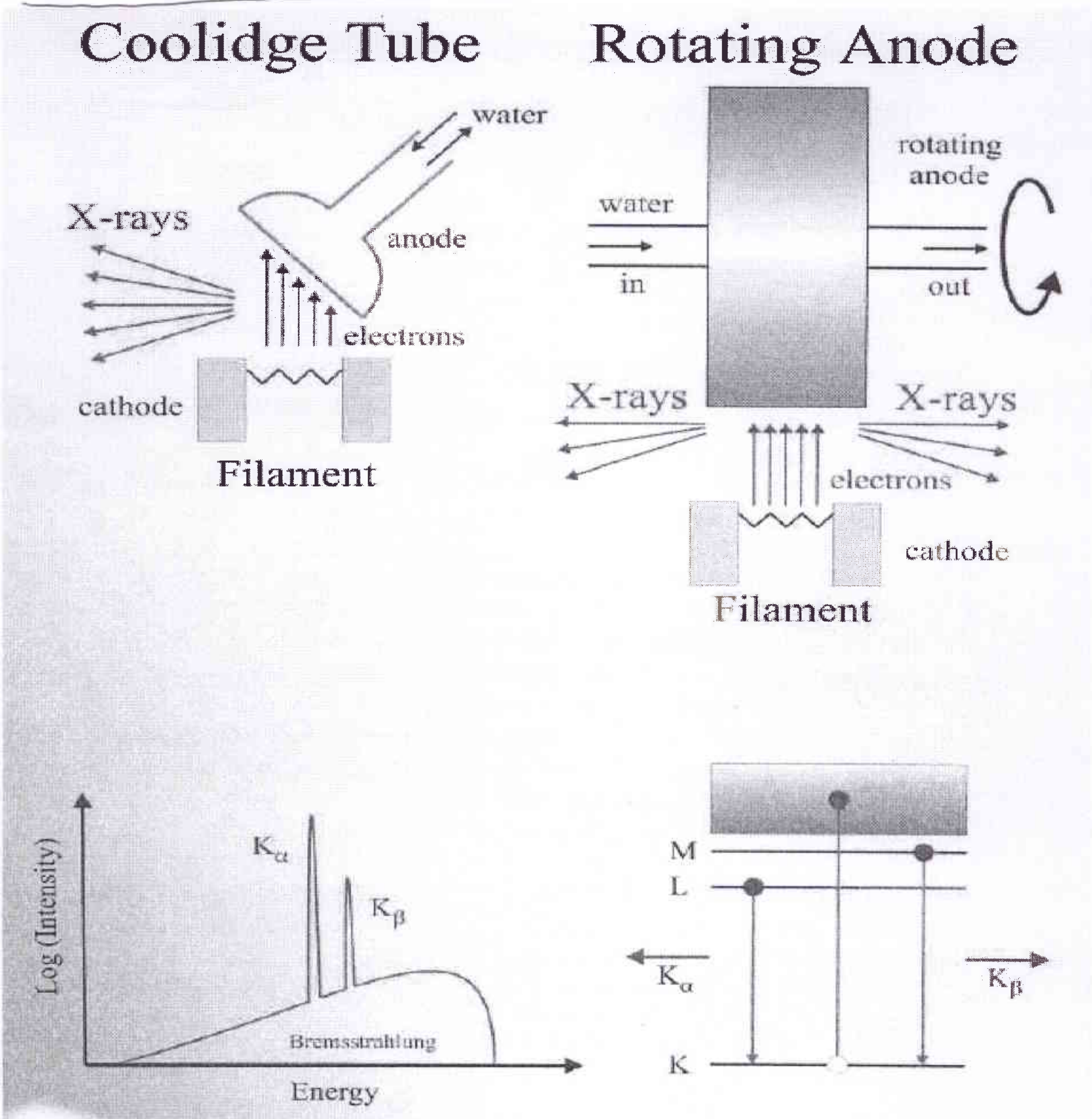
X-ray Sources:

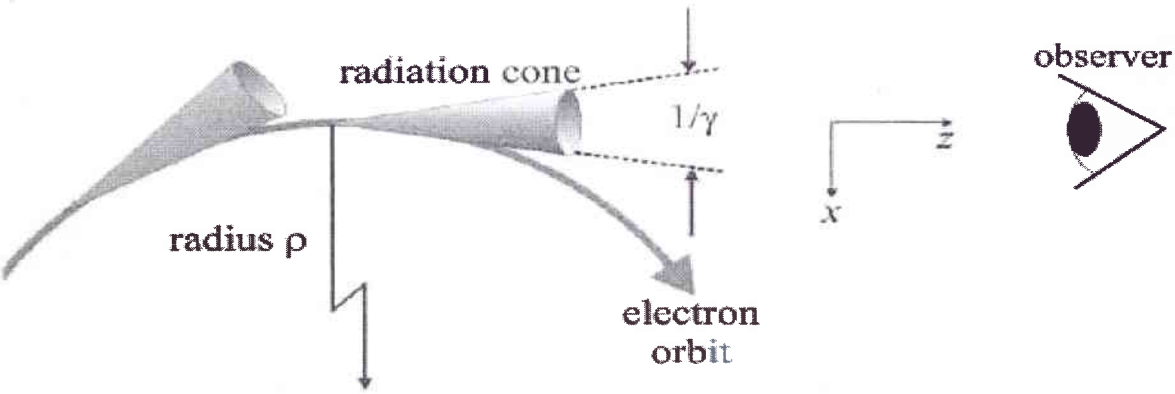


The standard X-ray tube (upper, left) was developed by Coolidge around 1912. The intensity limitation is set by the maximum power a cooled metal anode can withstand. The power can be increased by dissipating it over a larger volume which is achieved by rotating the anode (upper, right). The spectrum from an X-ray tube has discrete fluorescent lines superimposed on the continuous bremsstrahlung radiation (bottom, left). Schematic atomic energy level diagram (bottom, right): the K_α line results from transitions between an L and K shell, whereas the K_β comes from an M to K transition.

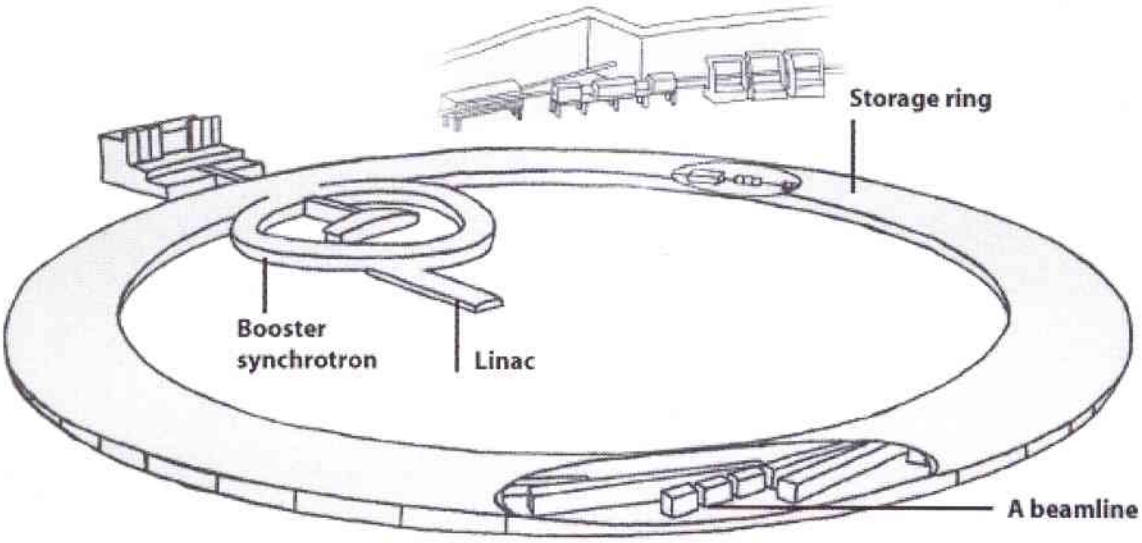
$\Rightarrow$ Discrete X-ray energies, for example Cu_{K_α}

2.

