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Small-Angle X-ray Scattering

Basics & Applications

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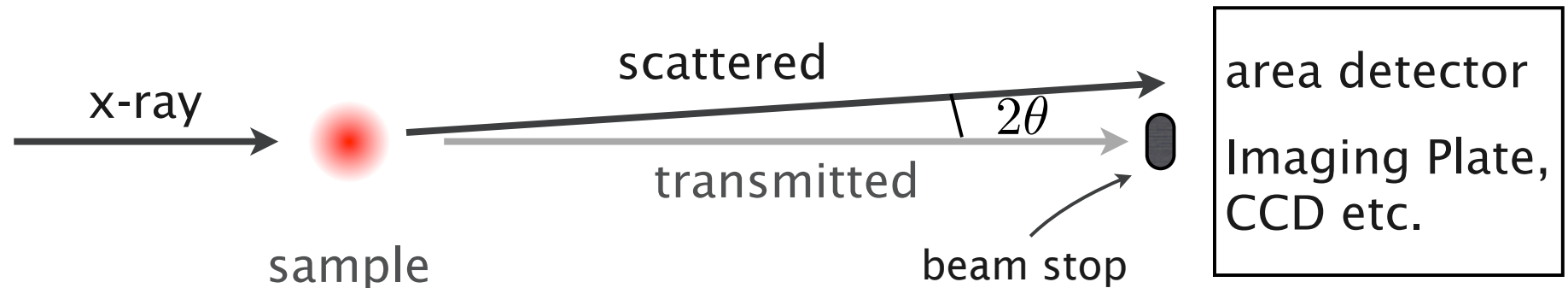
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The University of Tokyo

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What's Small-Angle X-ray Scattering ?

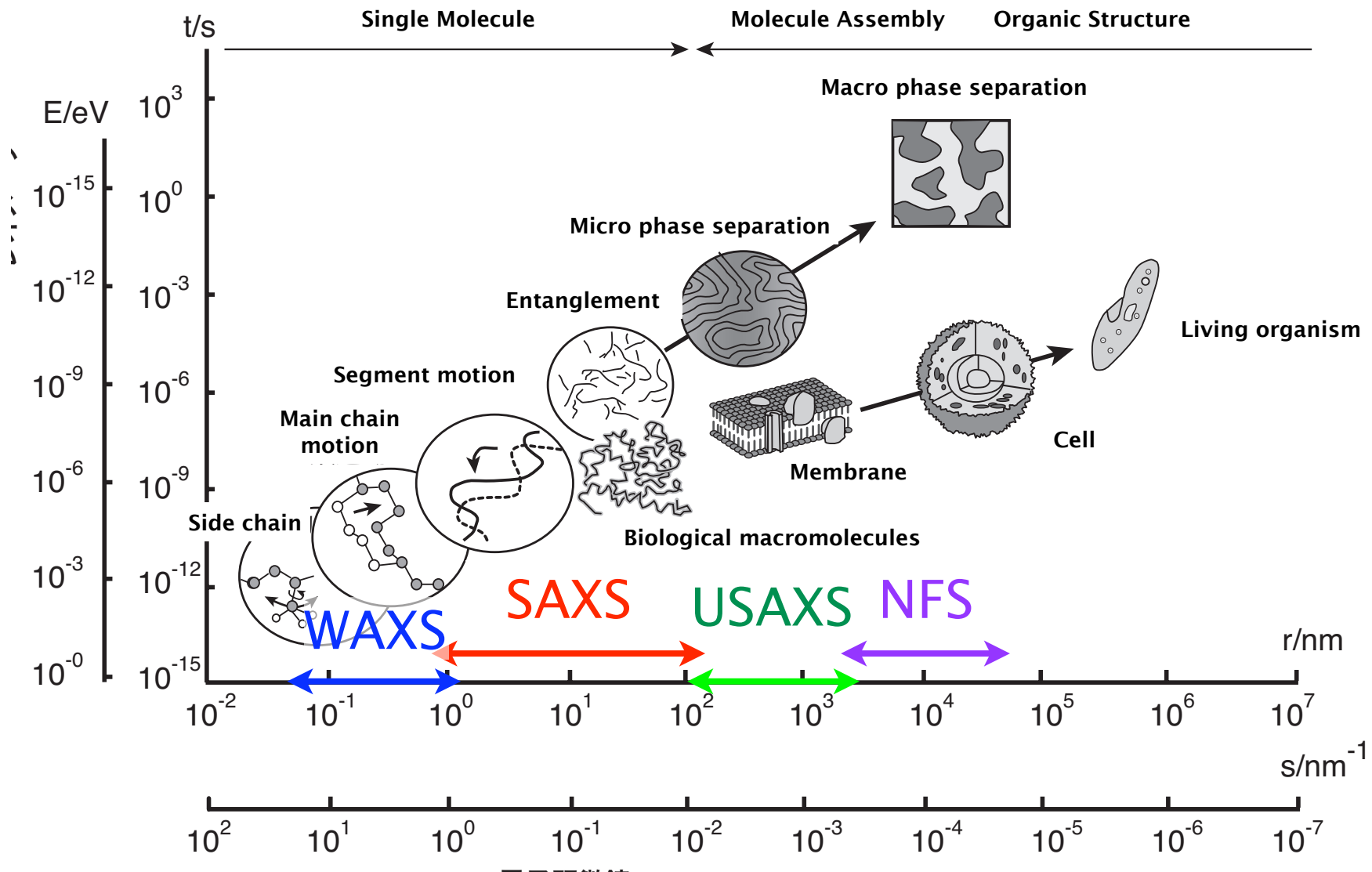


Bragg's law: $\lambda = 2d \sin \theta$

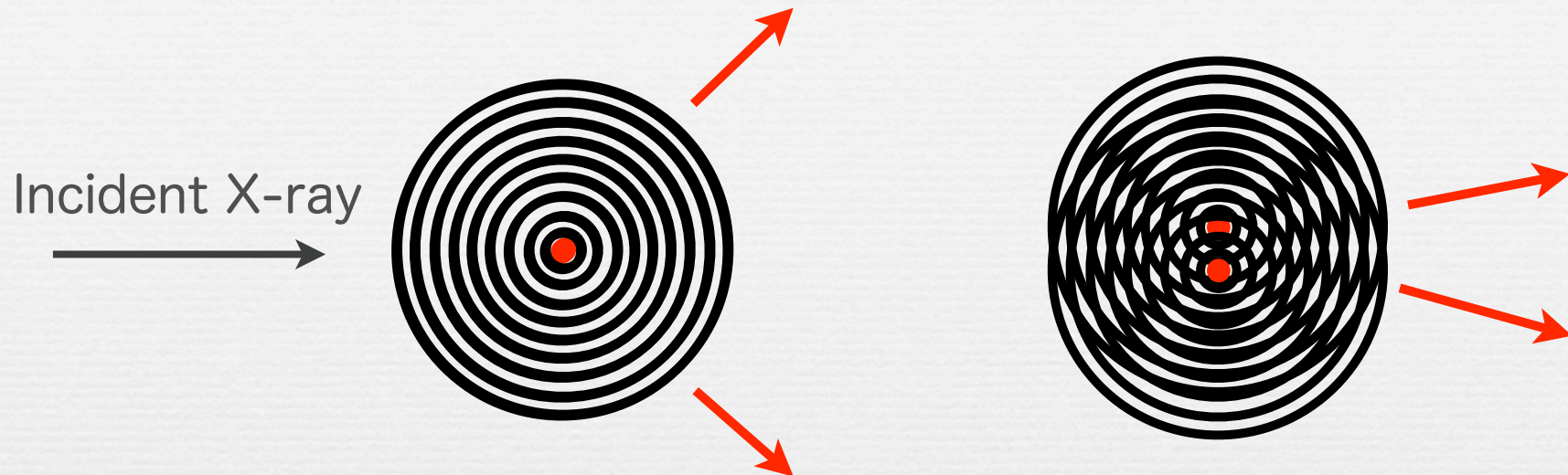
small angle \longrightarrow large structure
(1 – 100 nm)

crystalline sample --> small-angle X-ray diffraction: SAXD
solution scattering / inhomogeneous structure --> SAXS

Hierarchical Structure of Soft Matter



Interference of secondary waves



Each electron in materials vibrates and emits secondary spherical wave

When there are two electrons,

- interference between secondary waves from electrons
- when a distance between electrons is small
 - > scattering at large angle is intensified
- when a distance between electrons is large
 - > scattering at small angle is intensified --> **SAXS**



History of SAXS (< 1936)

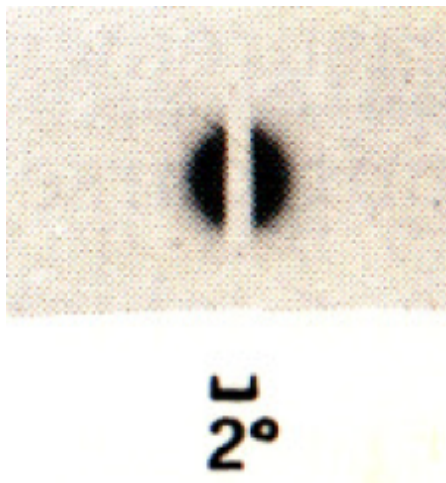
Krishnamurty (1930)

Hendricks (1932)

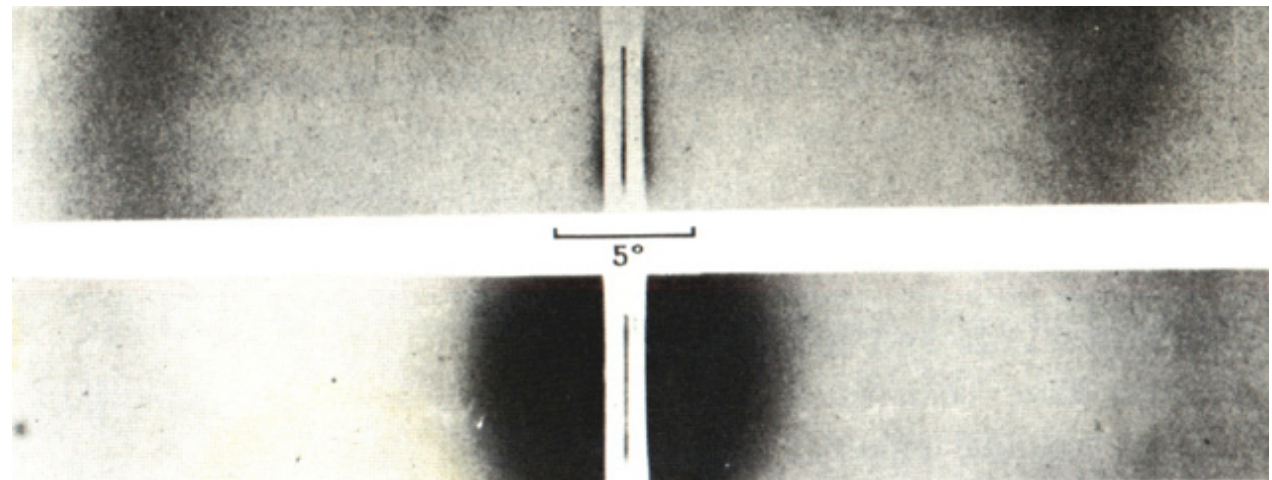
Mark (1932)

Warren (1936)

Observation of scattering
from powders, fibers, and colloidal
dispersions

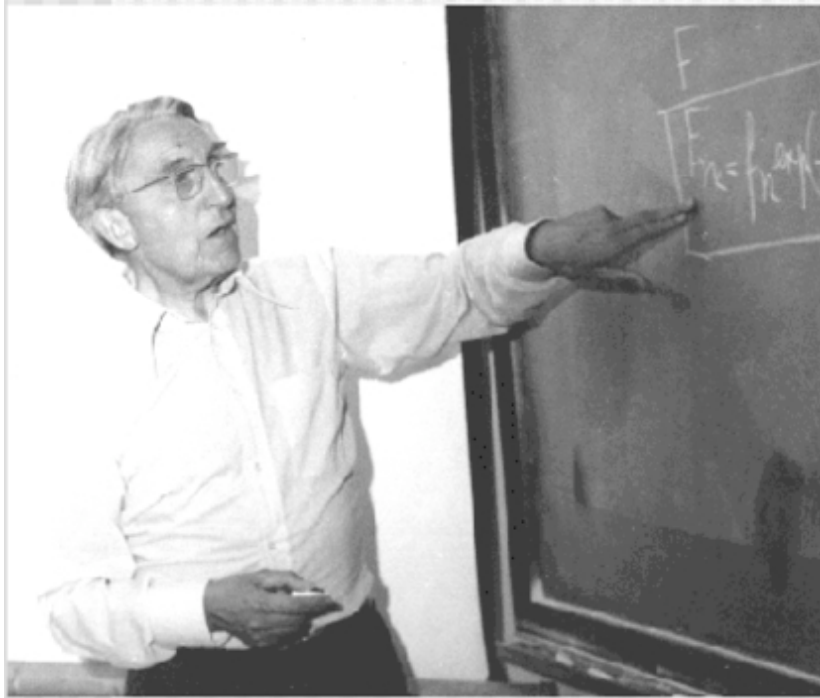


carbon black



Molten silica - silica gel
(above) (below)

History (> 1936)



[A. Guinier](#) (1937, 1939, 1943)

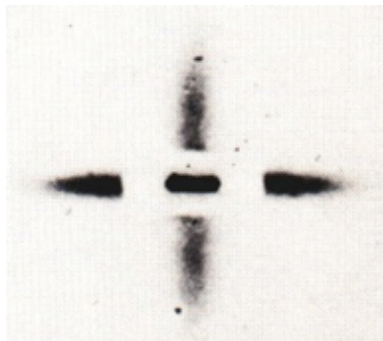
Interpretation of inhomogeneities in Al alloys “G-P zones”, introducing the concept of “particle scattering” and formalism necessary to solve the problem of a diluted system of particles.

[O. Kratky](#) (1938, 1942, 1962)

[G. Porod](#) (1942, 1960, 1961)

Description of dense systems of colloidal particles, micelles, and fibers.

Macromolecules in solution.



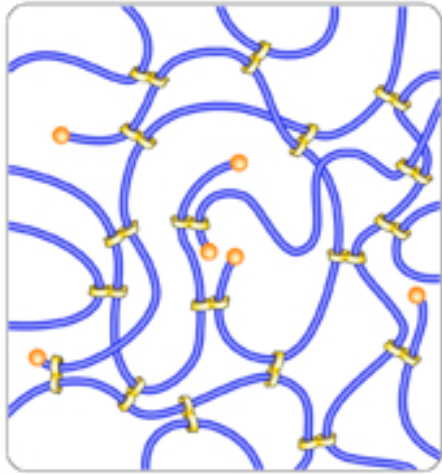
Single crystals of Al-Cu hardened alloy



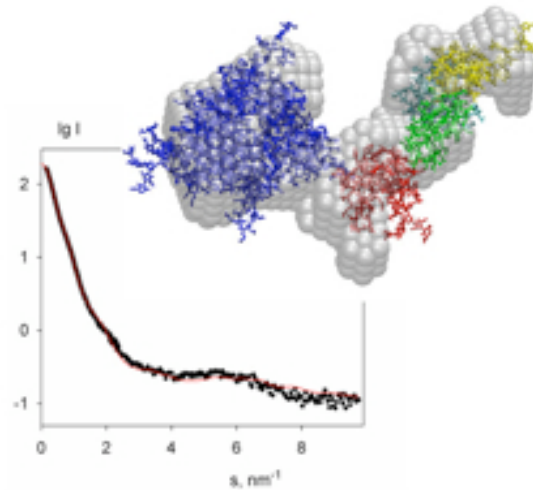
Hemoglobin

courtesy to Dr. I.L.Torriani

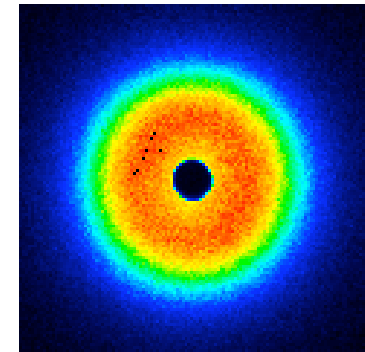
Application of SAXS



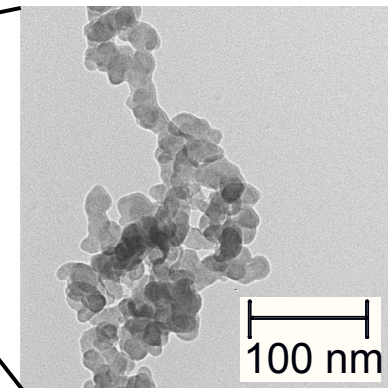
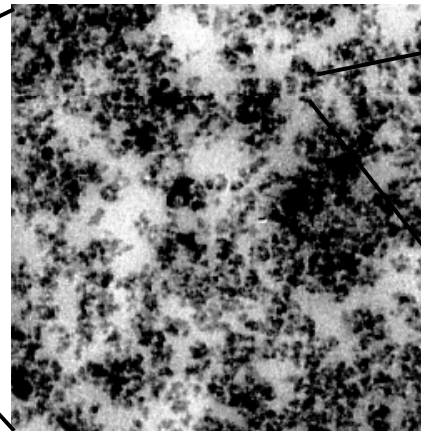
gel



Proteins in solution (Dr. Svergun, EMBL)



Typical SAXS image

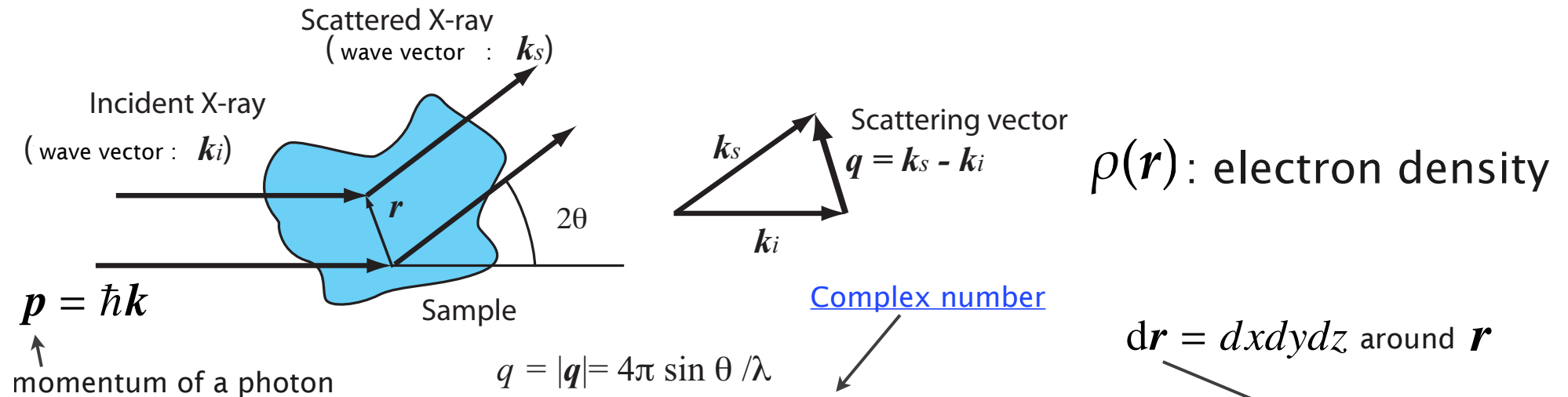


Nanocomposite

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Basic of X-ray scattering



Amplitude of scattered X-ray

$$A(\mathbf{q}) = \int_V \rho(\mathbf{r}) \exp(-i\mathbf{q} \cdot \mathbf{r}) d\mathbf{r}$$

Fourier transform of electron density

Intensity of scattered X-ray : $I(\mathbf{q}) = A(\mathbf{q})A^*(\mathbf{q}) = |A(\mathbf{q})|^2$

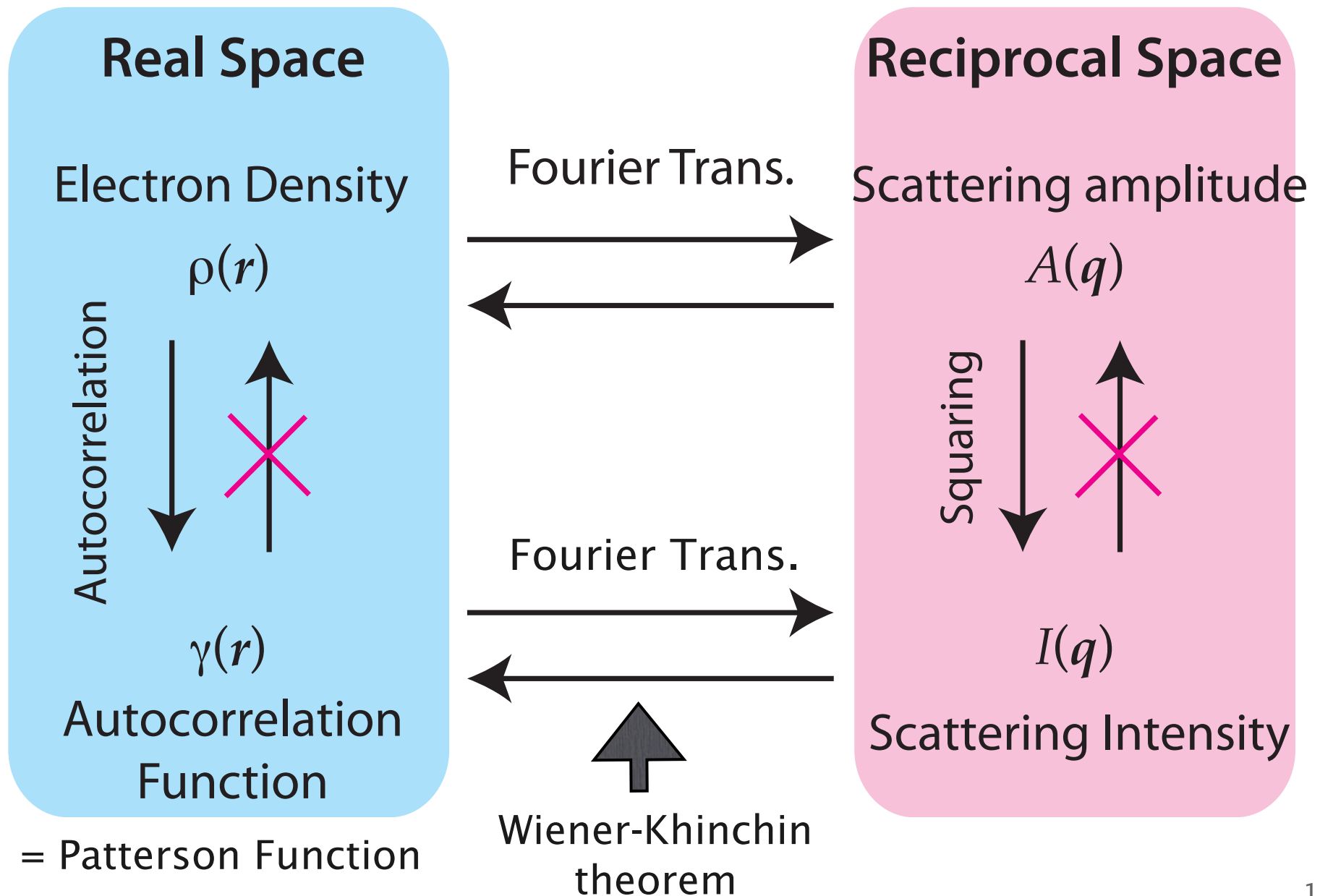
(Extensive variable)

Intensity of scattered X-ray per volume: $I(\mathbf{q}) = \frac{A(\mathbf{q})A^*(\mathbf{q})}{V} = \frac{|A(\mathbf{q})|^2}{V}$

(Intensive variable)

This doesn't depend on sample volume, and
is used to obtain the absolute intensity

