2008 Advanced Materials Characterization Workshop

X-ray analysis methods

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Sponsors:

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Outline

- *X-rays interactions with the matter*
- *Fundaments of diffraction*
- *Powder diffraction methods*
  - *Size / strain analysis*
  - *Search / Match, structure determination*
  - *Quantitative analysis, whole pattern fitting*
- *X-ray parallel beam methods*
  - *Thin film crystallographic orientation*
  - *Glancing / Grazing angle XRD methods*
- *Texture - preferred orientation methods*
- *Residual stress analysis methods*
- *High resolution XRD methods*
  - *Rocking curve analysis*
  - *Reciprocal lattice mapping*
- *X-ray reflectivity methods*
- *X-ray fluorescence methods*
- *X-ray analysis summary*
- *Comparison with other techniques*
- *Quick guide to the FS-MRL x-ray analysis facilities*
- *Recommended literature.*

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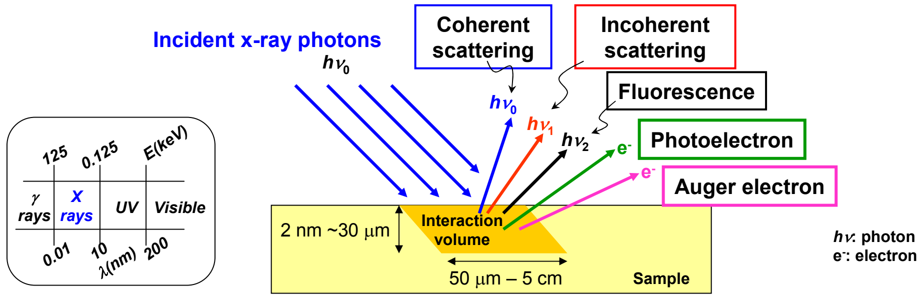
X-ray interactions with matter



$h\nu$: photon
e^-: electron

Coherent scattering (Thompson scattering / diffraction): incident photon $h\nu_o$ interacts with e^- with **no energy loss** and **no phase change**

Incoherent scattering (Compton scattering): (a) e^- absorbs incident energy $h\nu_o$ (excited photoelectron); (b) part of the energy is emitted at **different energy $h\nu_1$** and **different phase**.

Fluorescence: (a) K/L shell e^- absorb incident energy $h\nu_o$; (b) outer shell e- "cascade" down filling the "holes" causing secondary photons emission ($h\nu_2$).

Photoelectron emission: $h\nu_0$ energy is used to eject electron e^- with kinetic energy = $h\nu_o$ – B.E.(binding energy).

Auger electron emission: (a) incident $h\nu_0$ used to eject e^- from atom; (b) 2nd e^- "drops" to lower levels to fill the "hole" and a photon is emitted; (c) the emitted photon is absorbed by valance e^-, which ionizes and leaves the atom.

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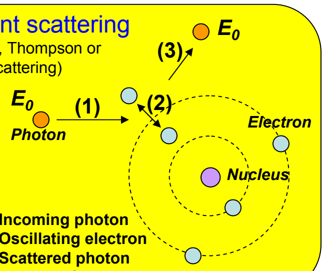
X-ray interactions with matter

Coherent scattering

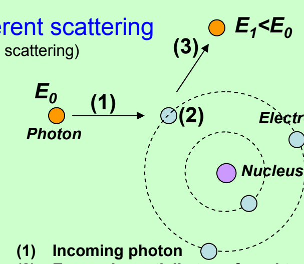
(Diffraction, Thompson or Rayleigh scattering)



(1) Incoming photon
(2) Oscillating electron
(3) Scattered photon
No loss of energy.

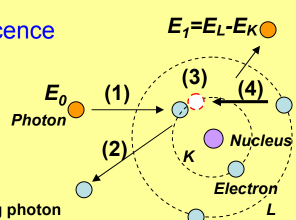
Incoherent scattering

(Compton scattering)



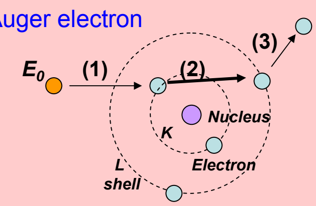
(1) Incoming photon
(2) Energy is partially transferred to electron
(3) Scattered photon (energy loss).

Fluorescence

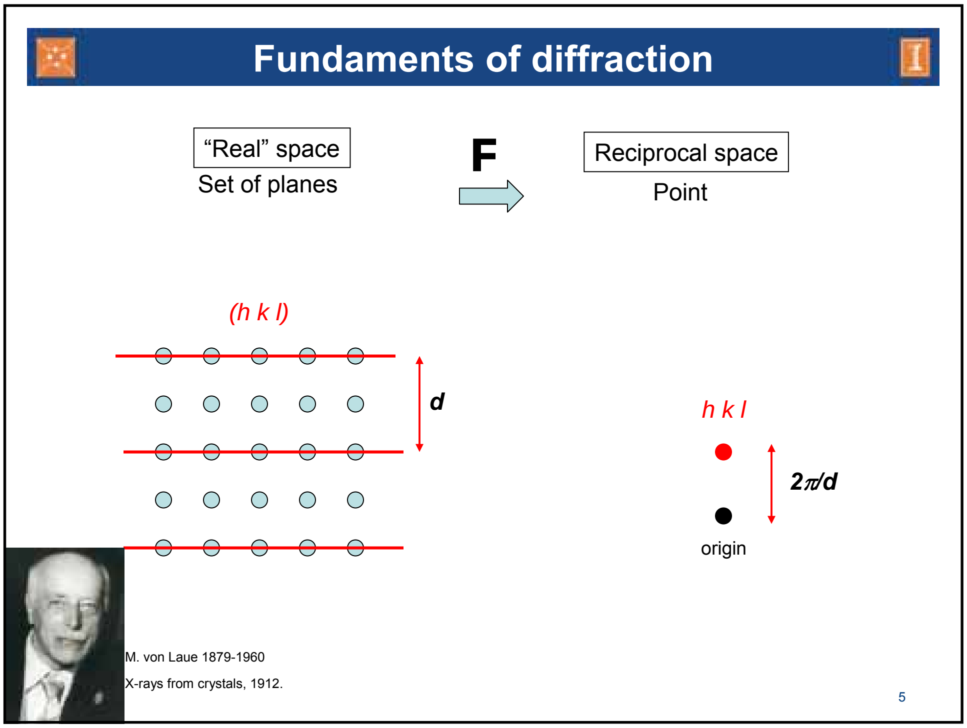


(1) Incoming photon
(2) Expelled electron (photoelectron)
(3) Hole is created in the shell
(4) Outer shell electron moves to the inner shell hole
(5) Energy excess emitted as characteristic photon.

Auger electron



(1) Incoming photon excites inner shell electron
(2) Excitation energy is transferred to outer electron
(3) Electron ejected from atom (Auger electron)


Fundaments of diffraction
“Real” space
Set of planes
F
Reciprocal space
Point
(h k l)
d
h k l
2π/d
origin
M. von Laue 1879-1960
X-rays from crystals, 1912.
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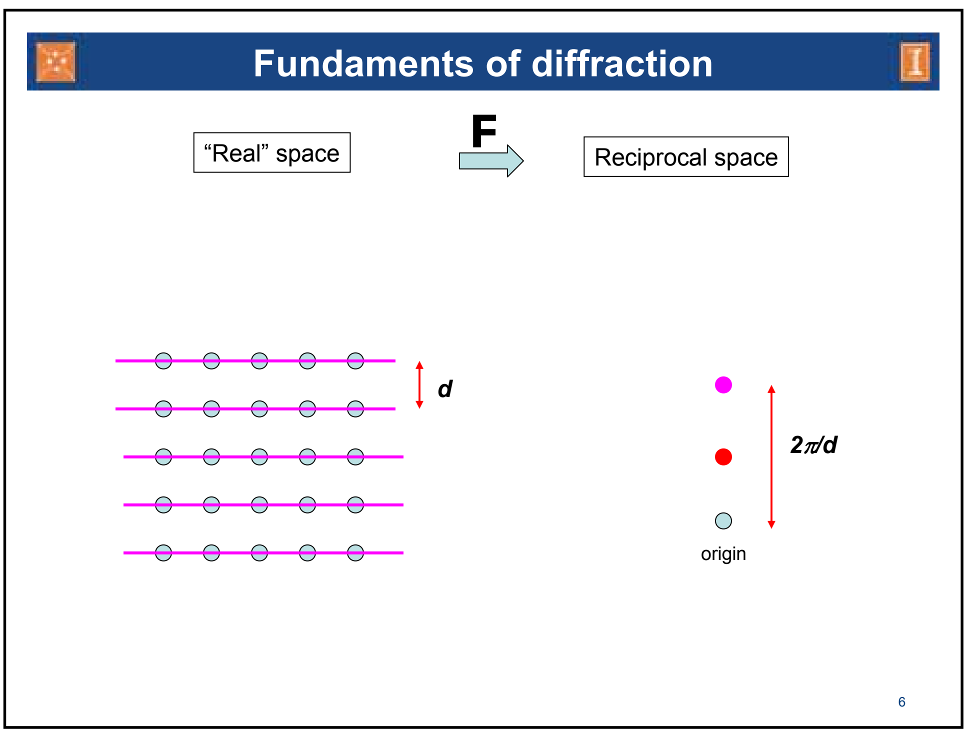
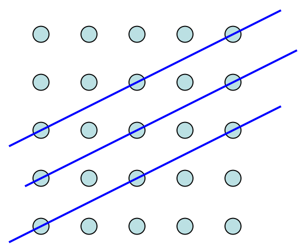
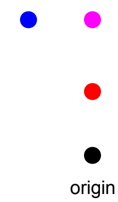

Fundaments of diffraction
“Real” space
F
Reciprocal space
d
2π/d
origin
6

Fundaments of diffraction

"Real" space **F** → Reciprocal space

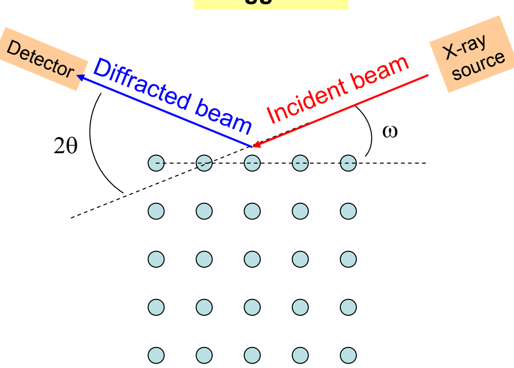


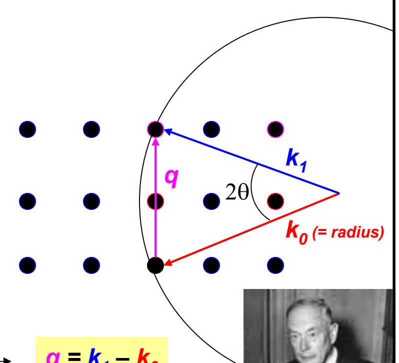


Fundaments of diffraction

"Real" space **F** → Reciprocal space

Bragg's law | **Ewald's sphere**



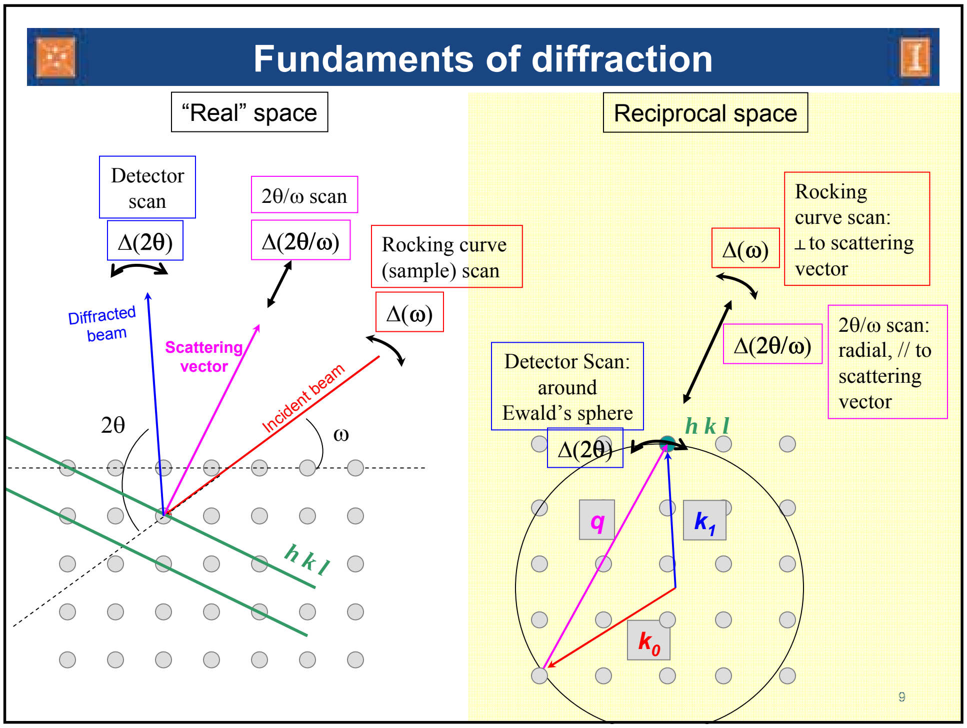


2 d sin θ ***= n*** λ

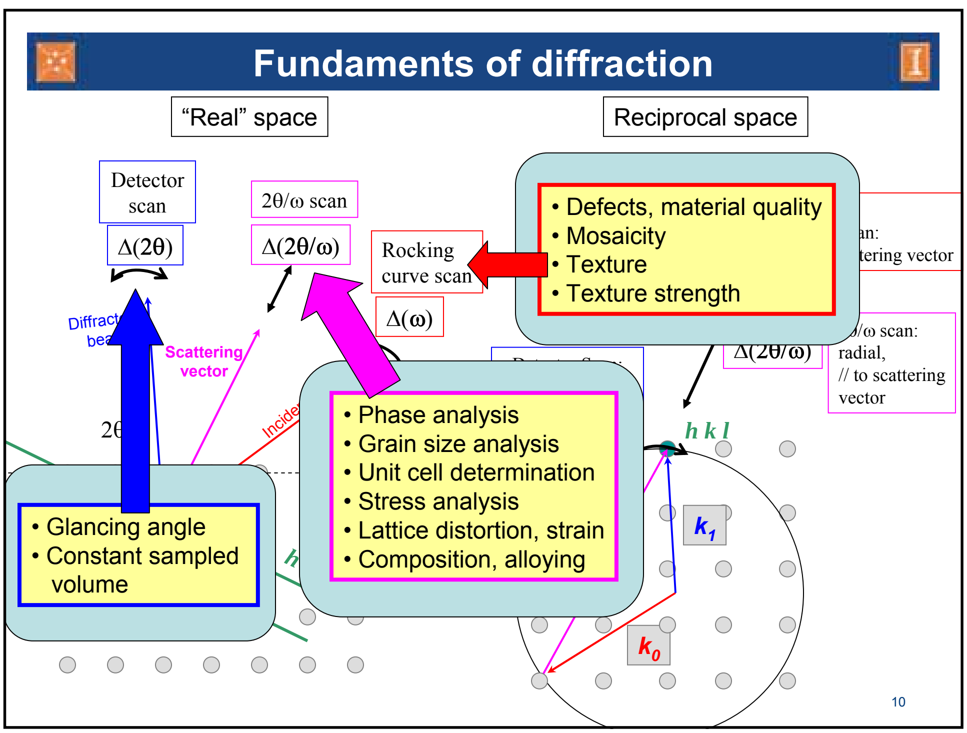
Elastic (Thompson's) scattering

$\mathbf{q} = \mathbf{k}_1 - \mathbf{k}_0$

q: scattering vector
$q = (4\pi/\lambda)\sin\theta$

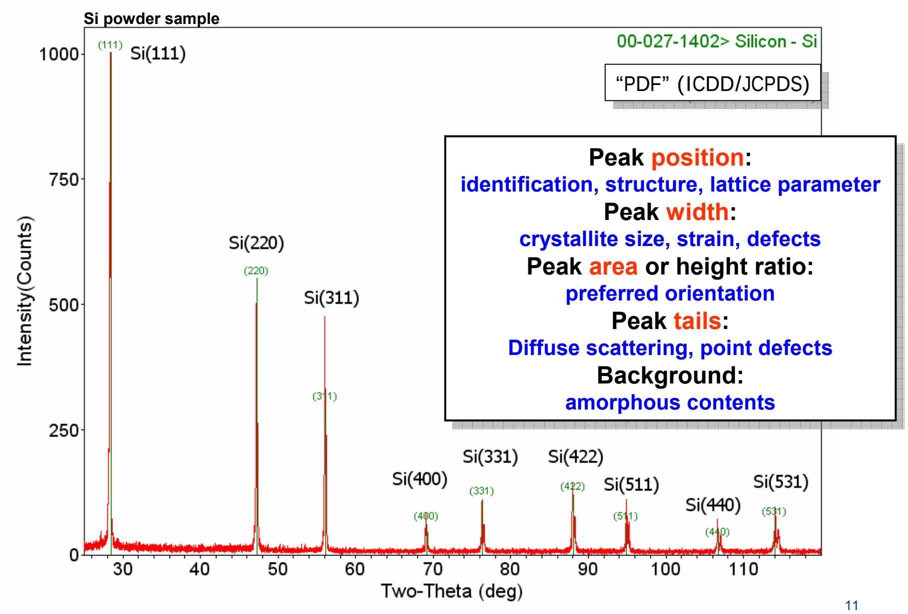


Fundaments of diffraction
"Real" space
Reciprocal space
Detector scan
Δ(2θ)
2θ/ω scan
Δ(2θ/ω)
Rocking curve (sample) scan
Δ(ω)
Diffracted beam
Scattering vector
Incident beam
2θ
ω
h k l
Rocking curve scan: ⊥ to scattering vector
Δ(ω)
Δ(2θ/ω)
2θ/ω scan: radial, // to scattering vector
Detector Scan: around Ewald's sphere
Δ(2θ)
h k l
q
k1
k0
9



