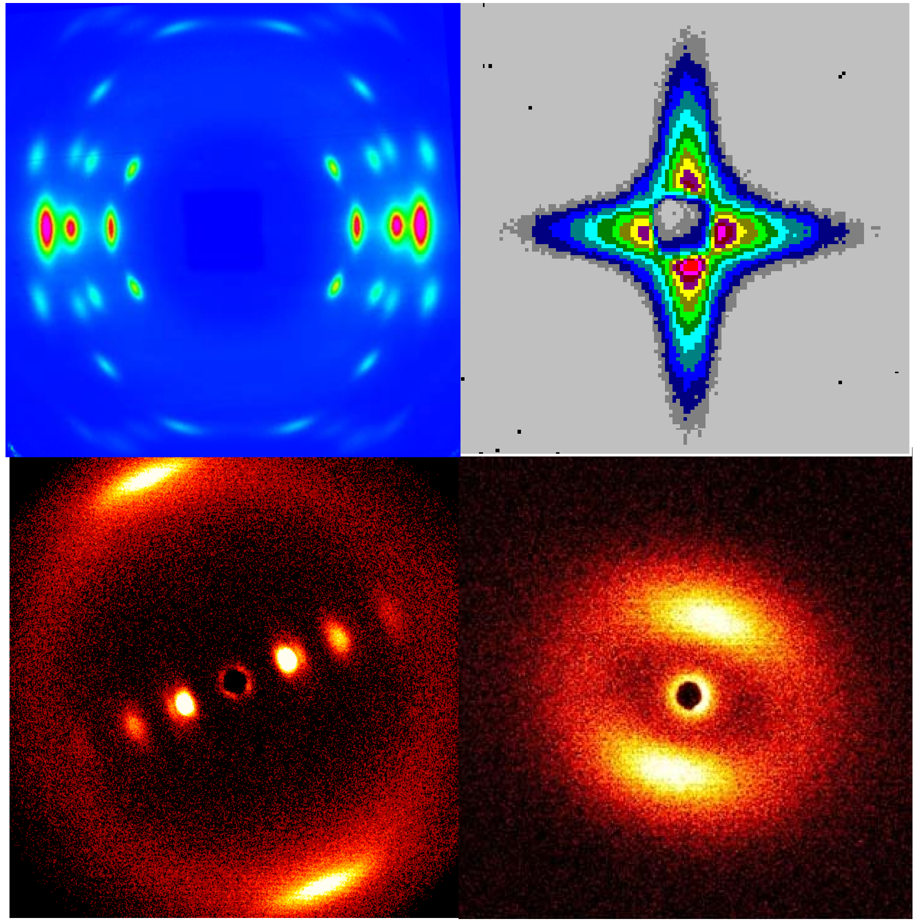


Phase contrast due to different absorption **capability**

Scattering of X-rays: visualizing the sub-micrometric structure

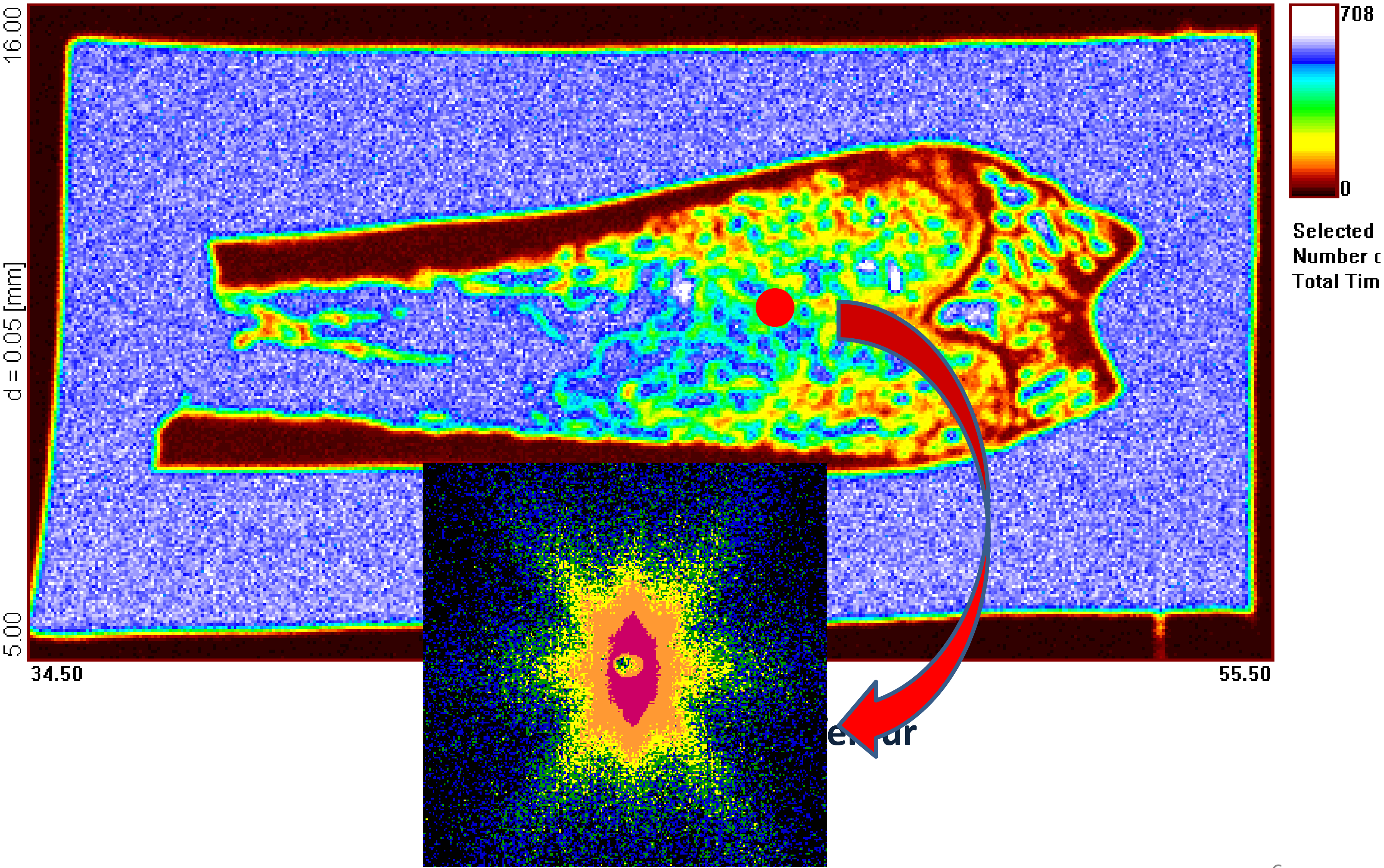
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS

- Polymers
- Biological materials
- Metals, alloys
- Nanopowders
- Solutions with sufficient scattering volume



Patterns caused by the nano-structure but not depicting the sample morphology directly !

Combination of real X-ray images and patterns



What are X-rays?

1. High-frequency (10^{16} – 10^{18} Hz) electromagnetic radiation caused by a change in the momentum of a charged particle (e.g., electron) by:

- Bombarding a metal target by high-speed electrons (vacuum tube);
- Accelerating electrons in vacuum and suddenly changing their path (synchrotron);
- Decelerating electrons by special coatings (old TV screens);
- Nuclear explosions and cosmic events.

2. In agreement with the wave-photon dualism, X-rays can be considered either as packages of waves or as flux of photons.

3. X-ray have extremely high penetration ability. Most materials (with the exception of the heaviest metals) are transparent for them.

What are X-rays?

1. High-frequency ($10^{16} – 10^{18}$ Hz) electromagnetic radiation caused by a change in the momentum of a charged particle (e.g., electron) by:

- Bombarding a metal target by high-speed electrons (vacuum tube);
- Accelerating electrons in vacuum and suddenly changing their path (synchrotron);
- Decelerating electrons by special coatings (old TV screens);
- Nuclear explosions and cosmic events.

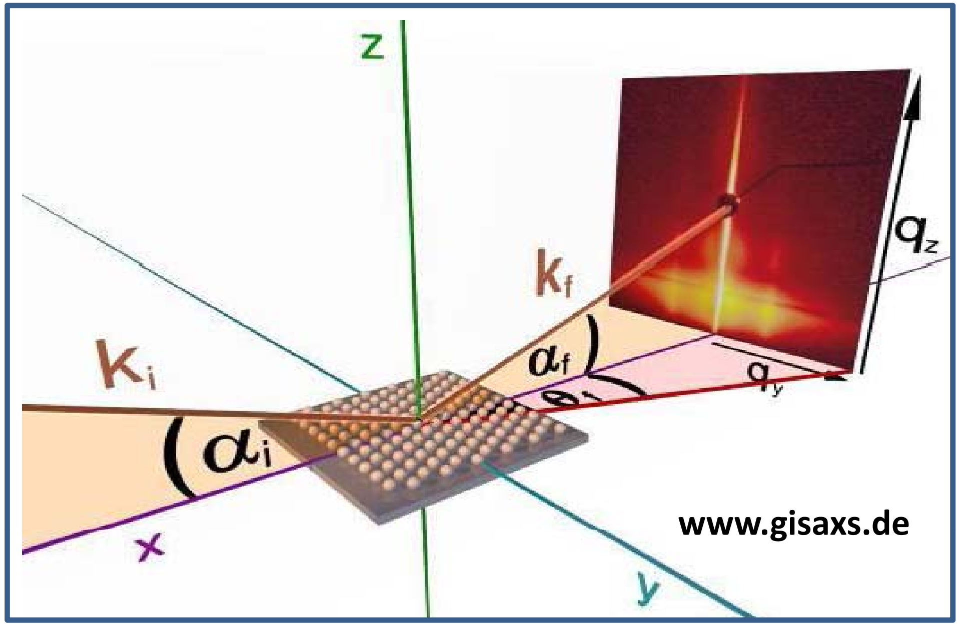
2. In agreement with the wave-photon dualism, X-rays can be considered either as packages of waves or as flux of photons.