Real space and Reciprocal Space

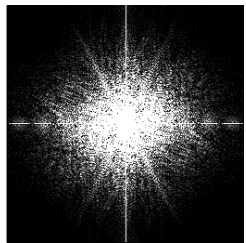


Importance of phase, $\theta(\mathbf{q})$ of complex amplitude

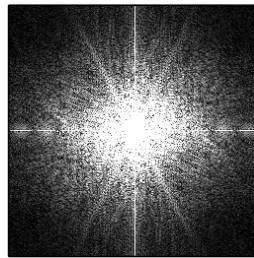
$$A(\mathbf{q}) = |A(\mathbf{q})| \exp(-i\theta(\mathbf{q}))$$



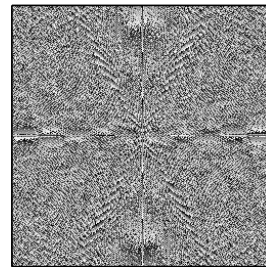
$\rho_1(\mathbf{r})$



$$I_1(\mathbf{q}) = |A_1(\mathbf{q})|^2$$



$|A_1(\mathbf{q})|$

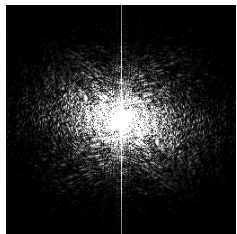


$\theta_1(\mathbf{q})$

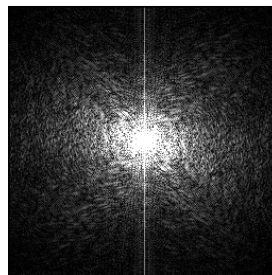
IFT



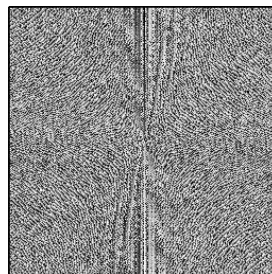
$\rho_2(\mathbf{r})$



$$I_2(\mathbf{q}) = |A_2(\mathbf{q})|^2$$



$|A_2(\mathbf{q})|$



$\theta_2(\mathbf{q})$

IFT

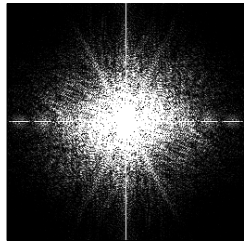


Importance of phase, $\theta(q)$ of complex amplitude

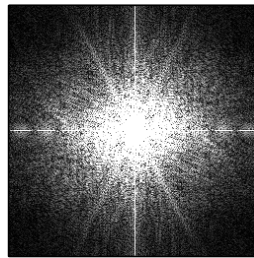
$$A(q) = |A(q)| \exp(-i\theta(q))$$



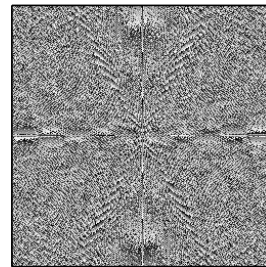
$\rho_1(\mathbf{r})$



$I_1(q) = |A_1(q)|^2$



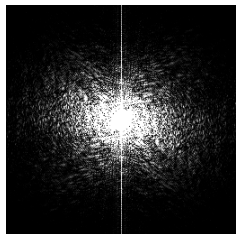
$|A_1(q)|$



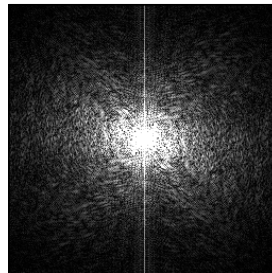
$\theta_1(q)$



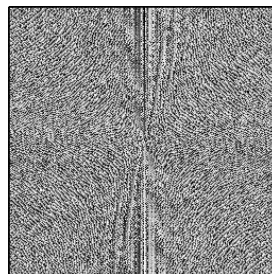
$\rho_2(\mathbf{r})$



$I_2(q) = |A_2(q)|^2$



$|A_2(q)|$



$\theta_2(q)$

IFT



IFT



Autocorrelation Function & Scattering Intensity

Autocorrelation function of electron density

$$\gamma(\mathbf{r}) = \frac{1}{V} \int_V \rho(\mathbf{r}') \rho(\mathbf{r} + \mathbf{r}') d\mathbf{r}' = \frac{1}{V} \underline{P(\mathbf{r})}$$

Patterson Function

(Debye & Bueche 1949)

asymptotic behavior of the autocorrelation function

$$\gamma(0) = \langle \rho^2 \rangle \qquad \gamma(\infty) = \langle \rho \rangle^2$$

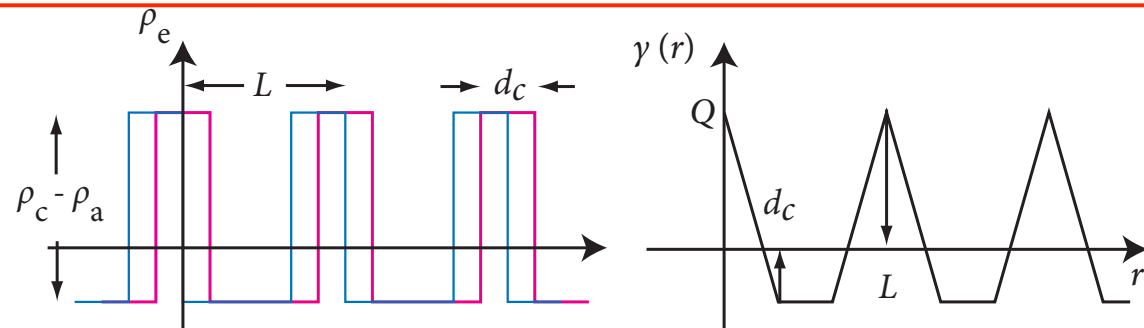
Scattering Intensity : Fourier Transform of autocorrelation function

$$I(\mathbf{q}) = \int_V \gamma(\mathbf{r}) \exp(-i\mathbf{q} \cdot \mathbf{r}) d\mathbf{r}$$

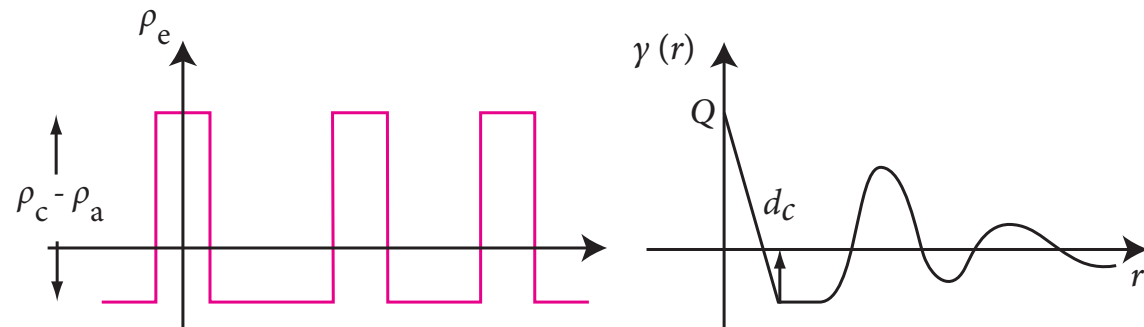
cf. Wiener-Khinchin theorem

Example of $\rho(r)$ & $\gamma(r)$: in case of lamellar

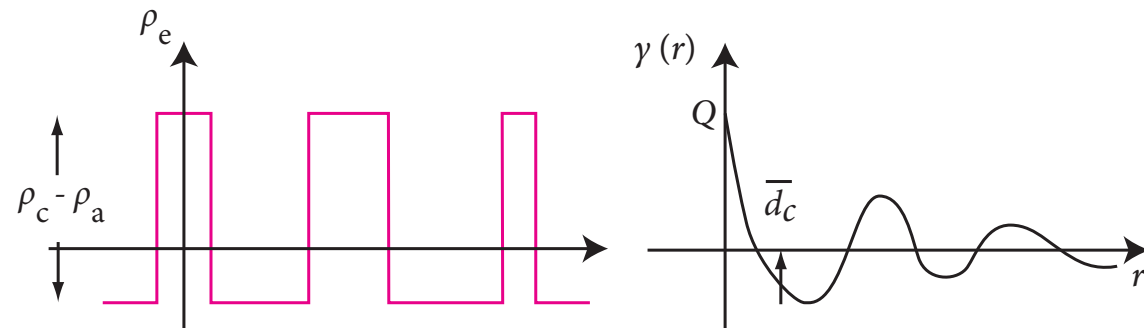
ideal ordering



Long period changes.



Thickness of crystal changes.



real space

autocorrelation

Normalized Autocorrelation Function:

Introducing the relative electron density, $\eta(\mathbf{r})$, as

$$\eta(\mathbf{r}) = \rho(\mathbf{r}) - \langle \rho \rangle \quad \longrightarrow \quad \langle \eta^2 \rangle = \langle (\rho(\mathbf{r}) - \langle \rho \rangle)^2 \rangle = \langle \rho^2 \rangle - \langle \rho \rangle^2$$

Introducing the normalized autocorrelation function, $\gamma_0(\mathbf{r})$, as

$$\gamma_0(\mathbf{r}) = \frac{1}{\langle \eta^2 \rangle} \frac{1}{V} \int_V \langle \eta(\mathbf{r}') \eta(\mathbf{r} + \mathbf{r}') \rangle d\mathbf{r}'$$


then, $\gamma_0(\mathbf{r}) = \frac{\rho(\mathbf{r}) - \langle \rho \rangle^2}{\langle \eta^2 \rangle}$ where $\gamma_0(0) = 1$ $\gamma_0(\infty) = 0$

then,
$$I(\mathbf{q}) = \underbrace{\langle \eta^2 \rangle}_{\text{observable}} \int_V \gamma_0(\mathbf{r}) e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} + \underbrace{\langle \rho \rangle^2}_{\text{Not observable}} \delta(\mathbf{q})$$

The average of the relative electron density fluctuations determines the magnitude of $I(\mathbf{q})$. The normalized autocorrelation function $\gamma_0(\mathbf{r})$ determines the shape of $I(\mathbf{q})$.

Not observable.

Invariant Q

Invariant: $Q = \int_0^\infty I(\mathbf{q}) d\mathbf{q} \Rightarrow \int_0^\infty I(q) 4\pi q^2 dq$
 (when isotropic)
 $= (2\pi)^3 \langle \eta^2 \rangle$  It doesn't depend on the structure

$(\because) Q = \int_0^\infty I(\mathbf{q}) d\mathbf{q} = \langle \eta^2 \rangle \int_0^\infty \int_V \gamma_0(\mathbf{r}) e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} d\mathbf{q}$
 $= \langle \eta^2 \rangle \int_V \gamma_0(\mathbf{r}) \int_0^\infty e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{q} d\mathbf{r}$
 $= (2\pi)^3 \langle \eta^2 \rangle \int_V \gamma_0(\mathbf{r}) \delta(0) d\mathbf{r} = (2\pi)^3 \langle \eta^2 \rangle \gamma_0(0) = (2\pi)^3 \langle \eta^2 \rangle$

$I(\mathbf{q}) = \langle \eta^2 \rangle \int_V \gamma_0(\mathbf{r}) e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} + \frac{\langle \rho \rangle^2 \delta(\mathbf{q})}{\text{Omitted.}}$

Parseval's theorem

Fourier Trans.

$$A(\mathbf{q}) \longleftrightarrow \eta(\mathbf{r})$$

$$\int |A(\mathbf{q})|^2 d\mathbf{q} = (2\pi)^3 \int |\eta(\mathbf{r})|^2 d\mathbf{r}$$

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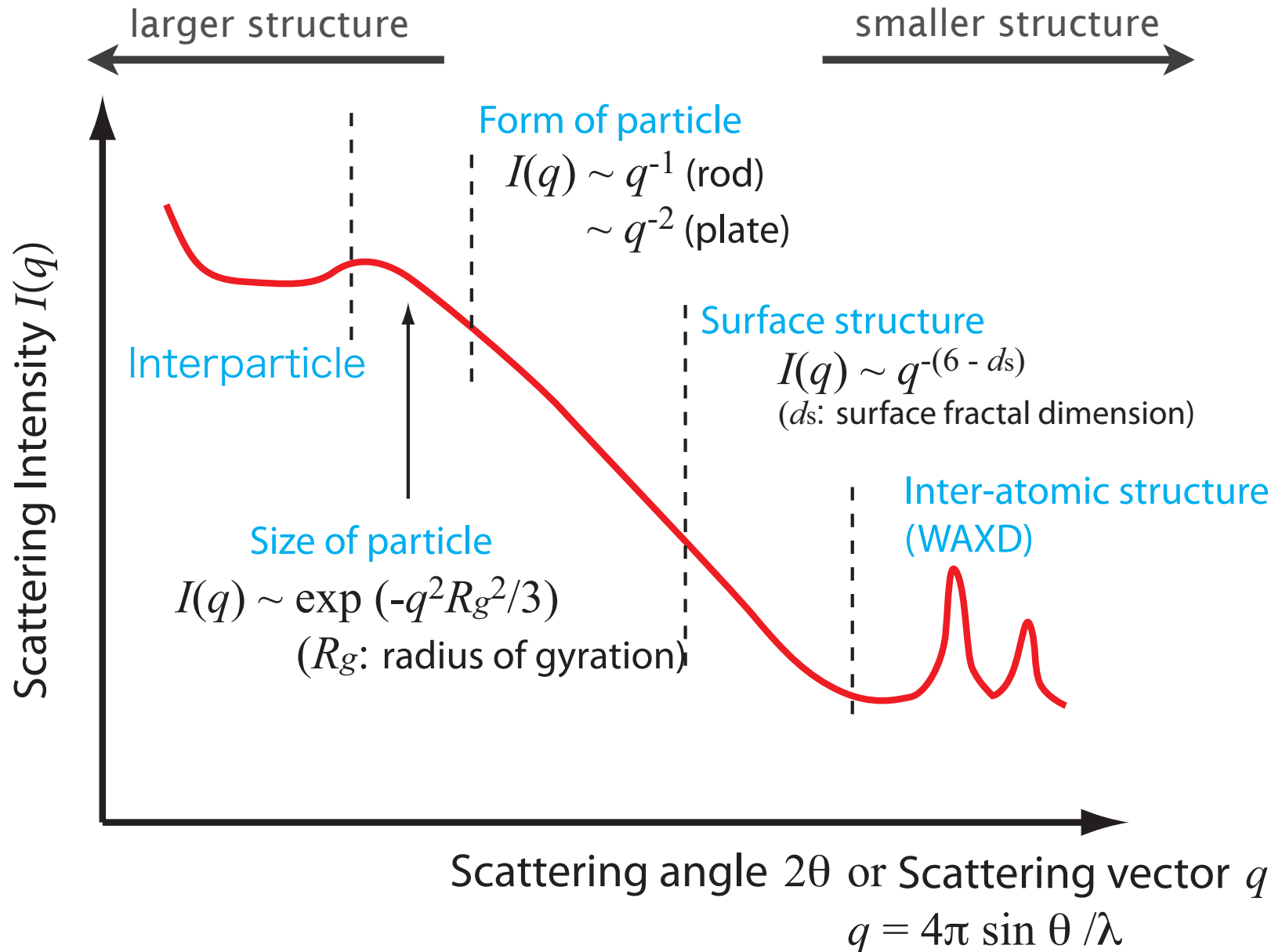
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Information obtained from SAXS

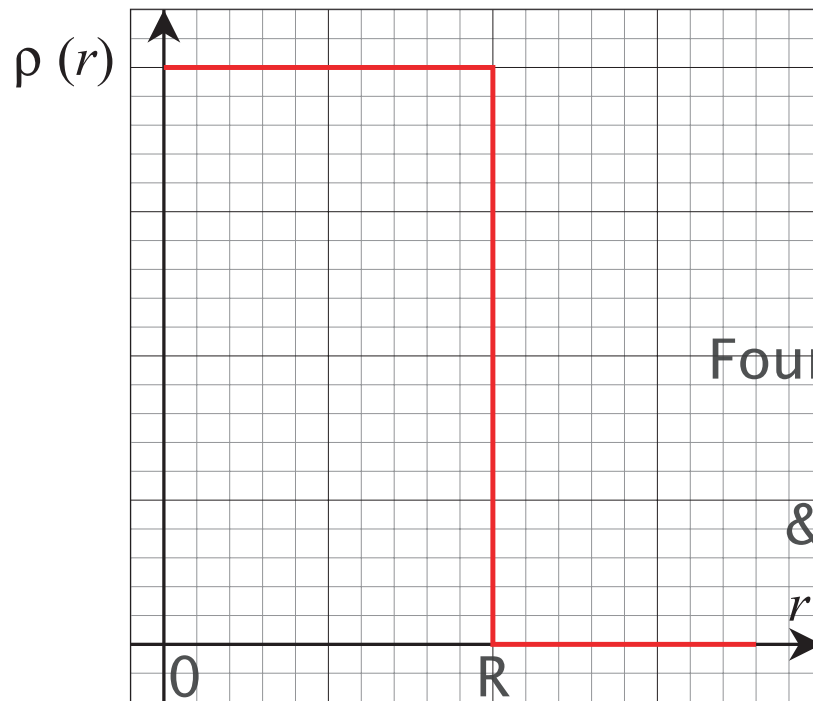
1. Size and form of particulate system
 - ✿ Colloids, Globular proteins, etc...
2. Correlation length of inhomogeneous structure
 - ✿ Polymer chain, two-phase system etc.
3. Lattice parameters of distorted crystals (para-crystal)
4. Degree of crystallinity, crystal size, crystal distortion
 - ✿ Crystalline polymer

SAXS of particulate system

Relation between SAXS pattern and Structural information



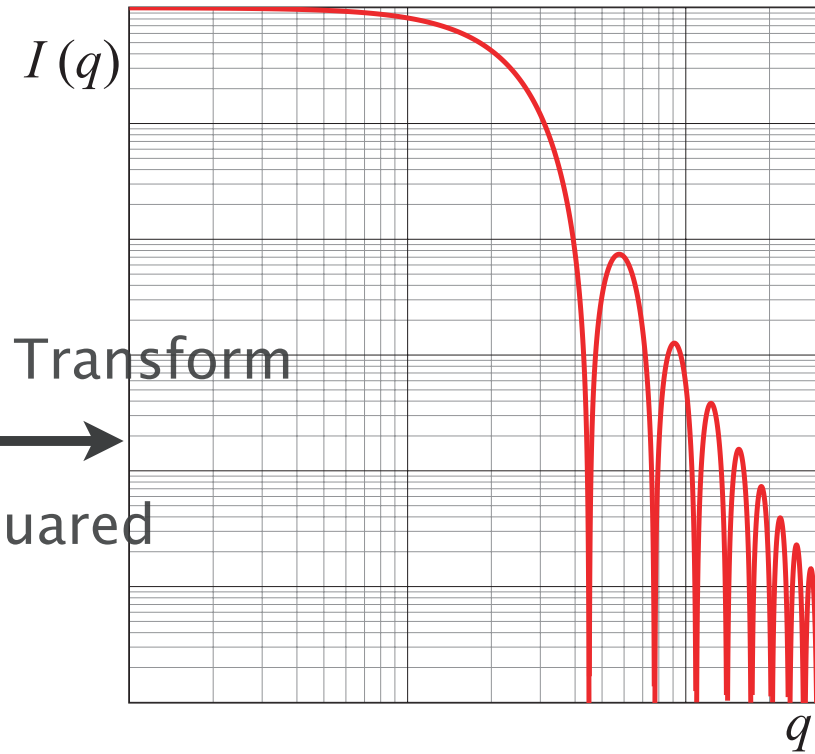
Spherical sample



Fourier Transform



& squared

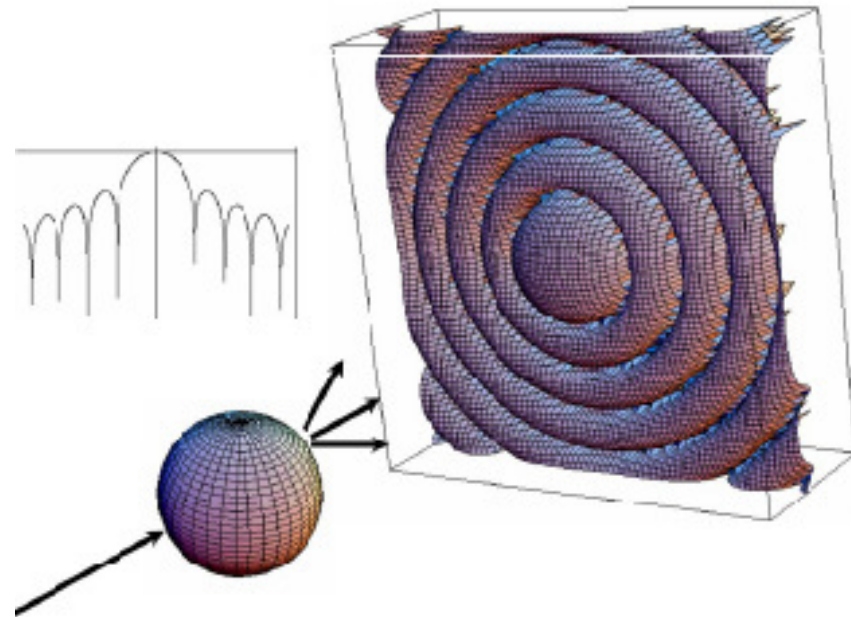
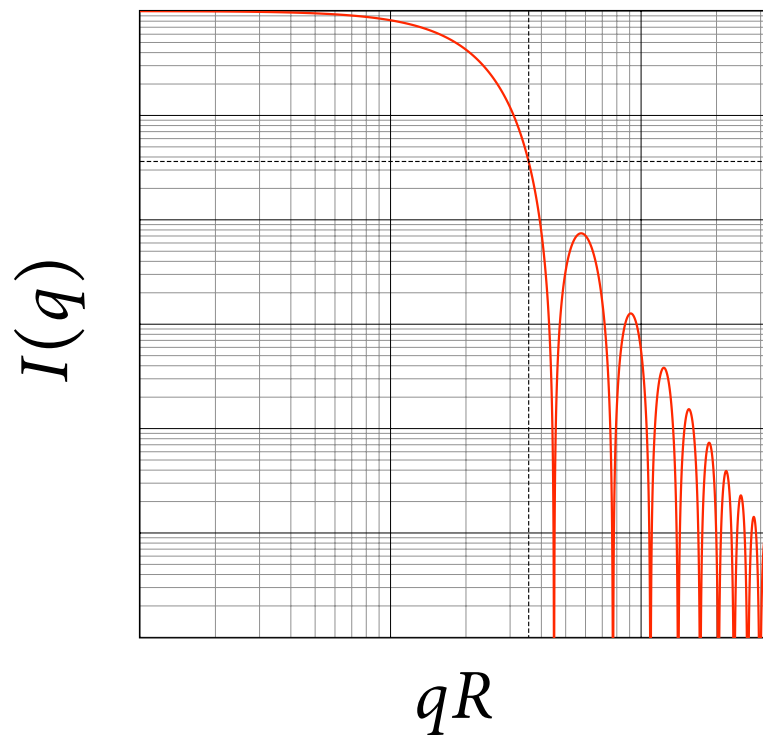


$$\rho(r) = \begin{cases} \Delta\rho & r < R \\ 0 & \text{else} \end{cases}$$

$$I(q) = \frac{(\Delta\rho)^2 V_{\text{particle}}^2}{V} \left[3 \frac{\sin qR - qR \cos qR}{(qR)^3} \right]^2$$

