

X-ray diffraction techniques for thin films

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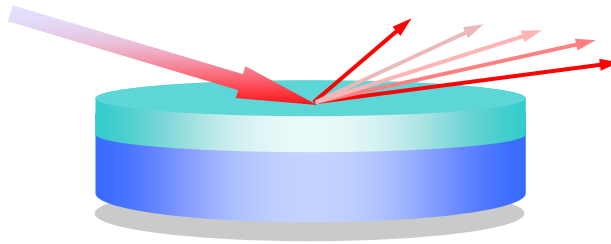
Today's contents (PM)

- **Introduction**
- X-ray diffraction method
 - Out-of-Plane
 - In-Plane
 - Pole figure
 - Reciprocal space mapping
 - High resolution rocking curve
- X-ray reflectivity



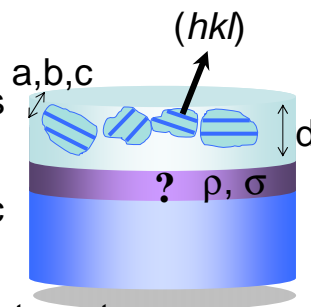
Advantage of X-ray diffraction (XRD) method

- Probed depth control by incidence angle
- Nondestructive
- Measurement under atmosphere pressure

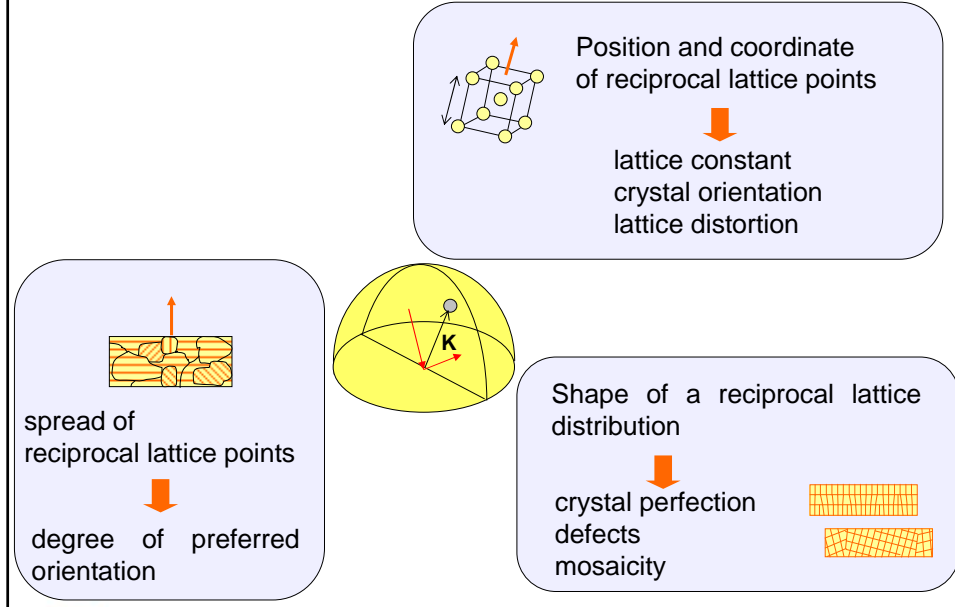


What can we see?

- Thickness, Density, Roughness
- Phase Identification
 - Interface, transition layer, etc
- Crystal structure
 - Crystal quality, lattice parameter, etc
- Crystal orientation
 - Single: orientation relation of substrate & film
 - Poly: preferred orientation