Fundaments of diffraction
"Real" space
Reciprocal space
Detector scan
Δ(2θ)
2θ/ω scan
Δ(2θ/ω)
Rocking curve scan
Δ(ω)
• Defects, material quality
• Mosaicity
• Texture
• Texture strength
• Phase analysis
• Grain size analysis
• Unit cell determination
• Stress analysis
• Lattice distortion, strain
• Composition, alloying
• Glancing angle
• Constant sampled volume
Scattering vector
Δ(2θ/ω)
radial,
// to scattering vector
h k l
k1
k0
10

Typical contents from XRD pattern (diffractogram)



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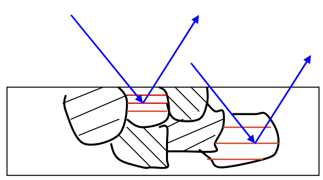
Powder diffraction methods



Crystalline? Amorphous?

What elements, compounds, phases are present?

Structure? Lattice constants?

Strain?

Grain sizes? Grain orientations?

Is there a mixture? What % ?

Powders, bulk materials, thin films, nanoparticles, soft materials.

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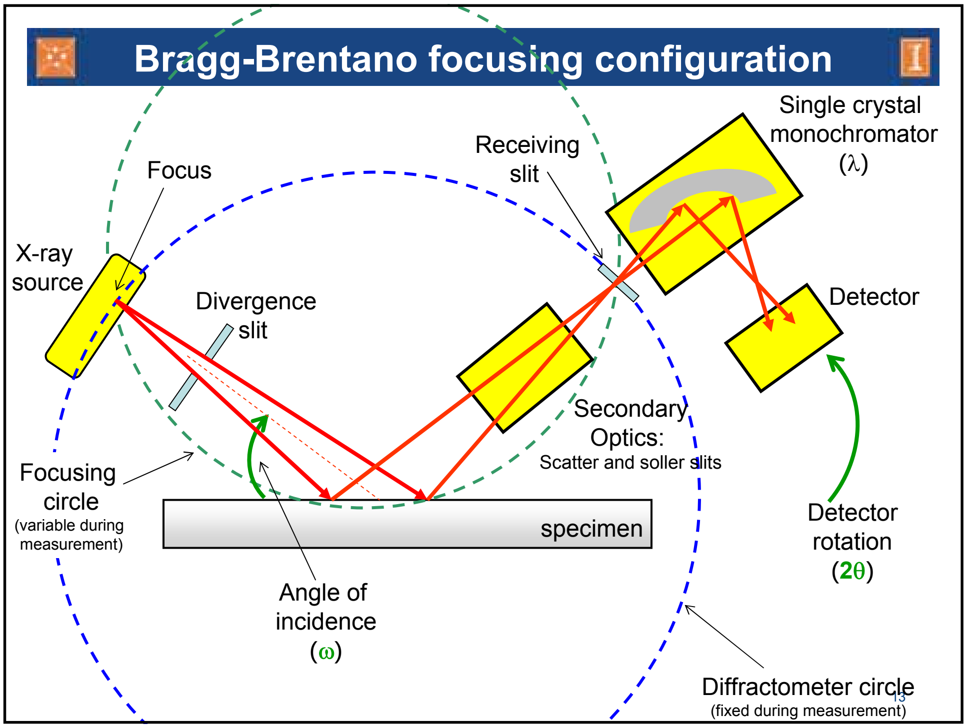
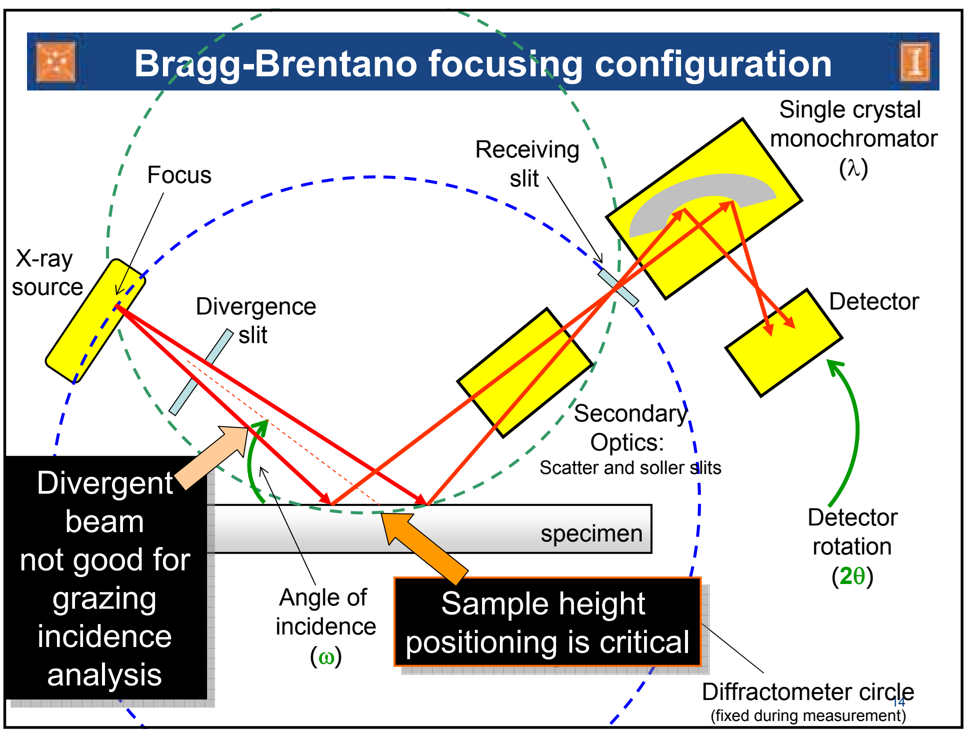
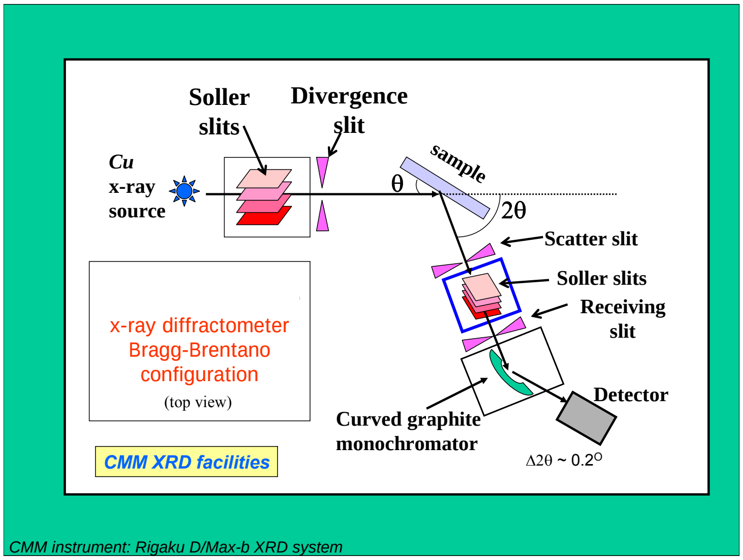

Bragg-Brentano focusing configuration
Single crystal monochromator (λ)
Receiving slit
Focus
X-ray source
Divergence slit
Detector
Secondary Optics:
Scatter and soller slits
Focusing circle
(variable during measurement)
specimen
Detector rotation (2θ)
Angle of incidence (ω)
Diffractometer circle
(fixed during measurement)

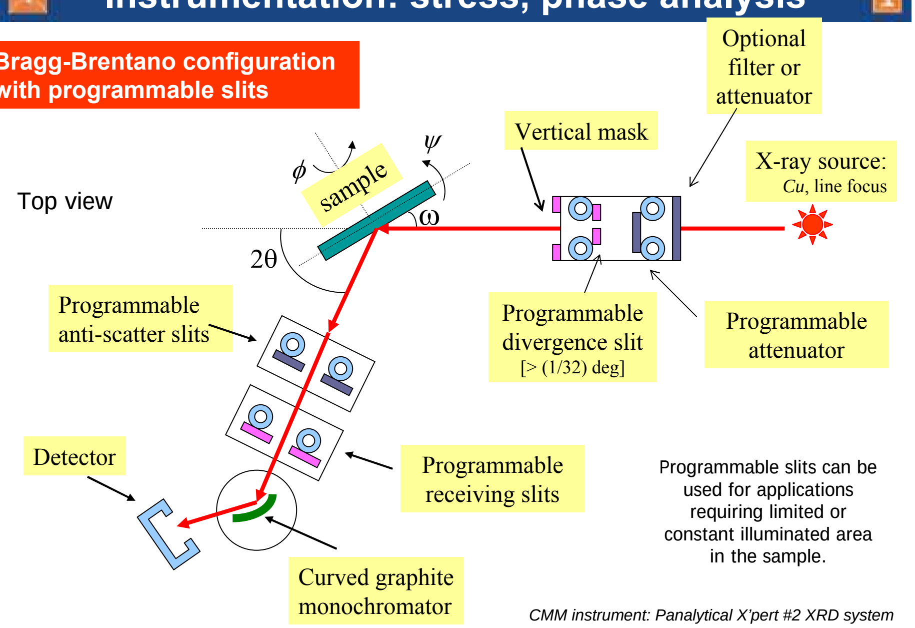

Bragg-Brentano focusing configuration
Single crystal monochromator (λ)
Receiving slit
Focus
X-ray source
Divergence slit
Detector
Secondary Optics:
Scatter and soller slits
Divergent beam not good for grazing incidence analysis
specimen
Detector rotation (2θ)
Angle of incidence (ω)
Sample height positioning is critical
Diffractometer circle
(fixed during measurement)

Instrumentation: powder diffraction



Instrumentation: stress, phase analysis

Bragg-Brentano configuration with programmable slits



Programmable slits can be used for applications requiring limited or constant illuminated area in the sample.

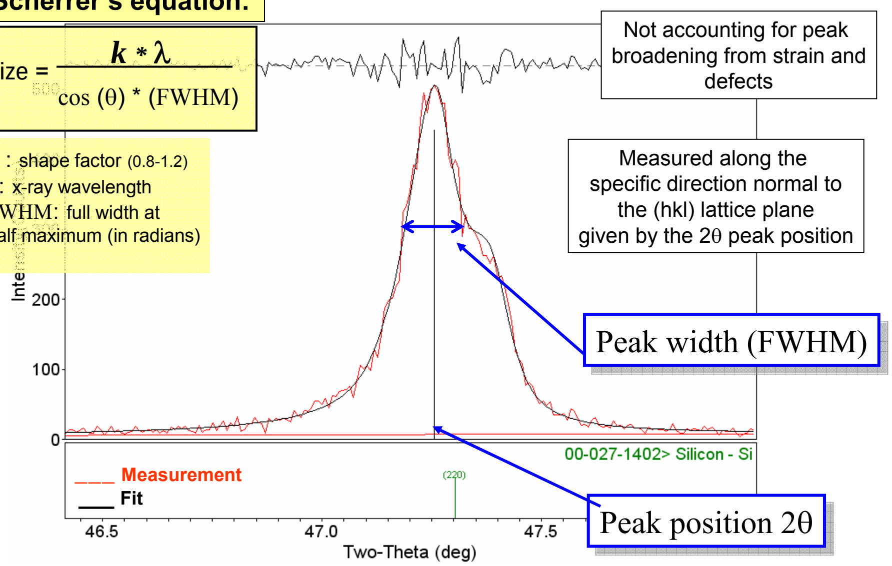
CMM instrument: Panalytical X'pert #2 XRD system

Crystallite size analysis

Scherrer's equation:

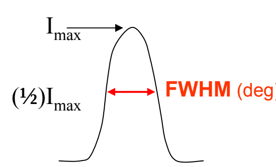
$$\text{Size} = \frac{k * \lambda}{\cos(\theta) * (\text{FWHM})}$$

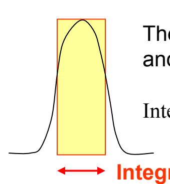
k : shape factor (0.8-1.2)
λ: x-ray wavelength
FWHM: full width at half maximum (in radians)



FWHM vs. integral breadth

It does not take into account the lower part of the profile



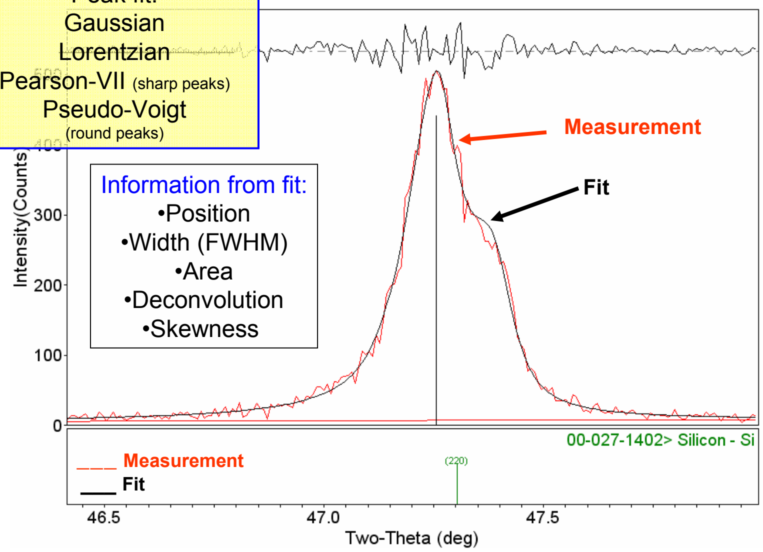


The square-topped profile has the same area and peak height as the measured curve.

