

Small Angle X-ray Scattering

SAXS & WAXS

Peak positions

d values > Phase ID

Lattice parameter

d shift >

Residual stress

Solid solution

Intensity vs. Orientation

Preferred orientation

Fiber structure

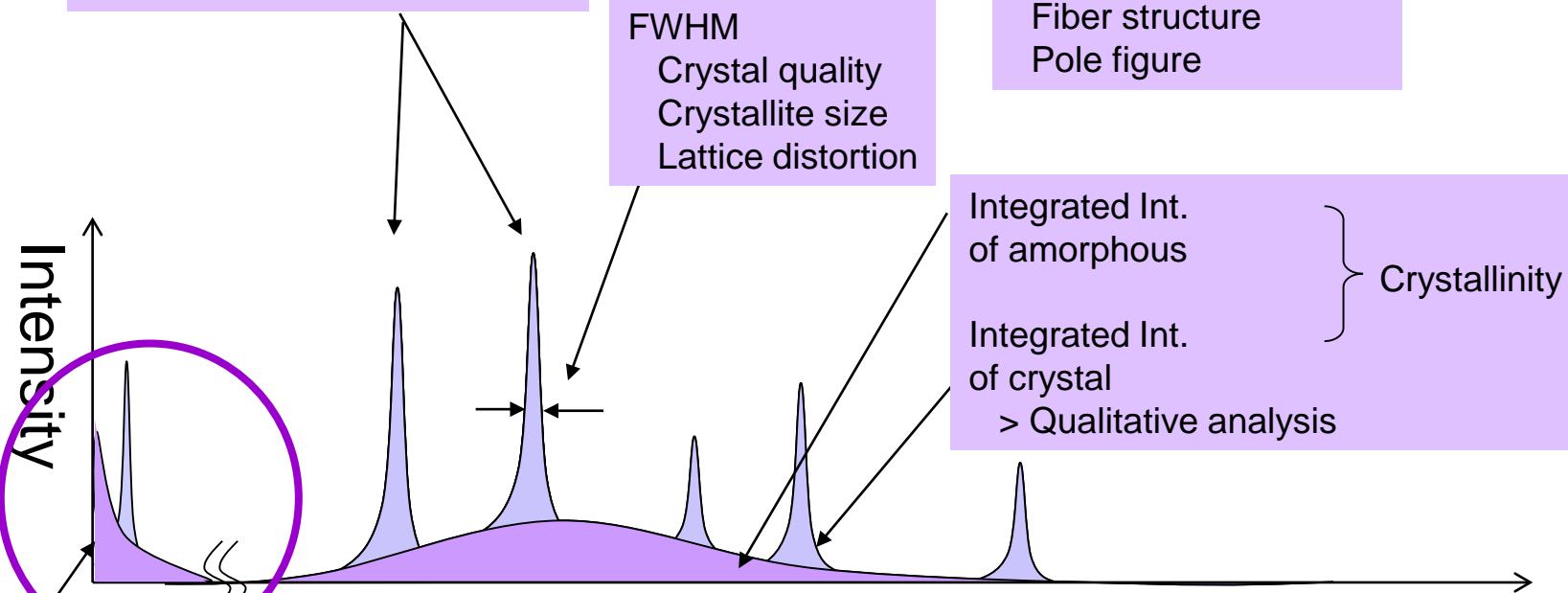
Pole figure

FWHM

Crystal quality

Crystallite size

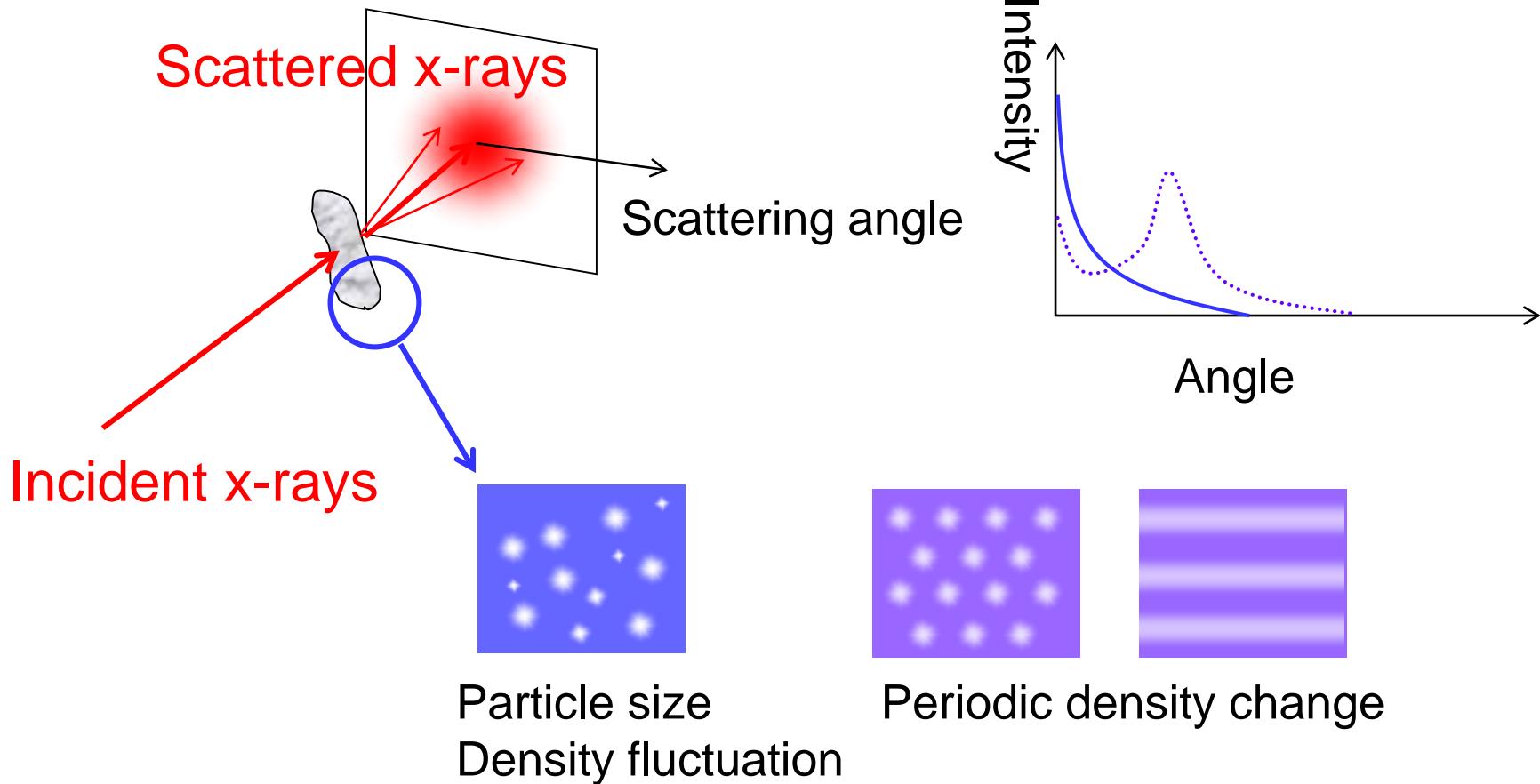
Lattice distortion



Primary beam divergence