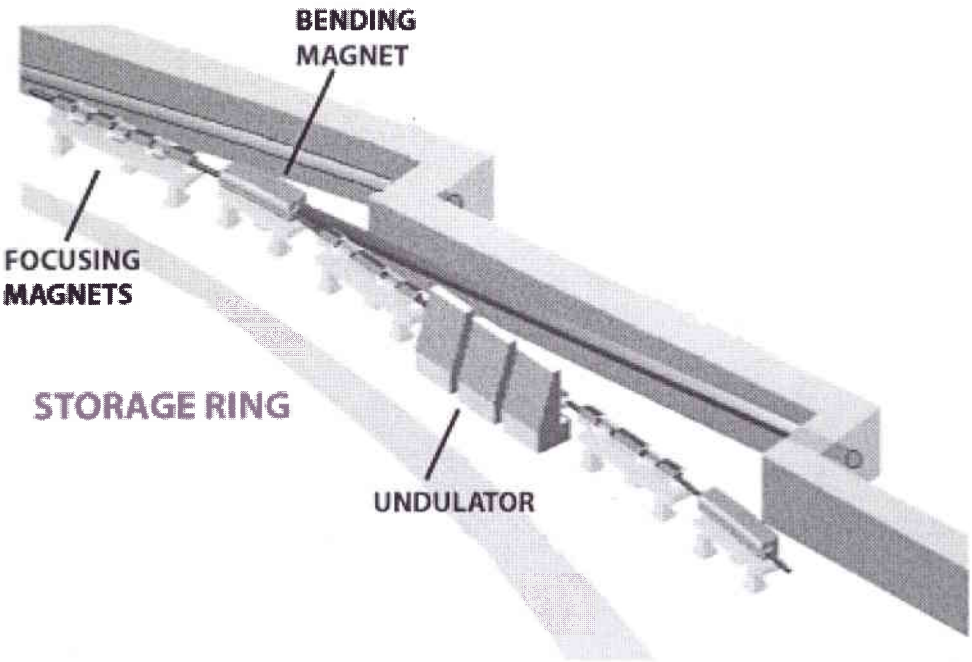
Synchrotron radiation:

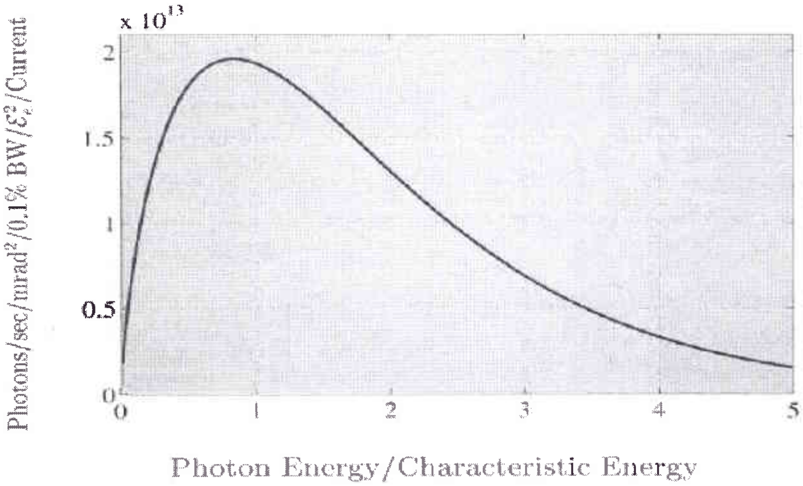


: The trajectory of an electron is approximated by a circle of radius ρ. The radiation is confined to a narrow cone with an opening angle of $1/\gamma$ around the instantaneous velocity.



3.



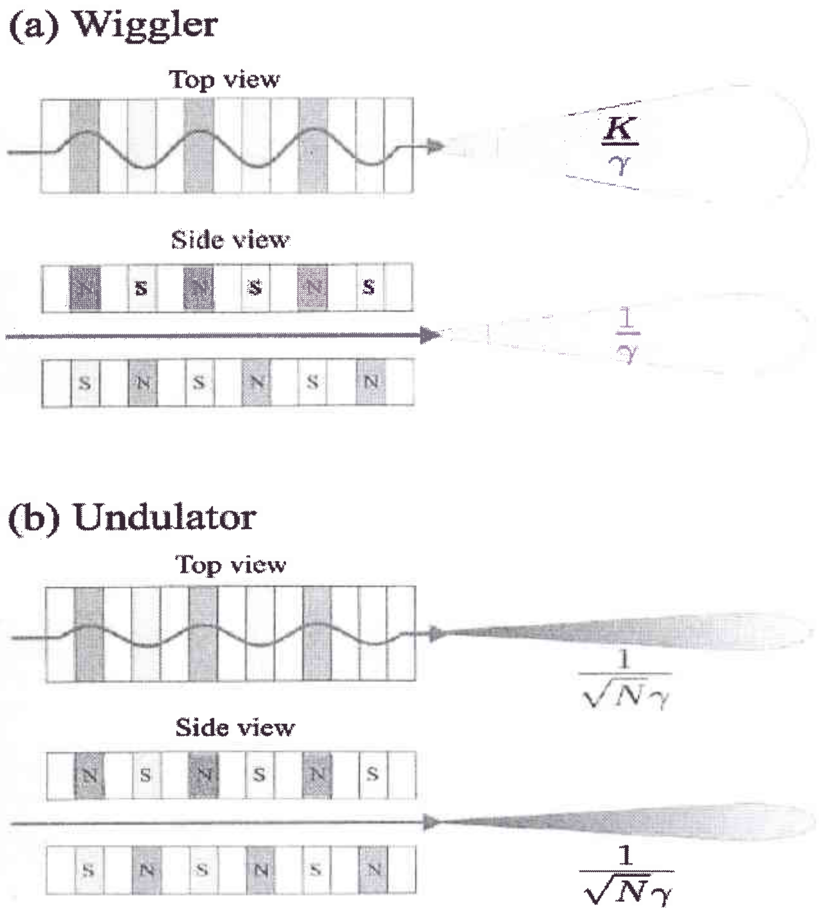


: The brilliance spectrum from a bending magnet, normalized by the square of the electron energy and the beam current. The abscissa is $x = \hbar\omega/(\hbar\omega_c)$, i.e. the photon energy normalized by the characteristic energy $\hbar\omega_c = \left(\frac{3}{2}\right)\gamma^3\hbar\omega_0$.

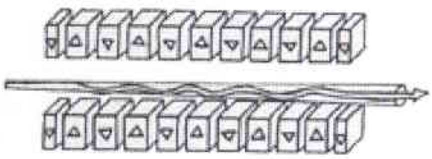
=> Synchrotron radiation gives continous wavelength - spectrum

=> Wavelength may be tuned at synchrotron

4. Insertion devices: Increase of X-ray Power.

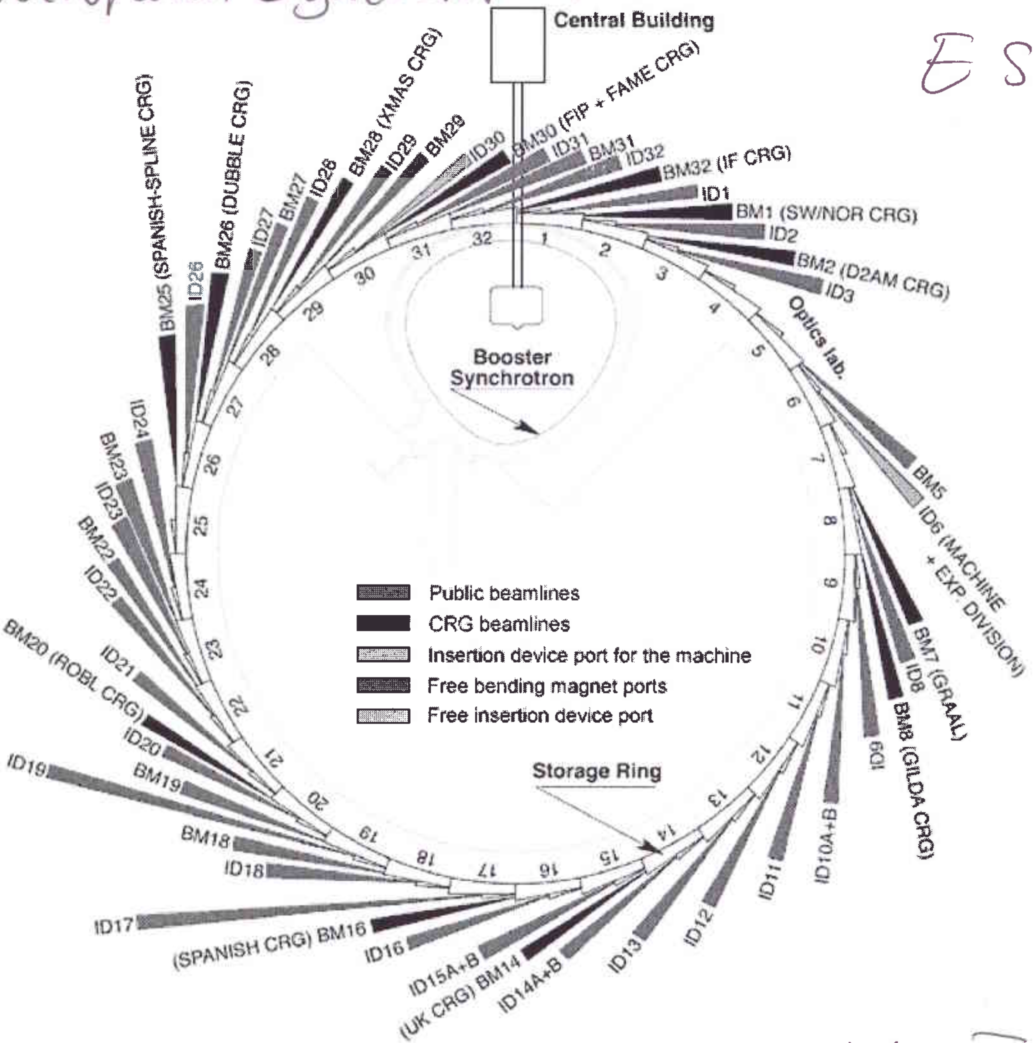


Radiation from a wiggler (top) and from an undulator (bottom). The difference in the performance of these devices arises from the difference in the maximum angle of the electron oscillations in the horizontal plane: K is around 20 for a wiggler, and 1 for an undulator. The consequence is that the radiation cone from an undulator is compressed by a factor of approximately $1/\sqrt{N}$ (Eq. (2.15)) relative to the natural opening angle of synchrotron radiation, $1/\gamma$. The number of periods N is typically around 50.