Integral breadth = (total area) / (peak height)

Peak shape analysis

Peak fit:
Gaussian
Lorentzian
Pearson-VII (sharp peaks)
Pseudo-Voigt (round peaks)



Information from fit:
- Position
- Width (FWHM)
- Area
- Deconvolution
- Skewness

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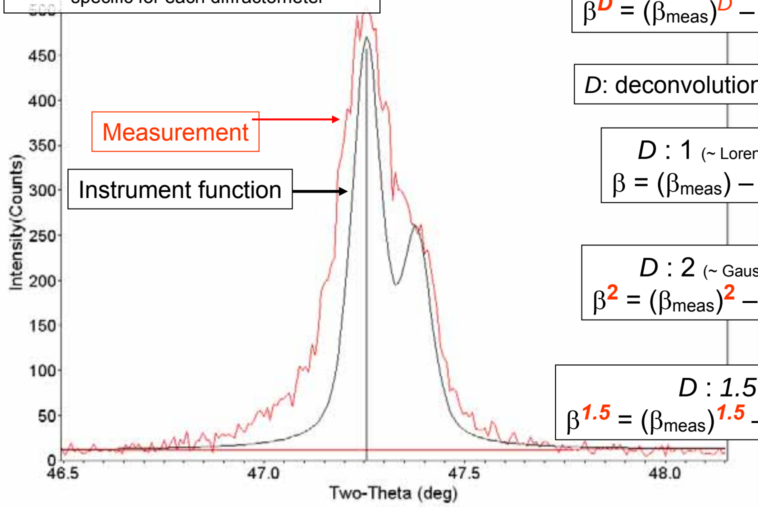
Correction for instrument resolution

Use FWHM curve as a function of 2θ from standard sample (NIST LaB_6)
- specific for each diffractometer



FWHM: β

$\beta^D = (\beta_{meas})^D - (\beta_{instr})^D$

D: deconvolution parameter

D : 1 (~ Lorentzian)

$\beta = (\beta_{meas}) - (\beta_{instr})$

D : 2 (~ Gaussian)

$\beta^2 = (\beta_{meas})^2 - (\beta_{instr})^2$

D : 1.5

$\beta^{1.5} = (\beta_{meas})^{1.5} - (\beta_{instr})^{1.5}$

Software: MDI's Jade + 8.0

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Potential artifacts in size determination

FWHM: β

$\beta^D = (\beta_{meas})^D - (\beta_{instr})^D$

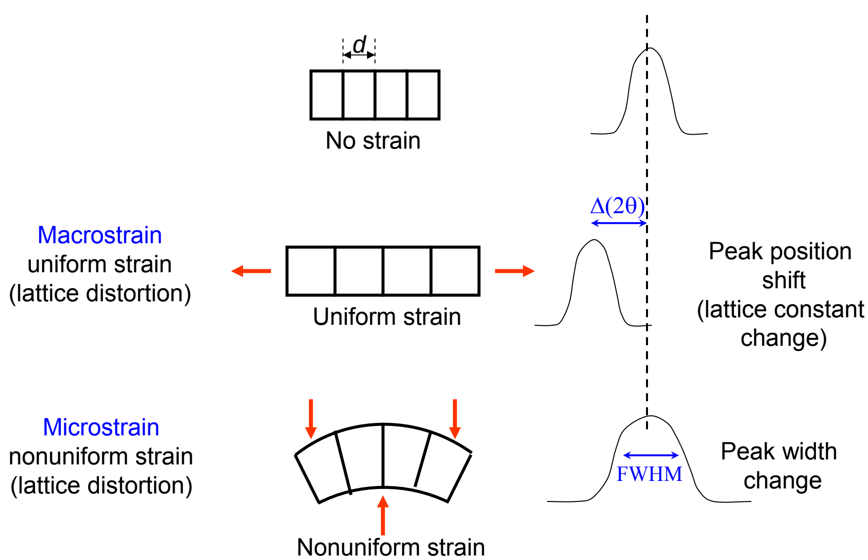
Assume:
- Instrument resolution ~ 0.15°
- 2θ = 40°
- Cu radiation

Measured peak width (°)	Size, nm *D = 1* (Lorentzian)	Size, nm *D = 1.5*	Size, nm *D = 2* (Gaussian)
0.3	56.4	38.0	32.6
0.5	24.2	19.2	17.7
0.75	14.1	12.0	11.5
1	10.1	8.8	8.6
1.5	6.3	5.8	5.7
2	4.6	4.3	4.2

Large difference (up to 48%!) for narrow peaks (large sizes)

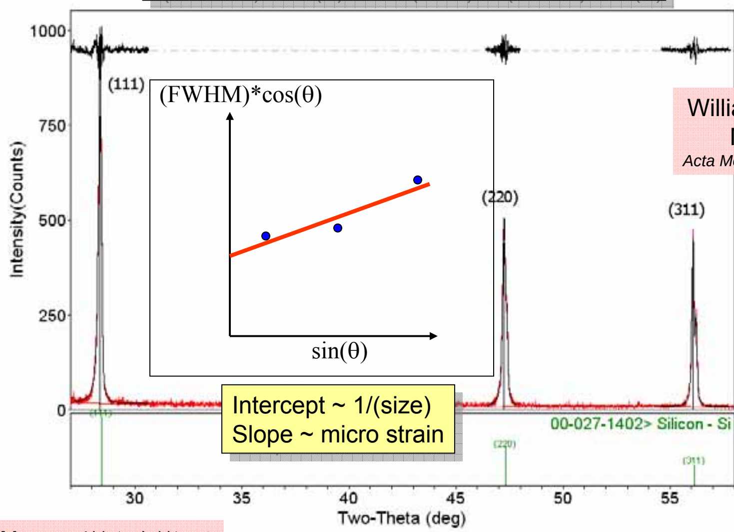
Smaller difference (~ 10%) for broad peaks (small sizes)

Strain effects in diffraction lines



Size and strain in peak shape analysis

$(FWHM)*\cos(\theta) = k\lambda/(size) + (strain)*\sin(\theta)$



Intercept ~ 1/(size)
Slope ~ micro strain

Williamson-Hall Method
Acta Metall. ***1*** *(1953) 22.*

$FWHM_{strain} = 4*(strain)*\tan\theta$

Other methods of grain size analysis

Rietveld refinement method:

H. Rietveld (1932-)

- Refines the whole diffraction pattern (including background)
- Needs detailed data over a wide angular range
- Gives one average size value
- Works better for powder samples (not well with films with strong preferred orientation)
- Data processing (refinement and "play" with parameters) is time consuming.

Warren-Averback method:

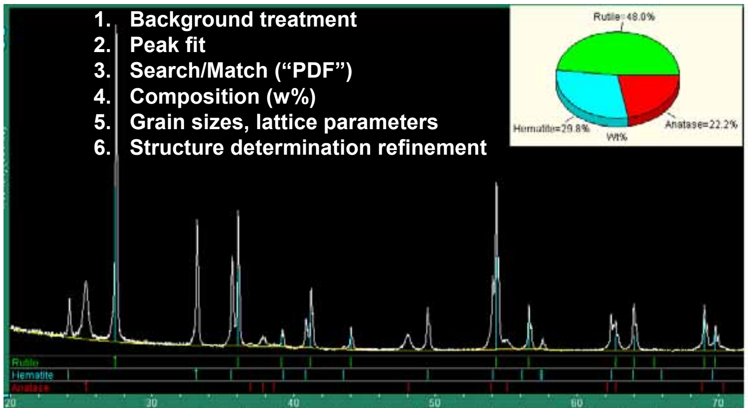
Standard sample -> Instrumental Broadening -> Correct measured peaks assuming "error"-type of function (main assumption) $I \sim \exp(-p\phi)^2$ -> Use Scherrer's equation

Jones' method:

Measured profile = convolution ("pure", instrument)
Use Fourier integrals for all the profiles (measured, "pure", instrument)
Use ratio of the integrals to determine sizes.

Pattern treatment, S/M and structure refinement

1. Background treatment
2. Peak fit
3. Search/Match ("PDF")
4. Composition (w%)
5. Grain sizes, lattice parameters
6. Structure determination refinement



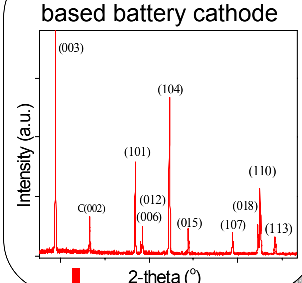
Weight % of parts in the mixture

25 Hits Sorted on Figure Of M...	Chemical Formula	FOM	J	D	PDF #	Hits	#d/I	I%	2T(0)	RIR	Space Group	a	b	c	Z	G
Rutile, syn	TiO2	2.0	•	D	00-021-1276	0	15	49	-0.020	3.40	P42/mnm (136)	4.593	4.593	2.959	2	
Iron Titanium Oxide	Fe9TiO15	5.2	•	D	00-054-1267	0	13	40	0.040		R-3c (167)	5.032	5.032	13.754	1.23	
Hematite, syn	Fe2O3	6.2	•	D	00-033-0664	0	19	18	-0.020	2.40	R-3c (167)	5.036	5.036	13.749	6	
Chromium Titanium Antimony ...	CrTiSbO6	8.2	•	D	00-050-0179	0	15	82	0.080		P42/mnm (136)	4.591	4.591	3.009	0.5	
Nickel Titanium Oxide	NiTiO3	10.2	•	D	00-033-0960	0	24	25	0.060		R-3 (148)	5.030	5.030	13.790	6	
Chromium Niobium Vanadium ...	CrVNbO6	10.5	•	D	00-036-0788	0	11	49	0.120		P42/mnm (136)	4.606	4.606	2.989		
Iron Titanium Antimony Oxide	(Ti1.80Fe0.10Sb0....	12.2	•	D	00-055-0051	0	12	97	0.120		P42/mnm (136)	4.601	4.601	2.980	1	
Iron Chromium Oxide	(Fe0.6Cr0.4)2O3	15.1	•	D	00-034-0412	0	20	23	-0.100		R	5.017	5.017	13.643	6	
Aluminum Tantalum Oxide	AlTaO4	19.6	•	V	00-012-0407	0	12	88	-0.120		P42/mnm (136)	4.568	4.568	2.964		
Gallium Antimony Titanium Oxi...	Ga0.33Sb0.33Ti0....	20.9	•	D	00-040-0912	0	11	67	0.120		P42/mnm (136)	4.598	4.598	3.015	2	
Iron Manganese Oxide Hydro...	(Fe0.67Mn0.33)OOH	22.1	?	V	00-014-0557	0	4	15	-0.060			2.905	2.905	4.555		
Magnesium Chromium Fluoride	MgCrF6	22.2	•	V	00-026-1215	0	11	10	0.100		R-3 (148)	5.091	5.091	13.143	3	
Chromium Tungsten Oxide	Cr2WO6	24.2	•	D	00-035-0791	0	27	34	-0.100		P42/mnm (136)	4.580	4.580	8.867	2	
Chromium Rhodium Oxide	CrRhO3	25.1	?	D	00-042-0410	0	15	37	0.080	1.40	R-3 (148)	5.031	5.031	13.767	6	
Aluminum Titanium Tantalum ...	AlTiTaO6	28.1		F	00-032-0028	0	12	25	-0.060			4.579	4.579	2.966	0.66	
Anatase, syn	TiO2	32.0	•	X	00-021-1272	0	14	10	0.040	3.30	I41/amd (141)	3.785	3.785	9.514	4	
Cobalt Germanium Oxide	CoGeO3	39.8	•	V	00-035-1055	0	10	12	-0.100		R	4.973	4.973	13.032	6	

Software:
MDI's Jade + 8.0

Whole pattern fitting and structure refinement

Pattern from a Li-Ni-based battery cathode

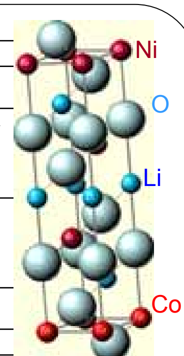


- Bragg-Brentano or parallel beam x-ray analysis.
- Powders, polycrystalline films or nanostructures, mineralogy, cements, ceramics, pharmaceutics, polymers, biomaterials.
- Identification, quantification (w%) and structure determination of mixtures, impurities, multiple phases and amorphous fractions.
- Quantification of crystallinity, texture, twinning, grain size and strain.
- Pattern indexing, lattice parameters determination, unit cell refinement, atomic positions, bonds distances and angles.
- Pattern simulation and structure refinement (Rietveld analysis).

Pattern treatment:
* Peak profile fit
* Search / Match
* Pattern Indexing
* Cell Refinement
* Pattern Simulation
* RIR w% quantification
* Rietveld structure refinement

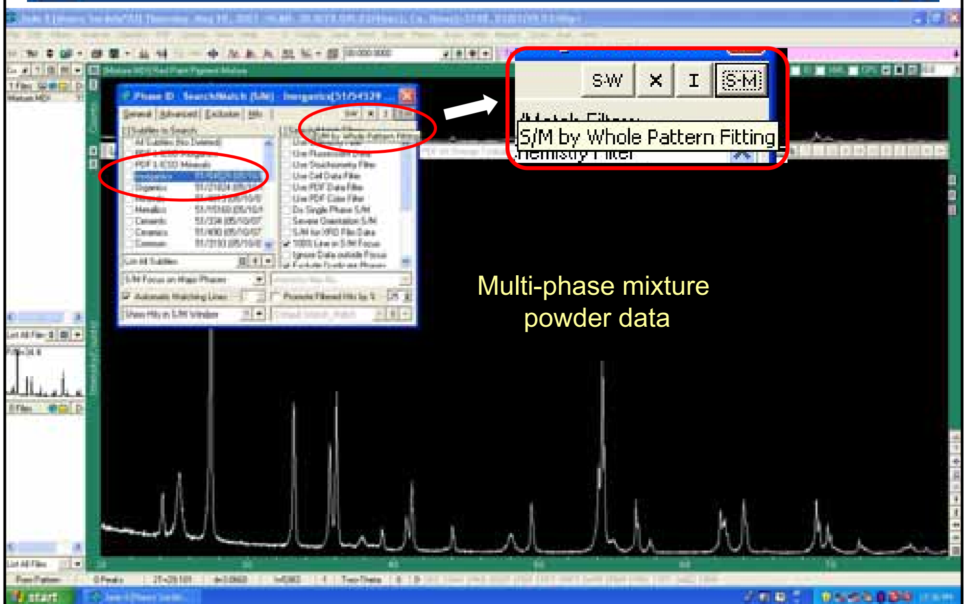
Results obtained from the measured pattern:

Search / Match:	$LiNi_{0.7}Co_{0.3}O_2$, major (minor: C graphite)
Structure:	Hexagonal R-3m (166) <1/3,2/3,2/3>, Z=1, hR4
Cell (Rietveld refinement):	a= 2.86285 Å; b= 2.86285 Å; c= 14.17281 Å α= 90° β= 90° γ= 120° Unit cell volume: 100.6 (Å)^3 Density: 4.8383 g cm^{-3} Linear Absorption Coefficient: 385.5 cm^{-1}
Average bond distances (Rietveld):	Ni-O: 1.9570 Å, Ni-Li: 2.8729 Å, Ni-Ni: 2.8629 Å, Co-O: 1.9570 Å, Co-Ni: 2.8629 Å, Co-Li: 2.8729 Å Li-O: 2.1117 Å, Li-Li: 2.8629 Å, O-O: 2.7983 Å
Quantitative analysis:	82.9 w% $LiNi_{0.7}Co_{0.3}O_2$, 17.1 w% C
Crystallite size:	> 1000 Å
Strain:	0.05%



Data: Sardela, UIUC
Sample: Abraham et al, ANL

New: S/M assisted by whole pattern fitting



Software: MDI's Jade + 8.0

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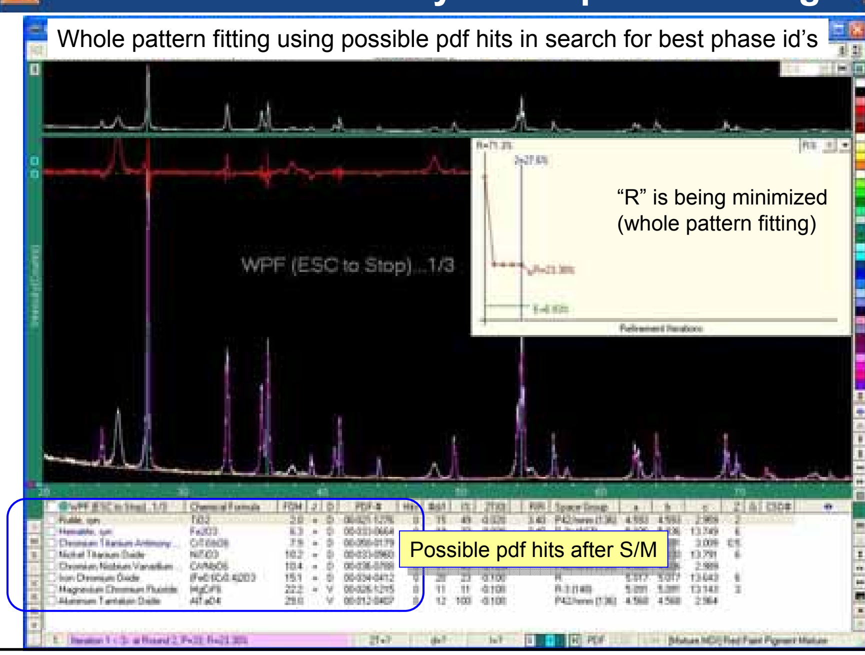
New: S/M assisted by whole pattern fitting



