

Small-Angle X-ray Scattering

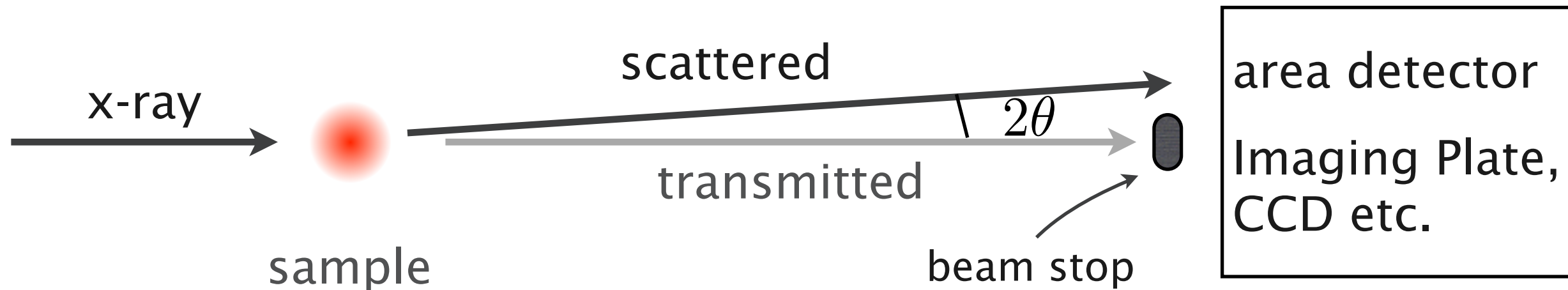
Basics & Applications

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The University of Tokyo

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What's Small-Angle X-ray Scattering ?



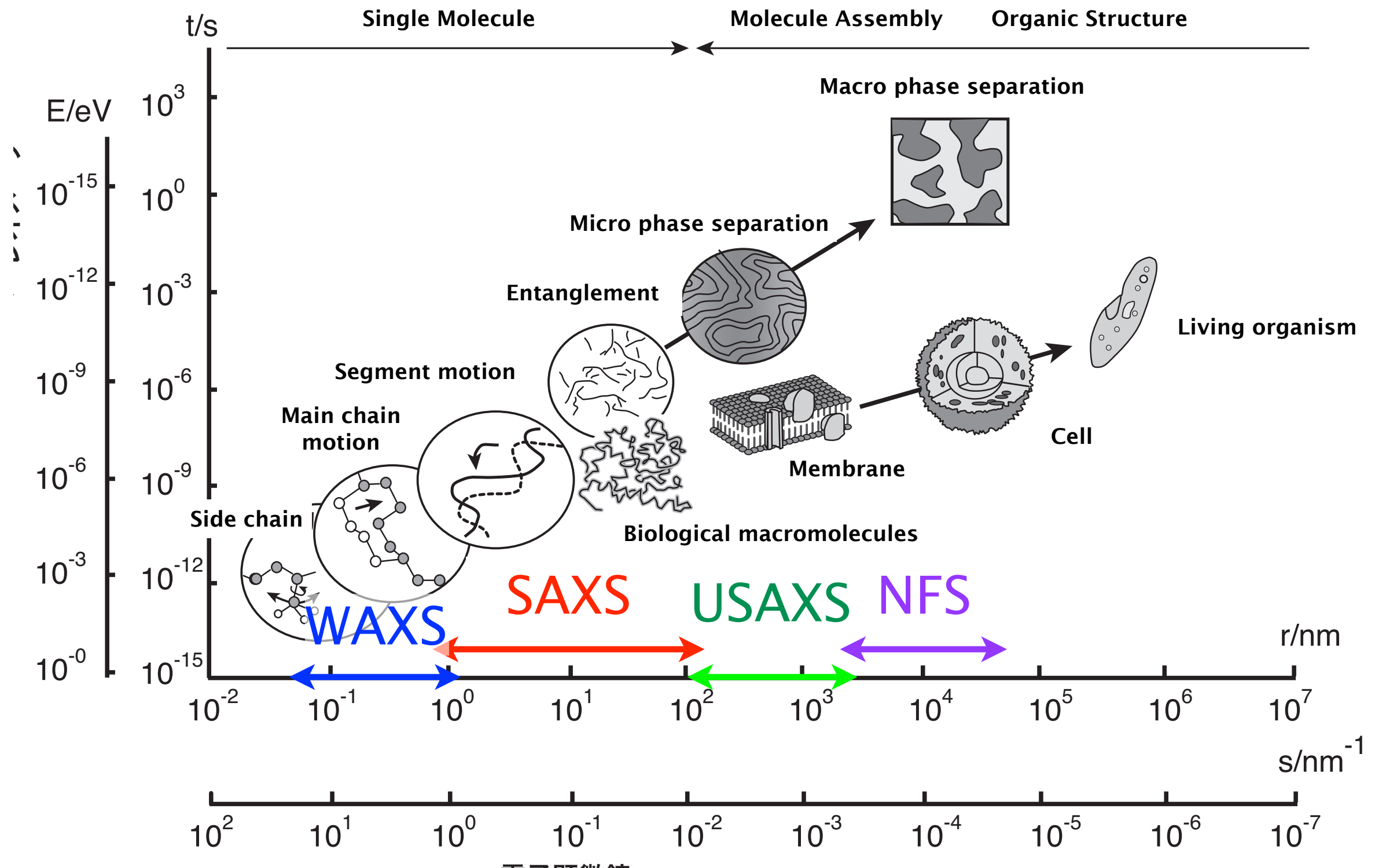
Bragg's law: $\lambda = 2d \sin \theta$

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

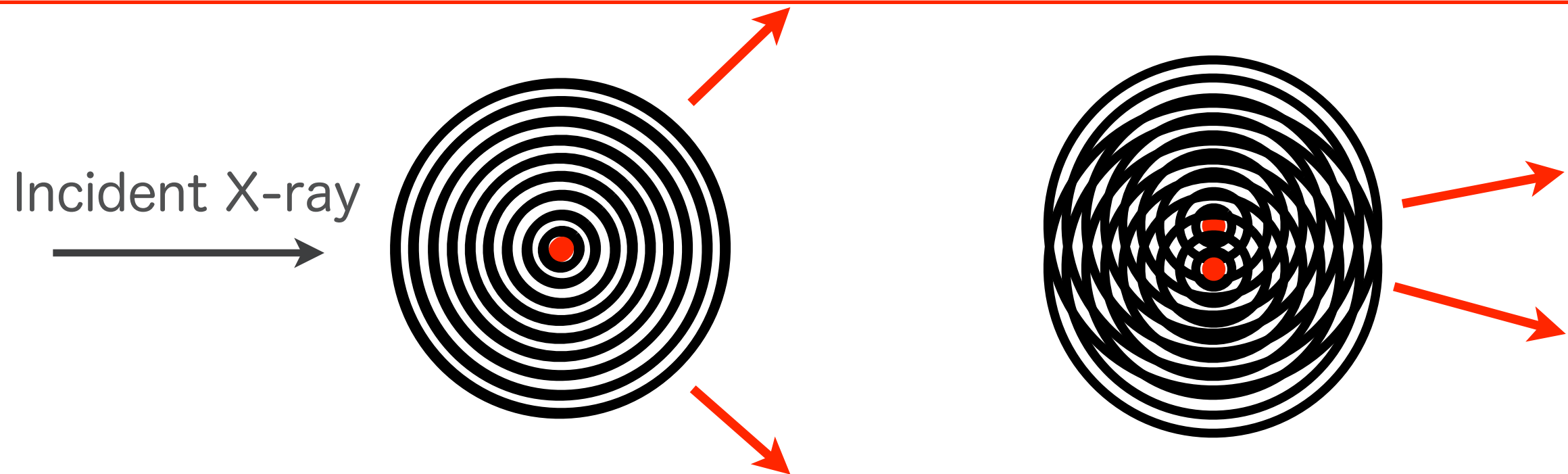
crystalline sample --> small-angle X-ray diffraction: SAXD

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 -->

History of SAXS (before 1936)

Krishnamurty (1930)

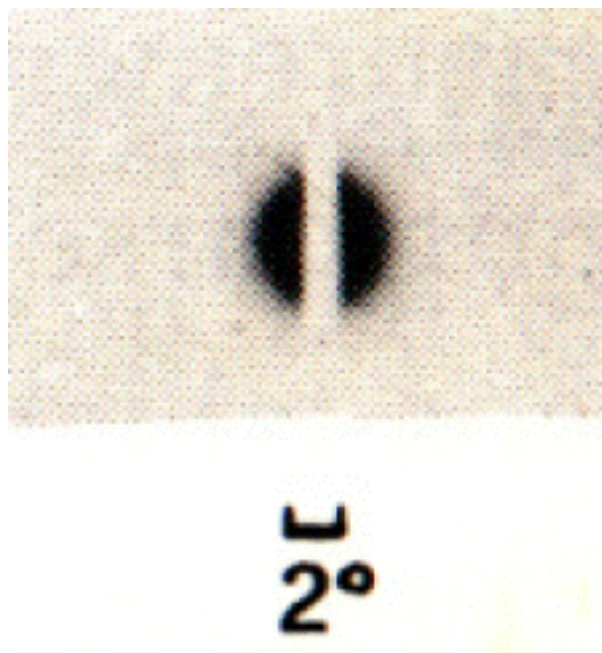
Hendricks (1932)

Mark (1932)

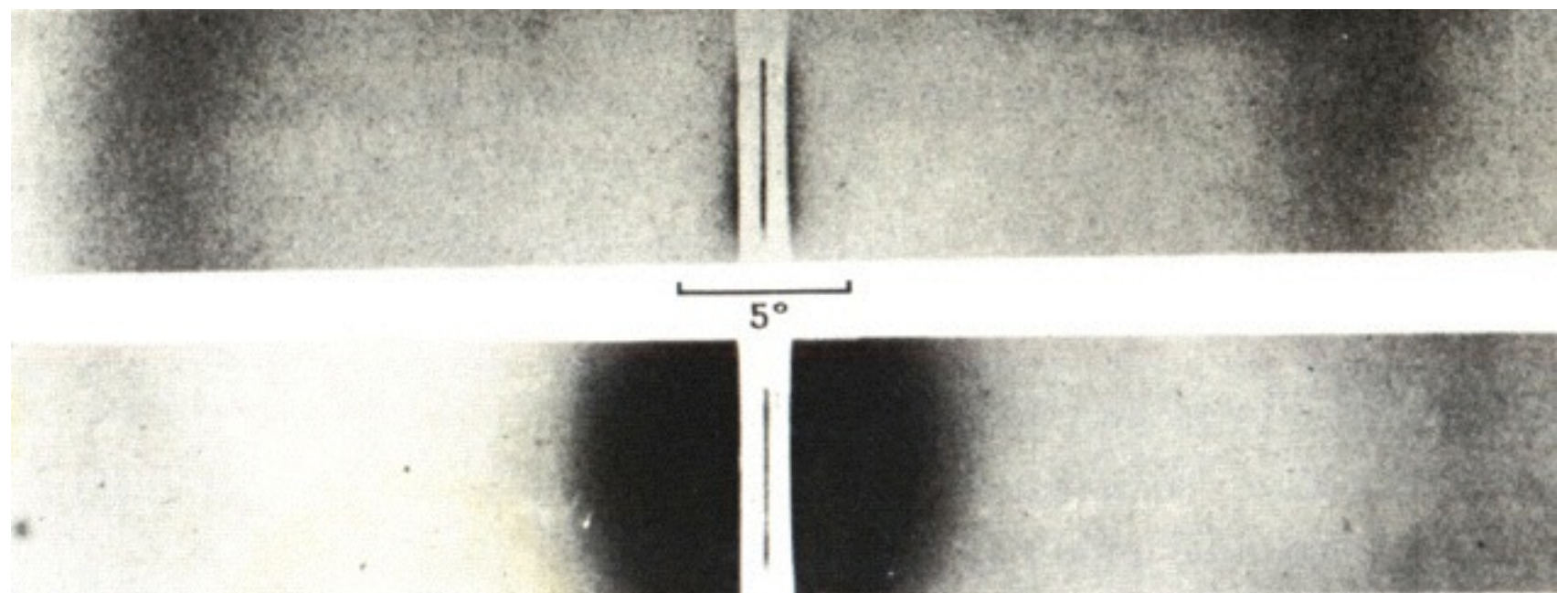
Warren (1936)

Observation of scattering

from powders, fibers, and colloidal dispersions

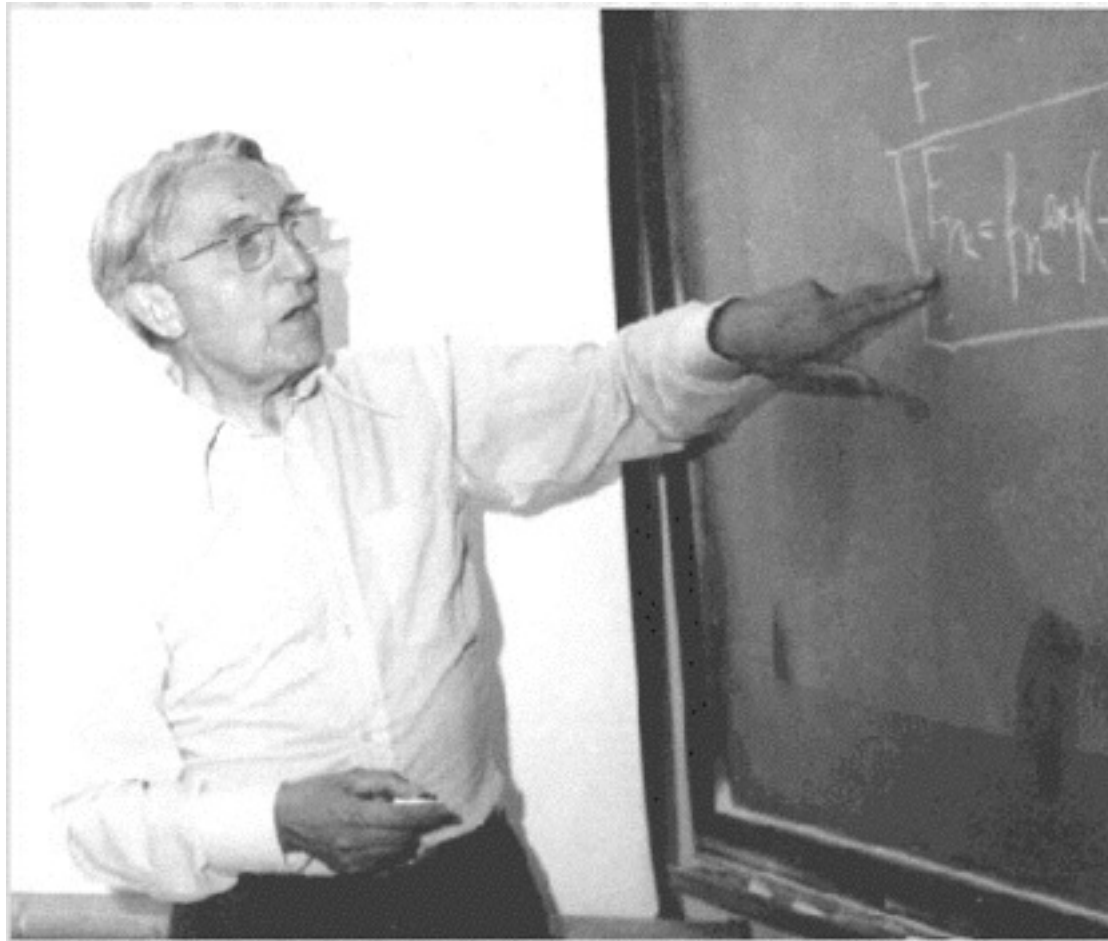


carbon black



Molten silica - silica gel
(above) (below)

History of SAXS (after 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)

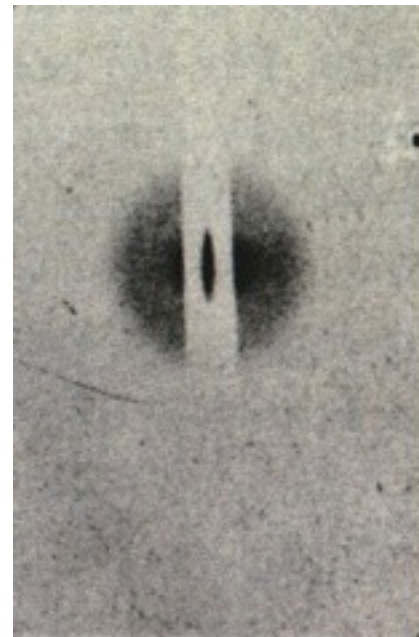
G. Polod (1942, 1960, 1961)

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

Macromolecules in solution.



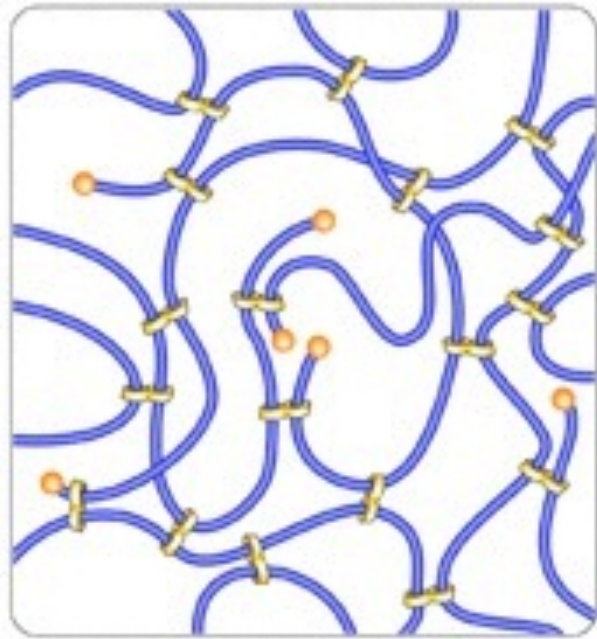
Single crystals of Al-Cu hardened alloy



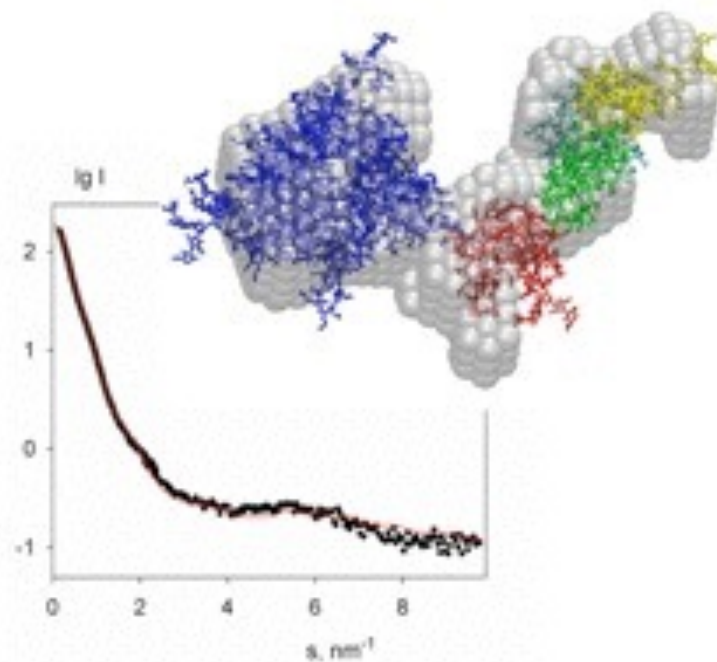
Hemoglobin

courtesy to Dr. I.L.Torriani

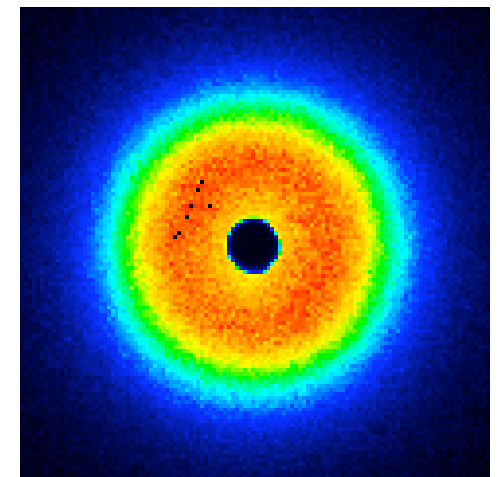
Application of SAXS



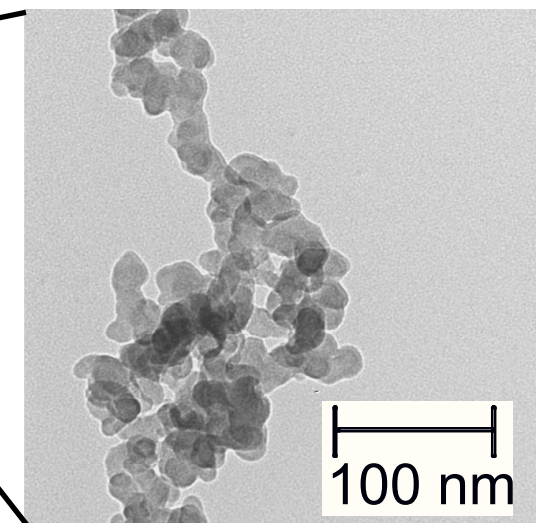
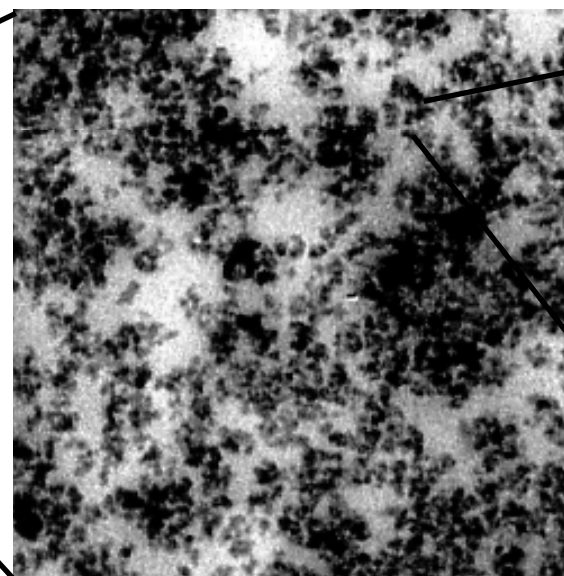
gel



Proteins in solution (Dr. Svergun, EMBL)



Typical SAXS image

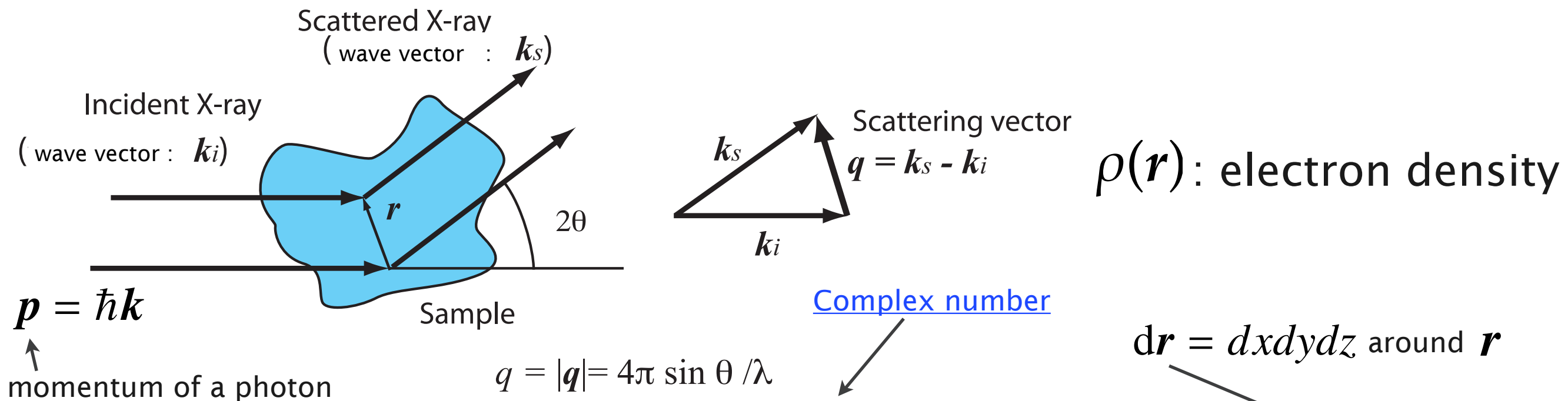


Nanocomposite

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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 : $I(\mathbf{q}) = A(\mathbf{q})A^*(\mathbf{q}) = |A(\mathbf{q})|^2$

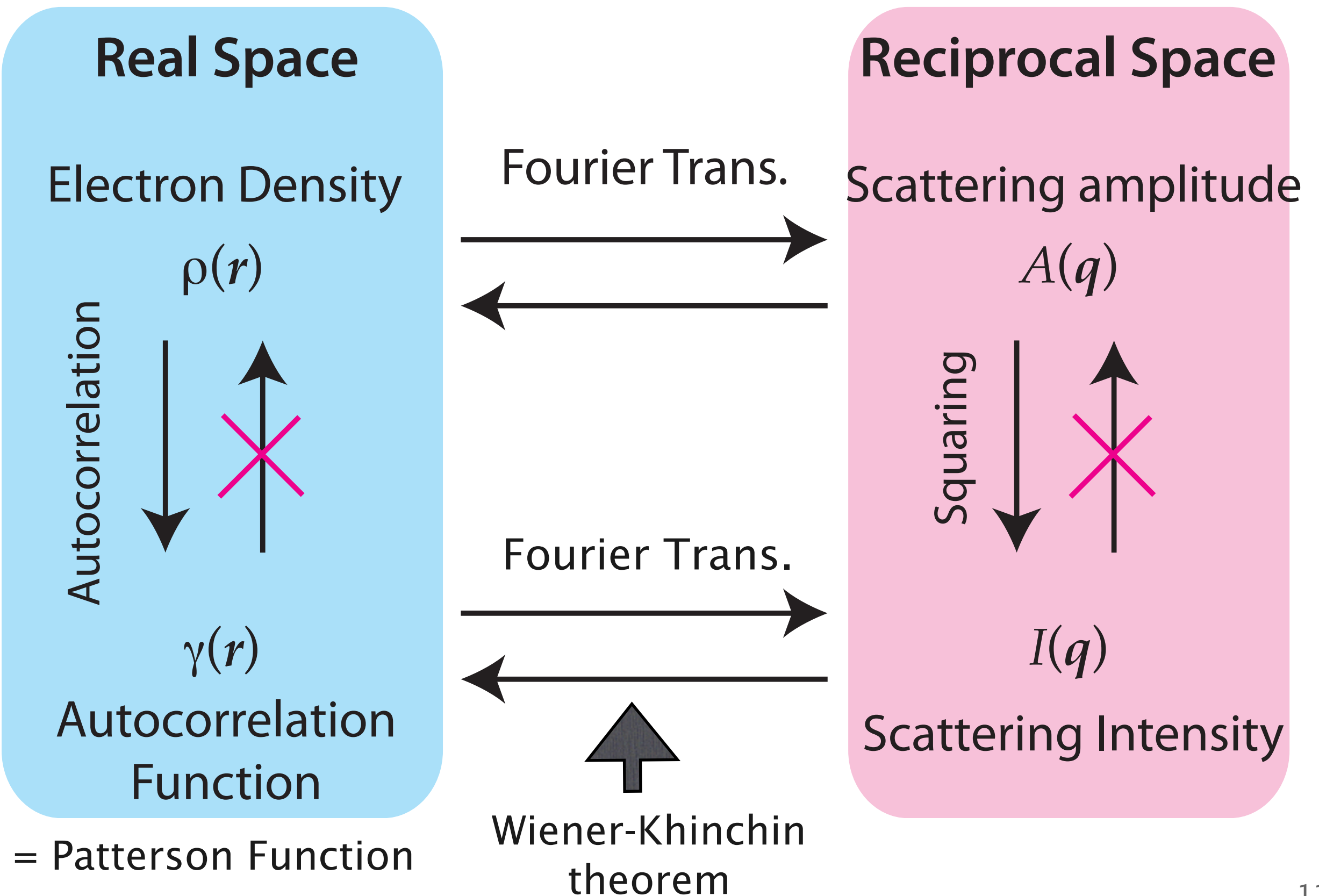
(Extensive variable)

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

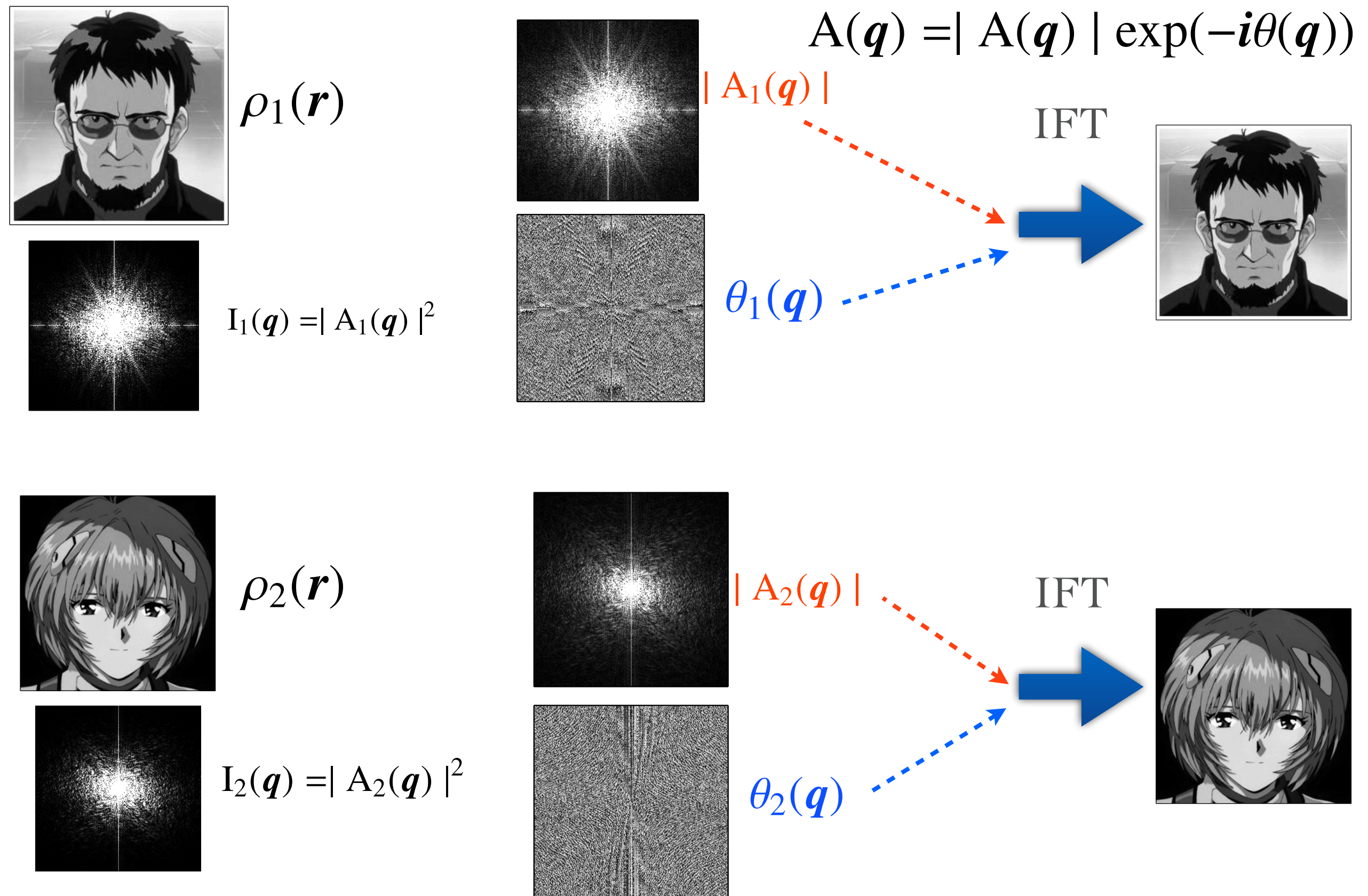
(Intensive variable)

