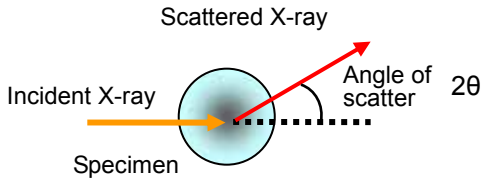
Structure Analysis Using Small-Angle X-ray Scattering (SAXS)

Overview



Overview of small-angle scattering

Bragg formula

$2d \sin\theta = n\lambda$
(d: Lattice constant; θ: Angle of scatter; n: Integer; λ: Wavelength)

In ordinary large-angle X-ray diffraction, diffraction is measured between 10° and 90°. The Bragg formula can be used to derive information concerning the molecular structure and atomic arrangement from this.

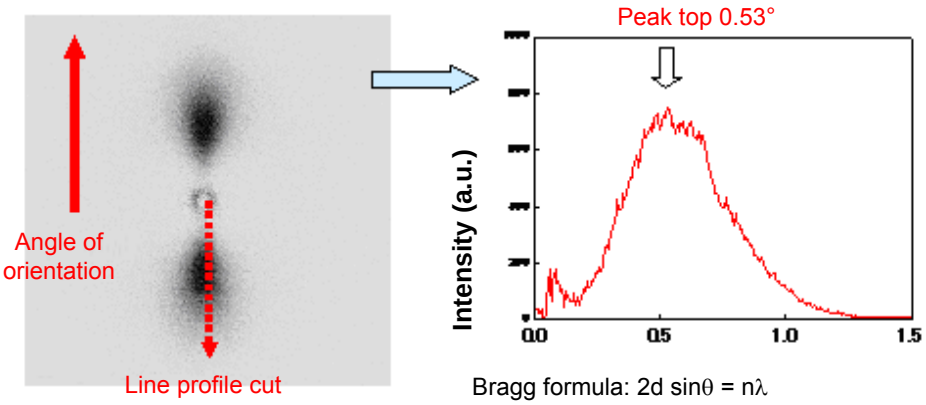
In Small-Angle X-ray Scattering (SAXS) measurements, scattering is measured in the region in which the angle of scattering (2θ) is 5° or less. This provides measurements that would not be possible with ordinary X-ray diffraction, and allows the evaluation of structures in the nm to tens of nm scale.

Features

1. Allows evaluation of structures in the 5 nm – 50 nm scale.
2. Allows rapid, non-destructive evaluation. (No staining or thin film pre-treatment required.)
3. Can be applied to a wide range of materials, including higher order polymer structures, rubbers, micelles, liquid crystals and foodstuffs. Can be used with film or powder etc, regardless of form. Measurements do not require a vacuum.

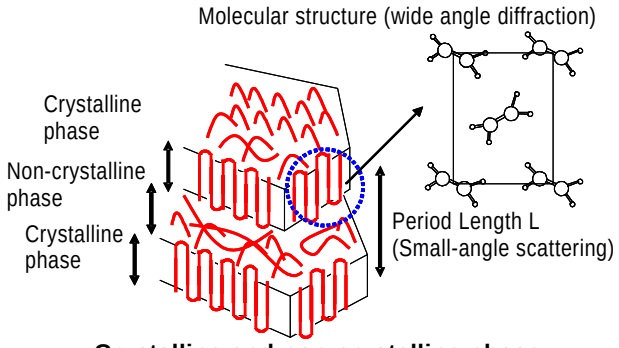
Example of analysis I: Analysis of higher order structure of all-purpose polymer material

This shows the results of SAXS measurement of a monoaxially oriented polypropylene film. A peak due to the periodic structure was found in the direction in which the sample was oriented only. This shows that the molecules are arranged in a specific direction and distributed in periodic structure size (repeated crystalline and non-crystalline phases).



SAXS Pattern of polypropylene

Bragg formula: $2d \sin\theta = n\lambda$
(d: Period length (nm); λ: X-ray wavelength)
From this, it is possible to calculate the period length.
Period length d = 17 nm



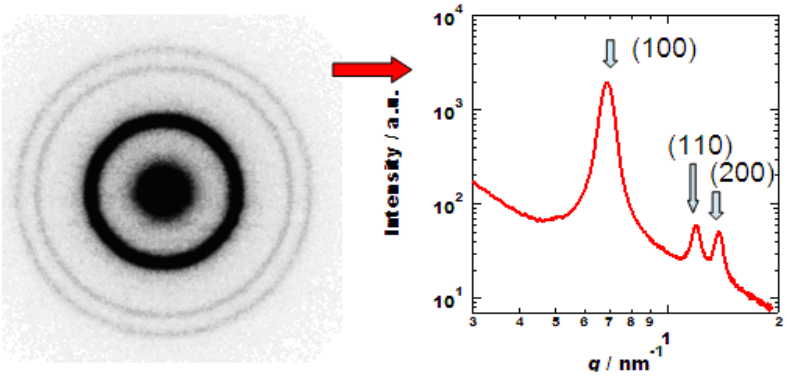
Crystalline and non-crystalline phase layer structure

Ordinary wide angle diffraction provides molecular structure information such as polymer packing etc. Small angle scattering allows observation of larger-scale structures such as the arrangement of the crystals and size (period length) of repetitions of the non-crystalline phase.

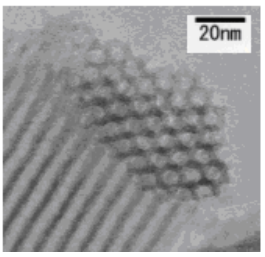
This makes it possible to establish discrete micro-phase structures in block polymers, the arrangement of polymers and the morphology and arrangement of discrete phase structures.

Example of analysis II: Analysis of structure of nano-scale porous material

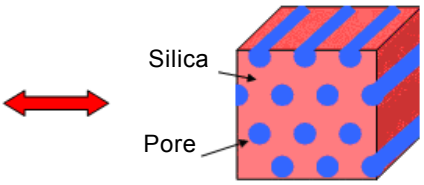
Mesoporous silica is a porous silica having uniform mesopores and a regular structure. It is used in catalyst carriers, separators and adsorbents. It is also being considered for application as a low induction inter-layer insulating film (low-k) making use of its porosity.



SAXS pattern and one-dimensional profile of mesoporous silica



Observed under TEM



Structural model inferred from SAXS
Two-dimensional cylindrical structure; pore diameter = 9.2 nm

Peaks corresponding to the pores appeared in the SAXS profile, which could be used to measure the size of the pores. Analysis of the position of the high order peaks confirmed the two-dimensional cylindrical structure. When the same material was observed under a TEM, it was found that the structural model derived from SAXS analysis was correct, and that the pores formed hexagonal cylindrical structures. This demonstrates that it is possible to derive information on high order structures such as size and orientation in such nano-scale materials. (Samples provided by Prof. H. Yajima, Tokyo University of Science)

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