Homogeneous sphere

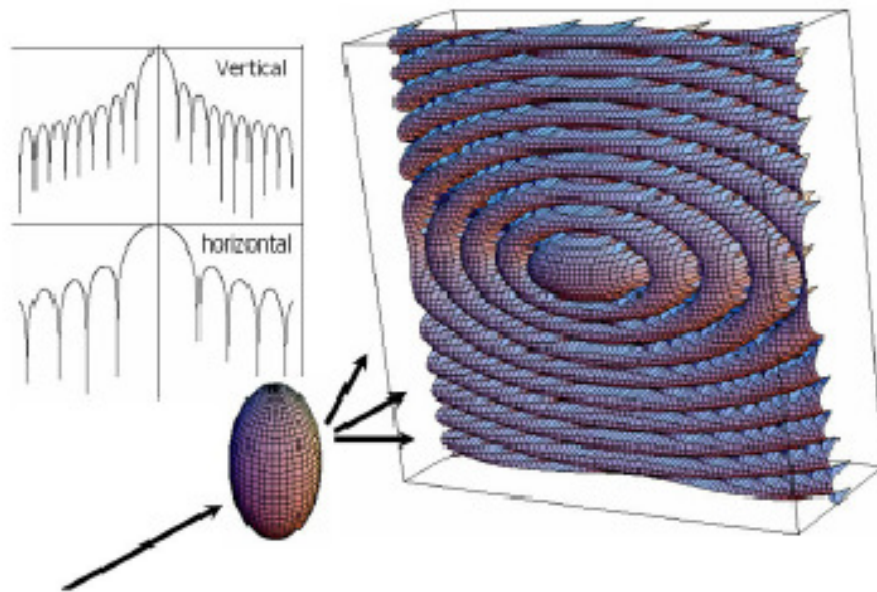
$$I(q) = \frac{(\Delta\rho)^2 V_{\text{particle}}^2}{V} \left[3 \frac{\sin qR - qR \cos qR}{(qR)^3} \right]^2$$



isotropic scattering

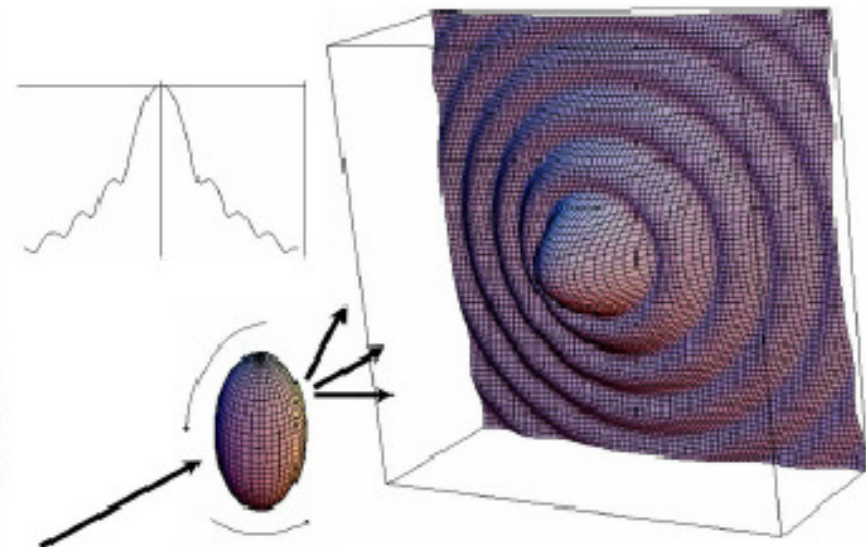
Homogeneous elipsoid

Fixed particle



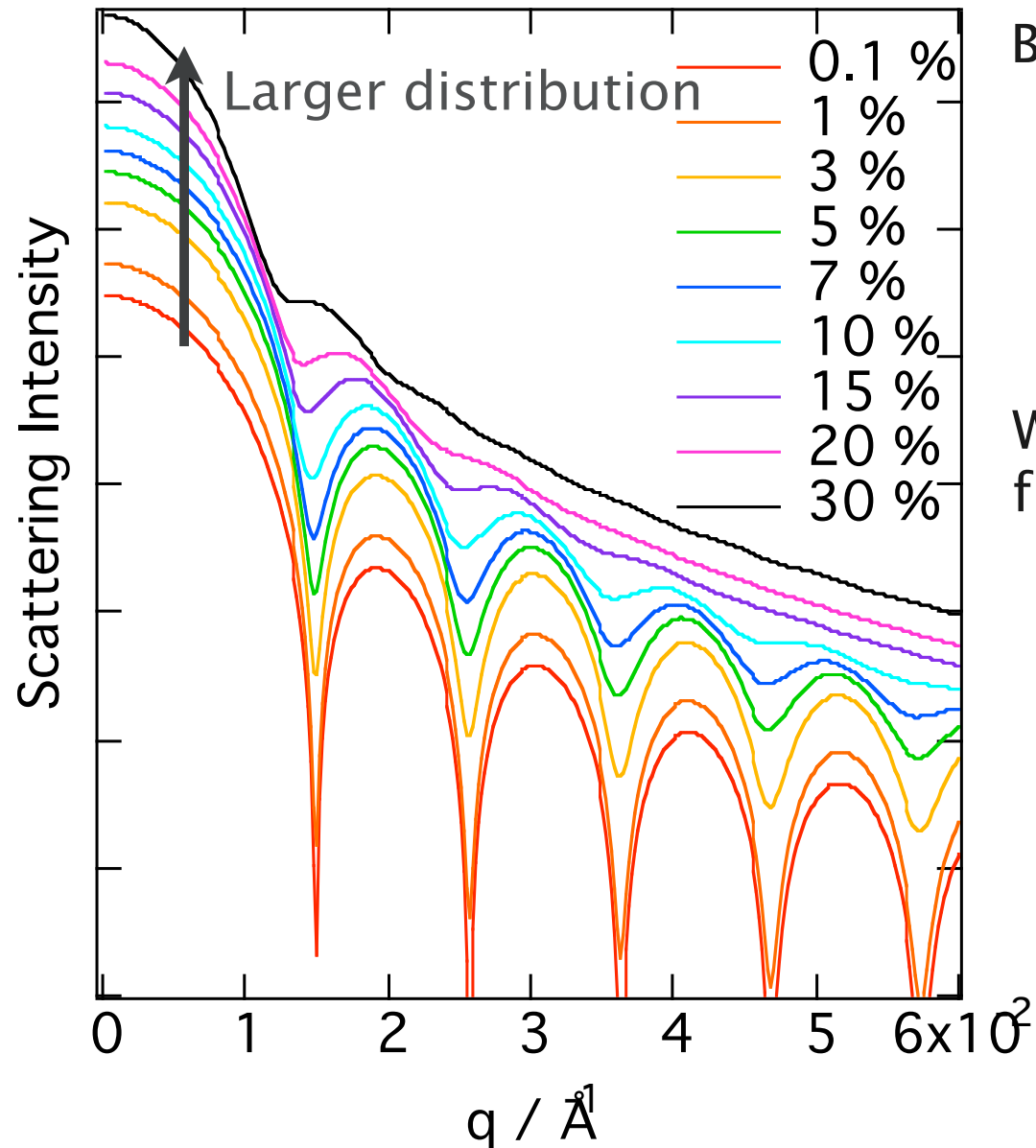
anisotropic scattering

Random orientation



isotropic scattering

Size distribution



Based on Gaussian distribution

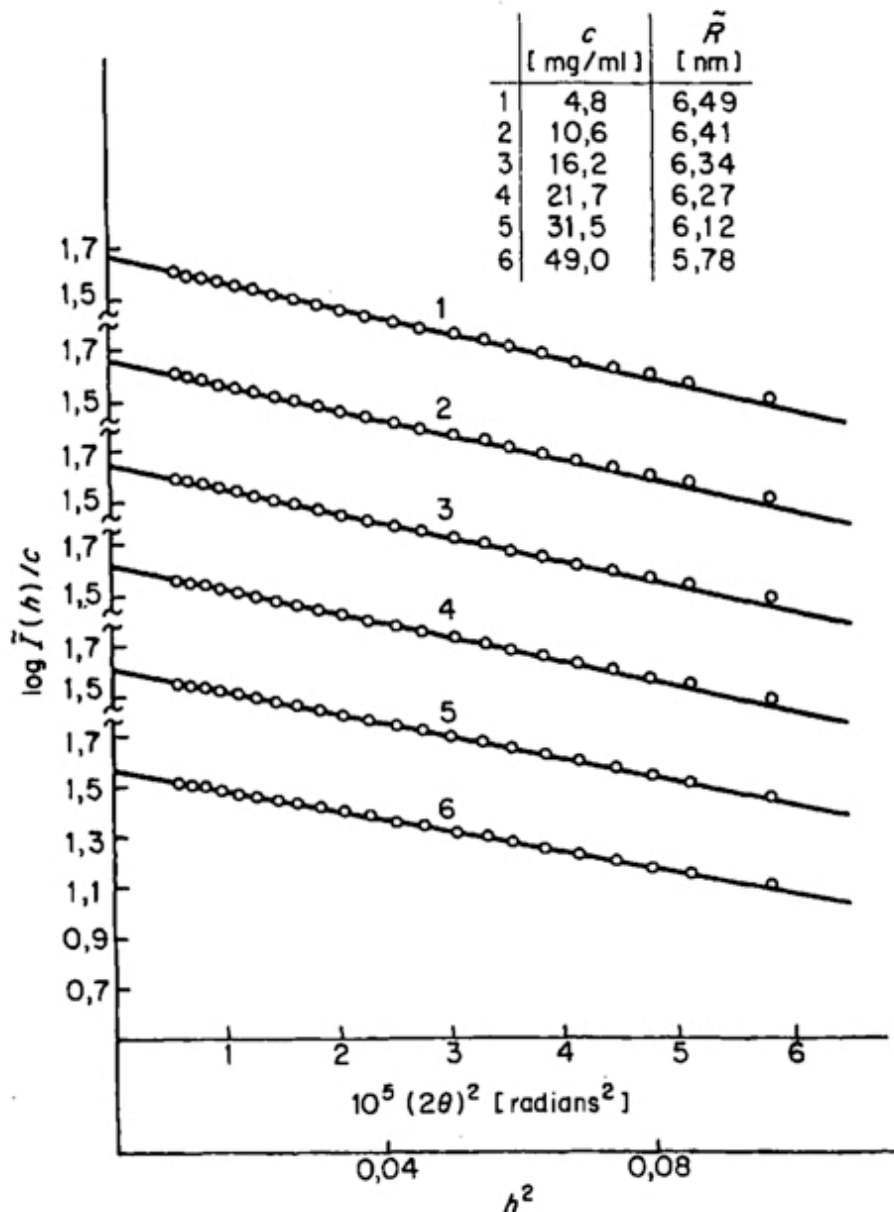
When the form has distribution, fringes are missed.

Radius of Gyration: R_g ($R_g^2 = \frac{\int r^2 \rho(r) dr}{\int \rho(r) dr}$) ----- Guinier Plot

$$I(q) \sim \exp\left(-\frac{q^2 R_g^2}{3}\right)$$

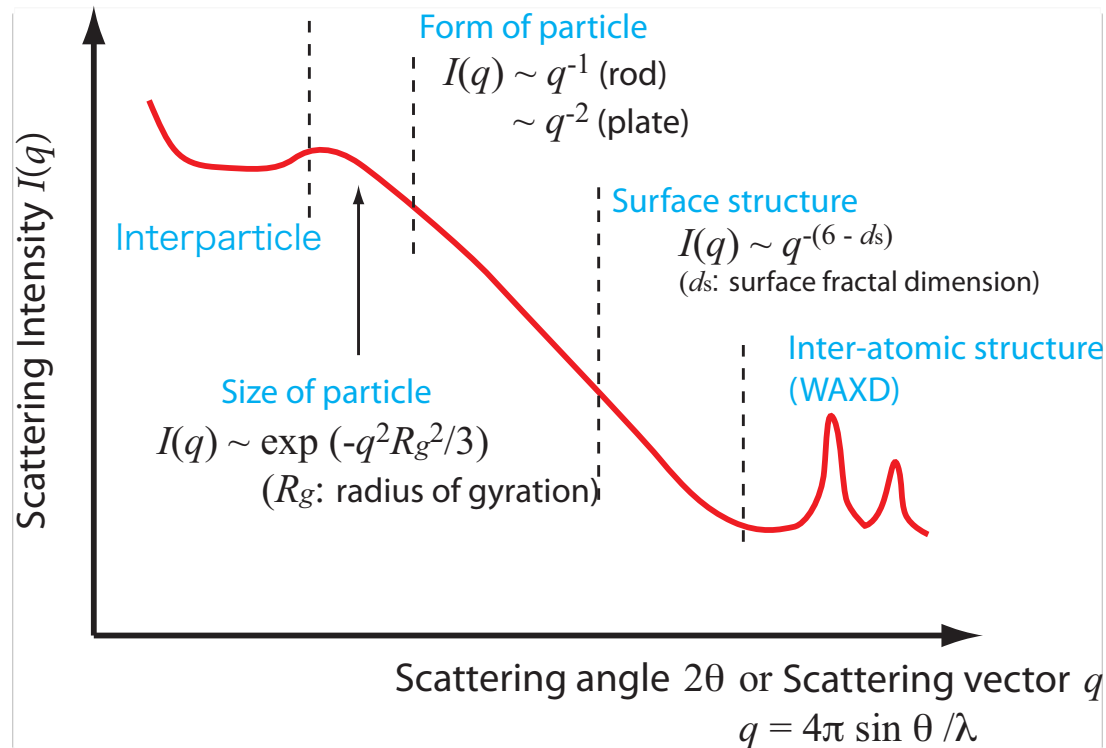
$$\log(I(q)) = -\frac{q^2 R_g^2}{3}$$

Guinier plot: $\log(I(q))$ vs q^2



O. Glatter & O. Kratky ed., "Small Angle X-ray Scattering", Academic Press (1982).

Structure Factor & Form Factor



$$I(q) = \phi V_{\text{particle}} \underline{S(q)} \underline{F(q)}$$

Structure Factor Form Factor

↓ ↓

inter-particle structure intra-particle structure

Separation of $S(q)$ & $F(q)$

→ Everlasting issue

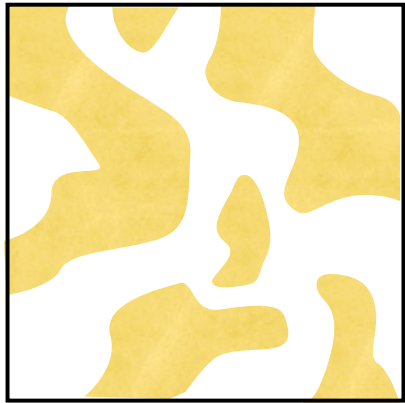
(especially, for non-crystalline sample)

Proposed remedy:

- GIFT (Generalized Inverse Fourier Trans.) by O. Glatter

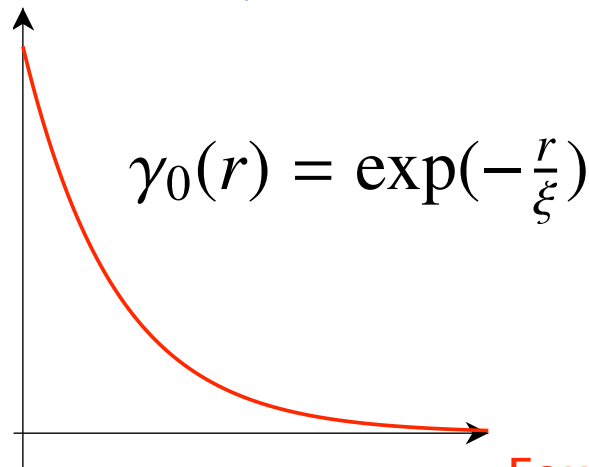
Scattering from Inhomogeneous Structure

Electron Density
 $\rho(r)$

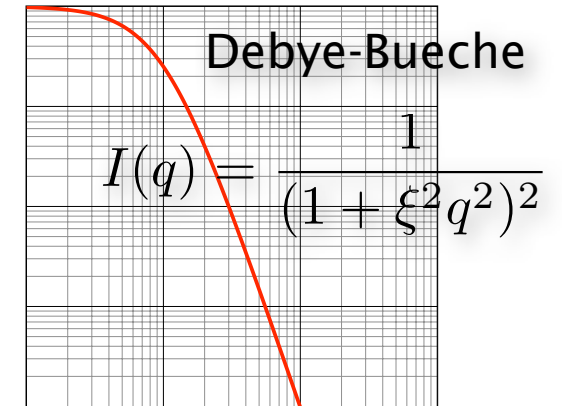


two phase system

Autocorrelation Function
 $\gamma_0(r)$



Scattering Intensity



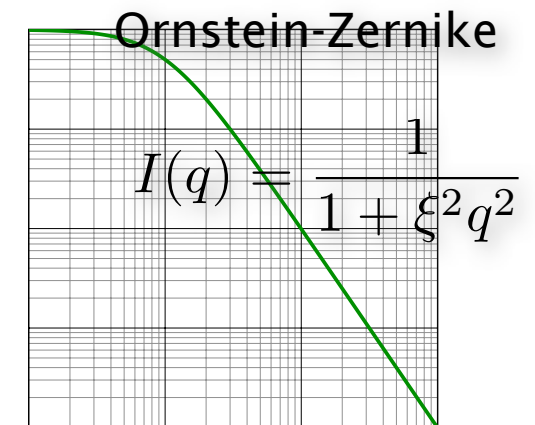
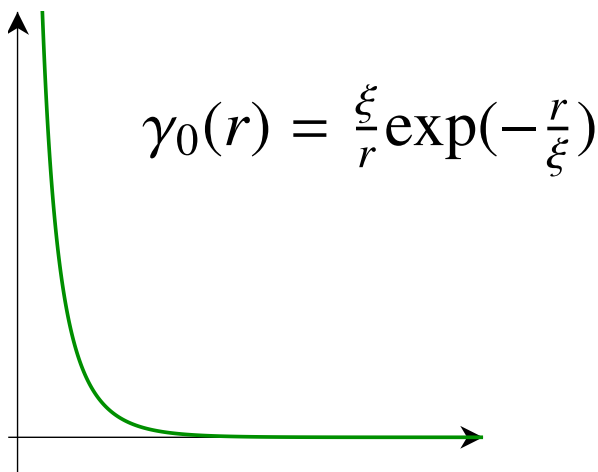
Autocorrelation



Fourier trans.



polymer chain etc.

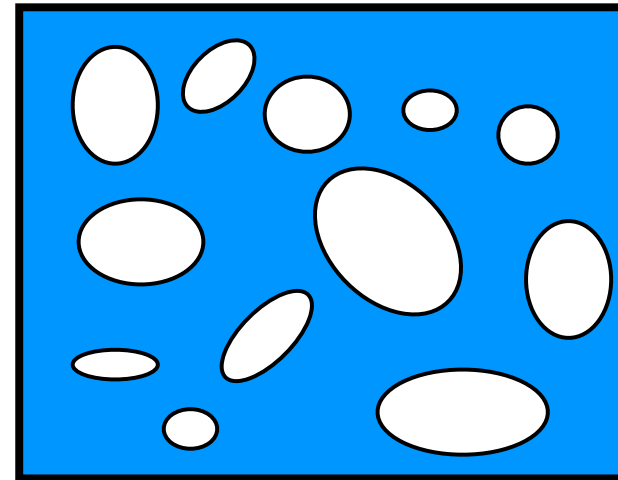
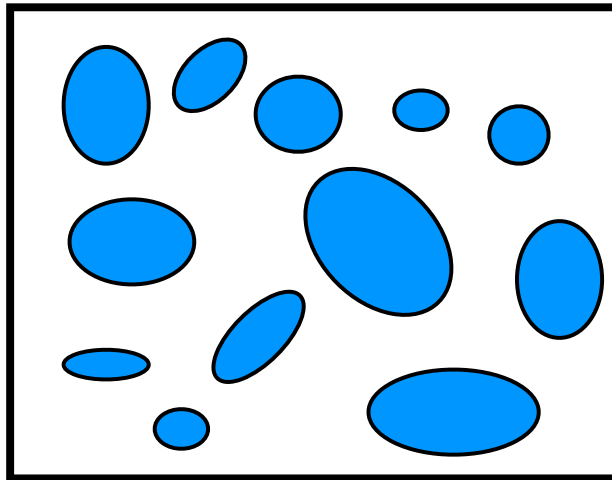


Two-phase system

Phase 1: ρ_1 , volume fraction ϕ Phase 2: ρ_2 volume fraction $1 - \phi$

$$\begin{aligned} A(\mathbf{q}) &= \int_{\phi V} \rho_1 e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} + \int_{(1-\phi)V} \rho_2 e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} \\ &= \int_{\phi V} (\rho_1 - \rho_2) e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} + \rho_2 \int_V e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} \end{aligned}$$

$$A(\mathbf{q}) = \int_V \Delta\rho e^{-i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} + \rho_2 \delta(\mathbf{q})$$



Babinet's principle

Two complementary structures produce the same scattering.

Two-phase system -- cont.

Averaged square fluctuation of electron density

$$\langle \eta^2 \rangle = \phi(1 - \phi)(\Delta\rho)^2 \quad \text{where} \quad \Delta\rho = \rho_1 - \rho_2$$

ϕ : volume fraction

$$I(q) = 4\pi\langle \eta^2 \rangle \int_0^\infty \gamma_0(r) \frac{\sin(qr)}{qr} r^2 dr$$

$$I(q) = 4\pi\phi(1 - \phi)(\Delta\rho)^2 \int_0^\infty \gamma_0(r) \frac{\sin(qr)}{qr} r^2 dr$$

$$Q = \int_0^\infty I(q) q^2 dq = 2\pi^2 \phi(1 - \phi)(\Delta\rho)^2$$

Invariant: does not depend on the structure of the two phases but only on the **volume fractions** and **the contrast between the two phases**.

Porod's law

For a sharp interface, the scattered intensity decreases as q^{-4} .

$$I(q) \rightarrow (\Delta\rho)^2 \frac{2\pi}{q^4} S/V$$

internal surface area

Combination of Porod's law & Invariant Q

$$\pi \cdot \frac{\lim_{q \rightarrow \infty} I(q) q^4}{Q} = \boxed{\frac{S}{V}}$$

surface-volume ratio

important for the characterization of porous materials

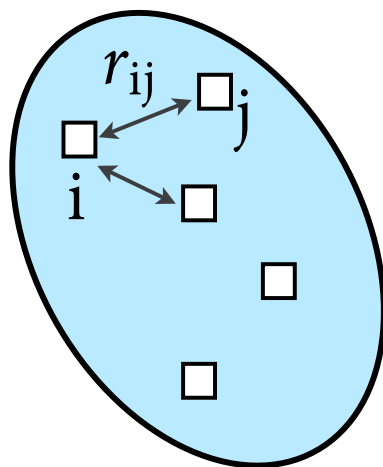
Pair Distance Distribution Function: PDDF

Scattering intensity:
(when isotropic) $I(q) = 4\pi \int_0^\infty \gamma_0(r) \frac{\sin(qr)}{qr} r^2 dr$

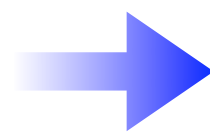
$$\text{PDDF : } p(r) = r^2 \gamma_0(r)$$

the set of distances joining the volume elements within a particle,
including the case of non-uniform density distribution.