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

Real space and Reciprocal Space

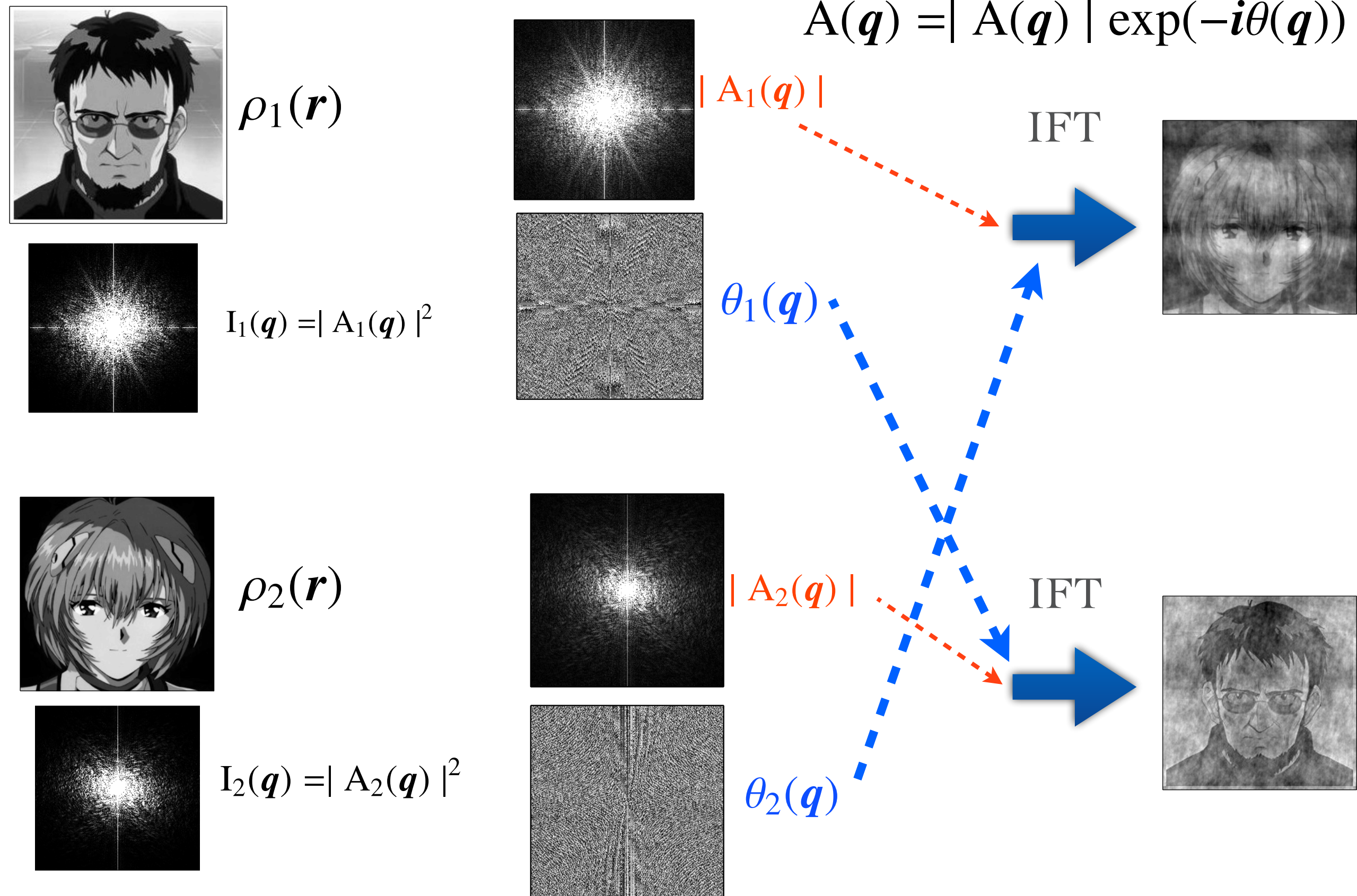


Importance of phase, $\theta(\mathbf{q})$ of complex amplitude



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

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



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

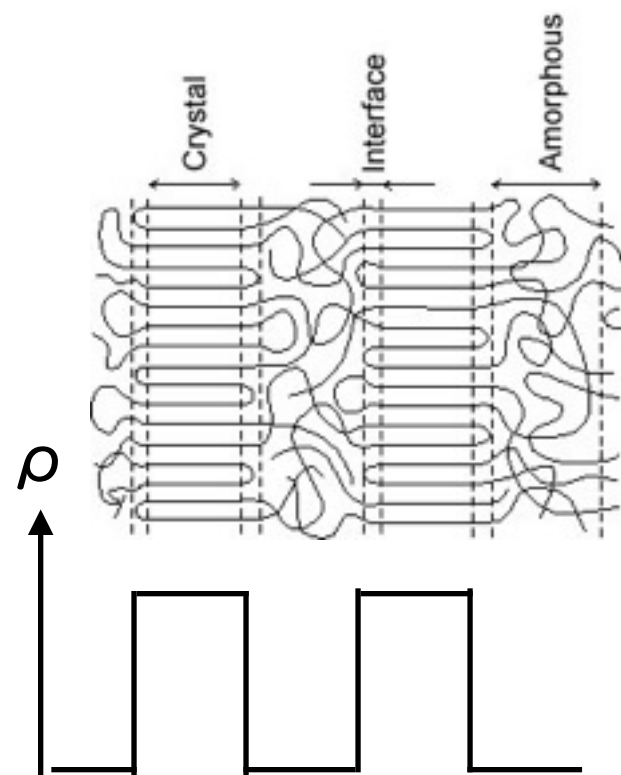
$$\gamma(0) = \langle \rho^2 \rangle \qquad \gamma(\infty) = \langle \rho \rangle^2$$

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: in case of lamellar



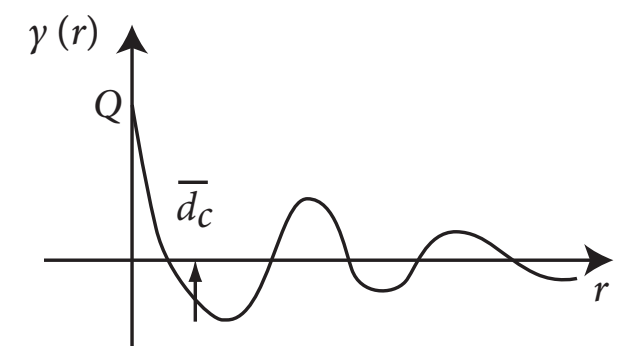
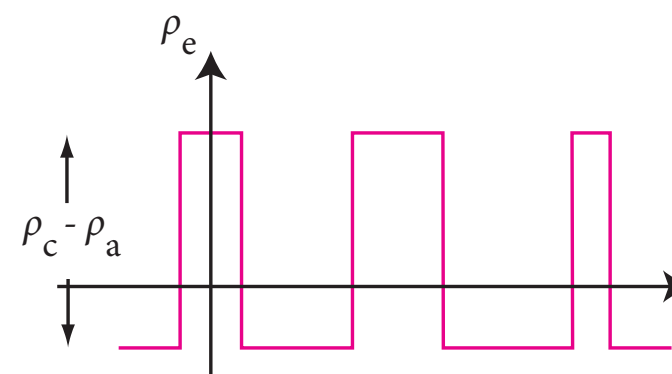
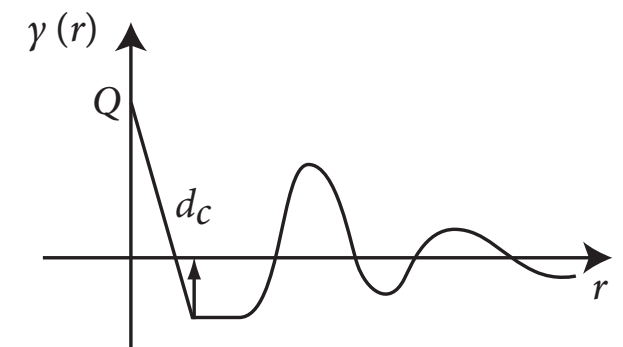
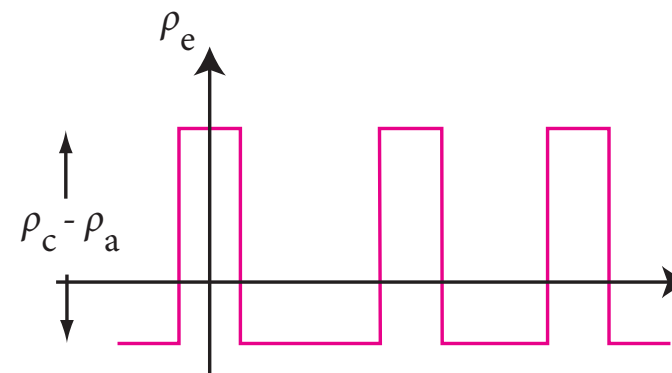
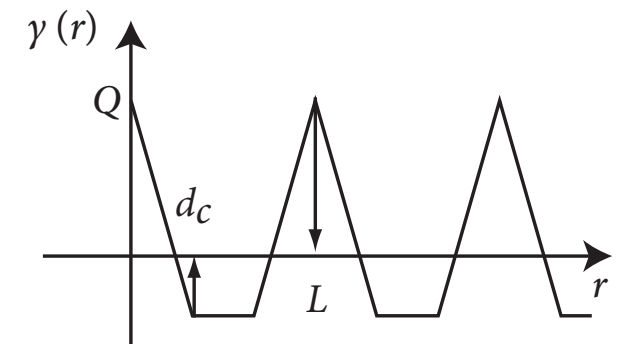
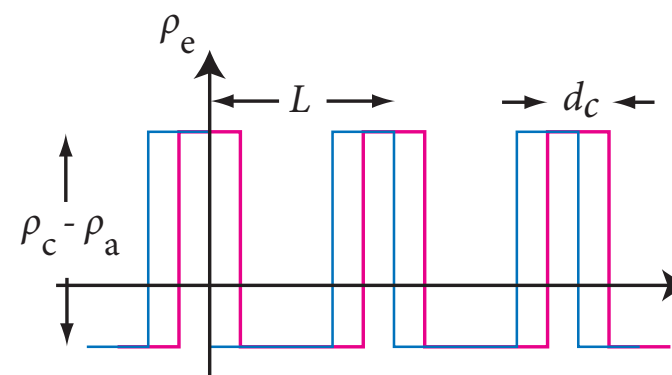
ideal ordering

Long period changes.

Thickness of crystal changes.

$\rho(r)$

$\gamma(r)$



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, $\gamma_0(\mathbf{r})$

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


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

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

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

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

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

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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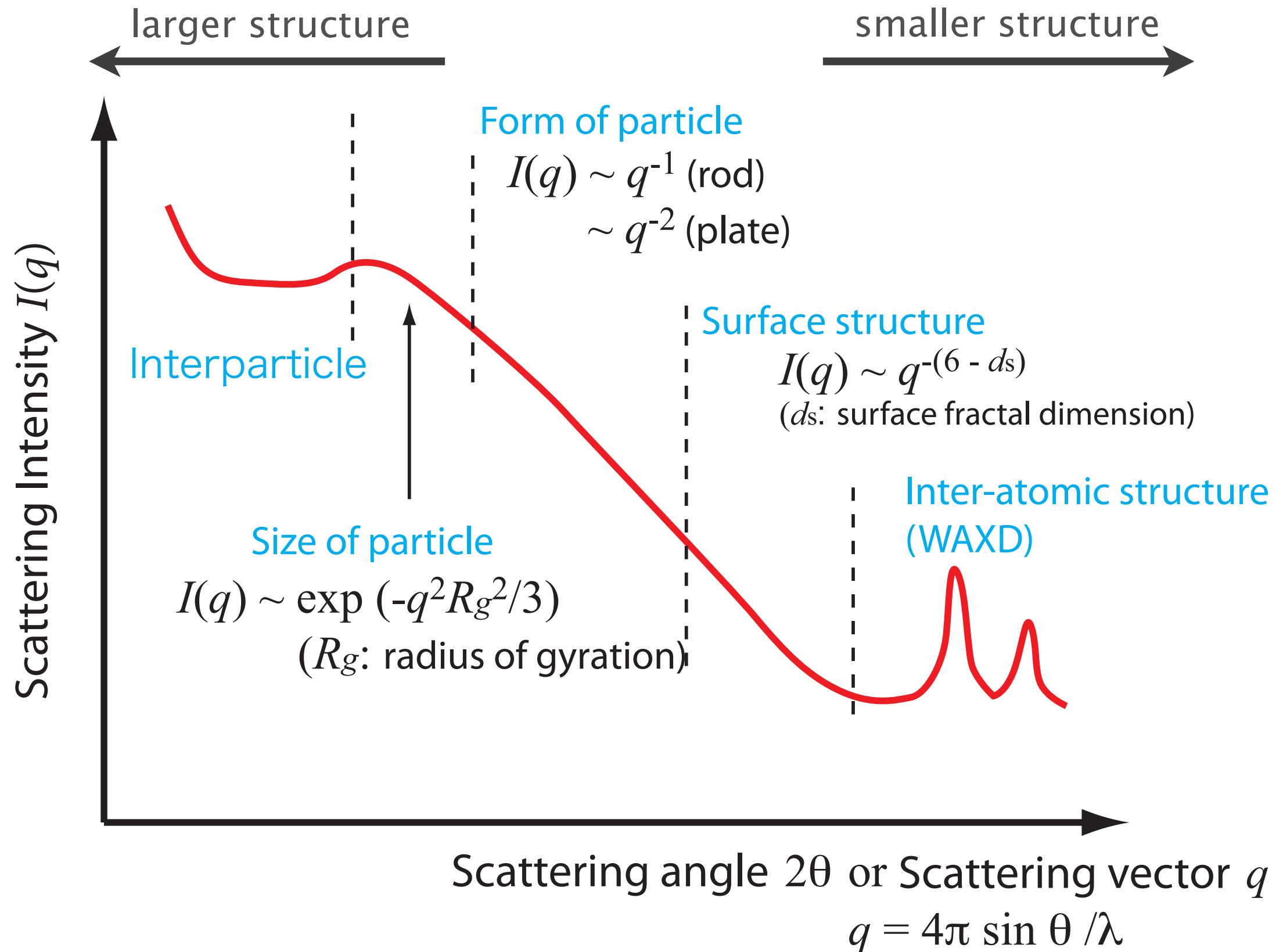
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Information obtained from SAXS

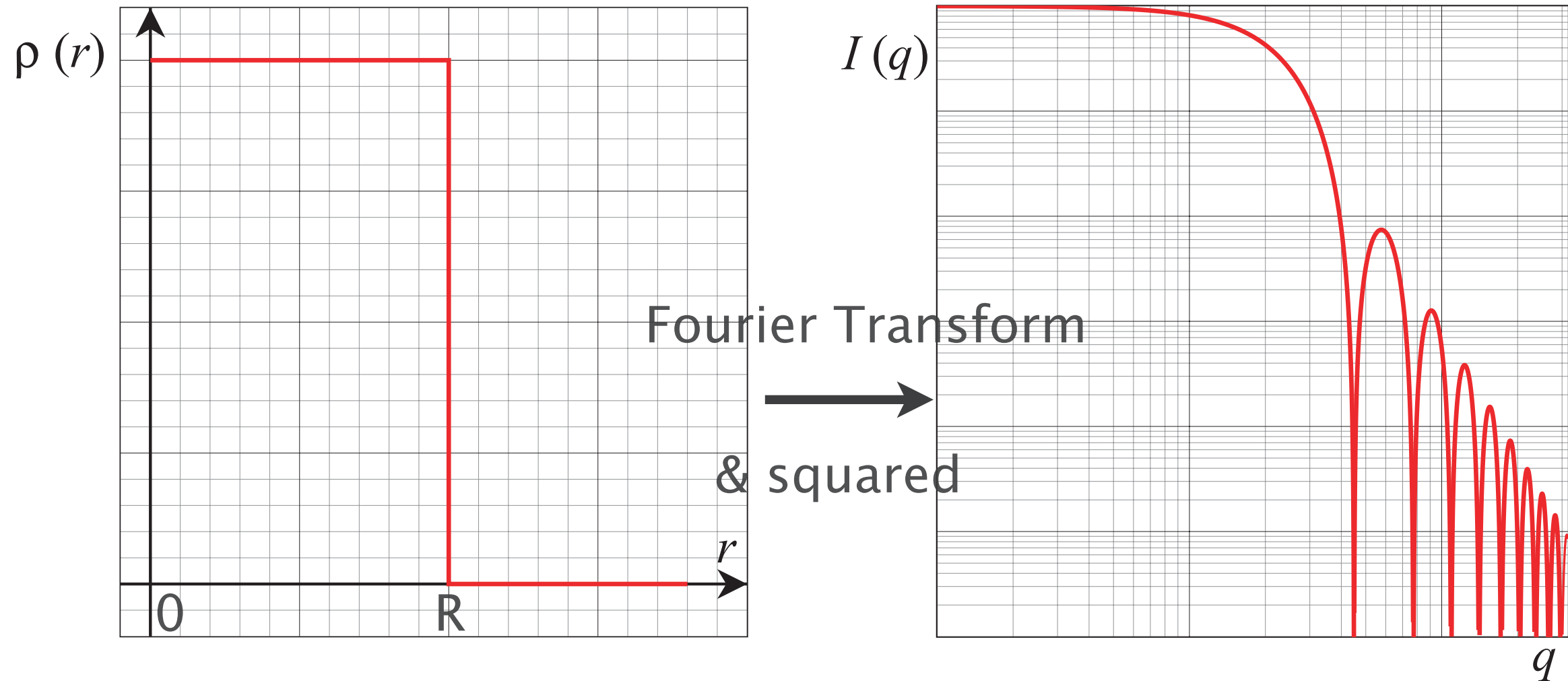
1. Size and form of particulate system
 - ❧ Colloids, Globular proteins, etc...
2. Correlation length of inhomogeneous structure
 - ❧ Polymer chain, two-phase system etc.
3. Lattice parameters of distorted crystals (para-crystal)
4. Degree of crystallinity, crystal size, crystal distortion
 - ❧ Crystalline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

SAXS of particulate system

Relation between SAXS pattern and Structural information



Spherical sample

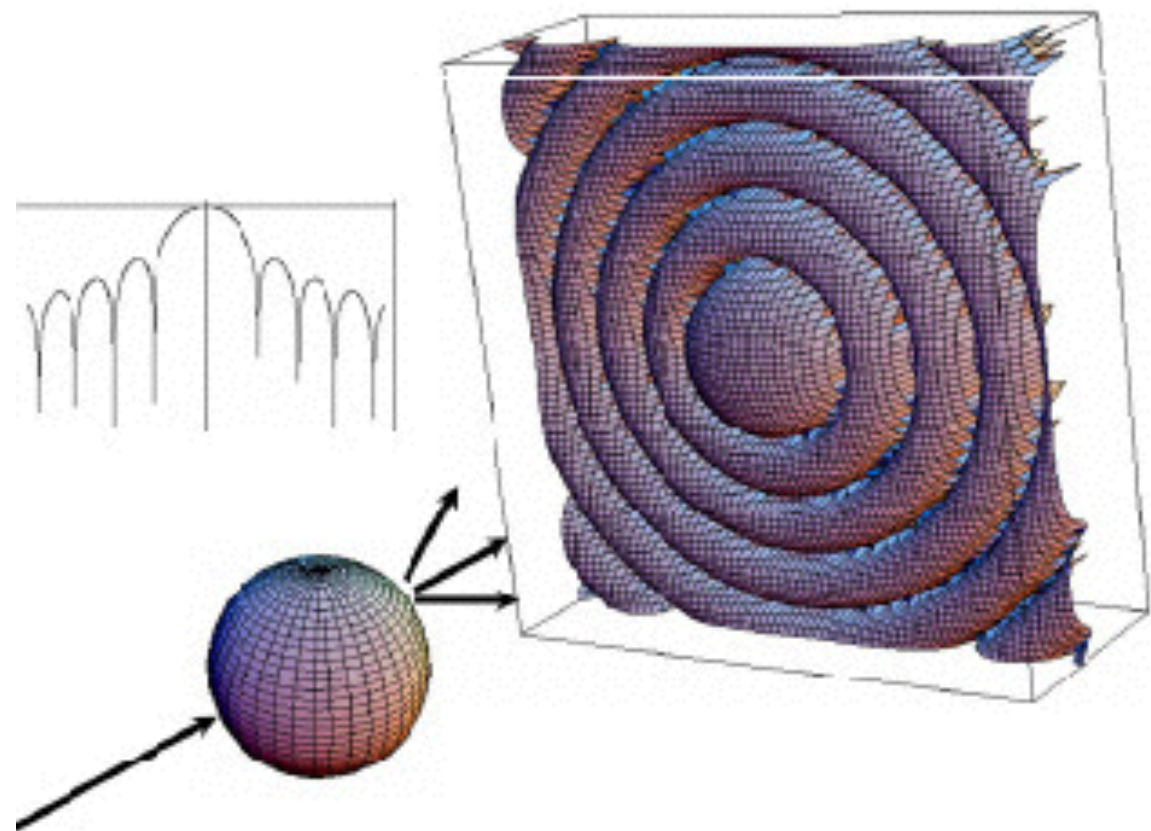
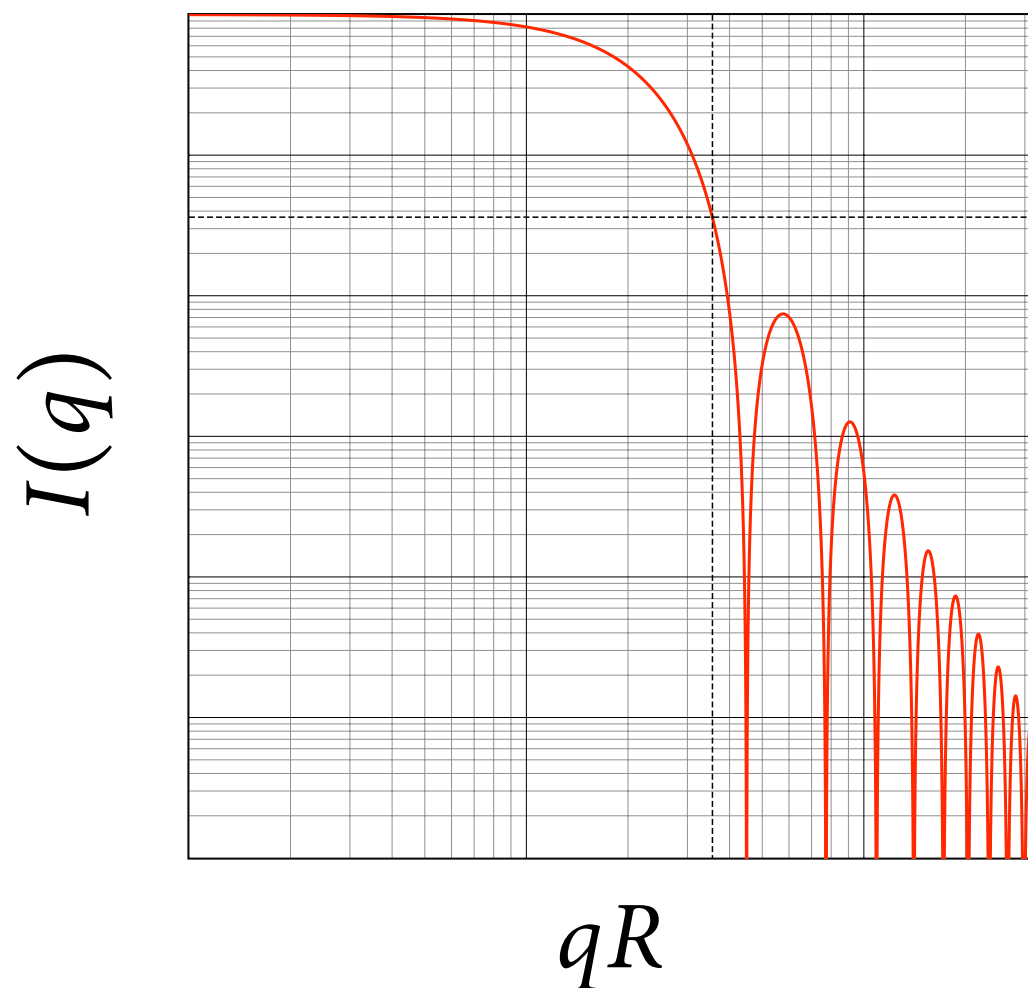


$$\rho(r) = \begin{cases} \Delta\rho & r < R \\ 0 & \text{else} \end{cases}$$

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

Homogeneous sphere

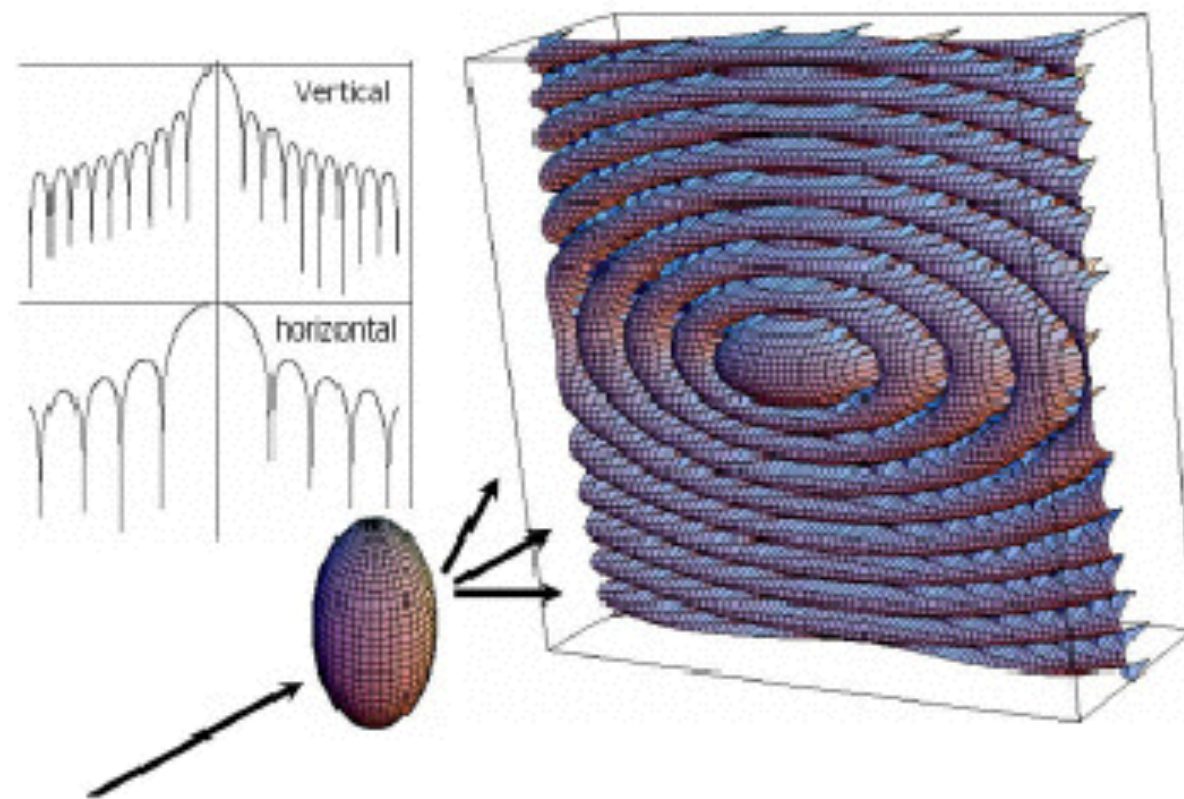
$$I(q) = \frac{(\Delta\rho)^2 V_{\text{particle}}^2}{V} \left[3 \frac{\sin qR - qR \cos qR}{(qR)^3} \right]^2$$