Particle/pore size
Density fluctuation

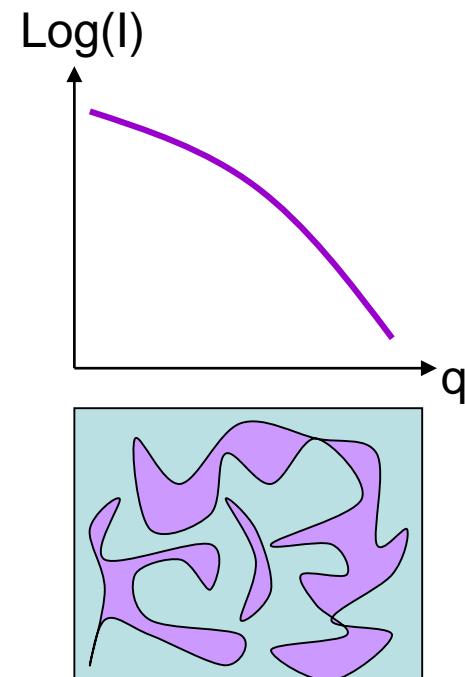
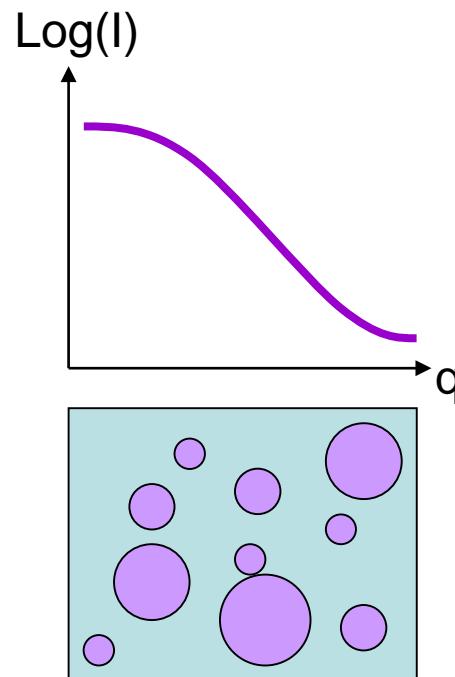
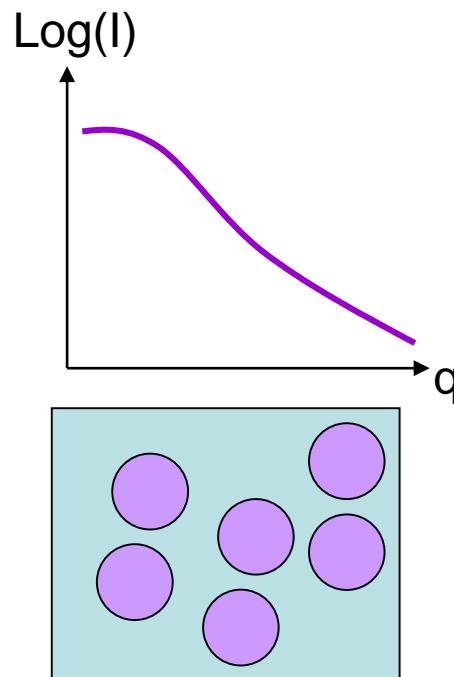
Scattering Angle [deg.]

Small Angle X-ray Scattering



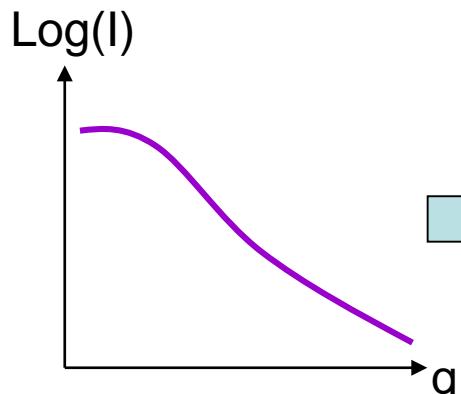
SAXS Profiles

- The distribution of primary beam reflects the fluctuation of electron density.
- But, they all look alike...