3. **X-ray have extremely high penetration ability. Most materials (with the exception of the heaviest metals) are transparent for them.**

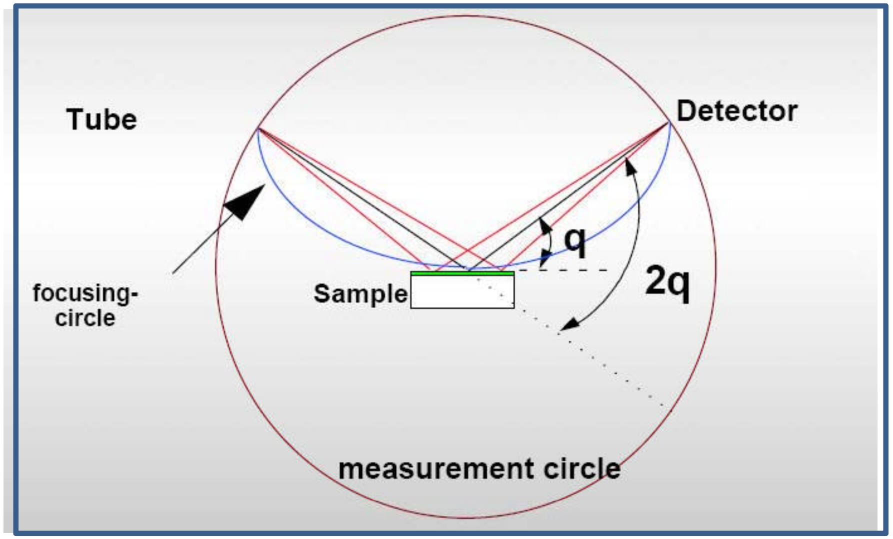
Setups for X-ray studies:

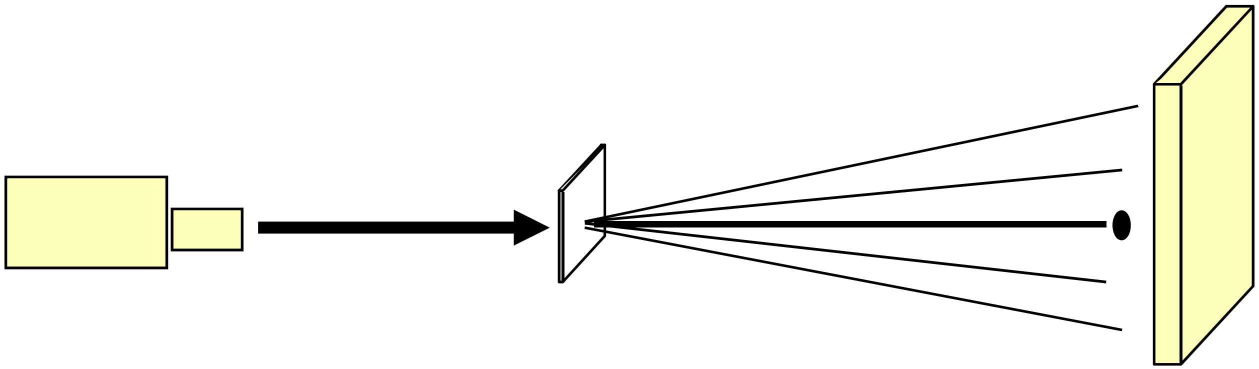
SEMAT
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS