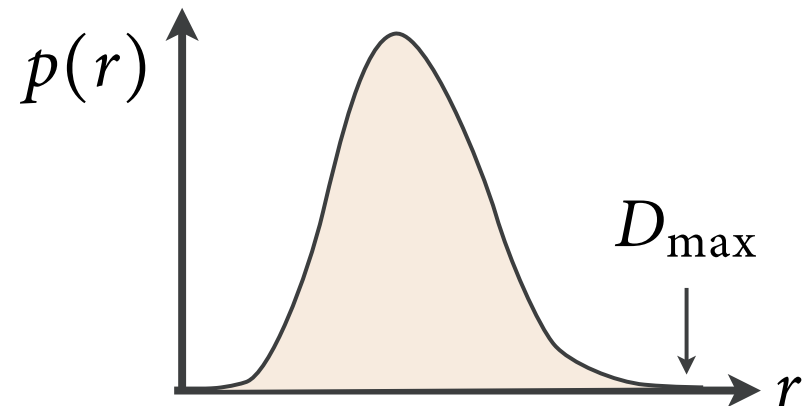
Particle's **SHAPE** and maximum **DIMENSION**.



pairs of volume
elements i-j

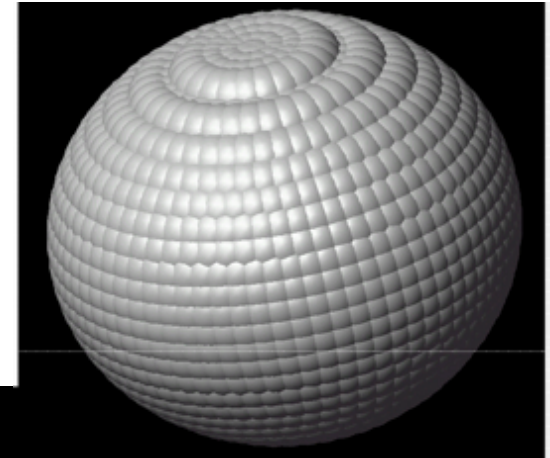
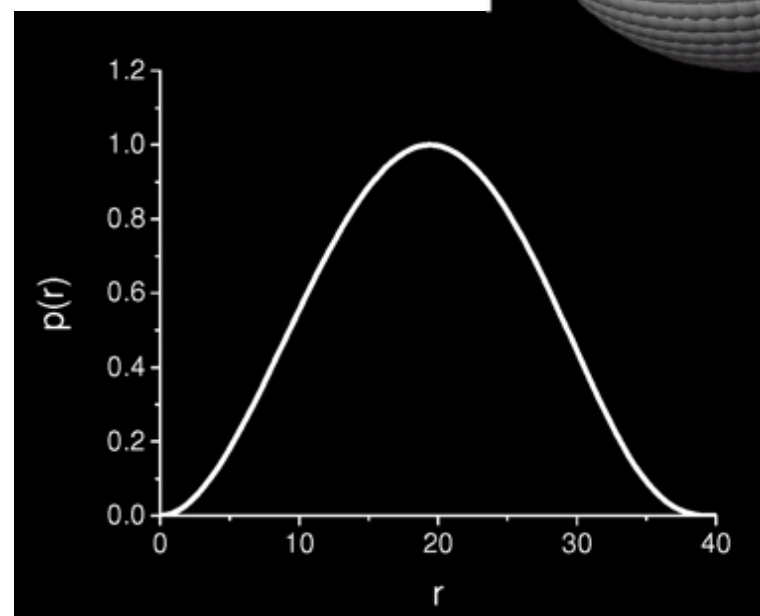
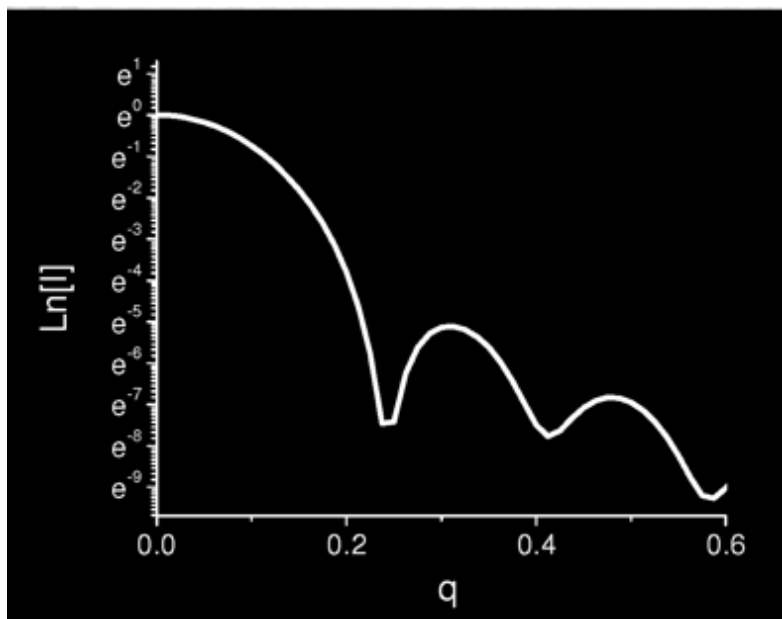


histogram of all intra-particle distances

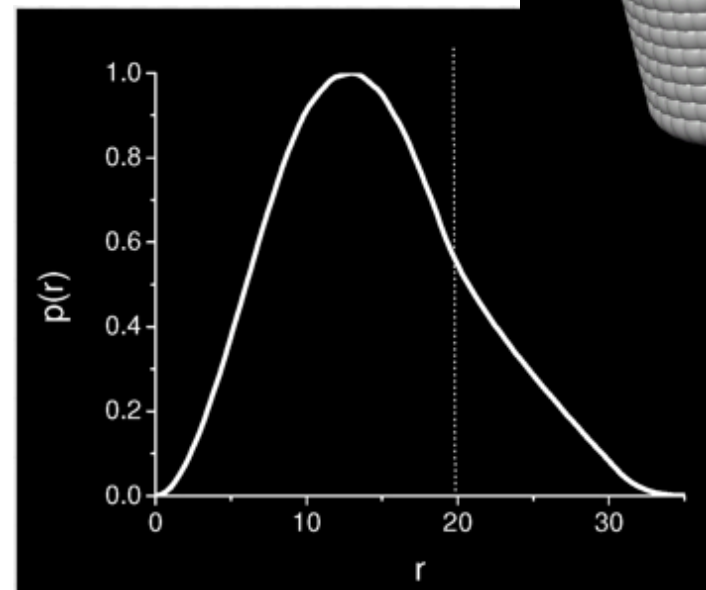
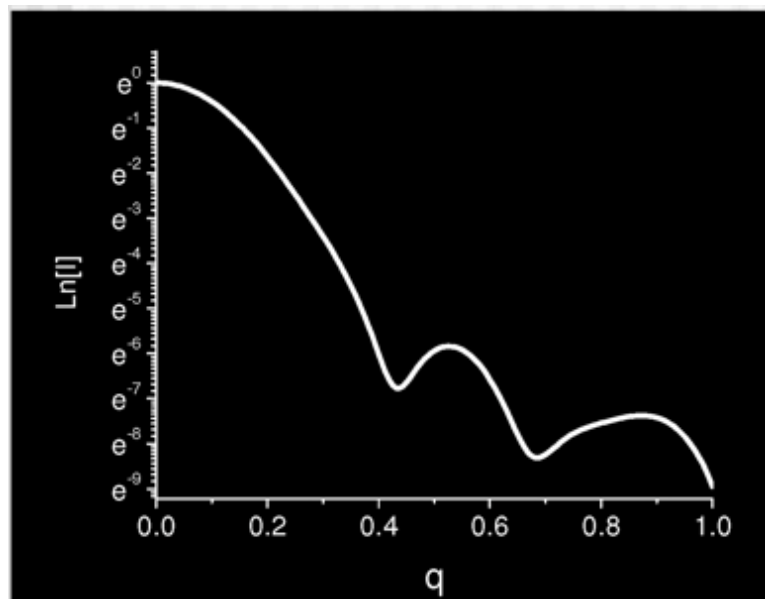


$$I(q) = 4\pi \int_0^\infty p(r) \frac{\sin(qr)}{qr} dr$$

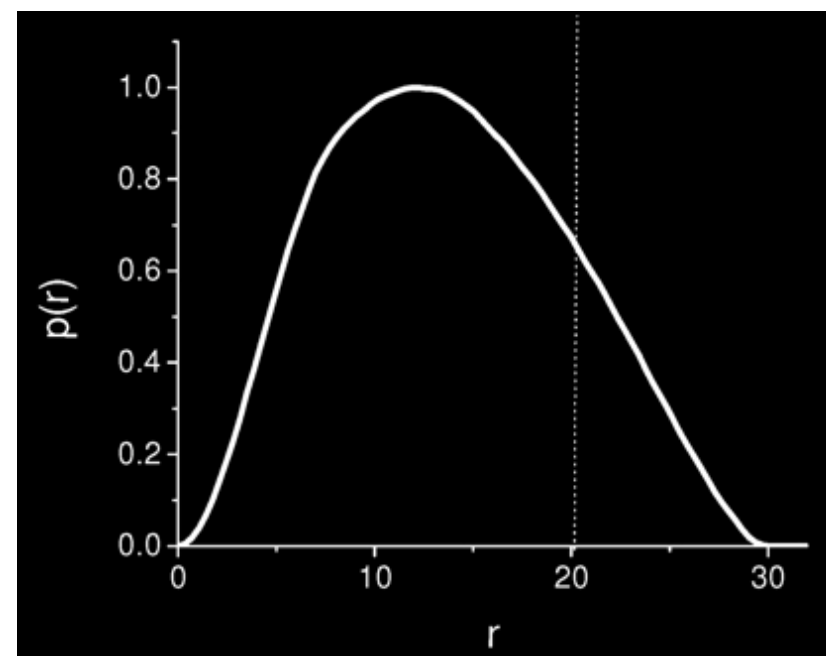
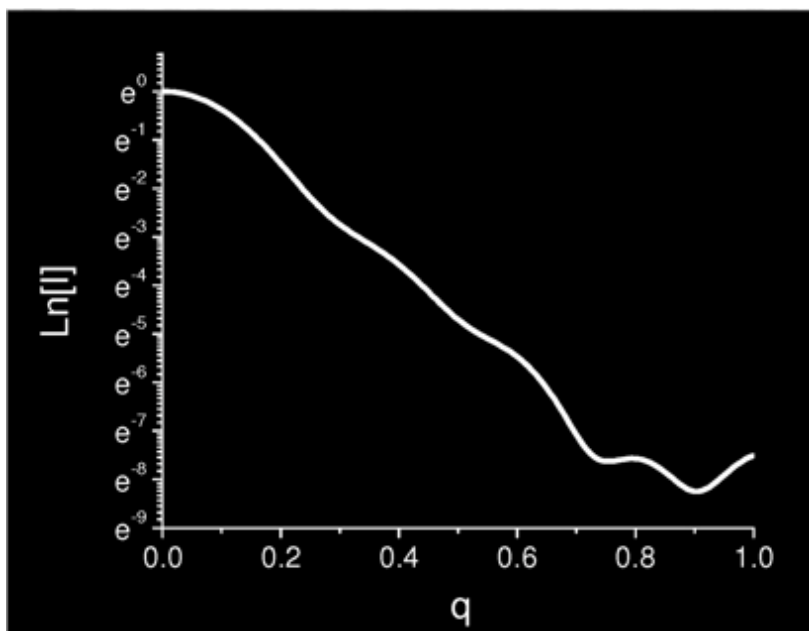
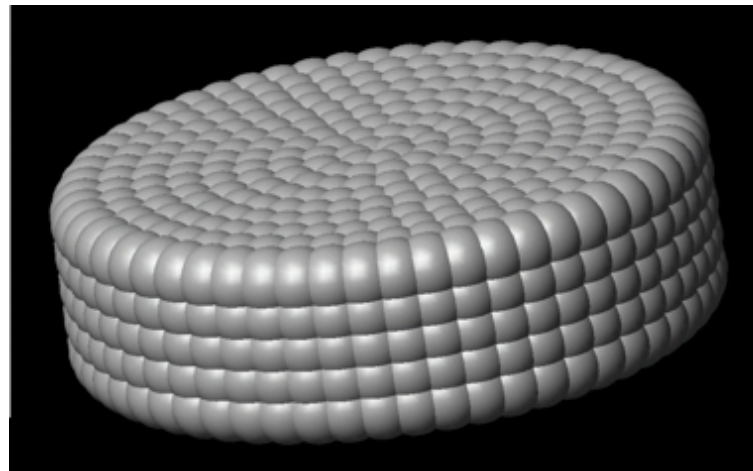
$I(q)$ & $P(r)$ of Spherical particle



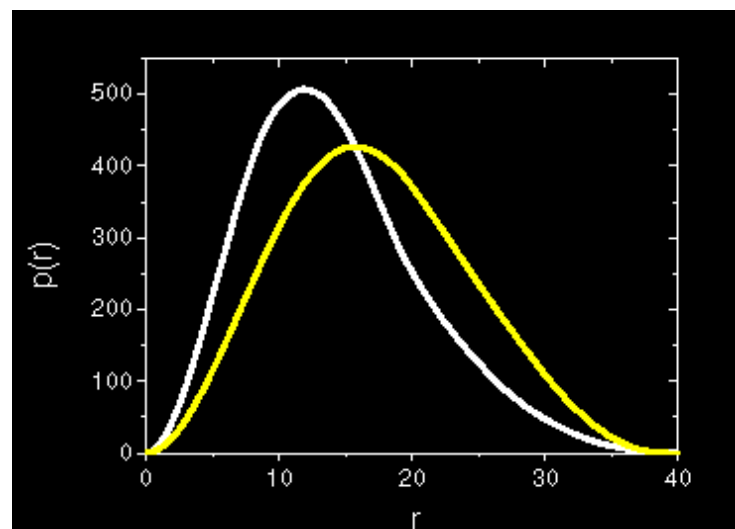
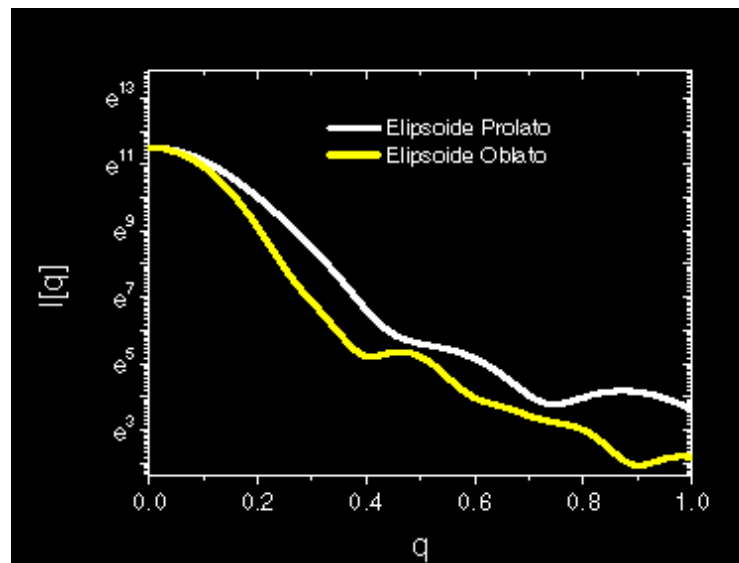
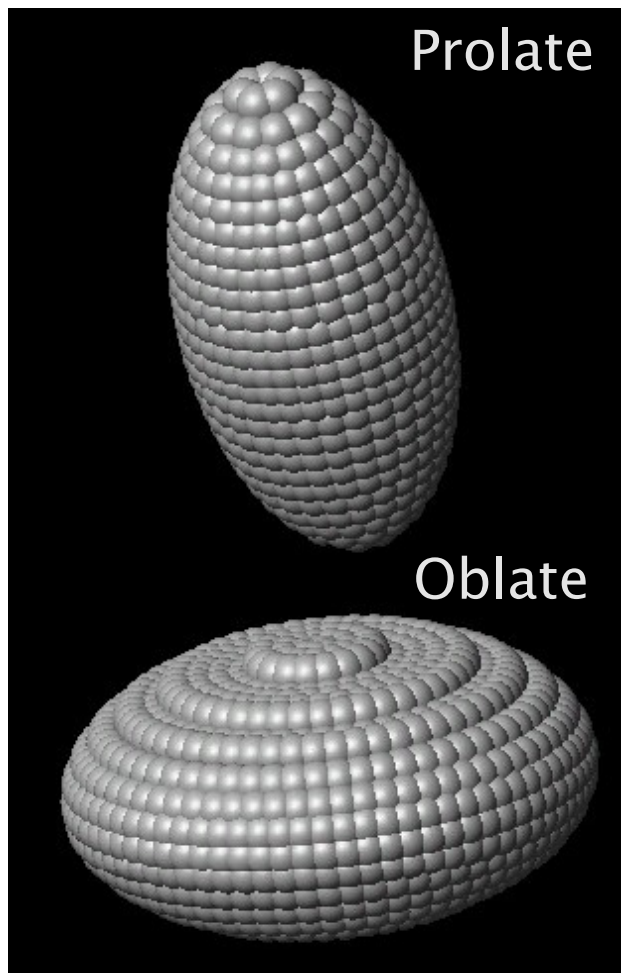
$I(q)$ & $P(r)$ of Cylindrical particle



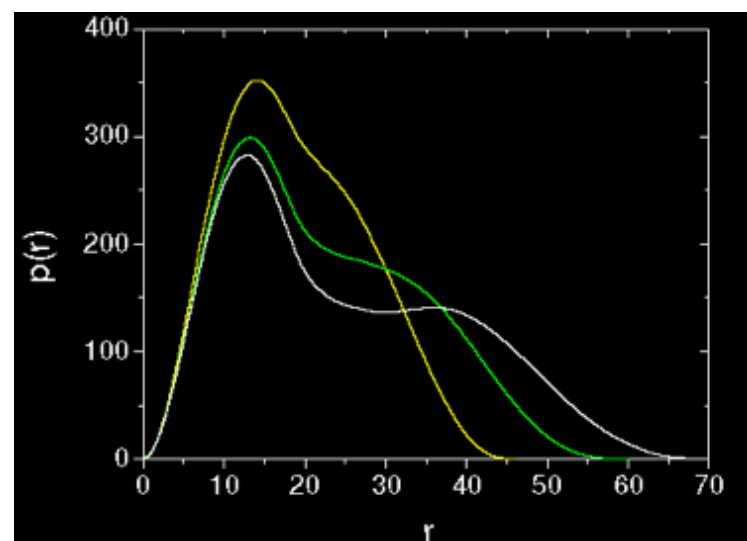
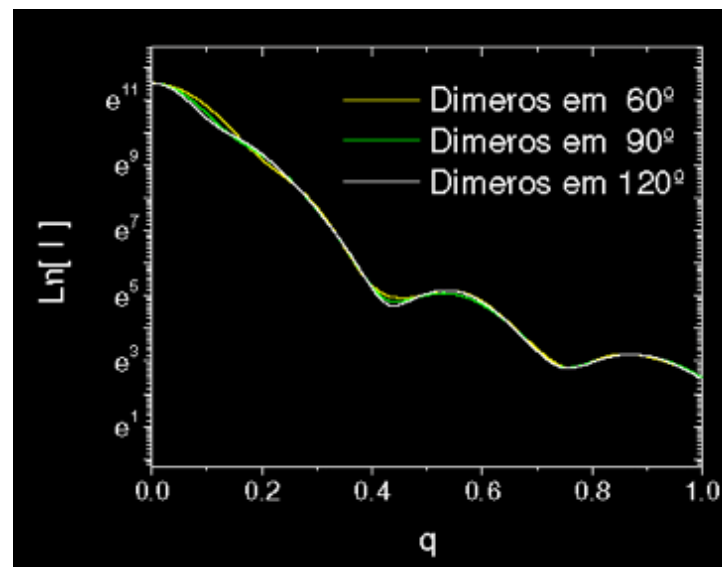
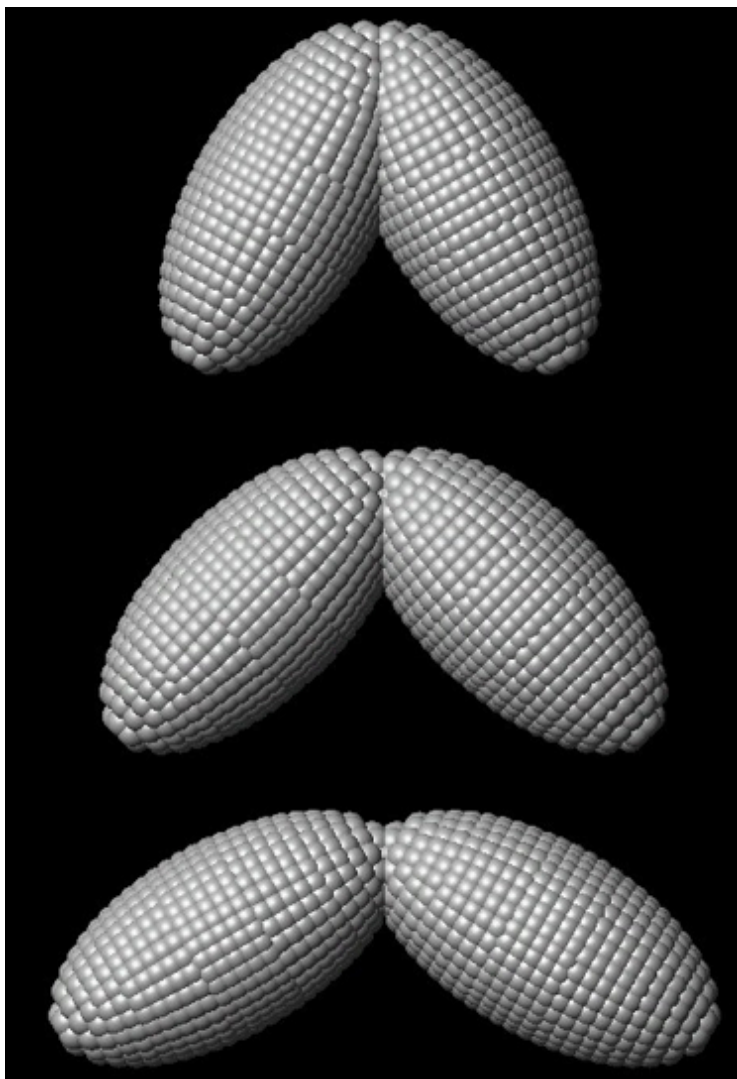
$I(q)$ & $P(r)$ of Flat particle



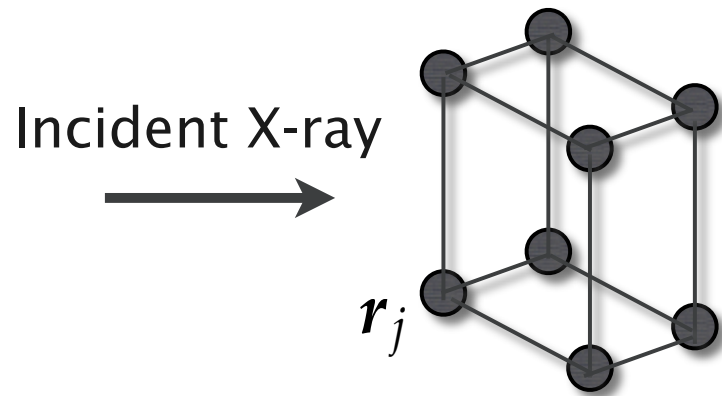
$I(q)$ & $P(r)$ of Ellipsoids



$I(q)$ & $P(r)$ of Two ellipsoid = dimer



Diffraction from Periodic Structure



Diffraction from Unit cell (Crystalline structure factor)

$$F(\mathbf{q}) = \sum_j f(\mathbf{q}) \exp(-i\mathbf{q} \cdot \mathbf{r}_j)$$

$f(\mathbf{q})$: Atomic Form Factor

Diffraction Intensity: $I(\mathbf{q}) \sim \underline{G(\mathbf{q})}^2 F(\mathbf{q})^2$

Laue function: $|G(\mathbf{q})|^2 = \frac{\sin^2(\pi N \mathbf{q} \cdot \mathbf{r})}{\sin^2(\pi \mathbf{q} \cdot \mathbf{r})}$

