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Practical XRD with Confidence

Episode 4 – Beyond Diffraction: Exploring Nanostructure with SAXS, WAXS, and X-Ray Reflectivity

Wednesday, June 17, 2026, at 1 pm CDT

Presenter: Keisuke Saito, PhD

Host: Tom Concolino, PhD

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Episode 4 – Beyond Diffraction: Exploring Nanostructure with SAXS, WAXS, and X-Ray Reflectivity

- *You will be muted during the workshop*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working*

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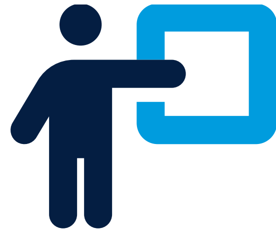

Presenter:
Keisuke Saito
Director, Application Science

Host:
Tom Concolino
Sales Manager, XRD

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You can ask questions during the presentation. Please use the Q&A to ask questions.

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Recording will be available tomorrow.

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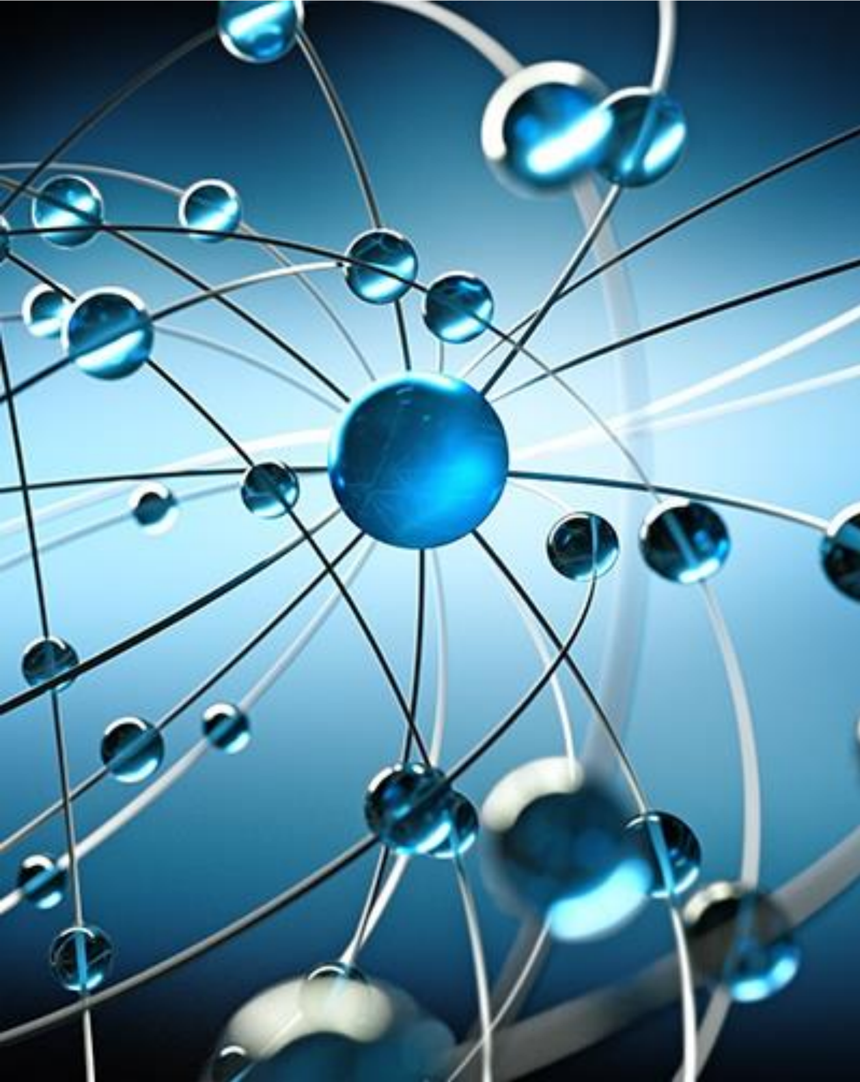

What I will cover today:

1. X-ray scattering fundamentals
2. SAXS fundamentals
3. SAXS setup & sample preparation
4. Sizing nanostructures with SAXS
5. Case study: oriented polymers
6. Case study: mesoporous silica
7. XRR fundamentals
8. XRR modeling & fitting

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1. X-ray scattering fundamentals

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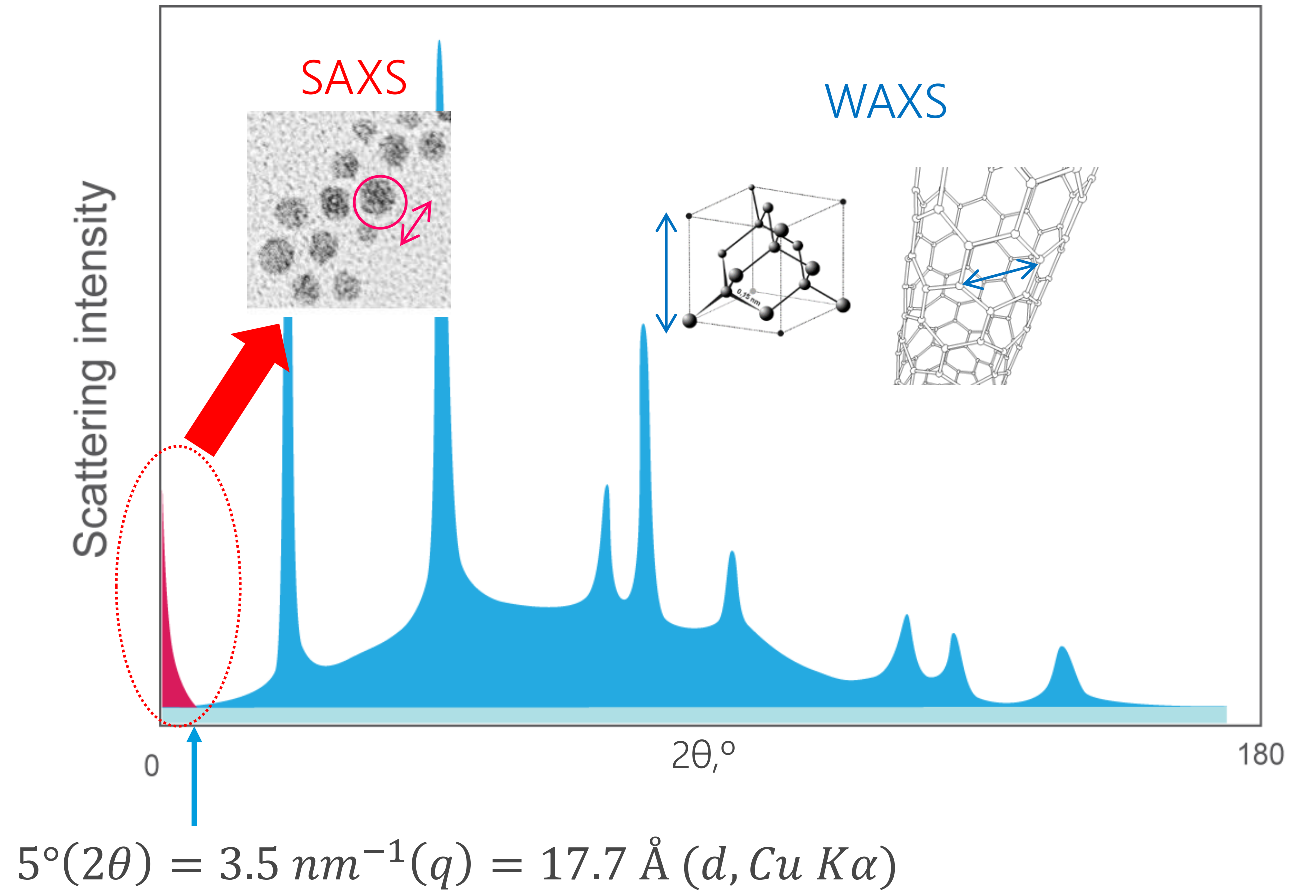
SAXS & WAXS



$$5°(2\theta) = 3.5\ nm^{-1}(q) = 17.7\ Å\ (d, Cu\ K\alpha)$$

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Comparison of X-ray scattering and diffraction techniques

Technique	Structure size	What causes signal?	Information obtained
SAXS	1 ~ 1,000 nm	Electron density fluctuations	Particle size, pores, aggregates
WAXS	0.1 ~ 10 nm	Atomic correlations	Crystal structure, phase, crystallinity, local order
XRD	0.1 ~ 1 nm	Bragg diffraction	Crystal structure, phase
XRR	0.1 ~ 1,000 nm	Reflection from interfaces	Thickness, density, roughness

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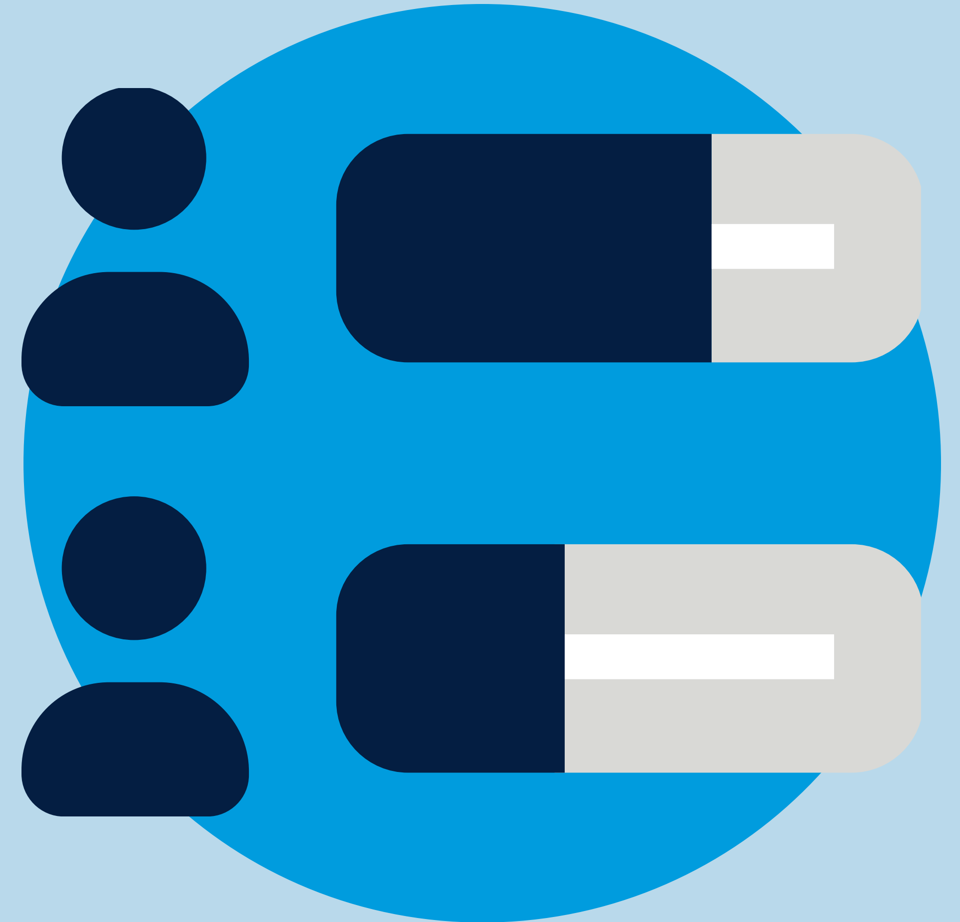
Polling Question

#1 Who has previously performed SAXS, WAXS, XRD or XRR?

1. SAXS
2. WAXS
3. XRD
4. XRR



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2. SAXS fundamentals

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Scattering vector

XRD

- $|\boldsymbol{k}| = \frac{1}{\lambda}$
- $\boldsymbol{K} = 2 \cdot |\boldsymbol{k}| \cdot \sin\theta = \frac{2}{\lambda} \cdot \sin\theta = \frac{1}{d_{hkl}} = \boldsymbol{g}_{hkl}$

XRD: reciprocal spacing $1/d$.

SAXS / WAXS

- $|\boldsymbol{k}| = \frac{2\pi}{\lambda}$
- $\boldsymbol{q} = 2 \cdot |\boldsymbol{k}| \cdot \sin\theta = \frac{4\pi}{\lambda} \cdot \sin\theta = \frac{2\pi}{d_{hkl}}$

SAXS/WAXS: Fourier-transform convention q

λ: wavelength of X-ray
$\boldsymbol{K}, \boldsymbol{q}$: scattering vector
$\boldsymbol{k}$: X-ray wave vector
$\boldsymbol{g}_{hkl}$: reciprocal lattice vector
θ: Bragg angle
d_{hkl}: interplanar spacing

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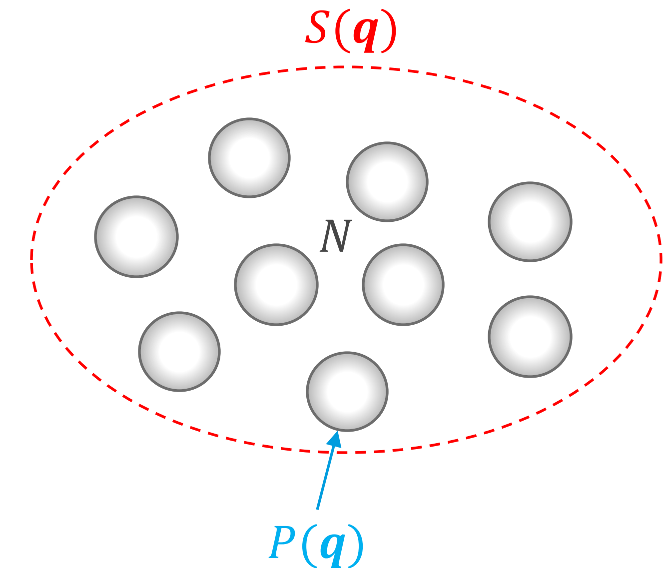
SAXS: scattering intensity - isotropic case

- $I(\boldsymbol{q}) \propto N \cdot P(\boldsymbol{q}) \cdot S(\boldsymbol{q})$
  - N: number of nano particles
  - $P(\boldsymbol{q})$: form factor
  - $S(\boldsymbol{q})$: structure factor



- What shape/size are the particles? → $P(\boldsymbol{q})$
- How are they arranged? → $S(\boldsymbol{q})$

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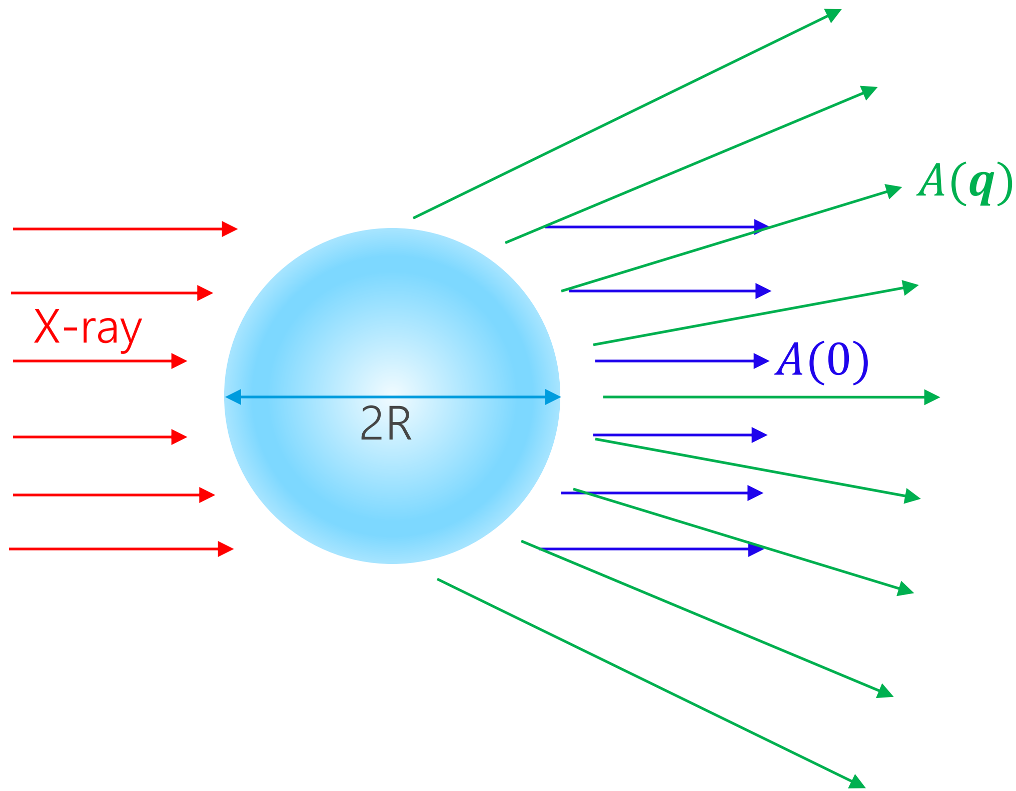
Form factor $P(q)$ for sphere



- Scattering amplitude for sphere
  - $A(\boldsymbol{q}) = \int \rho(\boldsymbol{r}) e^{-i\boldsymbol{q}\cdot\boldsymbol{r}} d\boldsymbol{r}$

$$= \rho V \left[\frac{\sin(qR) - qR\cos(qR)}{(qR)^3}\right]$$

- Form factor
  - $P(q) = \left|\frac{A(q)}{A(0)}\right|^2 = \left[3\frac{\sin(qR) - qR\cos(qR)}{(qR)^3}\right]^2$