Grazing incidence



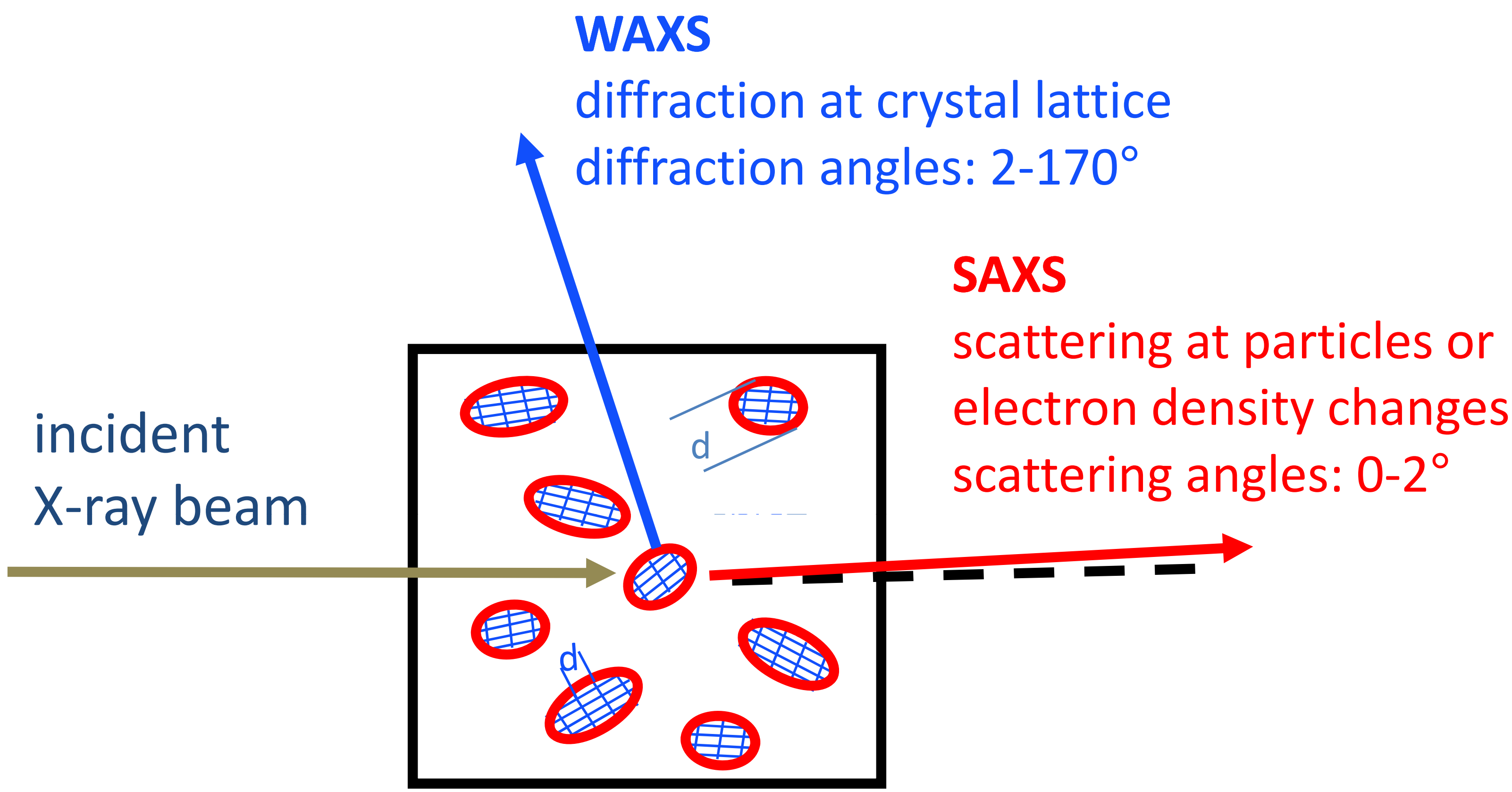
Bragg - Brentano



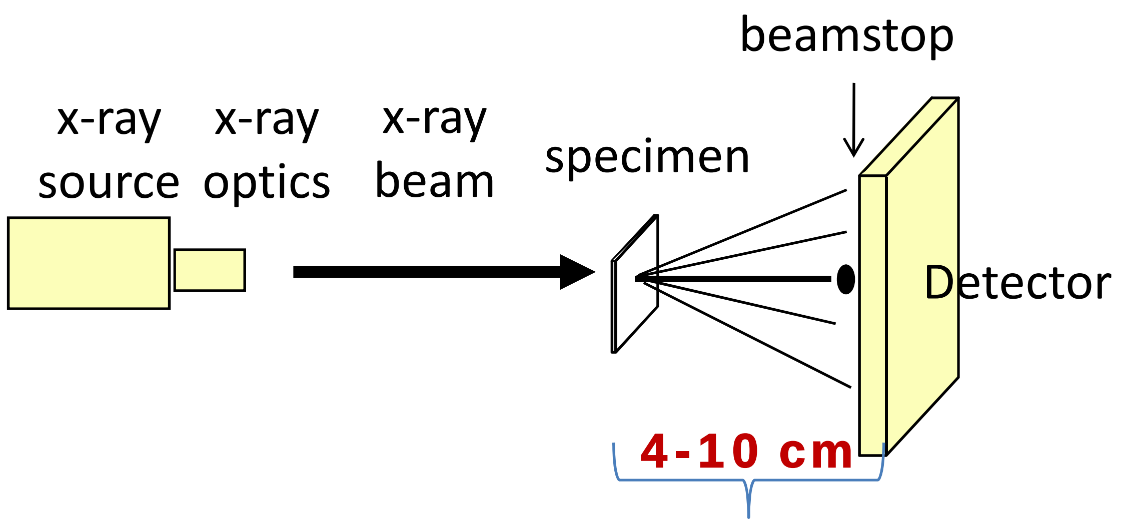


Transmission mode

X-ray experiments: WAXS and SAXS

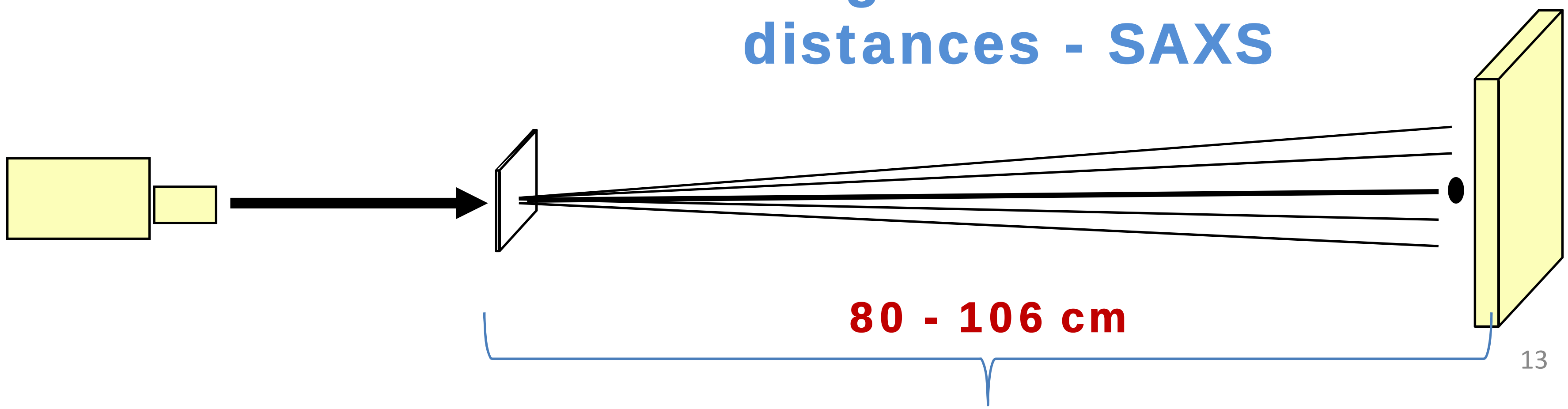


Setups for X-ray studies: WAXS and SAXS



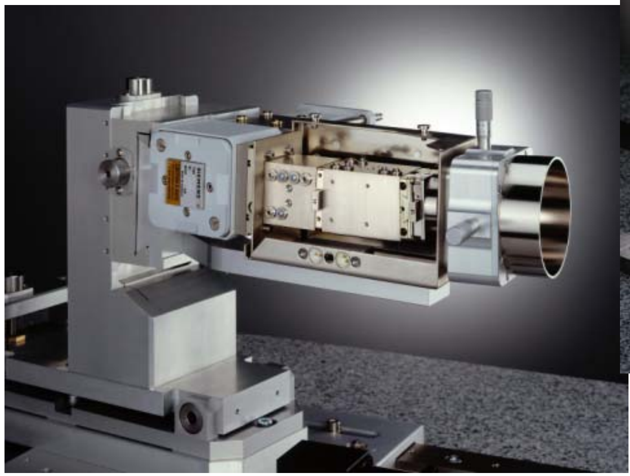
Short SD distances - WAXS

Large SD distances - SAXS



Bruker AXS NanoStar – Key Components

X-ray source with
X-coupled Göbel Mirrors



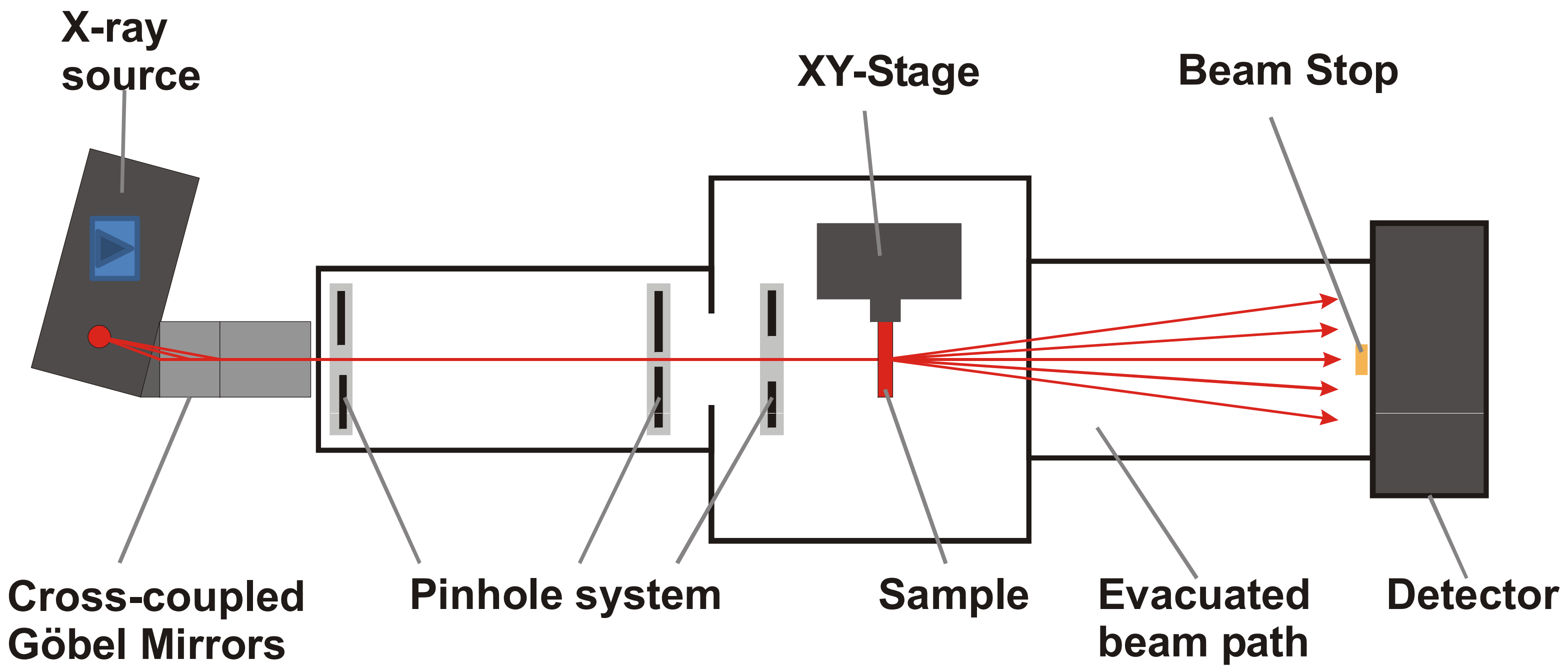
Versatile Pinhole
optics

Sample chamber

2-D detector
And beam stop

Bruker AXS NanoStar – Key Components

Bruker AXS NanoStar – Scheme



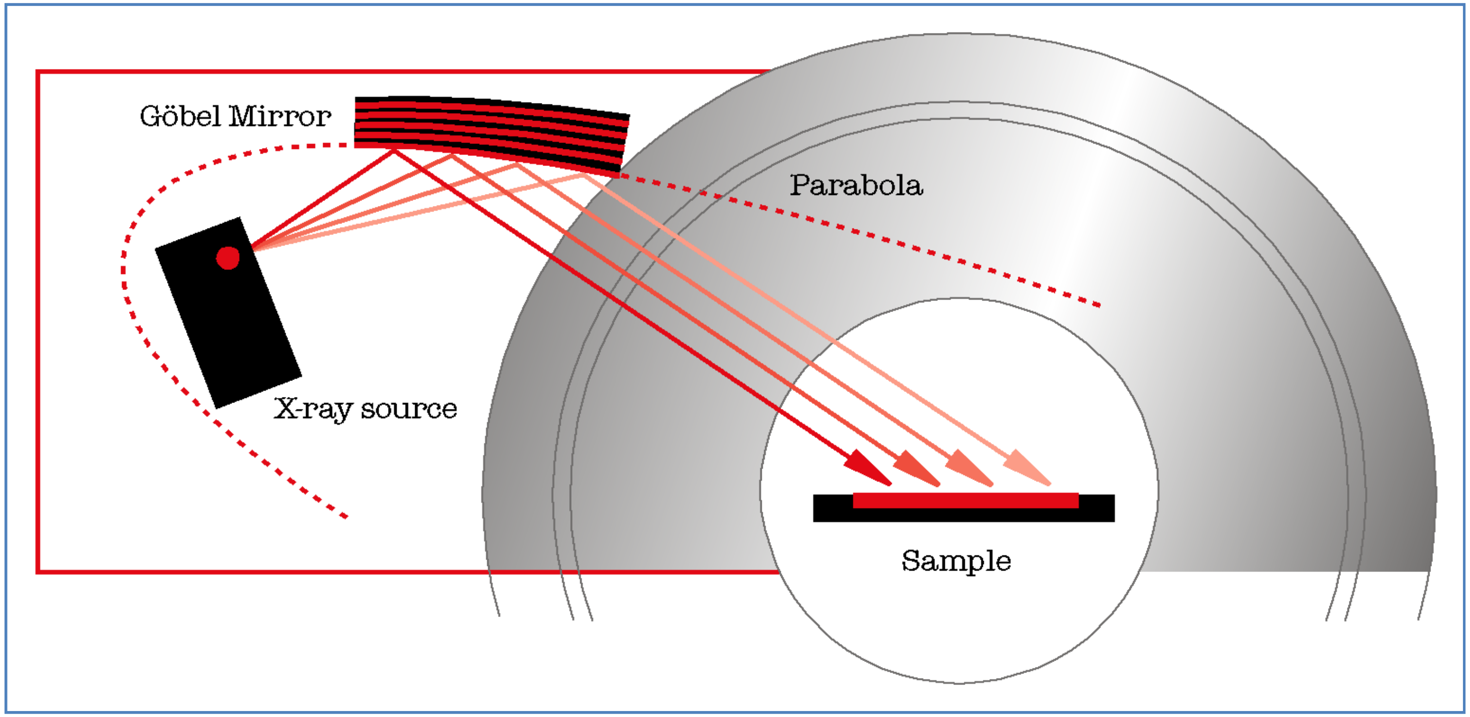
Distance 1st – 2nd pinhole: 92.5 cm

Distance 2nd – 3rd pinhole: 42.8 cm

(3-pinhole geometry based on an idea by Prof. Jan Skov Pedersen, Univ. Aarhus, DK)