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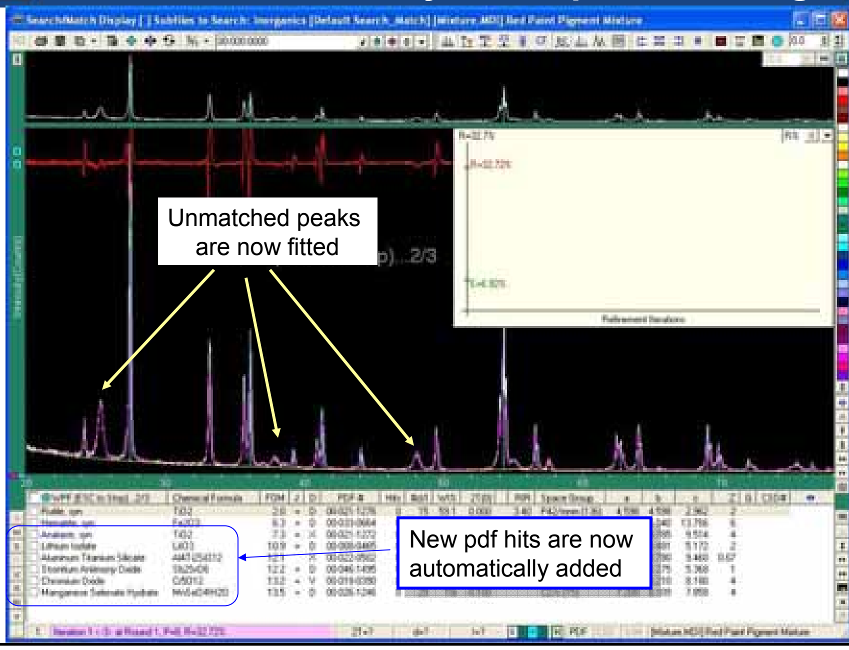
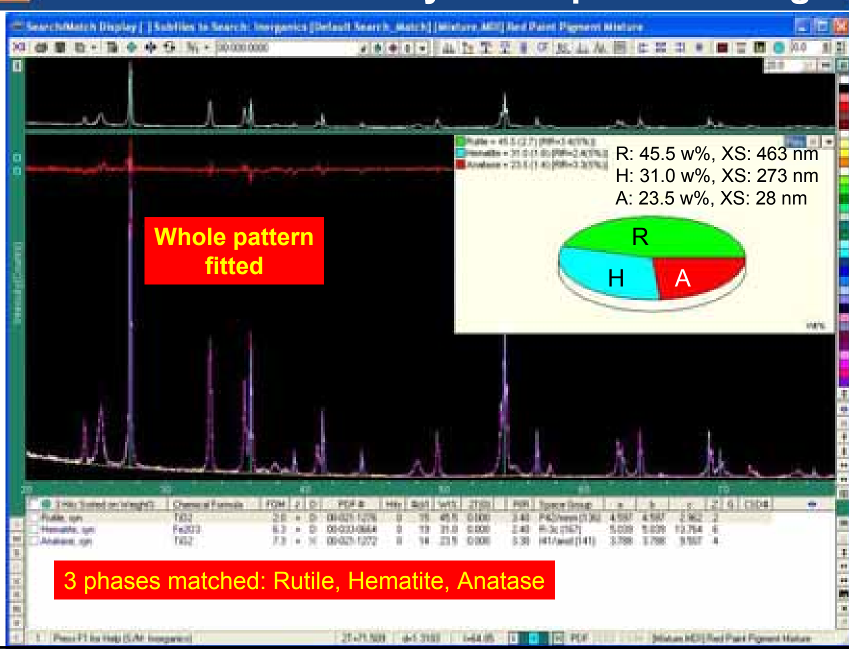
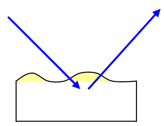


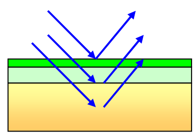
New: S/M assisted by whole pattern fitting



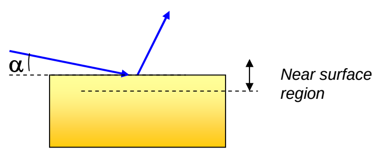
X-ray parallel beam methods



Rough, irregular surfaces



Film / Substrate systems



Glancing / grazing angle applications.
Phase, stress gradients (depth profiles)

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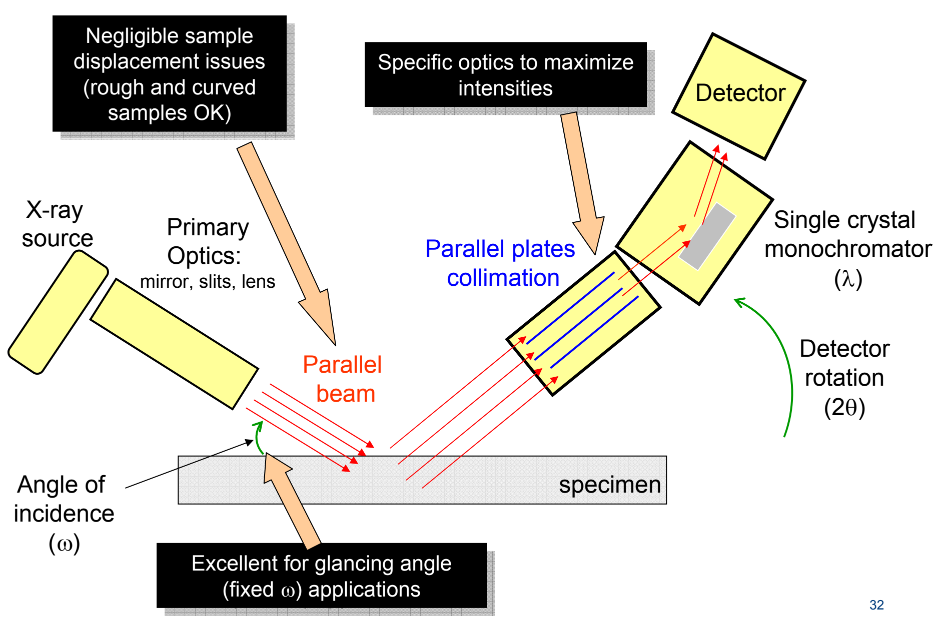
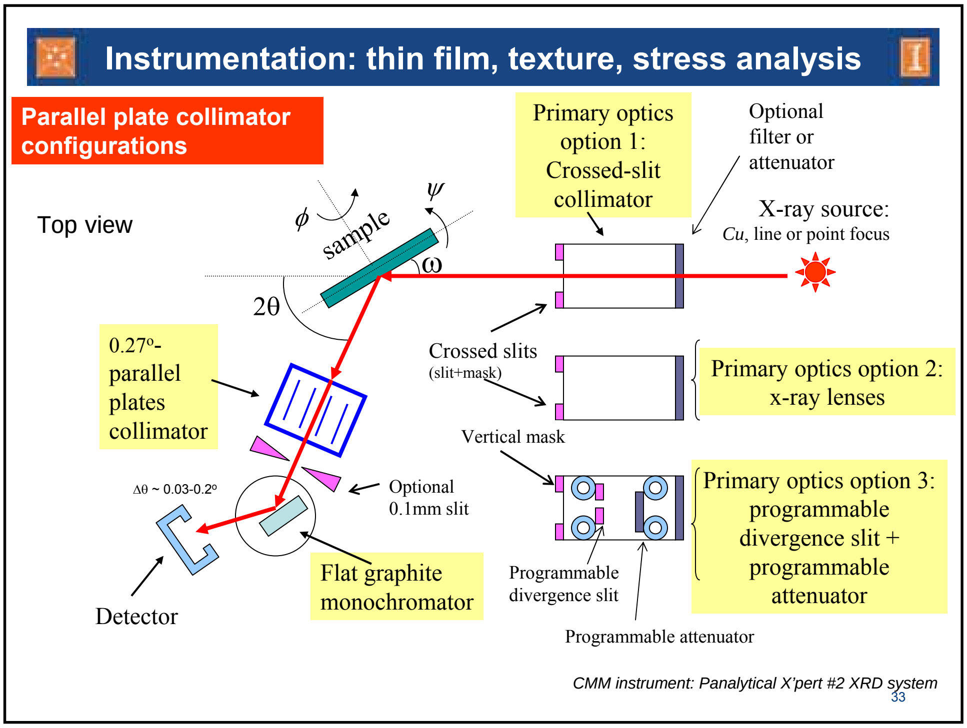
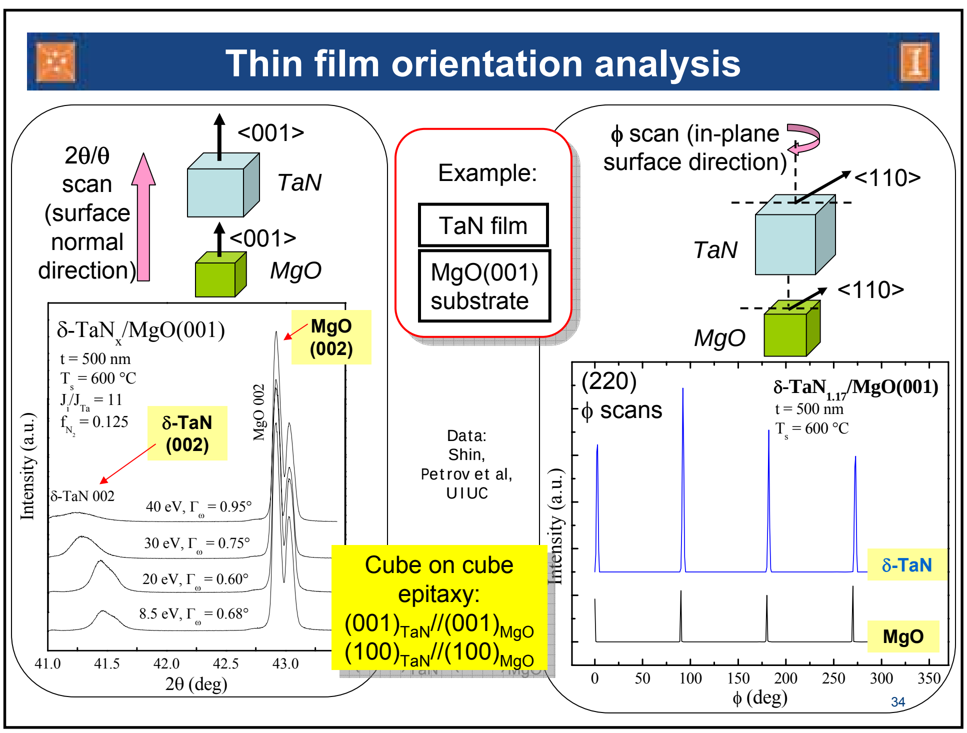
Parallel beam configuration

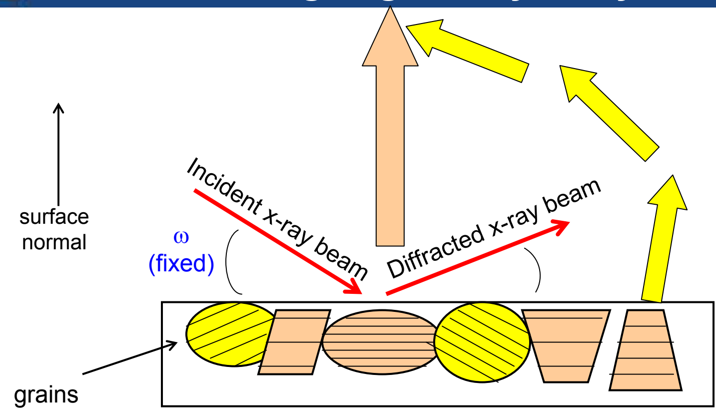


32


Instrumentation: thin film, texture, stress analysis
Parallel plate collimator configurations
Top view
Primary optics option 1: Crossed-slit collimator
Optional filter or attenuator
X-ray source:
Cu, line or point focus
sample
ω
2θ
0.27°-parallel plates collimator
Crossed slits
(slit+mask)
Primary optics option 2: x-ray lenses
Vertical mask
Δθ ~ 0.03-0.2°
Optional 0.1mm slit
Primary optics option 3: programmable divergence slit + programmable attenuator
Flat graphite monochromator
Programmable divergence slit
Detector
Programmable attenuator
CMM instrument: Panalytical X'pert #2 XRD system
33


Thin film orientation analysis
2θ/θ scan (surface normal direction)
<001>
TaN
<001>
MgO
δ-TaNx/MgO(001)
t = 500 nm
Ts = 600 °C
Ji/JTa = 11
fN2 = 0.125
MgO (002)
δ-TaN (002)
MgO 002
δ-TaN 002
40 eV, Γω = 0.95°
30 eV, Γω = 0.75°
20 eV, Γω = 0.60°
8.5 eV, Γω = 0.68°
Intensity (a.u.)
41.0
41.5
42.0
42.5
43.0
2θ (deg)
Example:
TaN film
MgO(001) substrate
Data: Shin, Petrov et al, UIUC
Cube on cube epitaxy:
(001)TaN//(001)MgO
(100)TaN//(100)MgO
ϕ scan (in-plane surface direction)
<110>
TaN
<110>
MgO
(220) ϕ scans
δ-TaN1.17/MgO(001)
t = 500 nm
Ts = 600 °C
δ-TaN
MgO
Intensity (a.u.)
0
50
100
150
200
250
300
350
ϕ (deg)
34

Glancing angle x-ray analysis



: conventional Bragg-Brentano configuration
2θ-ω scans probe only grains aligned parallel to the surface

\+ : parallel-beam grazing incidence configuration
2θ scans probe grains in all directions

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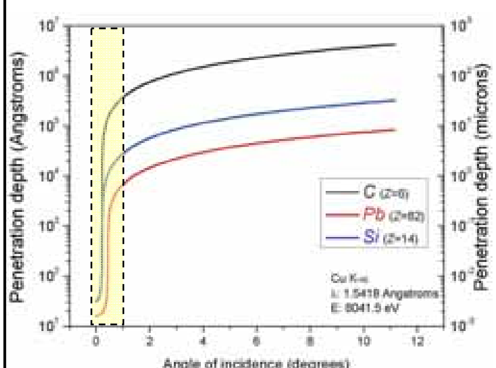
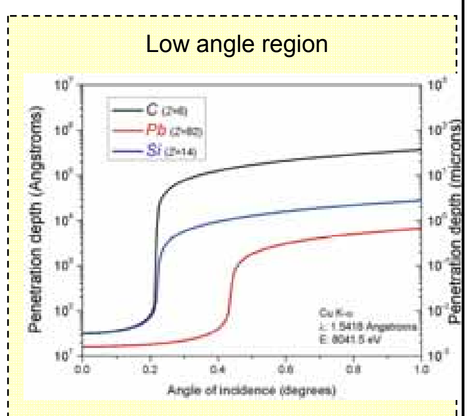
X-ray penetration depth vs. angle of incidence



-Type of radiation
- Angle of incidence
- Material (Z, A, ρ, μ)



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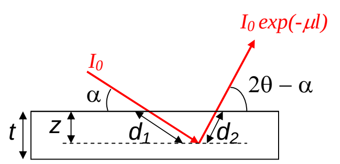
X-ray penetration and information depths



$$l = d_1 + d_2 = \frac{z}{\sin\alpha} + \frac{z}{\sin(2\theta - \alpha)}$$

$$\text{Absorption factor} = \frac{\text{Absorption (sample w/ finite thickness)}}{\text{Absorption (sample w/ infinite thickness)}}$$

For θ/2θ configuration: $A_{\theta 2\theta} = 1 - \exp(-2\mu/\sin\theta)$

For GIXRD configuration: $A_{GIXRD} = 1 - \exp(-\mu t k_\alpha)$

$k_\alpha = 1/\sin\alpha + 1/\sin(2\theta - \alpha)$

t: thickness
z: depth from surface
$\mu = \mu_m \rho$ = linear absorption coefficient
μ_m = mass absorption coefficient (tables)
ρ: mass density
α_c: critical angle
β: $\lambda\mu / 4\pi$ (imaginary part of refractive index)
$\delta = ½(\alpha_c^2)$

(1) Penetration depth $\tau_{1/e}$:
depth for Intensity = I_o/e ~ 37% I_o

$\tau_{1/e} = (\sin\alpha) / \mu$

(2) Penetration depth τ_{63}:
depth for A_{GIXRD} = 1-1/e (~ 63%, $\mu t k_\alpha$=1)

$$\tau_{63} = 1 / \mu k_\alpha = \frac{\sin\alpha \sin(2\theta-\alpha)}{\mu [\sin\alpha + \sin(2\theta-\alpha)]}$$

Bragg peak (2θ) dependent penetration depth!