What XRD reveals



Structure parameters

| Structure Parameter | | Order | Analysis Method |
|---------------------|-----------------------|---|---|
| Layer Structure | Thickness | 1~10 ³ nm Precision : ~several % | Xray Reflectivity |
| | Density | H ₂ O~Heavy Metals | Xray Reflectivity |
| | Roughness | 0.2~several nm | Xray Reflectivity |
| Crystal Structure | Phase ID | - | In-Plane XRD Out-of-Plane XRD etc |
| | Crystal System | - | In-Plane XRD Out-of-Plane XRD etc |
| | Lattice Constant | ~several nm Precision : 0.05~0.00005nm | In-Plane XRD Out-of-Plane XRD etc |
| | Crystal quality | Poly~Single, Perfect Crystals | In-Plane XRD Out-of-Plane XRD etc |
| | Preferred Orientation | Random~Preferred Orientation ~Single Crystal | Pole Figure ect |
| | Orientation Relation | Relation between Film & Substrate | Rocking Curve Reciprocal Space Map etc |

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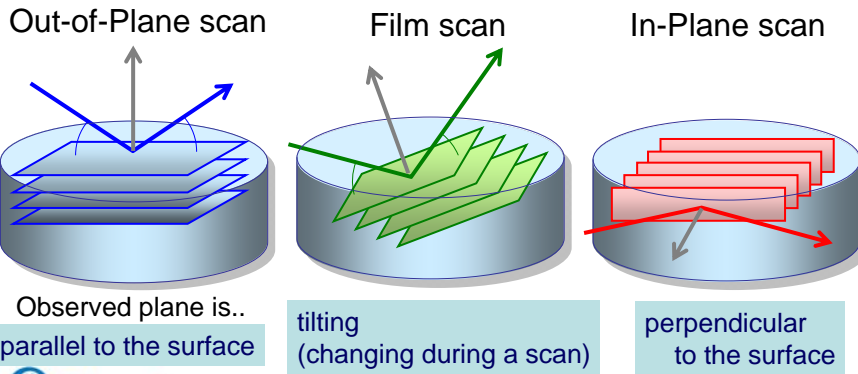
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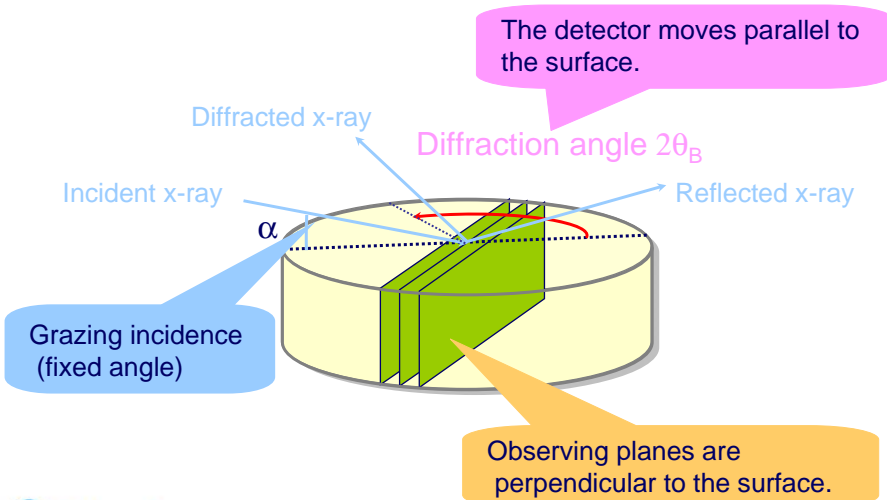
Difference between Scan Modes

- The orientation of observed crystal plane depends on scanning mode.