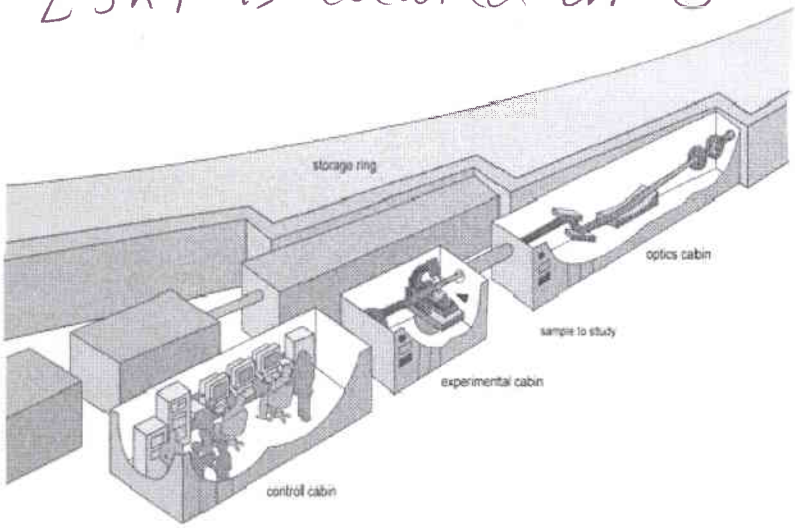


5. Example of Synchrotron source: European Synchrotron Radiation Facility

ESRF



ESRF is located in Grenoble, France



Radiation is "taken out" at beamlines tangential to the storage ring.

ESRF: Grenoble France

Storage ring diameter 270 m
=> Circumference ~ 844 m
688 bending magnets.

7.

Characterization of X-ray sources => Brilliance

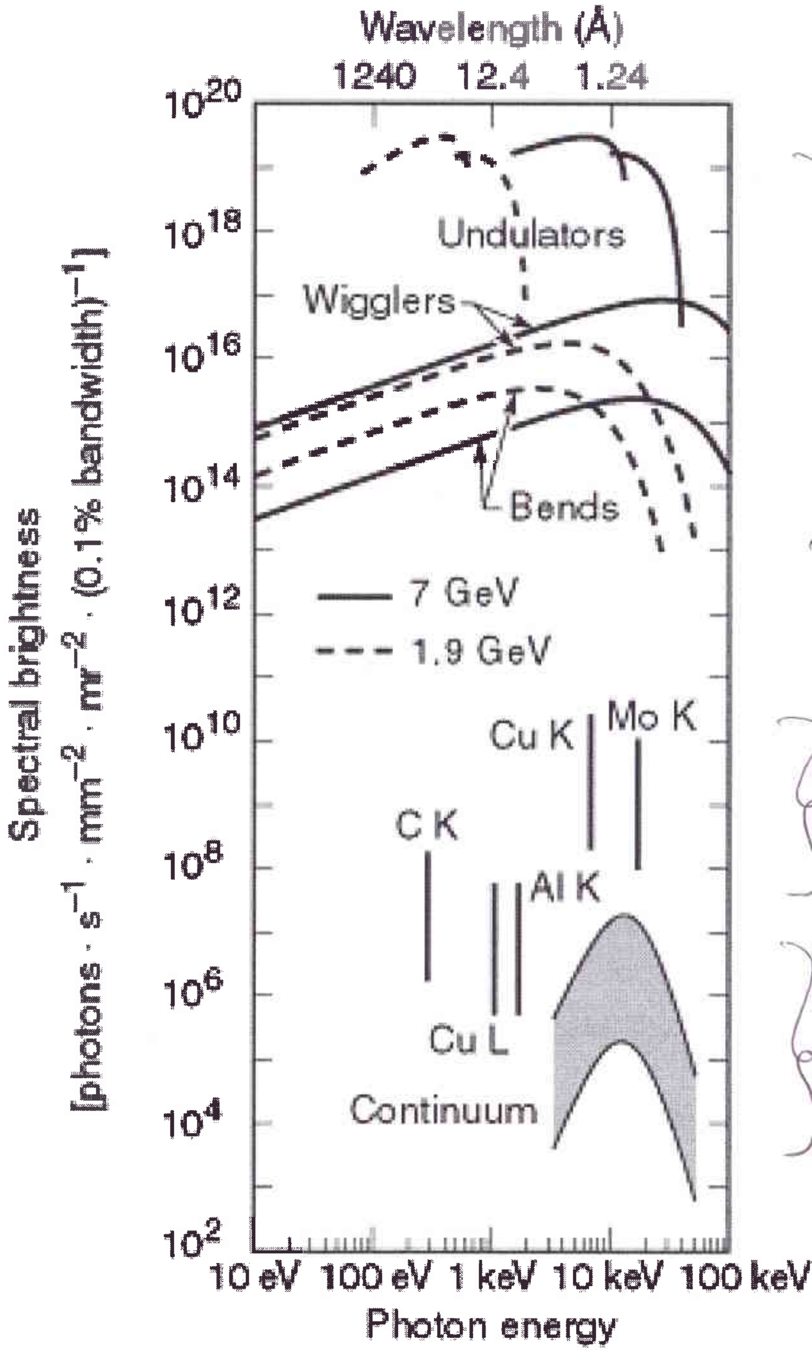
Several aspects of an X-ray source determine the quality of the X-ray beam it produces. These aspects can be combined into a single quantity, called the brilliance, which allows one to compare the quality of X-ray beam from different sources. First of all, there is the number of photons emitted per second. Next, there is the collimation of the beam. This describes how much the beam diverges, or spreads out, as it propagates. Usually the collimation of the beam is given in milli-radian, both for the horizontal and for the vertical direction. Third, it may be of importance how large the source area is – if it is small, one may be able to focus the X-ray beam to a correspondingly small image size. The source area is usually given in mm^2. Finally, there is the issue of the spectral distribution. Some X-ray sources produce very smooth spectra, others have peaks at certain photon energies. So it matters, when making comparisons, what range of photon energies contribute to the measured intensity. The convention is therefore to define the photon energy range as a fixed relative energy bandwidth, which has been chosen to be 0.1%. There are several reasons why the relative rather than the absolute bandwidth is chosen. One reason is that monochromator crystals are often perfect crystals, and

the relative bandwidth for a perfect crystal in symmetric reflection geometry is independent of the photon energy, and depends only on the Miller indices of the reflection. Altogether then, one defines the figure-of-merit for the source as:

$$\boxed{\text{Brilliance} = \frac{\text{Photons/second}}{(\text{mrad})^2\ (\text{mm}^2 \text{ source area})\ (0.1\% \text{ bandwidth})}}$$

The intensity in photons per second after the monochromator crystal is the product of the brilliance, angular divergences set by the horizontal and vertical apertures (in milli-radian), the source area (in mm^2), and the relative bandwidth of the monochromator crystal relative to 0.1%.

8.



Synchrotron Sources

Rotating anodes

Stationary tubes

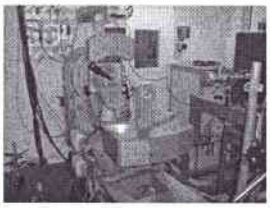
9.