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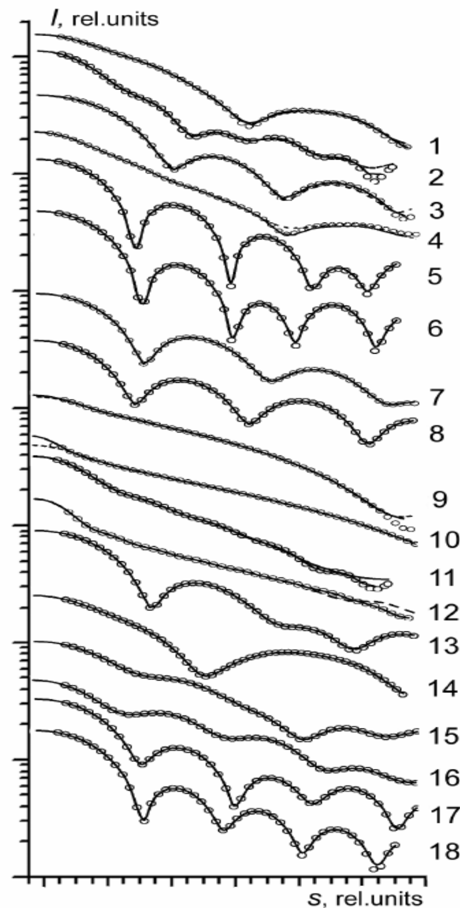
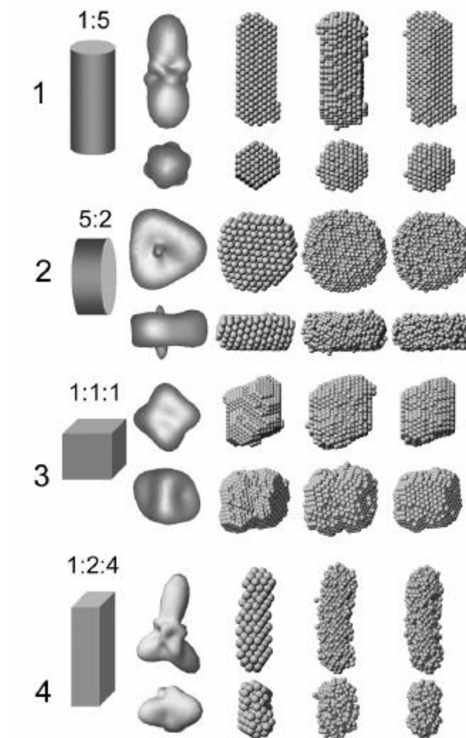
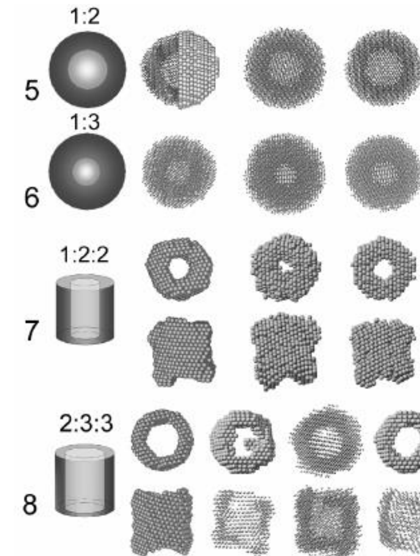
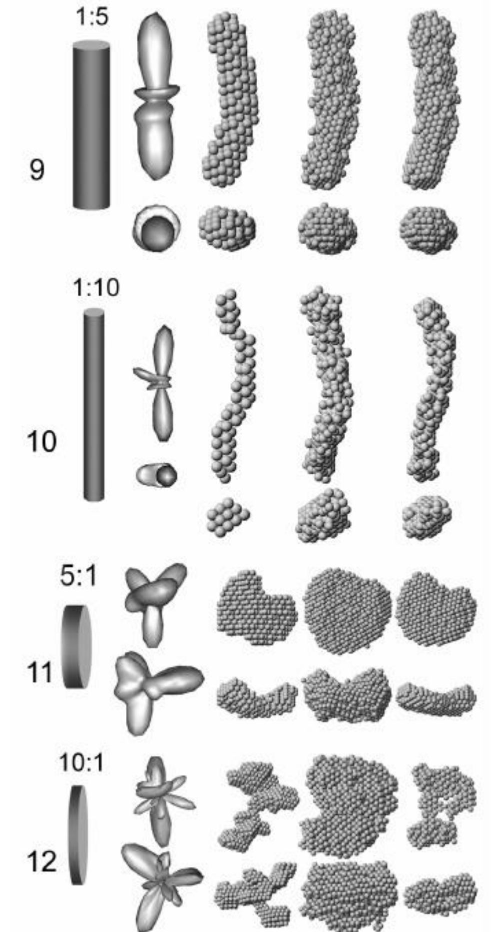
Form factor $P(q)$ for other shapes









[V. Volkov, D. Svergun, J.Appl.Cryst. (2003) 36, 860-864.]

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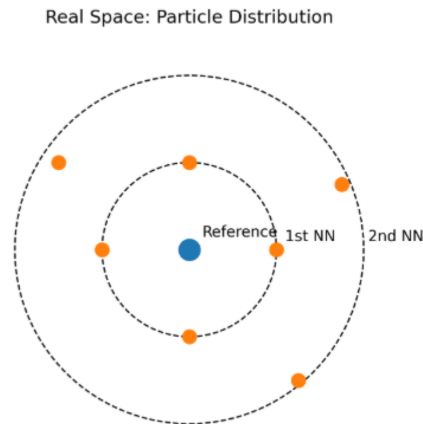
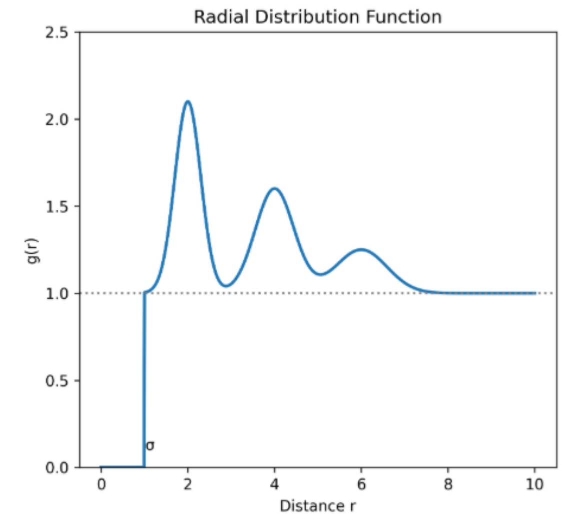
Structure factor $S(q)$

- $S(\boldsymbol{q}) = 1 + \int_v (n(\boldsymbol{r}) - n_0)\, e^{i\boldsymbol{q}\cdot\boldsymbol{r}} d\boldsymbol{r}$
  - $n(\boldsymbol{r})$: particle count density function
  - n_0: average particle density
- $n(\boldsymbol{r})$
  - Radial distribution function (RDF)
  - Percus-Yevick (PY) model for isotropic case

RDF





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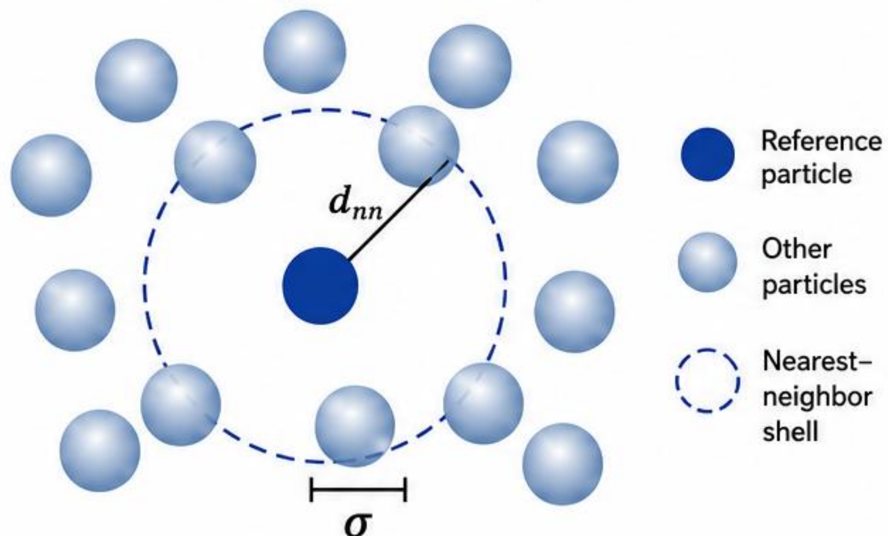
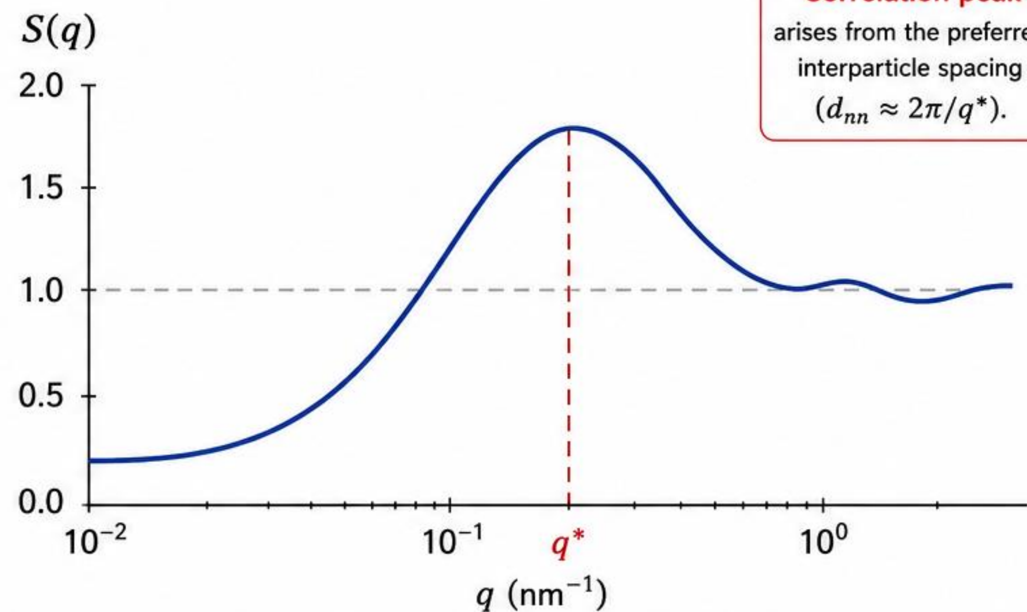
PY model





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3. SAXS setup & sample preparation

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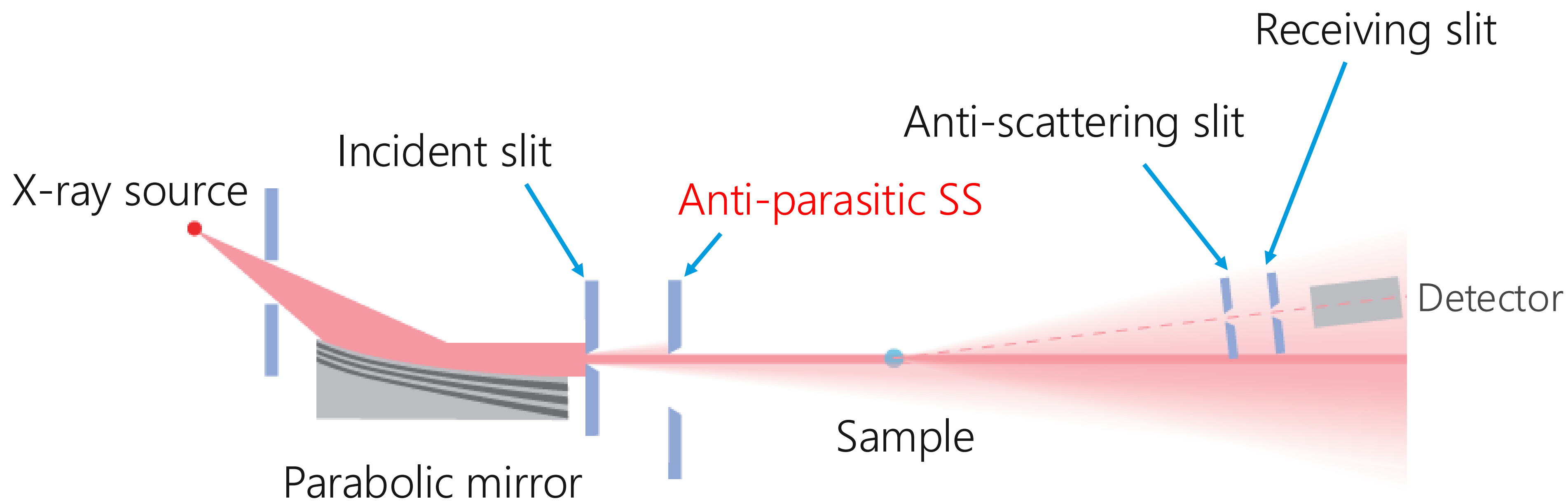
Instrument configurations



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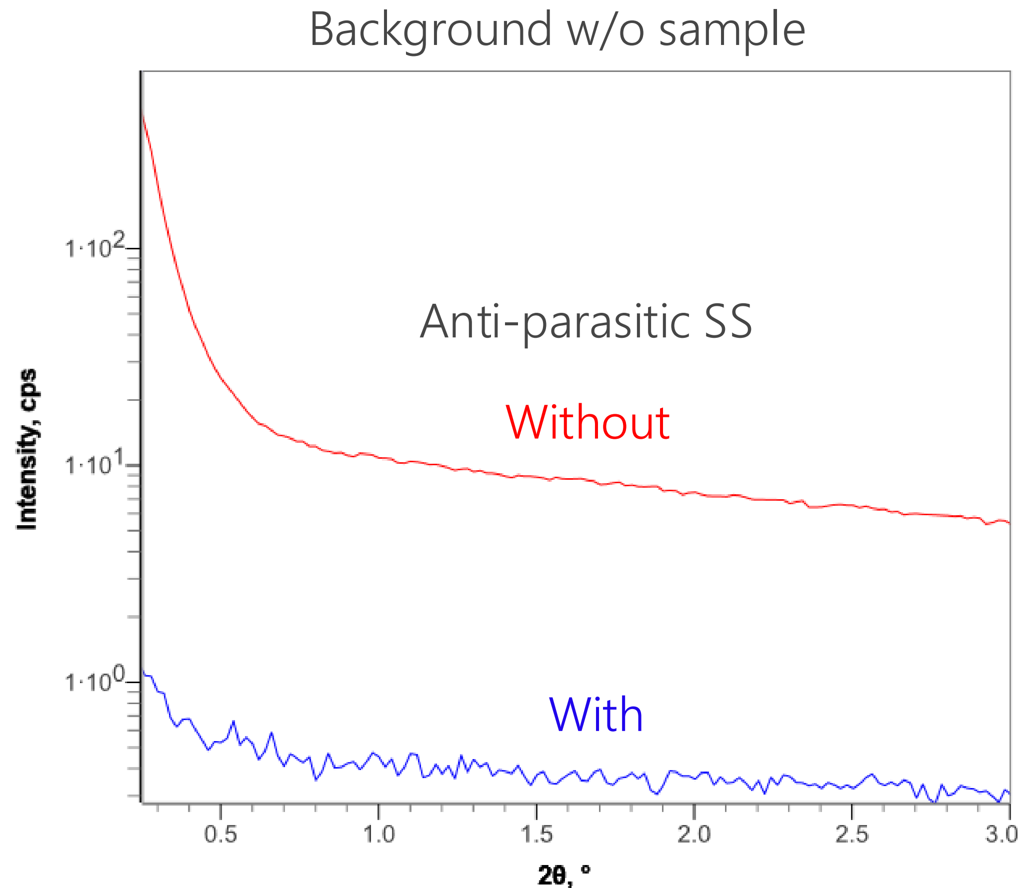
Parasitic scattering



- Parasitic scattering is unwanted low-angle scattering caused by optics such as slits.