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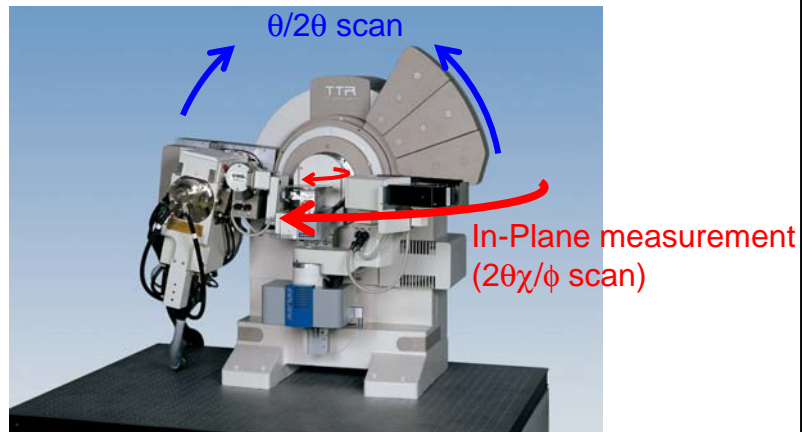
What is In-Plane XRD?



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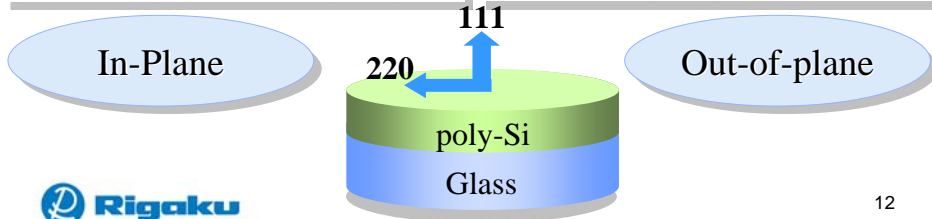
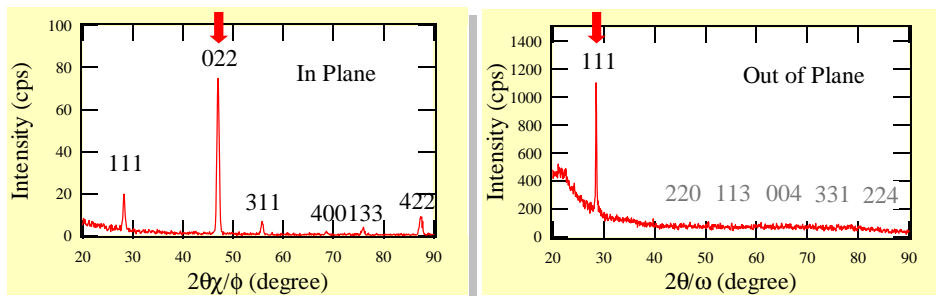
Outward of In-Plane Attachment

- Scanning motion is completely perpendicular to $\theta/2\theta$ scan.



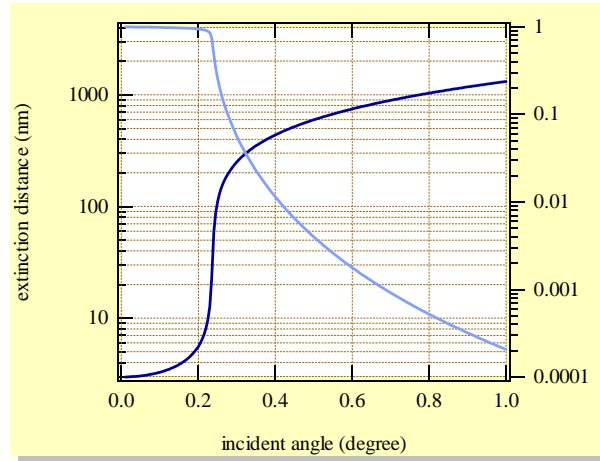
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In-plane effect