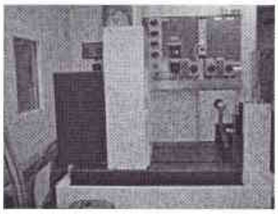
Two examples of specialized beamlines at ESRF:

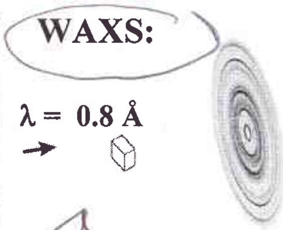
SNBL:

ESRF Grenoble:

Swiss Norwegian Beam Line SNBL/ERSF:







Synchrotron Small Angle x-ray Scattering (SAXS)

ESRF - The European Synchrotron Radiation Facility, Grenoble, France

DUBBLE:

The DUtch-Belgian BeamLinE (DUBBLE)

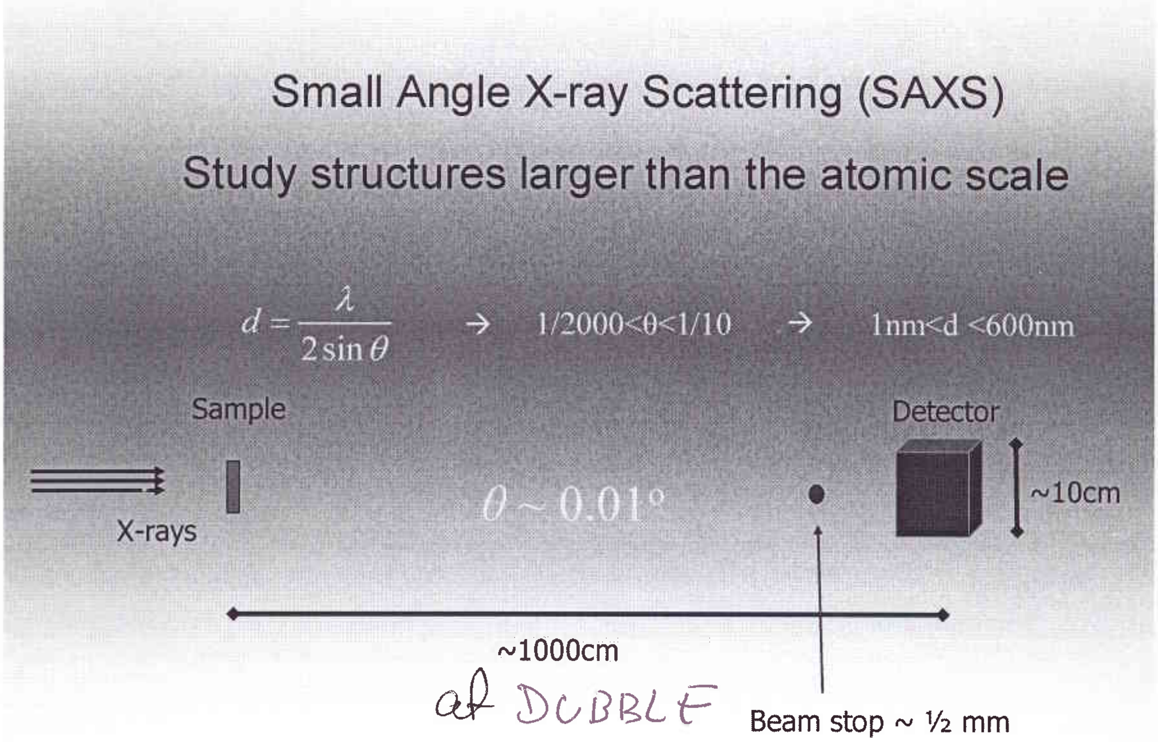
10.

What is SAXS?:

$\lambda \sim 1\,\text{Å}$

Braggs' law: $2d\sin\theta = \lambda$ $(n=1)$

$\Rightarrow$ At small angles (θ small) $\Rightarrow$ Large d's can be studied.



at DUBBLE

Disordered structures $\Rightarrow$ d is not sharp

$\Rightarrow f(d)$

$\Rightarrow g(\theta) = \frac{\lambda}{2f(d)}$ is measured

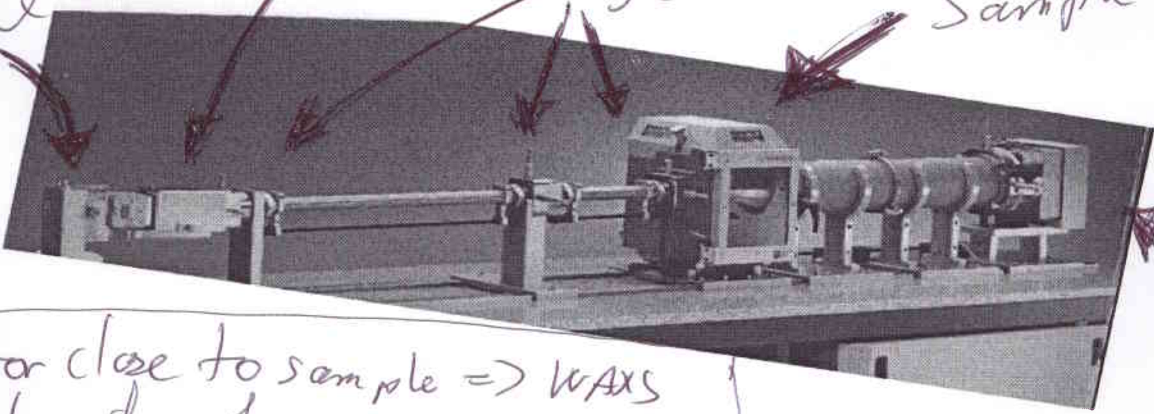
SAXS instrument in X-ray lab at NTNU:

Collimating monochromator => CuKα - parallel beam

3 collimating pinholes

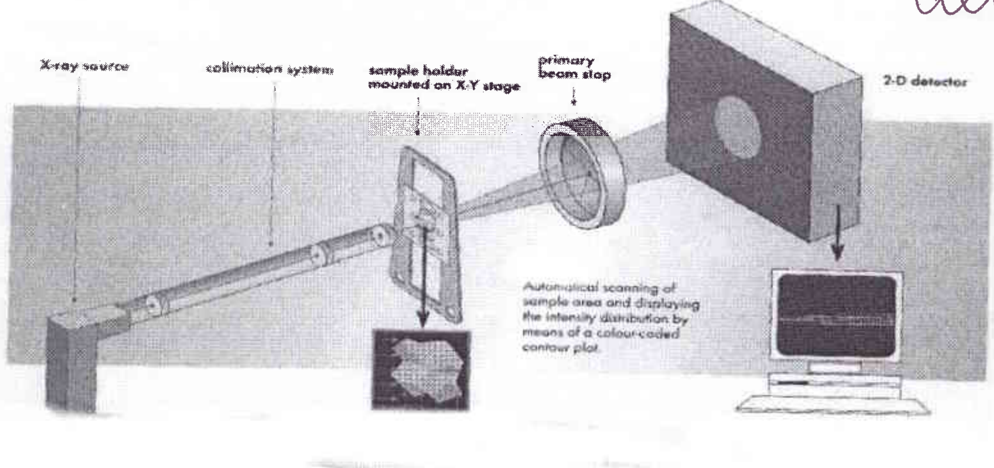
Rotating anode source

Sample chamber



$\boxed{\text{Detector close to sample => WAXS; Detector far from sample => SAXS}}$

2 dimensional detector



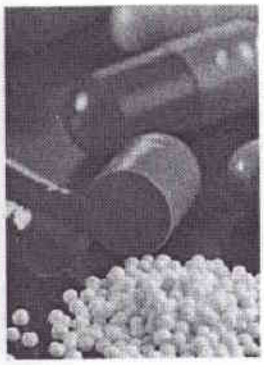
Types of 2d-detector: (Differences in resolution, speed etc.)

Image plate detector

Gas multi wire detector ← At NTNU

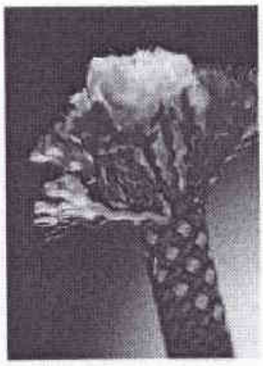
CCD detector

12. Examples of Systems one can study with SAXS:



Polymers:
For bulk- or semi-crystalline polymers and block copolymers, the mesophases are characterized by SAXS. For polymer solutions and block copolymer micelles in solution, the osmotic compressibility, molecular mass, and radius of gyration or correlation can be obtained. Additional informations on intermicellar interactions and on the structure and shape of the micelles can also be examined.

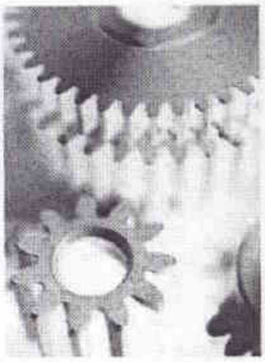
Biological materials:
In membranes or bones, e.g. cross-section structure and interlamellar ordering is analysed. In proteins, DNA, RNA, and complexes of these SAXS detects shape, structure, and aggregation state.