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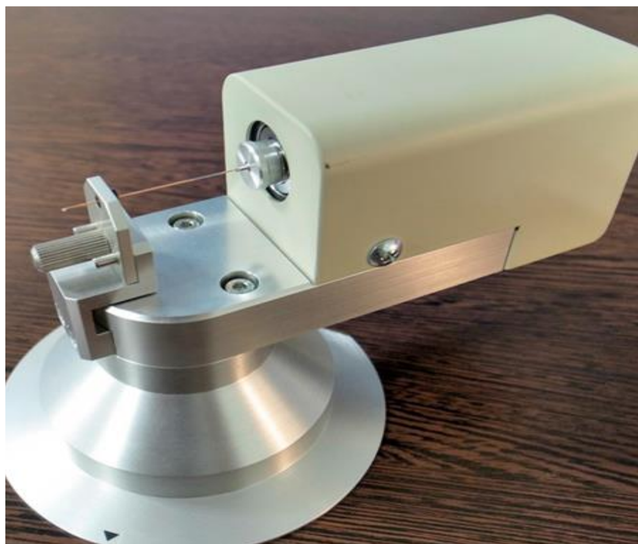
Sample preparation



- Fused silica capillary
- Liquid, powder sample
- 0.3, 0.5, 0.7, 0.9, 1.0 mmφ

- Foil-transmission
- Powder, liquid, gel sample
- Sample to be placed between two mylar / Kapton foils

- $20\ \% \leq T = \frac{I_{sample}}{I_{no\ sample}} \leq 80\ \%$
  - T: transmittance
  - I_{sample}: intensity transmits through sample and holder
  - $I_{no\ sample}$: intensity transmits through holder without sample

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4. Sizing nanostructures with SAXS

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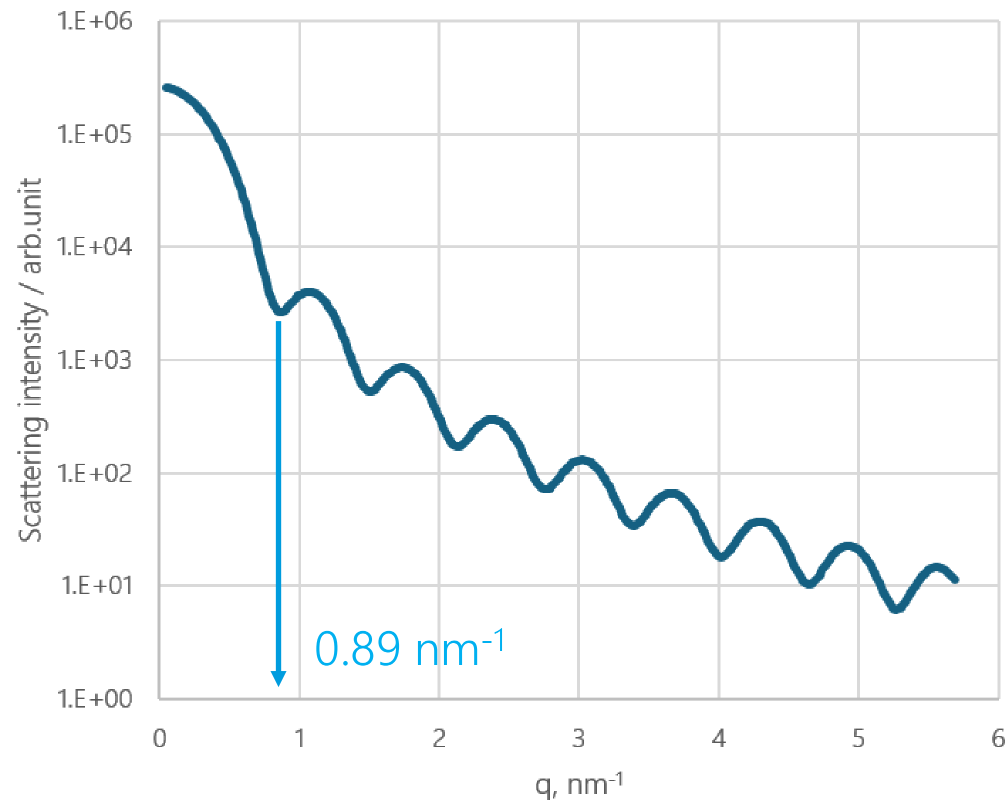
Particle size estimation (1): peak search



- Sphere model
  - $P(q) = \left[3\frac{\sin(qR) - qR\cos(qR)}{(qR)^3}\right]^2$
  - 1st minimum $qR \approx 4.493$
- Particle diameter: D (nm)
  - $D = 2R = \frac{4.493}{0.89} \sim 10$

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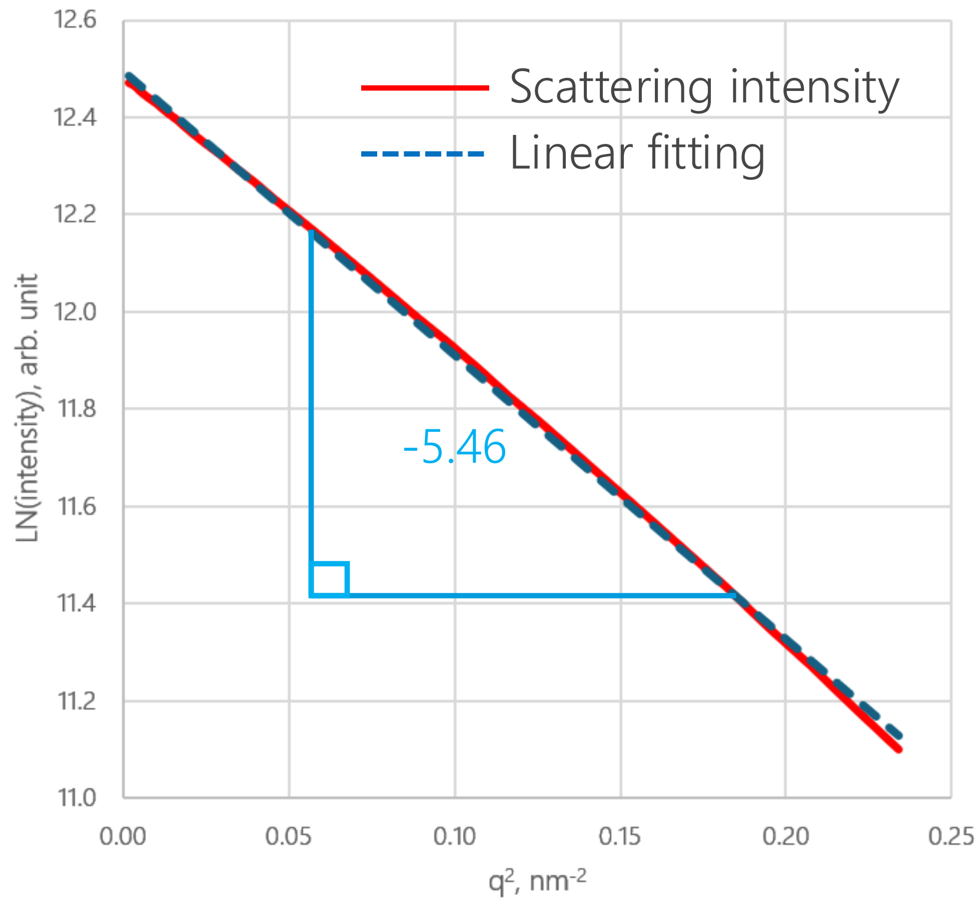
Particle size estimation (2): Guinier plot



- In q range, $qR_g < 1$
  - $I(q) = I(0)exp\left(-\frac{q^2R_g^2}{3}\right)$
  - R_g: radius of gyration
  - $D = 2R = 2\sqrt{\frac{5}{3}}R_g = 2\sqrt{\frac{5}{3}}\sqrt{-3 \cdot slope}$

    $= 10.4\ nm$ (sphere)
  - $q_{max} < \frac{1}{R_g} = 0.247\ nm^{-1}$

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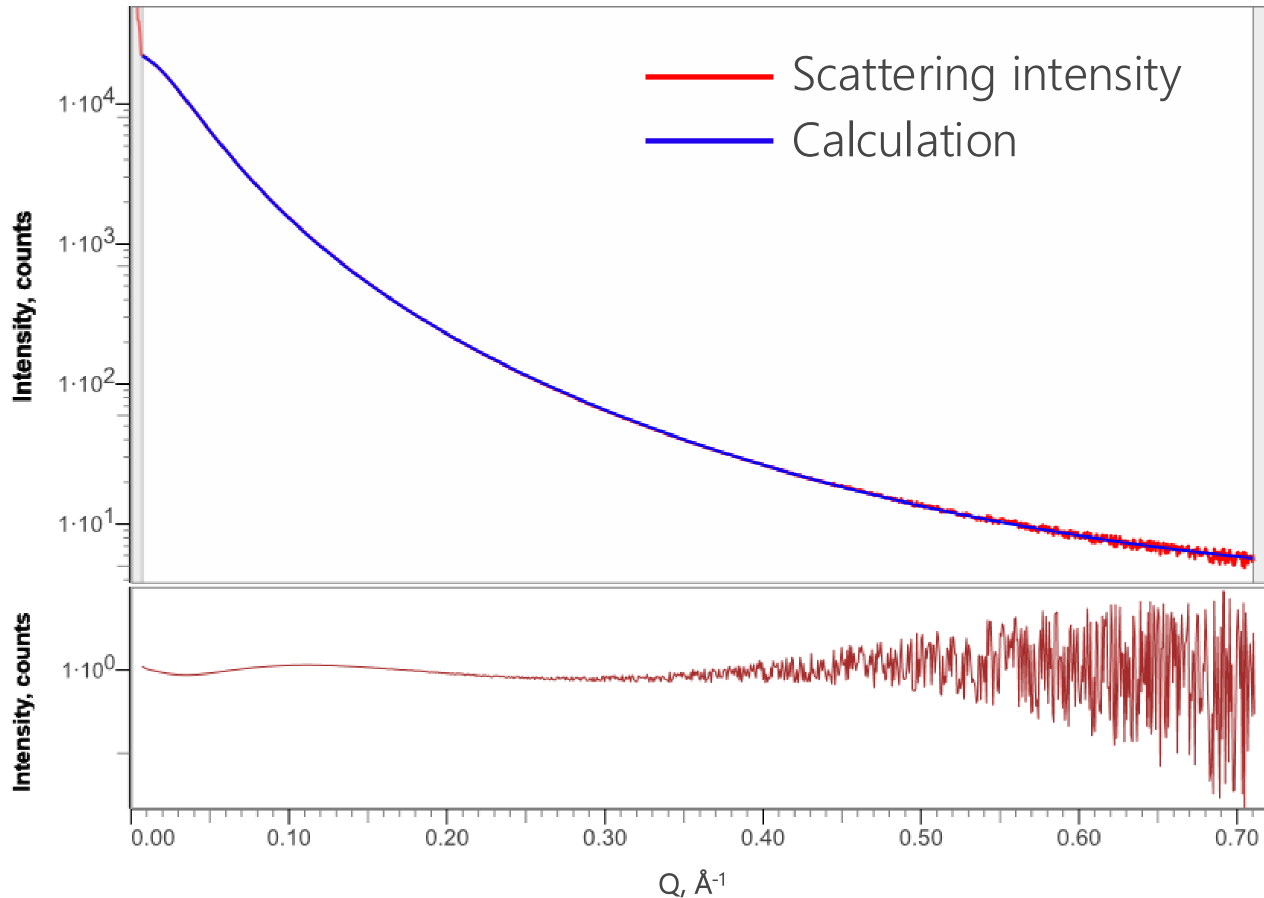
Particle size estimation (3): Model fitting



- Sample
  - Particles: gold (sphere)
  - Matrix: toluene
- Average diameter: 5.5 nm
- Size distribution: 70 %

Pedersen, J.S., "Analysis of Small-Angle Scattering Data from Colloids and Polymer Solutions: Modeling and Least-Squares Fitting," Advances in Colloid and Interface Science, 70, 171–210 (1997).

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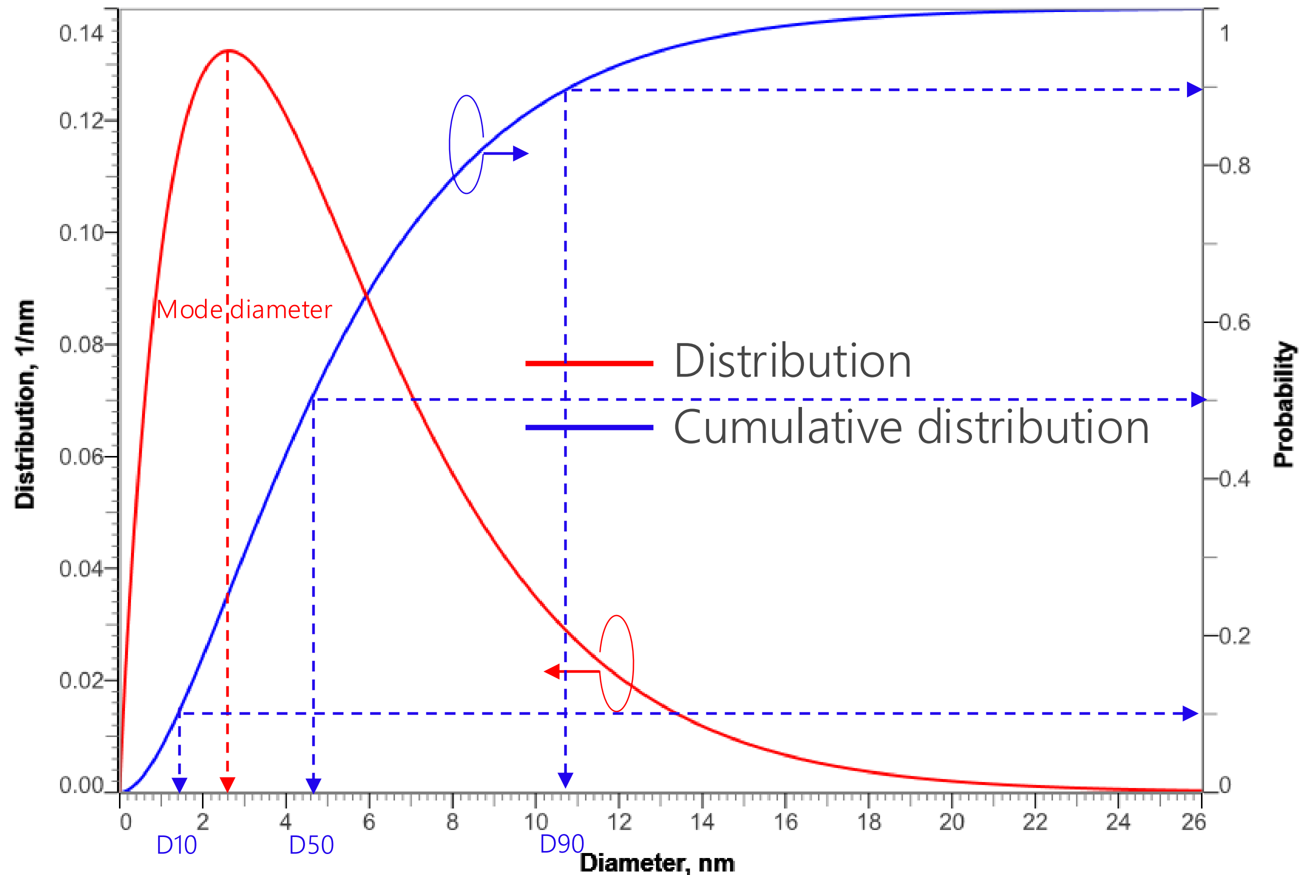
Particle size estimation (3): Model fitting



- Average diameter: 5.5 nm
- Size distribution: 70 %

- Mode diameter: 2.6 nm
- D10: 1.4 nm
- D50: 4.6 nm
- D90: 10.8 nm

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5. Case study: oriented polymers

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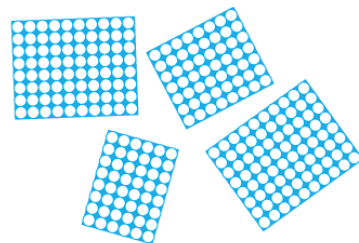
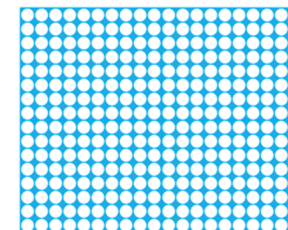
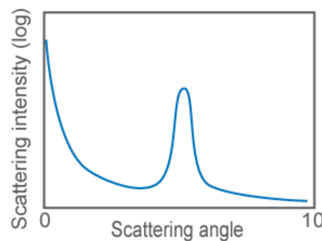
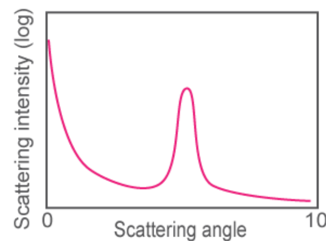
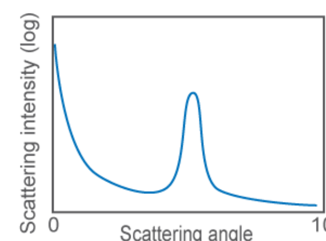
Texture effect

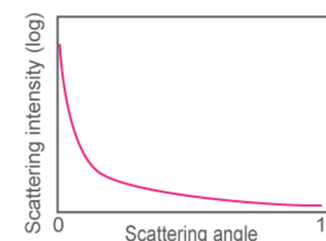


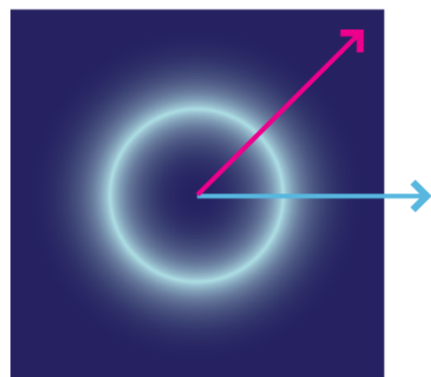


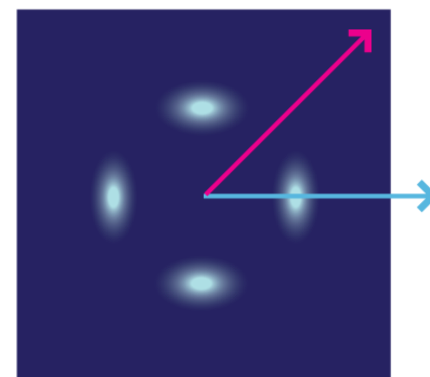












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Instrument configurations – 2D SAXS