(3) Information depth τ:
weighted average of sampling depth

$$\tau = \frac{1}{\mu k_\alpha} + \frac{1}{1 - \exp(\mu t k_\alpha)}$$

It will not exceed the thickness value!

(4) Penetration depth $\tau_{1/e}$ for $\alpha \sim \alpha_c$:
Includes correction with refractive index $n = 1 - \delta - i\beta$

$$\tau_{1/e} = \frac{\sqrt{2}\,\lambda}{4\pi} \sqrt{[(\alpha^2 - \alpha_c^2)^2 + \beta^2]^{-1/2} - (\alpha^2 - \alpha_c^2)}$$

Excel spreadsheet calculator available at:
http://www-ssrl.slac.stanford.edu/materialscatter/gixs-calculator.xls

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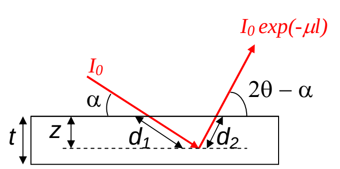
X-ray penetration and information depths



500 nm thick TiO_2 rutile film
(μ = 0.0528 μm^{-1}, Cu Kα radiation)
2θ range: 22 – 60°

Angle of incidence α (°)	Penetration depth $\tau_{1/e}$ (nm)	Information depth τ (nm)
0.9	*298*	*180 - 182*
1.8	*595 (>t!!)*	*212 – 214*
2.7	*892 (>t!!)*	*224 – 226*

From: M. Birkholz, "Thin Film Analysis by X-ray Scattering" Wiley-VCH 2006

(1) Penetration depth $\tau_{1/e}$:
depth for Intensity = I_o/e ~ 37% I_o

$\tau_{1/e} = (\sin\alpha) / \mu$

(2) Penetration depth τ_{63}:
depth for A_{GIXRD} = 1-1/e (~ 63%, $\mu t k_\alpha$=1)

$$\tau_{63} = 1 / \mu k_\alpha = \frac{\sin\alpha \sin(2\theta-\alpha)}{\mu [\sin\alpha + \sin(2\theta-\alpha)]}$$

Bragg peak (2θ) dependent penetration depth!

(3) Information depth τ:
weighted average of sampling depth

$$\tau = \frac{1}{\mu k_\alpha} + \frac{1}{1 - \exp(\mu t k_\alpha)}$$

It will not exceed the thickness value!

(4) Penetration depth $\tau_{1/e}$ for $\alpha \sim \alpha_c$:
Includes correction with refractive index $n = 1 - \delta - i\beta$

$$\tau_{1/e} = \frac{\sqrt{2}\,\lambda}{4\pi} \sqrt{[(\alpha^2 - \alpha_c^2)^2 + \beta^2]^{-1/2} - (\alpha^2 - \alpha_c^2)}$$

Excel spreadsheet calculator available at:
http://www-ssrl.slac.stanford.edu/materialscatter/gixs-calculator.xls

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Grazing angle vs. Bragg-Brentano configurations

Grazing incidence analysis with parallel beam optics

Detect grains in various orientations (including tilted to the sample surface).

Fixed incident angle: constant probed depth in the sample during analysis.

Low angle of incidence:
- high sensitivity to (ultra) thin films;
- avoid artifacts from substrates;
- can be used for "depth profiling" at different incidences.

Slightly lower 2θ resolution: ok for broad peaks.

Thin film collimation optics: enhanced sensitivity to thin films.

Parallel beam: insensitive to sample displacement errors.

Conventional analysis with focusing configuration

Detect only grains with orientation parallel to the sample surface.

Sample probed depth may vary during the analysis.

Lower sensitivity to (ultra) thin films; substrate artifacts are problem.

Very good 2θ resolution and well-known mathematical formalism.

Typical optics are ideal for powder samples and thick films with no preferred orientation.

Focusing configuration: very sensitive to sample displacement errors

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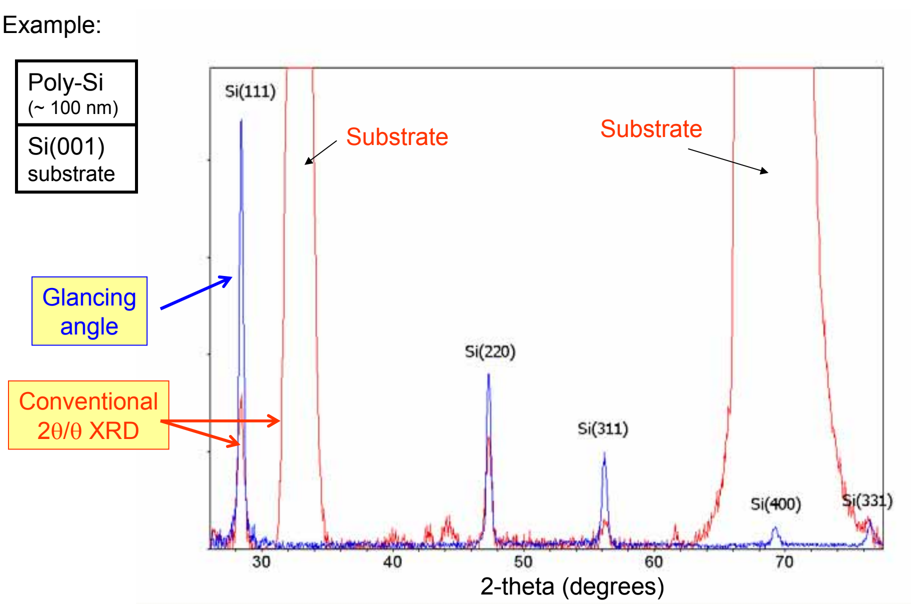
Glancing angle x-ray analysis

Example:

Poly-Si (~ 100 nm)
Si(001) substrate



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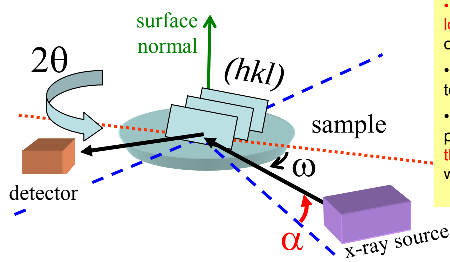
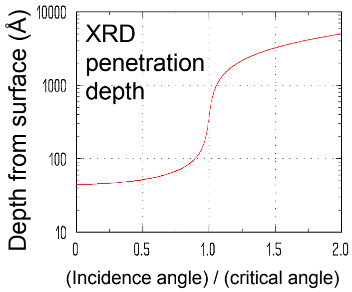
Grazing incidence x-ray analysis

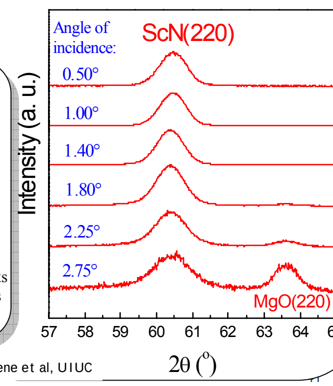


- Grazing incidence X-ray scattering (GIXS) analysis uses low angle of x-ray beam incidence relative to the surface in order to enhance diffraction from very thin layers.
- Analysis of ultra-thin films (<10 nm), nanostructures or topmost surface regions of the material.
- Allows structure determination and quantification of lattice parameters, rocking curve widths and crystallite size along the surface direction (different from conventional XRD, which probes surface normal directions).



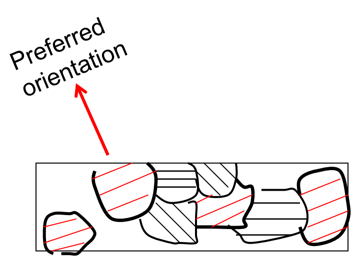
GIXS from a ScN thin film on MgO (001):

- The results allow the determination of in-plane lattice parameter and crystallite size;
- Notice the differences in the relative intensities of the film and substrate peaks at different in-plane angles of incidence α.



Data: Sardela, Shin, Petrov, Greene et al, UIUC

Texture and preferred orientation methods



Texture / Preferred orientation: anisotropy of grain orientation distribution.

Are the grain orientations distributed randomly? Or is there a preferred orientation?

What is the preferred orientation?

What is the % of random grains?

How strong and sharp is the texture?

For film / substrate systems:
what is the crystallographic orientation between the substrate and the layers?

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Determination of preferred orientation

Crystalline grains in a material may be preferentially distributed along one orientation (preferred orientation). This may complicate the analysis using conventional XRD methods derived from powder techniques.

Methods:

1. Compare relative peak height or area obtained from a 2θ/θ scan with the expected relative intensity from a standard (same material) with no preferred orientation (~ powder): Lotgering factors.
2. Use the relative intensity method above combined with March-Dollase preferred orientation corrections to obtain % grains that are more oriented in a specific direction.
3. Use the rocking curve analysis of a strong film diffraction. The width of the rocking curve peak is used as texture parameter.
4. Perform pole figures to determine the presence of grains of a certain orientation in all sample directions.
5. Use multiple pole figures from multiple orientations to obtain Orientation Distribution Functions: % of grain orientation distributions in all wafer directions.

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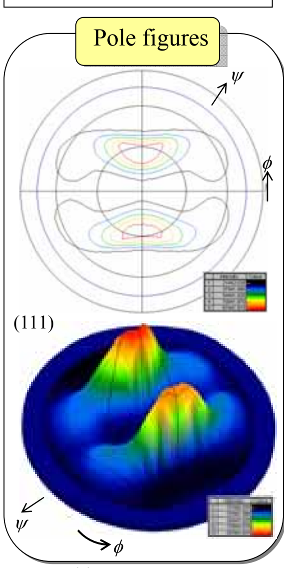
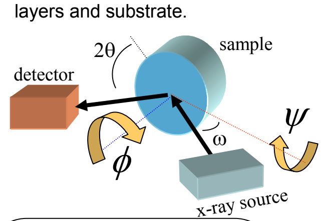
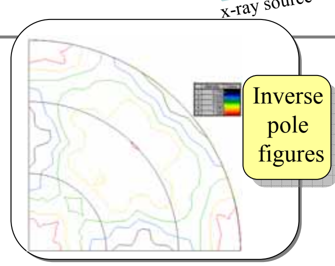
X-ray pole figure analysis of textured materials

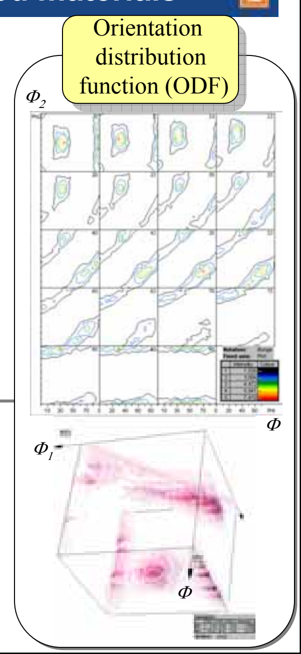
Texture results from a rolled Cu foil



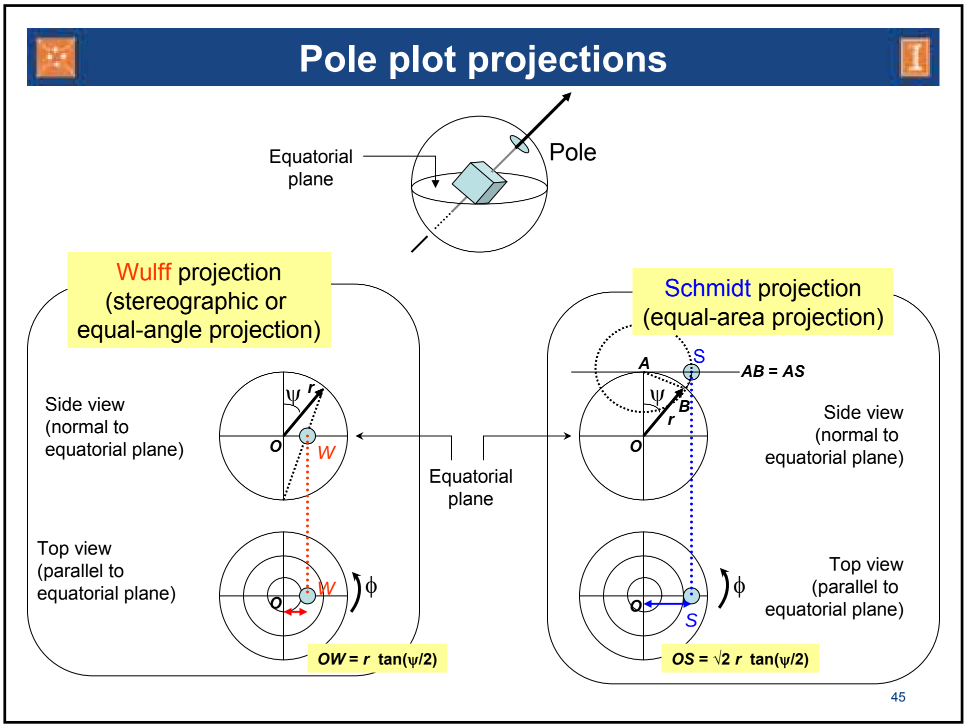
- Texture orientation and quantification.
- Volume fraction of textured grains, twinning and random distributions.
- Texture strength and sharpness.
- Crystallographic orientation.
- Crystallographic relationship between layers and substrate.

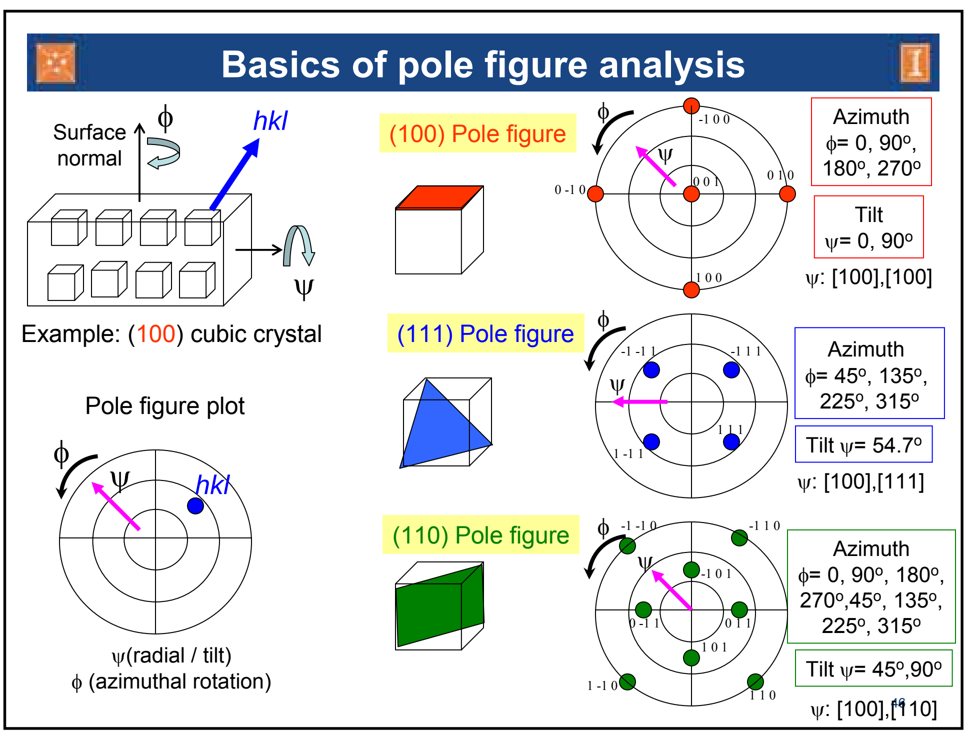




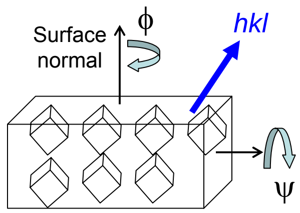


Data: Sardela, UIUC


Pole plot projections
Equatorial plane
Pole
Wulff projection (stereographic or equal-angle projection)
Side view (normal to equatorial plane)
Top view (parallel to equatorial plane)
ψ
r
O
W
ϕ
OW = r tan(ψ/2)
Equatorial plane
Schmidt projection (equal-area projection)
A
S
B
AB = AS
Side view (normal to equatorial plane)
Top view (parallel to equatorial plane)
OS = √2 r tan(ψ/2)
45