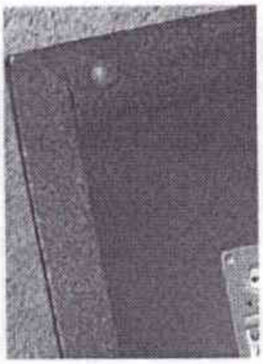


Fibres:
Polymeric fibres, composites, and natural fibres are analysed to obtain orientation and degree of ordering. Information on the internal structure of individual fibres can also be studied.

Colloids:
Gels, sols, aggregation processes, charge or sterically stabilized sols, and templating nanomaterials: these are samples where SAXS can help to derive information about size, size distribution, shape of particles, interparticle interactions, and geletation or aggregation state.

Metals:
Shape, size, composition, volume fraction as well as interparticle correlations of precipitations in alloys, nano-crystals, and nano-powders are information obtained by SAXS.





Complex fluids, liquid crystals:
SAXS provides information on size and shape of particles, interparticle interactions, degree of crystallinity, and phase transitions in liquid crystals.

13.

NTNU - System :

BRUKER ADVANCED X-RAY SOLUTIONS

BRUKER axs

Technical Data

X-ray source:	**Spot focus ceramic tube for Cu or Co radiation** Focus size: 0.4 x 0.8 mm maximum power: 1.5 KW for Cu 1.2 KW for Co
X-ray optics:	**Cross-coupled Göbel Mirrors, 3-pinhole collimation system** Pinhole diameters: 750 µm, 400 µm, 1000 µm: other diameters on request **2-pinhole collimation system** Pinhole diameters: 100 µm, 300 µm
Sample stages:	**XY-stage** Translations of 100 x 80 mm (vertical/horizontal) positional resolution: 10 µm **Heating units:** Cooling/heating unit: -30 °C ...+ 120 °C Heating unit: RT ...+ 300 °C
Sample holders:	**Liquid samples:** vacuum tight quartz capillaries with 1 mm or 2 mm diameter **Powder and bulk samples** **Reference sample holder** **Wide angle sample holder**
Detector:	**2-D HI STAR detector** Real time data collection and display, virtually background free. High quantum efficiency for high sensitivity, user-selectable 1024 x 1024 or 512 x 512 pixel frames, 11.5 cm diameter active area
Primary Beam Stop:	Made of fluorescence-free material, fixed with X-ray transparent wires, alignment accuracy better than 10 µm
Resolution:	For Copper Kα radiation the resolution capabilities for the various sample-detector distances are as follows:

Distance/mm	**q-min/Å^{-1}**	**Particle size/Å**
60	0.3	21
270	0.04	150
670	0.01	628
1070	0.00628* for NanoSTAR U	1000* for NanoSTAR U
	0.01 for NanoSTAR C	628 for NanoSTAR C

With wide angle configuration the maximum angle is 40° 2-theta. *dependent on pinhole combination and size

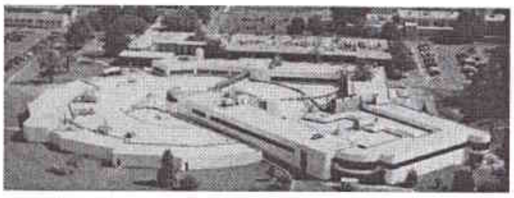
14. Two examples of use of X-ray scattering:

Example 1: A nano-layered material CLAY.

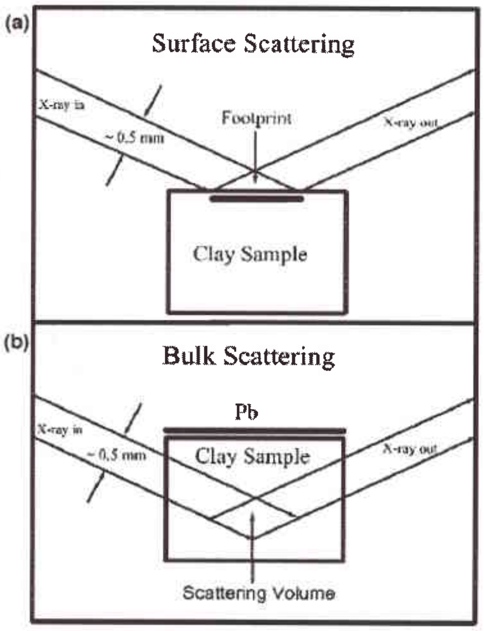
"Nanodiffusion" Experiments at Brookhaven National Lab:

$Na\text{-}(Mg_2Li)Si_4O_{10}F_2$: Sodium-fluorohectorite

Brookhaven National Lab/National Synchrotron Light Source BNL/NSLS:



Brookhave National Lab, L.I. New York

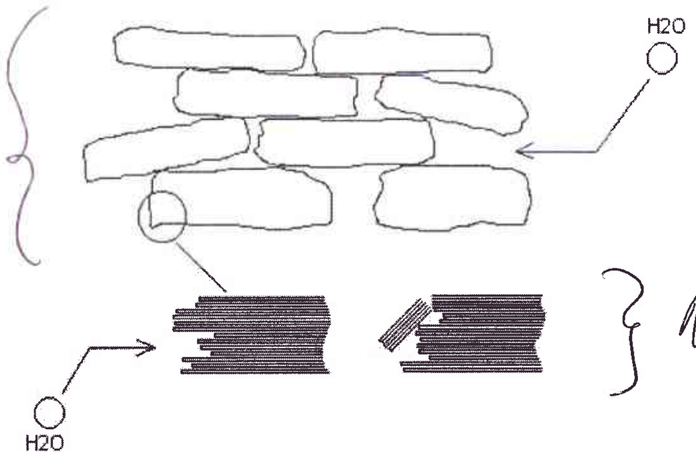


Schematic representation of the experimental arrangement utilized for the analysis of the surface scattering geometry (a) and of the bulk scattering geometry (b) of the sample.

15.

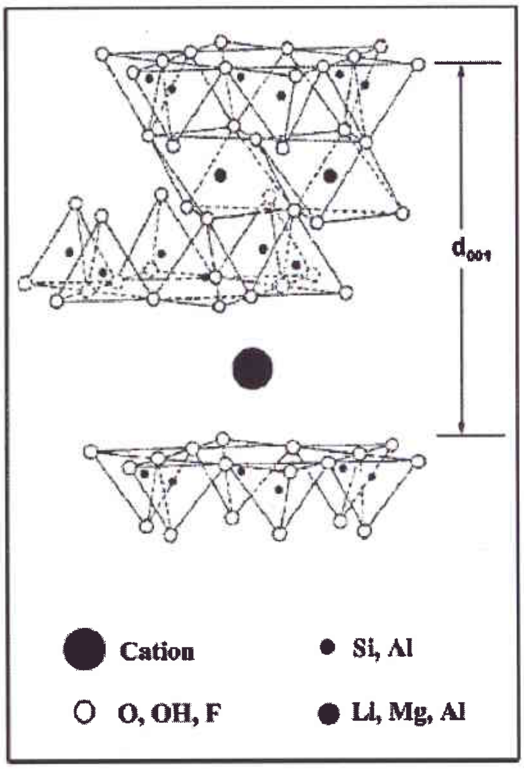
Ex 1.

Micro structure



} Nanostructure

A schematic drawing of a proposed internal organization of the dehydrated fluorohectorite. The upper part depicts a possible spatial organization of the clay grains. The insets (lower part) show how one may imagine stacking and surface irregularities/pores in the nm-range. Absorption and intercalation of water is also indicated.



Close-up of Å-nano structure

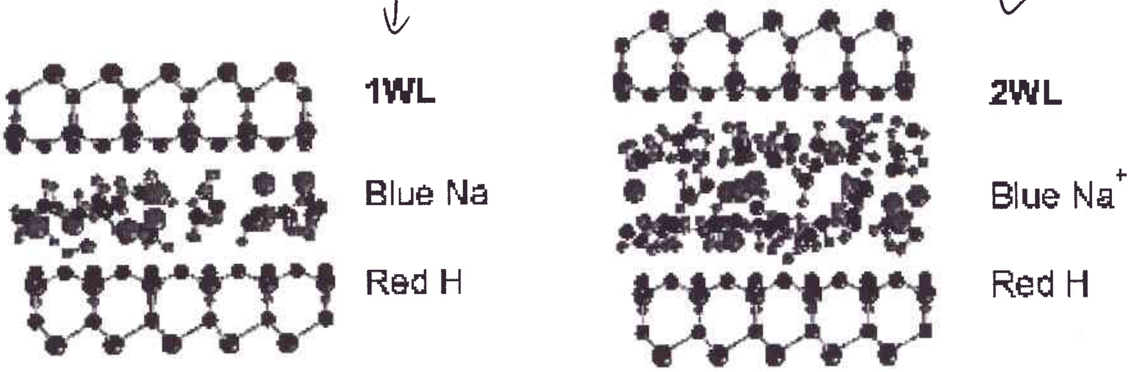
The structure of a typical 2:1 layered silicate clay. On the right the basal spacing d_{001} is indicated and below are the symbols for some typical elements of clay components.