isotropic scattering

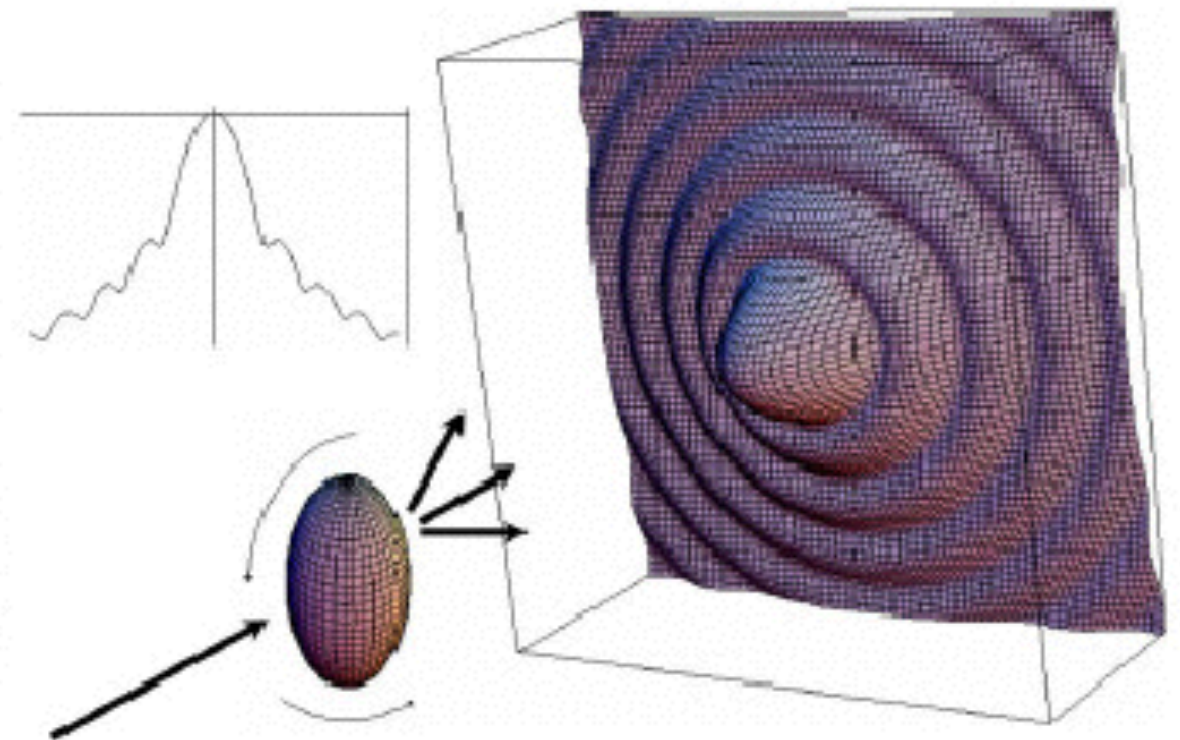
Homogeneous elipsoid

Fixed particle



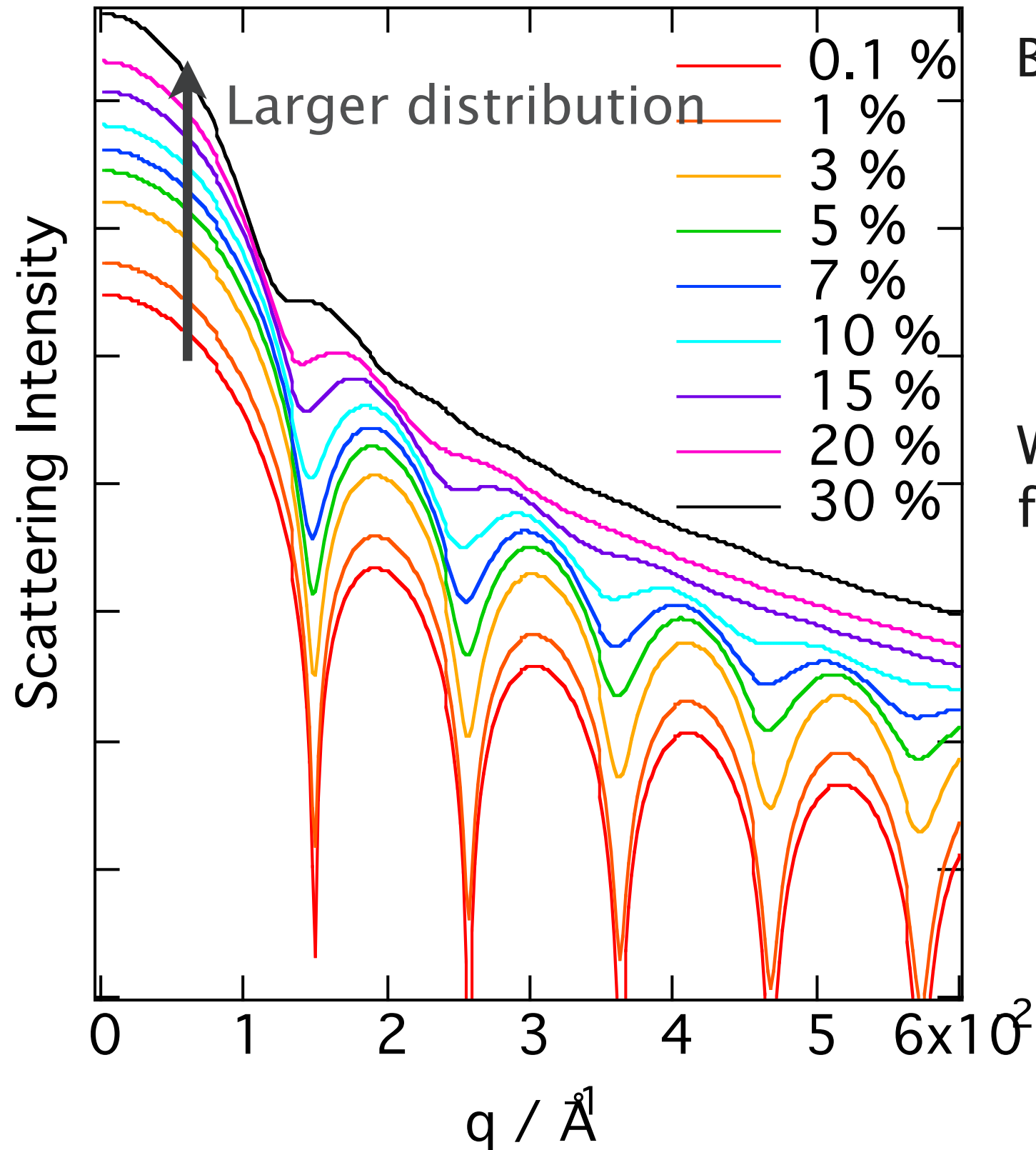
anisotropic scattering

Random orientation



isotropic scattering

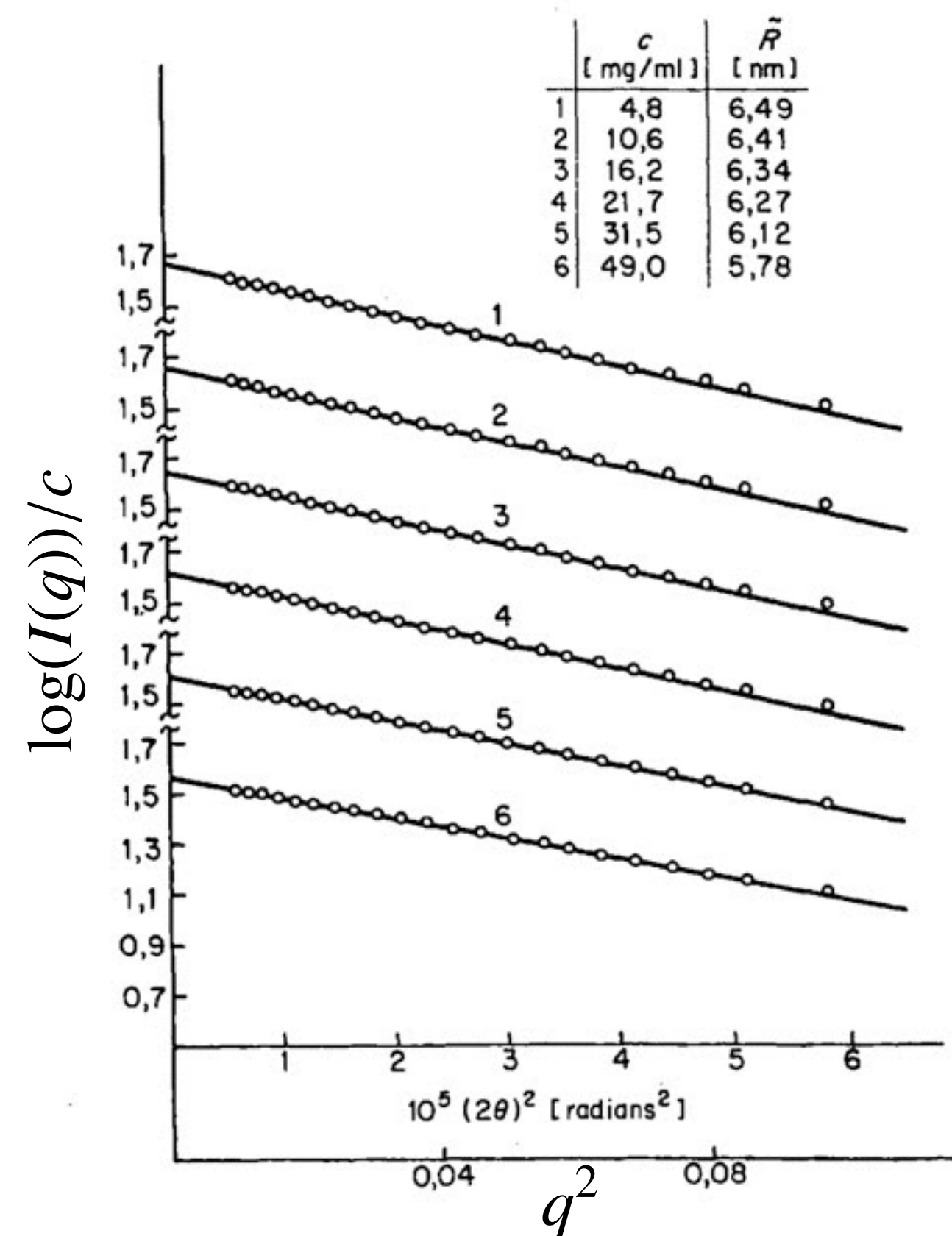
Size distribution



Based on Gaussian distribution

When the form has distribution, fringes are missed.

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



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

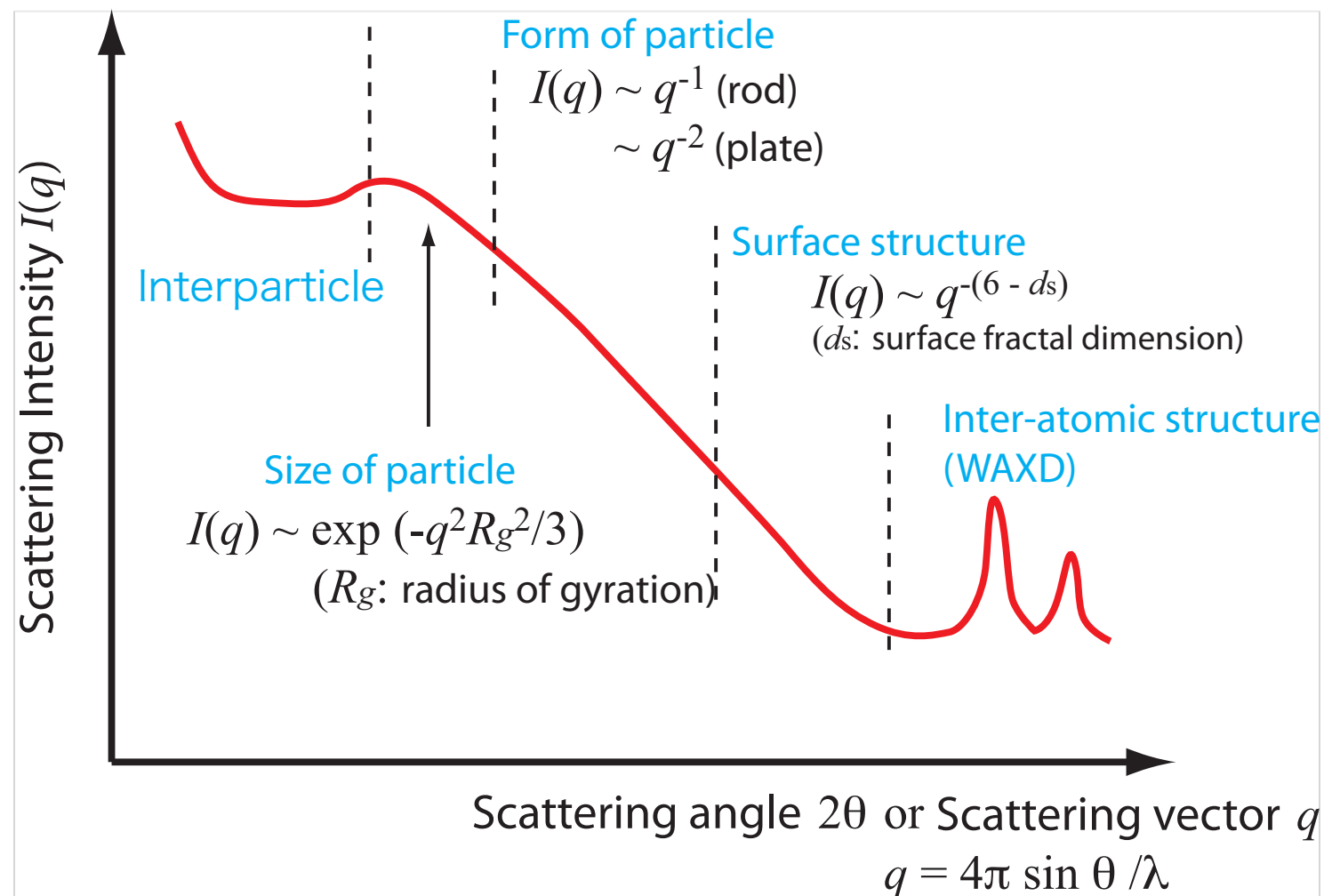
$$\log(I(q)) = -\frac{q^2 R_g^2}{3}$$

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

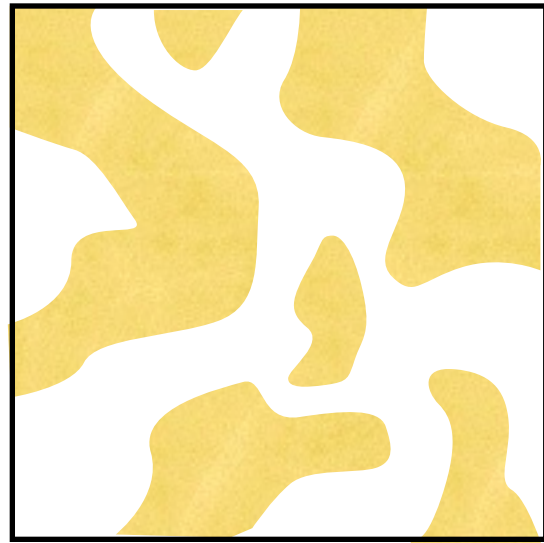
(especially, for non-crystalline sample)

Proposed remedy:

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

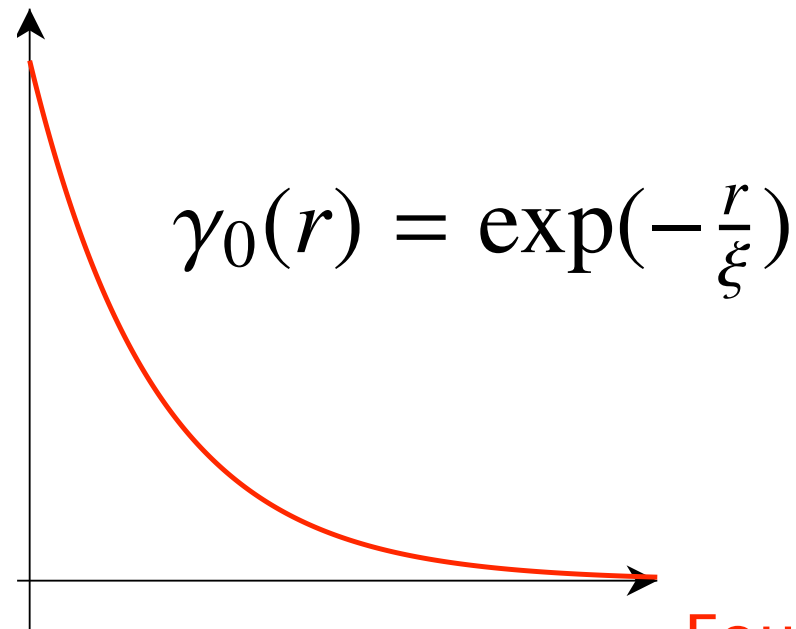
Scattering from Inhomogeneous Structure

Electron Density
 $\rho(r)$

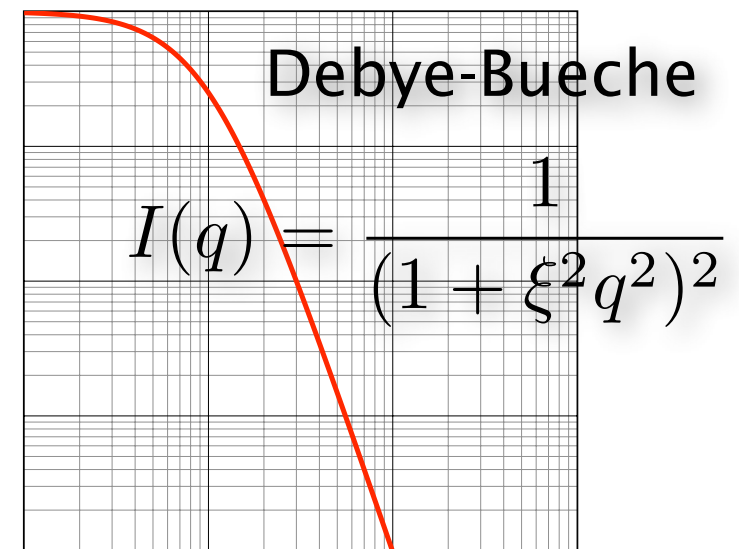


two phase system

Autocorrelation Function
 $\gamma_0(r)$



Scattering Intensity



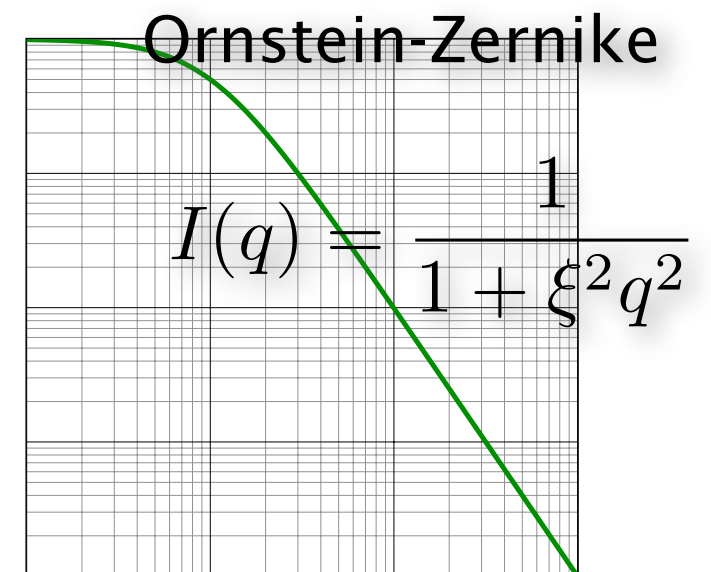
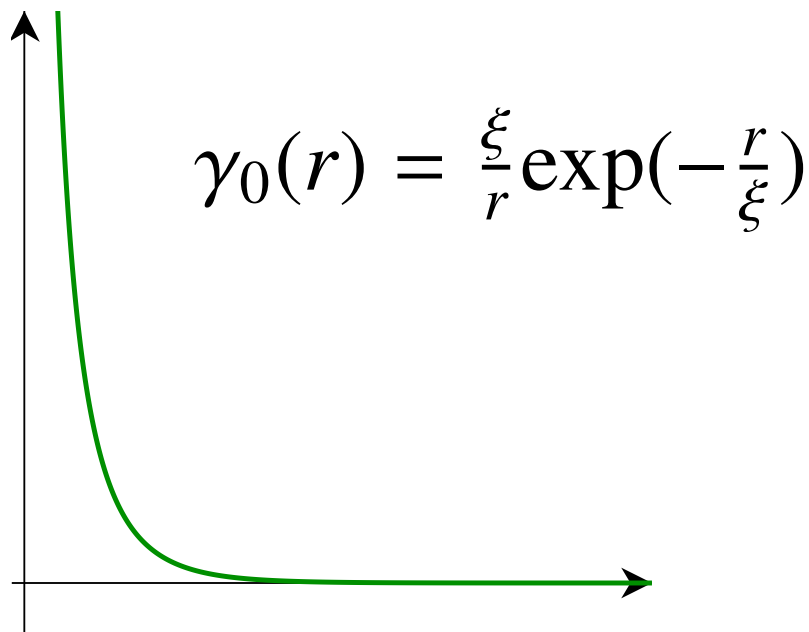
Autocorrelation



Fourier trans.



polymer chain etc.

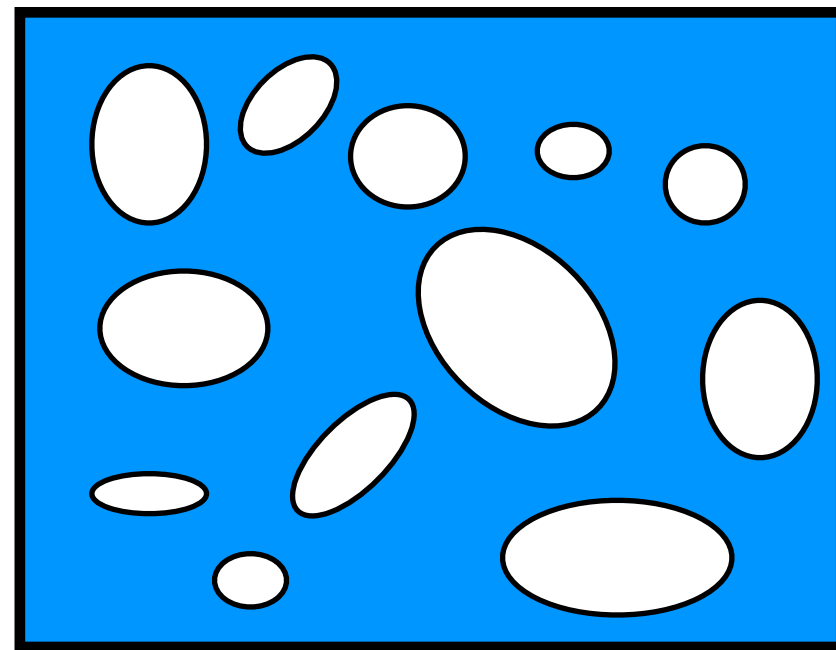
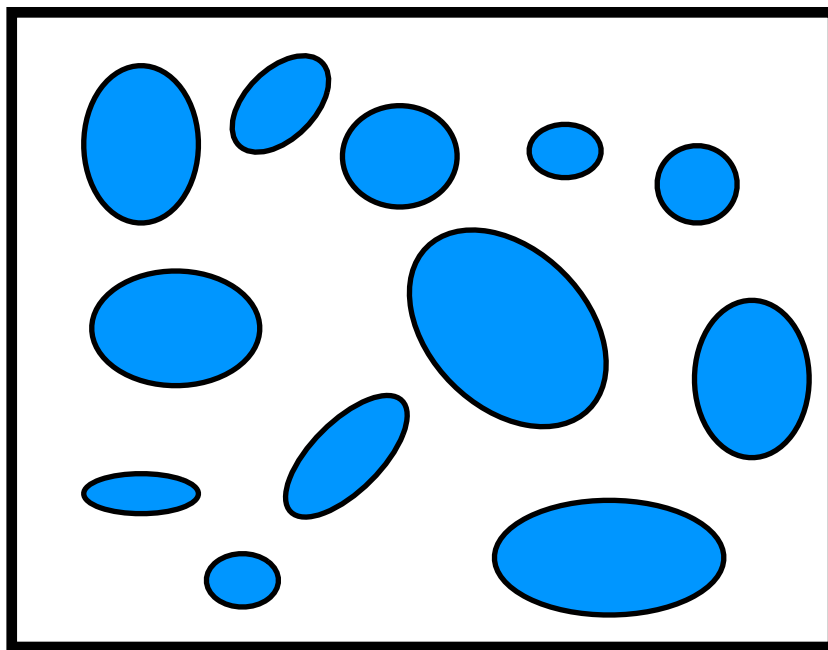


Two-phase system

Phase 1:

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

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



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

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

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

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

Invariant

only on the volume fraction and the contrast between the two phases.

Porod's law

For a sharp interface, the scattered intensity decreases as

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

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

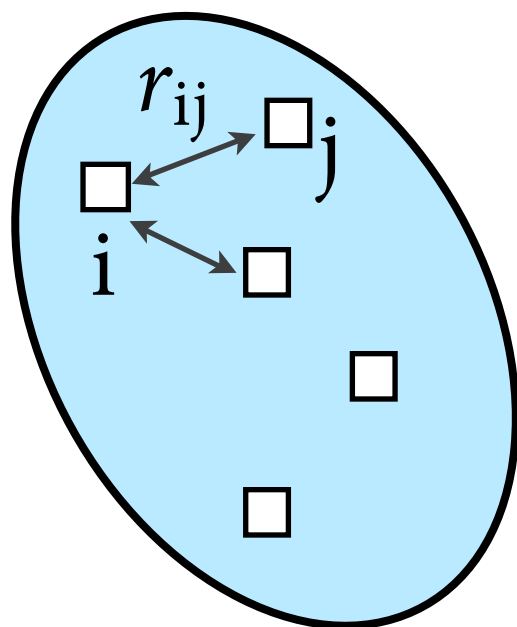
Pair Distance Distribution Function:

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

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

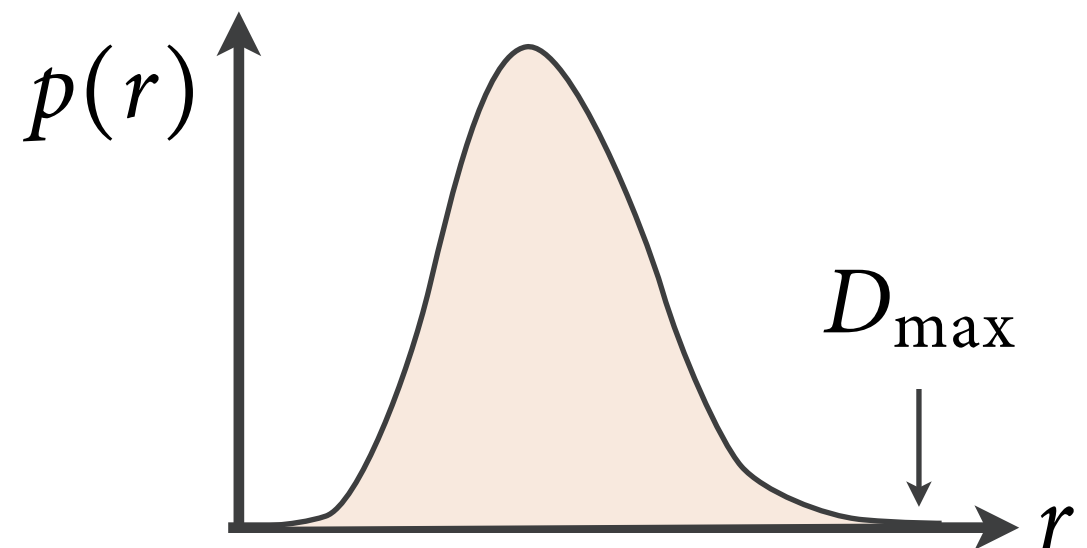
the set of distances joining the volume elements within a particle,
including the case of non-uniform density distribution.

Particle's SHAPE and maximum DIMENSION



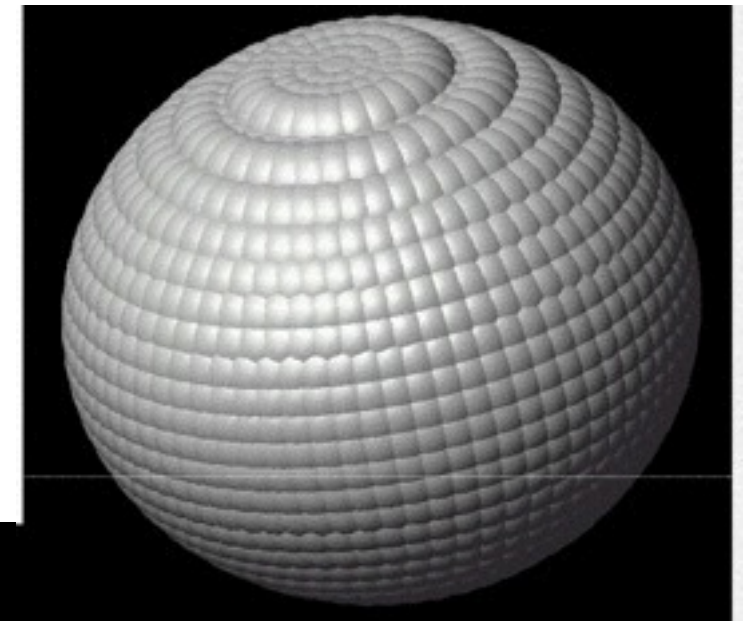
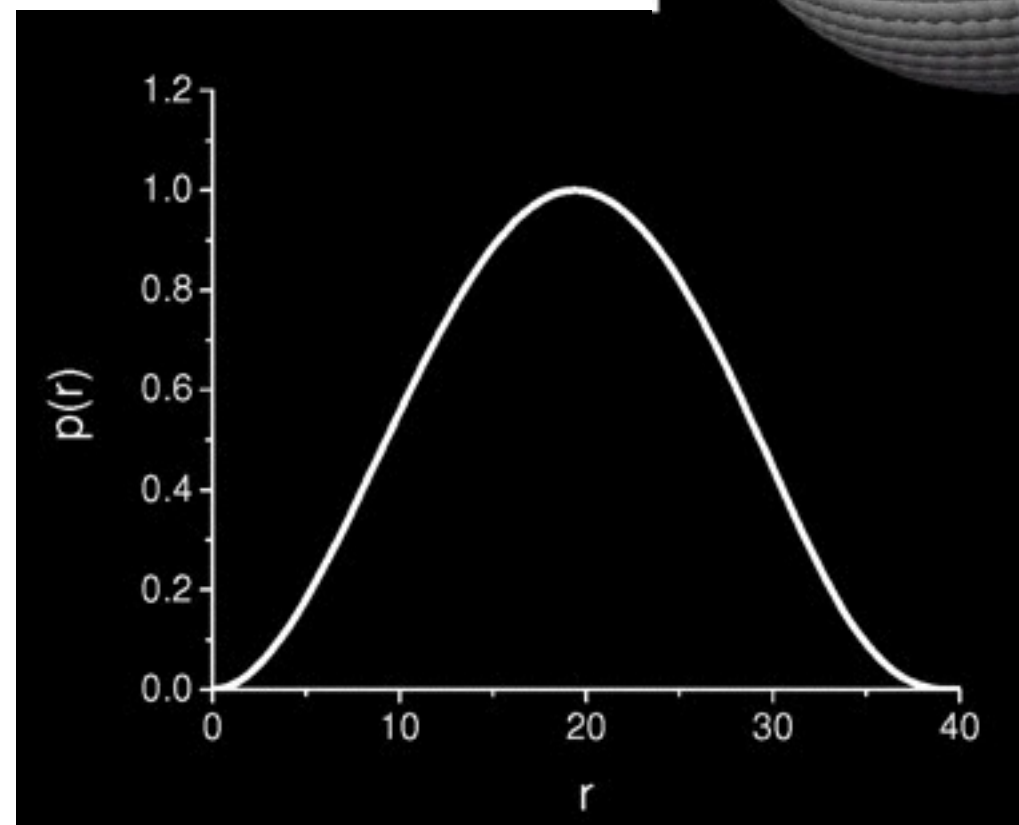
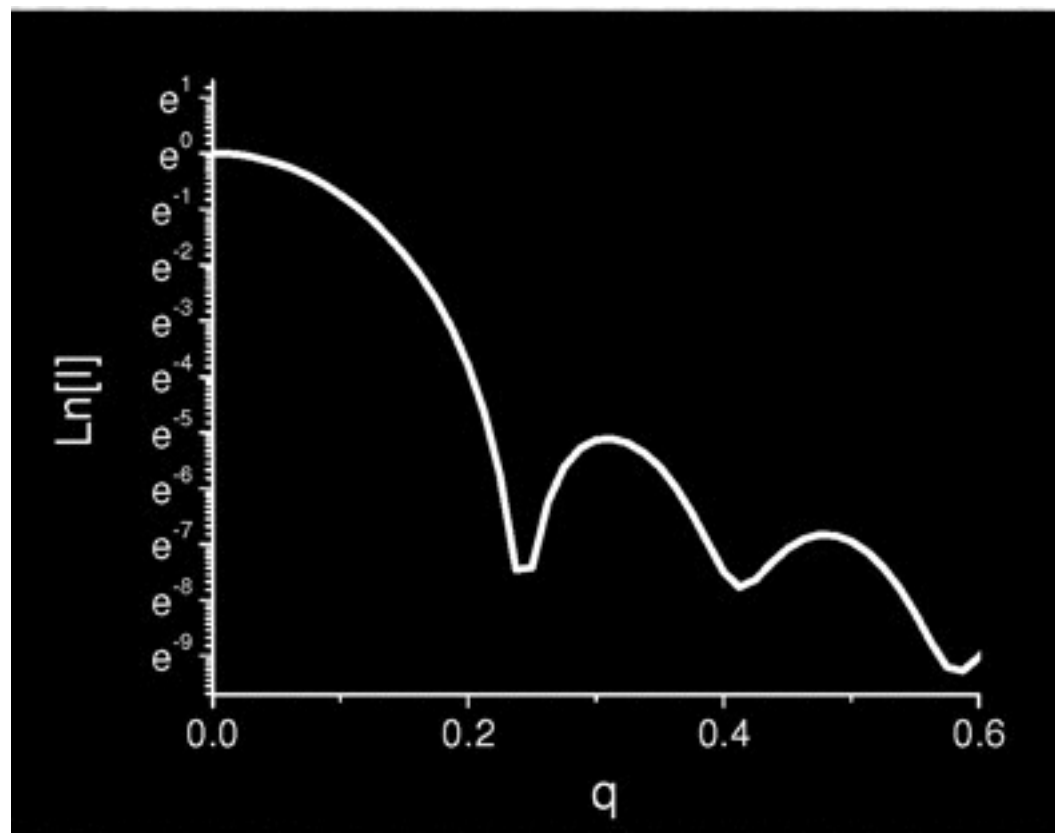
pairs of volume
elements i-j