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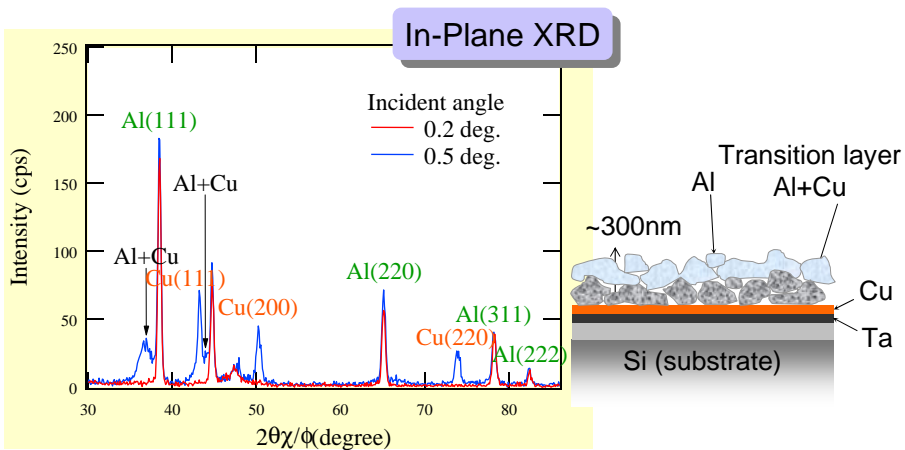
Probed depth control ?



Sample:Al Wavelength:1.54056Å (CuKα1)



Surface & Interface Structure

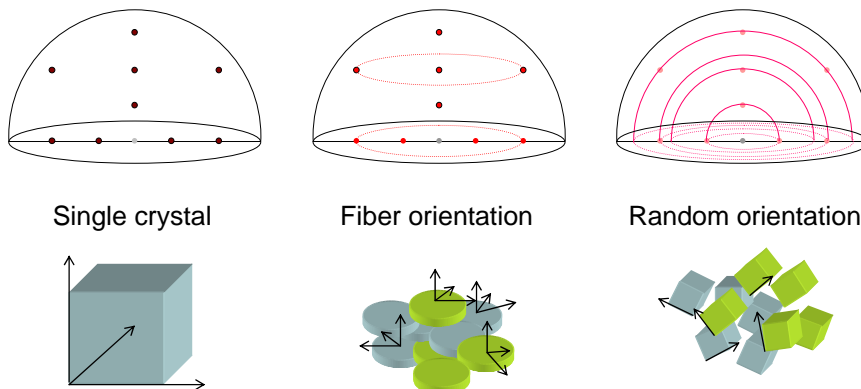


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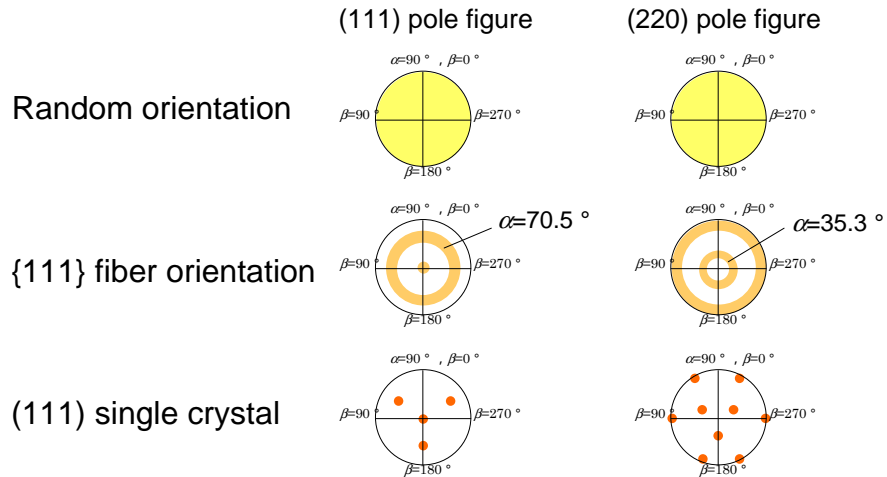
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Single crystal and random orientation