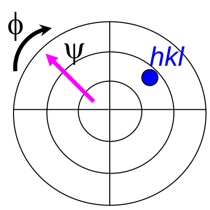

Basics of pole figure analysis
Surface normal
ϕ
hkl
ψ
Example: (100) cubic crystal
Pole figure plot
ψ(radial / tilt)
ϕ (azimuthal rotation)
(100) Pole figure
-1 0 0
0 -1 0
0 0 1
0 1 0
1 0 0
Azimuth ϕ= 0, 90º, 180º, 270º
Tilt ψ= 0, 90º
ψ: [100],[100]
(111) Pole figure
-1 -1 1
-1 1 1
1 1 1
1 -1 1
Azimuth ϕ= 45º, 135º, 225º, 315º
Tilt ψ= 54.7º
ψ: [100],[111]
(110) Pole figure
-1 -1 0
-1 1 0
-1 0 1
0 -1 1
0 1 1
1 0 1
1 -1 0
1 1 0
Azimuth ϕ= 0, 90º, 180º, 270º,45º, 135º, 225º, 315º
Tilt ψ= 45º,90º
ψ: [100],[110]

Basics of pole figure analysis



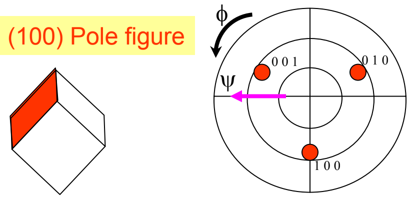
Example: (111) cubic crystal

Pole figure plot



ψ(radial / tilt)
ϕ (azimuthal rotation)

(100) Pole figure

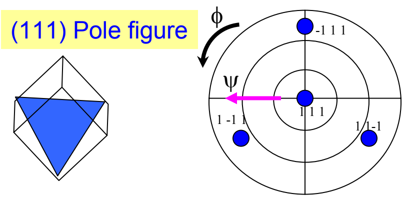


Azimuth
ϕ= 30º, 150º, 270º

Tilt ψ= 54.7º

ψ: [111],[100]

(111) Pole figure

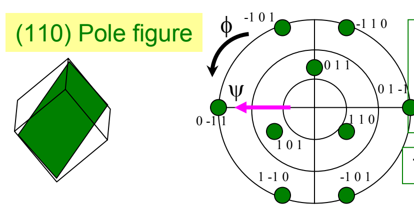


Azimuth
ϕ= 90º, 210º, 330º

Tilt ψ= 0, 70.5º

ψ: [111],[111]

(110) Pole figure



Azimuth
ϕ= 60º, 120º, 240º, 300º, 90º, 210º, 330º.

Tilt ψ= 35.3º, 90º

ψ: [111],[110]

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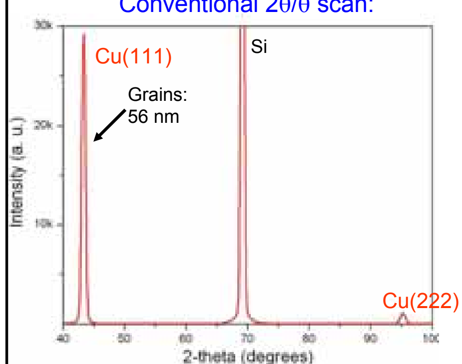
Fiber texture analysis

Example: Cu thin film on Si (001) substrate

Conventional 2θ/θ scan:



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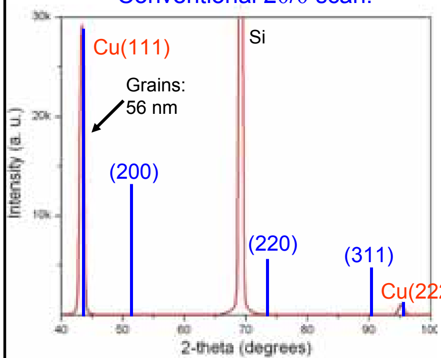
Fiber texture analysis

Example: Cu thin film on Si (001) substrate

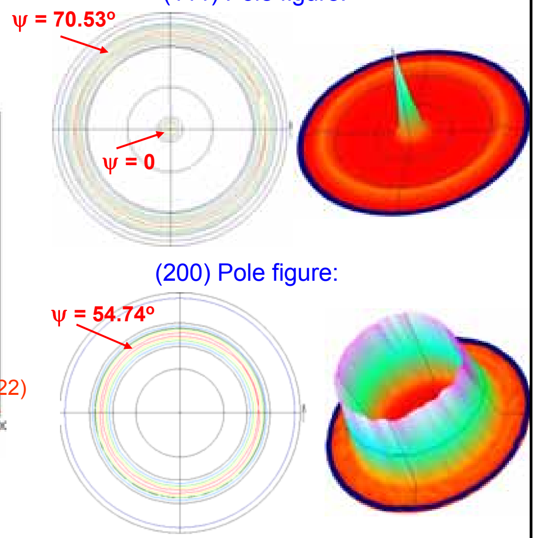
Conventional 2θ/θ scan:



All grains are <111> oriented along the film growth direction.

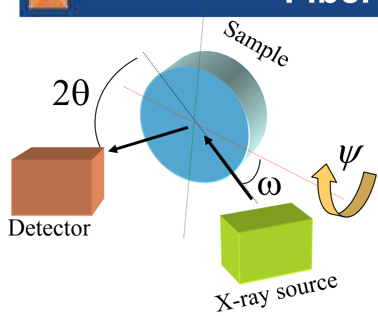
(111) Pole figure:

(200) Pole figure:

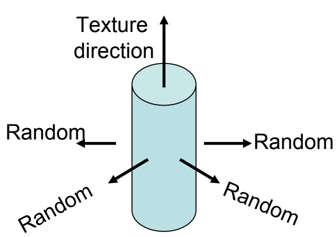


Grains are randomly oriented along the surface

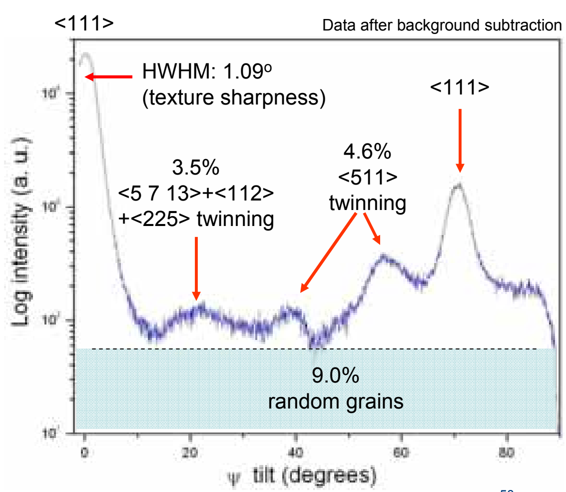
Fiber texture analysis



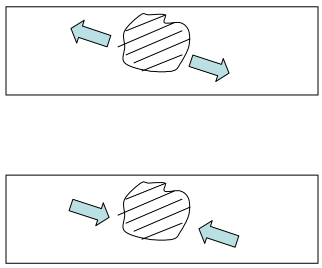
Fiber texture:



(111) Fiber plot
Cu film on Si (001) substrate



Residual stress analysis methods



Residual stress?

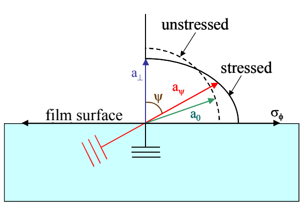
How much? (MPa – GPa)

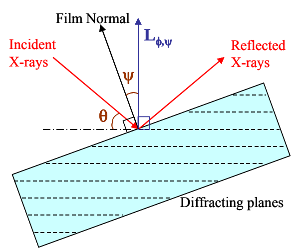
Type?

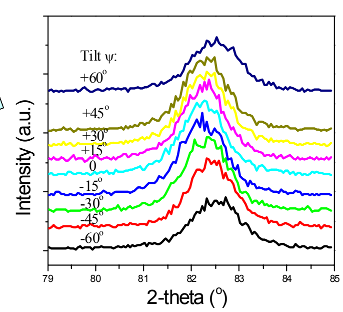
Direction (s)?

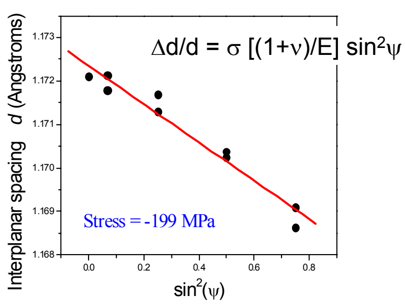
Stress gradients?

X-ray analysis of residual stress







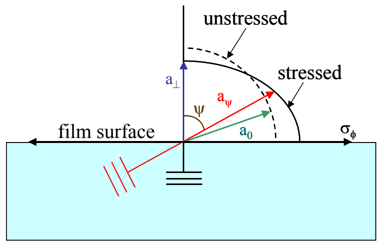


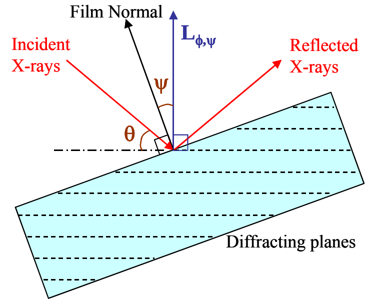
Stress results from a steel sample

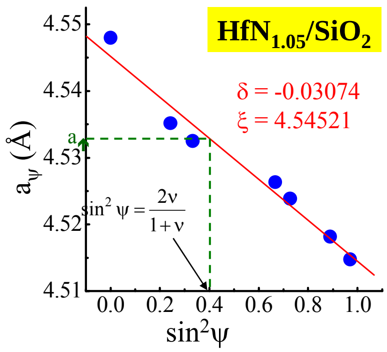
- Quantification of residual stress.
- Compressive (-) and tensile (+) stress.
- Crystallographic orientation of stress.
- $\mathrm{Sin}^2\psi$ and RIM methods.
- ψ and ω scan methods.
- New glancing angle method (texture).
- Determination of stress tensor.
- Requires crystallinity (no amorphous).

Data: Sangid et al, UIUC

Residual stress analysis: the sin$^2\psi$ method







$$\varepsilon_\psi = \frac{a_\psi - a_o}{a_o} = \frac{1+\nu}{E}\sigma_\phi \sin^2\psi - \frac{2\nu}{E}\sigma_\phi$$

$$a_\psi = \frac{a_o\sigma_\phi}{E}\left[(1+\nu)\sin^2\psi - 2\nu\right] + a_o = \delta\sin^2\psi + \xi$$

$$a_o = \xi + \frac{2\delta\nu}{1+\nu} = \mathbf{4.533\ \text{Å}}$$

$$\sigma_\phi = \frac{E\delta}{2\delta\nu + (1+\nu)\xi} = \mathbf{-1.47\ GPa}\ \textbf{(Compressive)}$$

Data: Petrov et al, UIUC

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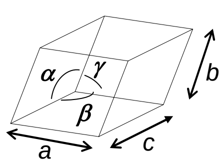
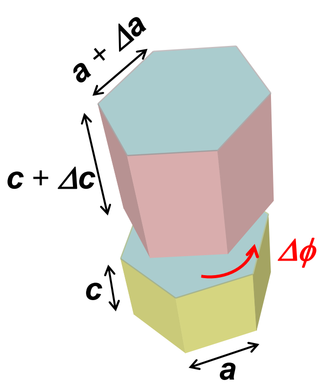
High resolution XRD methods



Single crystals:

Accurate measurements of ***a, b, c, α, β, γ***

Detailed peak shapes: defects, mosaicity.



Film / substrate epitaxial systems:

Measure small variations Δa, Δc,… (~ 10^{-5}).

Measure layer tilts $\Delta\phi$, …

Detailed peak shapes: defects, strain, mosaicity.

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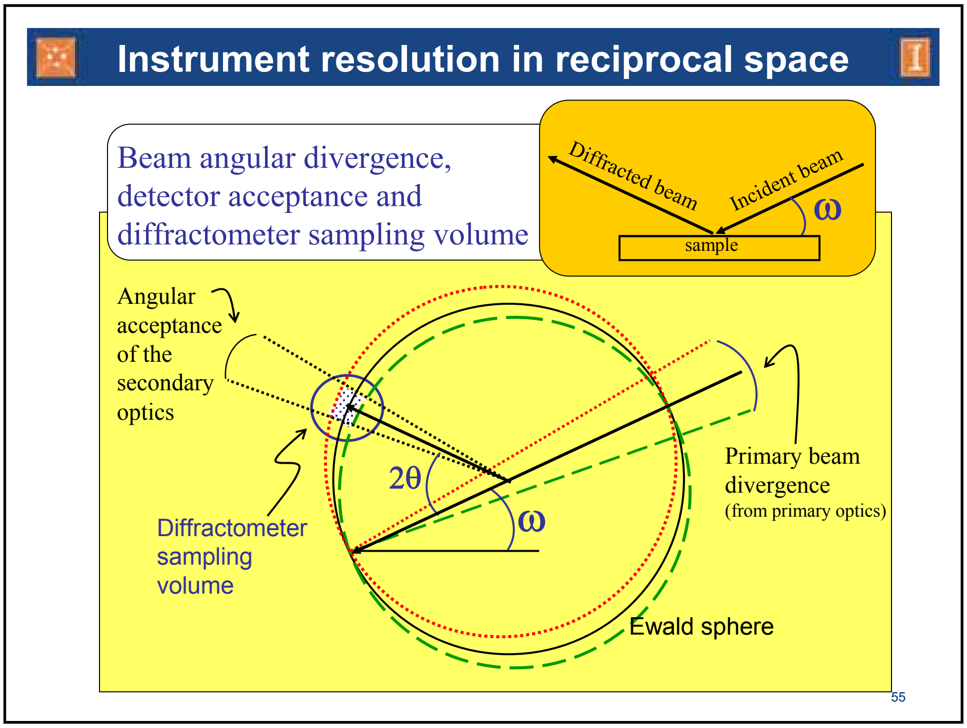
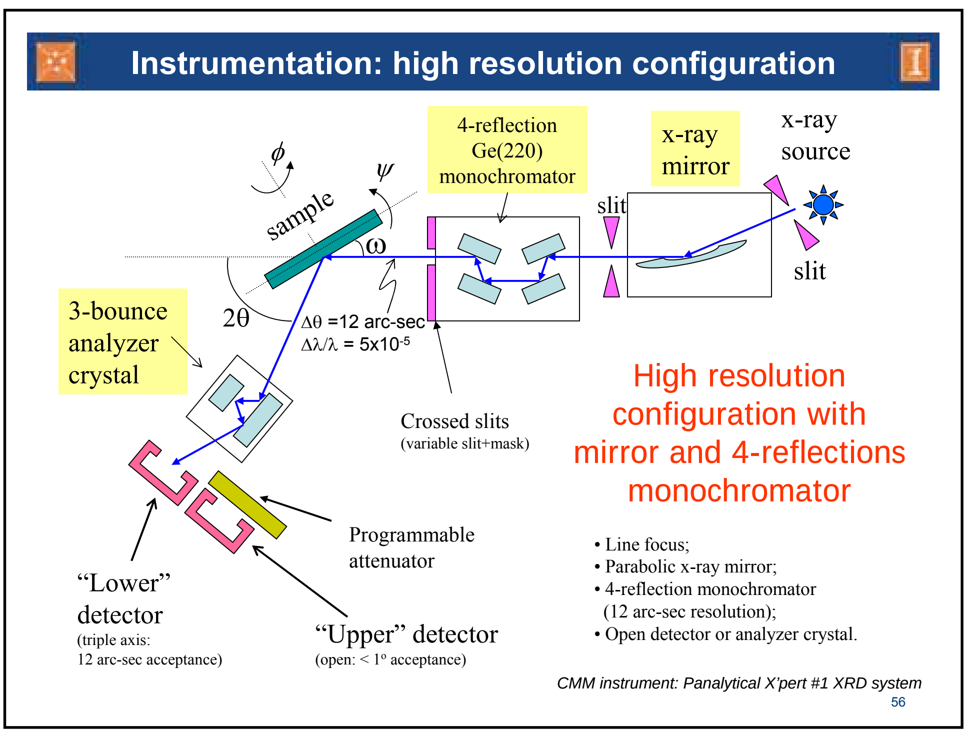