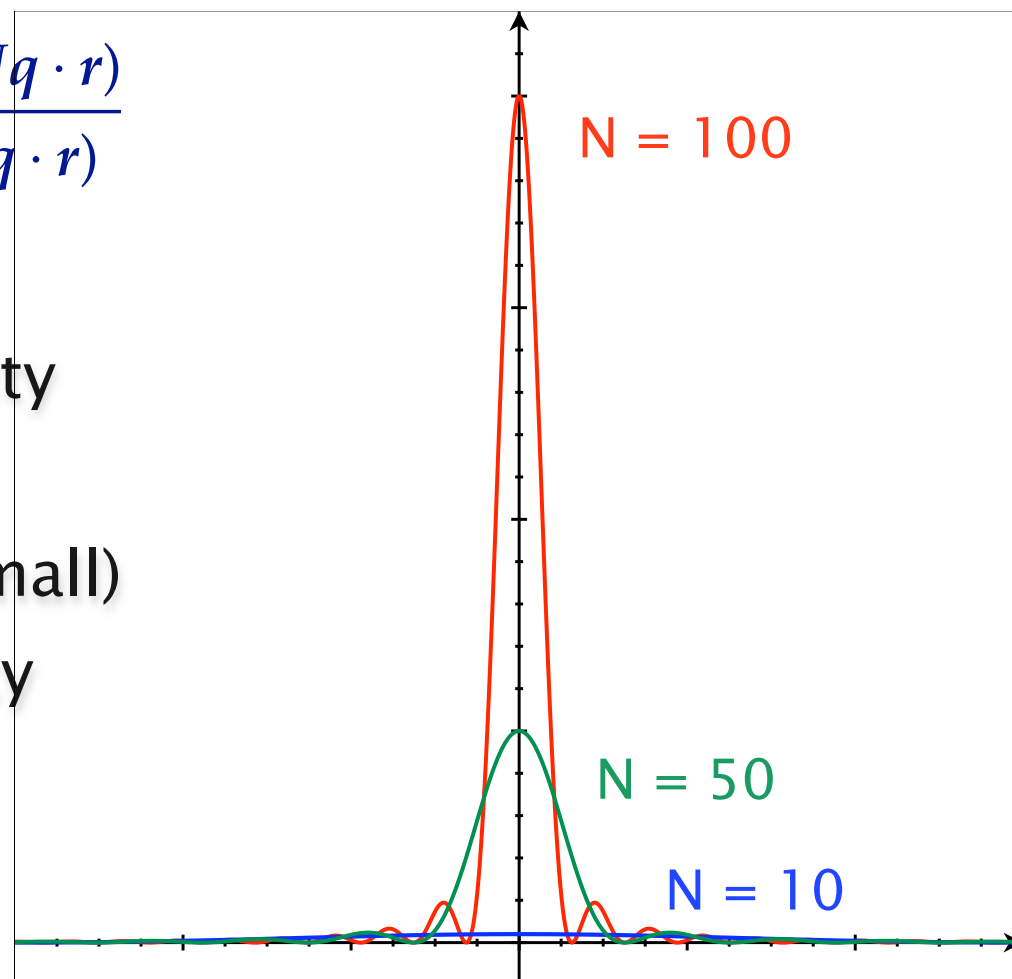
- Maximum $\sim N^2$
- FWHM $\sim 2\pi/N$
 - FWHM \rightarrow Size of crystal

Laue Function

Laue function: $|G(\mathbf{q})|^2 = \frac{\sin^2(\pi N \mathbf{q} \cdot \mathbf{r})}{\sin^2(\pi \mathbf{q} \cdot \mathbf{r})}$

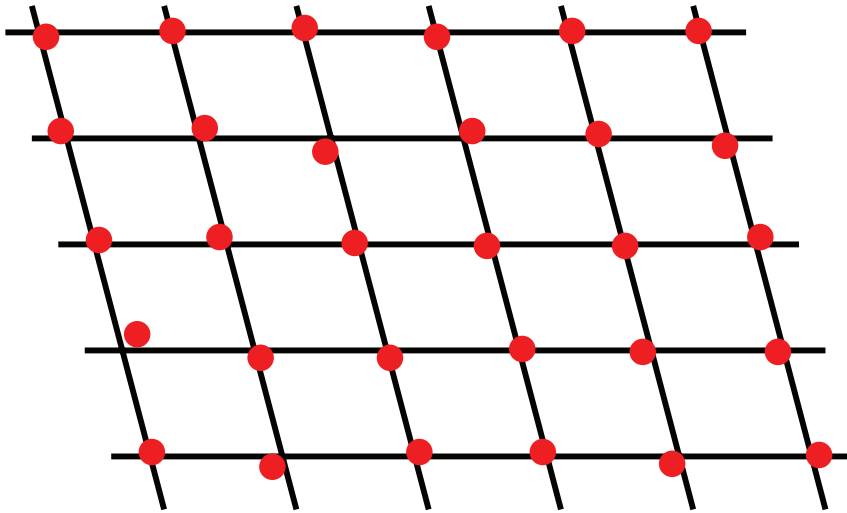
- Large crystal
 - High diffraction intensity
 - Narrow FWHM
- Soft matter (crystal size: small)
 - Low diffraction intensity
 - Wide FWHM

→ low S/N



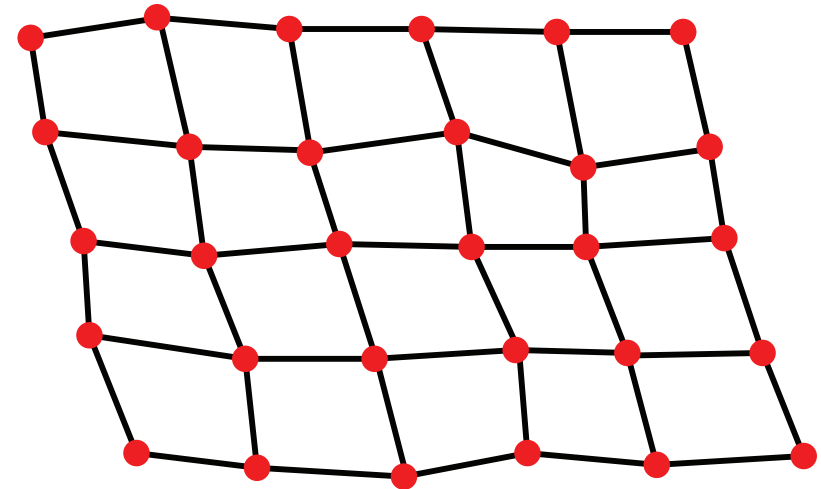
Crystal size --> Intensity & FWHM of diffraction

Imperfection of crystal (2D)



Imperfection of 1st kind

Thermal fluctuation etc.

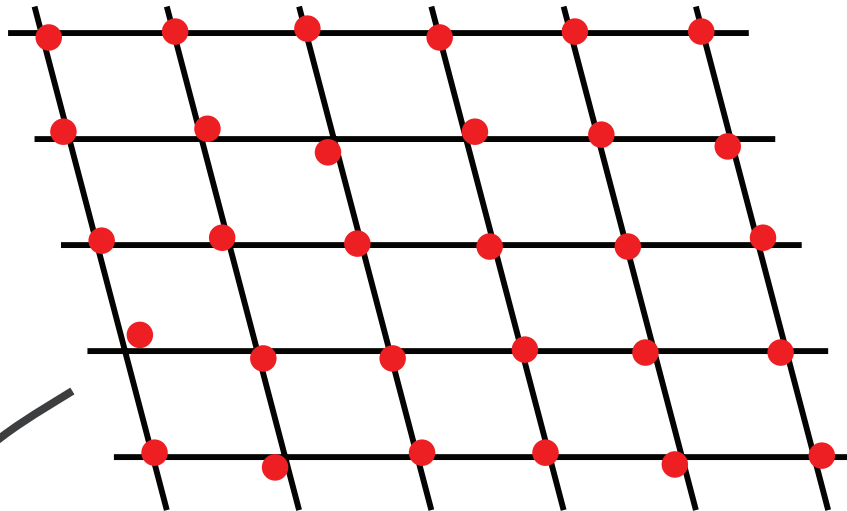


Imperfection of 2nd kind

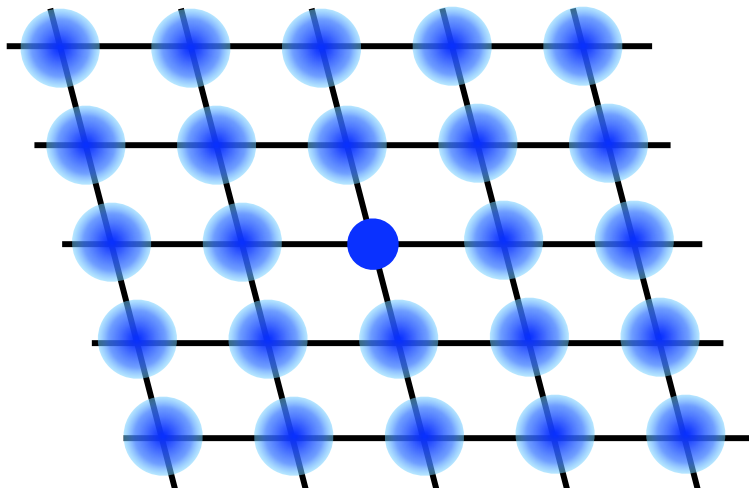
in the case of soft matter

Imperfection of crystal

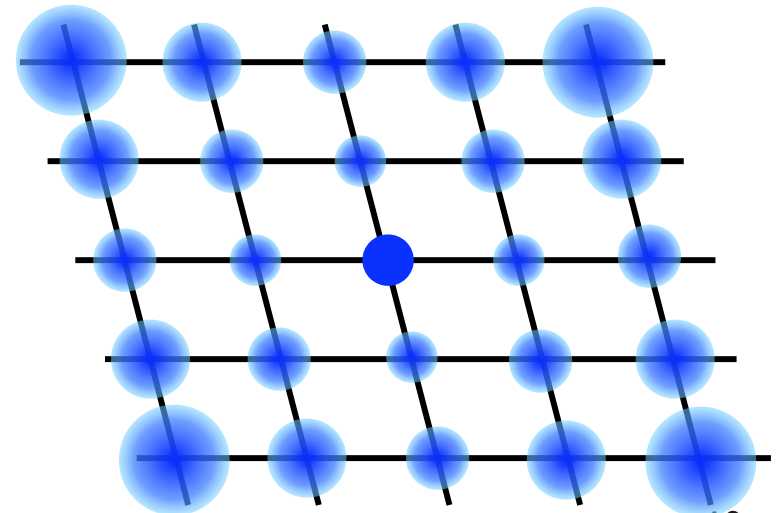
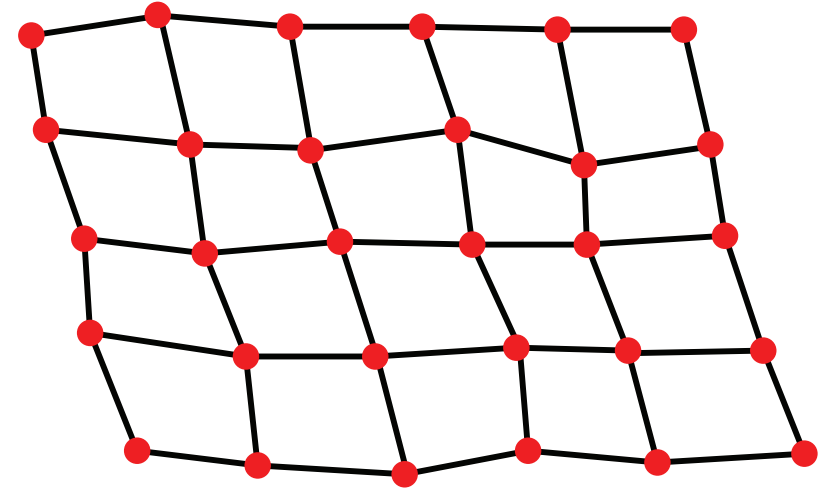
Imperfection of 1st kind




Autocorrelation




Imperfection of 2nd kind



Imperfection of lattice (1D)

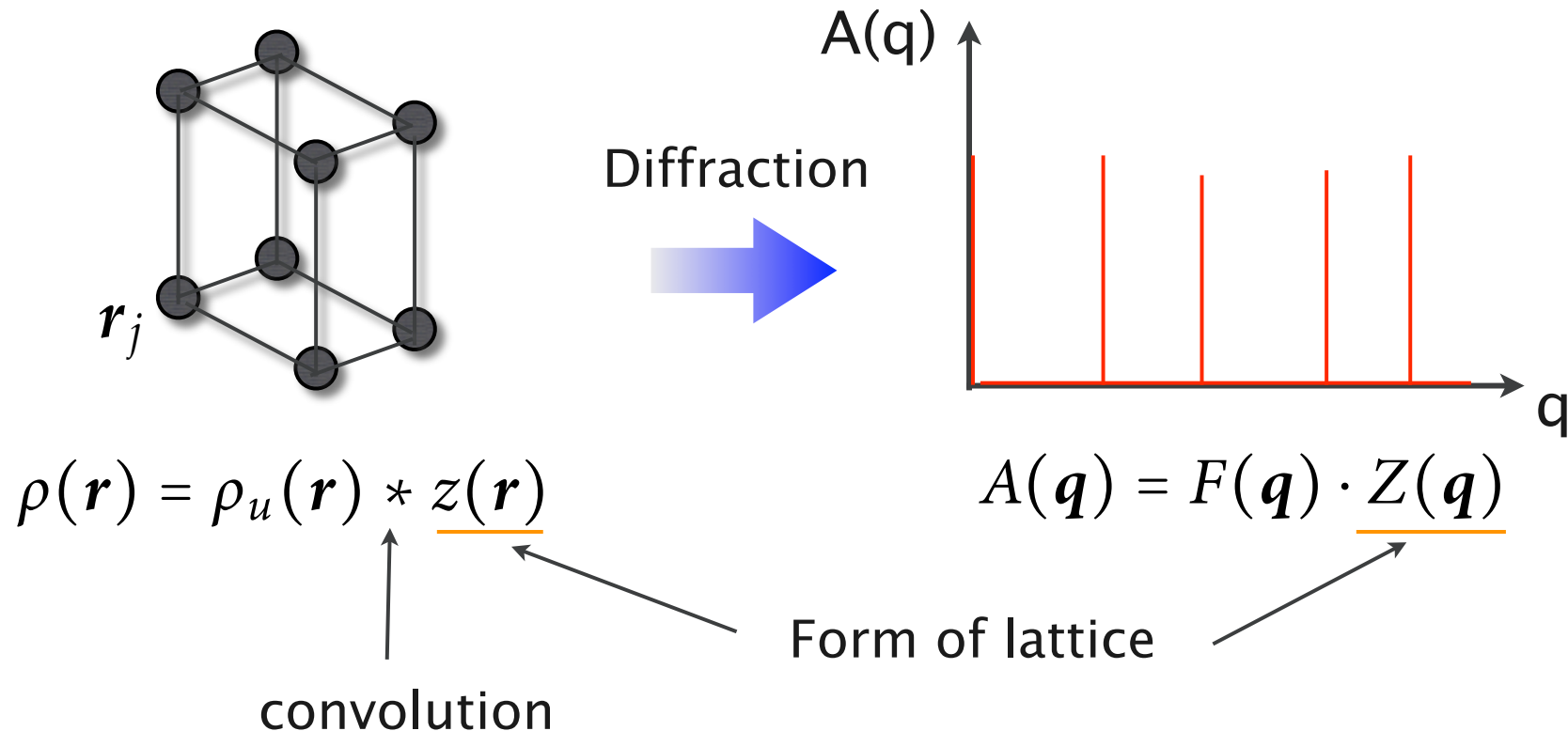
Perfect lattice 

Imperfection of 1st kind 

Imperfection of 2nd kind 

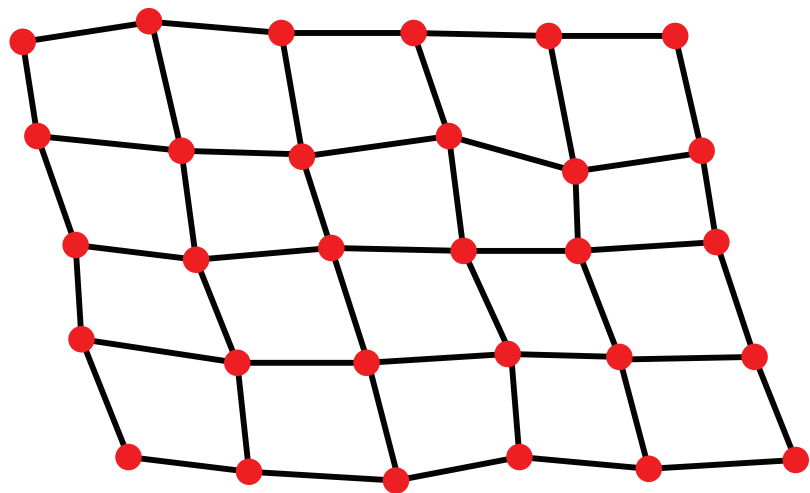
☞ Effect of imperfections on diffraction ?

Diffraction from lattice-structure



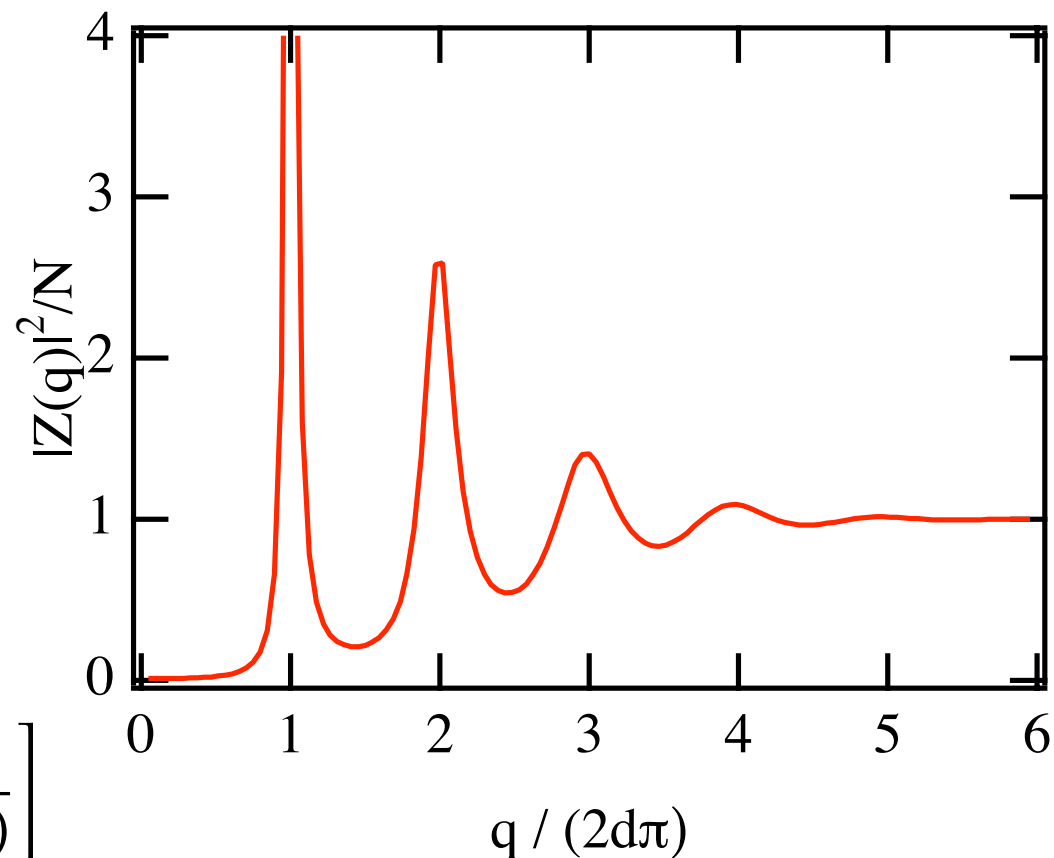
$z(\mathbf{r})$ with imperfection ---> calculate $Z(\mathbf{q})$

Imperfection of 2nd kind



Paracrystal theory

$$|Z(q)|^2 = N \left[1 + \frac{P(q)}{1 - P(q)} + \frac{P^*(q)}{1 - P^*(q)} \right]$$



Decrease of diffraction intensity and
Increase of FWHM

R. Hosemann, S. N. Bagchi, Direct Analysis of Diffraction
by Matter, North-Holland, Amsterdam (1962).

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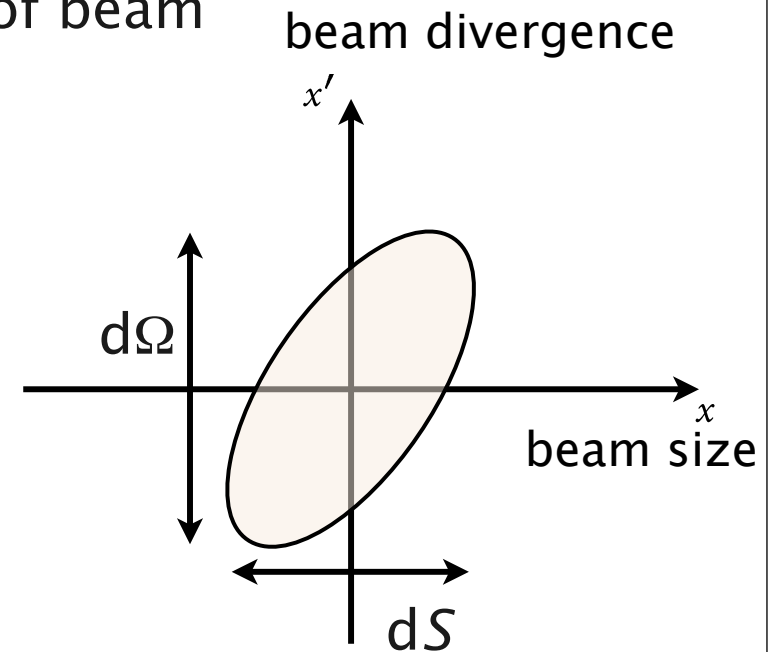
- ❧ Introduction
 - ❧ What's SAXS ?
 - ❧ History
- ❧ Theory
 - ❧ Basic of X-ray scattering
 - ❧ Structural Information obtained by SAXS
- ❧ Experimental Methods
 - ❧ X-ray Optics
 - ❧ X-ray Detectors
- ❧ Advanced SAXS
 - ❧ Microbeam, GI-SAXS, USAXS, XPCS etc...

X-ray Source for SAXS

Emittance -- Product of size and divergence of beam

$$\text{Brilliance} = \frac{d^4 N}{dt \cdot d\Omega \cdot dS \cdot d\lambda/\lambda}$$

[photons/(s · mrad² · mm² · 0.1% rel.bandwidth)]

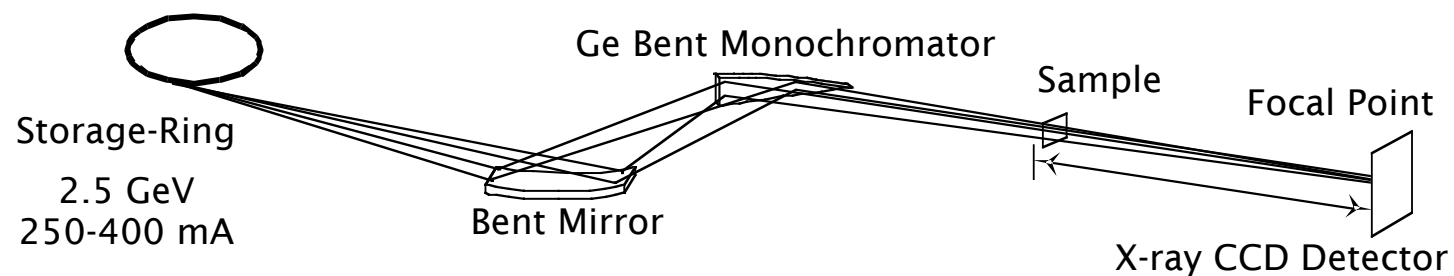


SAXS needs to use a low divergence and small beam

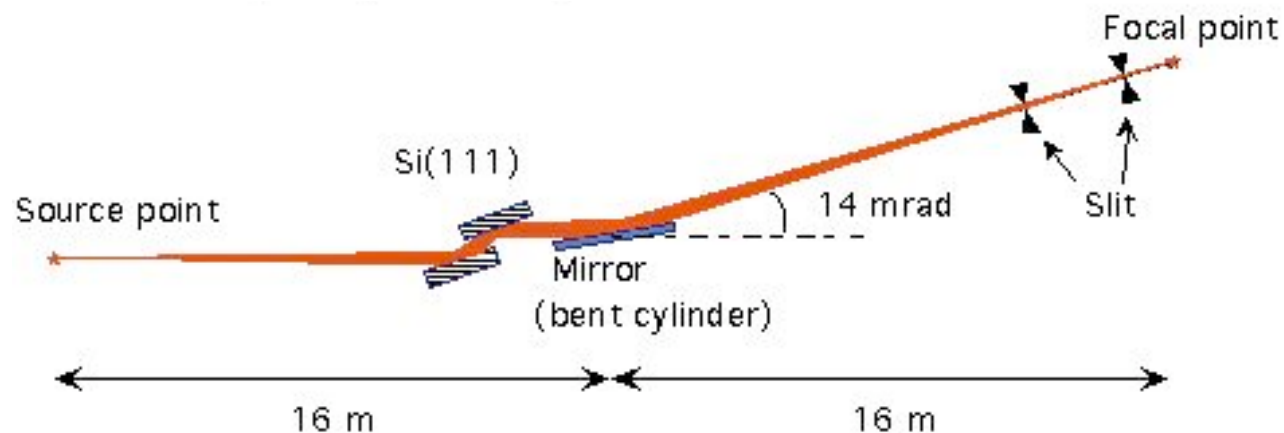
→ High brilliance beam is required !