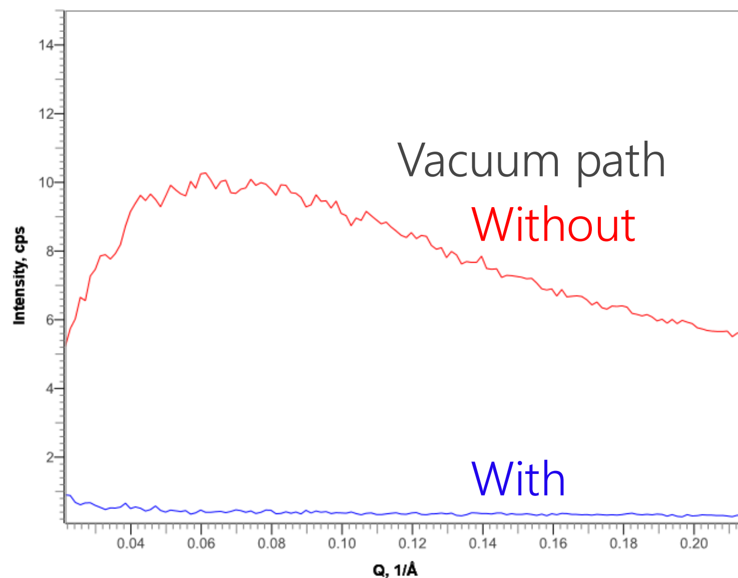
2D-SAXS

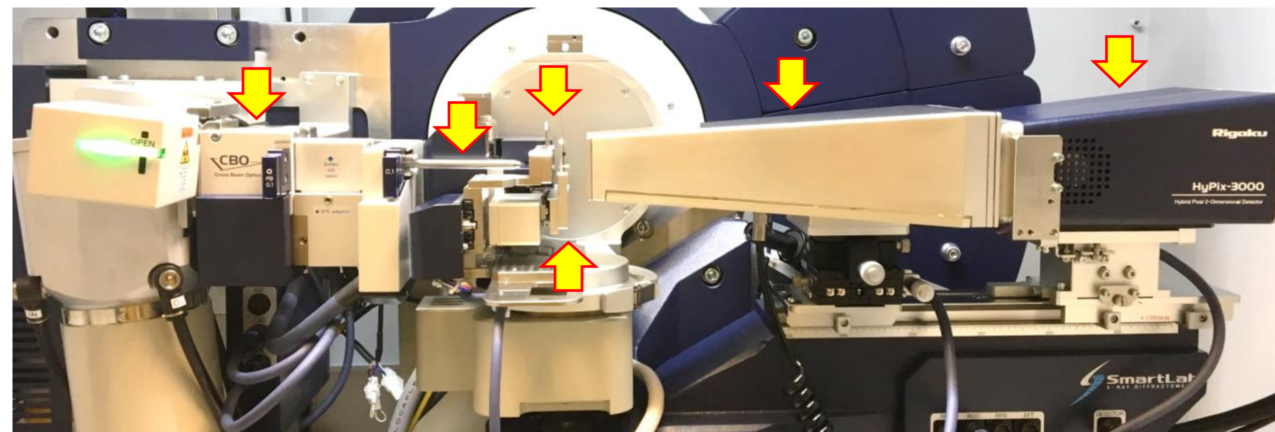
$0.2 < 2\theta < 7.0$ °

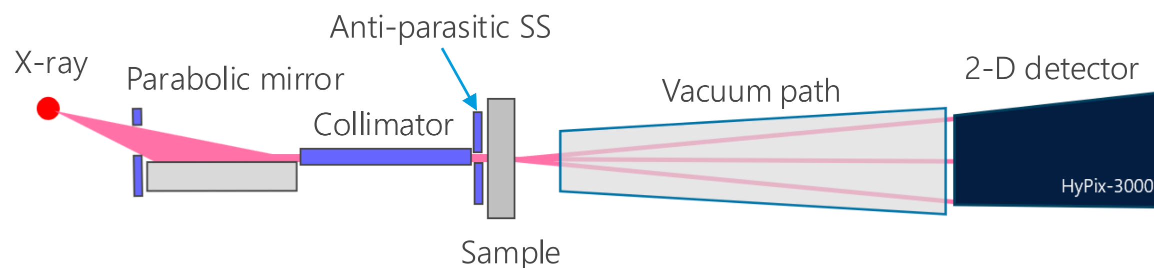
$12.6 < d < 441$ Å

$0.014 < Q < 0.498$ Å^{-1}

Scattering no-sample







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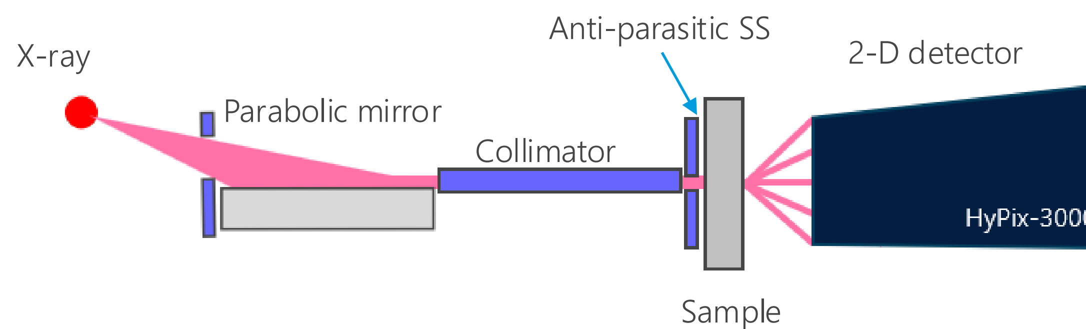
Instrument configurations – 2D WAXS

2D-WAXS

$6.5 < 2\theta < 35$ °

$2.56 < d < 13.59$ Å

$0.46 < Q < 2.45$ Å^{-1}



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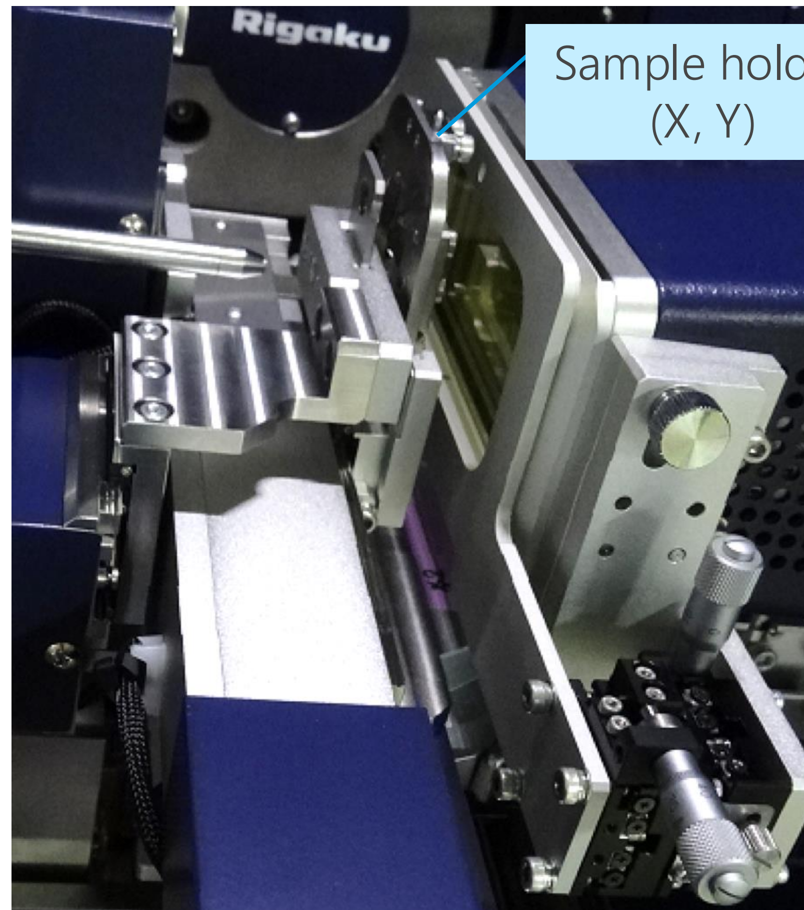
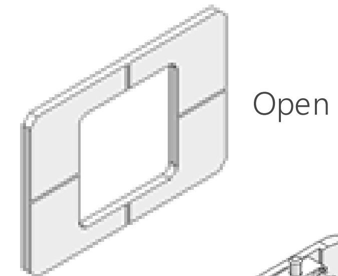
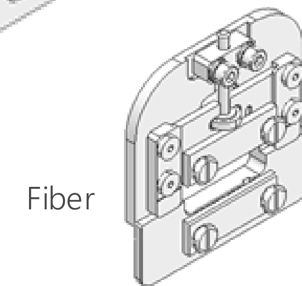
Sample holders



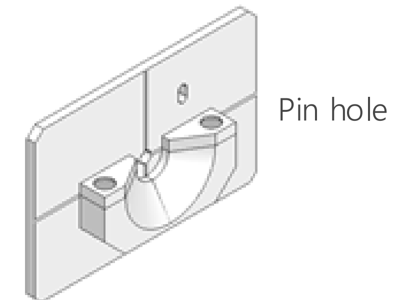
Sample holder
(X, Y)



Open



Fiber



Pin hole

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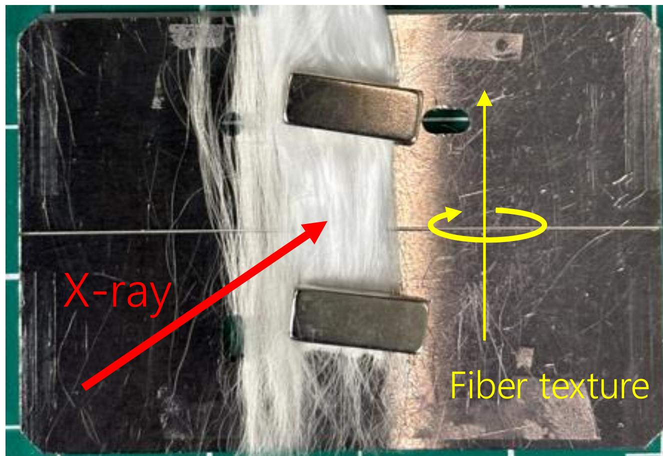
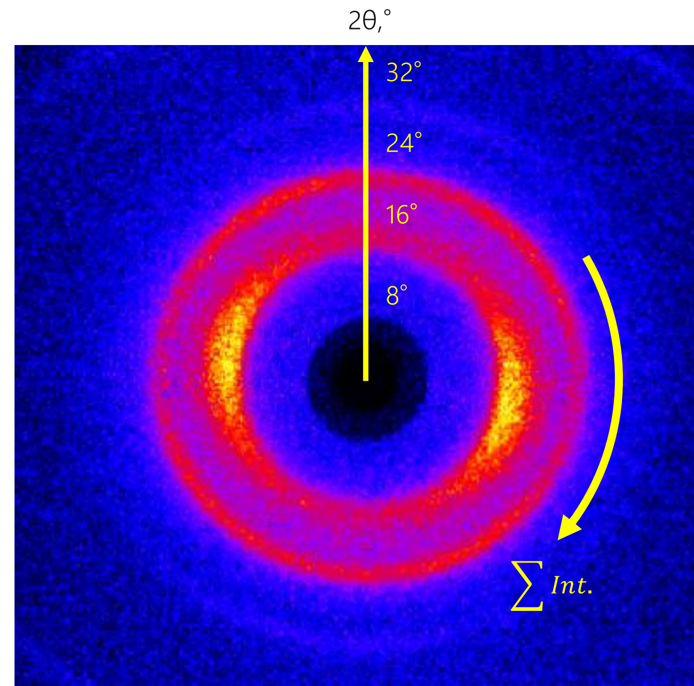
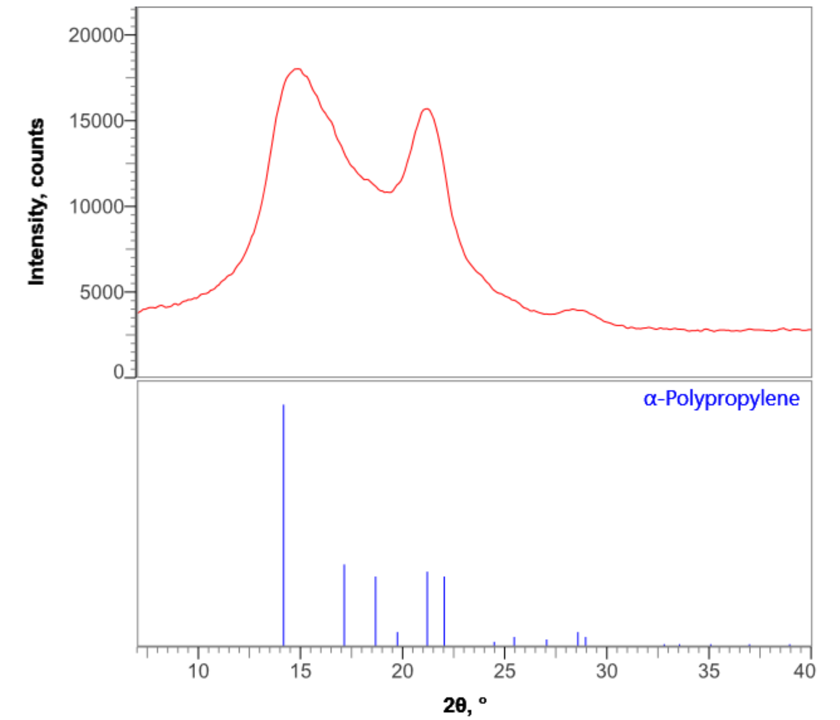
Polymer: WAXS







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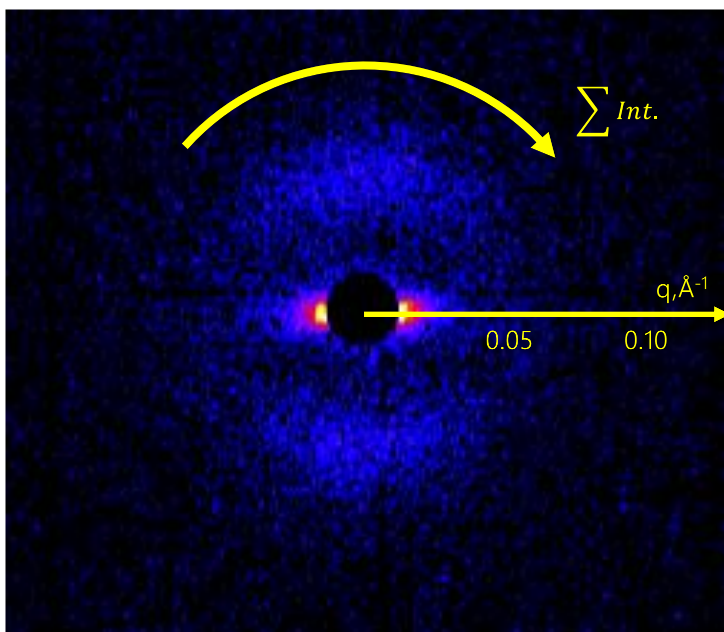
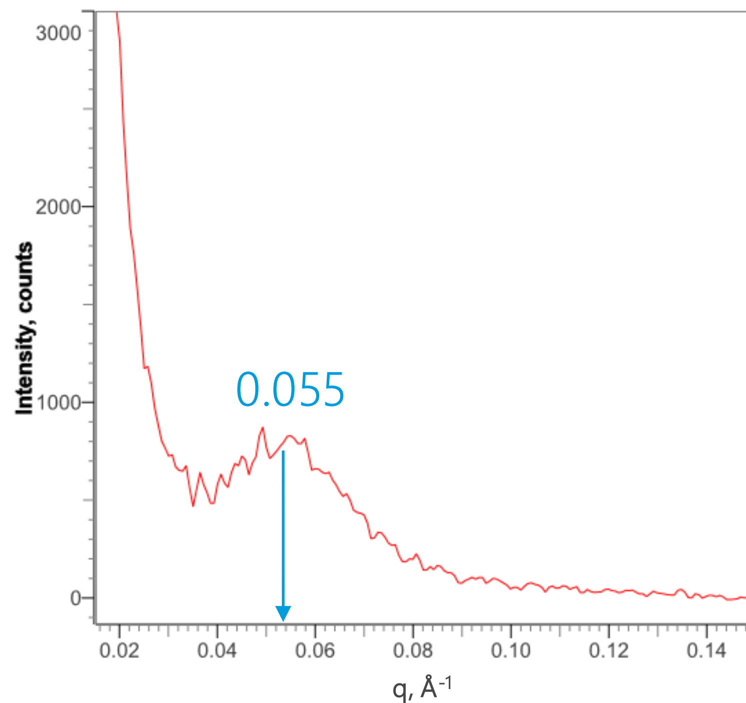
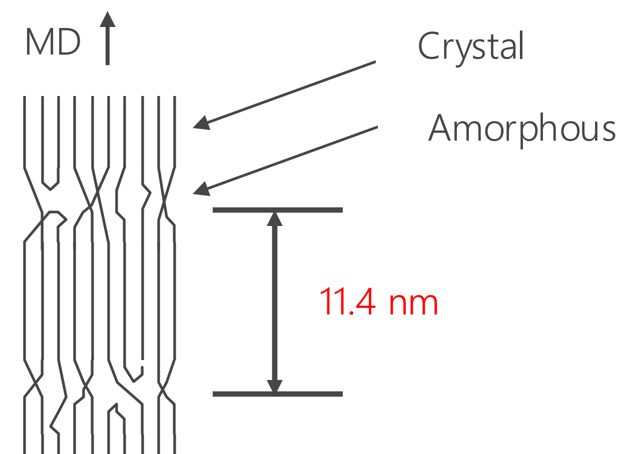
Polymer: SAXS





- $d = \frac{2\pi}{q} = 11.4\ nm$



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6. Case study: mesoporous silica