Bruker AXS NanoStar – Goebel Mirror

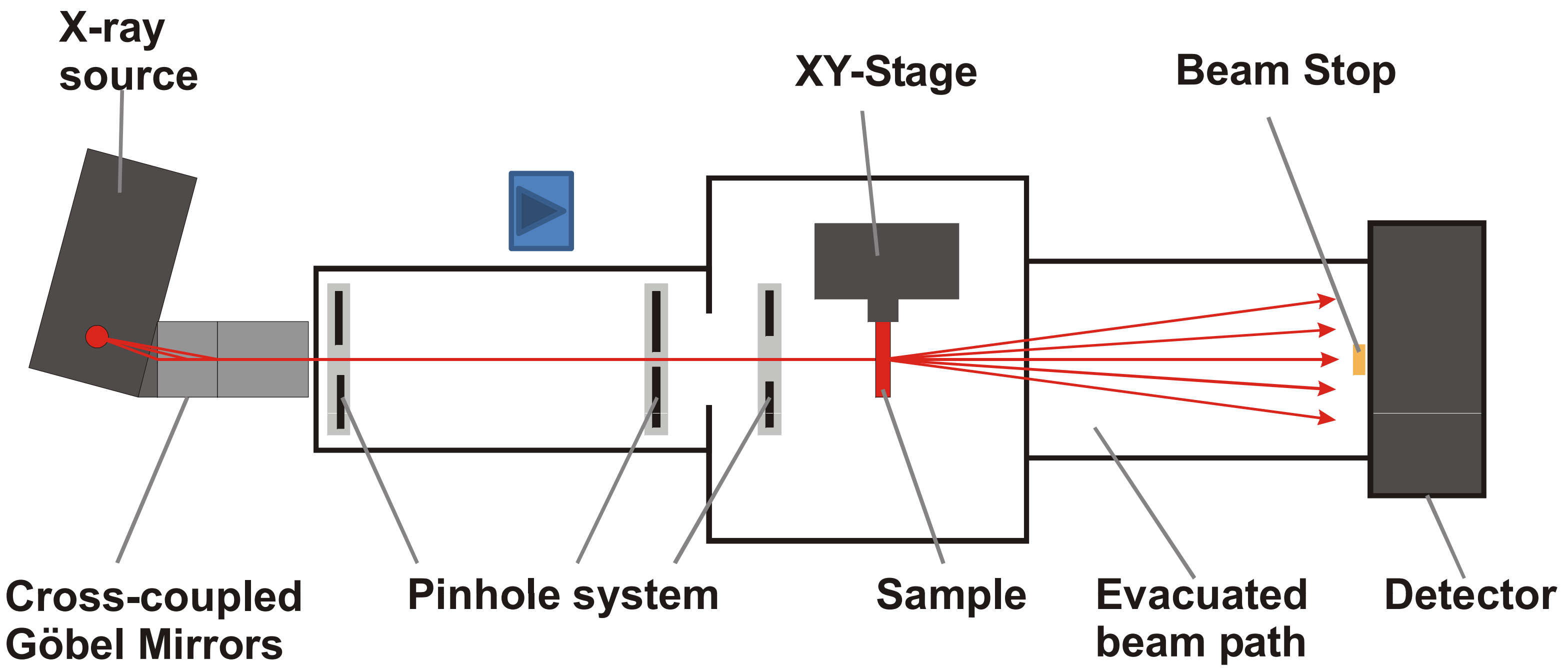
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS



Goebel Mirror: producing low divergence, parallel X-ray beams

- graded multilayer (e.g. W/Si or Ni/C)
- bent or pre-figured parabolically for parallel beam
- bent elliptically for focussed beam

Bruker AXS NanoStar – Slit System



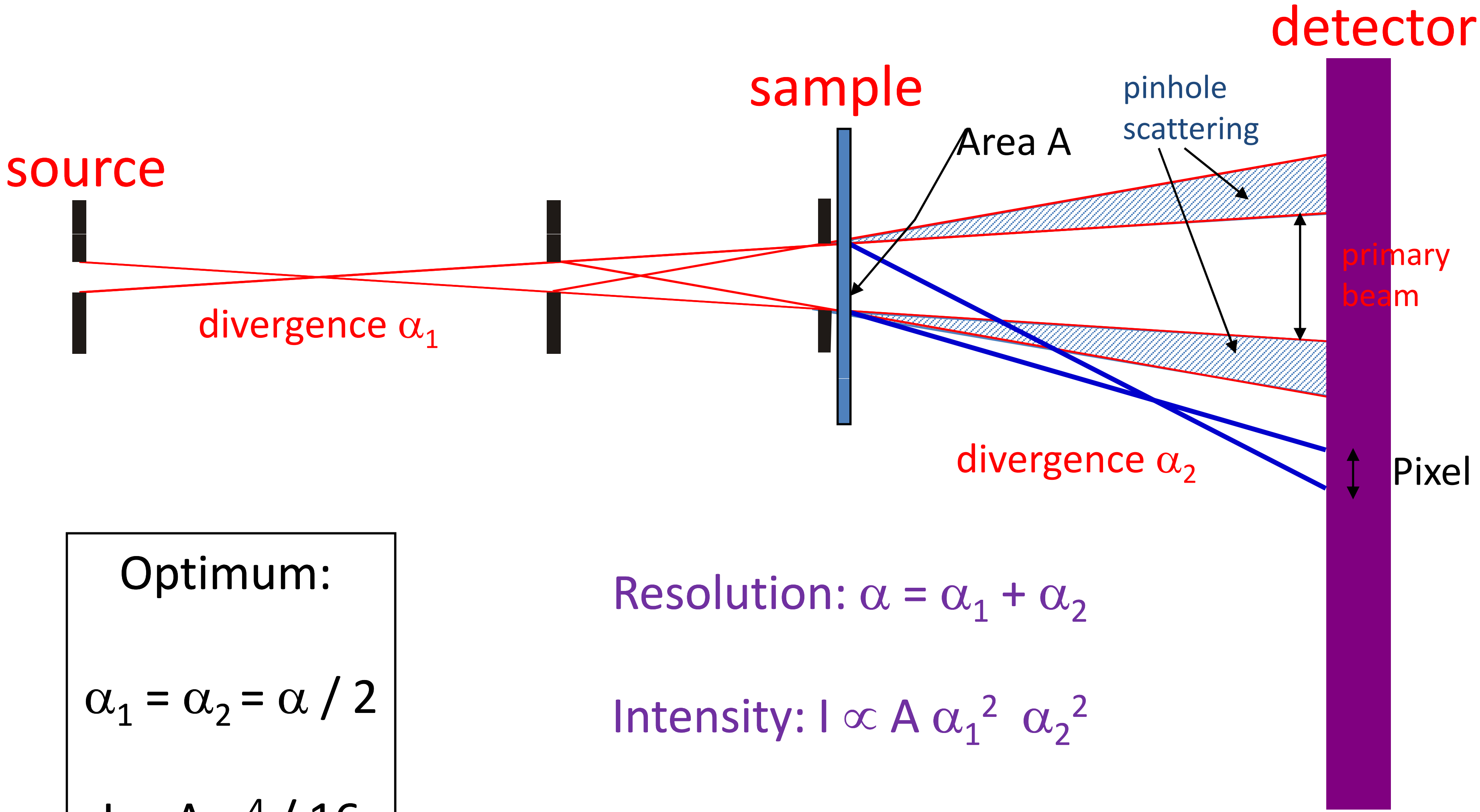
Distance 1st – 2nd pinhole: 92.5 cm

Distance 2nd – 3rd pinhole: 42.8 cm

(3-pinhole geometry based on an idea by Prof. Jan Skov Pedersen, Univ. Aarhus, DK)

Bruker AXS NanoStar – Resolution and Intensity

SEMAT
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS



Optimum:

$\alpha_1 = \alpha_2 = \alpha / 2$

$I \propto A\, \alpha^4 / 16$

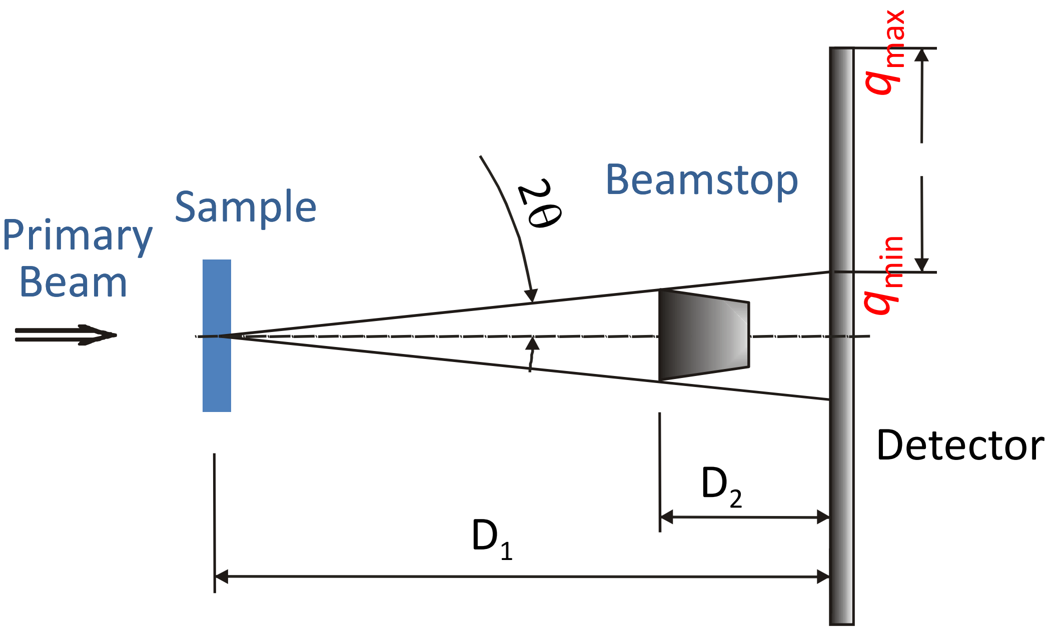
Resolution: $\alpha = \alpha_1 + \alpha_2$