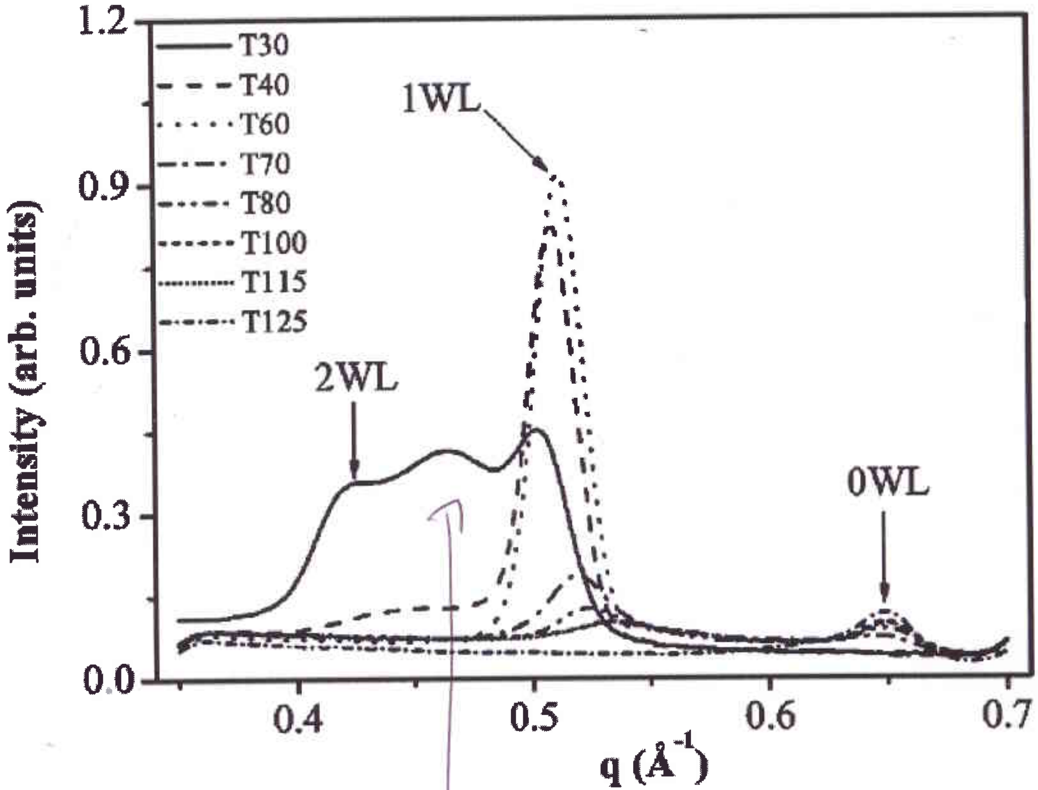
16.

Ex 1

1 Water layer

2 Water layers.

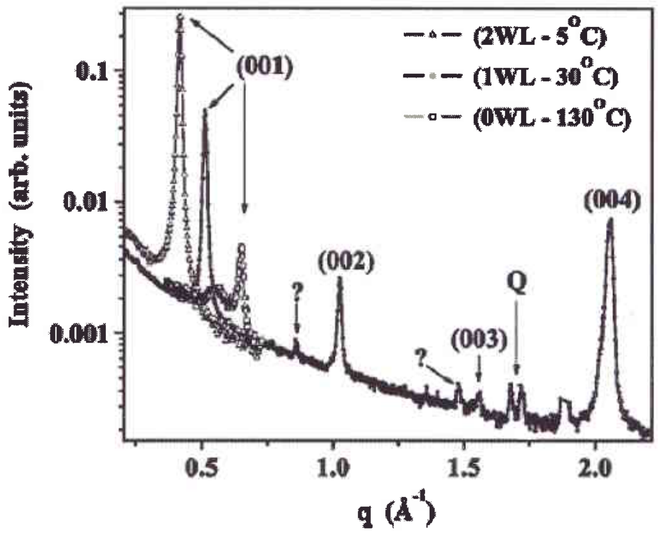




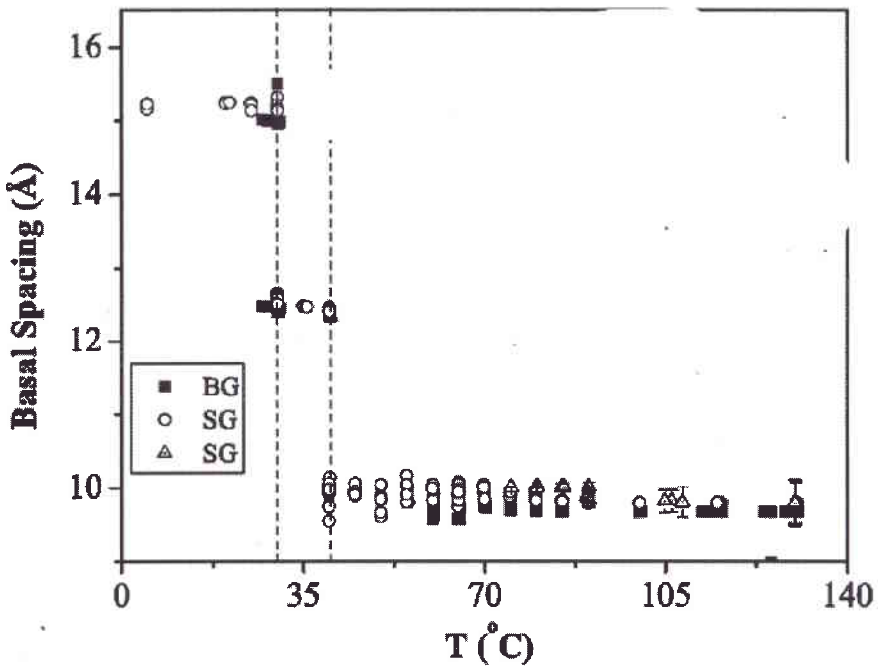
Mix of 1 and 2 Water layers

17.

Ex 1:



Bulk scattering geometry diffraction pattern. For one water layer (1WL) the peaks 001–004 are present. The symbols *Q* and "?" refer to quartz and some unidentified mineral impurities. For 0WL and 2WL only the (001) peaks are shown. The legend shows typical values of the temperature.



Basal spacing vs temperature for synthetic Na-fluorohectorite as determined from the (001) peaks.

EX 1.

WAXS: $2d\sin\theta = n\lambda$

Measure experimentally:

1. Peak positions ⇒ Lattice Spacing

2) Peak widths ⇒ Disorder

Arising from ↓

a) Temperature (D-W factor)

Williamson – Hall analysis {
b) Finite sample size
c) Lattice spacing disorder

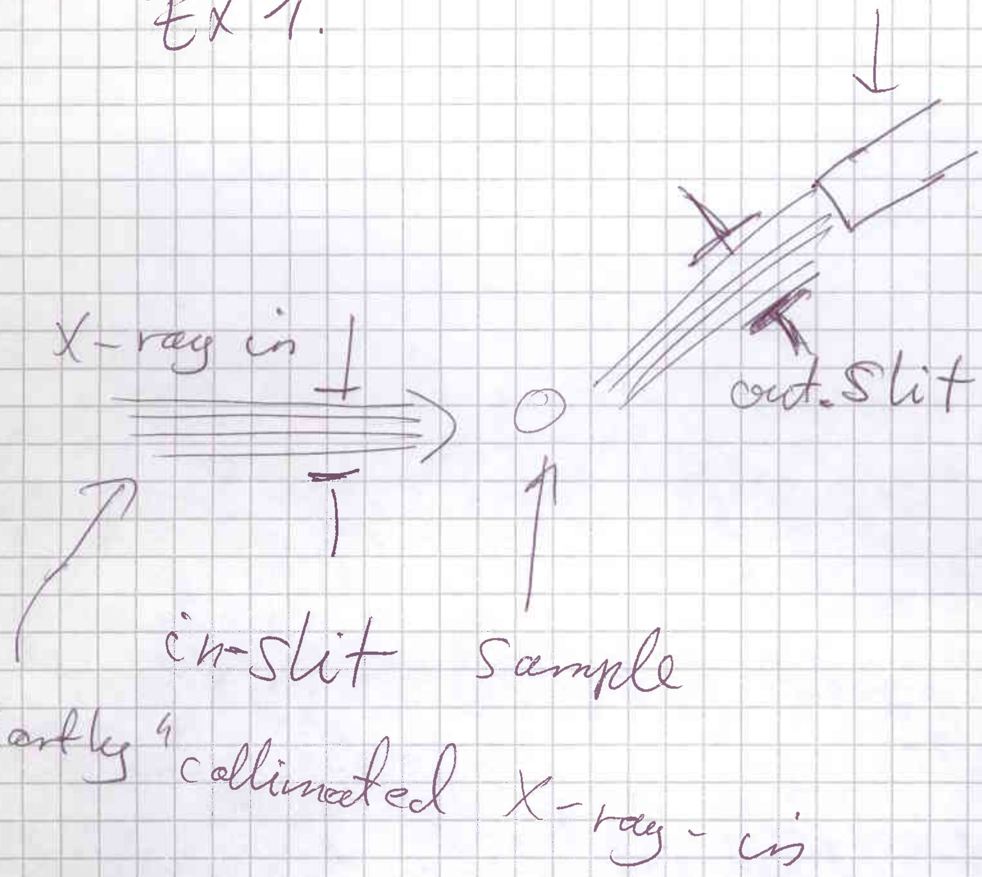
d) Instrument resolution
• Finite beam size
• Collimation of beam

$$I_{measured} = I_{sample} \otimes I_{instrument}$$

convolution

Approx: Pseudo-Voigt used in EX. 7.