Orientation conditions and pole figure



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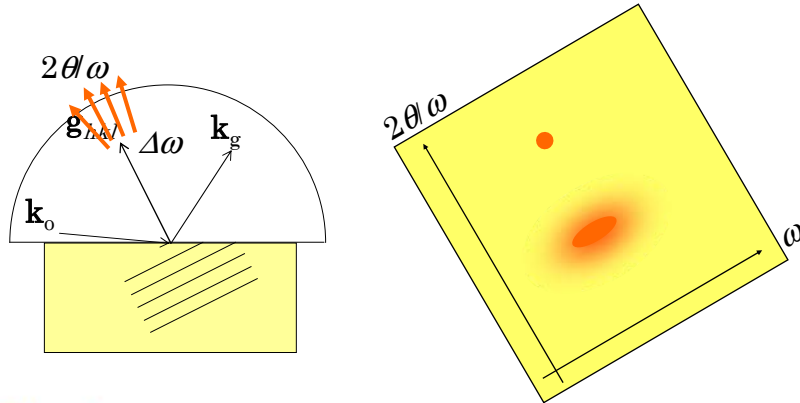
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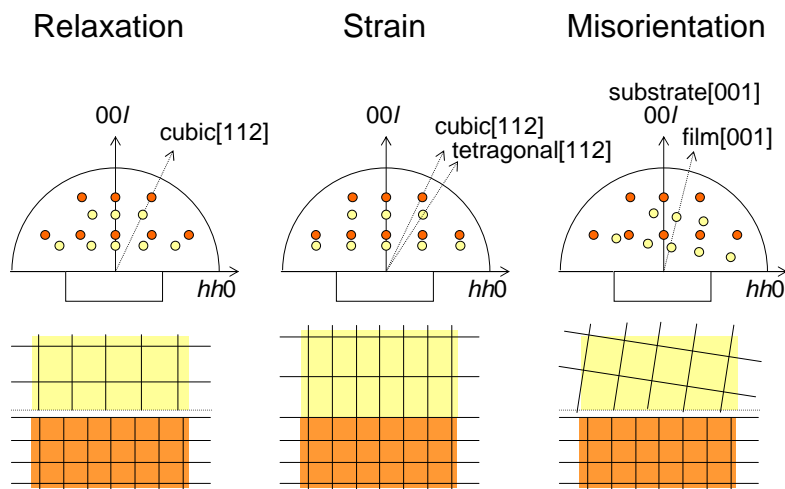
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Reciprocal space mapping

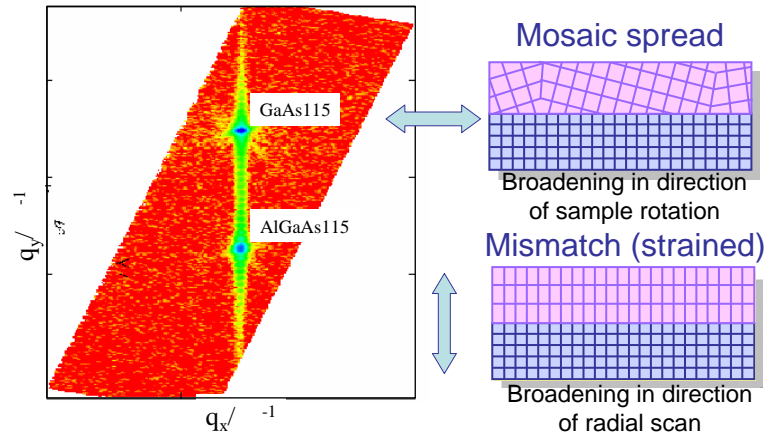
- Diffraction intensity distribution is plotted on reciprocal space.