Intensity: $I \propto A\, \alpha_1^2\ \alpha_2^2$

Hendricks J. Appl. Cryst. 1978)

Bruker AXS NanoStar – Maximum Resolution

SEMAT
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS



$$\text{minimum } 2\theta = \text{Arctan}\left(\frac{\frac{BS}{2}}{D_1 - D_2}\right)$$

$$\Rightarrow q_{min} = \frac{4\pi}{\lambda}\text{Sin}(\theta)$$

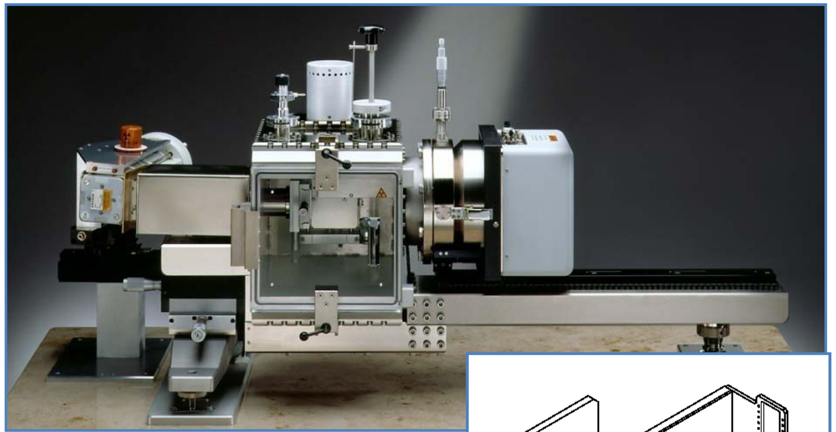
$$R_{max} = \frac{2\pi}{q_{min}}$$

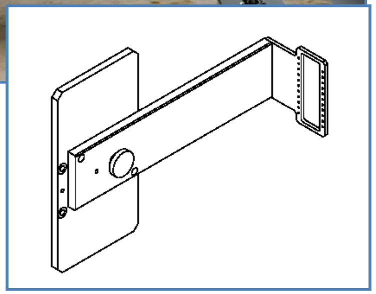
q denotes the scattering range in the reciprocal space

R_{max} gives the resolution limits of the NanoSTAR in real space

Bruker AXS NanoStar – Resolution and SD

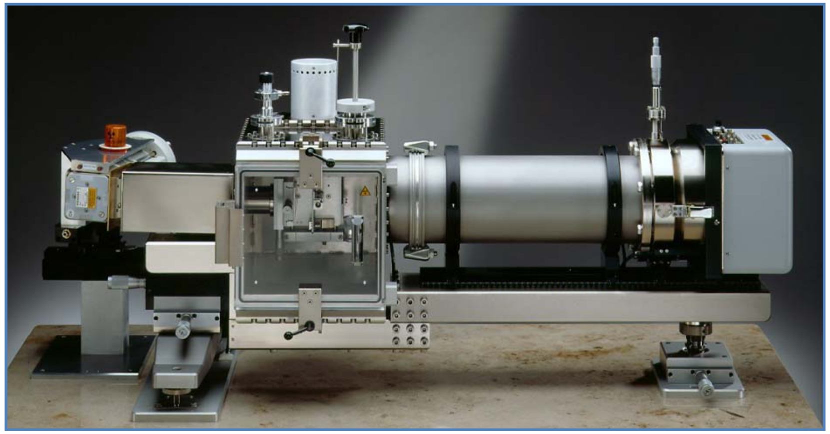
SEMAT SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS





- 100 mm
- 40 mm

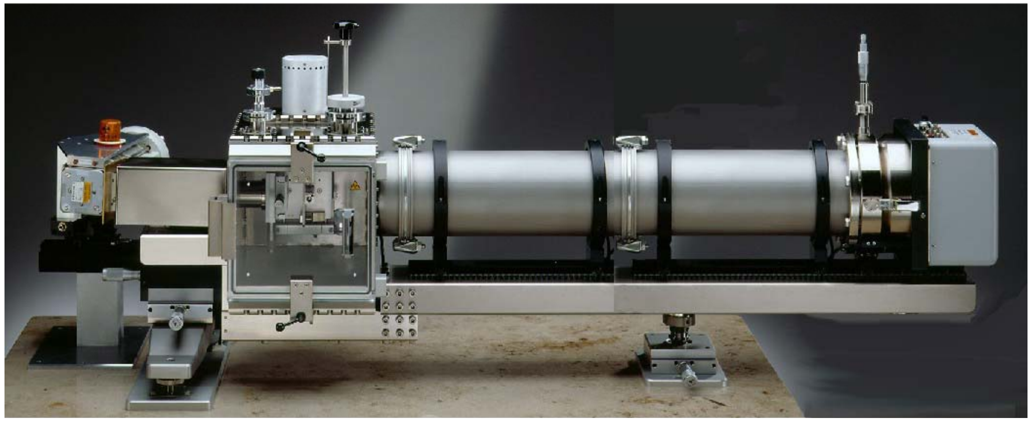
WAXS



- 650 mm

MAXS

Bruker AXS NanoStar – Resolution and SD



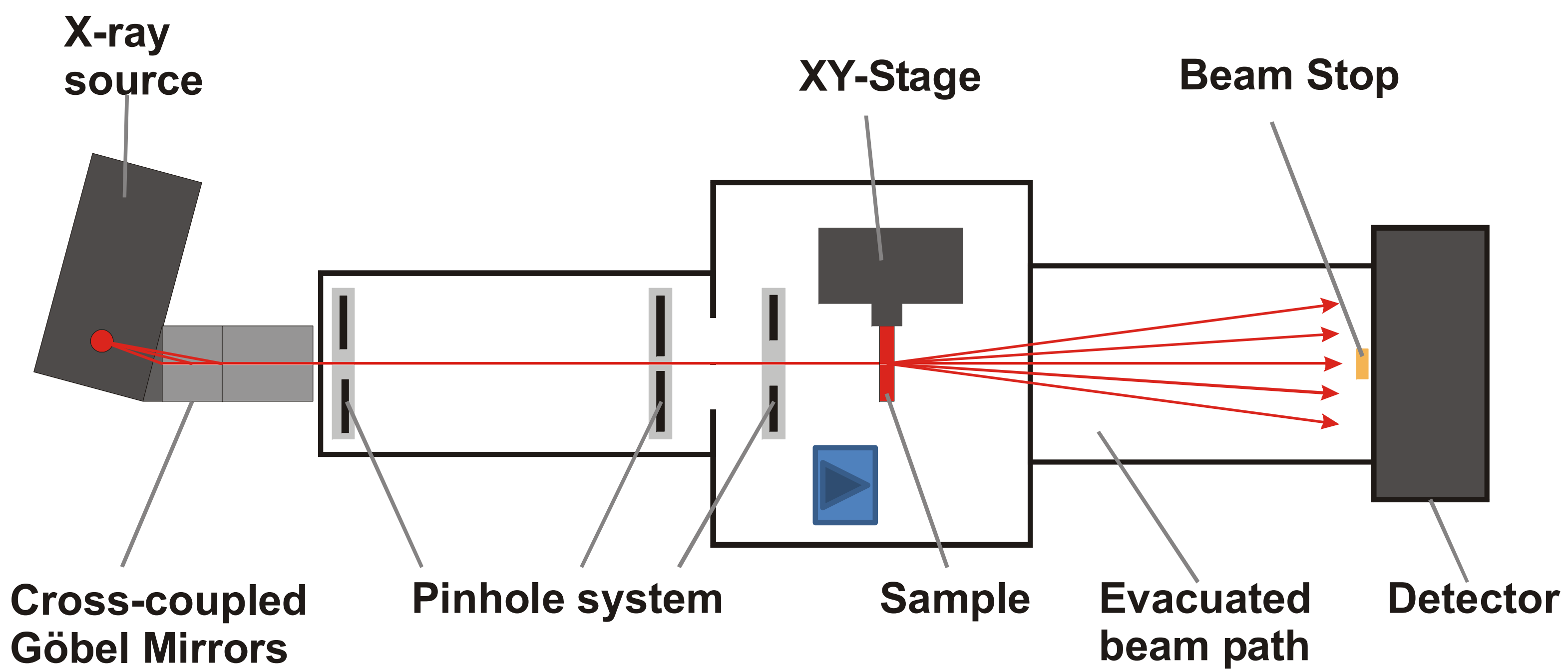
- 106 cm

SAXS

Typical resolution for the NanoSTAR with Cu radiation:

Distance Sample – Detector	Accessible q-range	Attainable Resolution R_{max}
1060 mm	$q_{min} = 0.008$ Å^{-1}	$R_{max} = 785$ Å
650 mm	$q_{min} = 0.01$ Å^{-1}	$R_{max} = 628$ Å
100 mm	0.07 Å^{-1} – 3.00 Å^{-1}	3 Å - 200 Å
40 mm	$q_{max} = 2.8$ Å^{-1}	$R_{min} = 2$ Å