Ex 1.



=) I instrument depends on

- Collimation
- In- slit
- Out - slit.

Ex. 2

Use of 2D-detector to study texture.

System:

Polarizable nanoparticles in oil suspension.

EACH is a nano layered stack

=> EACH particle gives a Bragg-peak when oriented correctly with respect to incoming beam.

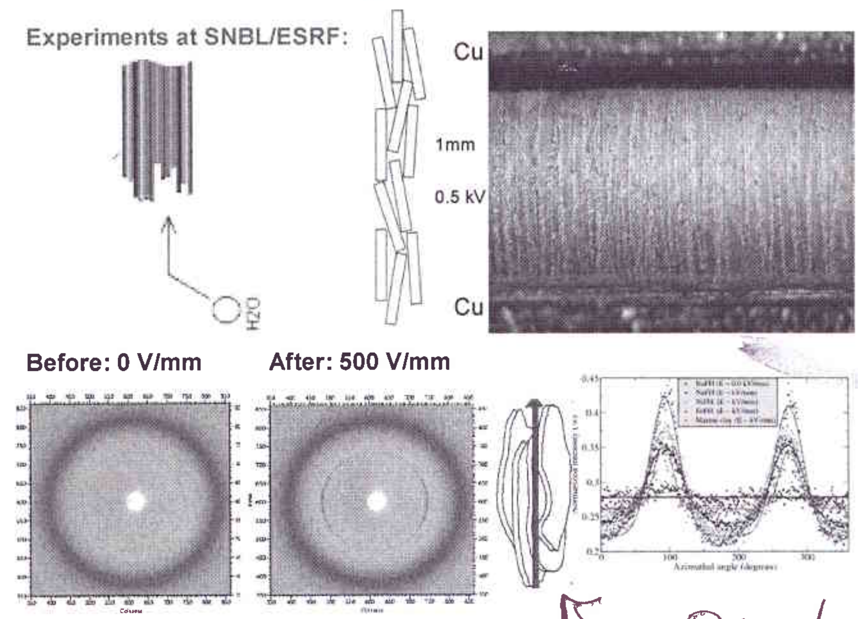
=> No preferred orientation gives rings at Bragg-scattering at 2D-detector.

=> Preferred orientation gives semi-circular Bragg-scattering on 2D-detector.

21. Ex2

Orientational distribution

Microscope image



Chain formation of dipolar nano-particles

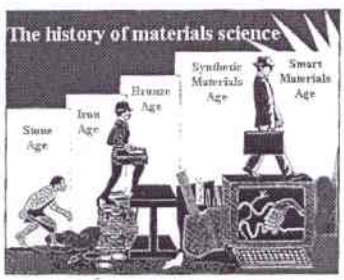
Dipole-direction

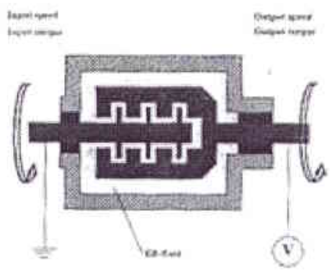
ring of scattering

semi circular scattering

Smart materials:

Smart Clay?

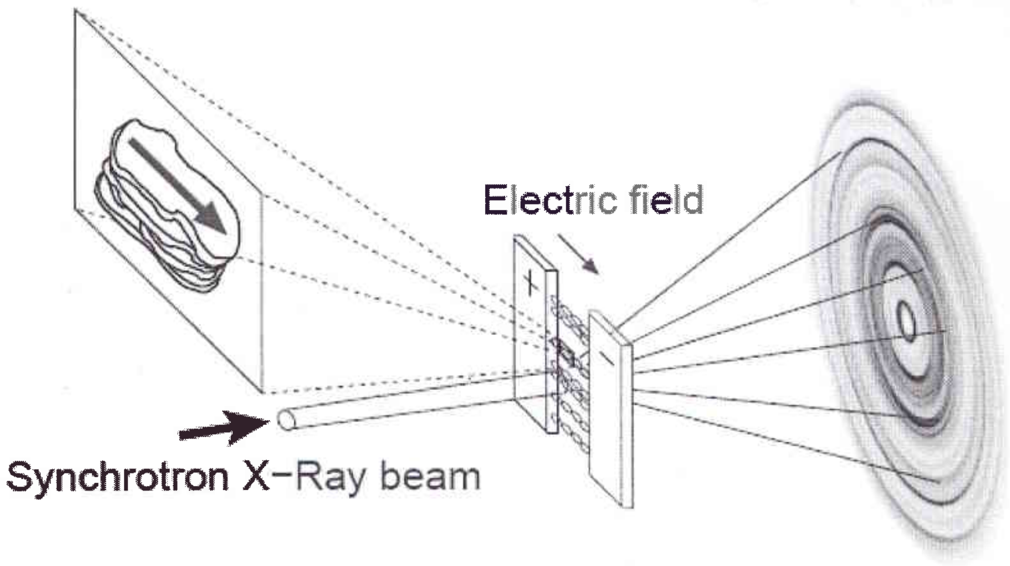




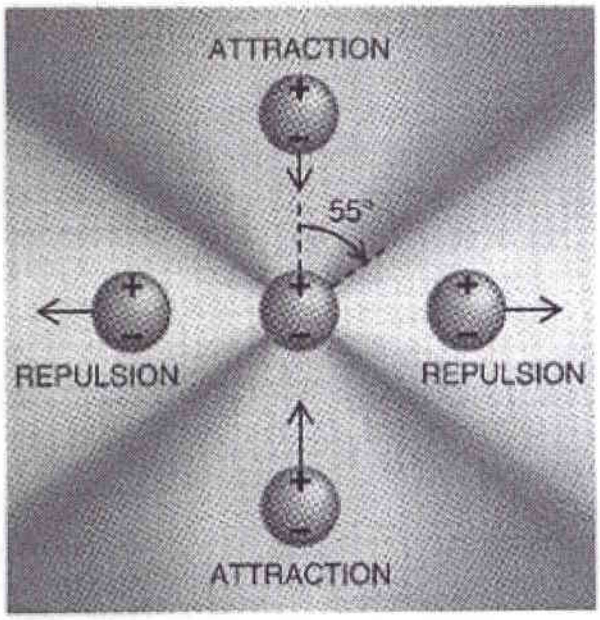
22. Ex 7

WAXS

$$2d \sin\theta = n\lambda$$



23. Ex7

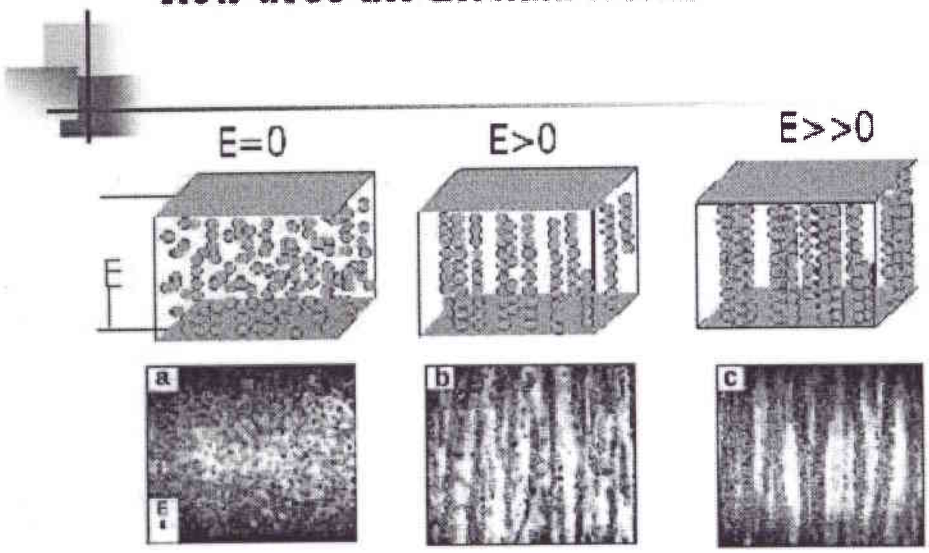


Dipolar interactions

Three-step dynamics:

How does the ER fluid work?