Epitaxial layer structures



Reciprocal space mapping

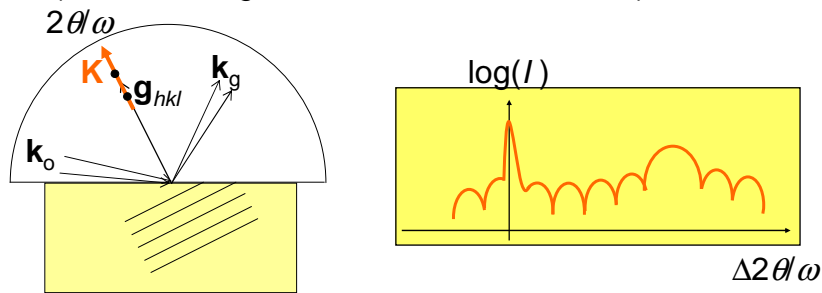


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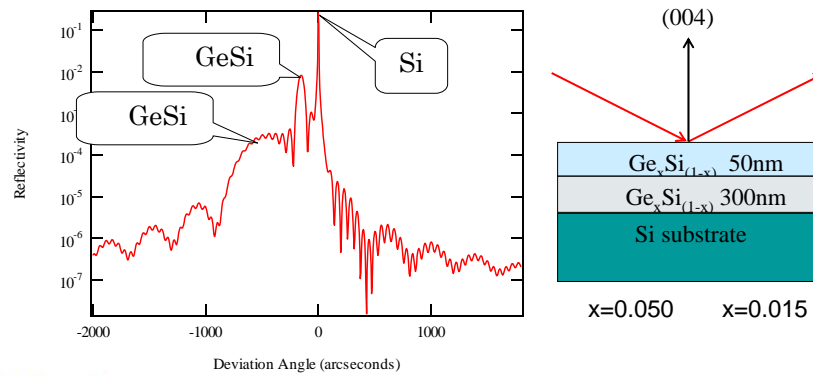
High- resolution rocking curve

- The differences of lattice spacing between the substrate and epitaxial films are observed.
 - Thickness and composition ratio of epitaxial films (when the degree of relaxation is known.)



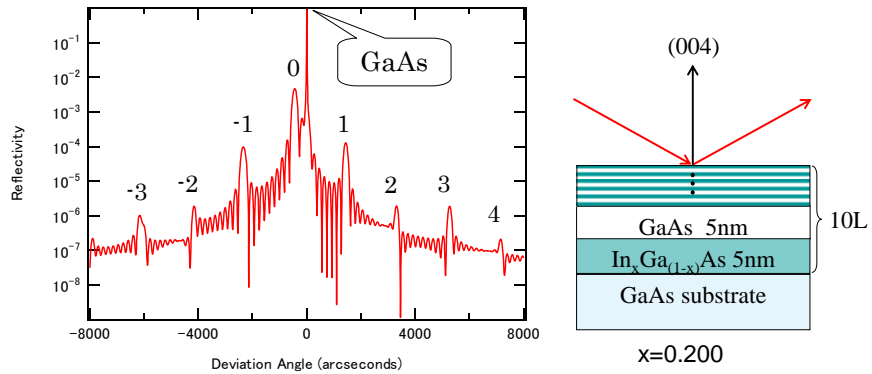
When the sample has multilayer structure...

- Complicated oscillation composed of oscillation from each layer is observed.

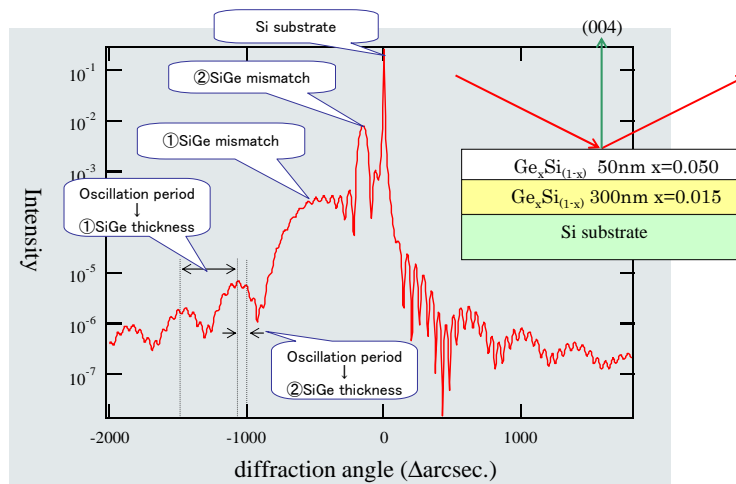


When the sample has superlattice structure...