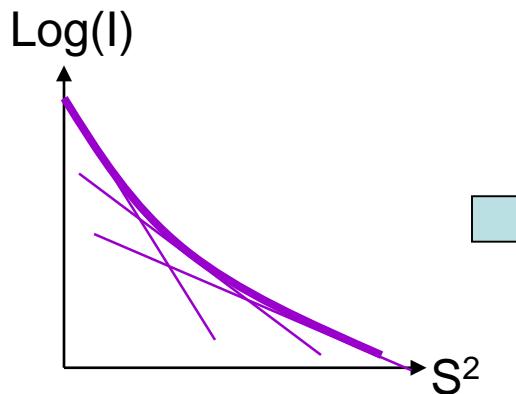


Conventional Approach

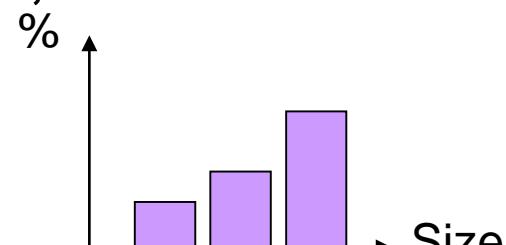
- 1. Simplify the observed profile**
- 2. Extract the structure information**
 - Guinier plot, Fourier transform, etc.



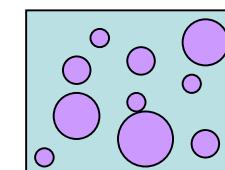
BG eliminated
Smoothed



Linear approximation
(Or FFT)



Analysis result



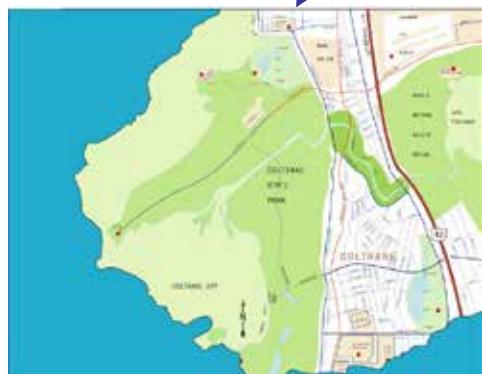
Structure model?

Details are lost in data processing



Simplified Profile

Phase information is lost and instrument function is convoluted during the observation. The raw data is processed for simplification and the details are lost.



Operator A's answer

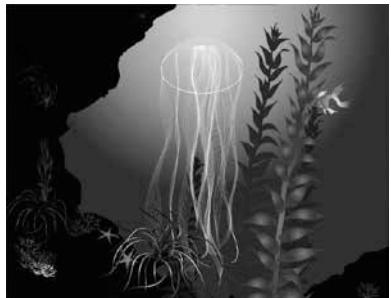
Operator B's answer

Operator C's answer

Data process to simplify the profile



True structure



Phase information is lost.
Instrument function is convoluted.



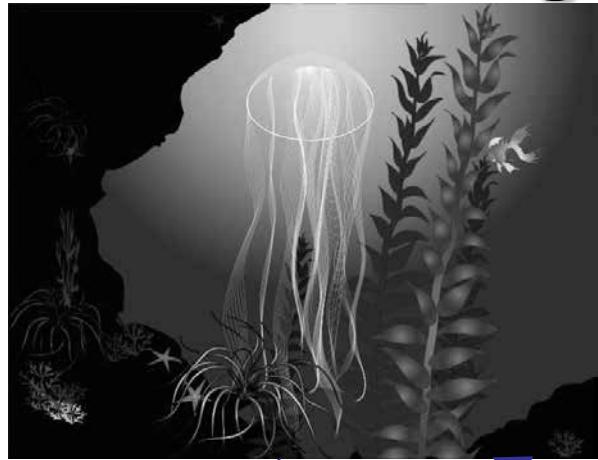
Smoothing



Linear approximation

New Approach

- Profile fitting with total modeling -

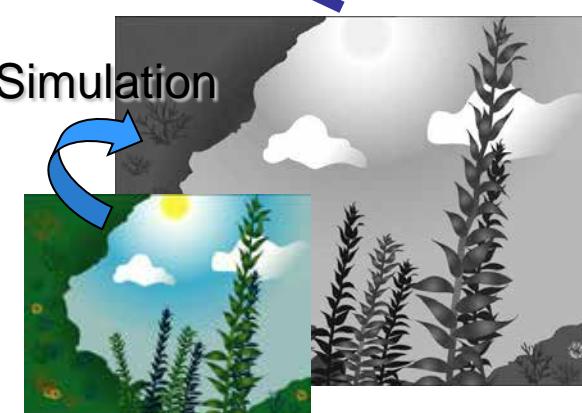


Raw Data Profile

Observed profile is reconstructed as a convolution of the scattering profile based on the model and the instrument function based on the measurement conditions.