X-ray Optics for SAXS

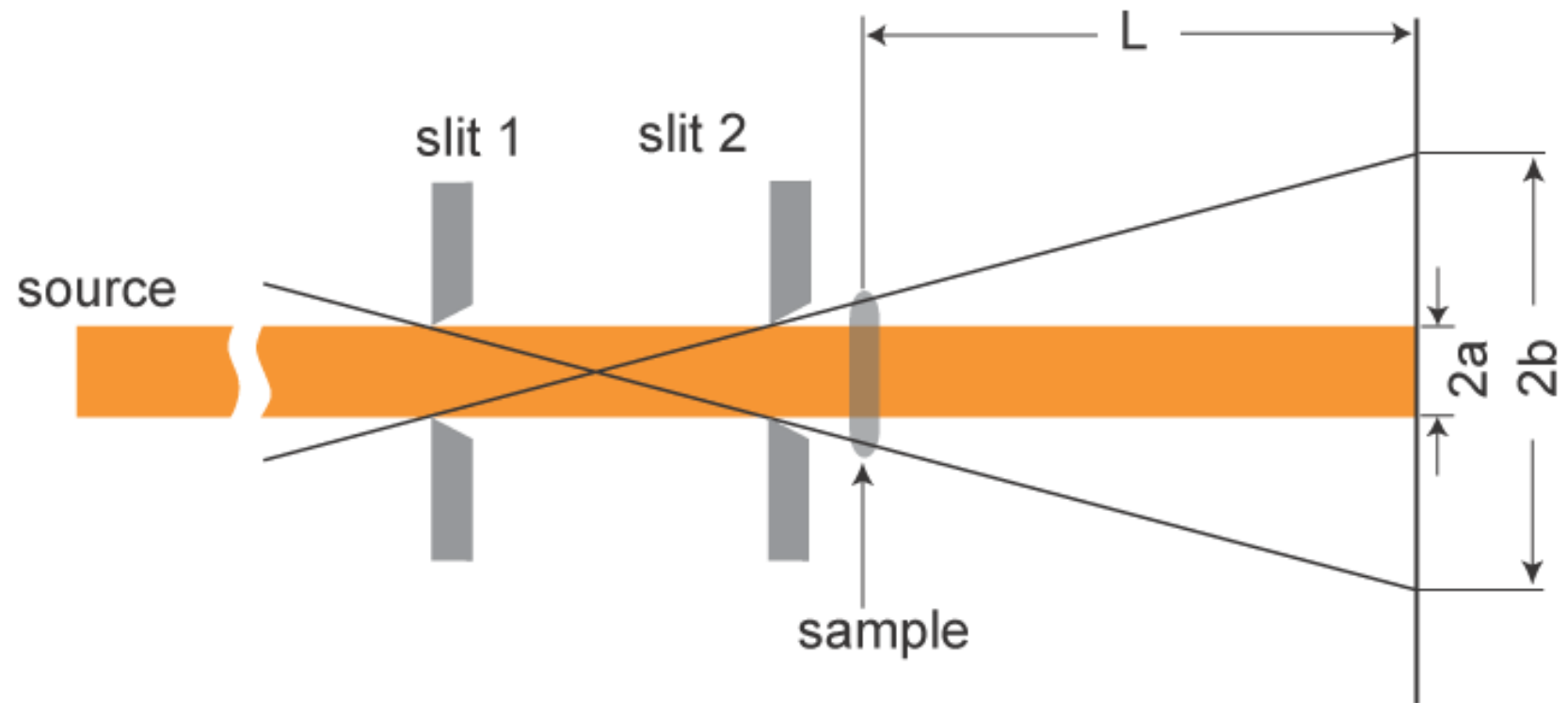
PF BL-15A



PF BL-10C



Slits for SAXS



Detectors for SAXS

	Good Point	Drawback
PSPC	<ul style="list-style-type: none">• time-resolved• photon-counting• low noise	<ul style="list-style-type: none">• counting-rate limitation
Imaging Plate	<ul style="list-style-type: none">• wide dynamic range• large active area	<ul style="list-style-type: none">• slow read-out
CCD with Image Intensifier	<ul style="list-style-type: none">• time-resolved• high sensitivity	<ul style="list-style-type: none">• image distortion• low dynamic range
Fiber-tapered CCD	<ul style="list-style-type: none">• fast read-out• automated measurement	<ul style="list-style-type: none">• not good for time-resolved

X-ray CCD detector with Image Intensifier

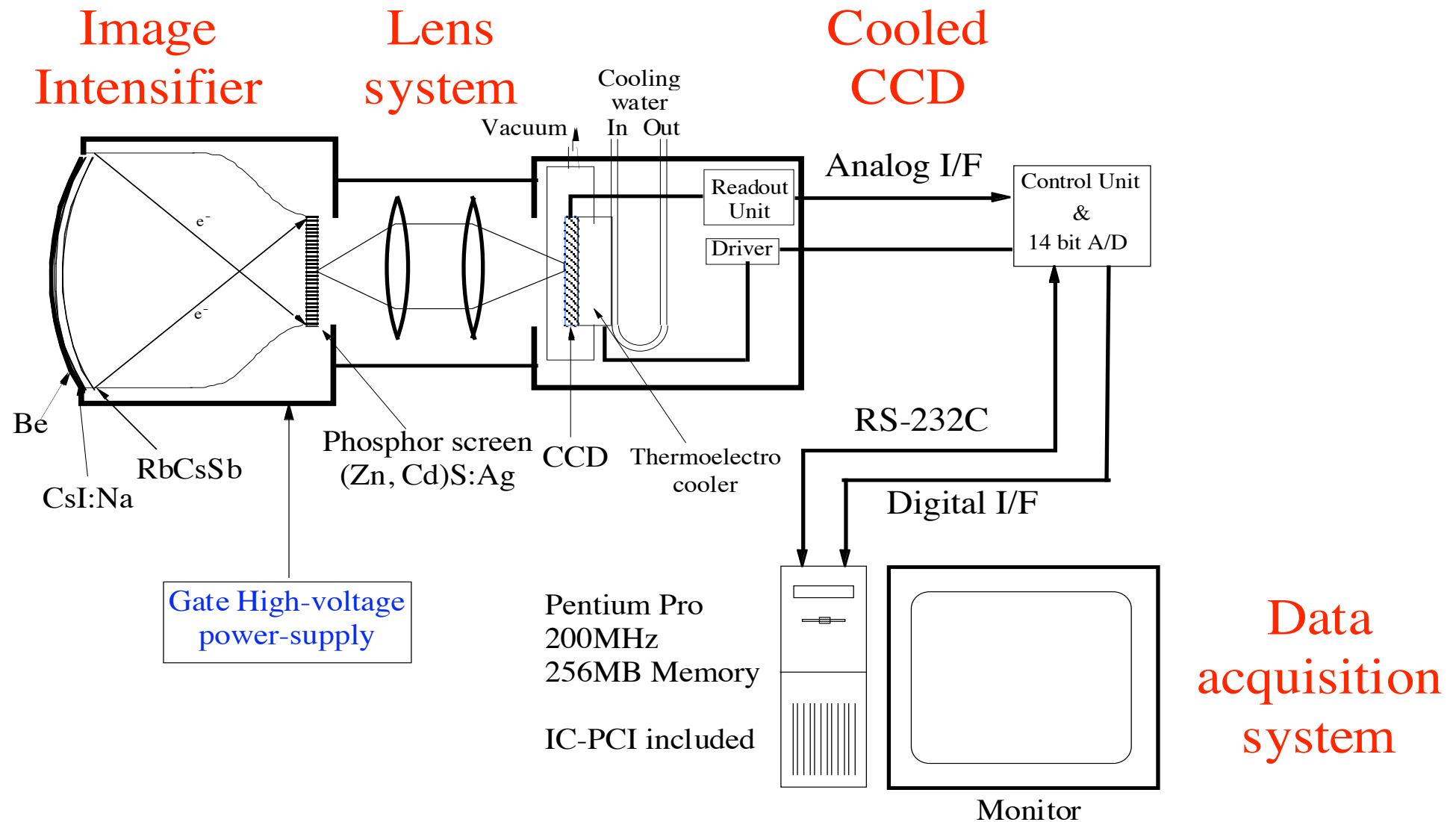
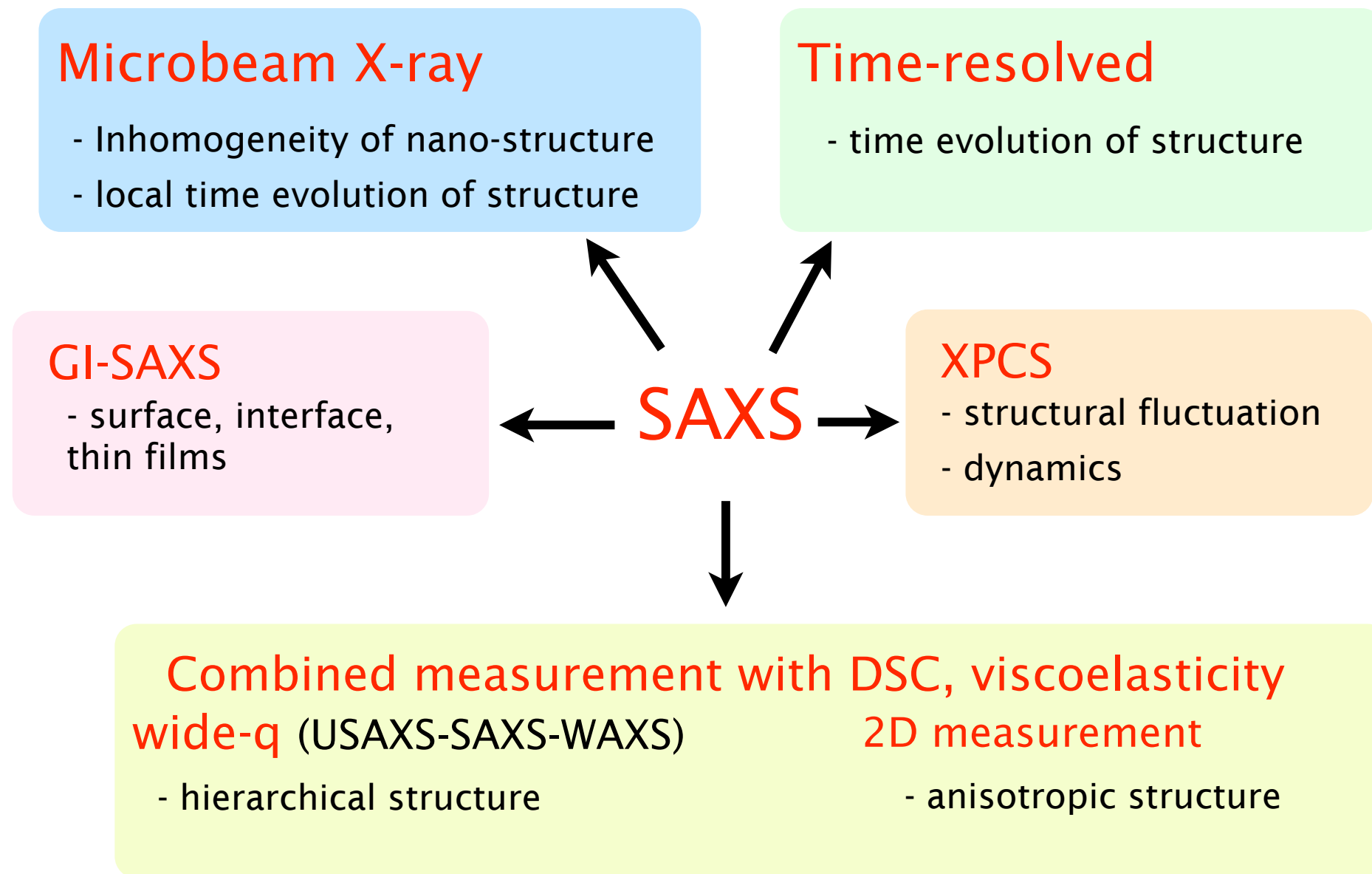


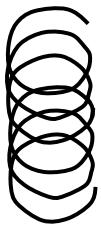
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 - ❧ X-ray Optics
 - ❧ X-ray Detectors
- ❧ Advanced SAXS
 - ❧ Microbeam, GI-SAXS, USAXS, XPCS etc...

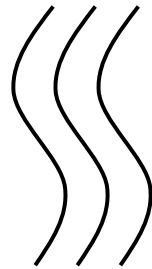
Advanced SAXS



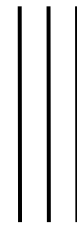
Application of paracrystal theory



African

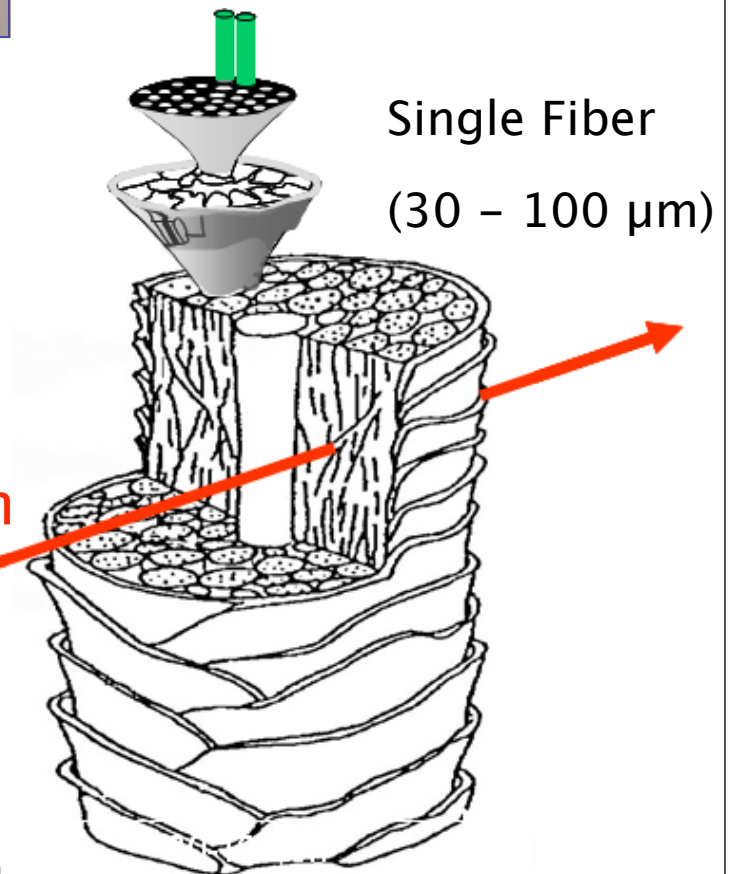


Caucasian



Asian

Collab. with Kao Ltd.



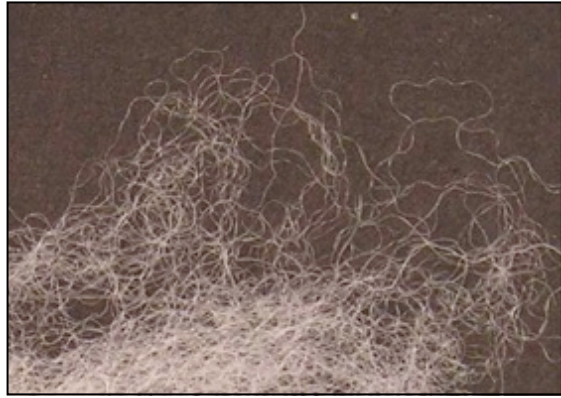
Single Fiber
(30 – 100 μm)

X-ray Microbeam
(5 μm x 5 μm)

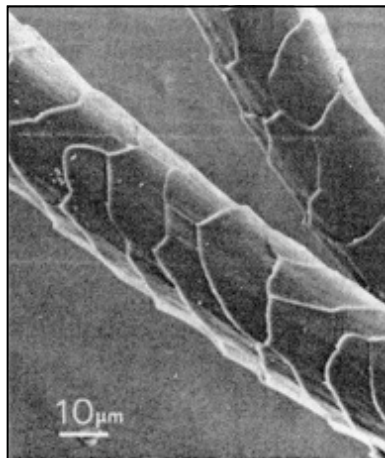
Relationship between macroscopic form
and nano structure?

Local observation with an X-ray microbeam

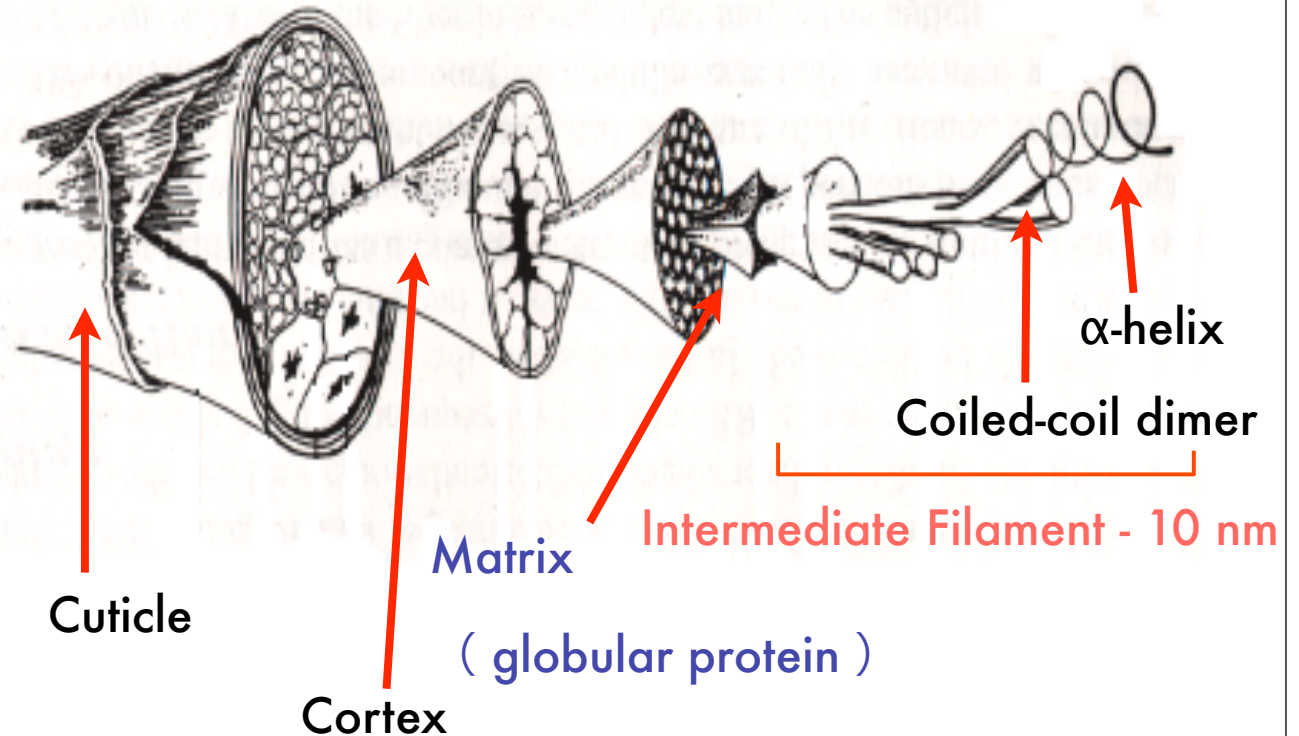
Internal structure of wool



SEM 像



H. Ito et al., Textile Res. J. 54, 397-402 (1986).

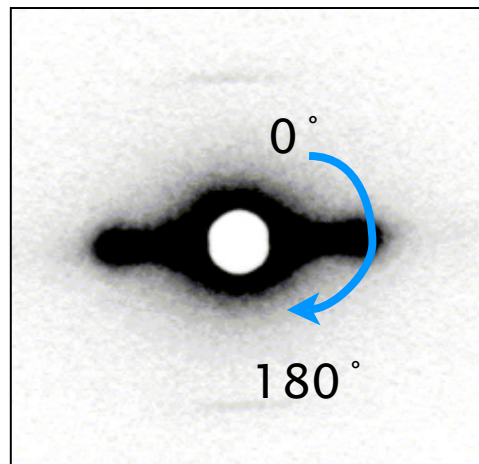
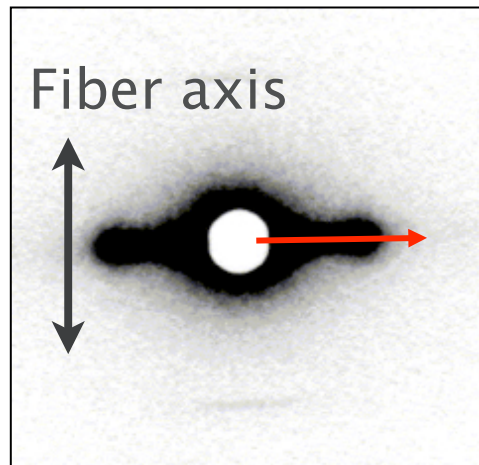


R. D. B. Fraser et al., Proc. Int. Wool Text. Res. Conf., Tokyo, II, 37, (1985) partially changed.

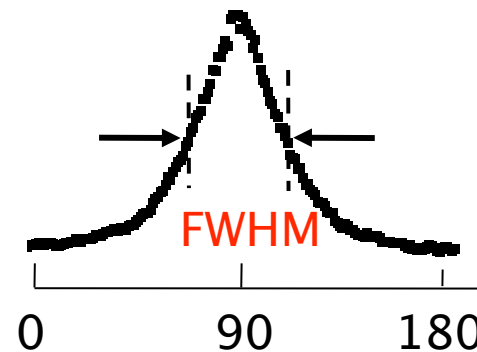
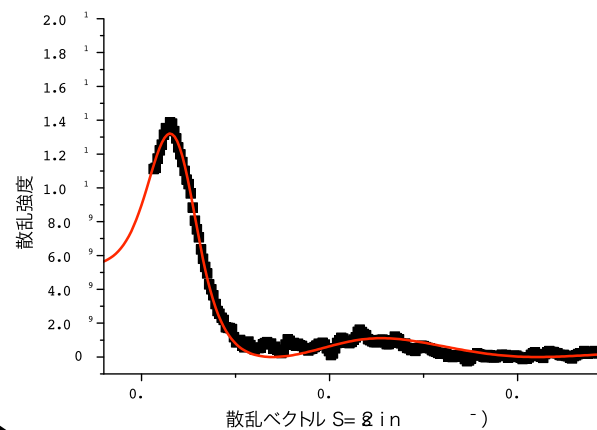
Relationship between IF distribution and hair curliness?