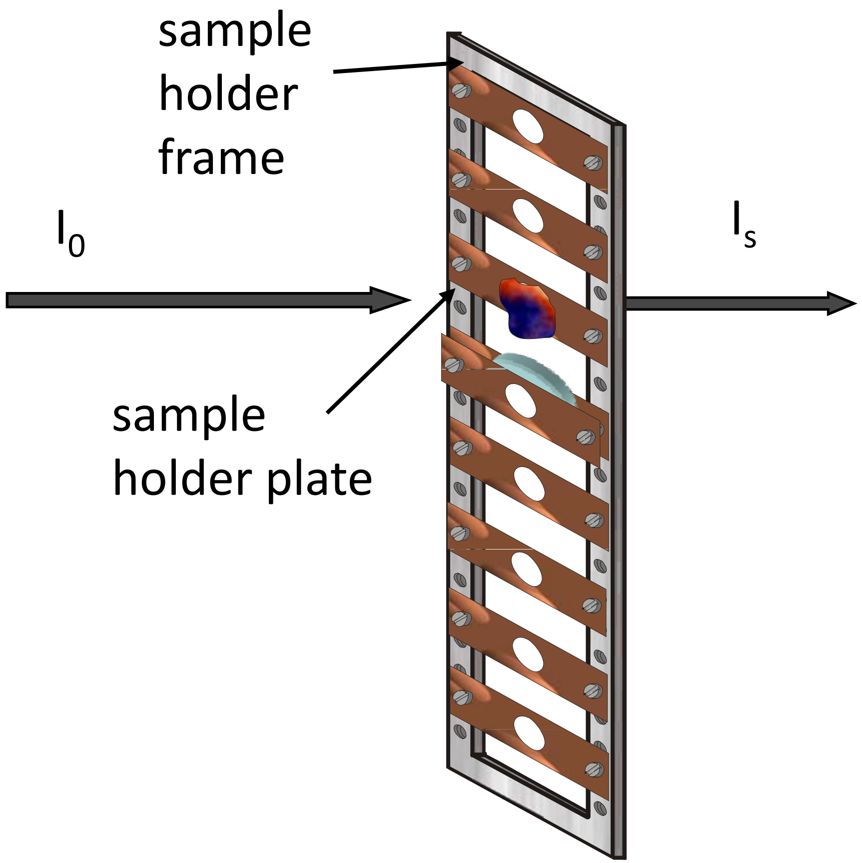
Bruker AXS NanoStar – Sample Environment



Distance 1st – 2nd pinhole: 92.5 cm

Distance 2nd – 3rd pinhole: 42.8 cm

Bruker AXS NanoStar – Sample Environment



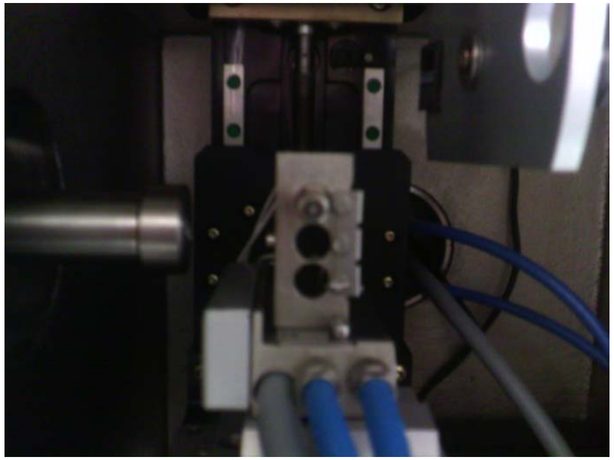
ideal sample thickness:

$$t = e^{-\mu . d} = I_s / I_0$$

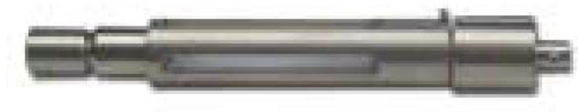
$$d_{ideal} = {}^{1}/_{\mu}$$

sample	typical thickness
polymers	2 mm
H_2O solutions	1 mm
wood	200 µm
metals	30 µm

Bruker AXS NanoStar – Sample Environment

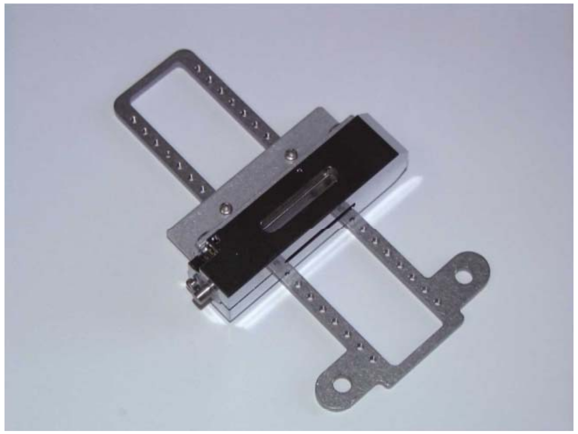


- Sample holder for controlled heating/cooling between *30-300ºC*

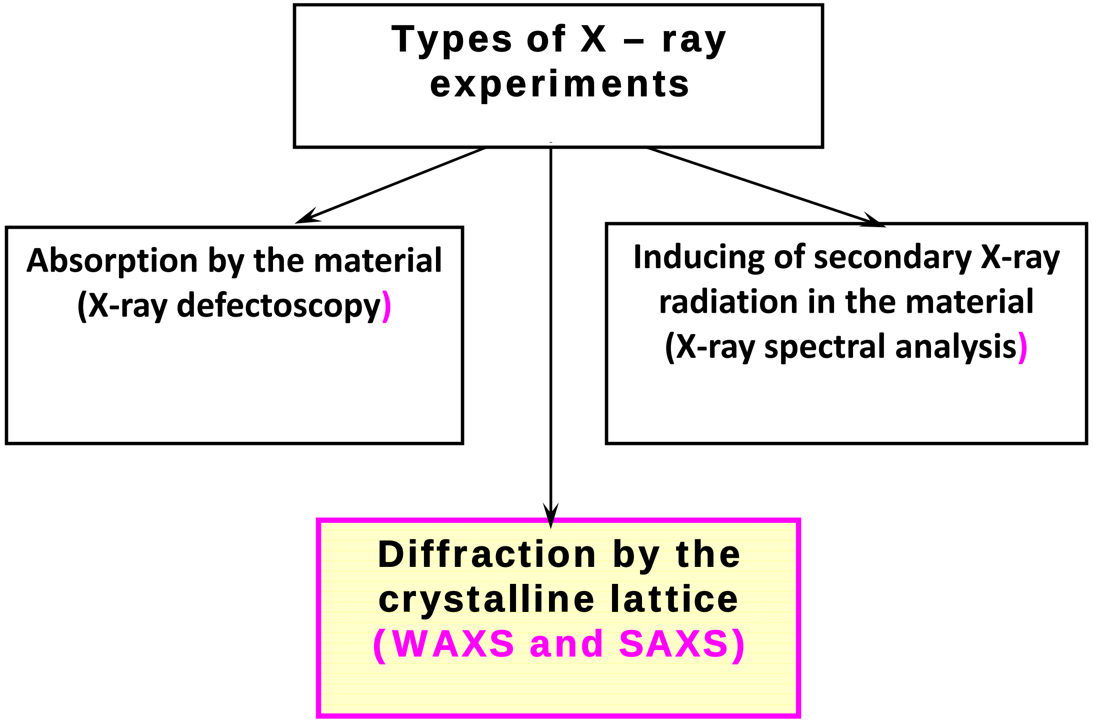


- Quartz Cuvettes

  A microliter syringe is used to fill the sample into the cuvette.