Model A



Model B



Model C

SAXS profile simulation

- **Intrinsic scattering intensity**

Scattering Intensity → Fourier Transform [$D(r)$]

- **Slit correction**

- **Transmission geometry**

- BG elimination

- **Reflection geometry**

- Refraction, total reflection correction

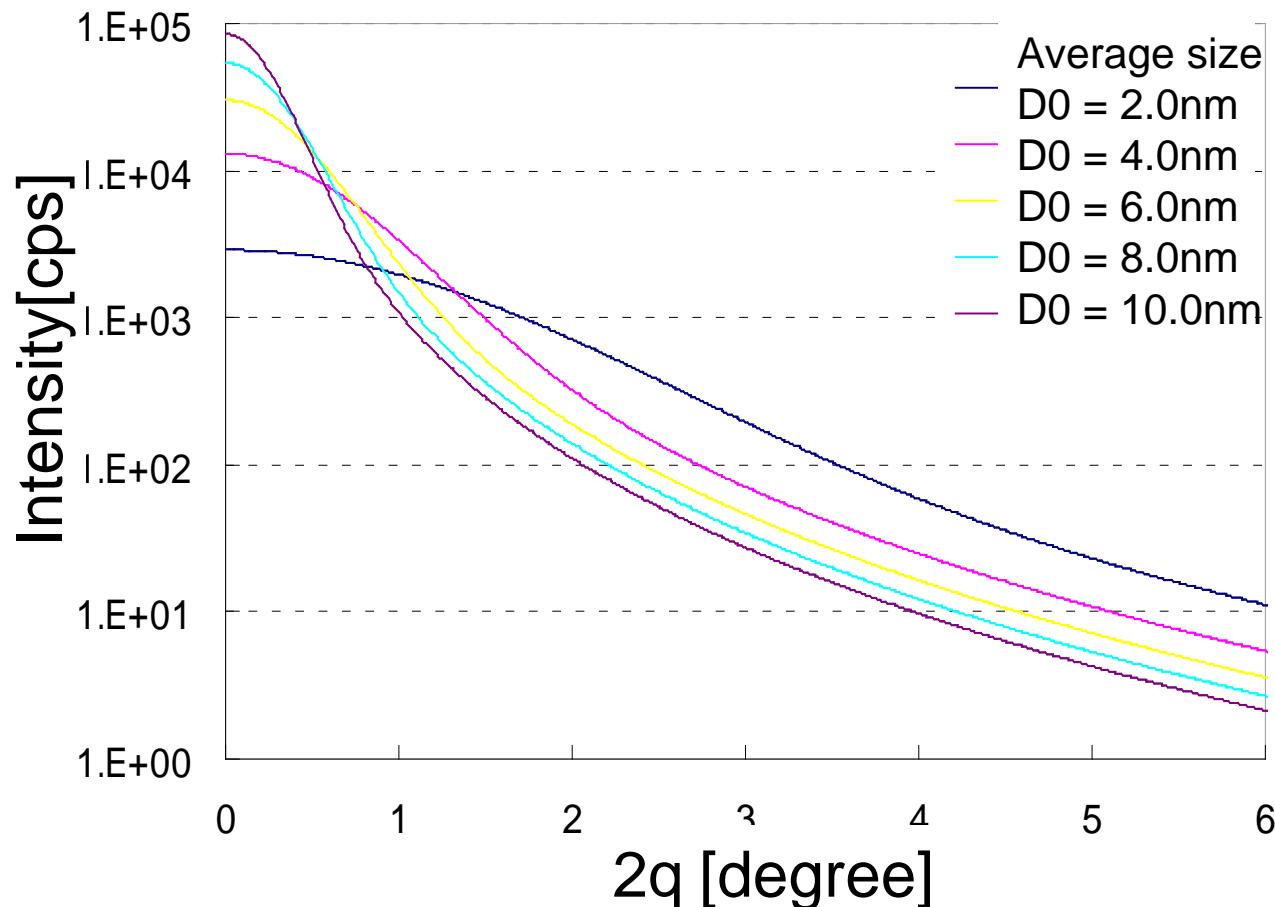
Modeling technique

- **Particle/pore size distribution**
 - >> Particle/pore size distribution function
- **Correlation length**
 - >> Correlation length distribution function
- **Model free**
 - >> Fourier transform
 - >> Radius distribution