histogram of all intra-particle distances

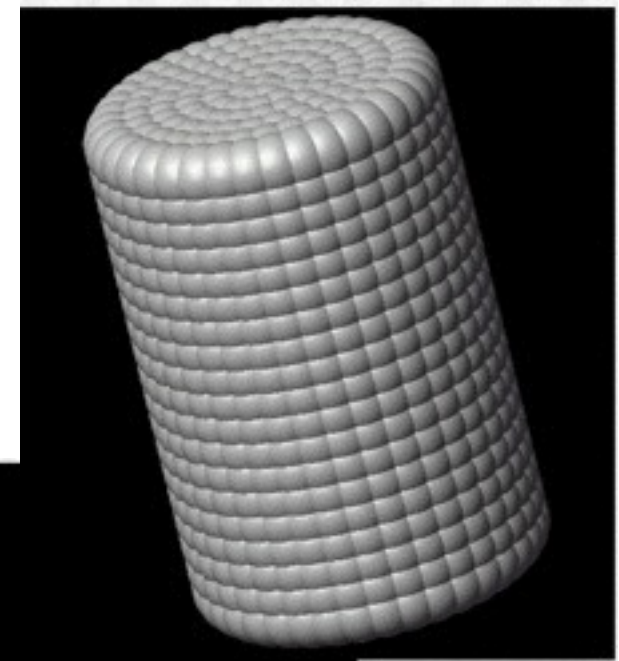
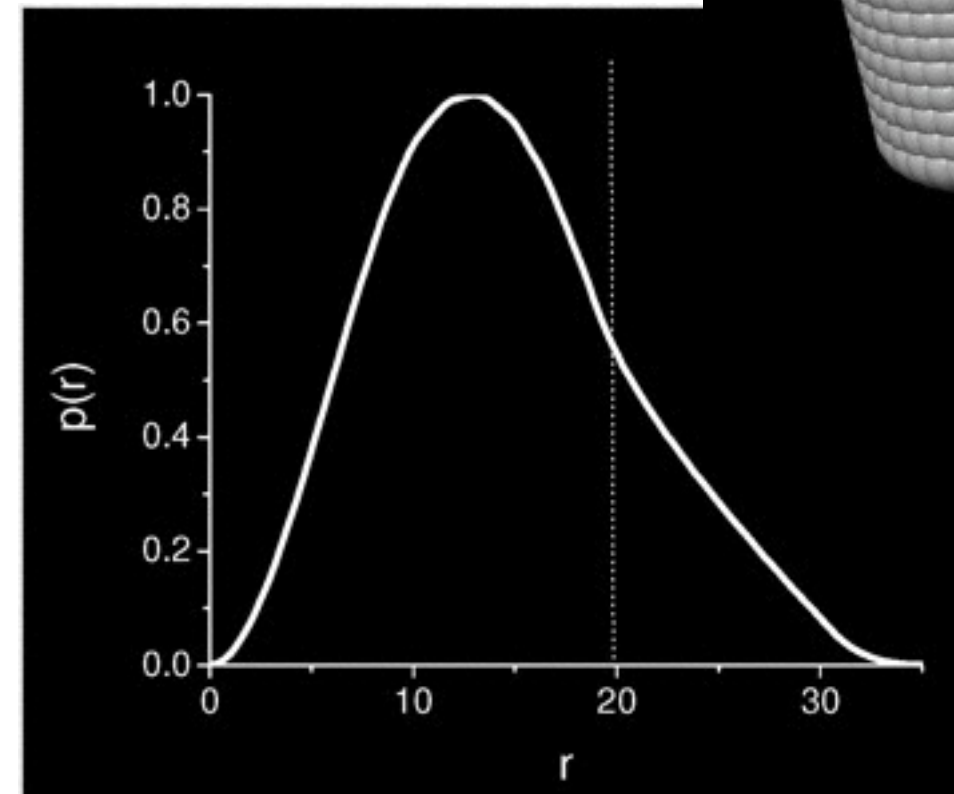
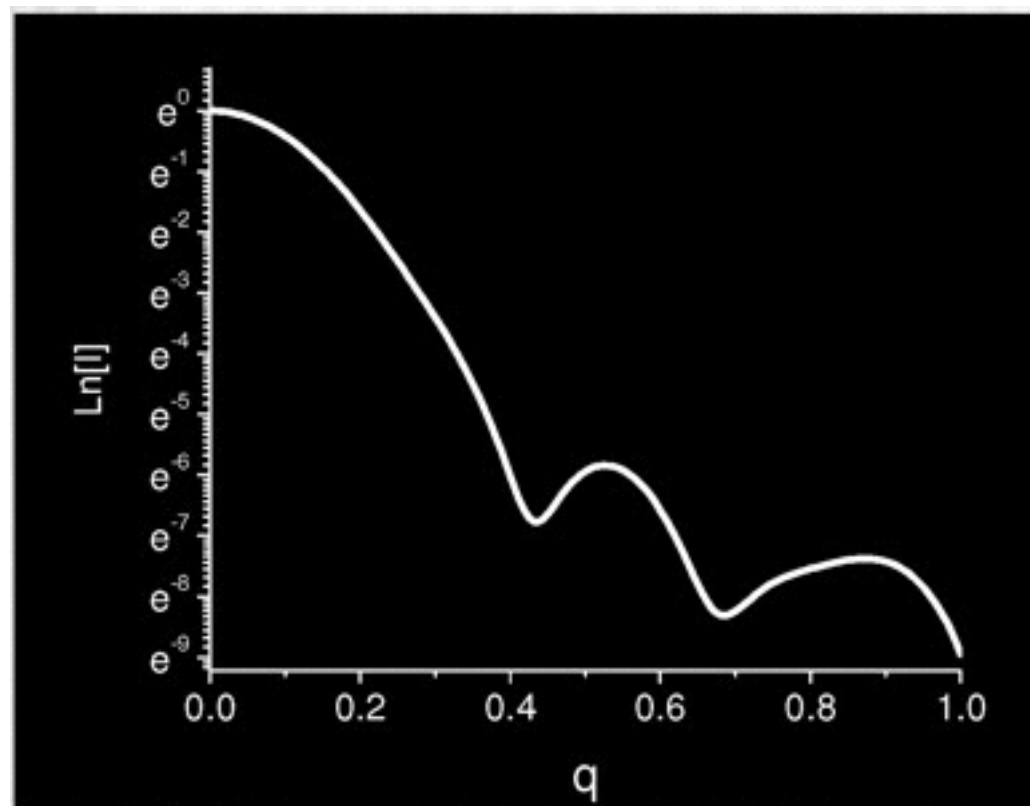


$$I(q) = 4\pi \int_0^\infty p(r) \frac{\sin(qr)}{qr} dr$$

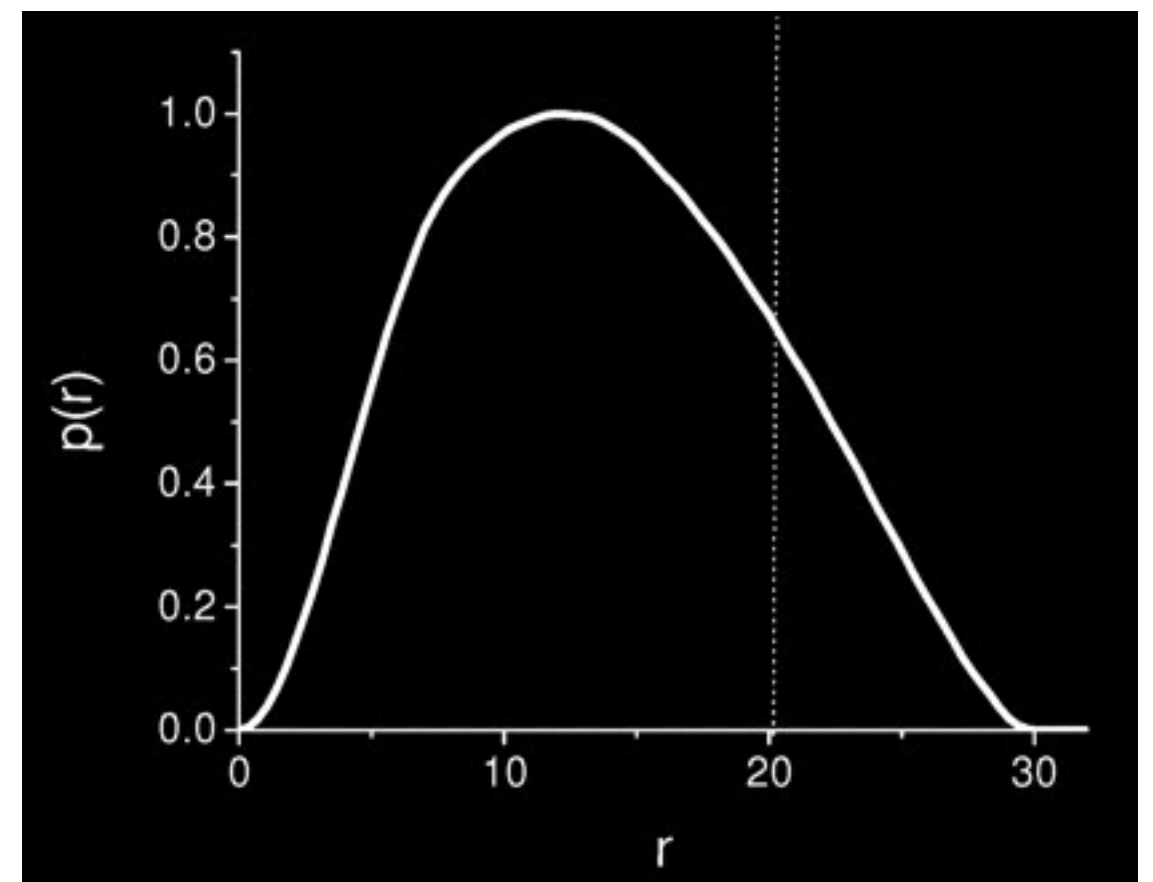
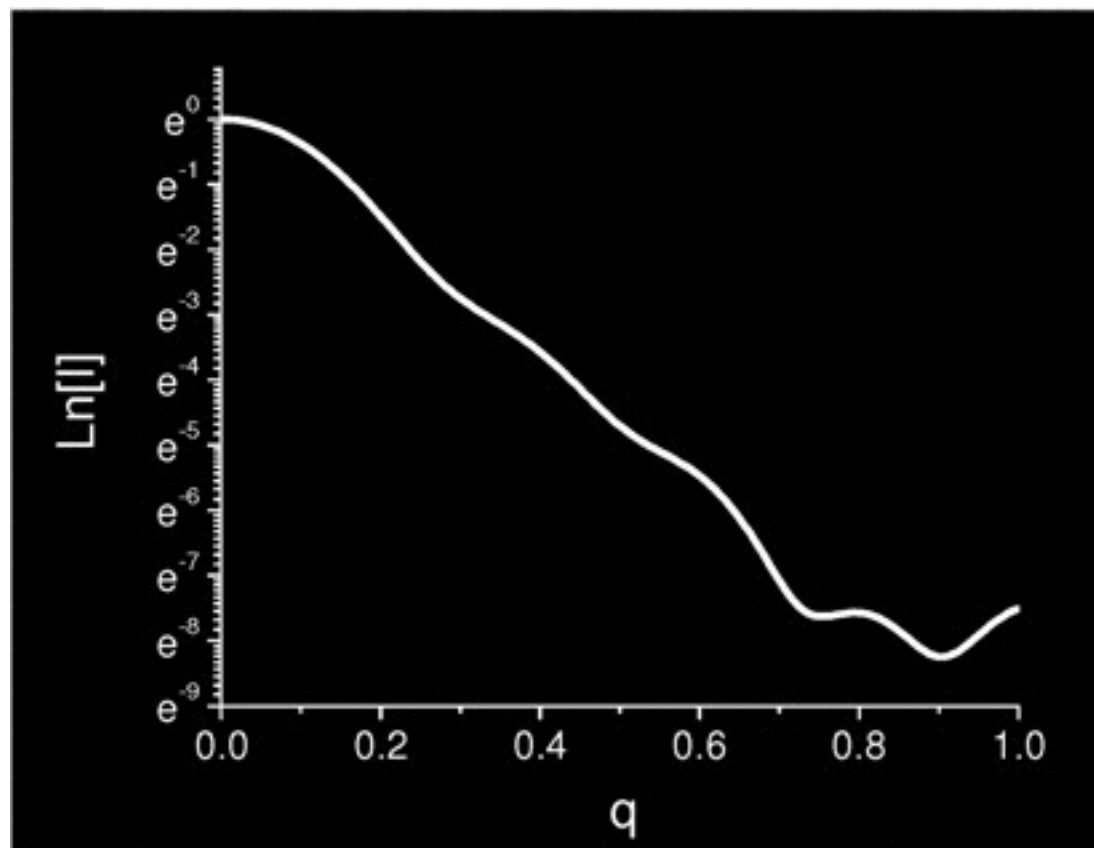
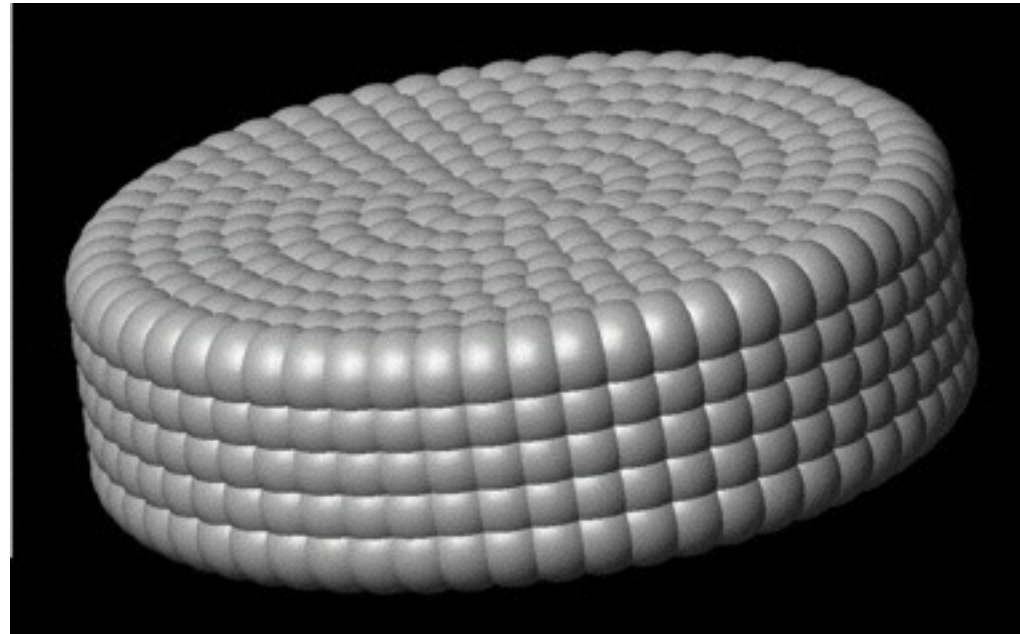
$I(q)$ & $P(r)$ of Spherical particle



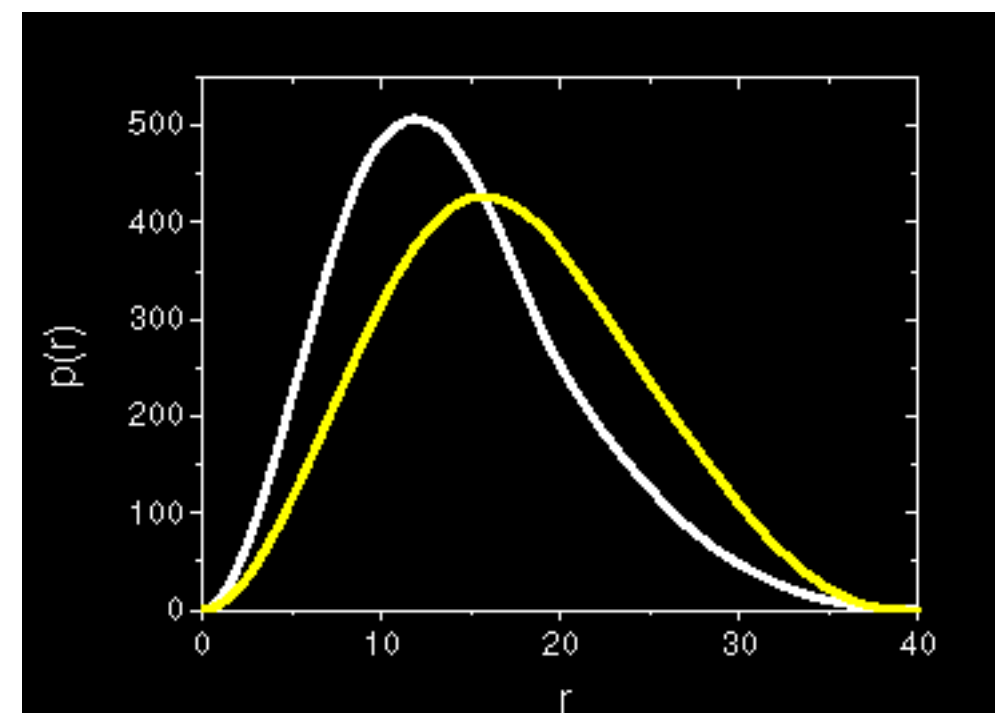
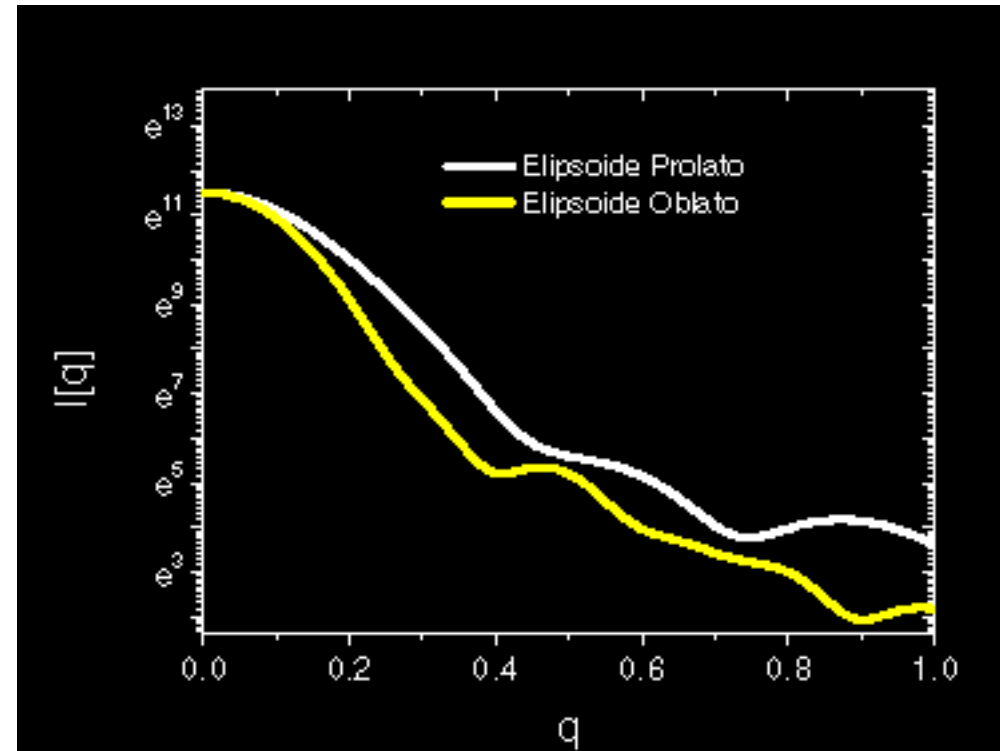
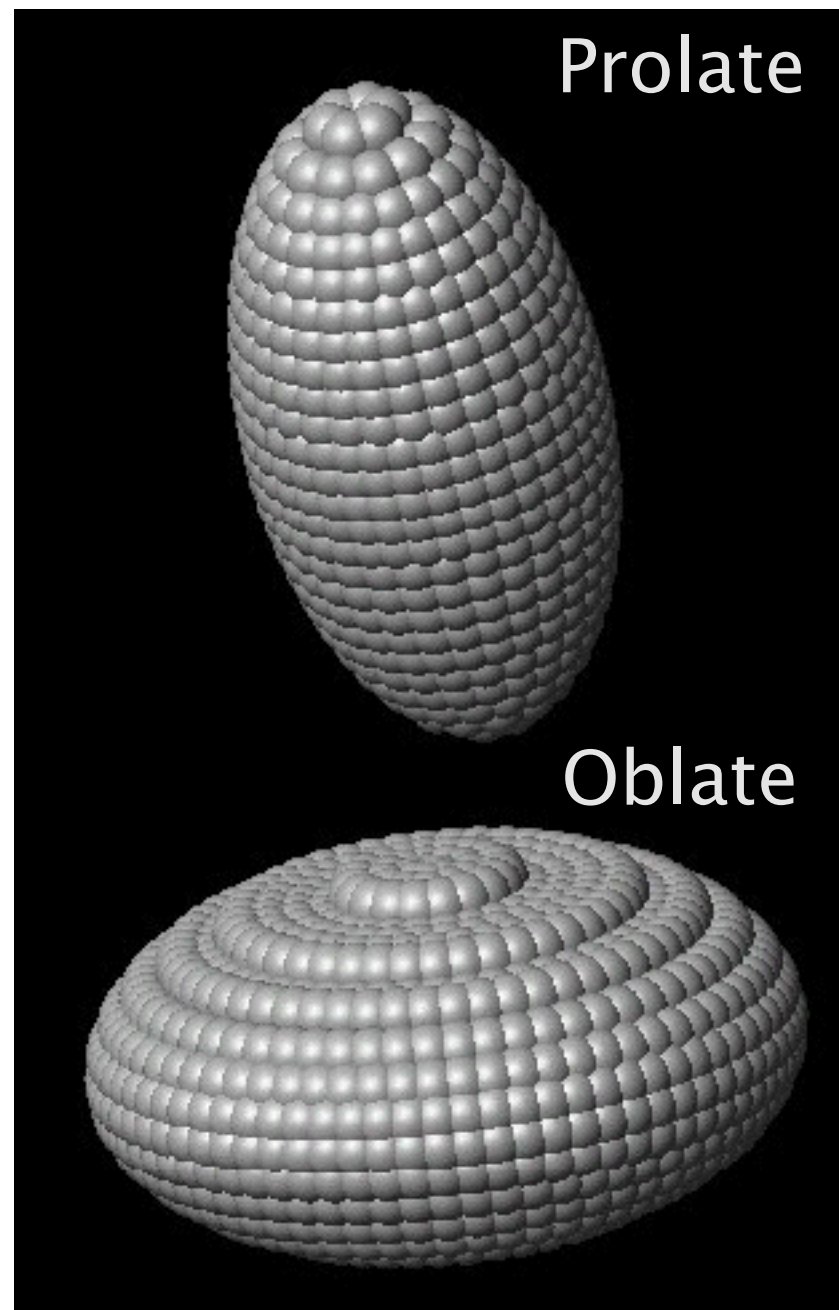
$I(q)$ & $P(r)$ of Cylindrical particle



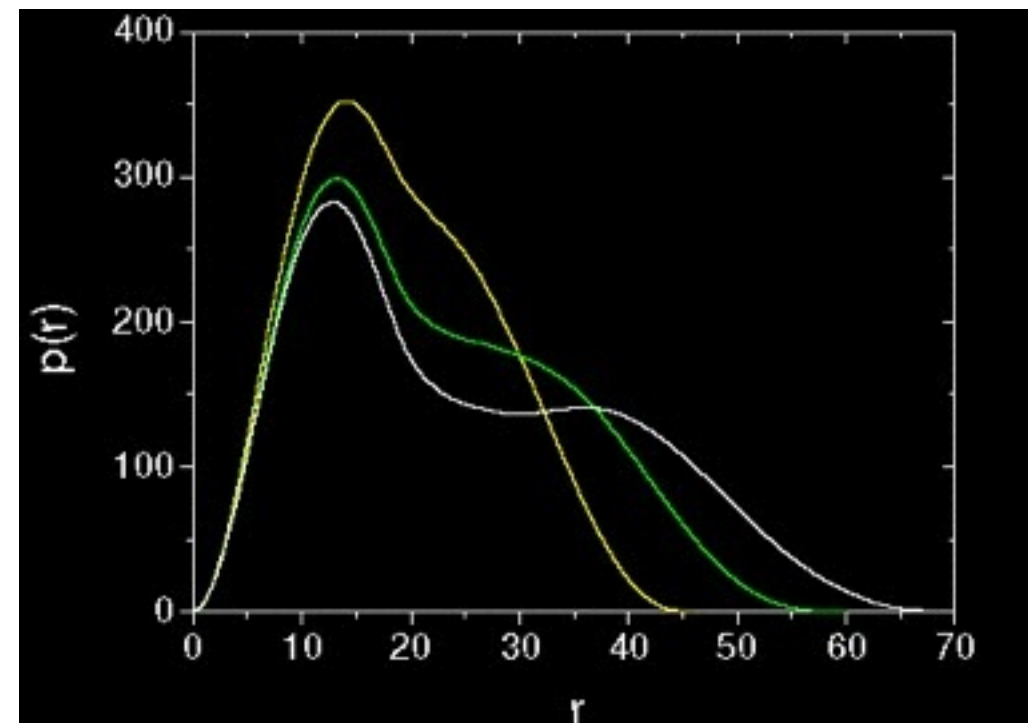
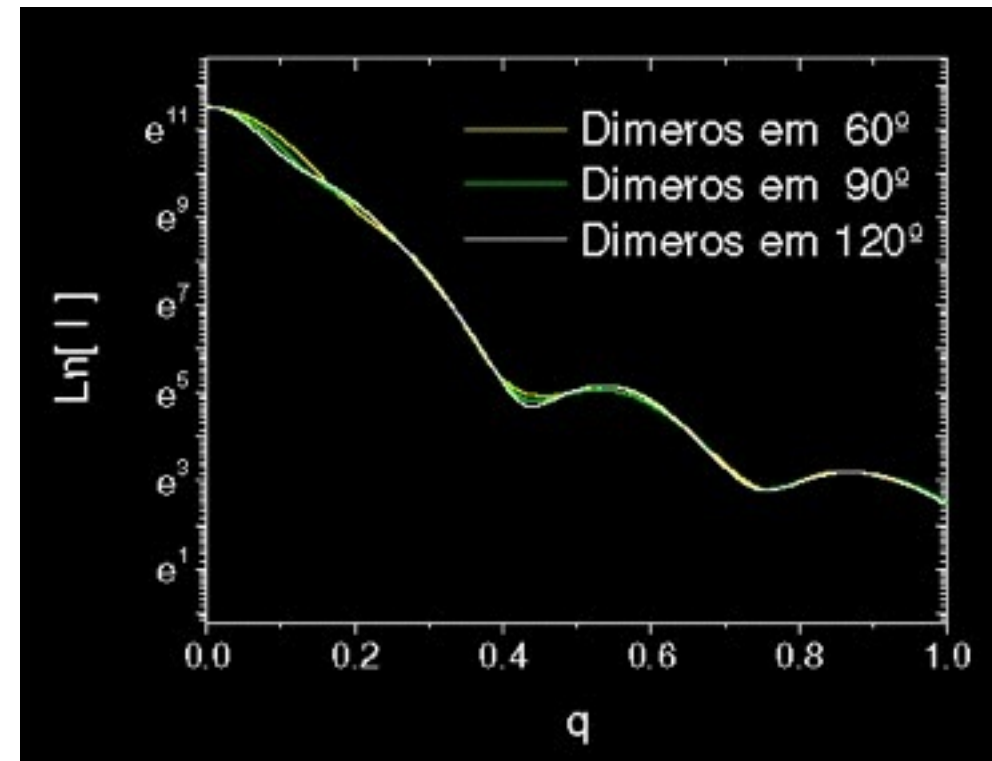
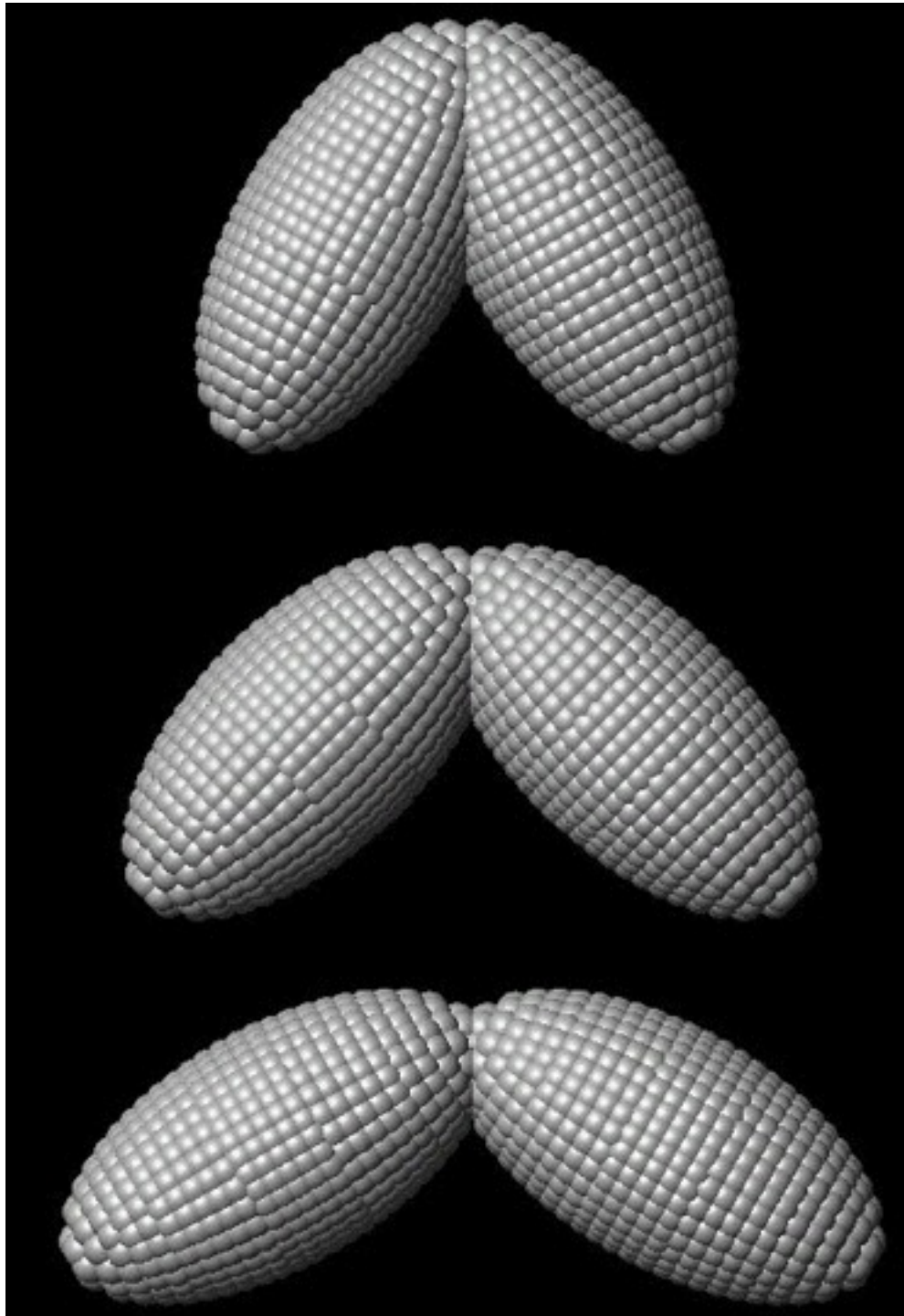
$I(q)$ & $P(r)$ of Flat particle