Instrument resolution in reciprocal space
Beam angular divergence, detector acceptance and diffractometer sampling volume
Diffracted beam
Incident beam
ω
sample
Angular acceptance of the secondary optics
2θ
ω
Diffractometer sampling volume
Primary beam divergence
(from primary optics)
Ewald sphere
55


Instrumentation: high resolution configuration
4-reflection Ge(220) monochromator
x-ray mirror
x-ray source
ϕ
ψ
sample
ω
slit
slit
3-bounce analyzer crystal
2θ
Δθ =12 arc-sec
Δλ/λ = 5x10-5
Crossed slits
(variable slit+mask)
High resolution configuration with mirror and 4-reflections monochromator
Programmable attenuator
"Lower" detector
(triple axis:
12 arc-sec acceptance)
"Upper" detector
(open: < 1° acceptance)
• Line focus;
• Parabolic x-ray mirror;
• 4-reflection monochromator
(12 arc-sec resolution);
• Open detector or analyzer crystal.
CMM instrument: Panalytical X'pert #1 XRD system
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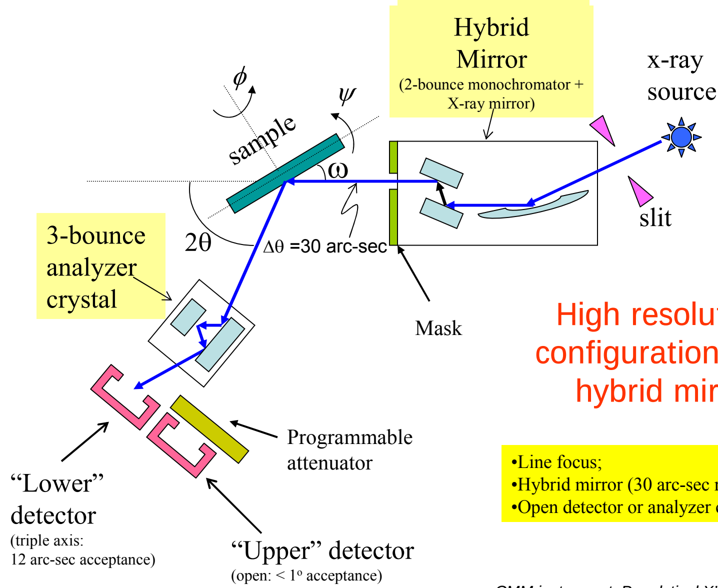
Instrumentation: high-resolution configuration



High resolution configuration with hybrid mirror

- Line focus;
- Hybrid mirror (30 arc-sec resolution);
- Open detector or analyzer crystal.

CMM instrument: Panalytical X'pert #2 XRD system

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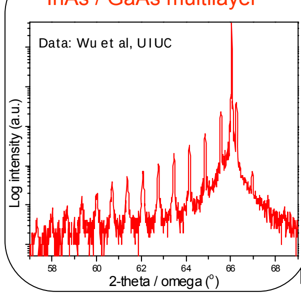
High resolution x-ray analysis

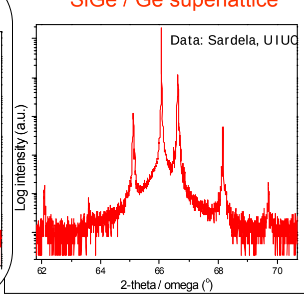
- High resolution methods using multi-reflection monochromator and possibly analyzer crystal ($\Delta\theta$ = 0.003°).
- Sensitive to lattice distortions within 10^{-5}.
- Rocking curve analysis.
- Film thickness measurements.
- Strain relaxation and lattice parameter measurements.
- Determination of alloy composition and superlattice periods.
- Dynamical scattering simulation to determine composition variations and interface smearing in heterostructures.

Single crystals
Epitaxial films
Heterostructures
Superlattices
Quantum dots

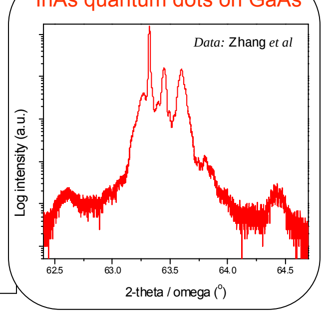
InAs / GaAs multilayer



SiGe / Ge superlattice



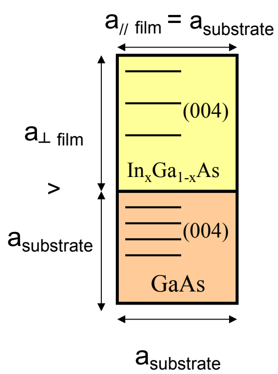
InAs quantum dots on GaAs



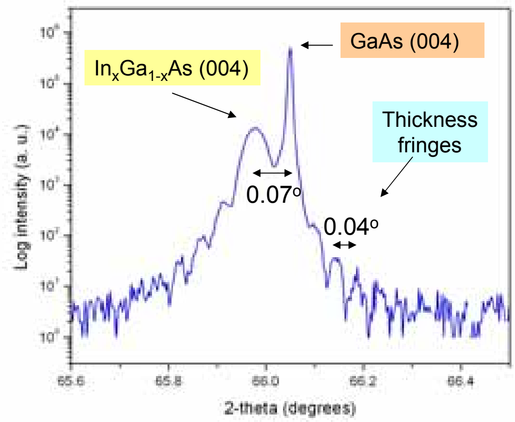
High resolution x-ray analysis

Example: strained $In_xGa_{1-x}As$ on GaAs (001) substrate

Lattice structure



High resolution 2θ/θ scan near GaAs(004)



$$\frac{\Delta a}{a} = \frac{\sin\theta(\text{substrate})}{\sin\theta(\text{film})} - 1 \qquad \text{Thickness} = \frac{\lambda}{2\,\Delta\theta\,\cos\theta}$$

Data: Sardela
Sample: Highland, Cahill, Coleman et at, UIUC

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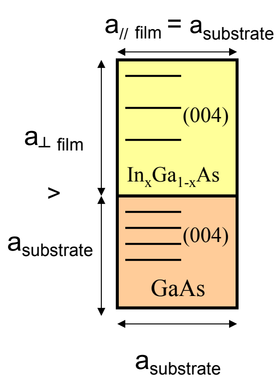
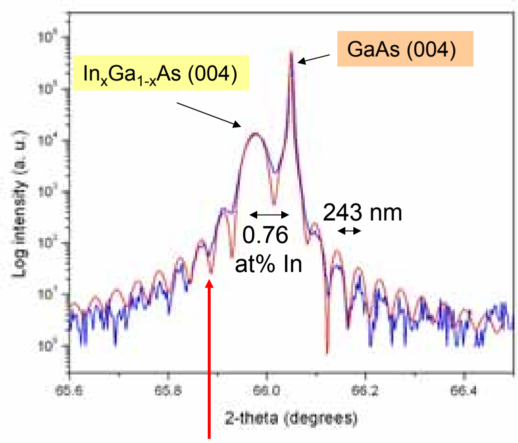
High resolution x-ray analysis

Example: strained $In_xGa_{1-x}As$ on GaAs (001) substrate

Lattice structure



High resolution 2θ/θ scan near GaAs(004) and dynamical scattering simulation



Takagi-Taupin dynamical scattering simulation

Data: Sardela
Sample: Highland, Cahill, Coleman et at, UIUC

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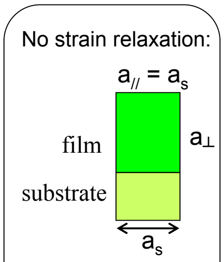
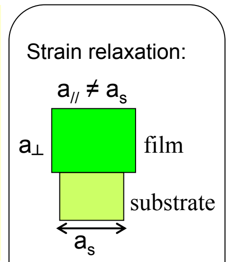
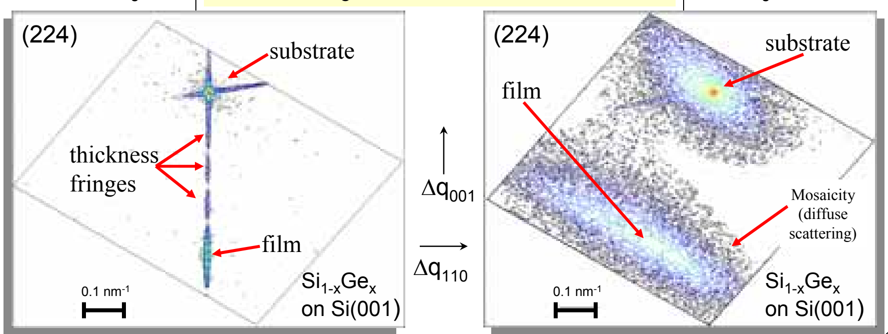
High resolution reciprocal space mapping



- High resolution reciprocal lattice mapping requires multi-reflection monochromator and analyzer crystal in order to separate strain from mosaicity.
- Sensitive to lattice distortions within 10^{-5}.
- Accurate lattice parameter determination (*in* and *out* of plane).
- Determination of strain and composition variations, strain relaxation, mosaic size and rotation, misfit dislocation density, nanostructure dimensions, lattice disorder and diffuse scattering.





Data: Sardela et al

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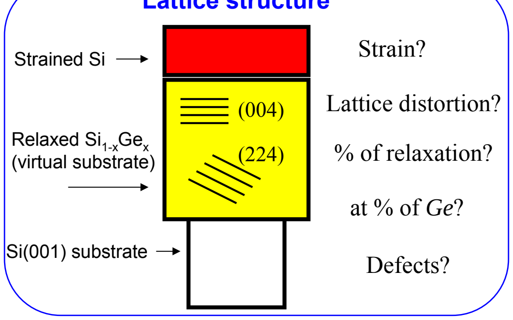
High resolution reciprocal space mapping

Layer structure

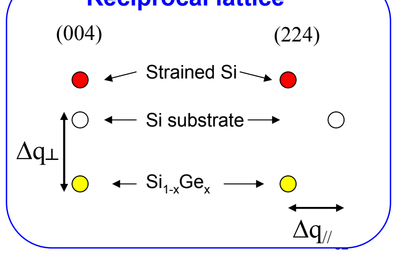
Strained Si (top layer, very thin)
Relaxed $Si_{1-x}Ge_x$ (thick, many microns)
Si(001) substrate

Example: strained Si layer on $Si_{1-x}Ge_x$ / Si substrate

Lattice structure

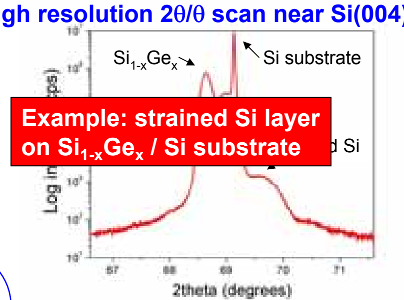


Reciprocal lattice



High resolution reciprocal space mapping

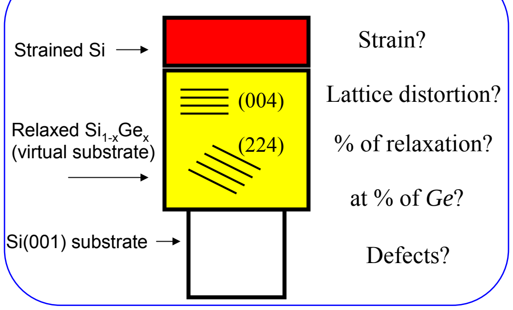
High resolution 2θ/θ scan near Si(004)



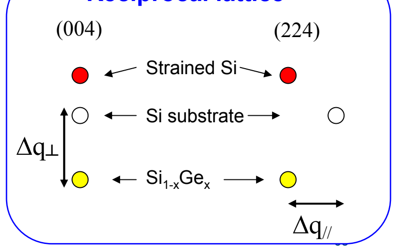
Layer structure

Strained Si (top layer, very thin)
Relaxed $Si_{1-x}Ge_x$ (thick, many microns)
Si(001) substrate

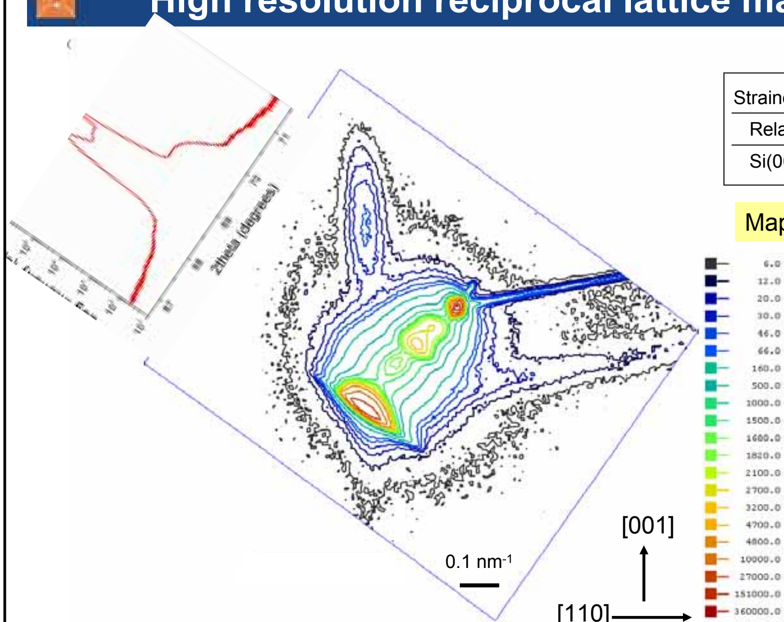
Lattice structure



Reciprocal lattice



High resolution reciprocal lattice map



Strained Si (top layer)
Relaxed $Si_{1-x}Ge_x$
Si(001) substrate

Data: Sardela
Sample: Zuo, UIUC

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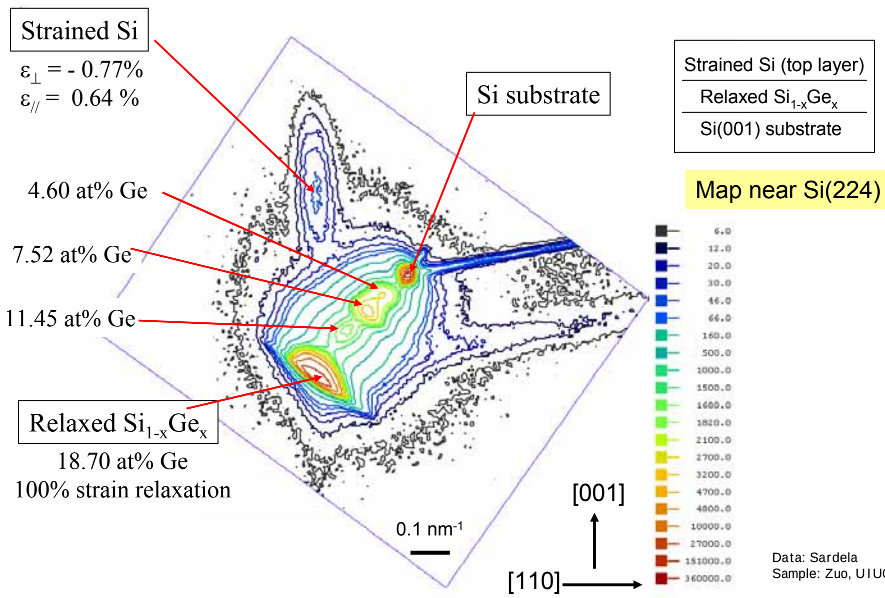
High resolution reciprocal lattice map



