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Small Angle X-ray Scattering (SAXS)

Synchrotron Radiation Course

Project work

Introduction

Small angle x-ray scattering (SAXS) is a technique for studying features with sizes in the order of 1 nm to several hundreds of nm. The scattered waves can give information about sizes, ordering and to some extent shape of the scattering objects which can be for example particles or pores. The measurement is usually set up in transmission, where a 2-D detector is placed after the sample. Since the central beam needs to be blocked not to damage the detector, scattering in very small angles can be difficult to detect. This gives a limit for the largest features that can be studied. For ordinary lab x-ray tubes, there will also be a problem with divergence of the beam which is over come in synchrotrons where the beam can be focused into a small spot.

Scattering

The incident wave on a sample and the scattered wave can be described with their respective wave vectors \mathbf{k}_0 and \mathbf{k} . For elastic scattering the wave vectors both have the magnitude $2\pi/\lambda$. The difference between the two are usually referred to as the scattering vector and is defined by $\mathbf{k} - \mathbf{k}_0 = \mathbf{q}$.

The amplitude of the scattered wave depends on the electron density distribution $\rho(\mathbf{r})$ in the sample. The amplitude of the scattered wave from a small volume dV in the sample can then be written as

$$f(\mathbf{q}) = \int \int \int dV \cdot \rho(\mathbf{r})e^{-i\mathbf{q}\cdot\mathbf{r}} \quad (1)$$

For a particle imbedded in a matrix the scattering amplitude will depend on the difference in electron density between the particle and the matrix, $\Delta\rho = \rho_1 - \rho_2$ where ρ_1 and ρ_2 is the electron density of the particle and matrix respectively. The difference in density between two points separated by \mathbf{r} can be described by the correlation function $\gamma(\mathbf{r})$. We can then write the scattered intensity as

$$I(q) = V \int_0^\infty 4\pi r^2 \gamma(r) \frac{\sin qr}{qr} dr \quad (2)$$

where V is the scattering volume.

Porod limit

For small distances r compared to the size of the scattering regions D in the sample the scattering will reflect interfaces between the particles and the

matrix. At large scattering vectors q , i.e. large angles, the scattered intensity will be proportional to the surface area (S) per unit volume (V), $A = S/V$. For an infinitely sharp interface Porod's law

$$A = \frac{1}{2\pi(\Delta\rho)^2V}K \quad (3)$$

is valid where $K \propto q^4 \cdot I(q)$ is the so called Porod limit. The law is valid for all sharp interfaces and for $r \gg r_0$ where r_0 is the interface width. In principle the width of the interface can be obtained by observing at which point the law fails but the scattering at these large angles are often too weak wherefore good data can be difficult to achieve. The Porod limit is independent on the geometry of the scattering particles and will not give any structural information.

Guinier's law

Isolated particles, that is no interaction between them, can be described using Guinier's law. For small scattering vectors the scattered intensity can be described by

$$I(q) = I_0 \exp - \frac{q^2 R_G^2}{3} \quad (4)$$

where R_G is the radius of gyration of the scattering particle and I_0 is the scattered intensity not related to the shape of the particle. If the shape of the particles are known, their sizes can be determined from the radius of gyration. For example for a spherical particle the radius of the particle is

$$R = \sqrt{\frac{5}{3}} R_G \quad (5)$$

Guinier's law is only valid for small angles, i.e. $qR_G \ll 1$. If the particles in the sample have different sizes the sizes of the smaller particles will be difficult to determine since the scattering will be dominated by the larger particles.

Simultaneously study of the Guinier and Porod regions

For samples containing more than one order of characteristic size of scatterers, Guinier regions can be hidden between two Porod regimes. By assigning one Guinier regime and one Porod regime to one characteristic size of parti-

cles this can be described as one structural level. Using the unified function

$$I(q) = G \exp\left(\frac{q^2 R_G^2}{3}\right) + B \left(\operatorname{erf}\frac{(q R_G / 6^{1/2})^3}{q}\right)^P \quad (6)$$

where $G = n^2 N_p I_e$ and $B = 2\pi N_p \rho^2 S_p I_e$, N_p is the number of particles in the scattering volume, I_e the scattering factor for a single electron, n the number of electrons in a particle [4].

Contrast variation by anomalous scattering

To enhance the contrast of scatterers in the sample anomalous scattering of x-rays can be used. For x-rays with energies close to the absorption edge of the element the atomic scattering is reduced compared to the scattering far from the absorption edge. Since each element has different absorption edges the contribution of a single element of the scattering can be effected.

Simultaneously WAXS study

By recording the WAXS pattern, i.e. the ordinary x-ray diffraction pattern, simultaneously as the SAXS data the atomic structure of the sample can be determined. For a sample with crystalline regions the diffraction pattern can then be used to determine the crystalline phases of the scattering regions.

References

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