Structure of Intermediate Filament

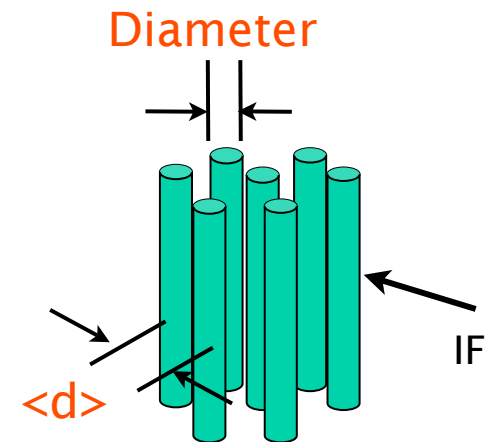
Scattering pattern



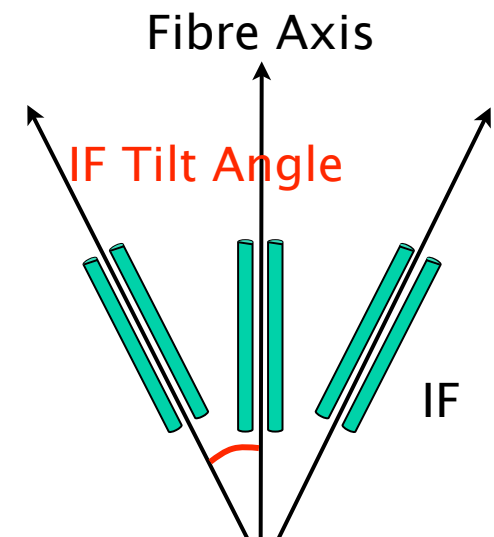
1D intensity profile



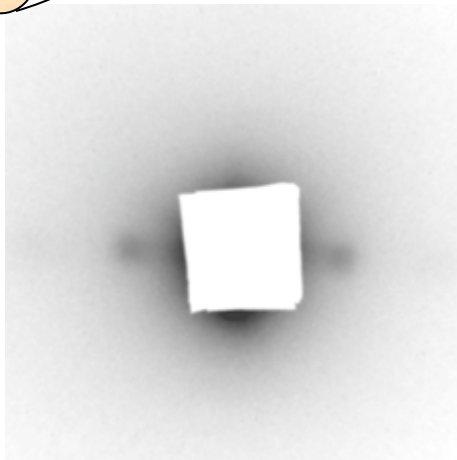
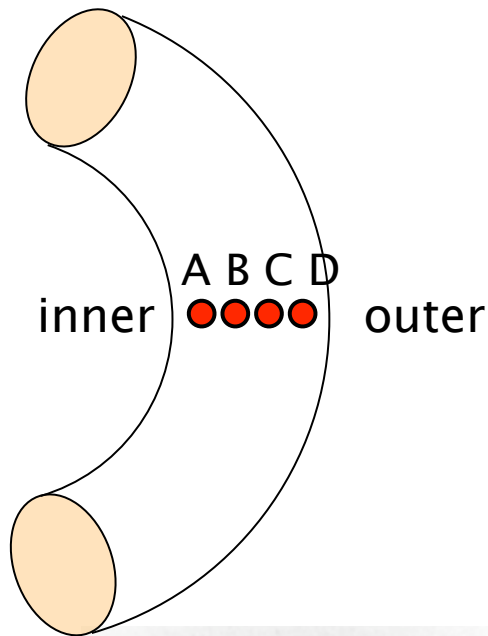
Real space structure



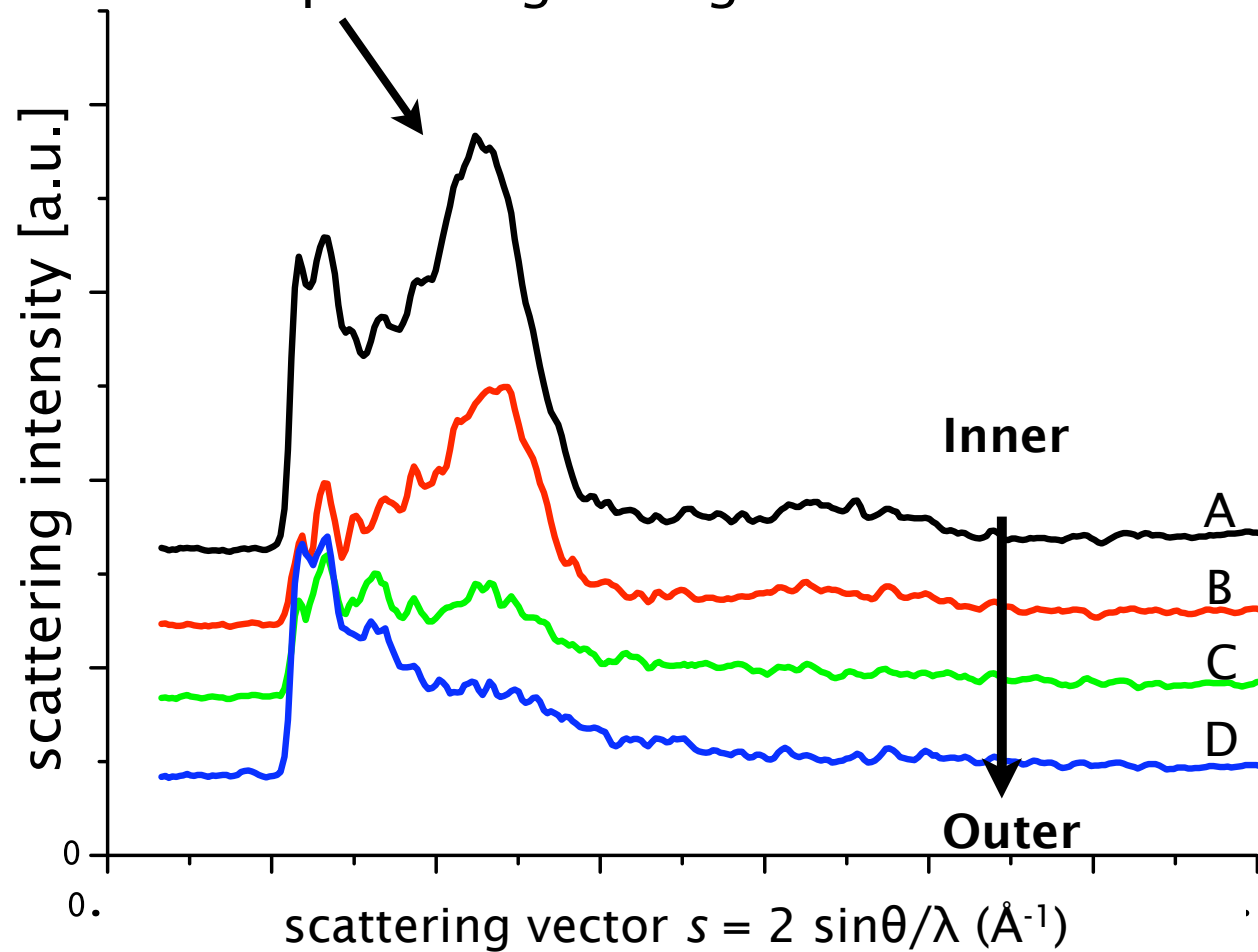
IF-IF Distance



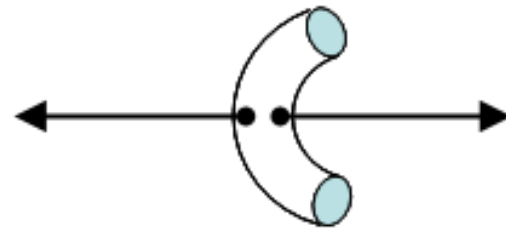
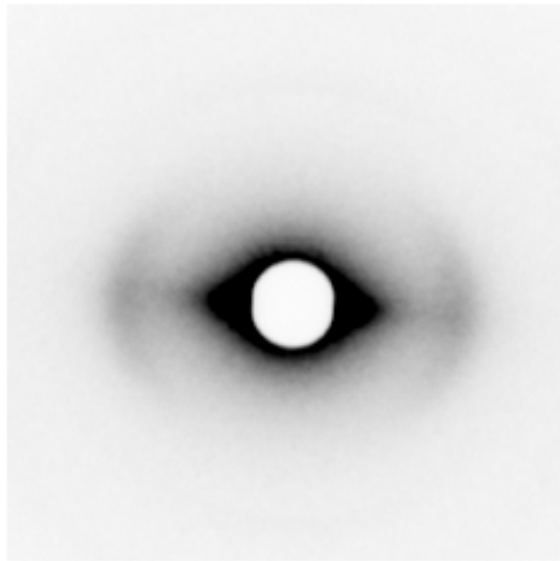
Diffraction intensity profiles



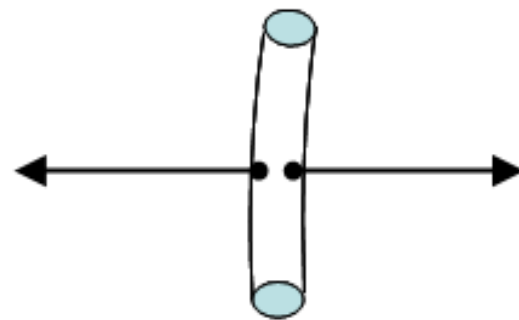
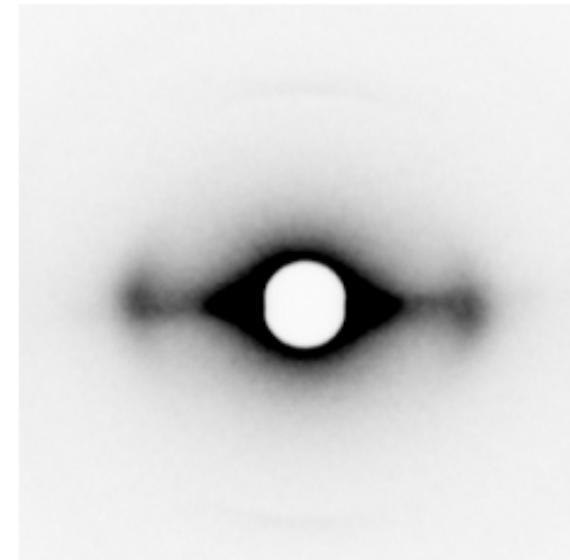
Diffraction peak originating from IF



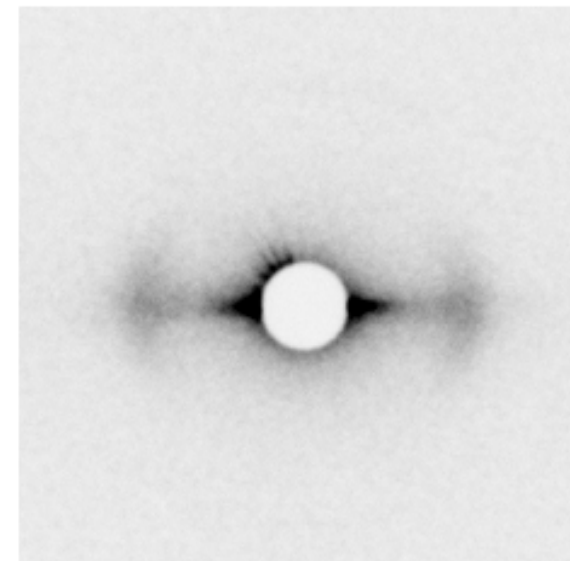
Difference in diffraction intensity
--> Structural difference in cortex.



Curly
(ROC = 1.5cm)

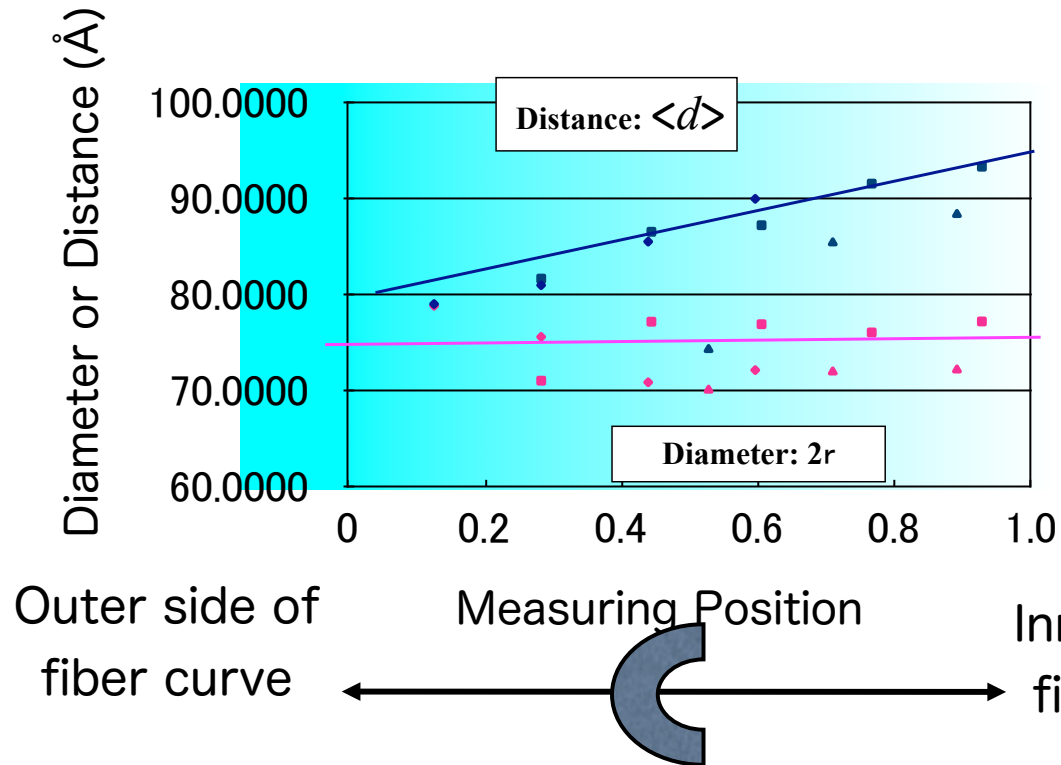


Nearly Straight
(ROC ~ 10cm)

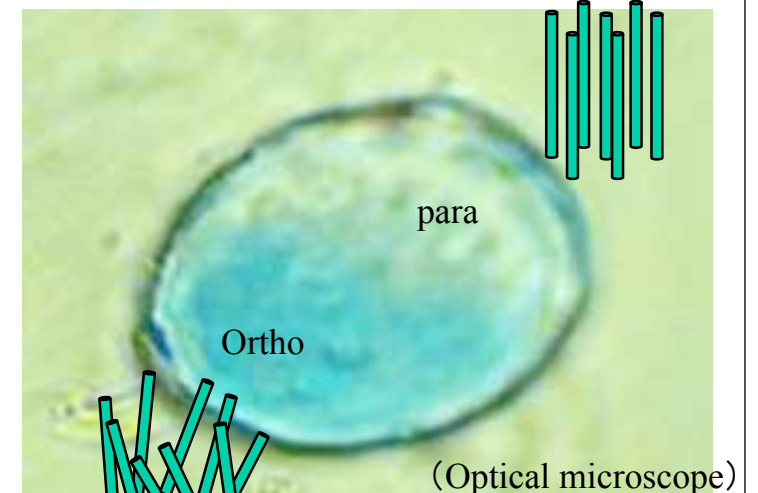


ROC: Radius of Curvature

Distribution of Intermediate Filament (IF)

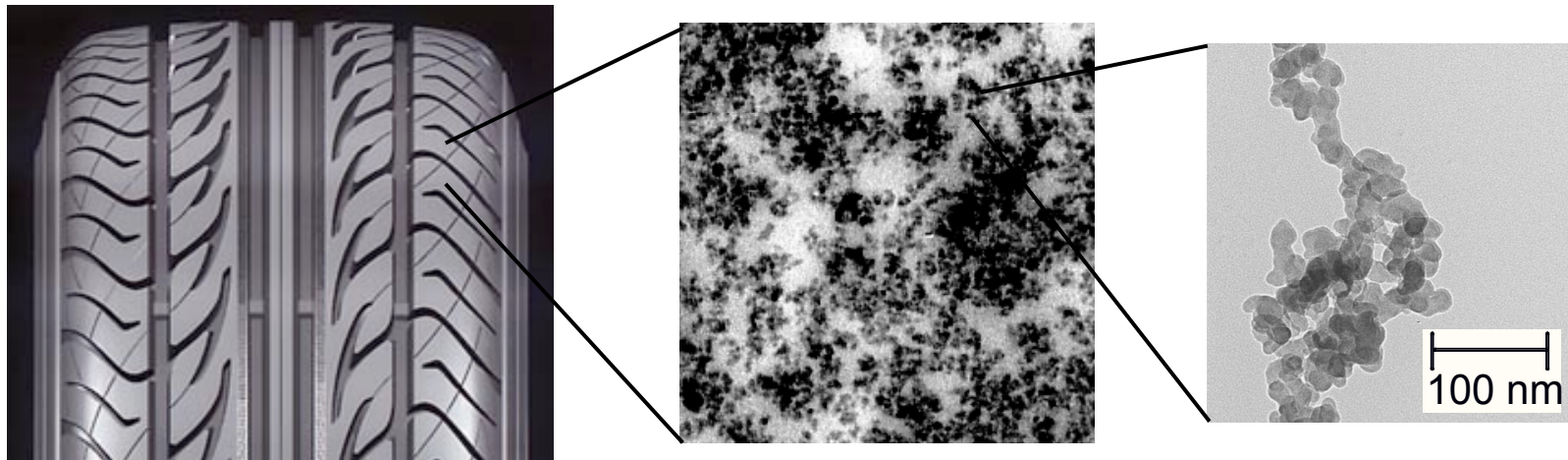


Stained with Methylene blue dye



- Diameter of Intermediate filament (IF) is almost constant
- Distance between IFs increases from outer to inner sides.

USAXS for nano-composite in rubber

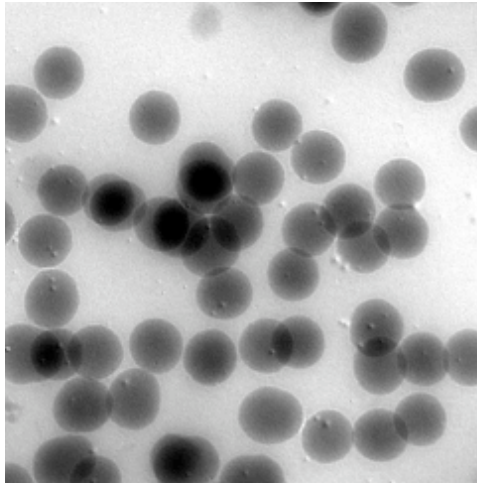


Nanocomposite

USAXS using medium-length beamline

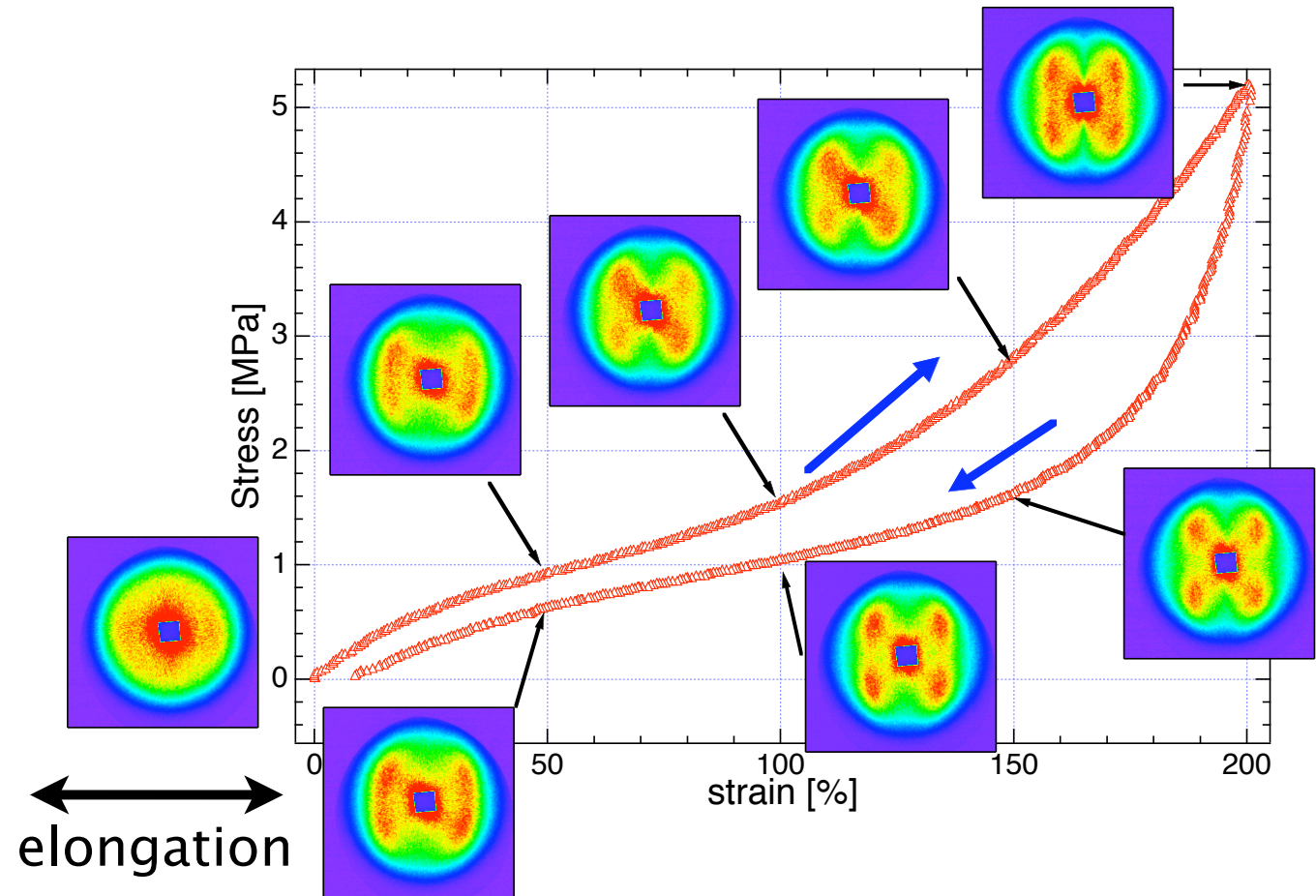


USAXS patterns from elongated rubber



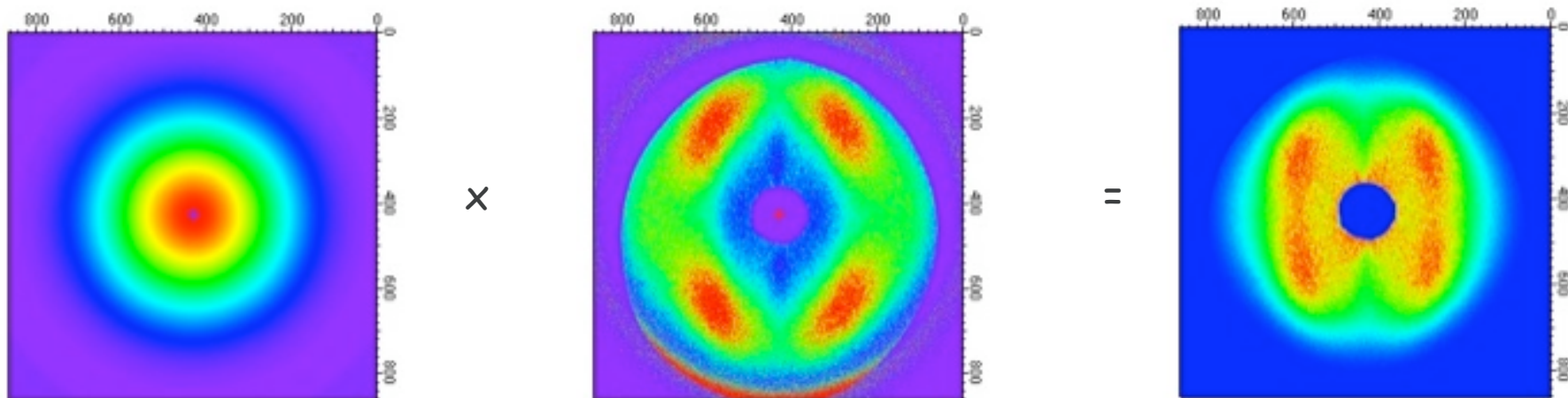
TEM image

Rubber filled with spherical silica



Scattering pattern also shows hysteresis.