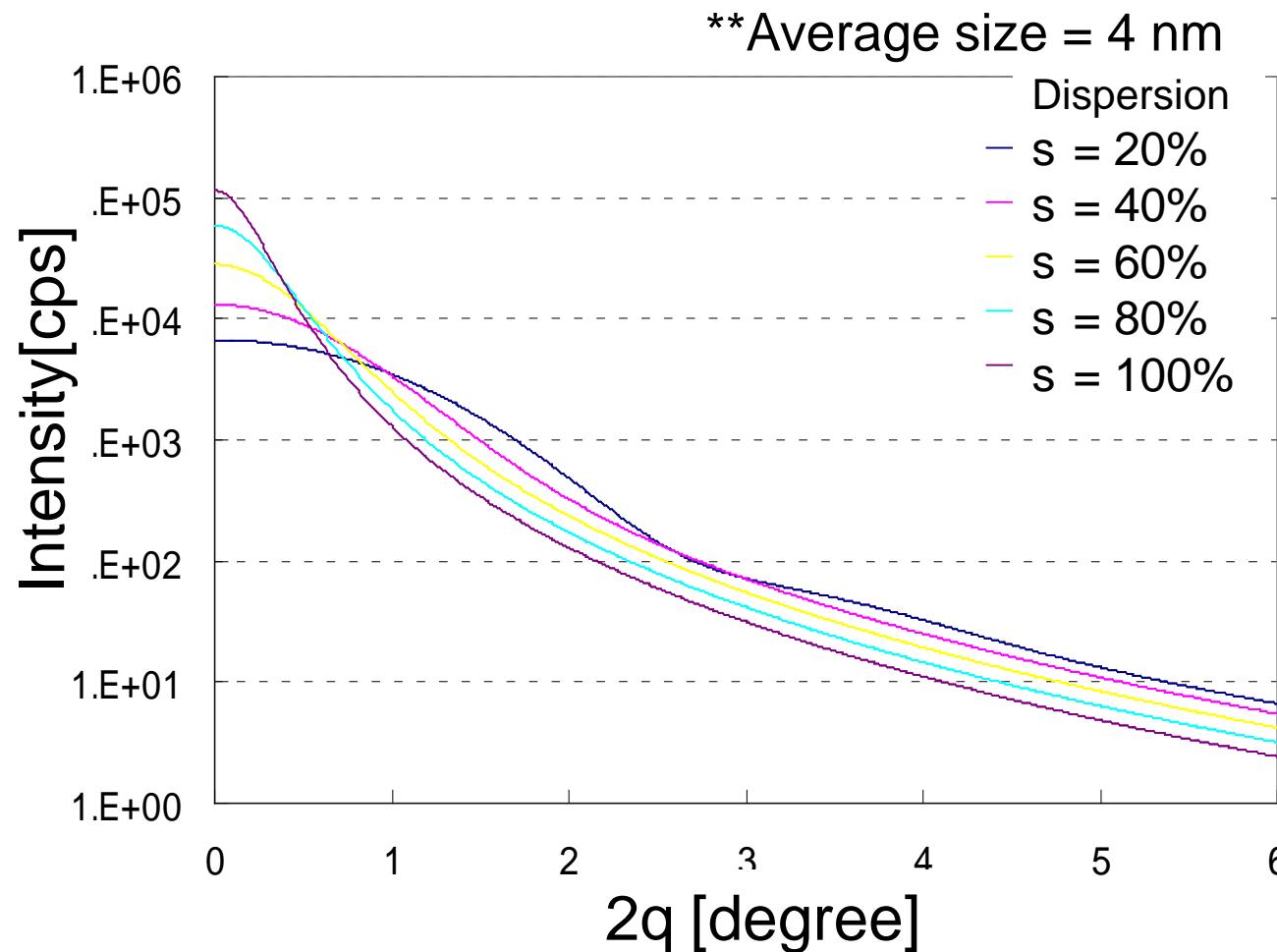


SAXS profiles (particle size)

**s (dispersion) = 40 %



SAXS profiles (distribution width)



Does it really work?

- **Whatever the technique,
SAXS profiles all look alike.**
- **The technique needs to be verified
by comparing it with other techniques.**

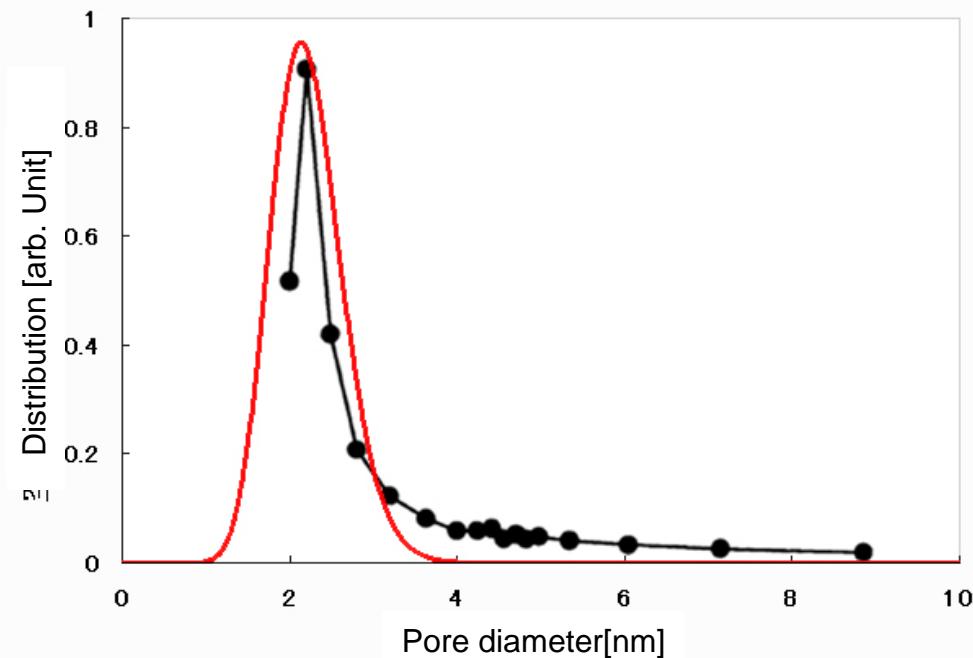
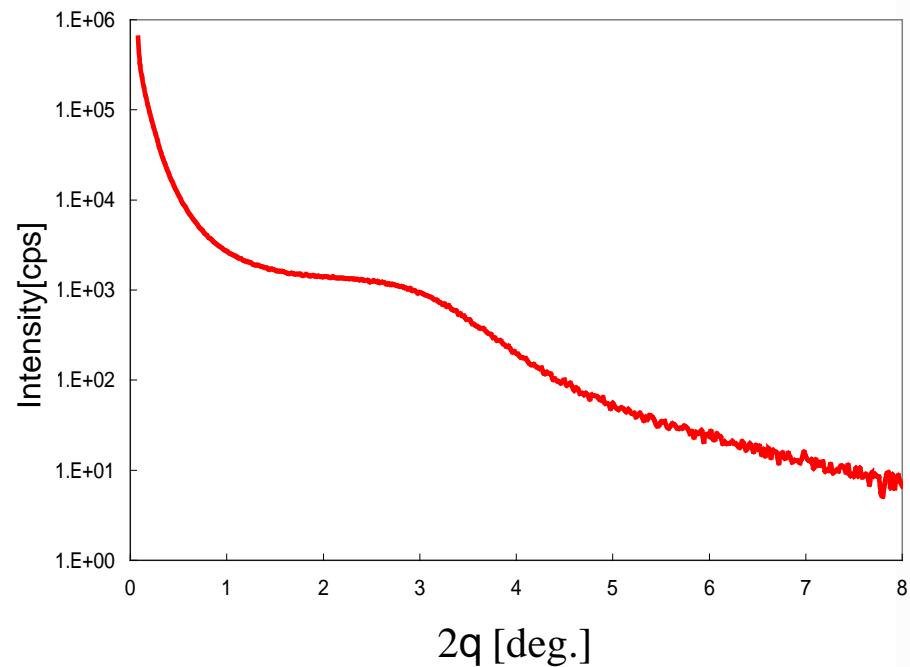
Size analysis techniques