HKUST



Electric field E=0, the particles suspend in the liquid randomly

Electric field E>0, the particles form chains along the field direction. Viscosity of ER fluids increases

Electric field E>>0, the columns are formed. ER fluids change form liquid to solid state.

1. Polarizing particles

Chain formation

Chain bundling

APPENDIX X-ray lecture

Storage ring synchrotron radiation sources both planned and operating (October 1999).

Location	Ring (Institution)	Energy (GeV)	Internet address
Australia	Boomerang	3	—
Brazil			
Campinas	LNLS-1	1.35	—
	LNLS-2	2	—
Canada			
Saskatoon	CLS (Canadian Light Source)	2.5–2.9	cls.usask.ca/
China (PRC)			
Beijing	BEPC (Inst. High Energy Phys.)	1.5–2.8	www.friends-partners.org/~china/ins/IHEP/bsrf/bsrf.html
	BLS (Inst. High Energy Phys.)	2.2–2.5	—
Hefei	NSRL (Univ. Sci. Tech. China)	0.8	—
Shanghai	SSRF (Inst. Nucl. Res.)	3.5	—
Denmark			
Aarhus	ASTRID (ISA)	0.6	www.isa.au.dk/
	ASTRID II (ISA)	1.4	www.isa.au.dk/
England			
Daresbury	SRS (Daresbury)	2	srs.dl.ac.uk/index.htm
	DIAMOND (Daresbury/Appleton)	3.0	srs.dl.ac.uk/top/diamond.html
	SINBAD (Daresbury)	0.6	—
France			
Grenoble	ESRF	6	www.esrf.fr/
Orsay	DCI (LURE)	1.8	www.lure.u-psud.fr/
	SuperACO (LURE)	0.8	—
	SOLEIL	2.5–2.75	www.sol.cnrs-gif.fr/
Germany			
Berlin	BESSY I	0.8	www.bessy.de/
	BESSY II	1.7–1.9	www.bessy.de/BII/
Bonn	ELSA (Bonn Univ.)	1.5–3.5	www-elsa.physik.uni-bonn.de/elsahome.html
Dortmund	DELTA (Dortmund Univ.)	1.5	www.delta.uni-dortmund.de/home_e.html
Hamburg	DORIS III (HASYLAB/DESY)	4.5–5.3	www-hasylab.desy.de/
	PETRA II (HASYLAB/DESY)	7–14	www-hasylab.desy.de/
Karlsruhe	ANKA (FZK)	2.5	www.fzk.de/anka/english/welcome.html

Storage ring synchrotron radiation sources (continued).

Location	Ring (Institution)	Energy (GeV)	Internet address
India			
Indore	INDUS-I (Ctr. Adv. Tech.)	0.45	www.ee.ualberta.ca/~naik/cataccel.html
	INDUS-II (Ctr. Adv. Tech.)	2.5	www.ee.ualberta.ca/~naik/cataccel.html
Italy			
Frascati	DAFNE	0.51	www.lnf.infn.it/acceleratori/
Trieste	ELETTRA (Synch. Trieste)	1.5–2	waxa.elettra.trieste.it/ELETTRA.html
Japan			
Hiroshima	HISOR (Hiroshima Univ.)	0.7	www.hiroshima-u.ac.jp/Organization/src.html
Ichihara	Nano-hana (Japan SOR Inc.)	1.5–2	www.iijnet.or.jp/NANO-HANA/en.html
Kashiwa	VSX (Univ. of Tokyo–ISSP)	2–2.5	www.issp.u-tokyo.ac.jp/labs/sor/vsxs/index.html
Kusatsu	AURORA (Ritsumaiken Univ.)	0.6	www.ritsumei.ac.jp/se/d11/index-e.html
Kyoto	KSR (Kyoto University)	0.3	wwwal.kuicr.kyoto-u.ac.jp/www/Buttons-e.imagemap?112,64
Nishi Harima	SPring-8 (JASRI)	8	www.spring8.or.jp/
	Subaru (Himeji Inst. Tech.)	1–1.5	www.lasti.himeji-tech.ac.jp/NS/Index.html
Okasaki	UVSOR (Inst. Mol. Science)	0.75	www.ims.ac.jp/about_ims/facilities.html
	UVSOR-II (Inst. Mol. Science)	1.0	—
Sendai	TLS (Tohoku Univ.)	1.5	—
Tsukuba	TERAS (ElectroTech. Lab.)	0.8	www.etl.go.jp/etl/linac/e/
	NIJI II (ElectroTech. Lab.)	0.6	www.etl.go.jp/etl/linac/e/
	NIJI IV (ElectroTech. Lab.)	0.5	www.etl.go.jp/etl/linac/e/
	Photon Factory (KEK)	2.5–3	pfwww.kek.jp/
	Accumulator Ring (KEK)	6	pfwww.kek.jp/
Korea			
Pohang	Pohang Light Source	2	pal.postech.ac.kr/
Seoul	CESS (Seoul Nat. Univ.)	0.1	—
Middle East	SESAME	1	www.weizmann.ac.il/home/sesame/
Russia			
Moscow	Siberia I (Kurchatov Inst.)	0.45	—
	Siberia II (Kurchatov Inst.)	2.5	—
Dubna	DELSY (JINR)	0.6–1.2	—
Novosibirsk	VEPP-2M (BINP)	0.7	ssrc.inp.nsk.su/
	VEPP-3 (BINP)	2.2	ssrc.inp.nsk.su/
	VEPP-4 (BINP)	5–7	ssrc.inp.nsk.su/english/load.pl?right=vepp.html
	Siberia-SM (BINP)	0.8	ssrc.inp.nsk.su/
Zelenograd	TNK (F.V. Lukin Inst.)	1.2–1.6	—
Singapore	Helios2 (Univ. Singapore)	0.7	www.nus.edu.sg/NUSinfo/SSLS/

Storage ring synchrotron radiation sources (continued).

Location	Ring (Institution)	Energy (GeV)	Internet address
Spain			
Barcelona	Catalonia SR Lab	2.5–3	www.lls.ifae.es/report/report.html
Sweden			
Lund	MAX I (Univ. Lund)	0.55	www.maxlab.lu.se/welcome.html
	MAX II (Univ. Lund)	1.5	www.maxlab.lu.se/welcome.html
	New Ring (Univ. Lund)	0.7	—
Switzerland			
Villigen	SLS (Paul Scherrer Inst.)	2.4	www1.psi.ch/www_sls_hn/
Taiwan (ROC)			
Hsinchu	SRRC (Synch. Rad. Res. Ctr.)	1.3–1.5	www.srrc.gov.tw/en/main2000.htm
Thailand			
Nakhon Ratchasima	SIAM (Suranaree Univ. Tech.)	1.0	—
Ukraine			
Kharkov	Pulse Stretcher/Synch. Rad.	0.75–2	—
Kiev	ISI-800 (UNSC)	0.7–1.0	—
USA			
Argonne, IL	APS (Argonne Nat. Lab.)	7	epics.aps.anl.gov/welcome.html
Baton Rouge, LA	CAMD (Louisiana State Univ.)	1.4	www.camd.lsu.edu/
Berkeley, CA	ALS (Lawrence Berkeley Nat. Lab.)	1.5–1.9	www-als.lbl.gov/als/
Durham, NC	FELL (Duke Univ.)	1–1.3	monk.fel.duke.edu/
Gaithersburg, MD	SURF III (NIST)	0.4	physics.nist.gov/MajResFac/SURF/SURF.html
Ithaca, NY	CESR (CHESS/Cornell Univ.)	5.5	www.chess.cornell.edu/
Raleigh, NC	NC STAR (N. Carolina State Univ.)	2.5	—
Stanford, CA	SPEAR2 (SSRL/SLAC)	3	www-ssrl.slac.stanford.edu/welcome.html
	SPEAR3 (SSRL/SLAC)	3	www-ssrl.slac.stanford.edu/welcome.html
Stoughton, WI	Aladdin (Synch. Rad. Ctr.)	0.8–1	www.src.wisc.edu/
Upton, NY	NSLS I (Brookhaven Nat. Lab.)	0.80	www.nsls.bnl.gov/
	NSLS II (Brookhaven Nat. Lab.)	2.5–2.8	www.nsls.bnl.gov/