Separation of Structure factor $S(q)$



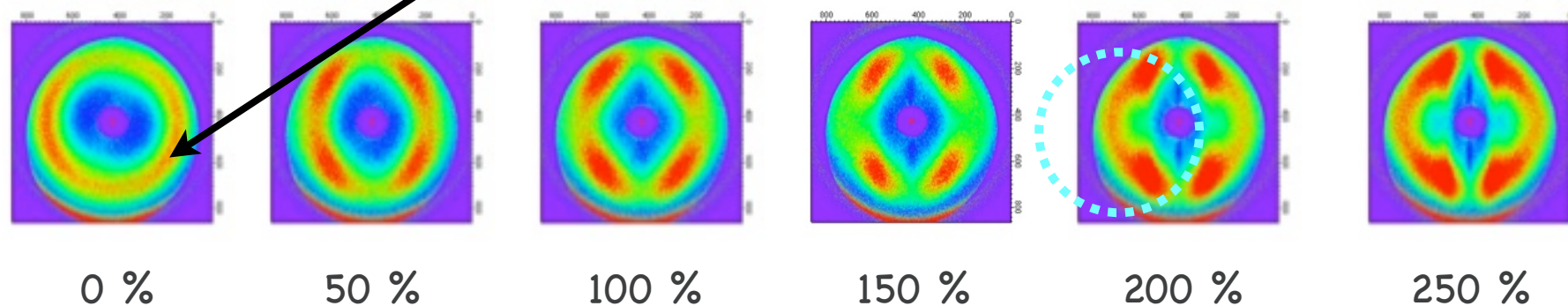
$F(q)$: form factor

$S(q)$: structure factor

$I(q)$: intensity

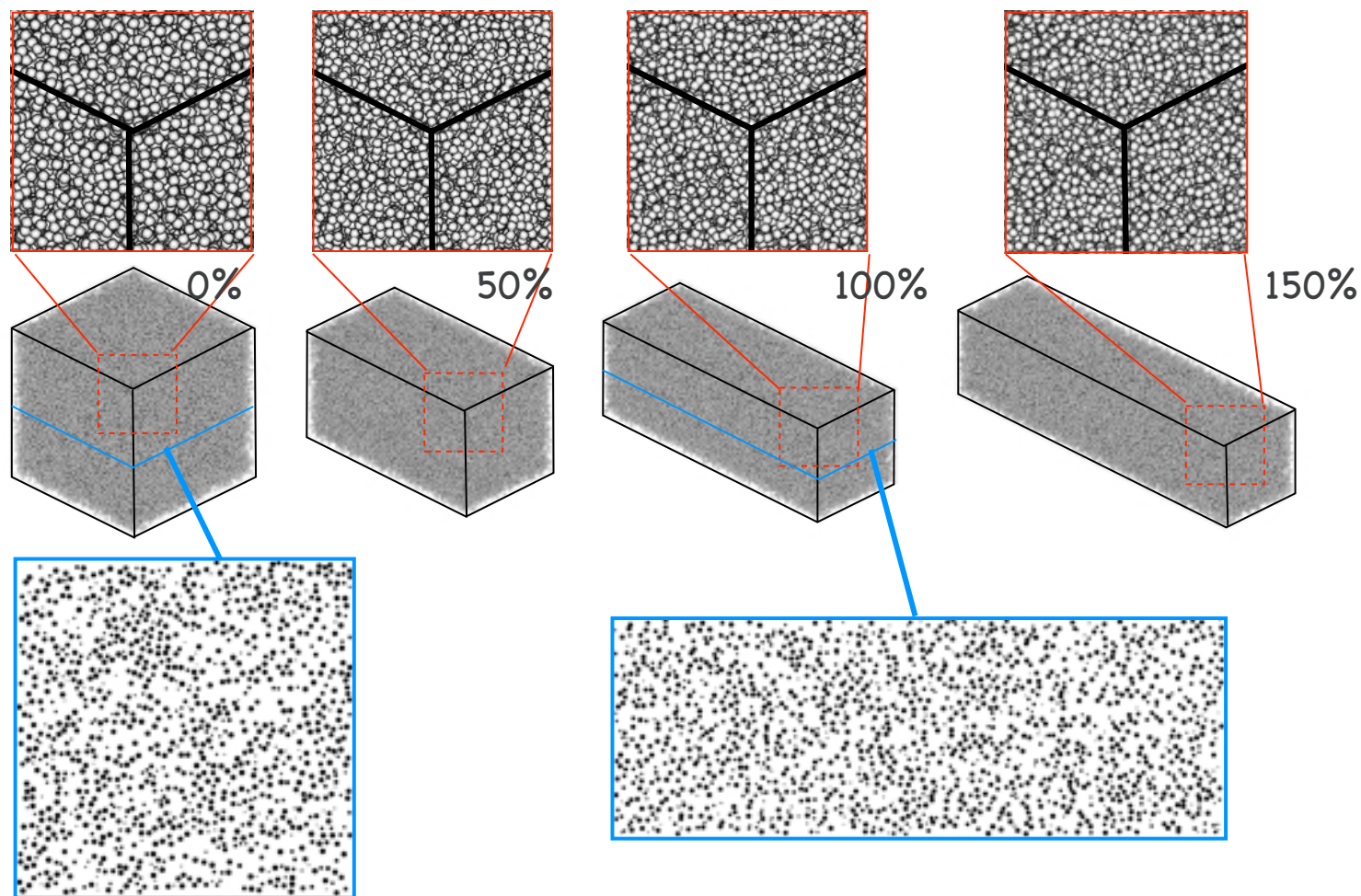
$D_{ave} = 282.9 \text{ nm}$

corresponding to distances between silica particles



Analysis by RMC (Reverse Monte Carlo)

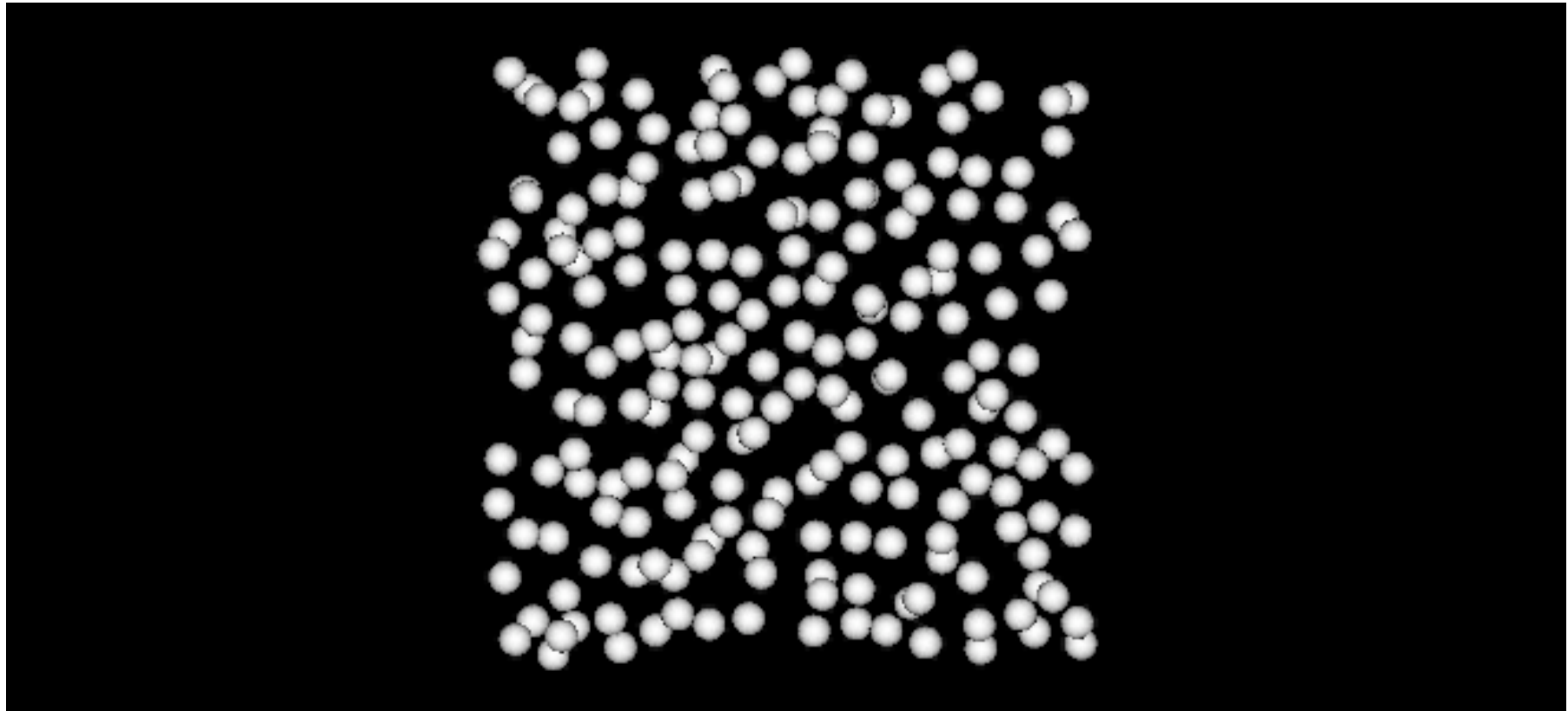
Sample : monodisperse silica spheres in rubber



Courtesy to Dr.Hagita & Prof. Arai

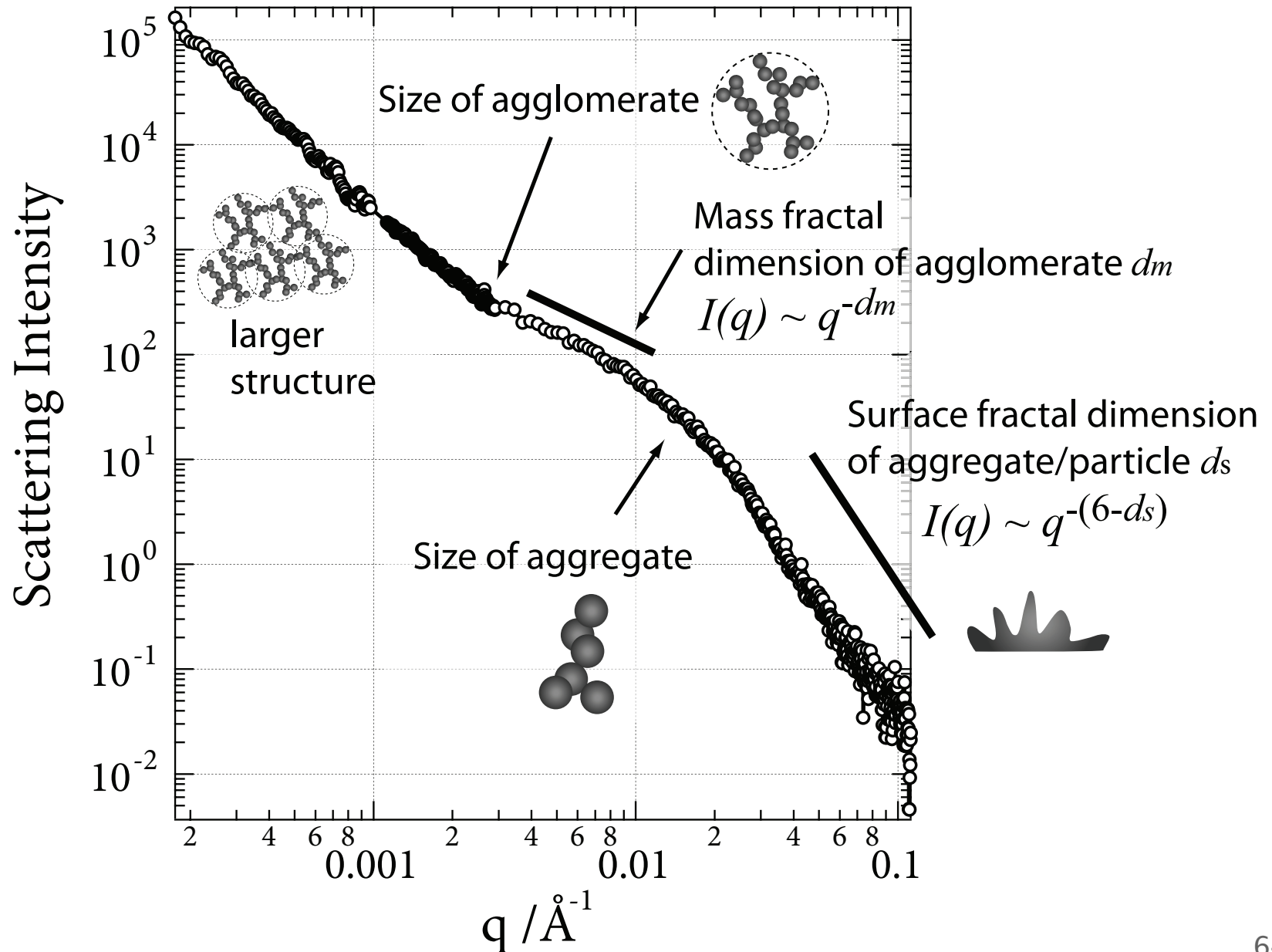
Visualization of structure change of fillers during elongation of rubber by using SAXS and RMC

0 → 150% : elongation process



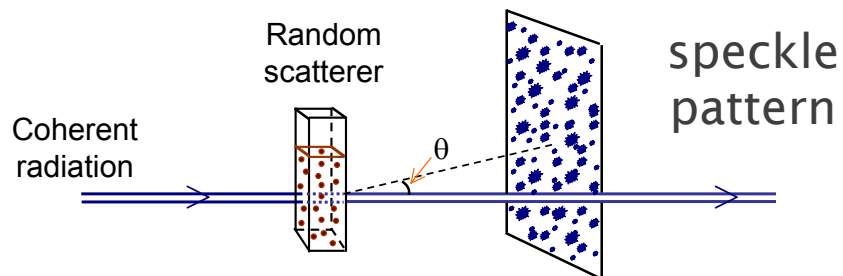
non-uniformity increases along the elongation direction.

Structural information from USAXS



X-ray Photon Correlation Spectroscopy: XPCS

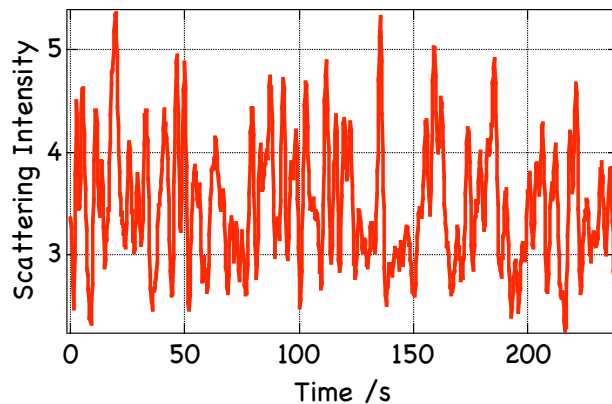
- Measurement of fluctuation of X-ray scattering intensity
--> Structural fluctuation in sample



$$g^{(2)}(q, \tau) = \frac{\langle I(q, 0)I^*(q, \tau) \rangle}{\langle I(q) \rangle^2}$$

Time-resolved SAXS with coherent X-ray

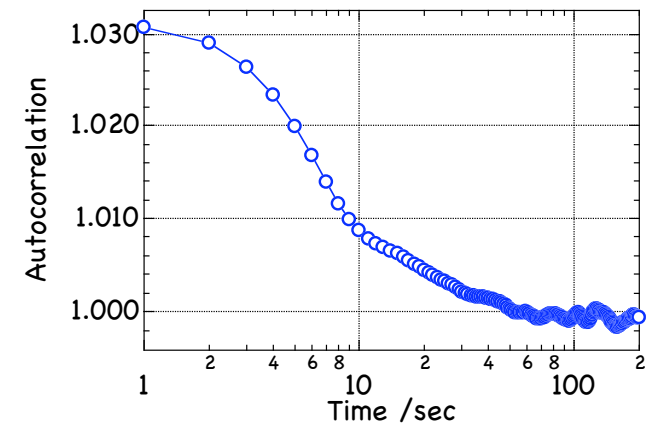
Fluctuation of intensity



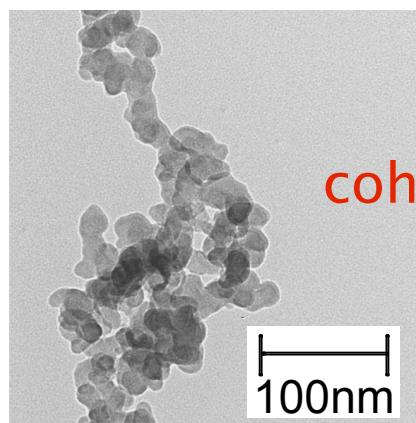
Autocorrelation



relaxation time in system

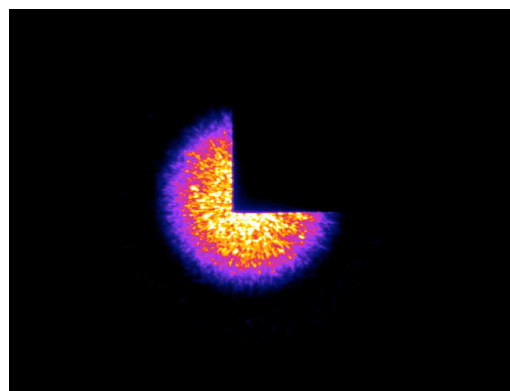


Dynamics of nanoparticles observed with XPCS

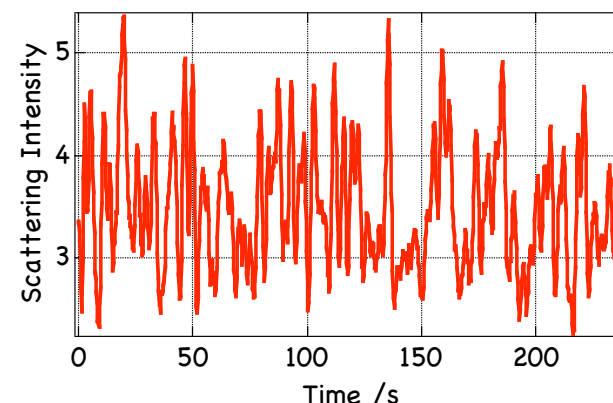


nano-particles in rubber

coherent x-ray



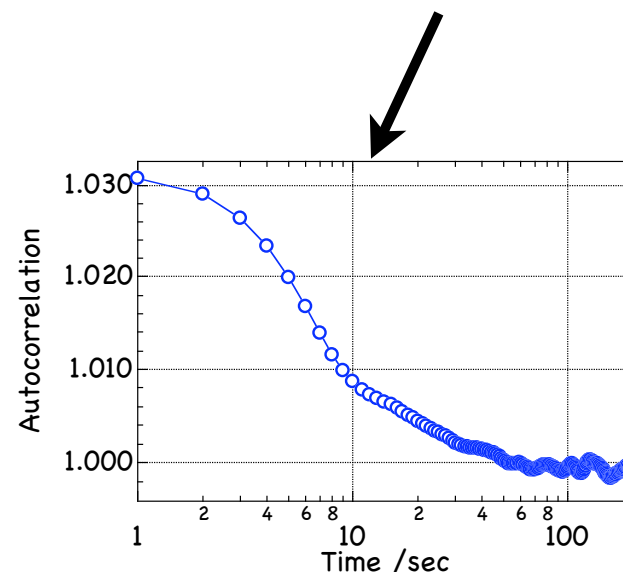
speckle pattern



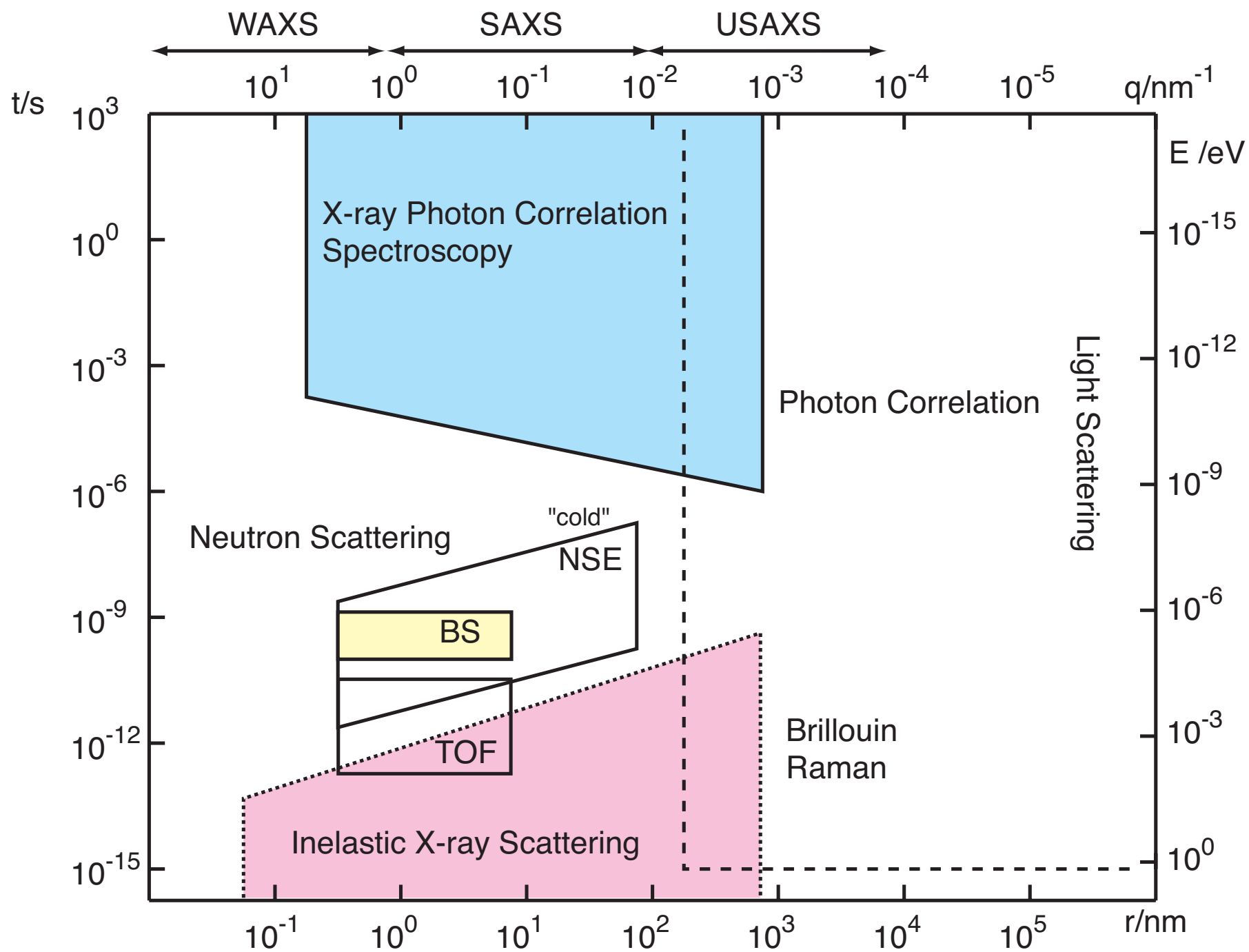
fluctuation of scattering intensity

Dependence of dynamics on...

- Volume fraction of nano-particles
- Vulcanization (cross-linking)
- Type of nano-particles
- Temperature etc.



Dynamics of Filler in Rubber



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