Technique	Pros	Cons	Size
SAXS	Non-destructive Quick and easy measurement Estimates density of thin film	Cannot distinguish between particles and pores	0.5~100 nm
Gas adsorption	Easy measurement Reasonable-priced hardware	Measures only open pores Cannot estimate smaller pores than molecules of gas	0.7~100nm
Ellipsometric porosimetry	Easy measurement at r.t.	Measures only open pores Cannot estimate smaller pores than molecules of gas	2nm~100nm
Positron Annihilation	Measures both open and closed pores	Requires large-scale equipment	~10nm
Dynamic light scattering	Reasonable-priced hardware Quick and easy measurement Needs	Measures only particles in dilute solution	1~6000nm
Electron microscope (SEM,TEM)	Observes real image	Cannot estimate average structure	0.1~100nm

Comparison / N₂ adsorption

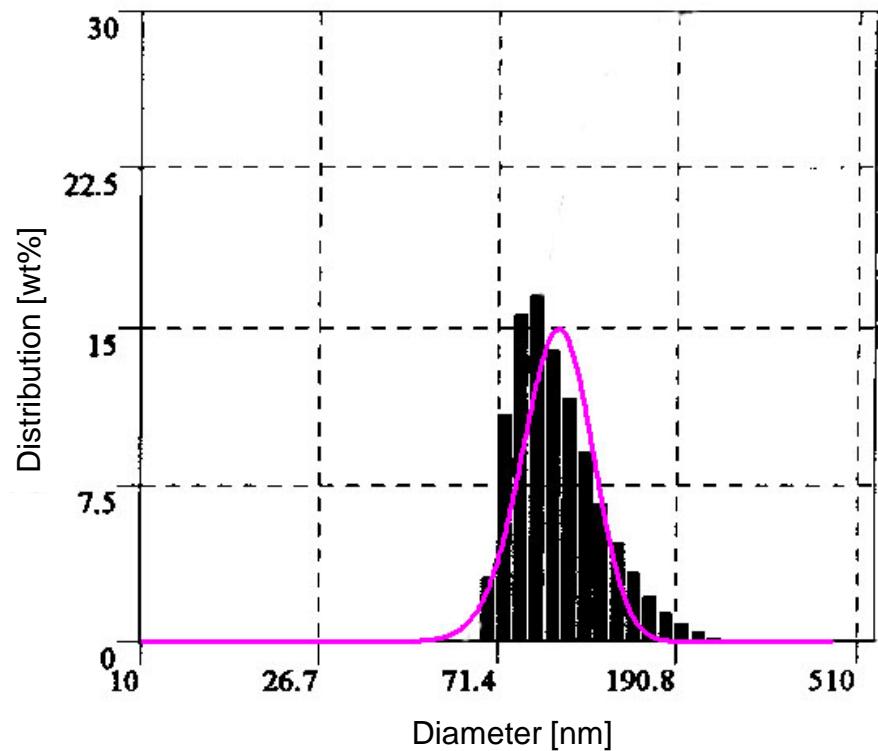
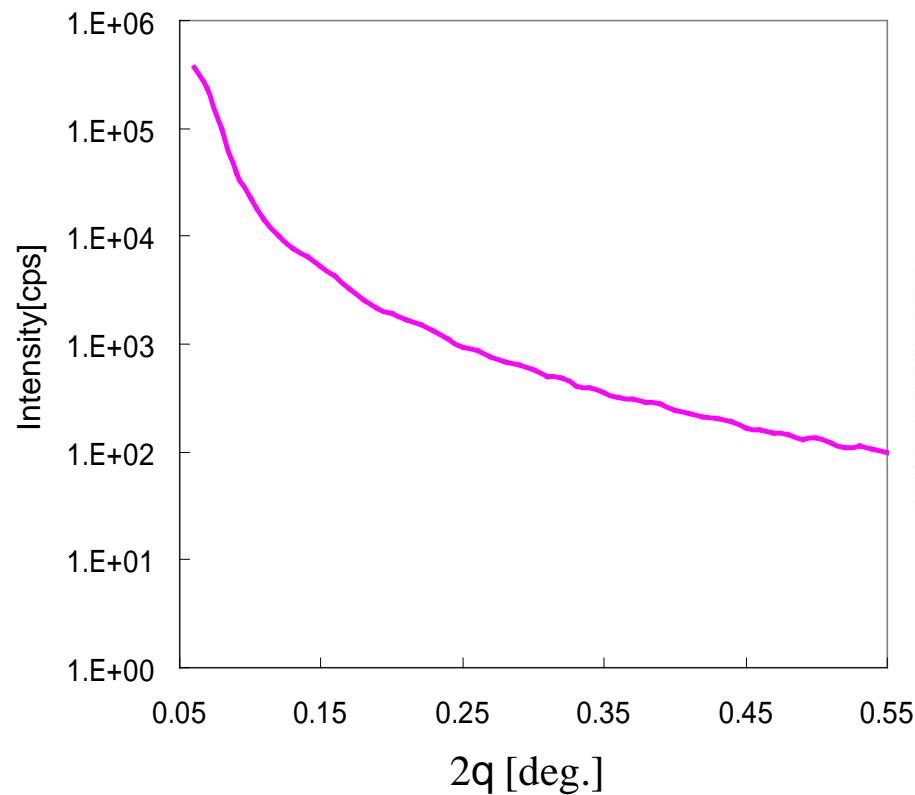
Pore-size distribution of mesoporous silica(powder)