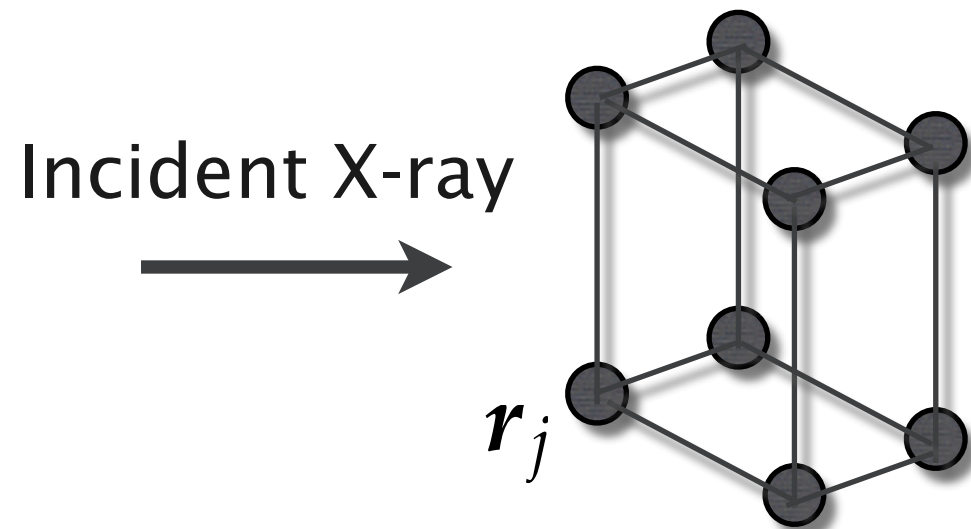
$I(q)$ & $P(r)$ of Ellipsoids



$I(q)$ & $P(r)$ 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:

$$I(\mathbf{q}) \sim \left| \underline{G(\mathbf{q})} \right|^2 \left| F(\mathbf{q}) \right|^2$$

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

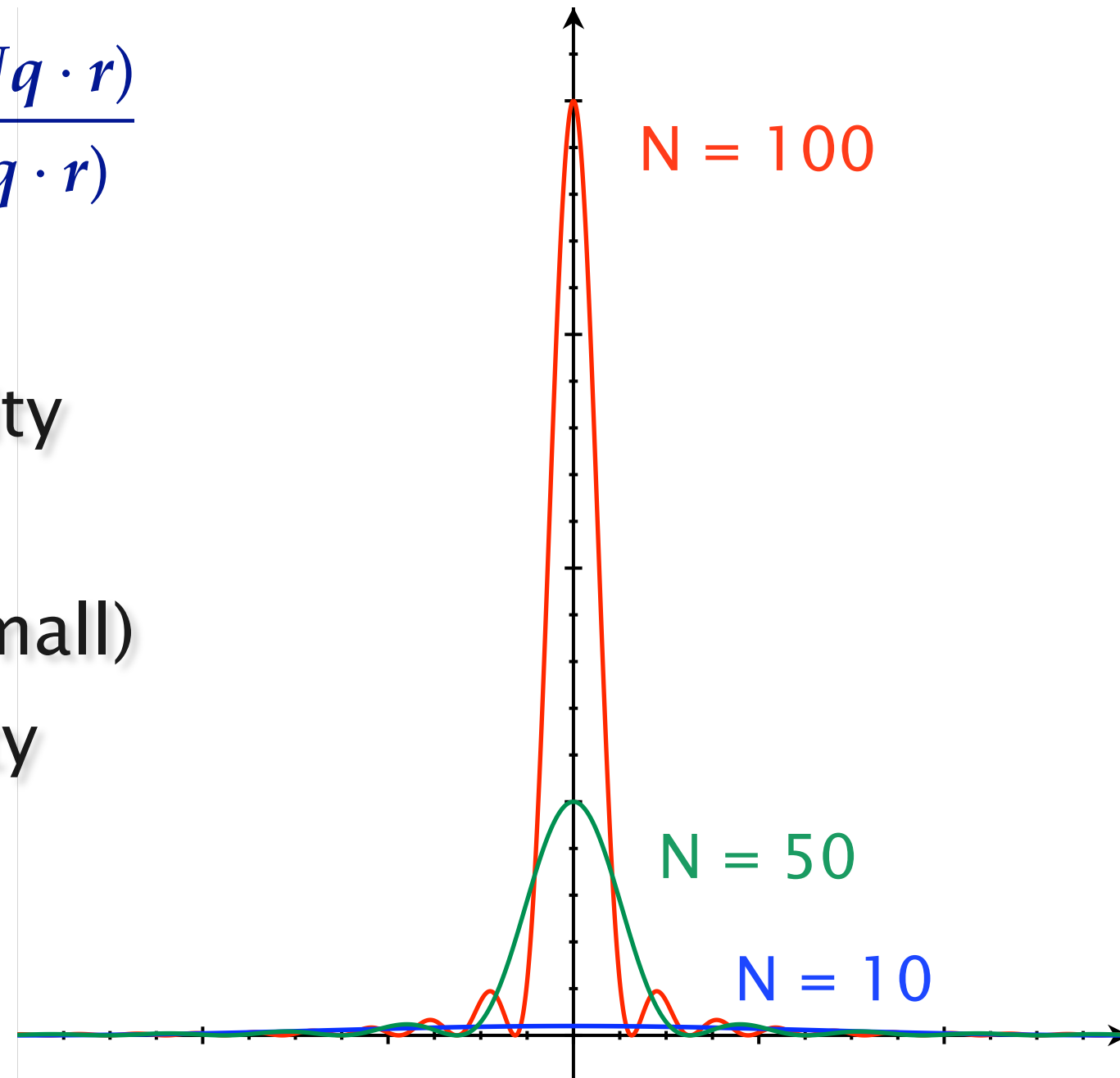
- Maximum $\sim N$
- FWHM $\sim 2\pi/N$
 - FWHM \rightarrow Size of crystal

Laue Function

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

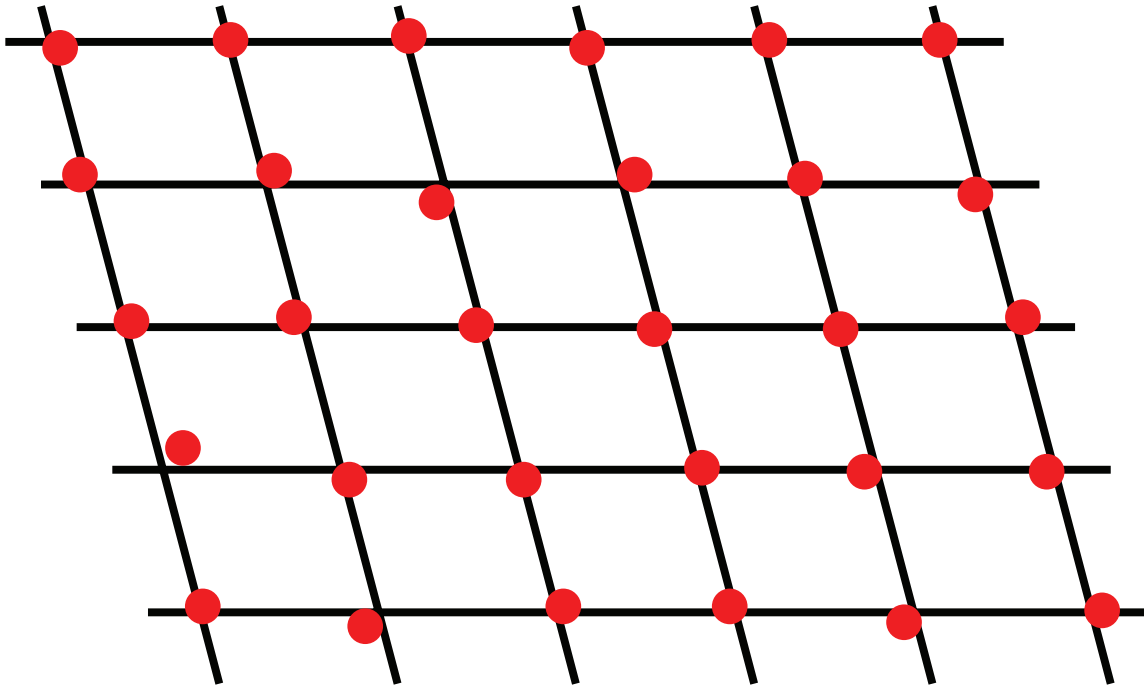
- Large crystal
 - High diffraction intensity
 - Narrow FWHM
- Soft matter (crystal size: small)
 - Low diffraction intensity
 - Wide FWHM

→ low S/N

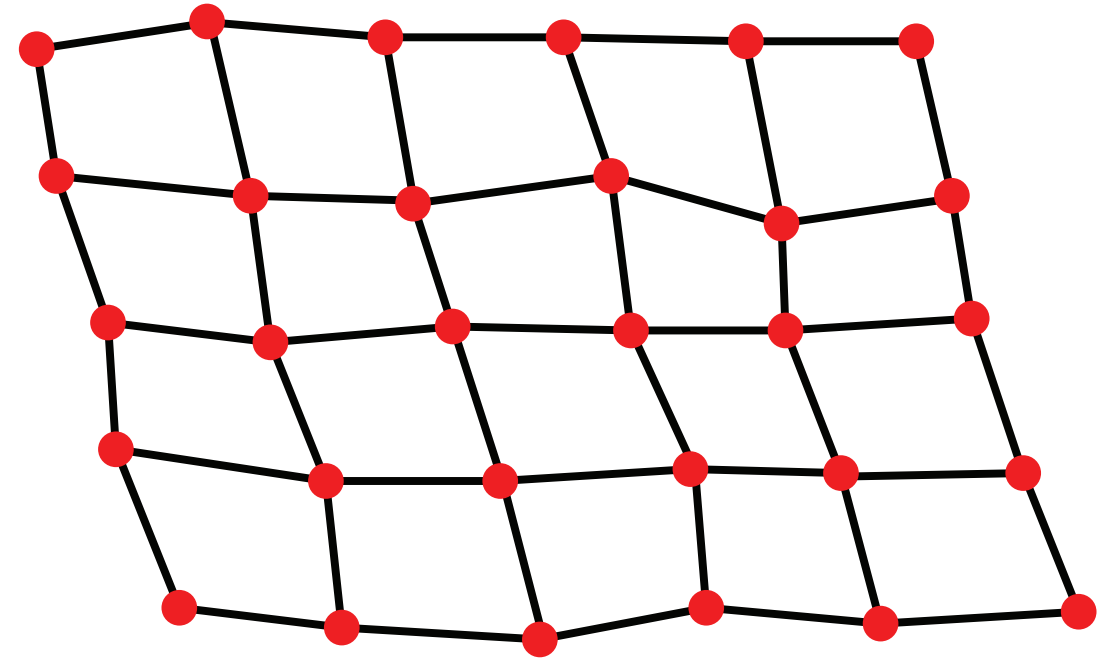


Crystal size --> Intensity & FWHM of diffraction

Imperfection of crystal (2D)



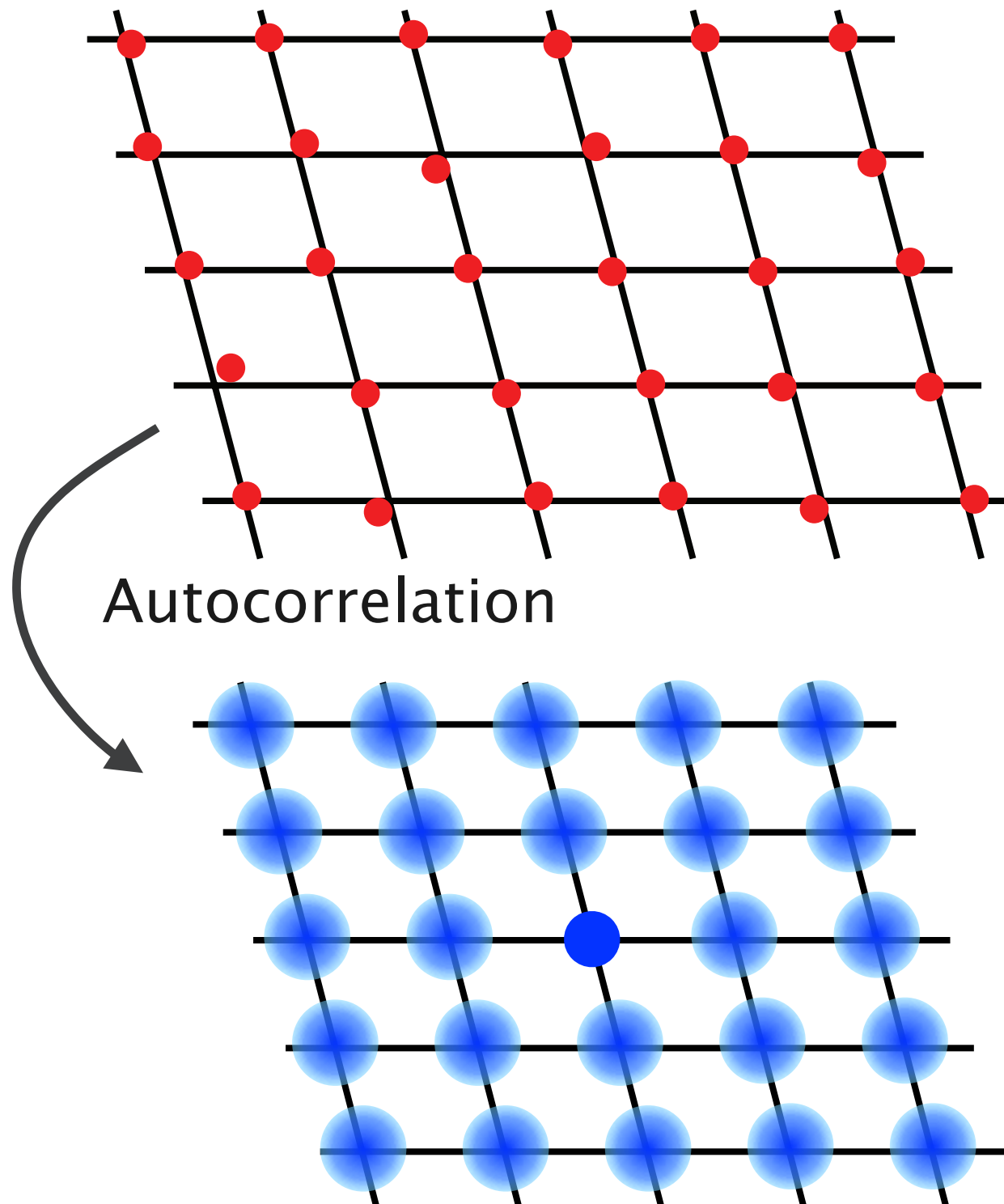
Imperfection of 1st kind
Thermal fluctuation etc.



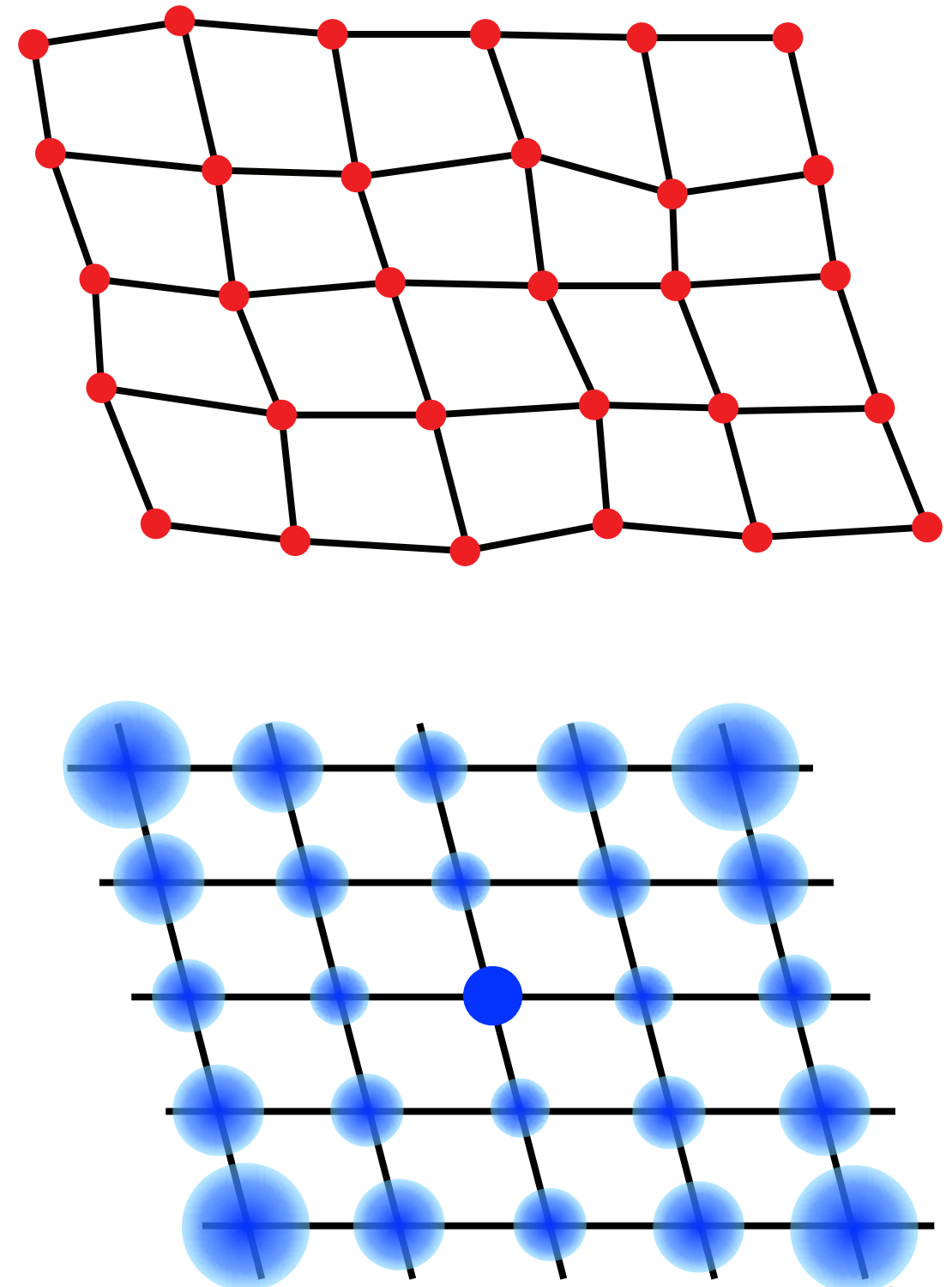
Imperfection of 2nd kind
in the case of soft matter

Imperfection of crystal

Imperfection of 1st kind




Imperfection of 2nd kind



Imperfection of lattice (1D)

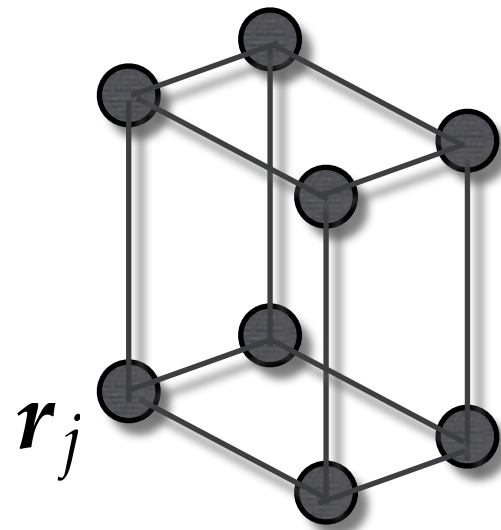
Perfect lattice 

Imperfection of 1st kind 

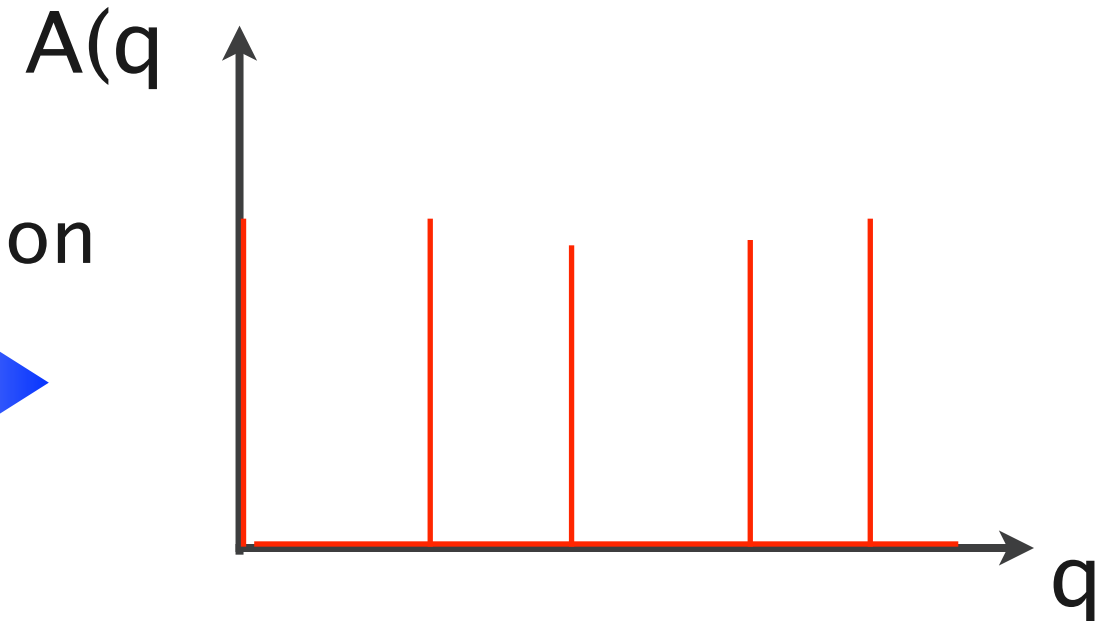
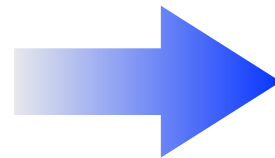
Imperfection of 2nd kind 

☛ Effect of imperfections on diffraction ?

Diffraction from lattice-structure



Diffraction



$$\rho(\mathbf{r}) = \rho_u(\mathbf{r}) * \underline{z(\mathbf{r})}$$

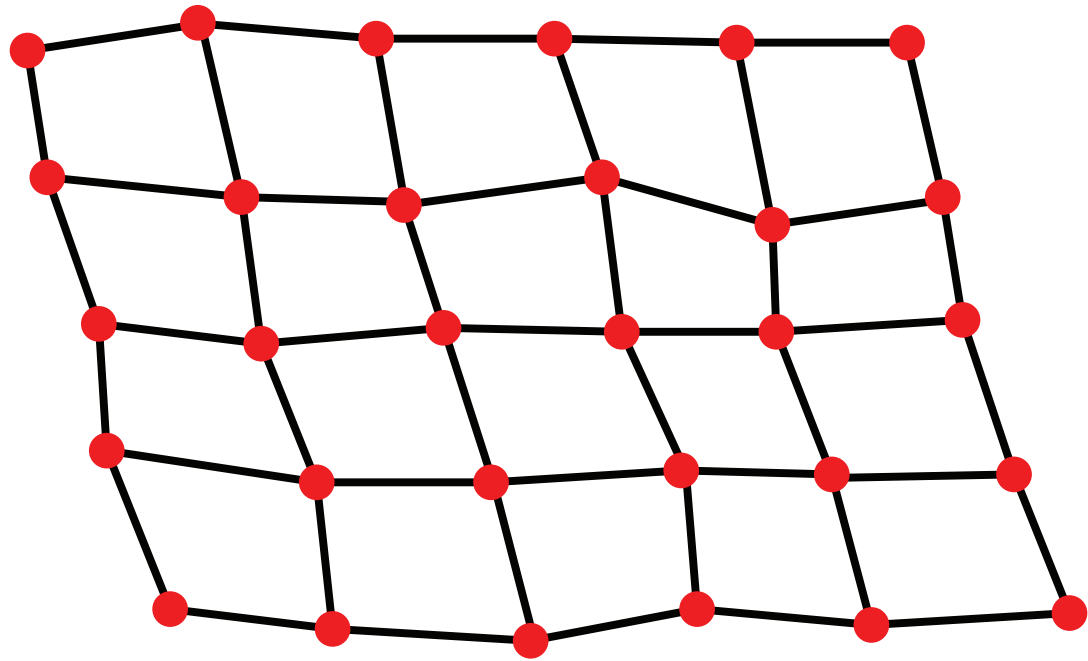
convolution

$$A(\mathbf{q}) = F(\mathbf{q}) \cdot \underline{Z(\mathbf{q})}$$

Form of lattice

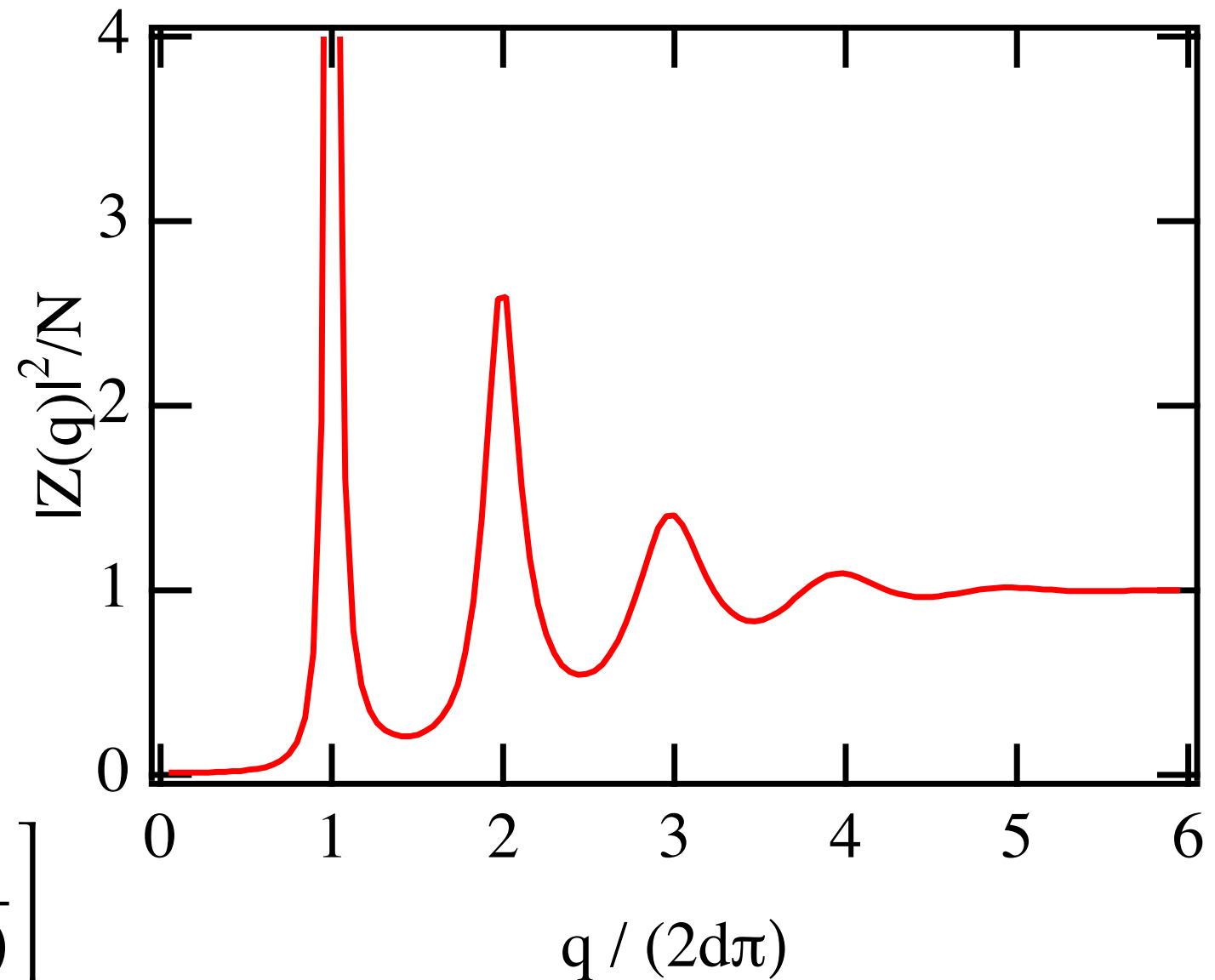
$z(\mathbf{r})$ with imperfection \rightarrow calculate $Z(\mathbf{q})$

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

Increase of FWHM

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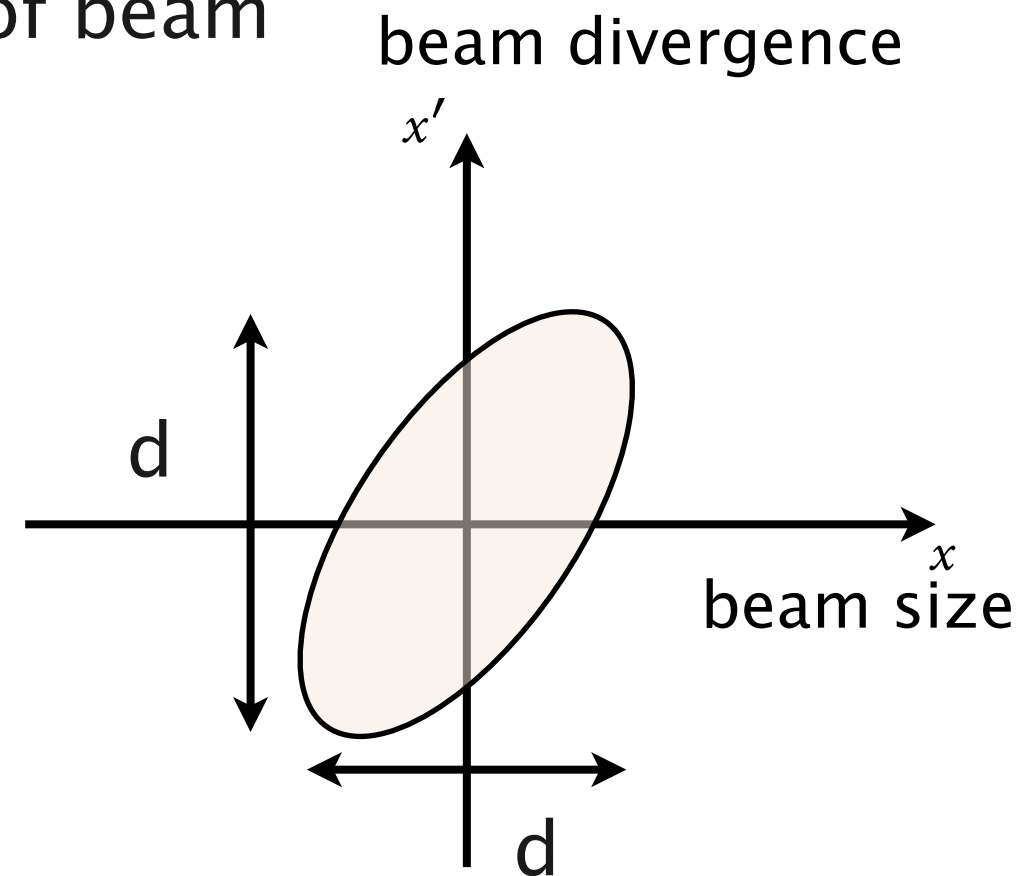
- ❧ Introduction
 - ❧ What's SAXS ?
 - ❧ History
- ❧ Theory
 - ❧ Basic of X-ray scattering
 - ❧ Structural Information obtained by SAXS
- ❧ Experimental Methods
 - ❧ X-ray Optics
 - ❧ X-ray Detectors
- ❧ Advanced SAXS
 - ❧ Microbeam, GI-SAXS, USAXS, XPCS etc...