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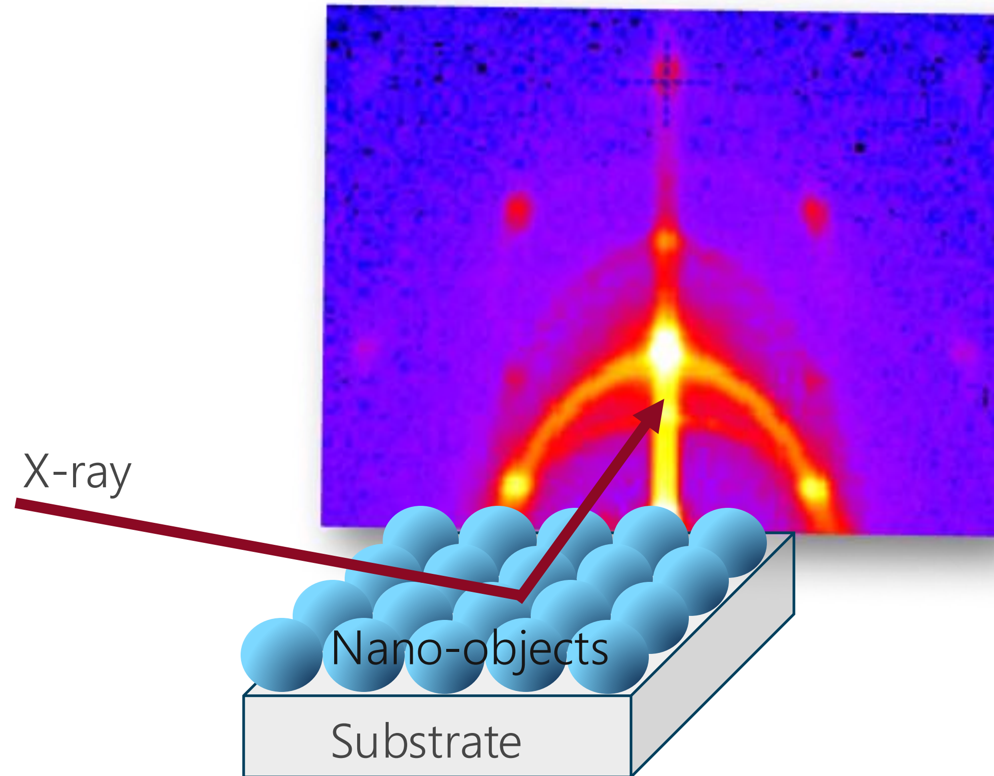
Mesoporous silica: GIWAXS



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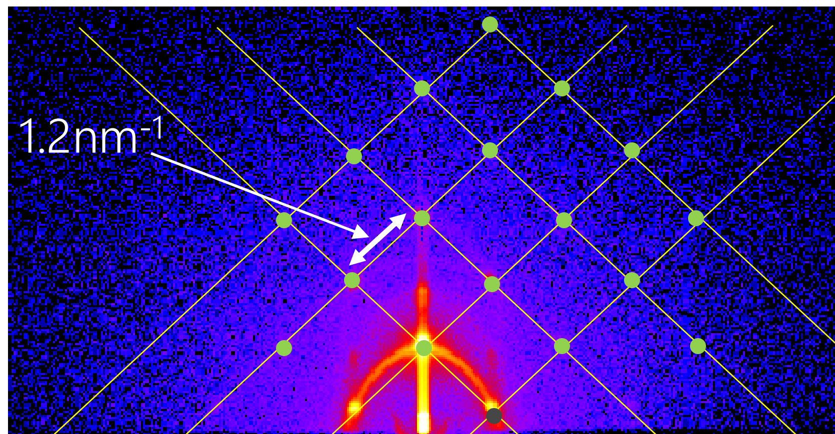
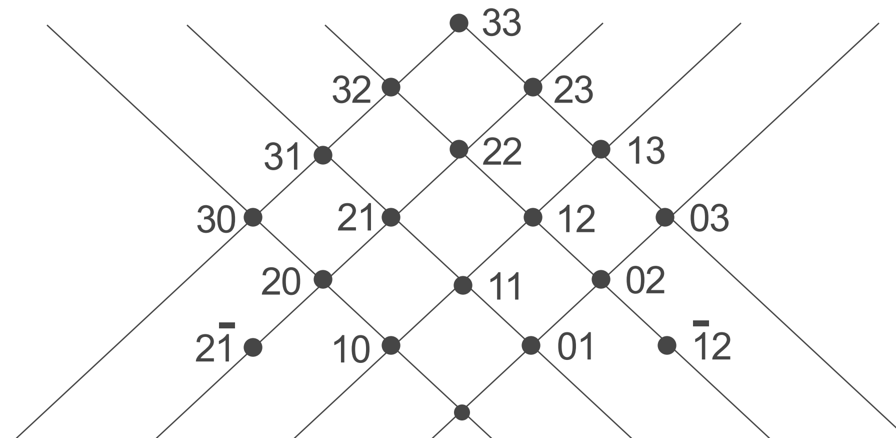
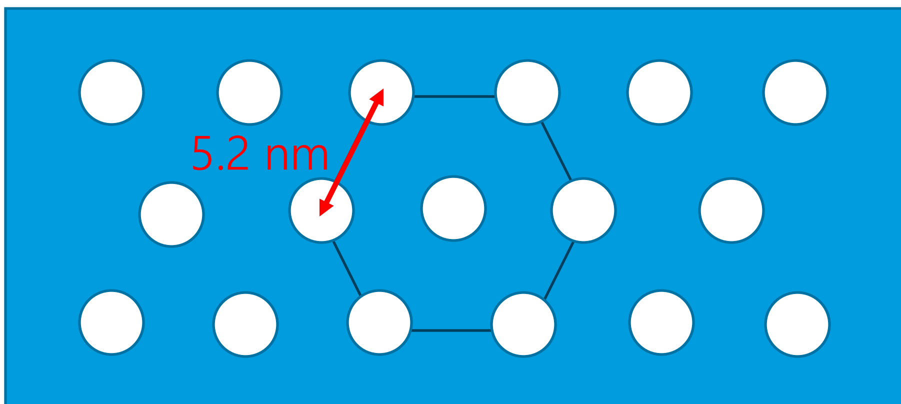
Mesoporous silica: GIWAXS







- $d = \frac{2\pi}{q} = 5.2\ nm$

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Rigaku

Summary

- SAXS and WAXS provide complementary structural information across multiple length scales.
- SAXS profiles contain quantitative information on particle size, shape, size distribution, and interparticle spacing.
- 2D SAXS/WAXS reveals anisotropy and orientation, enabling characterization of textured materials such as fibers, films, and polymers.

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Questions?

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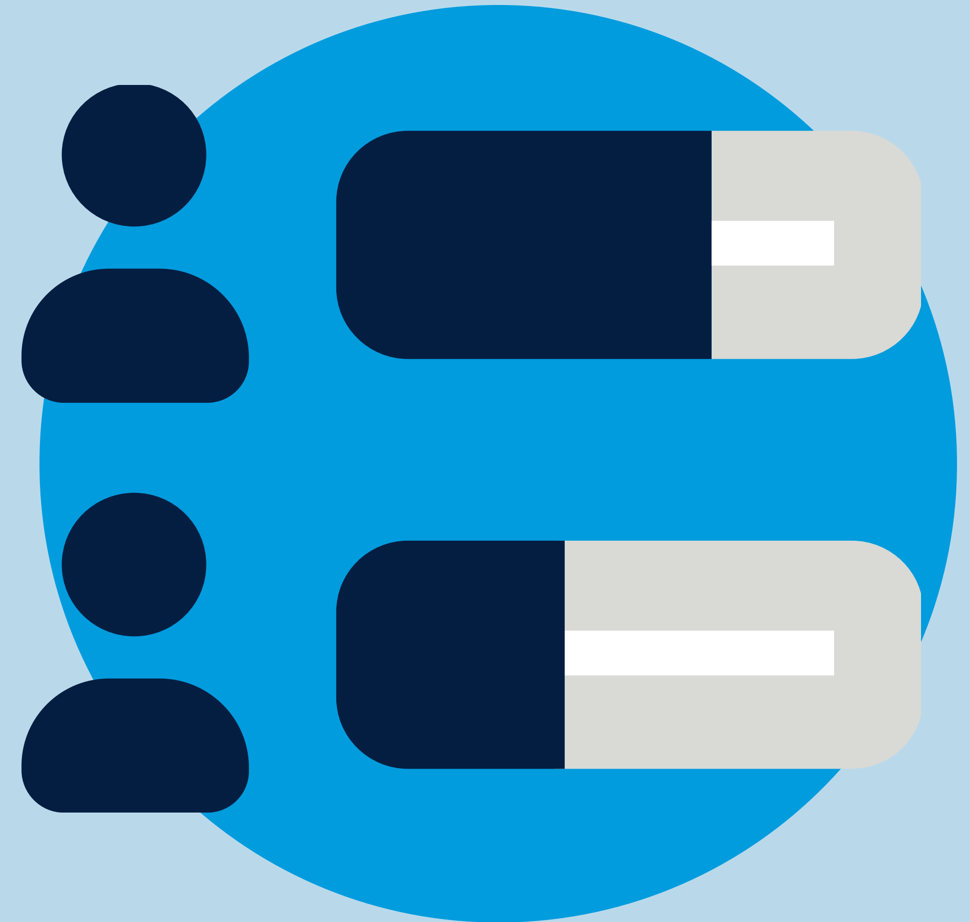
Polling Question

#2 Have you done 0D and 2D SAXS?

1. Yes
2. No
3. Both
4. I don't do SAXS.



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7. XRR fundamentals

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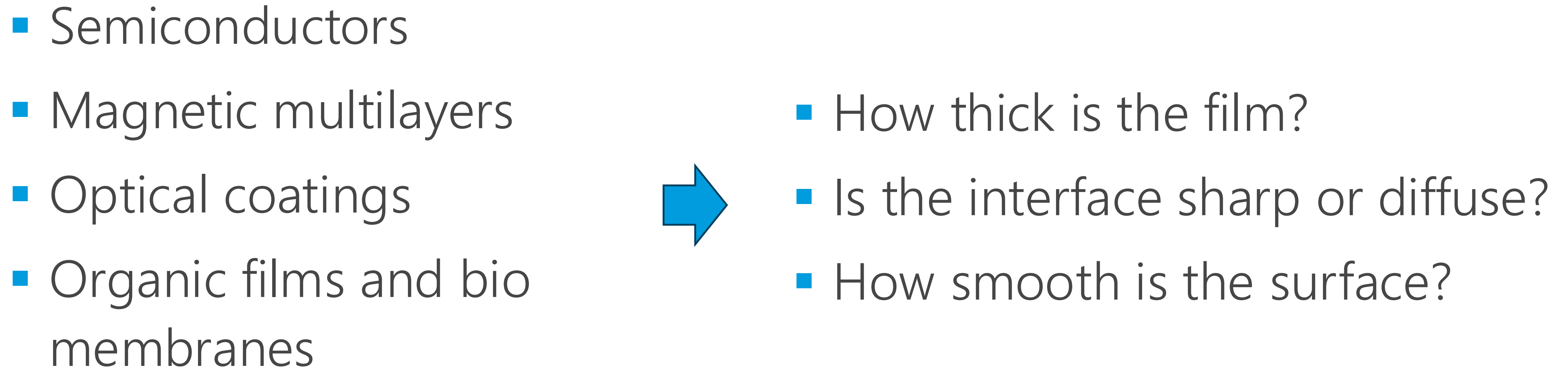
Why do we care about surfaces and thin films?



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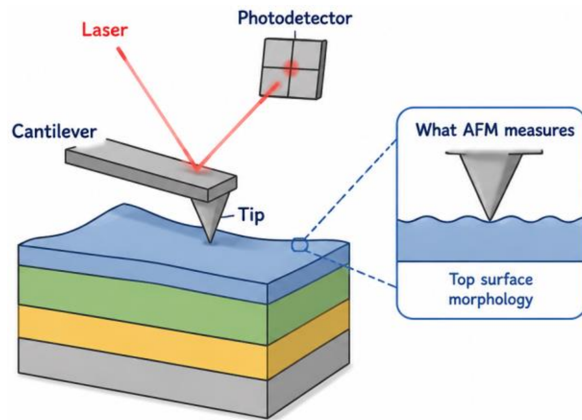
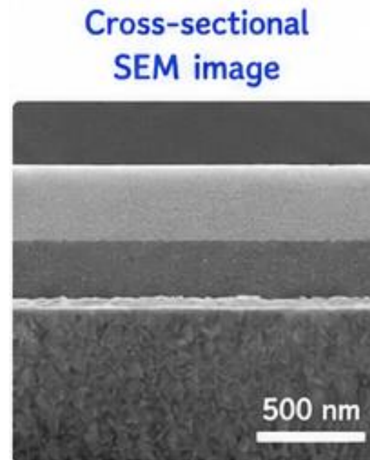
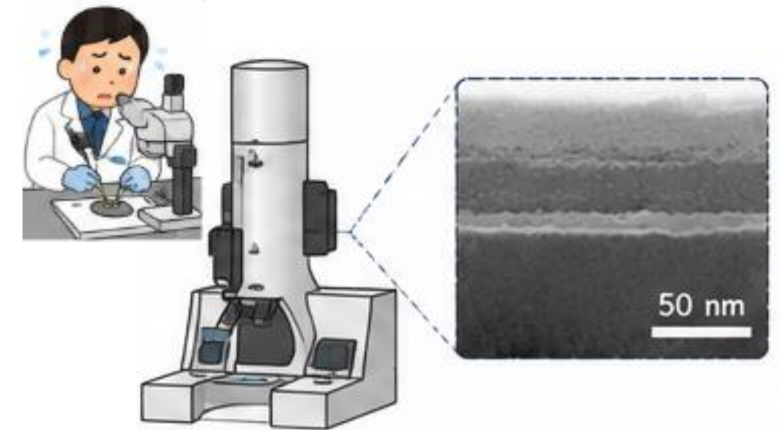
The challenge







- AFM only probes the top surface.
- SEM requires cross-section preparation.
- TEM is destructive and labor-intensive.

XRR non-destructively characterizes the entire thin-film stack over a large area.

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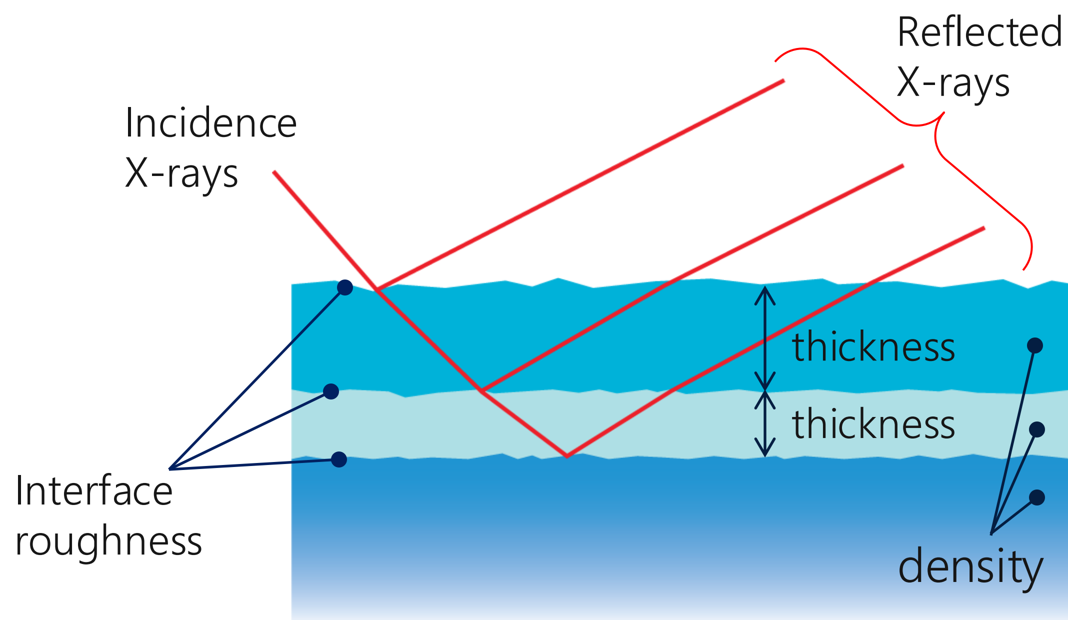
What is X-ray Reflectivity?



XRR measures how X-rays reflect from a surface as a function of incident angle.

- Thickness (0.1 ~ 1,000nm)
- Density
- Surface & interface roughness (0 ~ 3 nm)
  - $q_{z-max}\sigma_{max} \gg 1$
  - $\sigma_{max} \approx 3$ nm

S. K. Sinha, E. B. Sirota, S. Garoff, H. B. Stanley
"X-ray and neutron scattering from rough surfaces"
Phys. Rev. B 38, 2297 (1988).