- Satellite peaks are observed.



How to interpret the profile



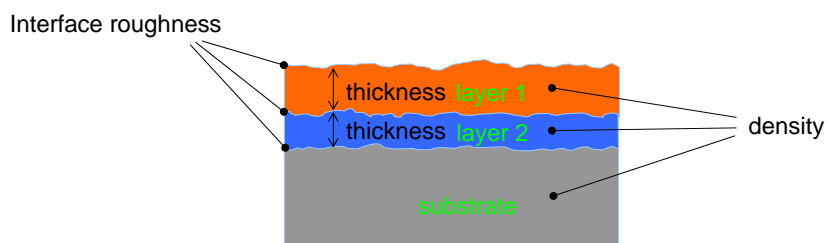
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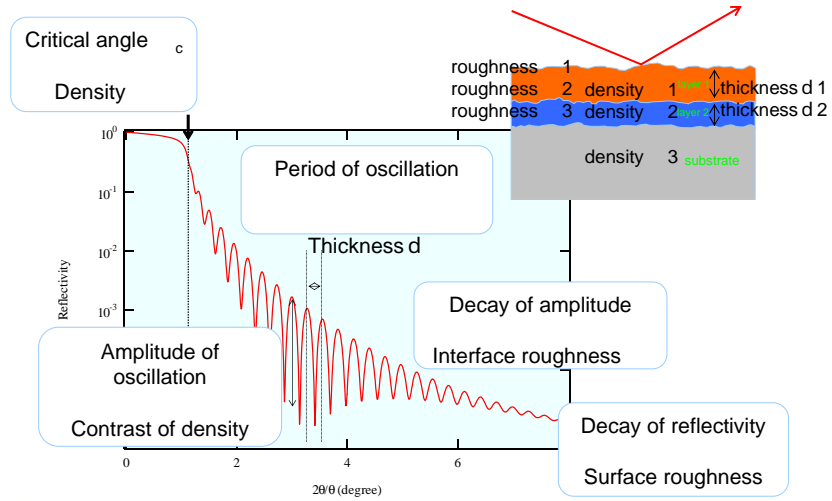
What reflectivity reveals

X-ray reflectivity nondestructively reveals

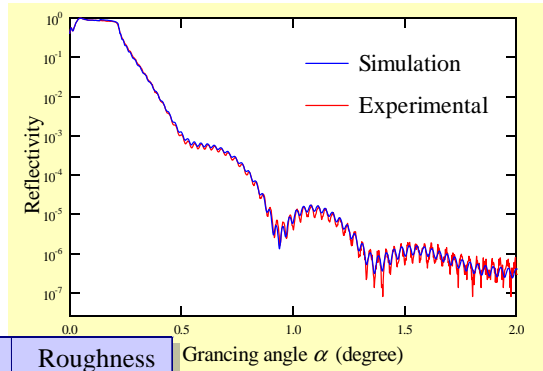
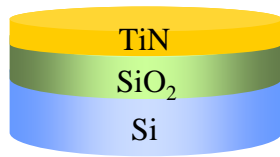
- layer structure of multi layers
- thickness (1 to 1000nm)
- density as an absolute value
- surface and interface roughness



How to interpret the profile



X-ray reflectivity measurement of TiN film



| Layer | density (g/cm ³) | Thickness (nm) | Roughness (nm) |
|------------------|------------------------------|----------------|----------------|
| TiN | 3.680 | 1.230 | 1.420 |
| TiN | 2.900 | 8.400 | 1.000 |
| SiO ₂ | 2.260 | 127.700 | 0.220 |
| Si | substrate | | |

Coating layer