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)]

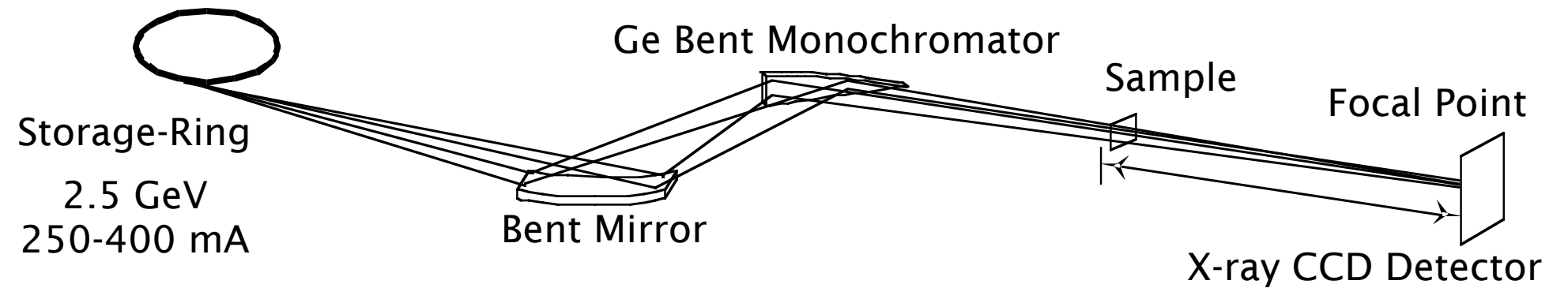


SAXS needs to use a low divergence and small beam

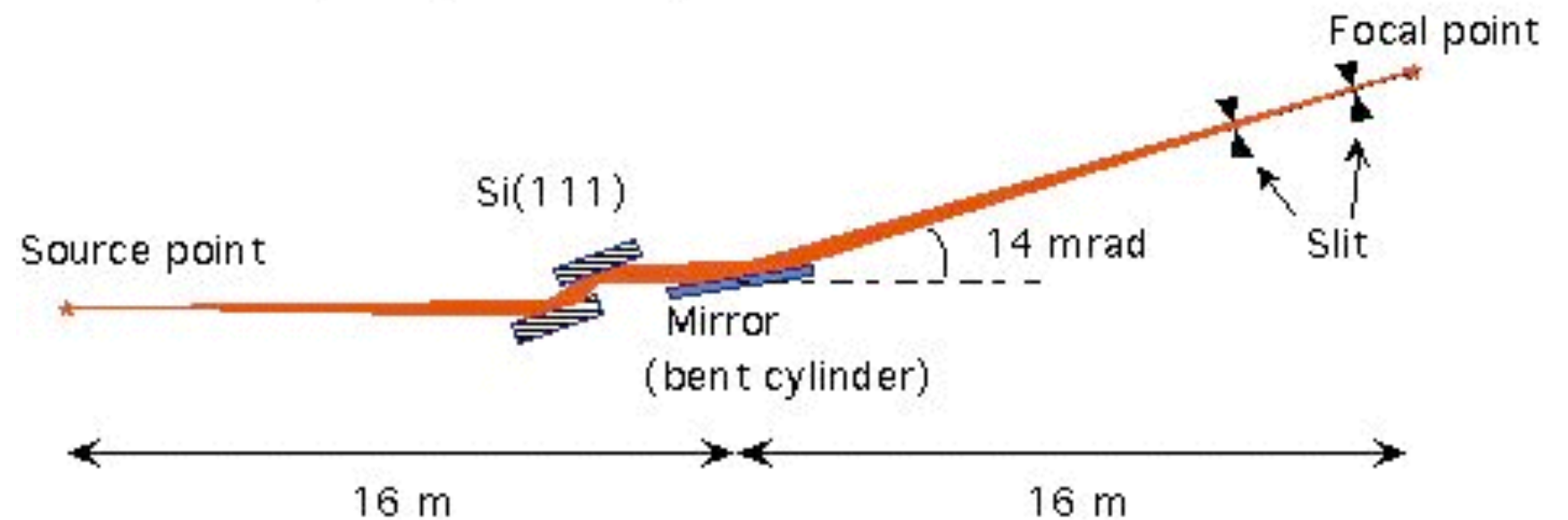
→ High brilliance beam is required !

X-ray Optics for SAXS

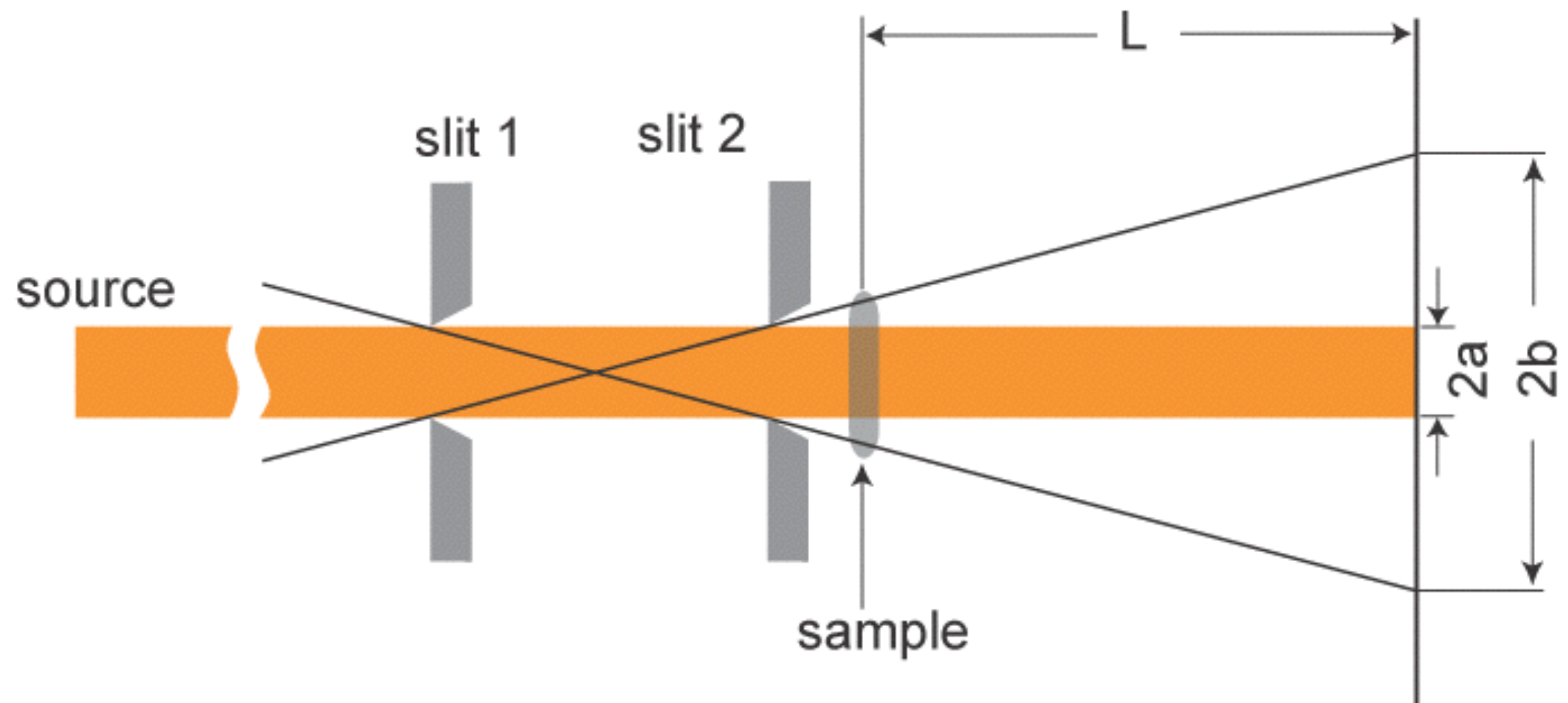
PF BL-15A



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

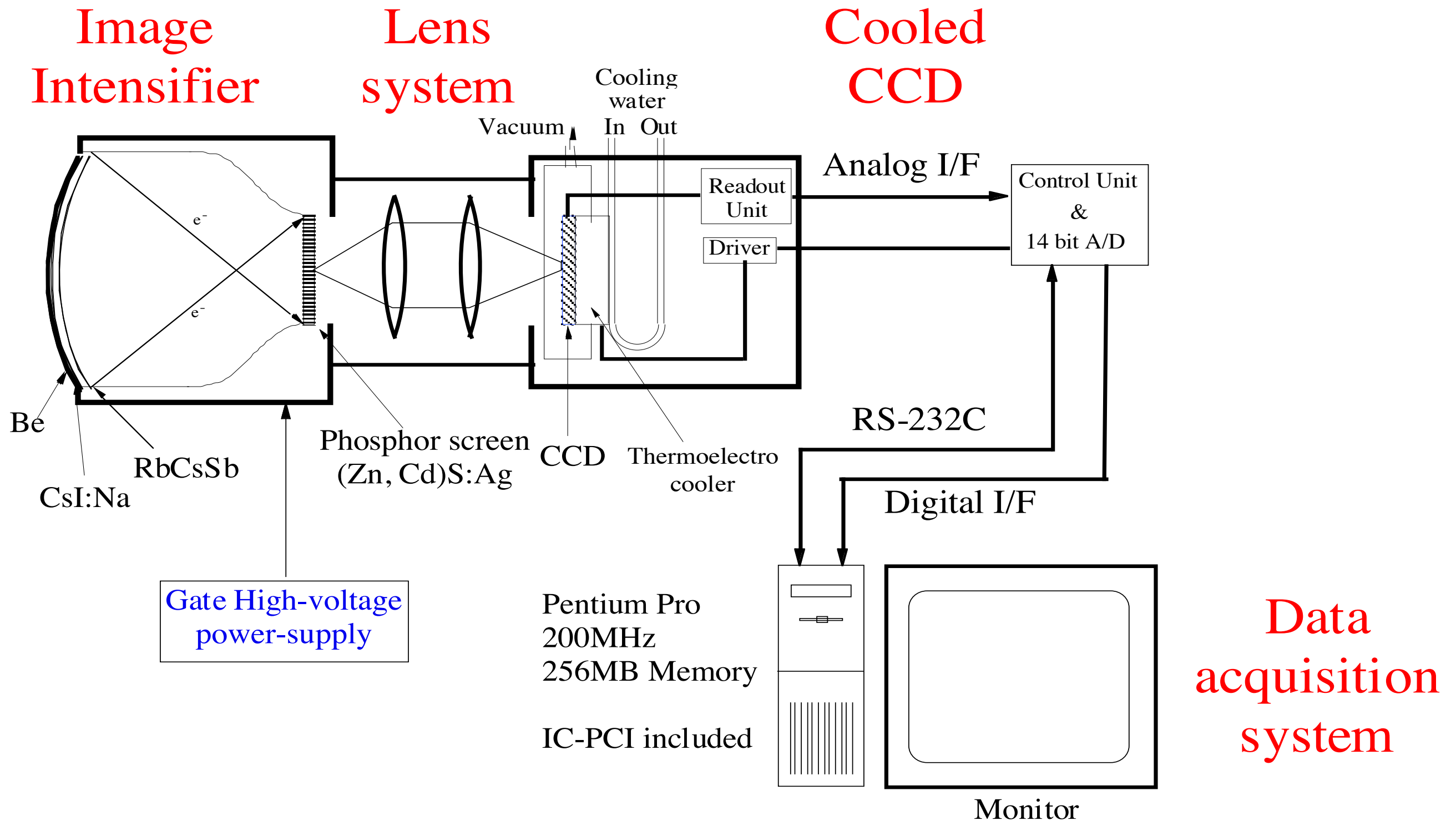


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Advanced SAXS

Microbeam X-ray

- Inhomogeneity of nano-structure
- local time evolution of structure

Time-resolved

- time evolution of structure

GI-SAXS

- surface, interface, thin films

SAXS

XPCS

- structural fluctuation
- dynamics

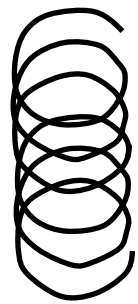
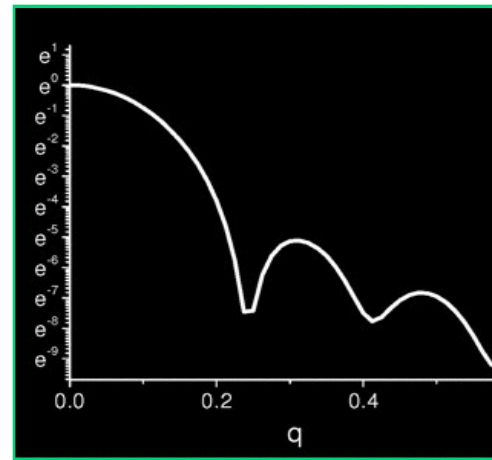
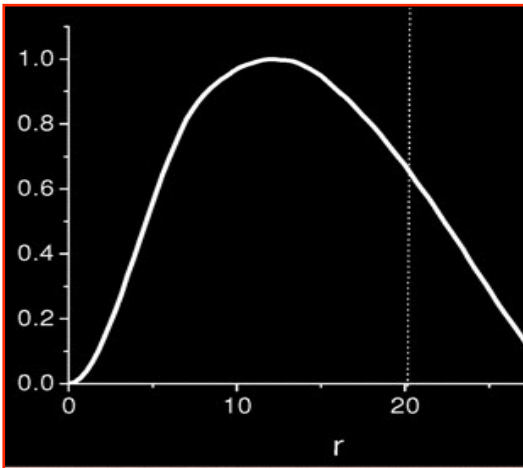
Combined measurement with DSC, viscoelasticity wide-q (USAXS-SAXS-WAXS) 2D measurement

- hierarchical structure

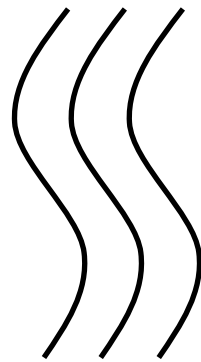
- anisotropic structure

Application of paracrystal theory

Collab. with Kao Ltd.



African



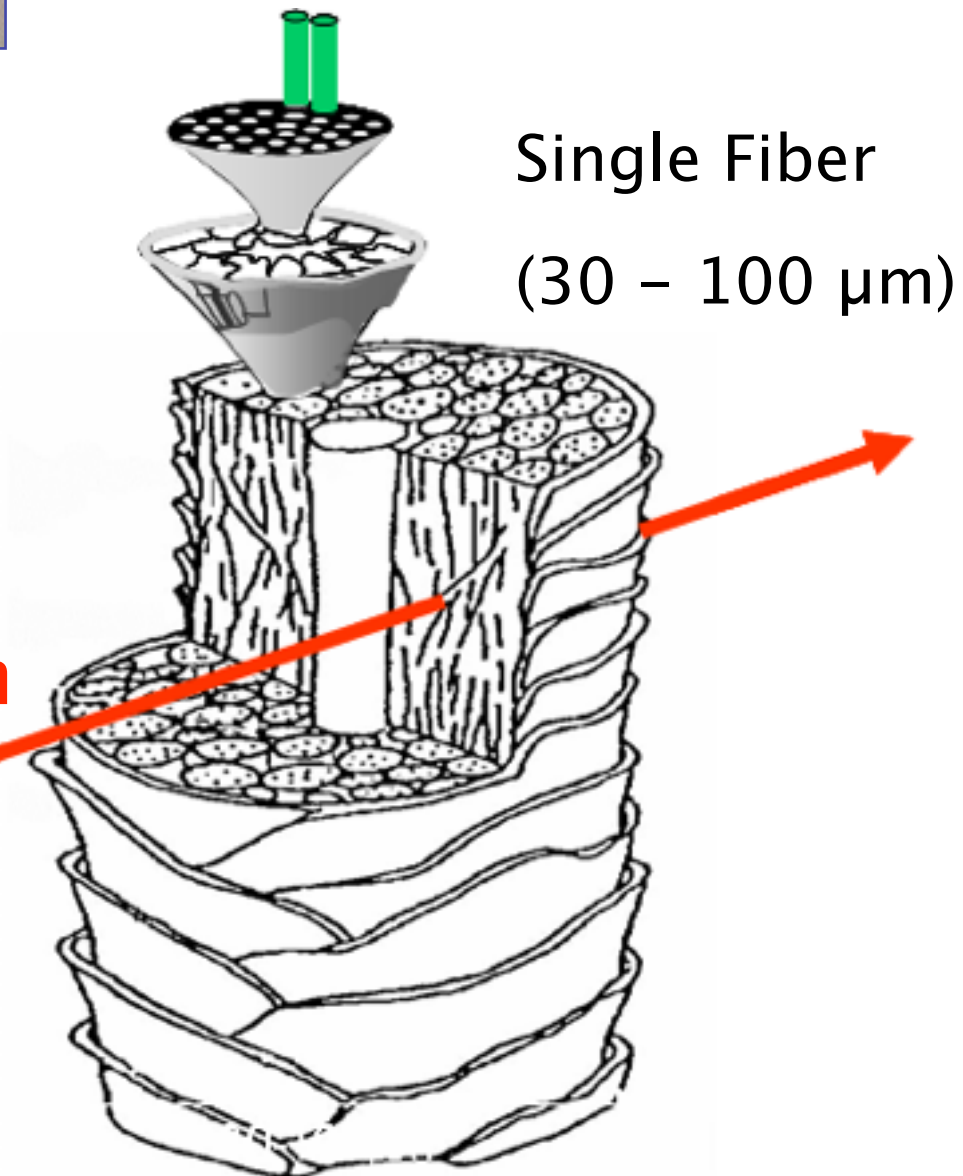
Caucasian



Asian

Relationship between macroscopic form and nano structure?

X-ray Microbeam
(5 μm x 5 μm)

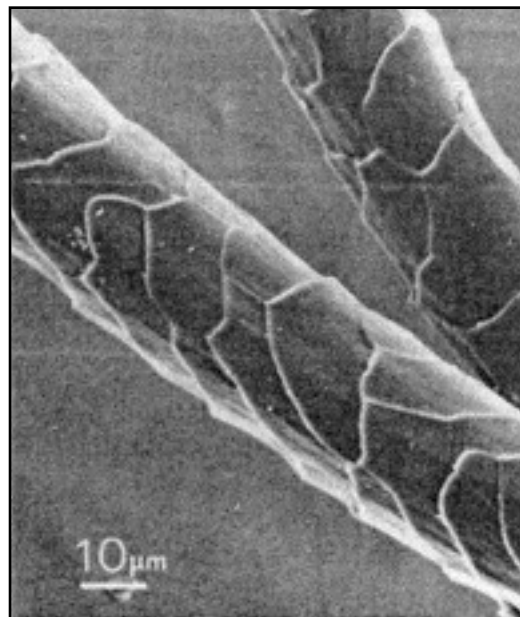


Local observation with an X-ray microbeam

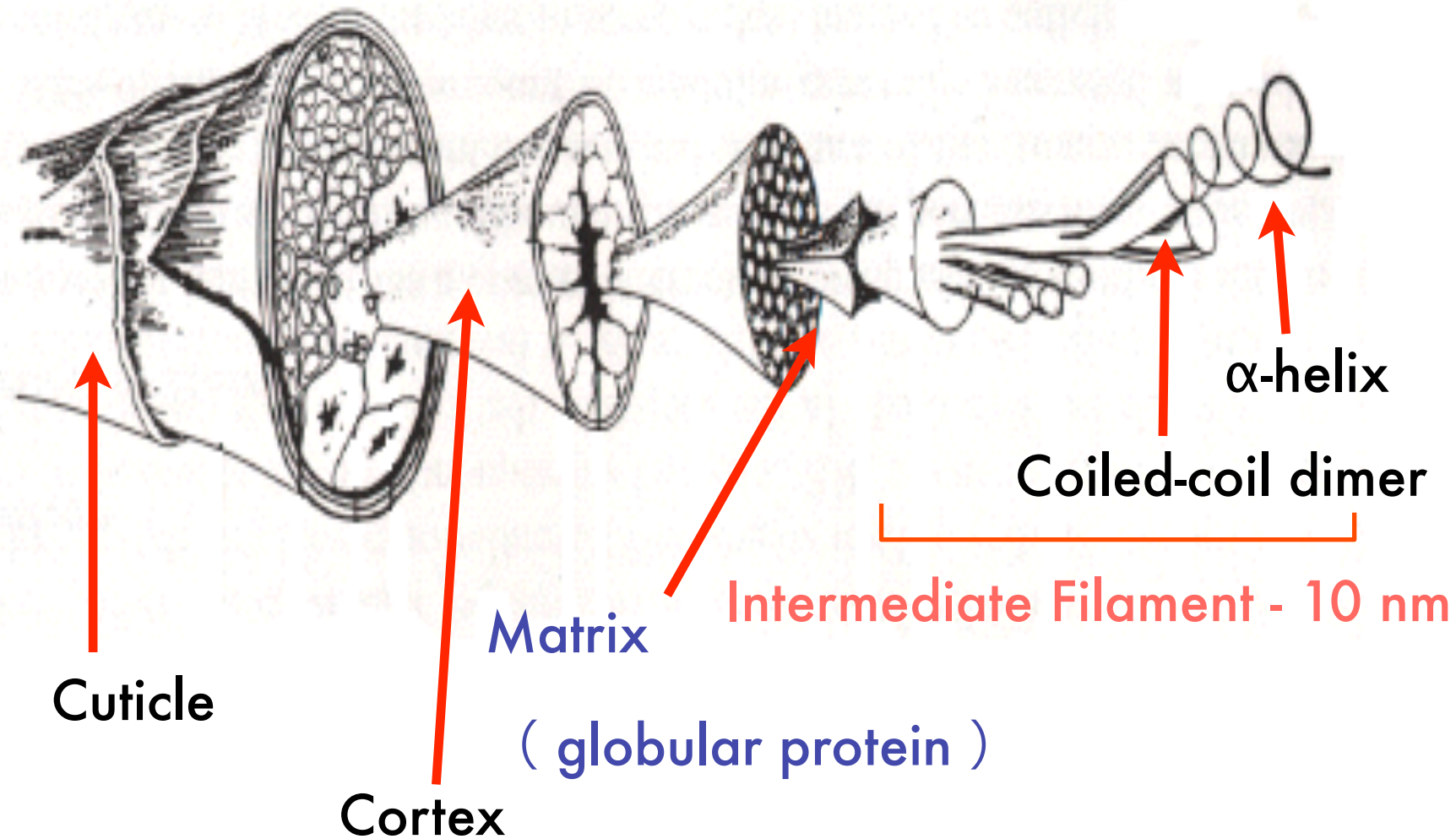
Internal structure of wool



SEM 像



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

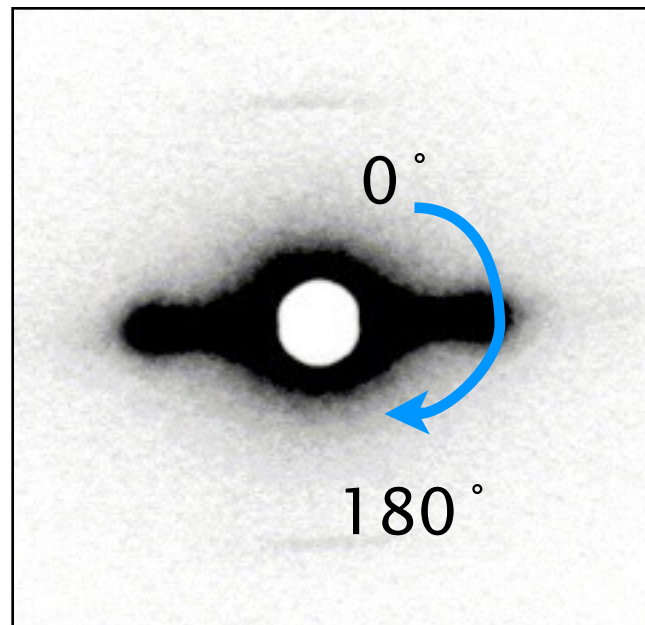
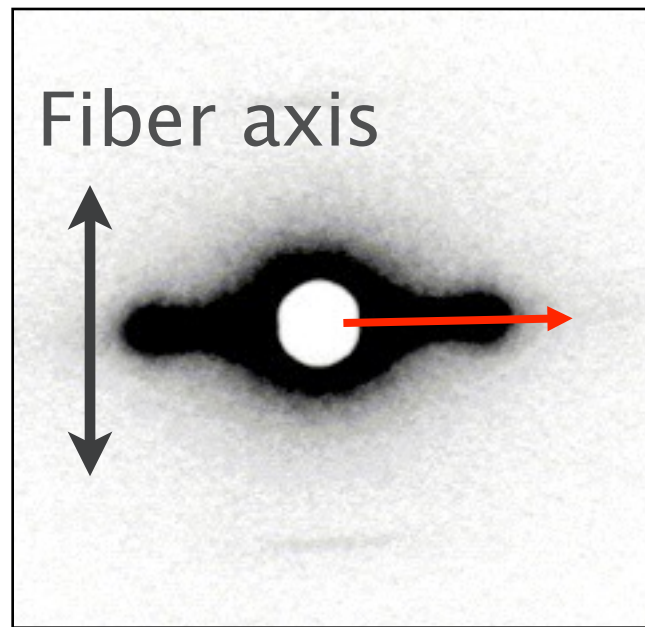


R. D. B. Fraser et al., Proc. Int. Wool Text. Res. Conf., Tokyo, II, 37, (1985) partially changed.

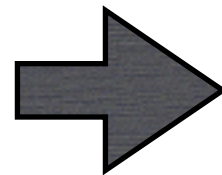
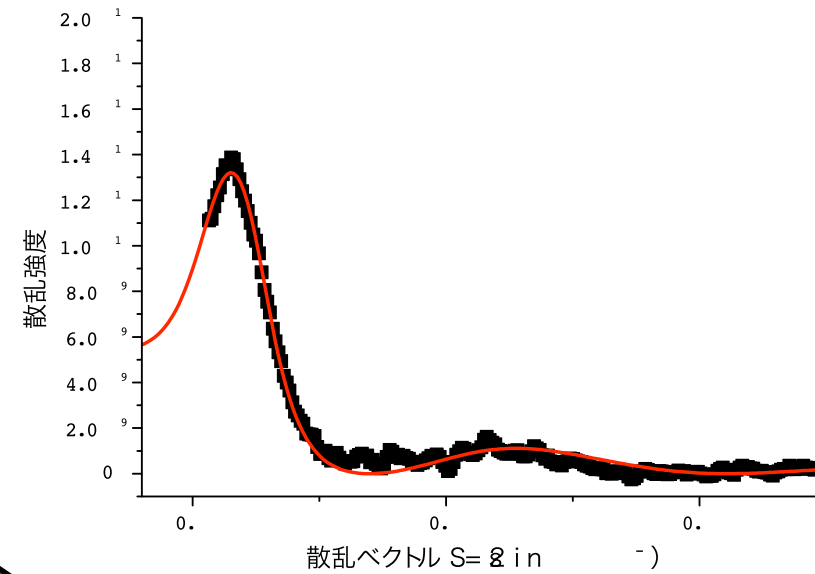
Relationship between IF distribution and hair curliness?