Onaka et al. , University of Tokyo

Comparison / Light scattering

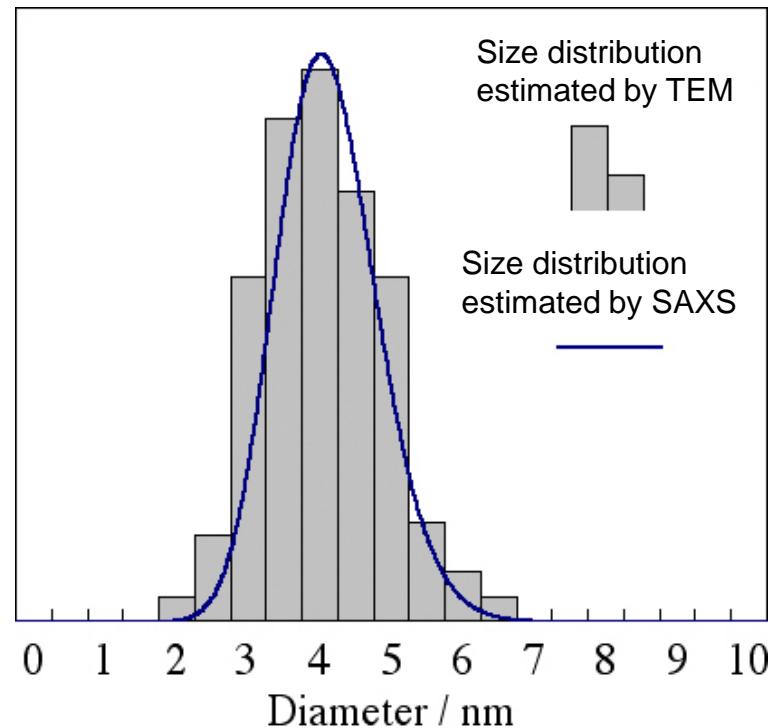
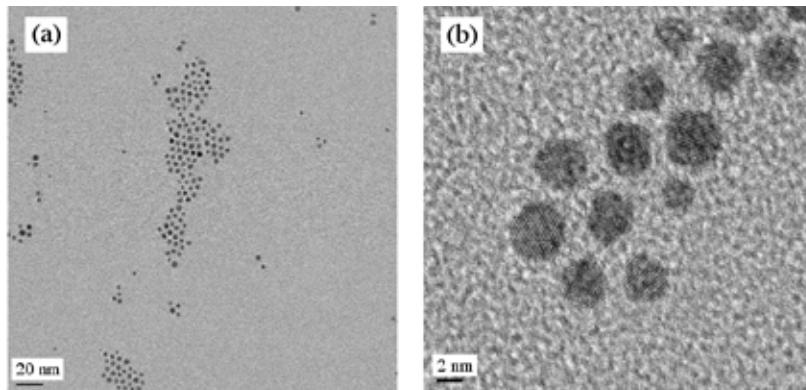
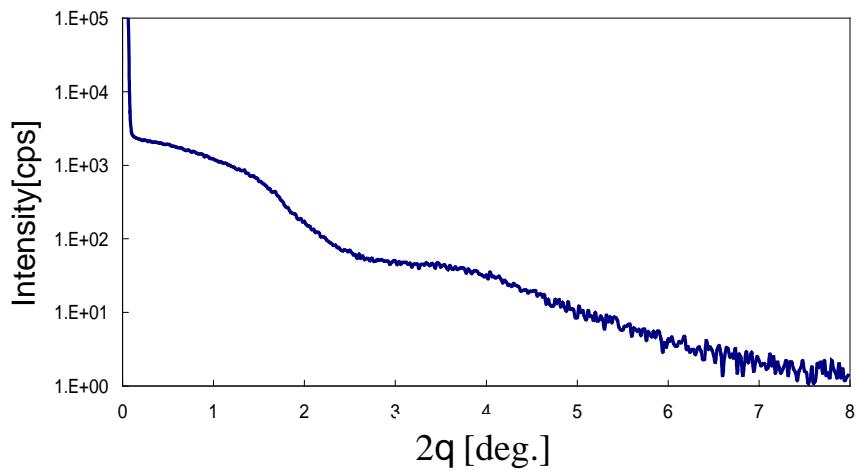
Size distribution of SiO_2 nanoparticles in ethanol



Nissan Chemical Industries, Ltd.