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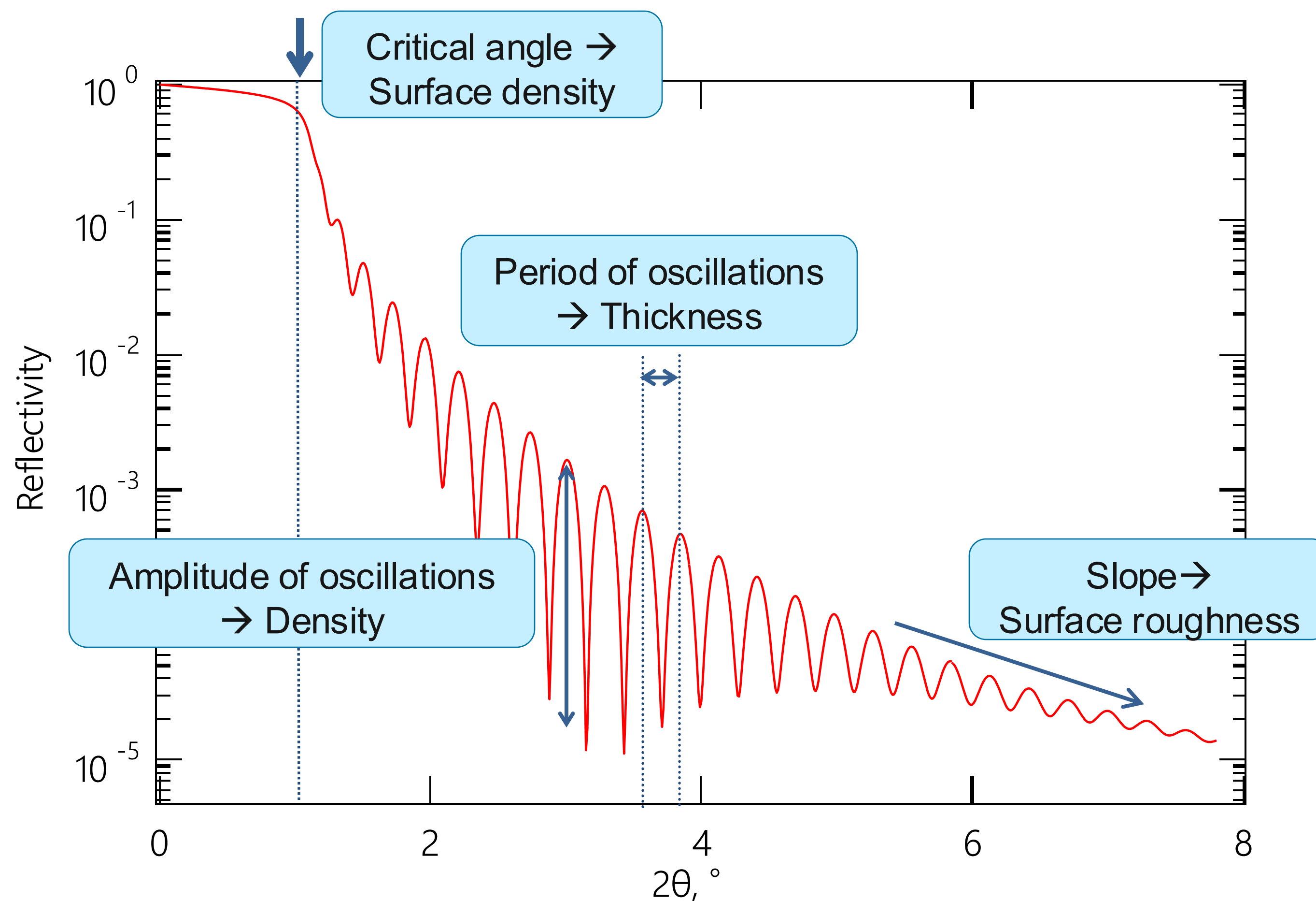
X-ray reflectivity profile

XRR
Specular reflection
$\boldsymbol{q} \perp$ surface



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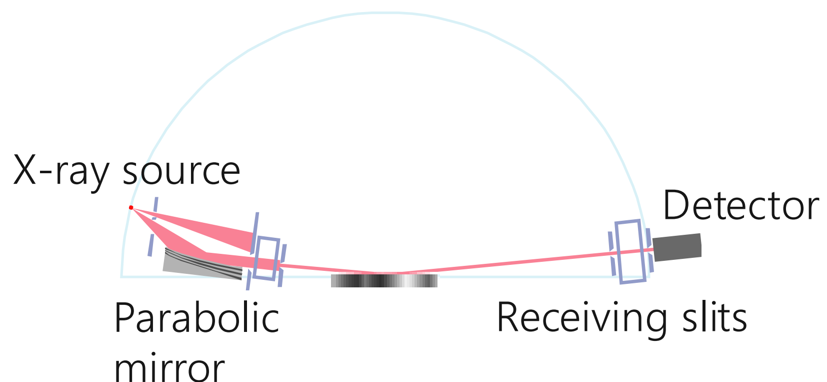
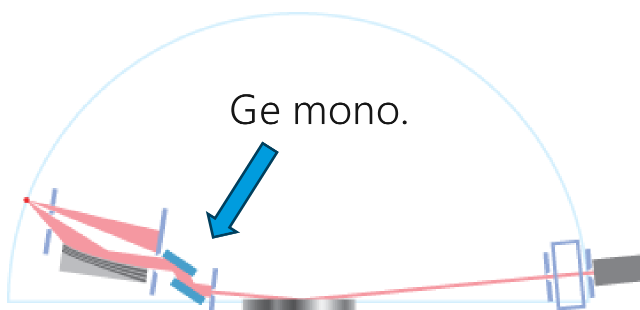
Instrument configurations



- **Parallel beam geometry (0.1 ~ 220 nm)**
  - Parabolic mirror
  - Beam divergence: $\Delta\theta$ ~ 0.02 °



- **High resolution geometry (1 ~ 440 nm)**
  - Parabolic mirror + Ge(400)x2
  - Beam divergence: $\Delta\theta$ ~ 0.01 °

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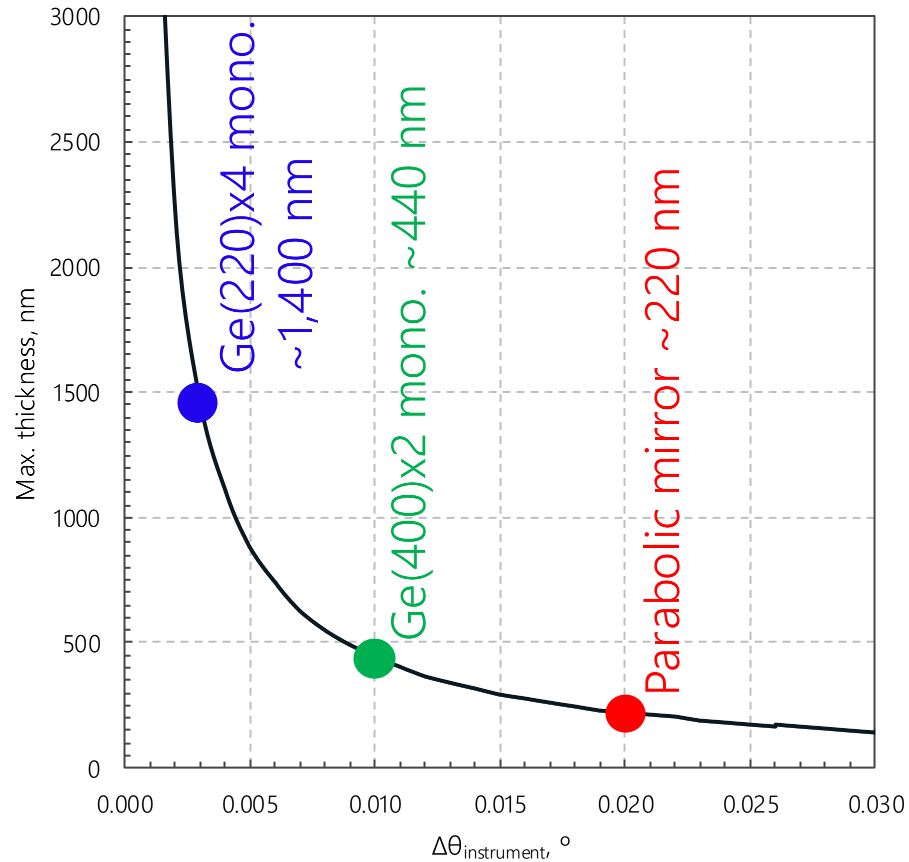
Measurable thickness range



- $\Delta\theta_{thickness} \approx \frac{\lambda}{2 \cdot t}$

  $\Delta\theta$: fringe spacing

  t: thickness of film (nm)

  λ: wavelength of X-ray 0.15406 (nm)

- Maximum thickness $t_{max.}$ measured

  $$t_{max.} \approx \frac{\lambda}{2 \cdot \Delta\theta_{instrument}}$$

  $\Delta\theta_{instrument}$: instrumental resolution

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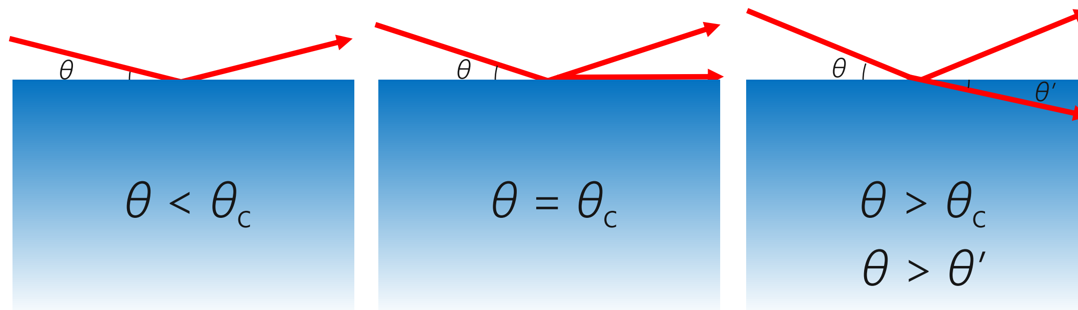
Critical angle of external total reflection



- Refractive index: $n = 1 - \delta + i\beta$
  - $\delta = \frac{\lambda^2 r_e}{2\pi V_c} \sum_j (Z_j + f_j')$ ➡ Density
  - $\beta = \frac{\lambda^2 r_e}{2\pi V_c} \sum_j (-f_j'')$ ➡ Absorption
  - $\theta_c = \sqrt{2\delta}$ ➡ ~Density

V_c = unit cell volume
Z_j = number of electrons of atom j
f_j' = real anomalous dispersion correction
f_j'' = imaginary anomalous dispersion correction
r_e = classical electron radius
θ_c = critical angle for total reflection

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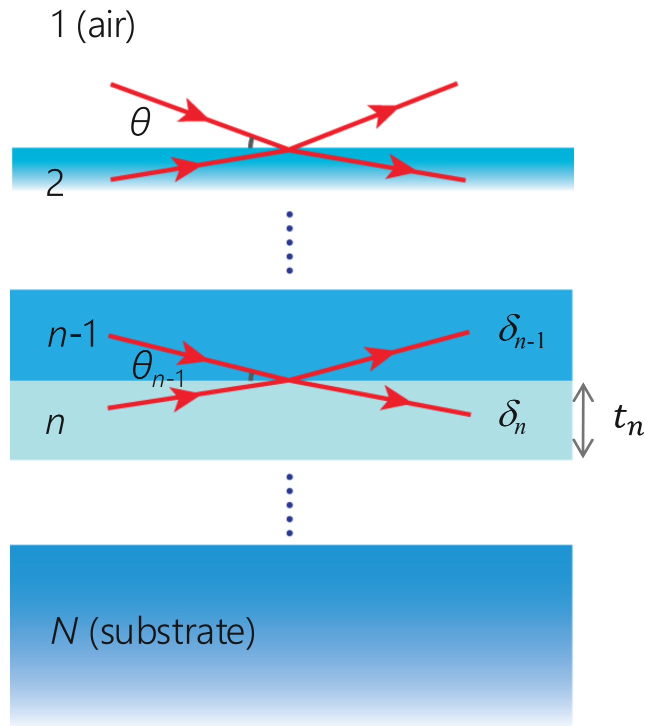
Parratt's recursive formula

L.G.Parratt, Phys.Rev.**95**(1954)359.



- Recursive equation of reflectivity

$$R_{n-1,n} = a_{n-1}^4 \left[\frac{R_{n,n+1} + F_{n-1,n}}{R_{n,n+1} \cdot F_{n-1,n} + 1}\right] \times exp\left\{-\frac{1}{2}\sigma_n^2\left(\frac{4\pi\sin\theta}{\lambda}\right)^2\right\}$$

Roughness

  - $a_n = exp\left(-ik_1 f_n \frac{t_n}{2}\right)$ Thickness
  - $F_{n-1,n} = \frac{f_{n-1} - f_n}{f_{n-1} + f_n}$
  - $f_n = \sqrt{\theta^2 - 2\delta_n - 2i\beta_n}$ Density
  - σ_n: roughness of nth interface
  - t_n: thickness of nth layer
  - θ: Bragg angle
  - λ: wavelength of X-ray

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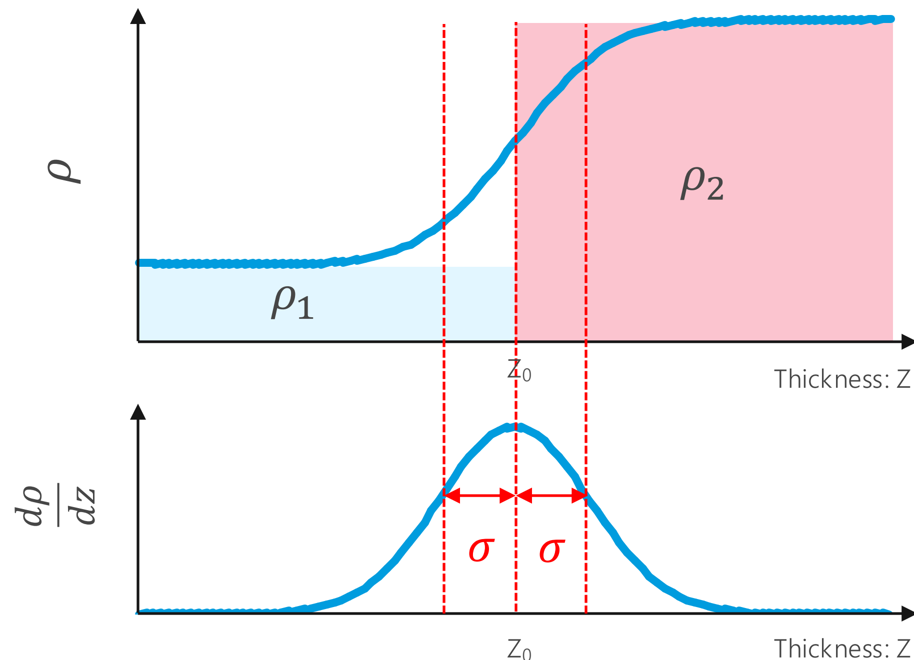
Nevot-Croce roughness model

L. Nevot & P. Croce, Rev.Phys.Appl.**15**(1980)761.



- Electron density profile across an interface is described by an error function.
  - $\rho(z) = \rho_{avg} + \frac{\Delta\rho}{2}\,\mathrm{erf}\left(\frac{z-z_0}{\sqrt{2}\sigma}\right)$ → Roughness
- Roughness parameter σ corresponds to the standard deviation of the Gaussian that is the 1st derivative of the density profile.
  - $\frac{d\rho}{dz} = \frac{\Delta\rho}{\sqrt{2\pi}\sigma}\exp\left[-\frac{(z-z_0)^2}{2\sigma^2}\right]$

Roughness

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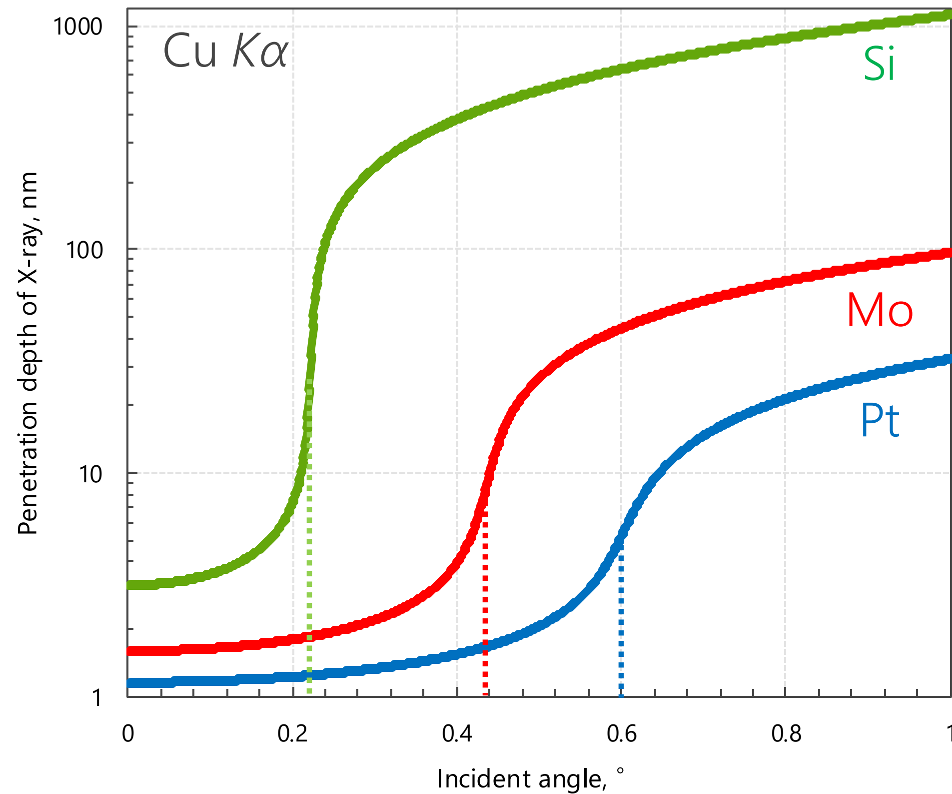
Penetration depth



- Penetration depth D around θ_c
  - $D = \dfrac{\lambda}{4\pi B}$
  - $2B^2 = (-\theta^2 + 2\delta) + [(\theta^2 - 2\delta)^2 + 4\beta^2]^{1/2}$

Density   Absorption

- θ_c
  - Si (ρ =2.33 g/cm^3): 0.22 °
  - Mo (ρ = 10.28 g/cm^3): 0.44 °
  - Pt (ρ =21.46 g/cm^3): 0.60 °

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Questions?