Structure of Intermediate Filament

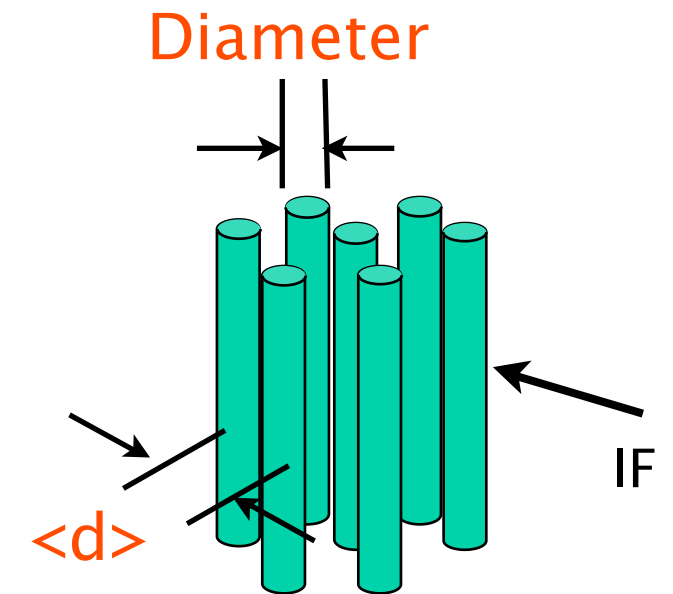
Scattering pattern



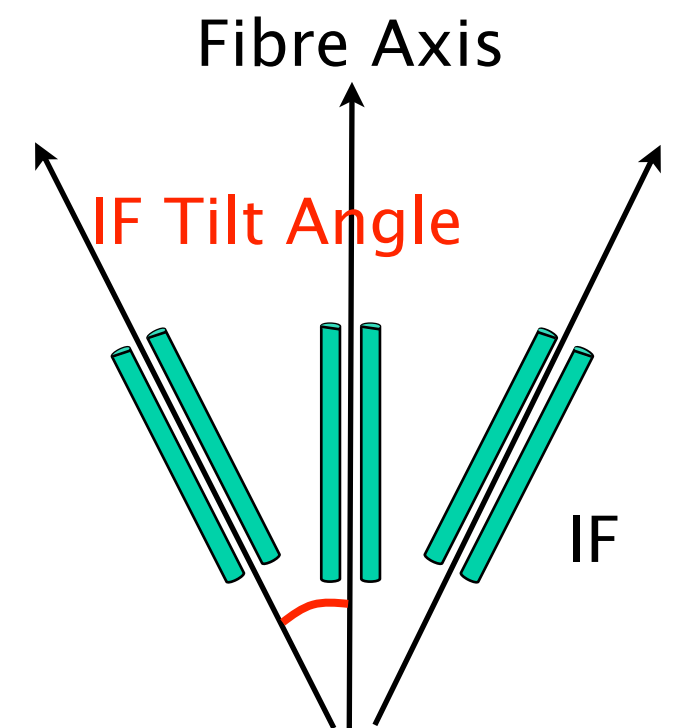
1D intensity profile



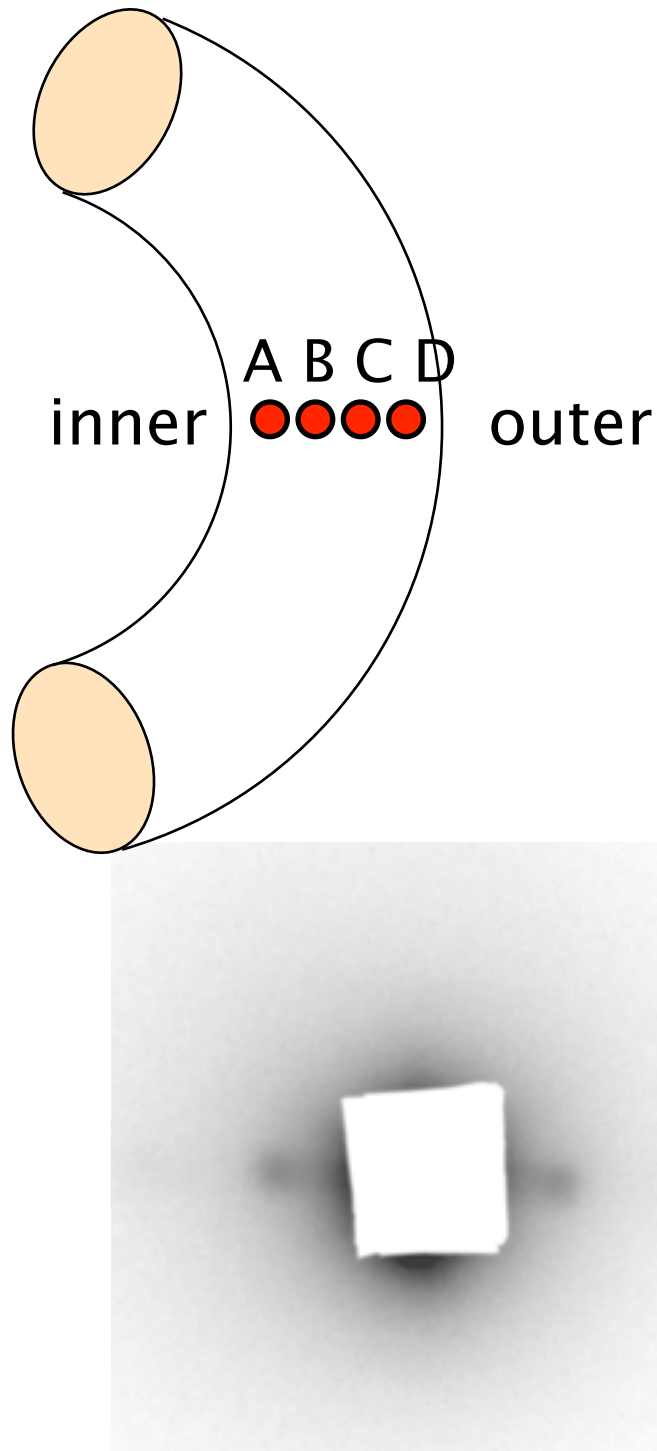
Real space structure



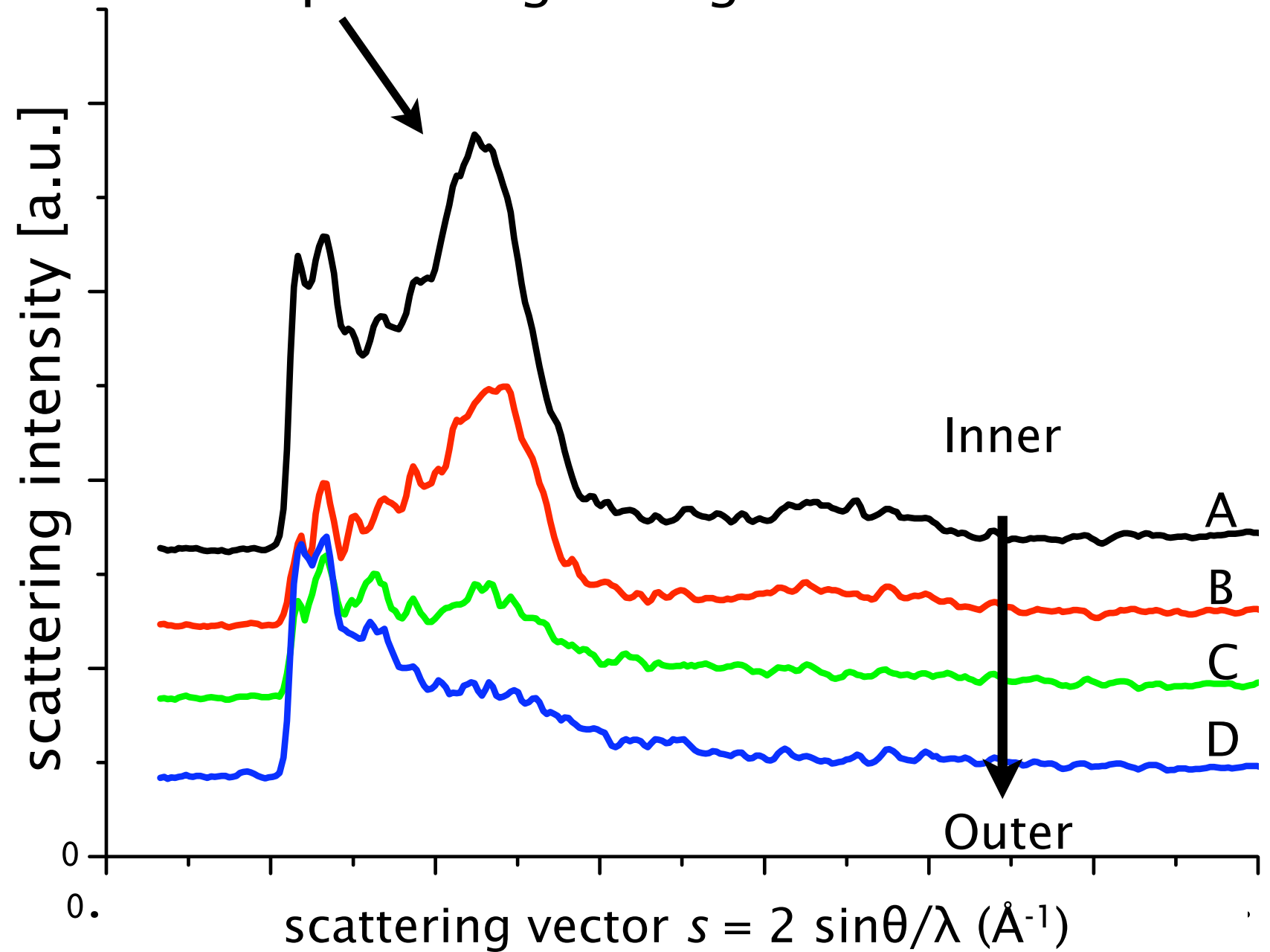
IF-IF Distance



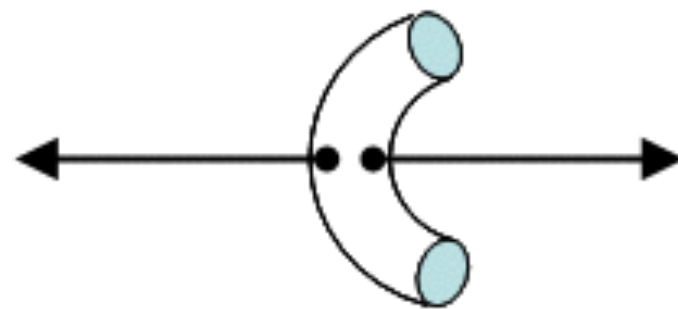
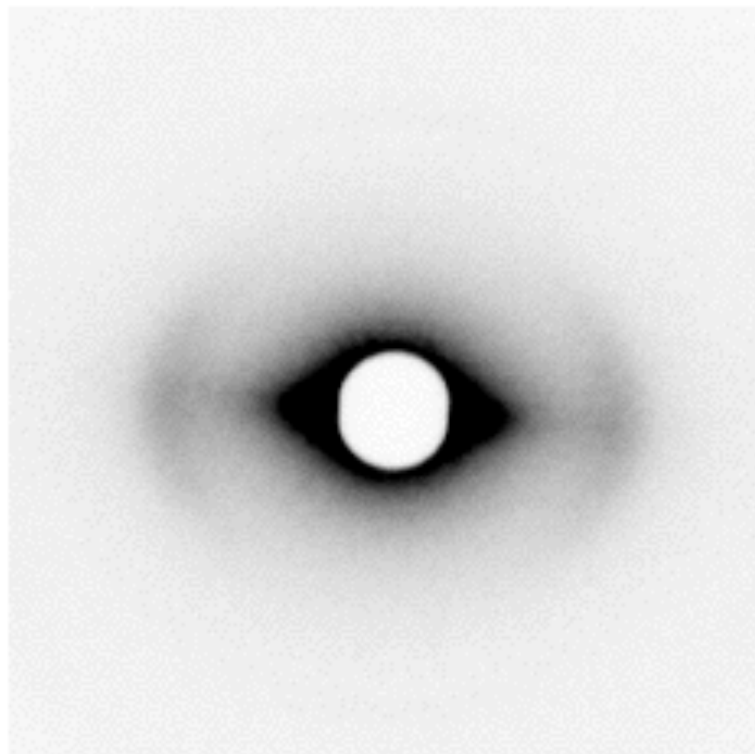
Diffraction intensity profiles



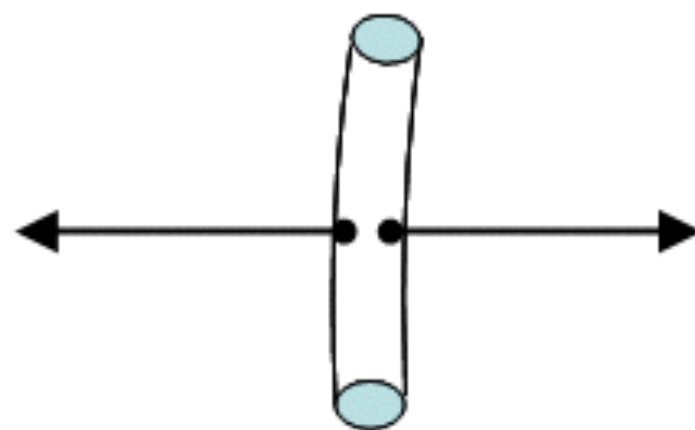
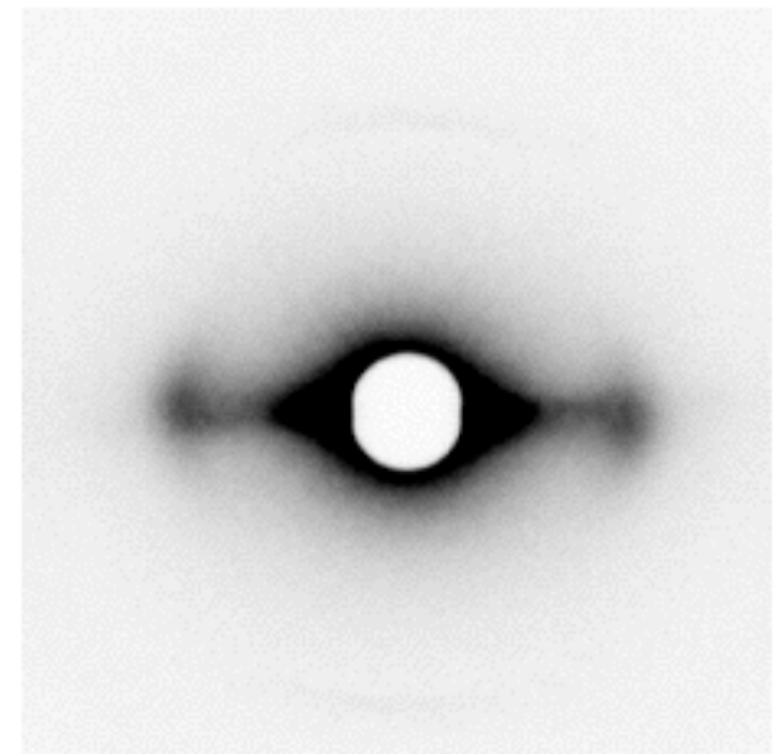
Diffraction peak originating from IF



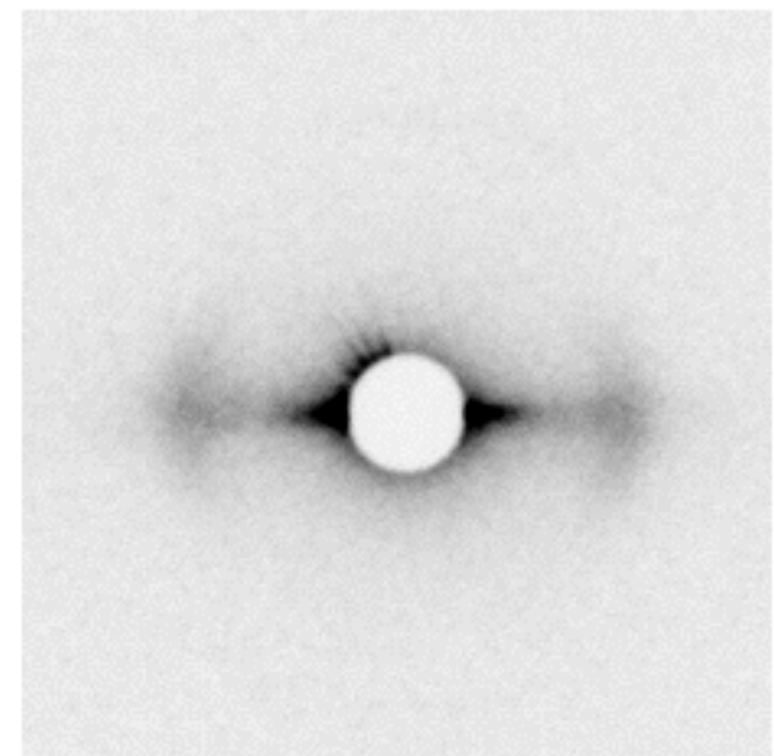
Difference in diffraction intensity
--> Structural difference in cortex.



Curly
(ROC = 1.5cm)

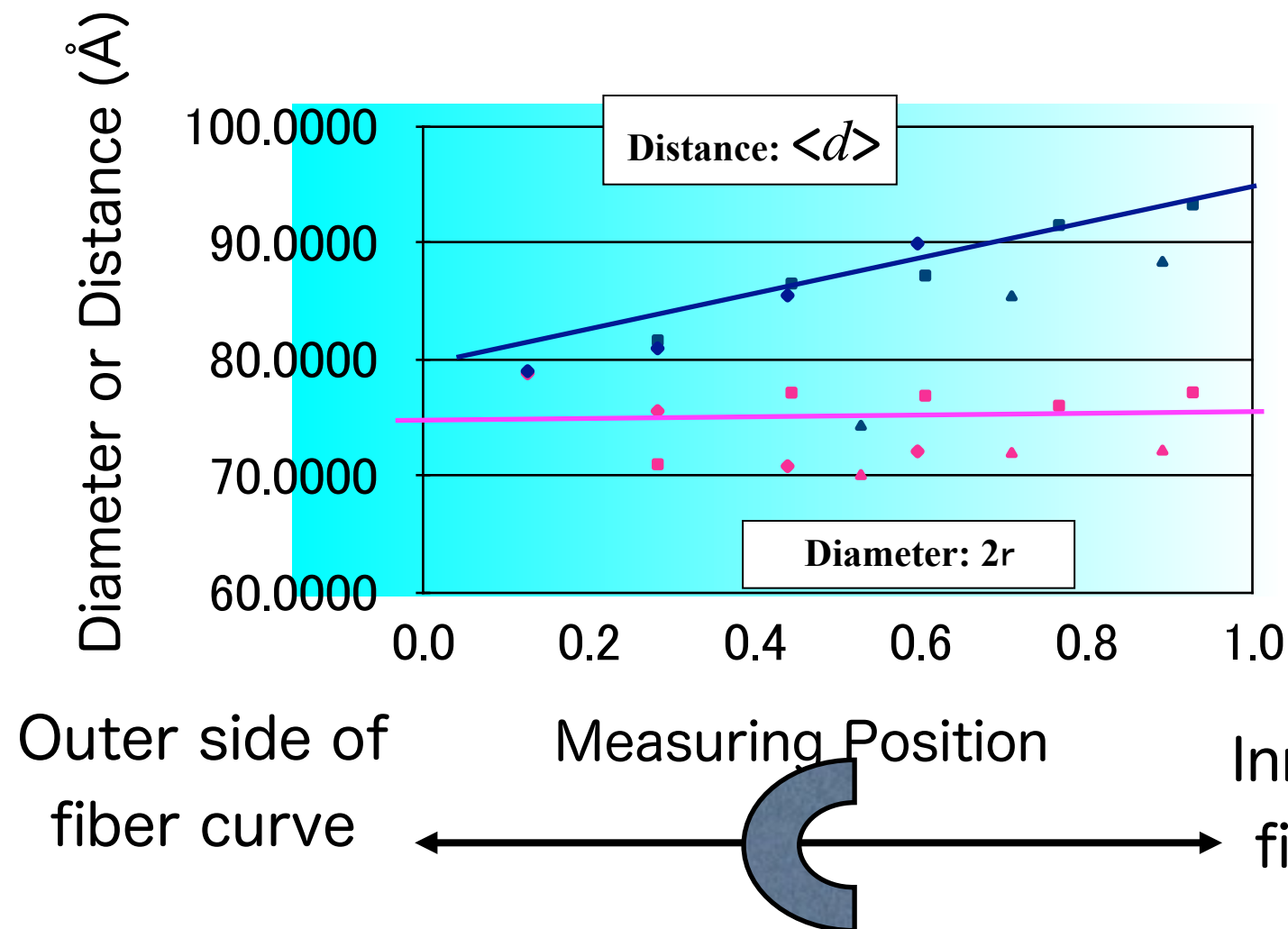


Nearly Straight
(ROC ~ 10cm)

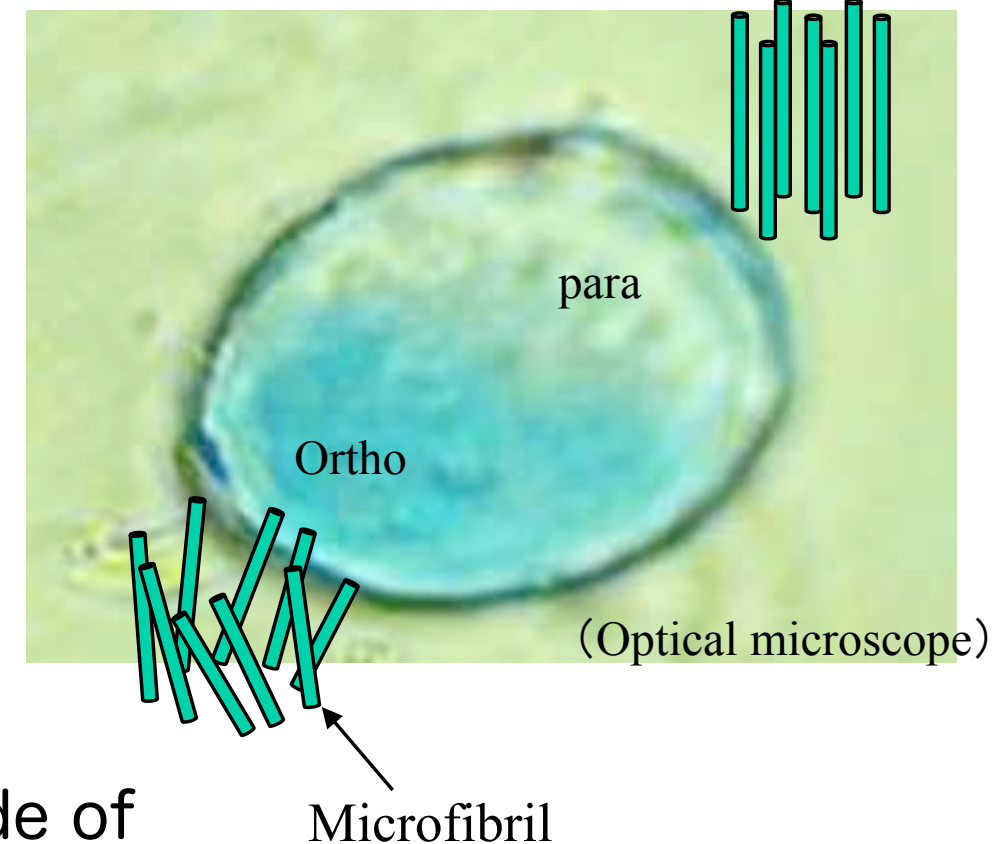


ROC: Radius of Curvature

Distribution of Intermediate Filament (IF)

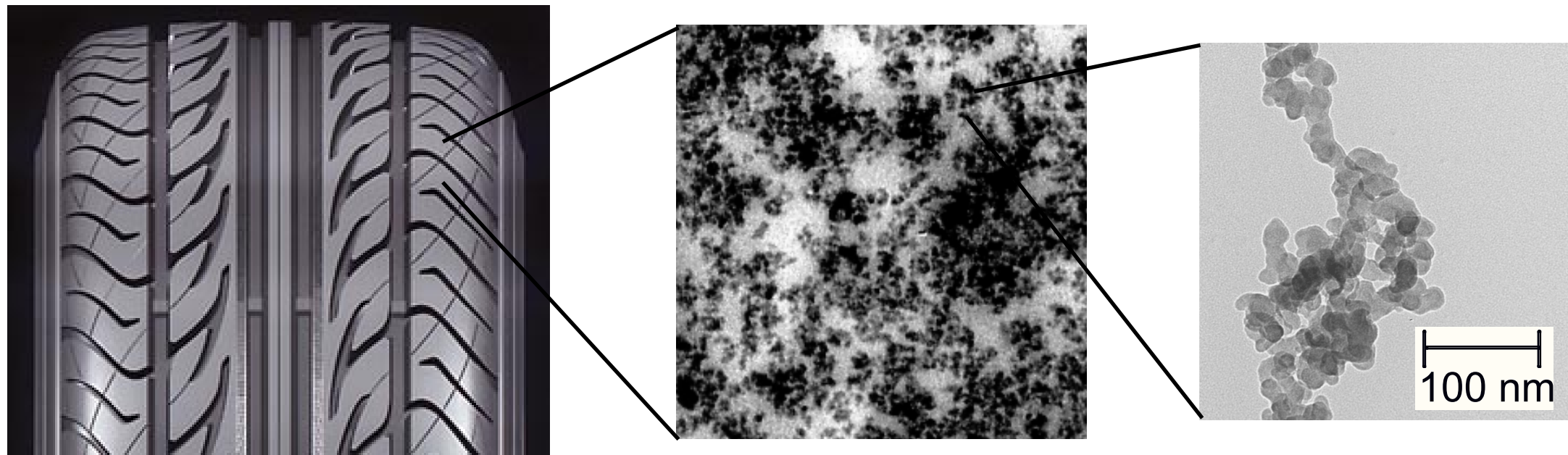


Stained with Methylene blue dye



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

USAXS for nano-composite in rubber

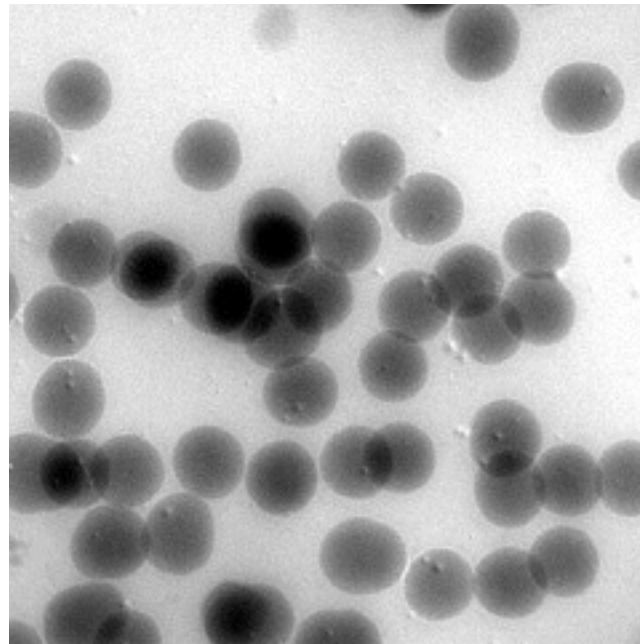


Nanocomposite

USAXS using medium-length beamline

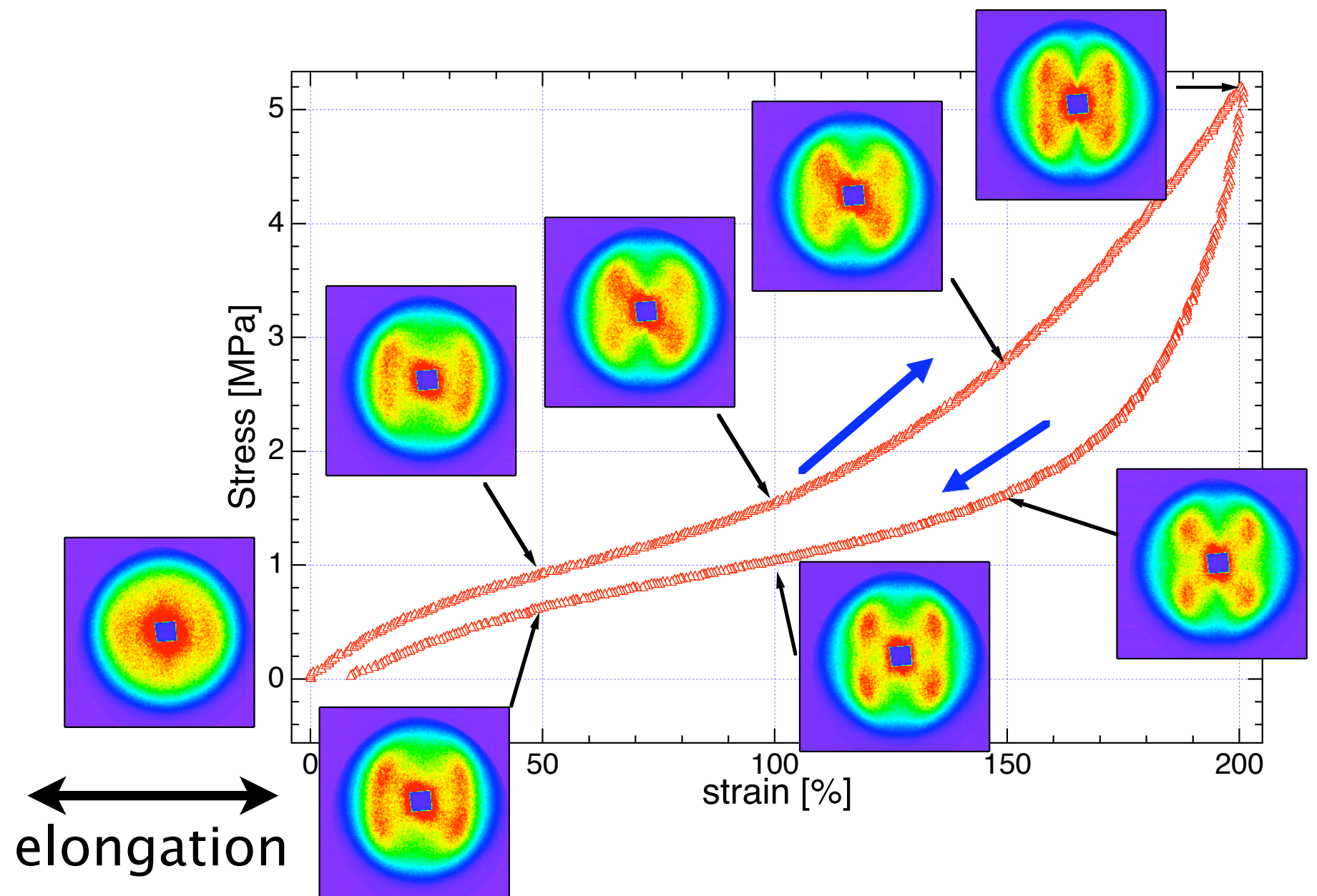


USAXS patterns from elongated rubber



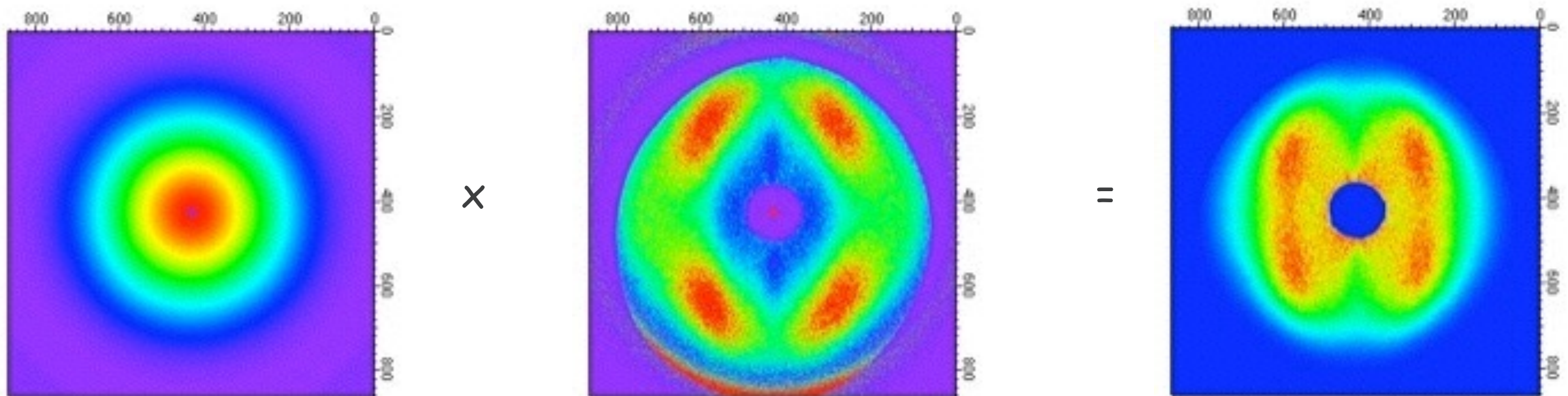
TEM image

Rubber filled with spherical silica



Scattering pattern also shows hysteresis.