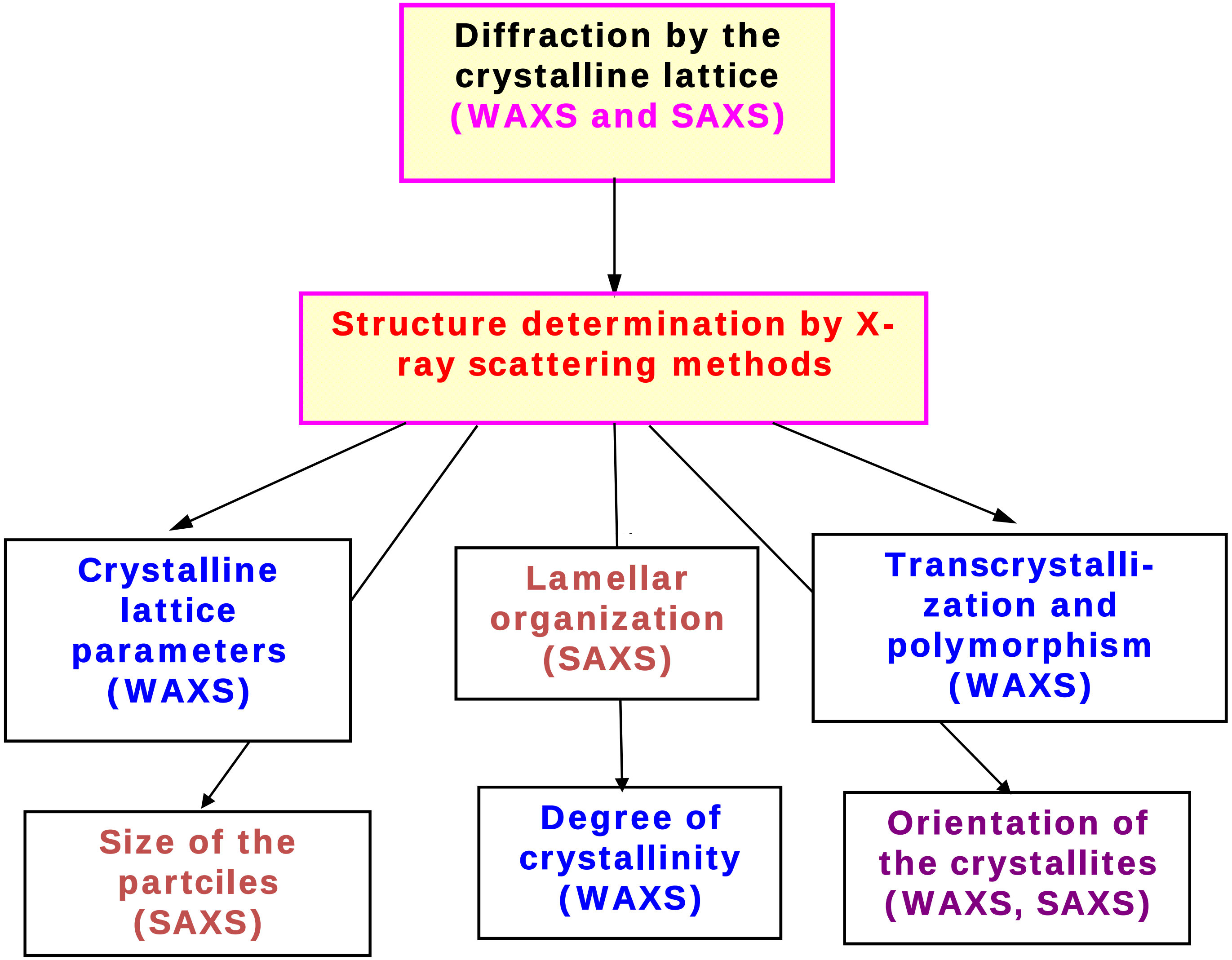


Bruker AXS NanoStar – Structure investigations in polymers

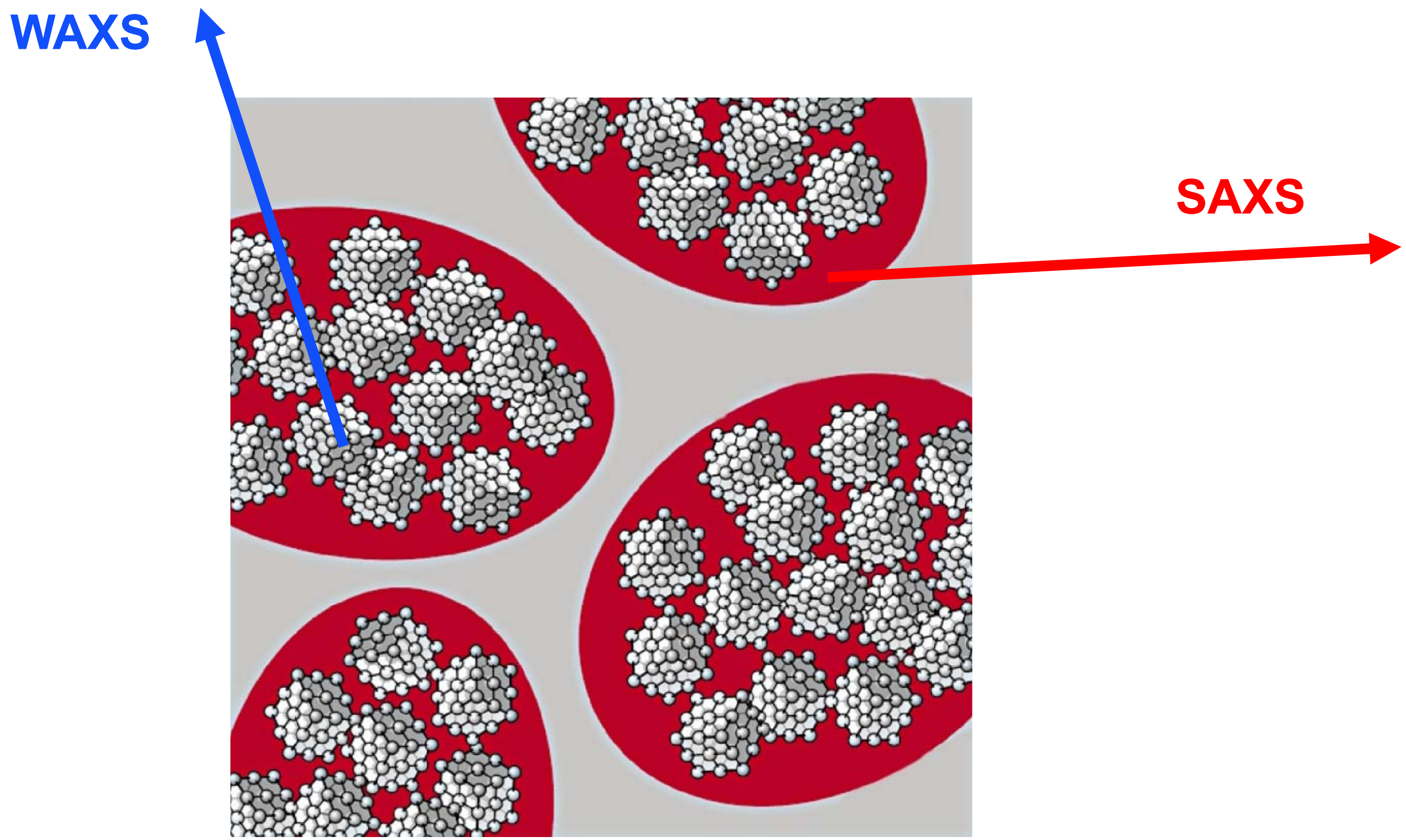


Bruker AXS NanoStar – Structure investigations in polymers

SEMAT
SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS



X-ray experiments: WAXS and SAXS



Orders of magnitude in polymer nanostructure

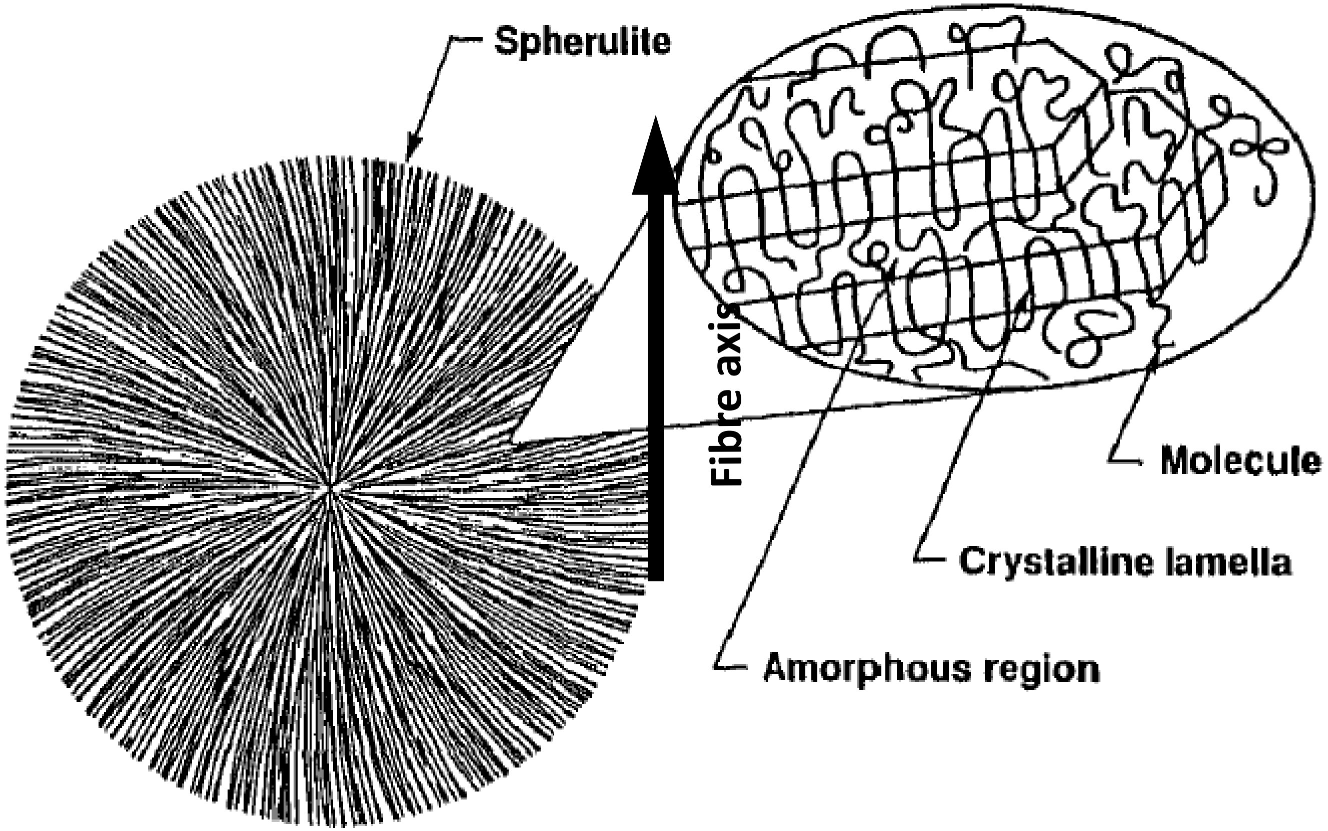
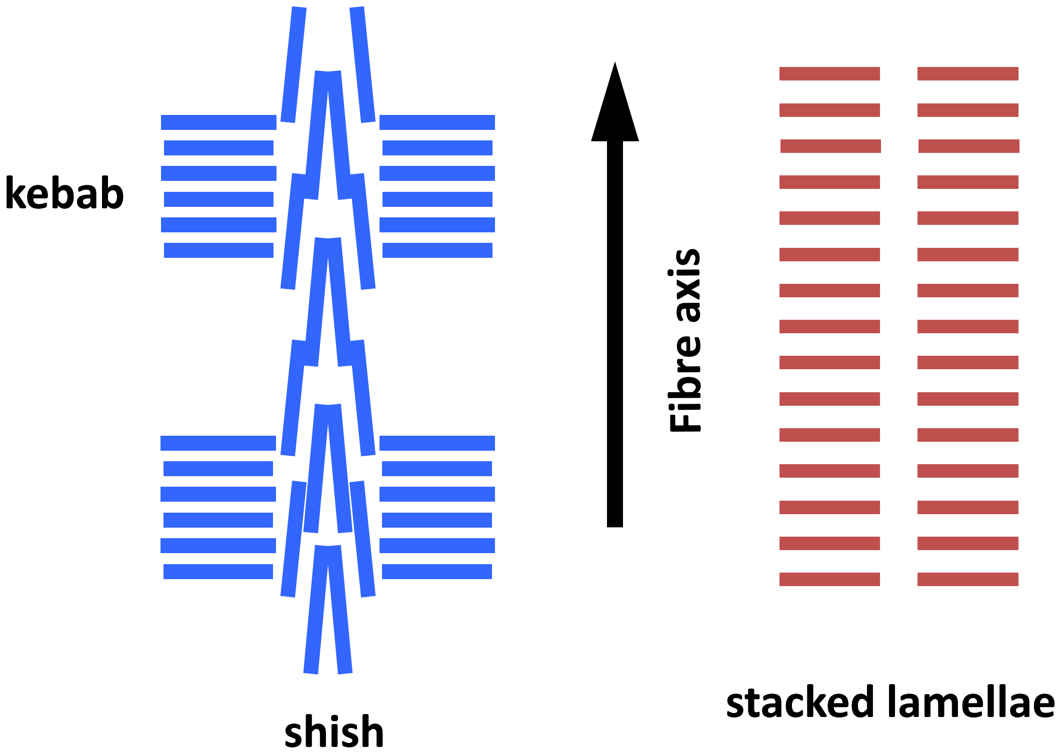


Figure 7. Schematic diagram of the spherulitic morphology of semicrystalline polymers. (Adapted from Broadhurst and Davis [17] and Lovinger [204].)