Comparison / TEM

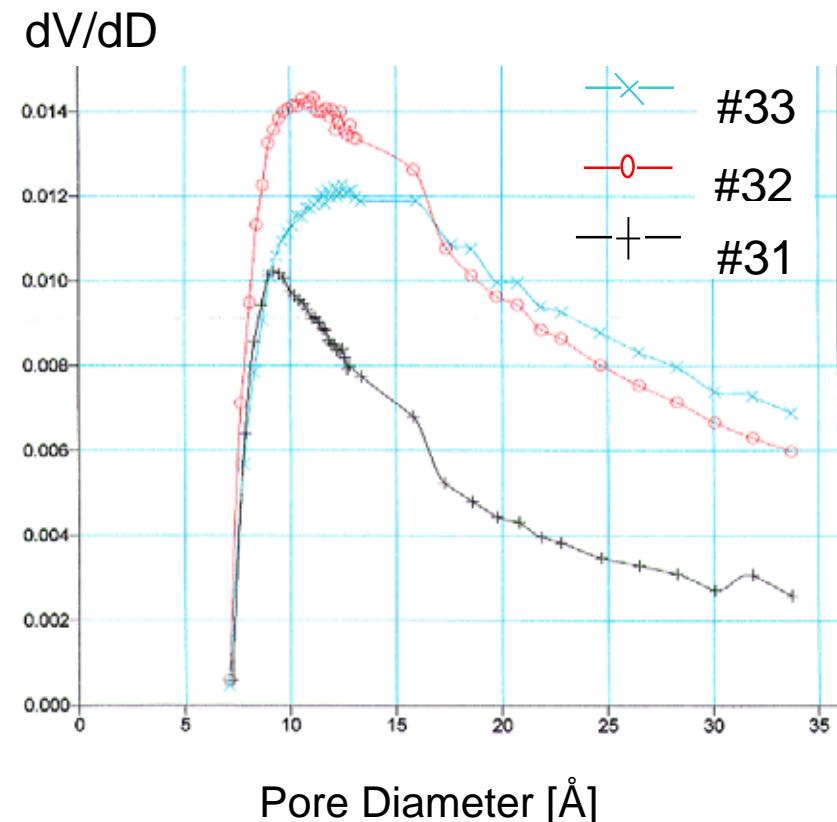
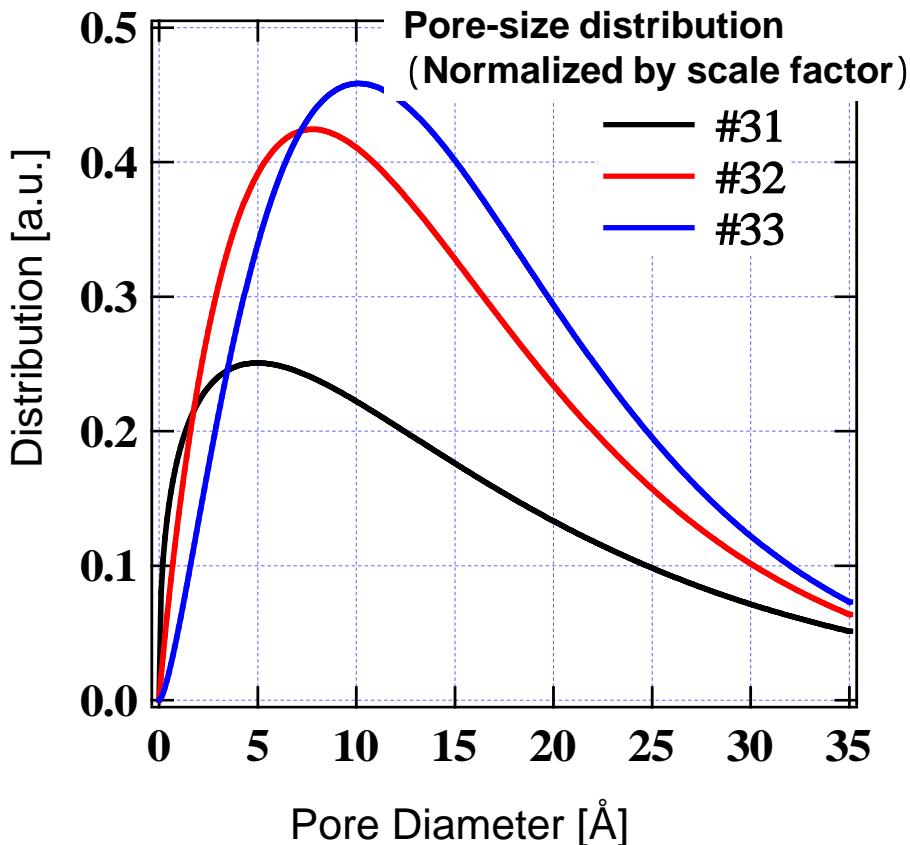
Size distribution of gold nanoparticles in toluene



T. Sugawara et al., Chem. Lett., 10, 1030-1031 (2002).

Comparison / Ar adsorption

Pore-size distribution of porous MSQ film on Si substrate



Summary

- **Use SAXS to see;**
 - Nano-scale periodic structure, texture, particle/pore size, density fluctuation of various materials.
- **Choose the correct instrument.**
 - Optimized configurations depend on what you want to see.
- **Respect raw data.**
 - Details can be lost by simplification of profiles.