Separation of Structure factor $S(q)$



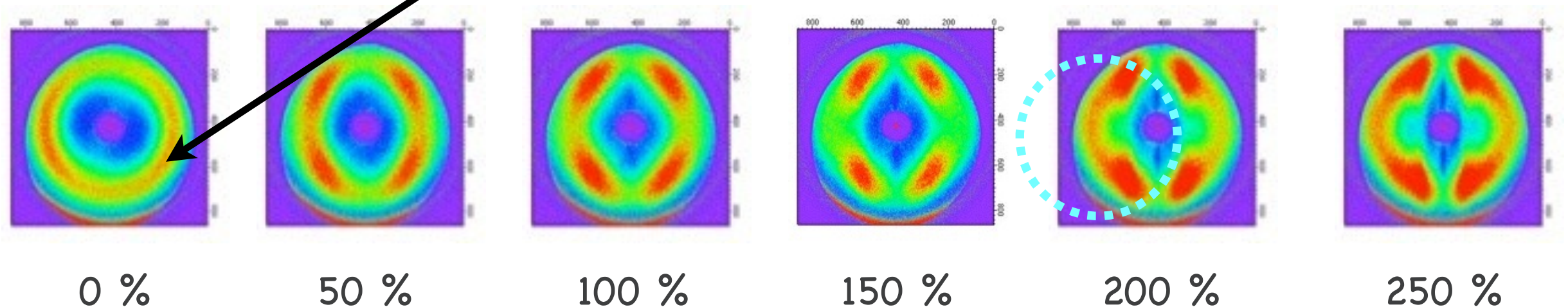
$F(q)$: form factor

$S(q)$: structure factor

$I(q)$: intensity

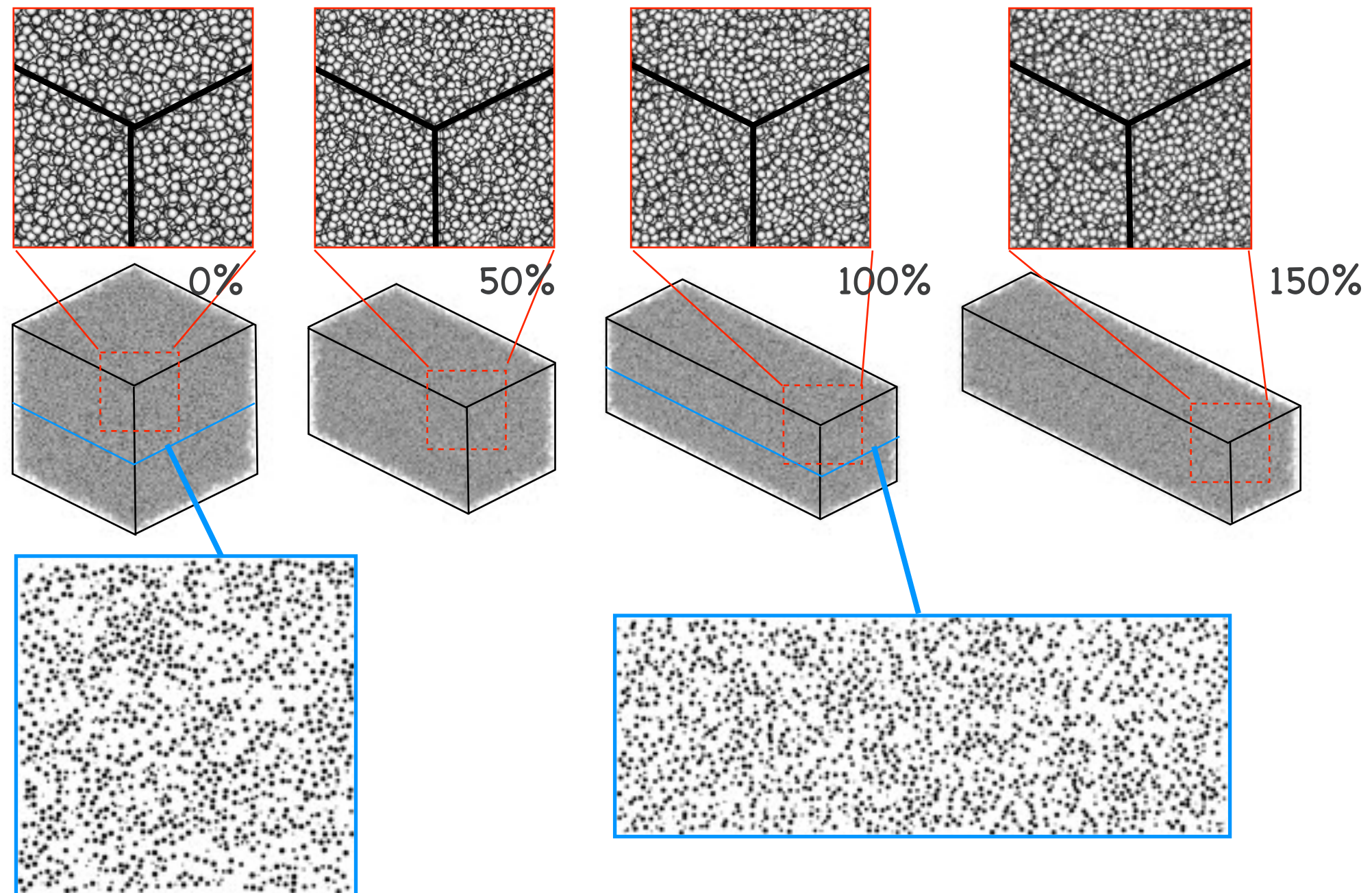
$D_{ave} = 282.9 \text{ nm}$

corresponding to distances between silica particles



Analysis by RMC (Reverse Monte Carlo)

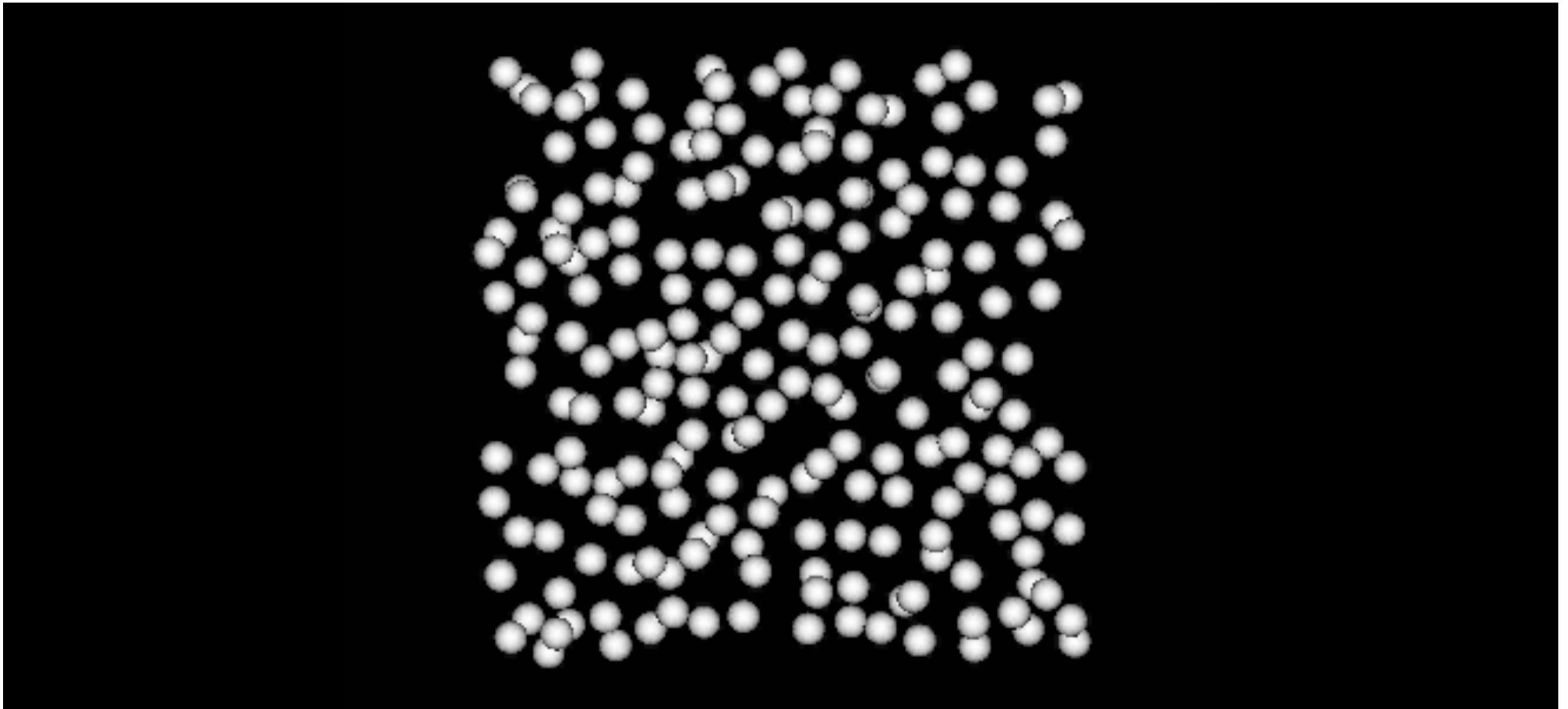
Sample



Courtesy to Dr.Hagita & Prof. Arai

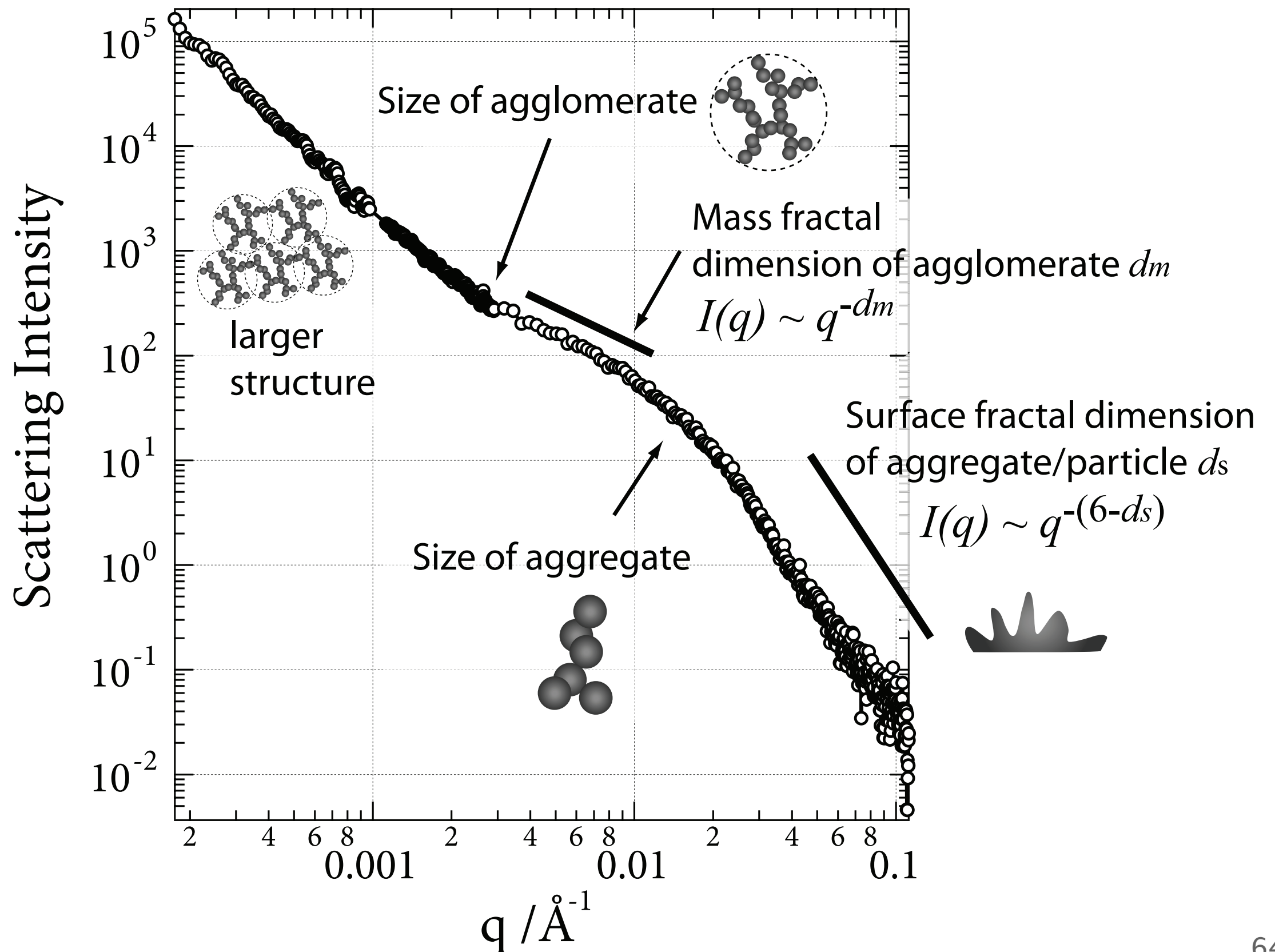
Visualization of structure change of fillers during elongation of rubber by using SAXS and RMC

0 → 150% : elongation process



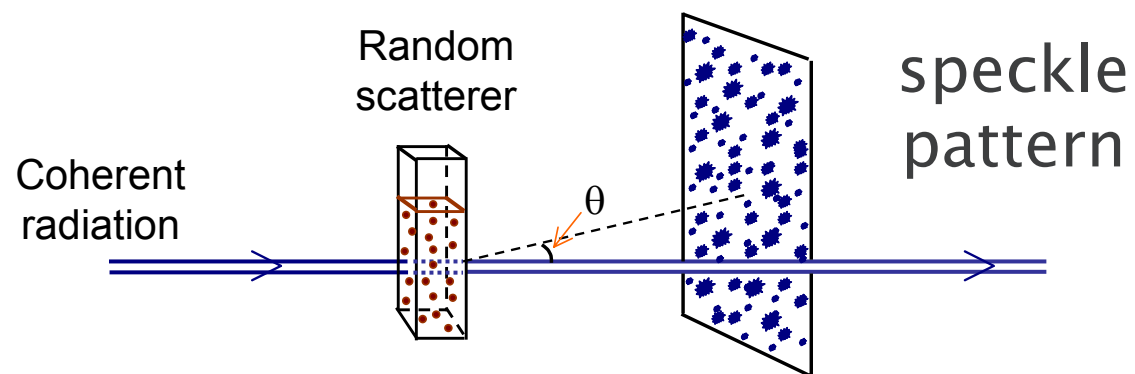
non-uniformity increases along the elongation direction.

Structural information from USAXS



X-ray Photon Correlation Spectroscopy: XPCS

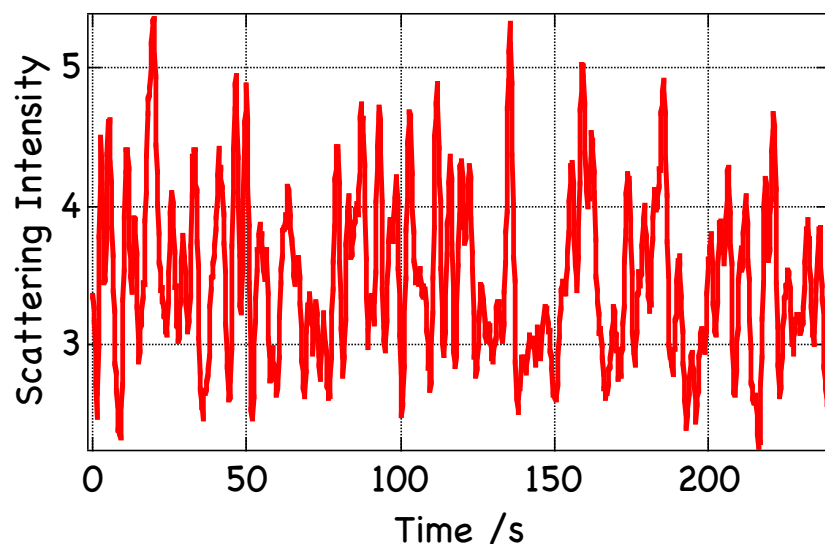
- Measurement of fluctuation of X-ray scattering intensity
--> Structural fluctuation in sample



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

Time-resolved SAXS with coherent X-ray

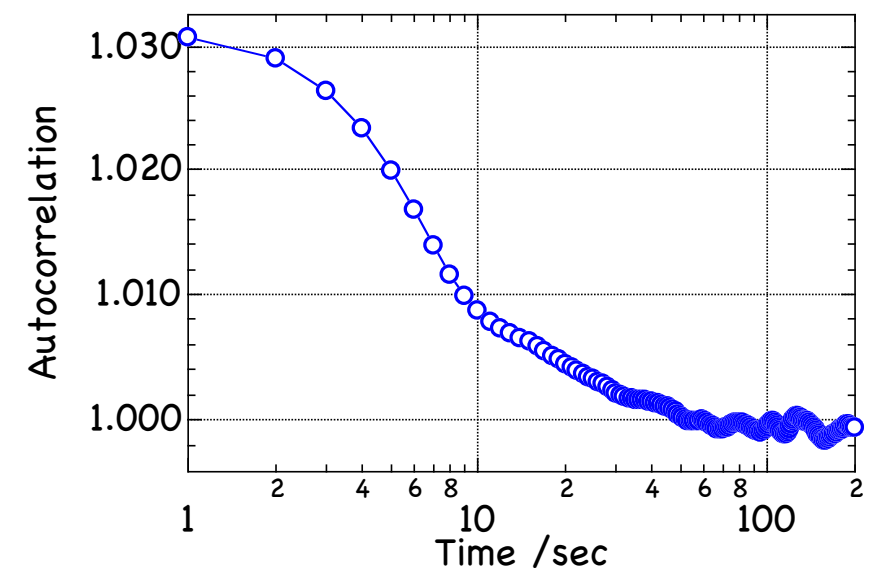
Fluctuation of intensity



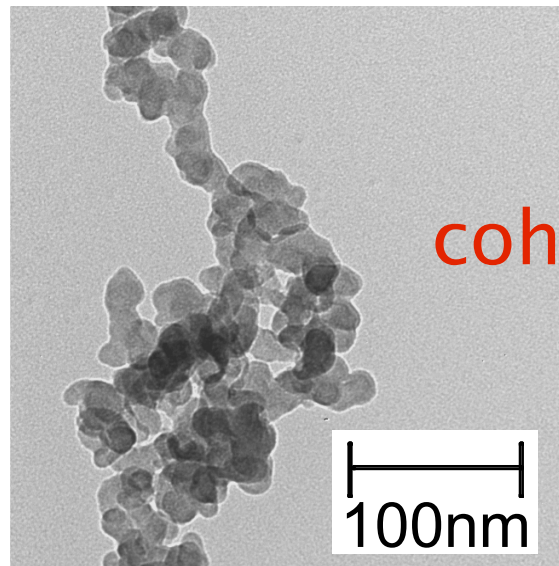
Autocorrelation



relaxation time in system

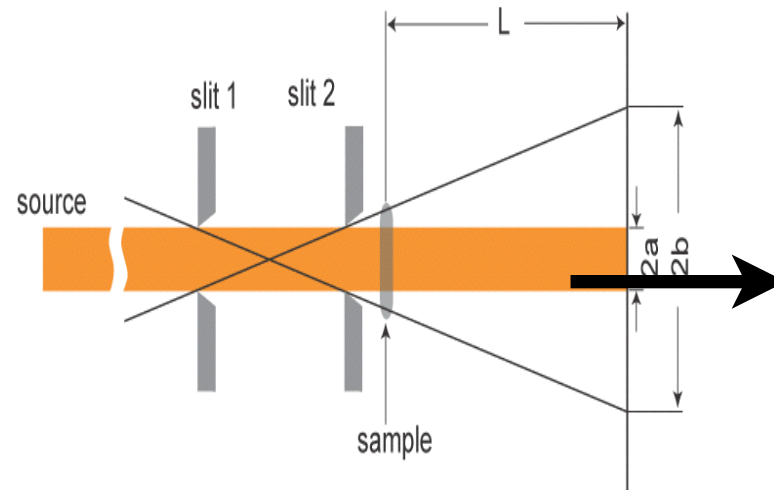


Dynamics of nanoparticles observed with XPCS

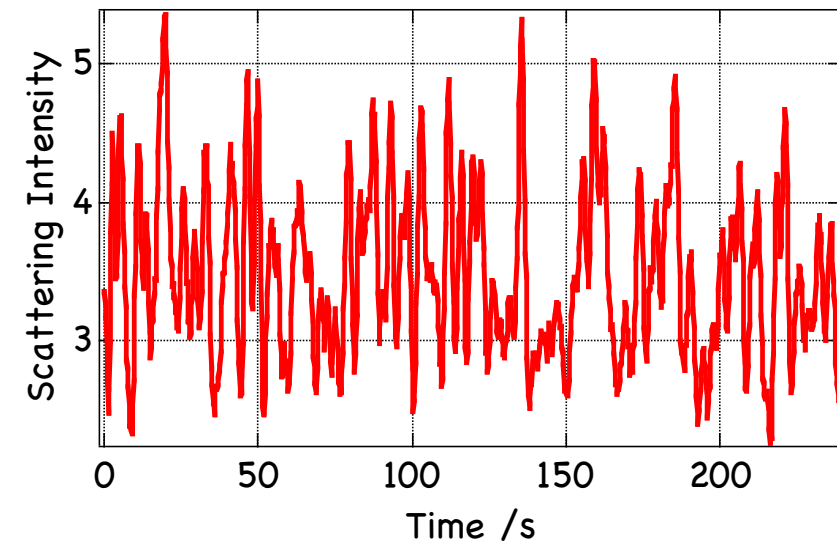


nano-particles in rubber

coherent x-ray



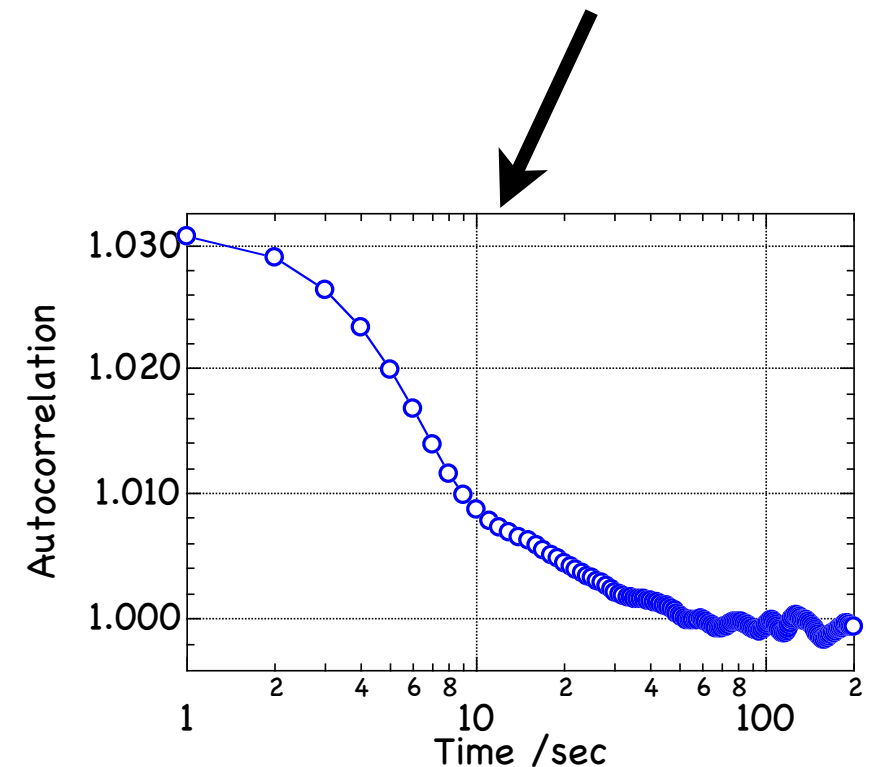
speckle pattern



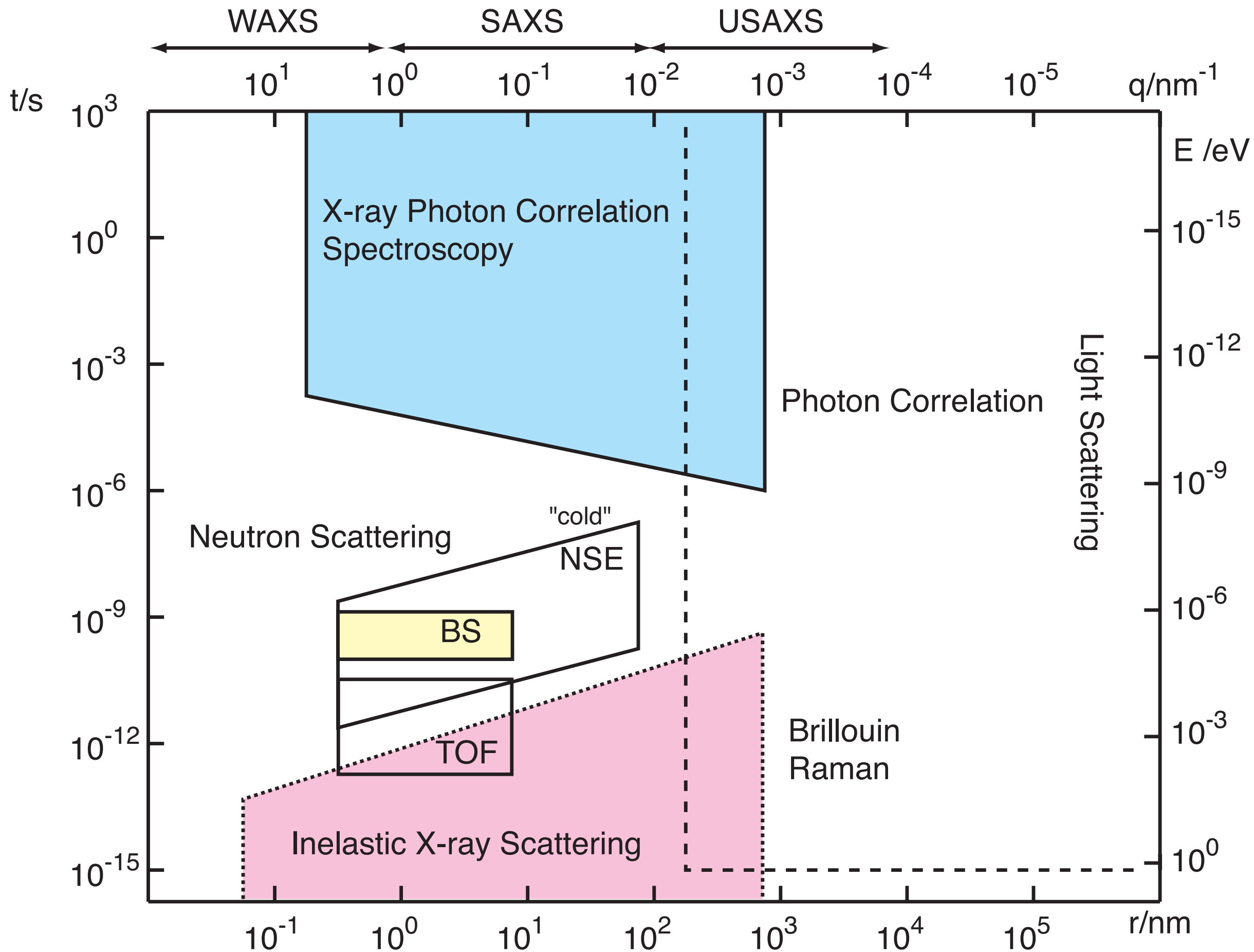
fluctuation of scattering intensity

Dependence of dynamics on...

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



Dynamics of Filler in Rubber



Bibliography

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Sons, New York.
- ❧ O. Glatter and O. Kratky ed. (1982) "Small Angle X-ray Scattering" Academic Press, London.
- ❧ L. A. Feigin and D. A. Svergun (1987) "Structure Analysis by Small Angle X-ray and Neutron Scattering" Plenum Press.
- ❧ P. Lindner and Th. Zemb ed. (2002) "Neutron, X-ray and Light Scattering: Soft Condensed Matter", Elsevier.
- ❧ Proceedings of SAS meeting (2003 & 2006). Published in J. Appl. Cryst.
- ❧ R-J. Roe (2000) "Methods of X-ray and Neutron Scattering in Polymer Science", Oxford University Press.