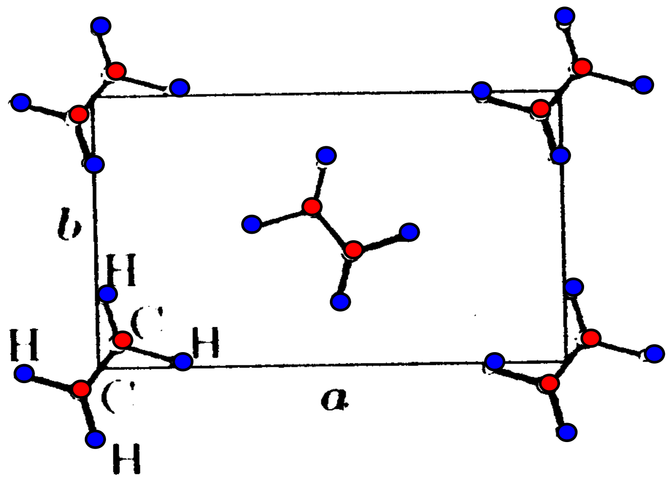
Kepler & Anderson, Adv. In Physics, 1992, 41, 1, 1-57

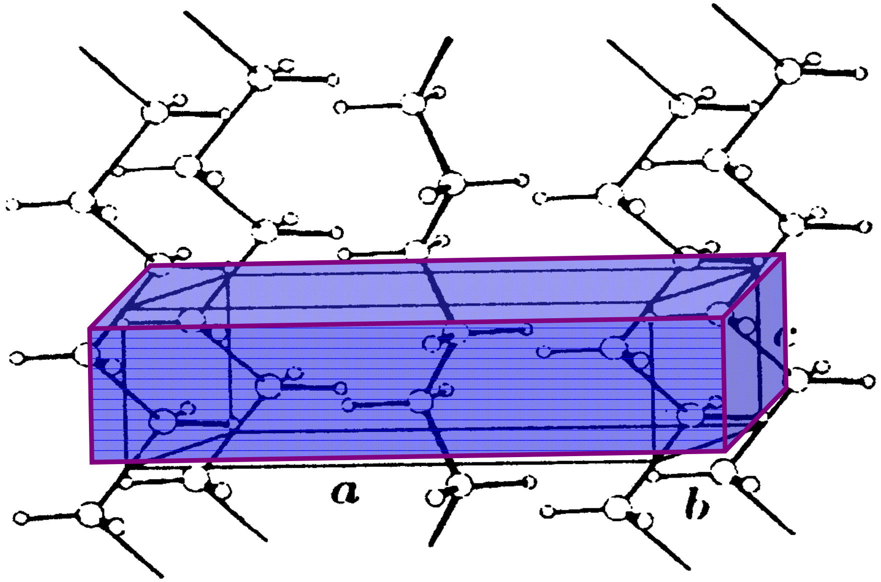
Orders of magnitude in polymer nanostructure



Lamellar structure and orientation – 2D SAXS

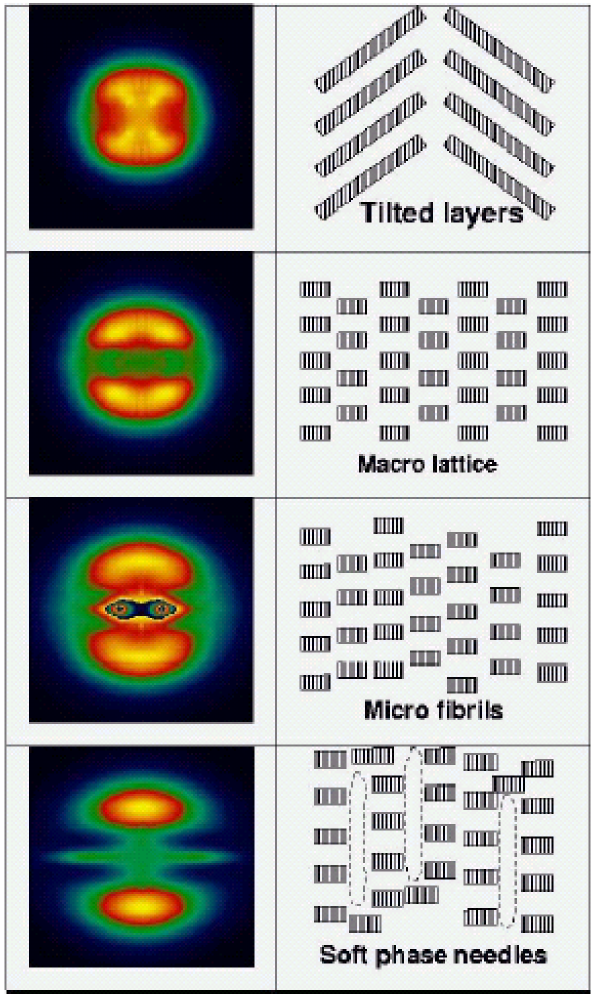
Orders of magnitude in polymer nanostructure

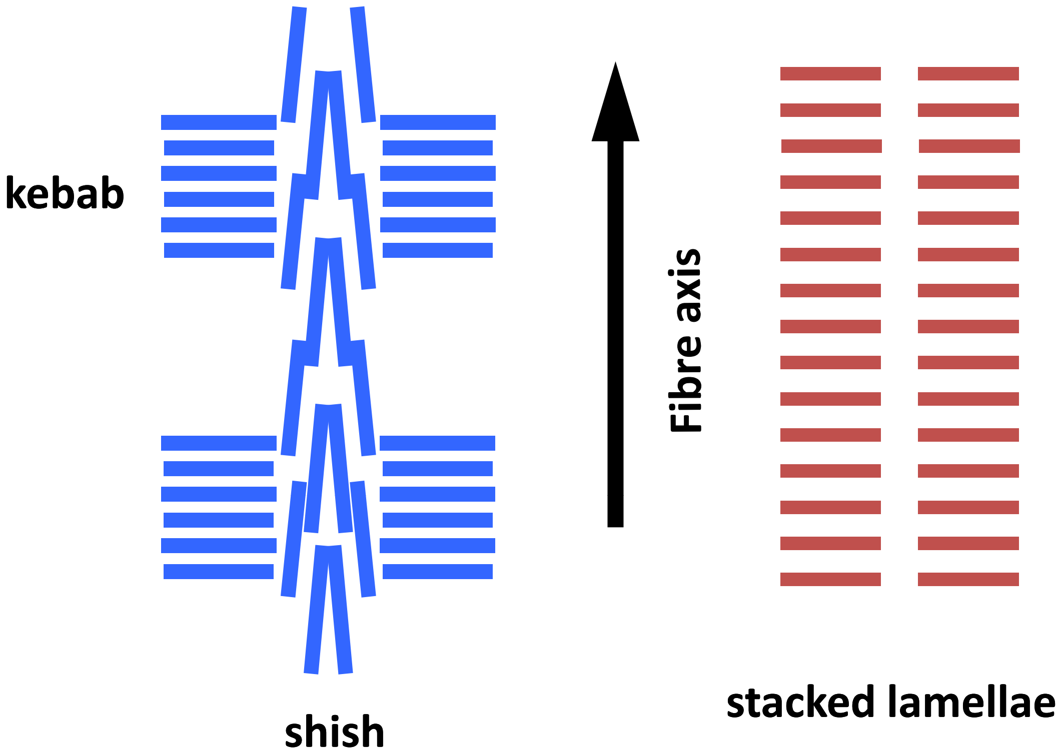




Crystallite structure and orientation - WAXS

Models of lamellar nanostructure





For more information see the index page of Prof. N. Stribeck at
http://www.chemie.uni-hamburg.de/tmc/stribeck/focus/index_e.html

Bruker AXS NanoStar X-ray data station - Summary

SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS

<u>In WAXS mode</u>:

(+) all what a normal diffractometer does plus studies related to orientation of the crystalline phase.

(-) Limited 2Θ range.

<u>In SAXS mode</u>

(+) particles size and size distribution;

(+) particles shape (spherical, cubic, cylindrical…)

(+) orientation

(+) main distance between particles….

(-) Skills and additional software needed for rigorous data evaluation.

Some practical hints for the users:

SERVIÇOS DE CARACTERIZAÇÃO DE MATERIAIS

1.Changing the setup:

- 2 weeks for WAXS setup, 2 weeks for SAXS setup
- Allow 1 entire day for the change.

2. "Machine studies":

-Allow 2 more days per month for optical system adjustments, calibrations, machine testing and maintenance.

3. Booking the equipment:

- In a weekly users' meeting