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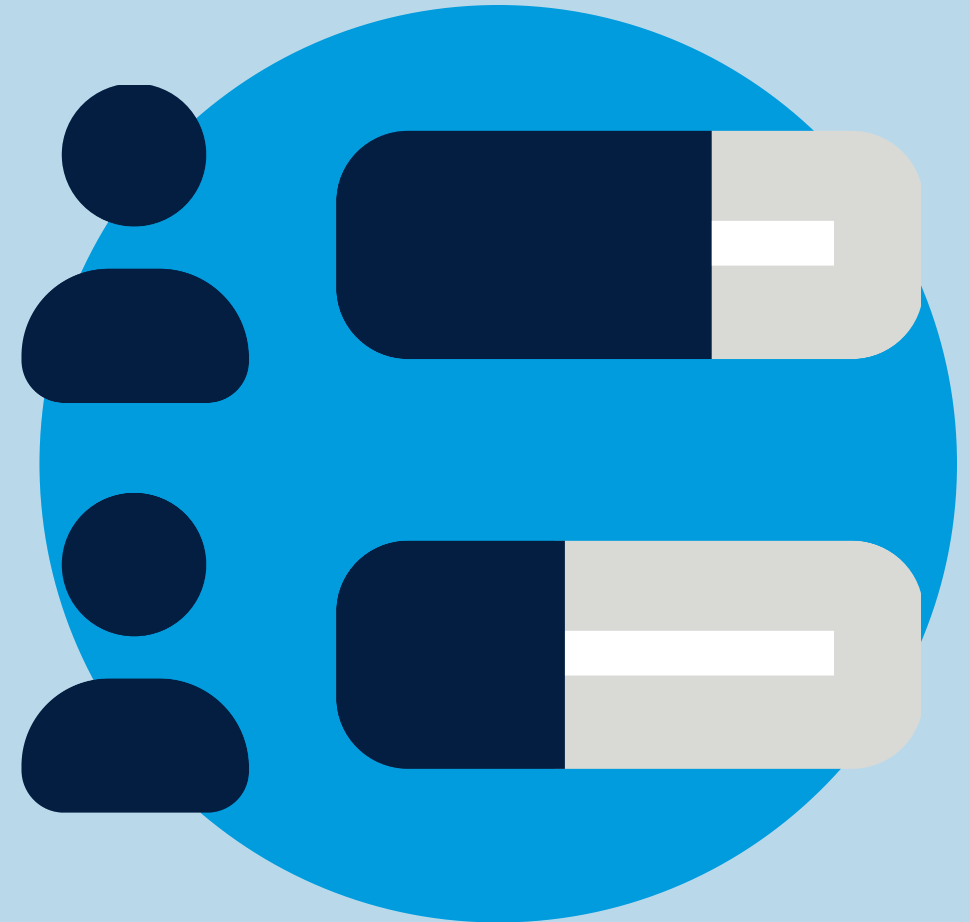
Polling Question

#3 Had you previously considered XRR modeling to be nanostructural analysis?

1. Yes
2. No



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8. XRR modeling & fitting

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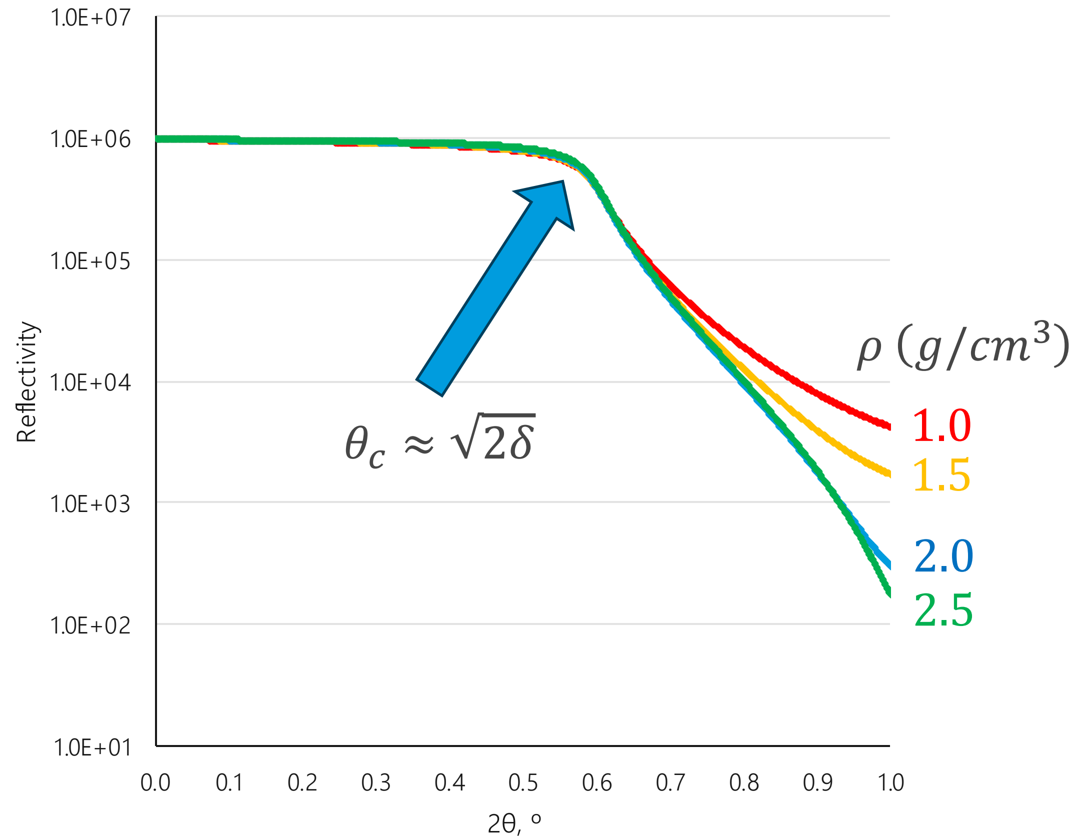
Critical angle for total reflection



- SiO_2(5nm)/GaAs sub.
- SiO_2 with different densities show the same θ_c.
- 5 nm-thick SiO_2 is too thin to show θ_c.

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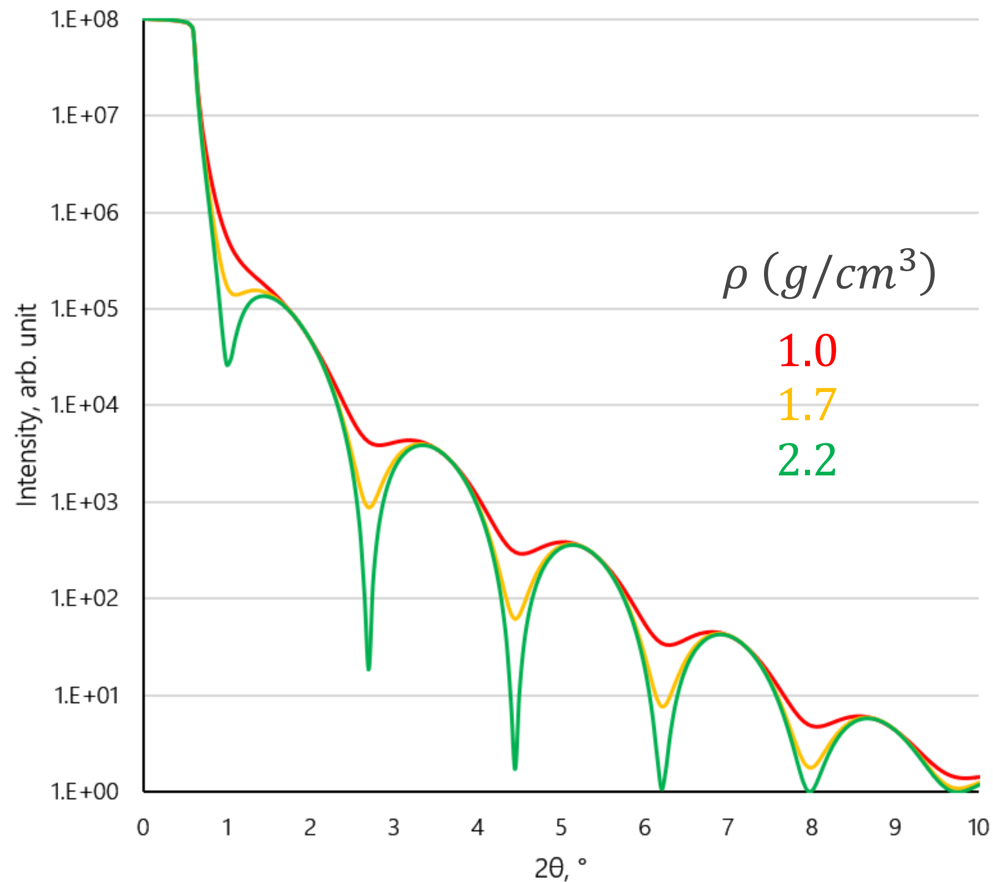
Profile vs. density of SiO_2 layer



- SiO_2(5nm)/GaAs sub.
- Roughness: 0.3 nm.
- Density of SiO_2 changes amplitude of the thickness fringes.

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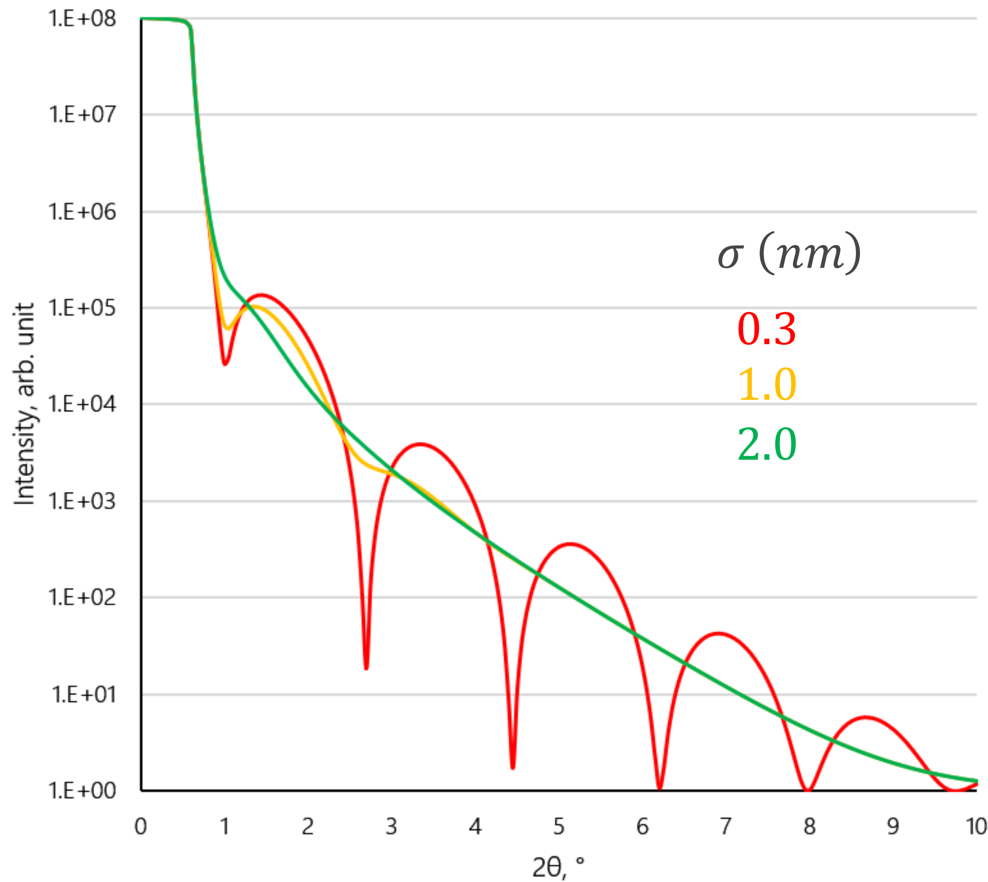
Profile vs. roughness of SiO_2 layer



- SiO_2(5nm)/GaAs sub.
- Density: 2.2 g/cm^3.
- σ changes maximum 2θ angle for fringes.

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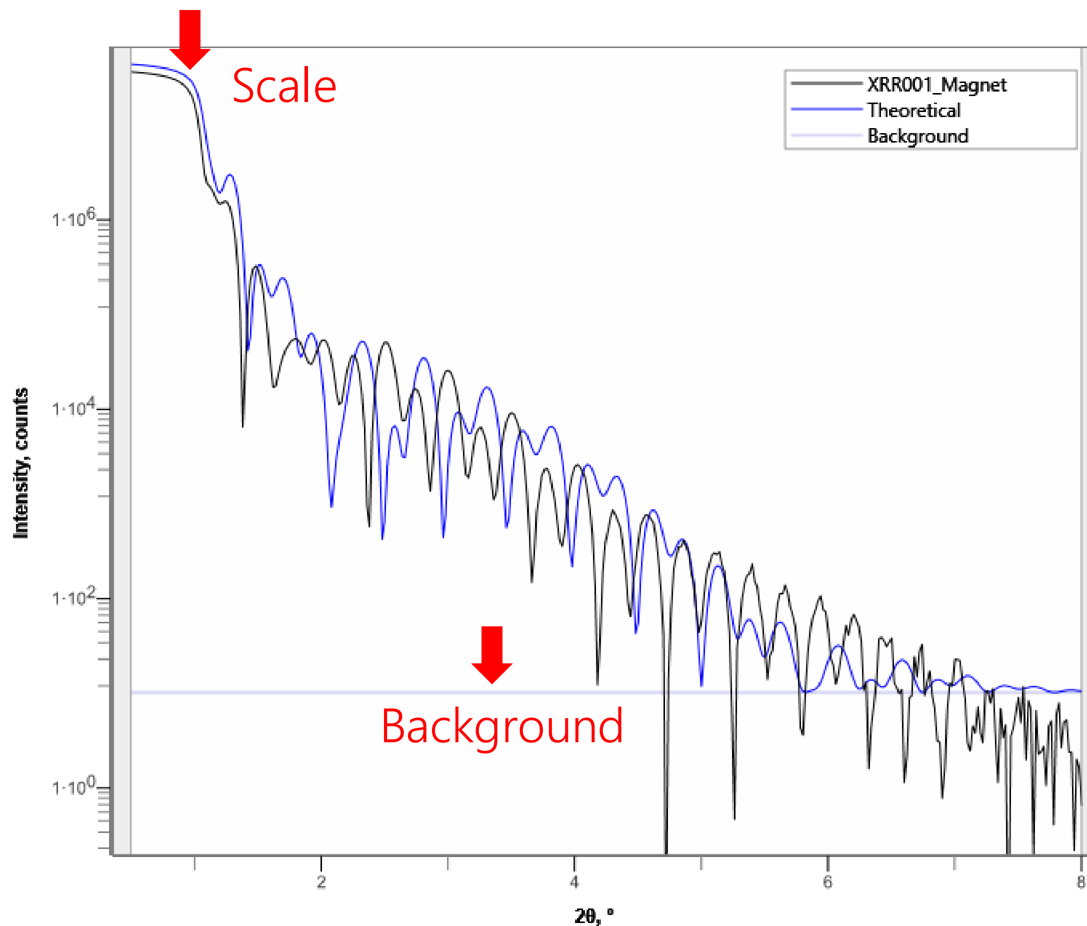
XRR profile fitting



Layer	Thickness [nm]	Density [g/cm^3]	Roughness [nm]
TaO	2.30	8.70	0.50
Ta	16.00	16.70	0.50
NiFe	16.00	8.70	0.50
Si sub.	n.a.	2.33	0.50

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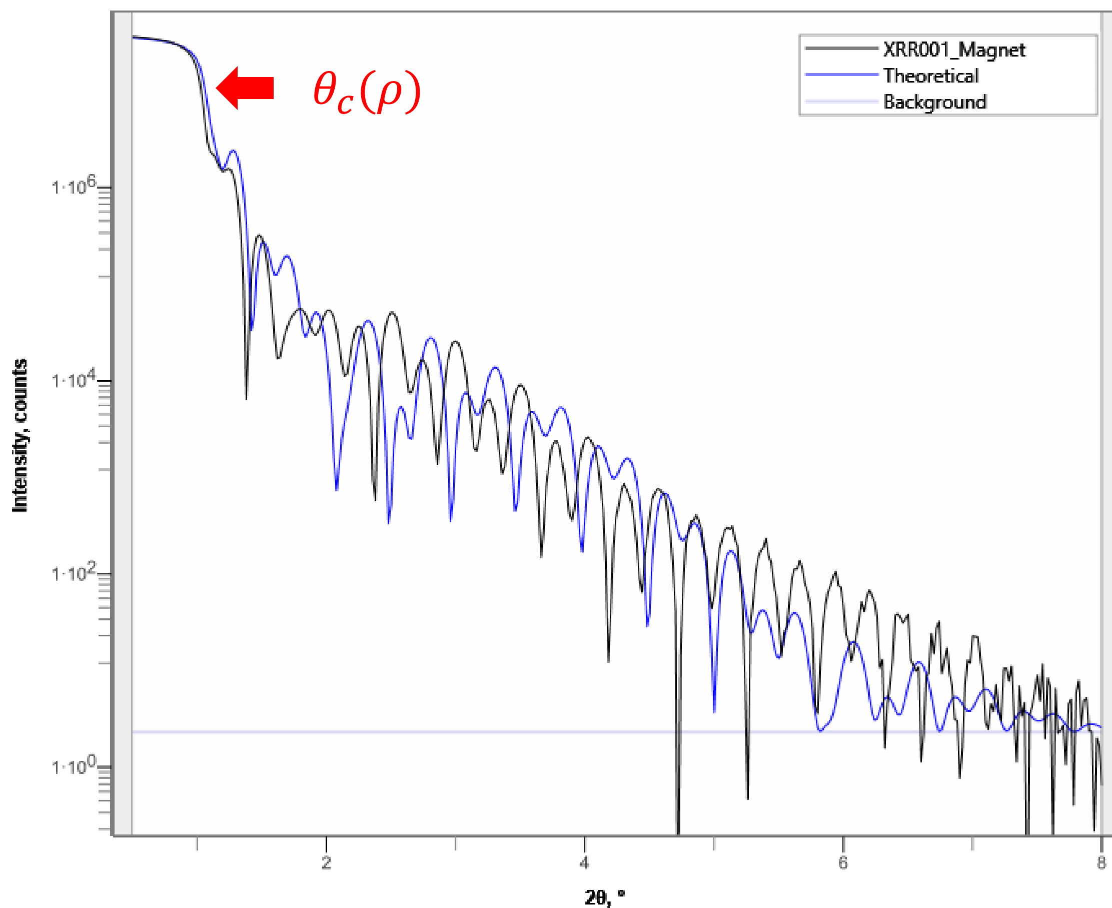
XRR profile fitting: step (1)



Layer	Thickness [nm]	Density [g/cm^3]	Roughness [nm]
TaO	2.30	8.70	0.50
Ta	16.00	16.70	0.50
NiFe	16.00	8.70	0.50
Si sub.	n.a.	2.33	0.50

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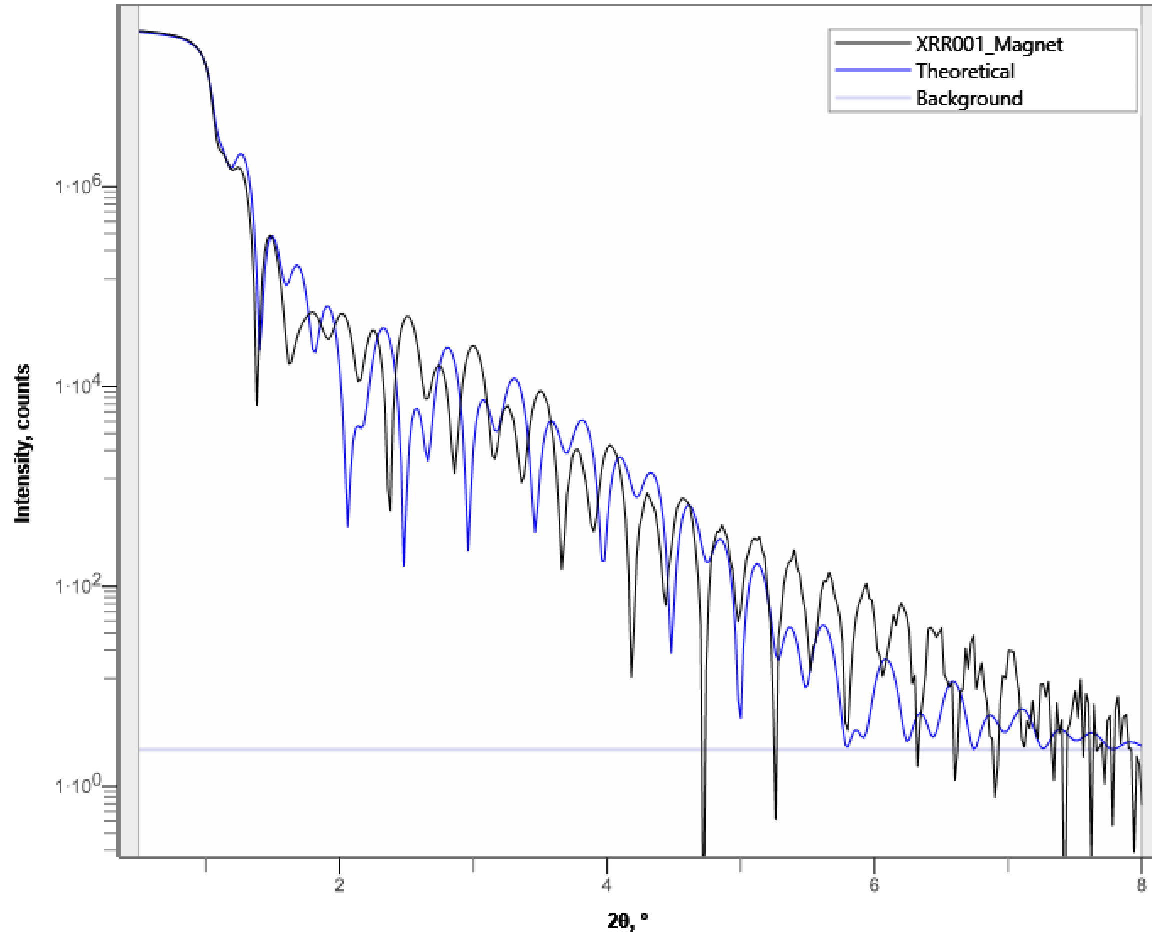
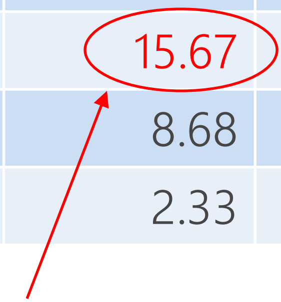
XRR profile fitting: step (2)



Layer	Thickness [nm]	Density [g/cm³]	Roughness [nm]
TaO	2.30	8.70	0.50
Ta	16.00	15.67	0.50
NiFe	16.00	8.68	0.50
Si sub.	n.a.	2.33	0.50



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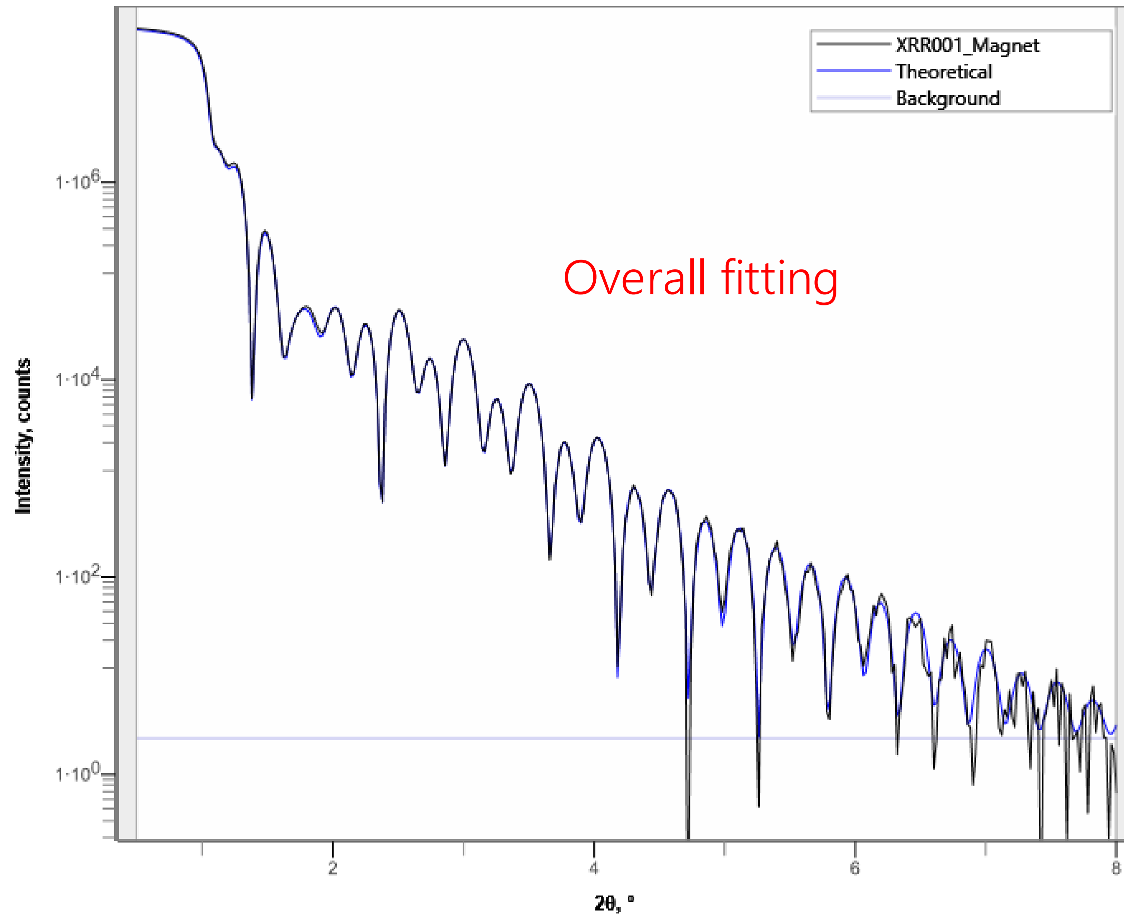
XRR profile fitting: step (3)



Layer	Thickness [nm]	Density [g/cm³]	Roughness [nm]
TaO	2.80	8.11	0.72
Ta	17.10	15.67	0.38
NiFe	15.30	8.61	0.56
Si sub.	n.a.	2.33	0.42

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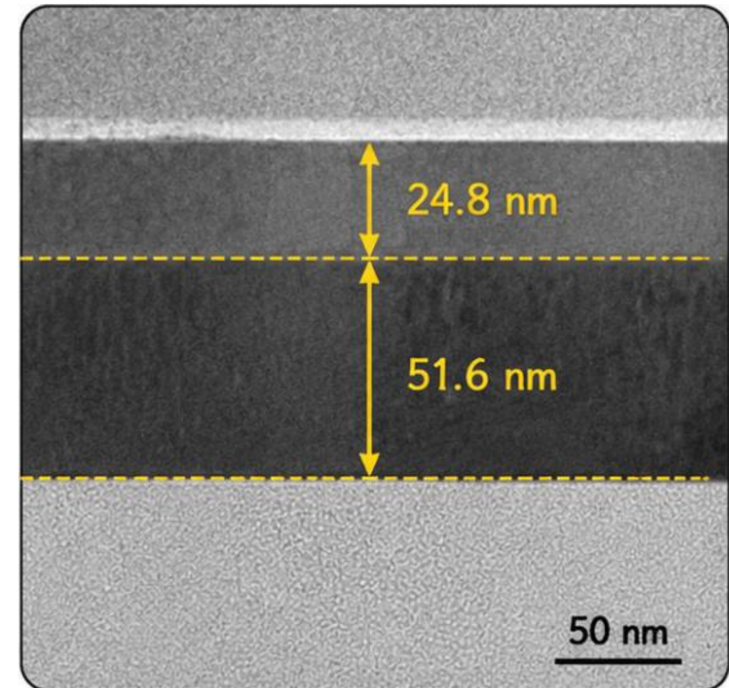
When the fit doesn’t converge (1)

- Try TEM to get an idea about thickness(es).



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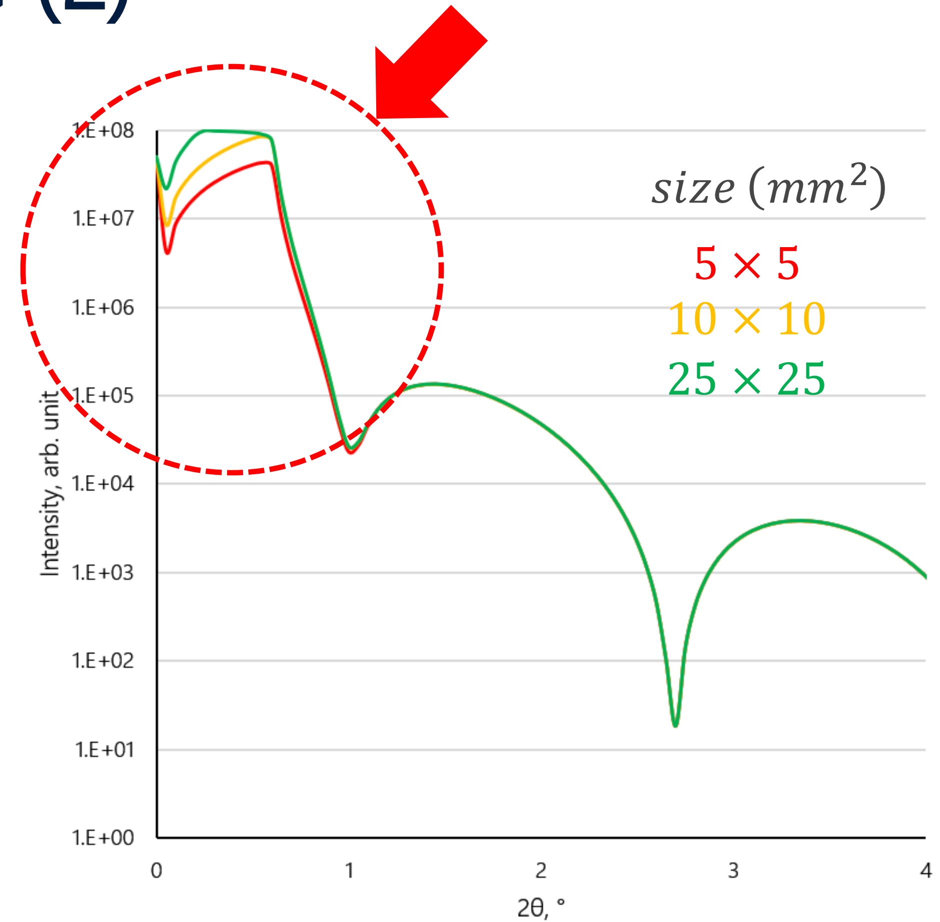
When the fit doesn't converge (2)

- Prepare a sample with surface area larger than 10x10 mm^2.



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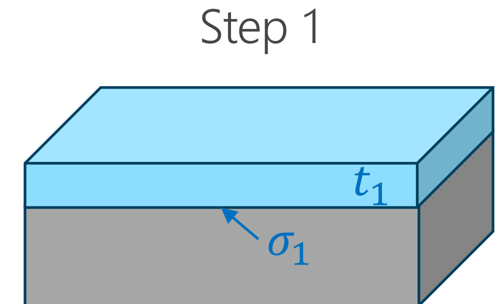
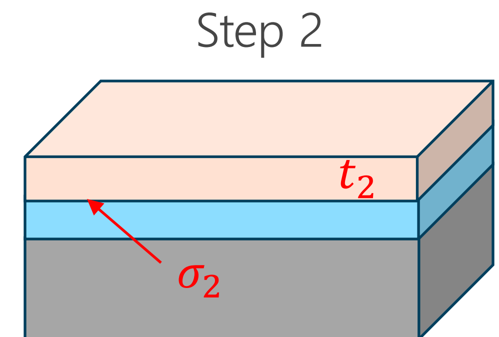
When the fit doesn't converge (3)

- Characterize individual layers before building the full stack

Step 1



Step 2



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Summary

- XRR is a non-destructive technique for characterizing thin films and interfaces, providing thickness, density, and surface/interface roughness with nanometer-scale sensitivity.
- Instrument configuration determines the measurable thickness range, extending from a few nanometers to over one micrometer with high-resolution optics.

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Questions?

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We'll follow up with your questions.

Recording will be available tomorrow.

More new content is coming soon.

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Capillary

- Fused silica (quartz)
  - Low background
  - Excellent uniformity
  - Low parasitic scattering
- Borosilicate
  - B, Na, Al, K
  - Diffuse scattering
  - Higher background
  - Parasitic scattering

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Distance to nearest neighbor: $\boldsymbol{d_{nn}}$

- Average center-to-center distance between neighboring particles
  - $\therefore d_{nn} \approx \left(\frac{\pi\sigma^3}{6\eta}\right)^{\frac{1}{3}}$
  - d_{nn} is a function of σ and η.

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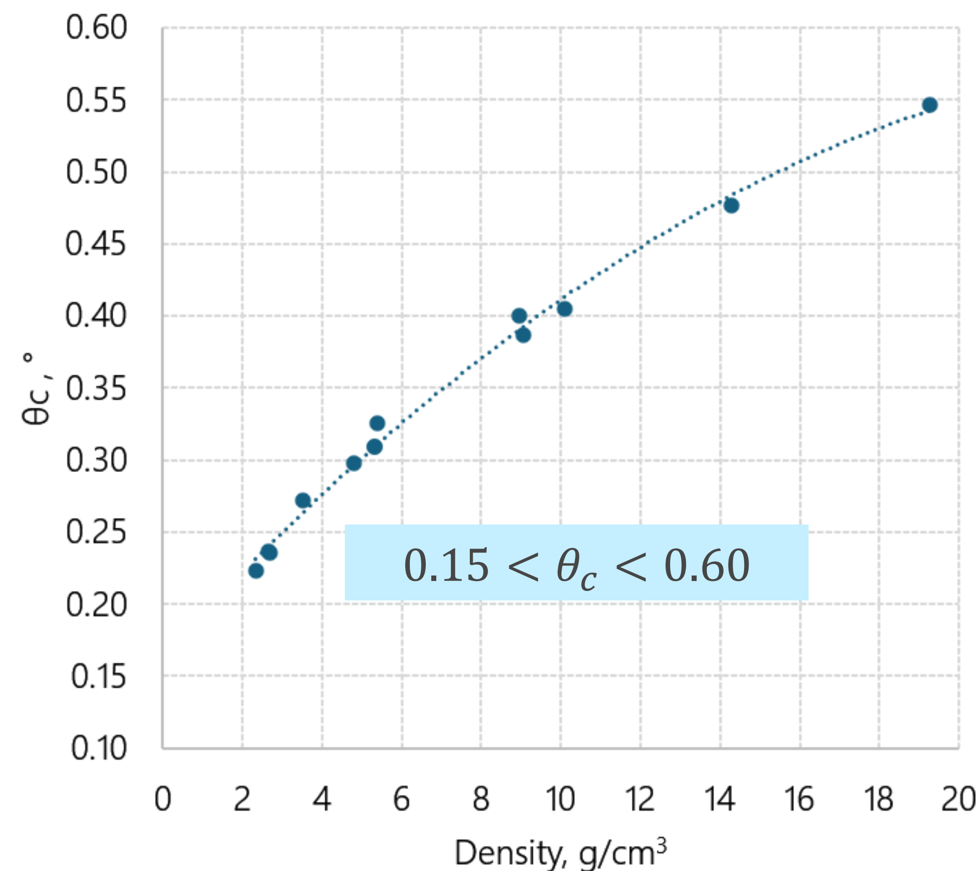
Example of δ, β and θ_c for Cu-Kα

	ρ [g/cm^3]	δ [10^{-6}]	β [10^{-7}]	θ_c [°]
Si	2.33	7.57	1.75	0.223
GaAs	5.32	14.5	4.20	0.309
InP	4.79	13.5	1.15	0.298
Ge	5.33	14.5	4.17	0.309
C	3.52	11.3	16.9	0.272
SiO_2	2.65	8.57	1.11	0.237
HfO_2	10.11	25.05	15.51	0.405
Ta_2O_5	9.07	22.8	14.1	0.387
TiN	5.39	16.2	10.2	0.326
TaN	14.3	34.6	24.9	0.477
Cu	8.94	24.3	5.31	0.400
W	19.26	45.6	37.4	0.547
Al	2.70	8.47	1.58	0.236



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