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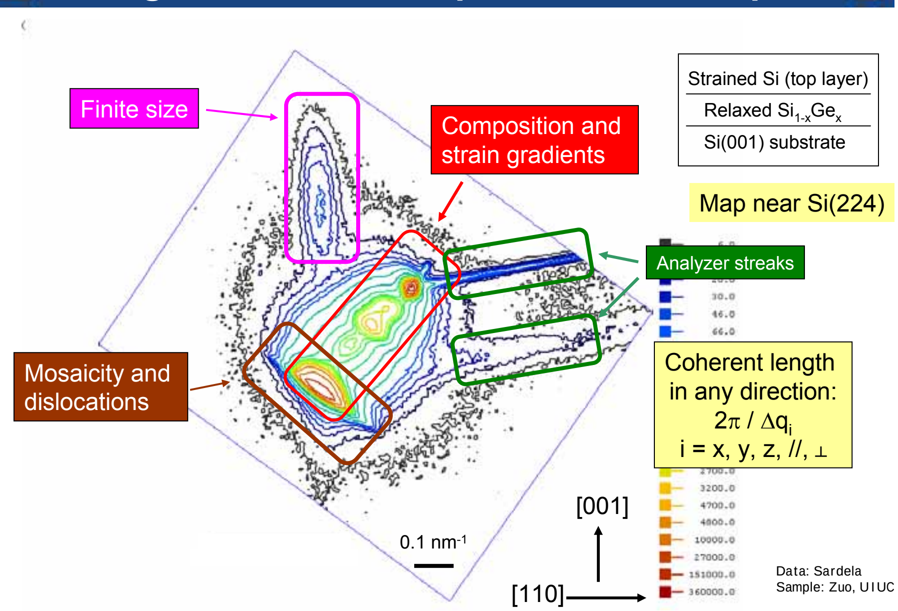
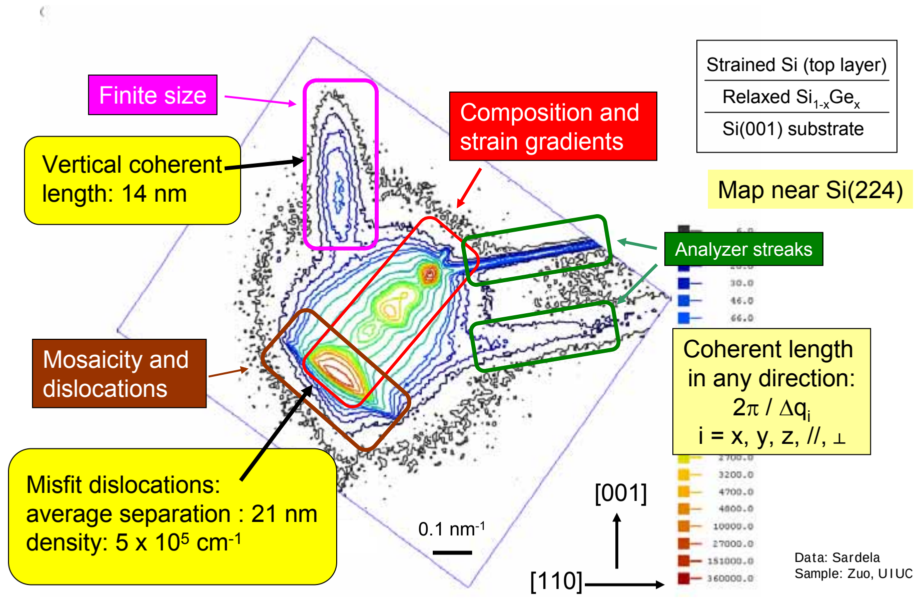
High resolution reciprocal lattice map



66

High resolution reciprocal lattice map



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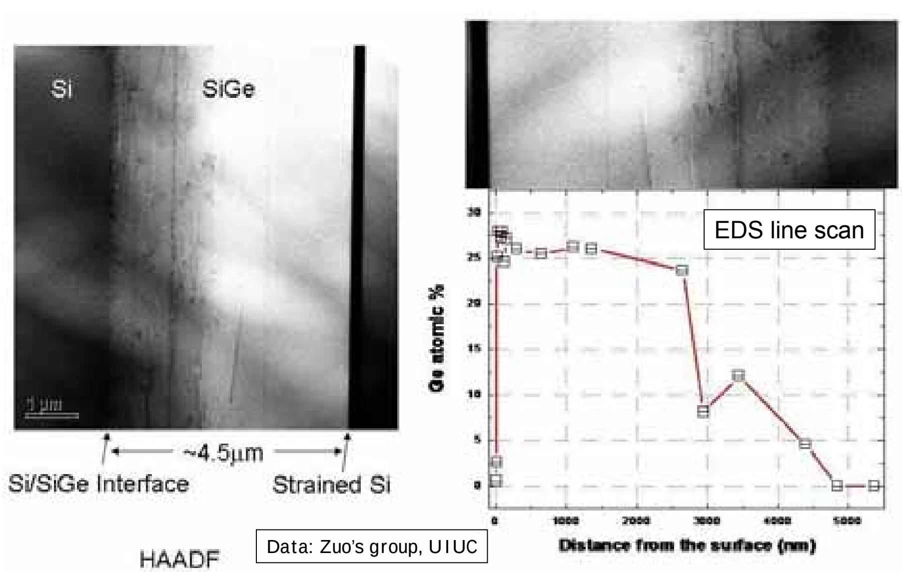
Comparison with TEM



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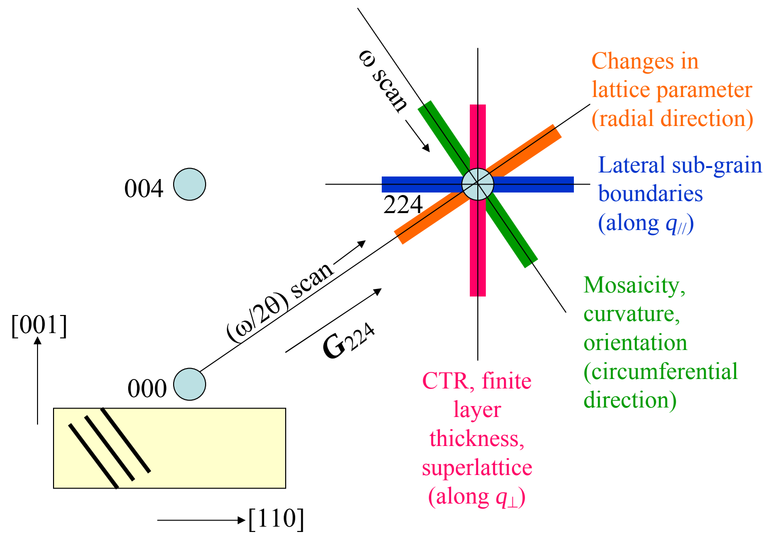
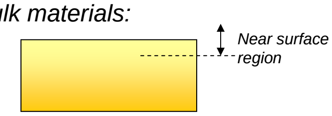
The “shape” of the reciprocal lattice point

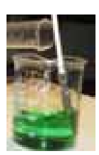


X-ray reflectivity methods

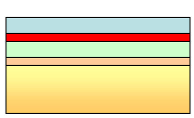
Bulk materials:



Liquids:



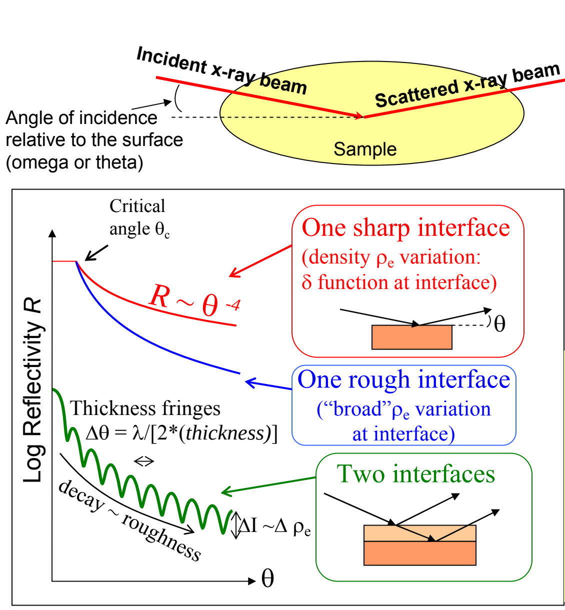
Multilayered systems:

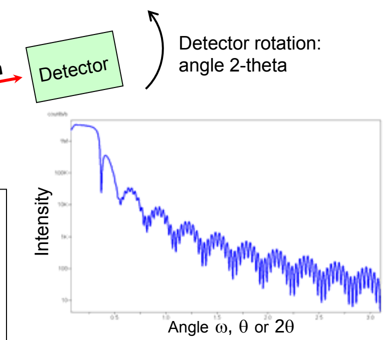


Near surface and interface information on:

Density
Porosity
Roughness
Thickness in films (ultra thin to thick)
Amorphous or crystalline materials

X-ray reflectivity





- Film thickness measurements: 2 – 300 nm.
- Applicable to ultra-thin films, amorphous or crystalline materials, multilayers and liquids.
- Simulation and fitting: determination of interface roughness (rms) at each interface, roughness correlation and film porosity.
- Very sensitive to density variations.
- Determination of critical angle, refractive index and density.

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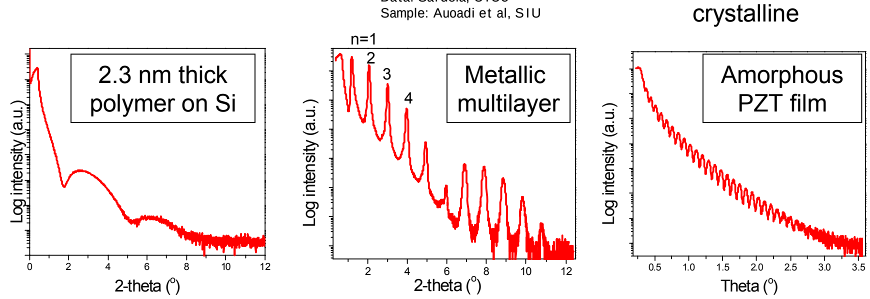
X-ray reflectivity analysis of thin films

Ultra-thin

Complex multilayers

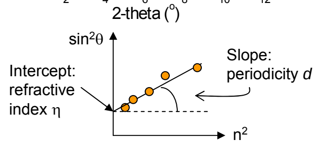
Data: Sardela, UIUC
Sample: Auoadi et al, SIU

Non crystalline



Data: Heitzman et al, UIUC

Data: Mikalsen et al, UIUC



$\sin^2\theta = (\lambda^2/2d)^2\, n^2 - (\eta^2 - 1)$

(modified Bragg's law to include refractive index)

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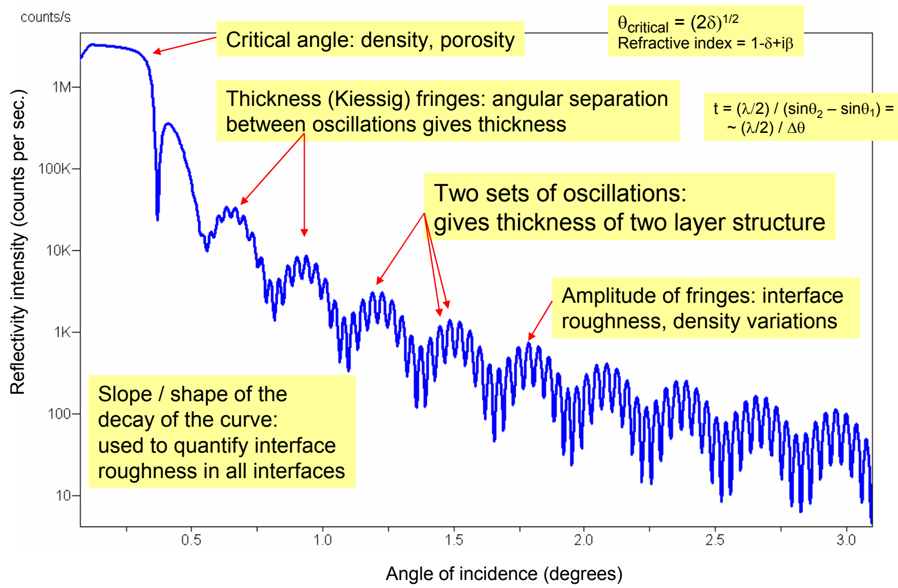
X-ray reflectivity information content



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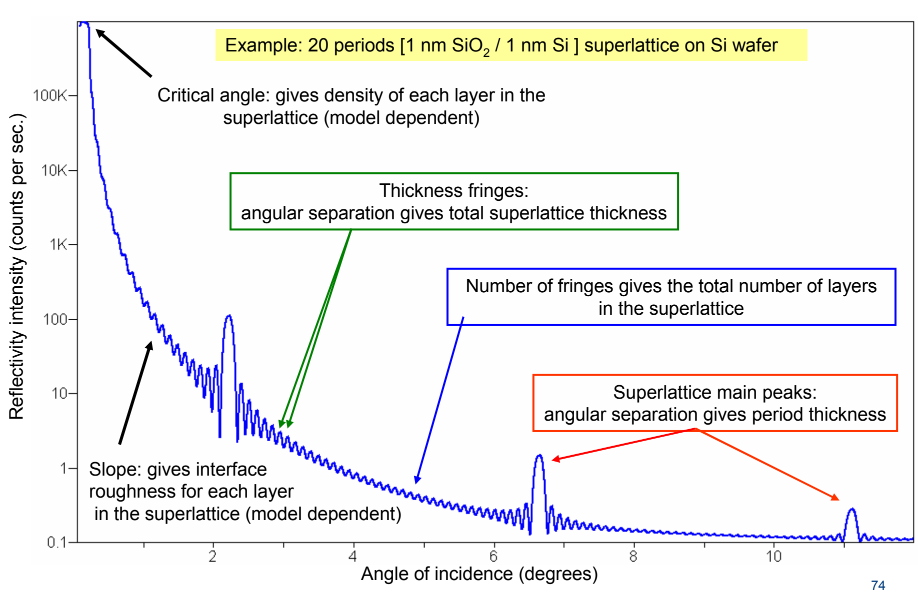
X-ray reflectivity from superlattices



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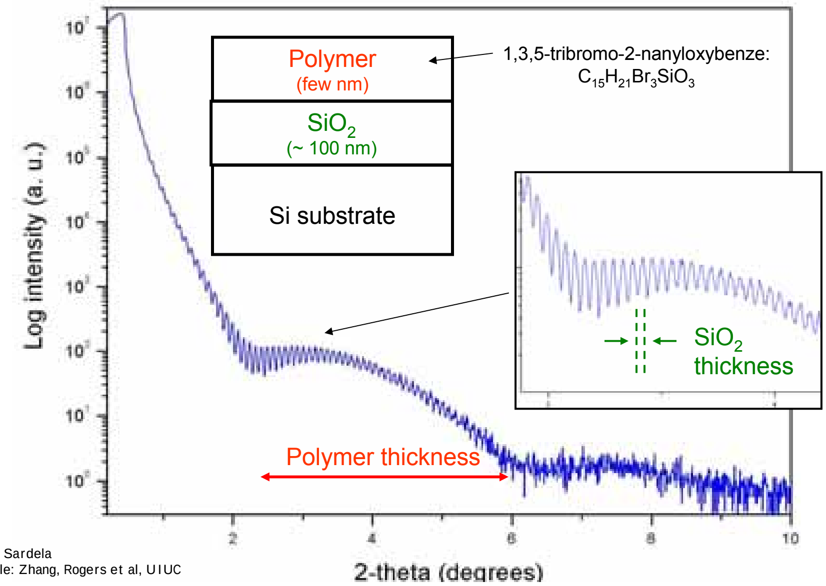
X-ray reflectivity data fitting in ultra-thin films



Data: Sardela
Sample: Zhang, Rogers et al, UIUC

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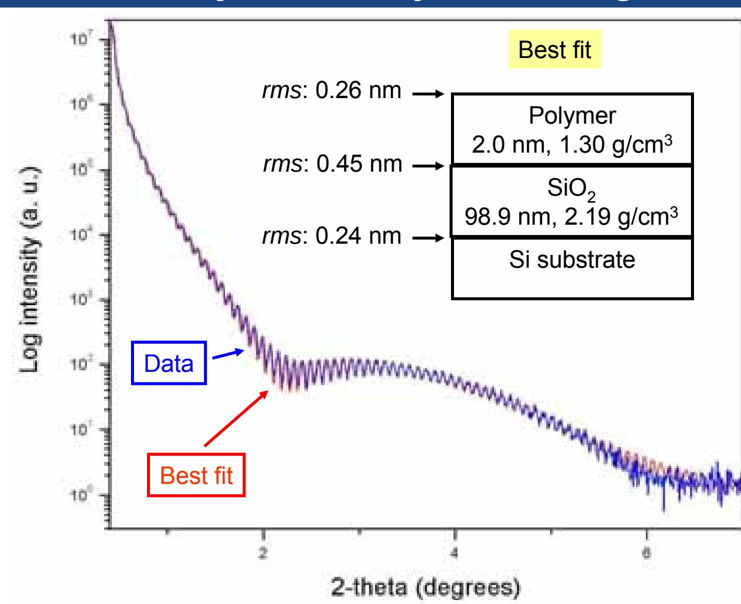
X-ray reflectivity data fitting



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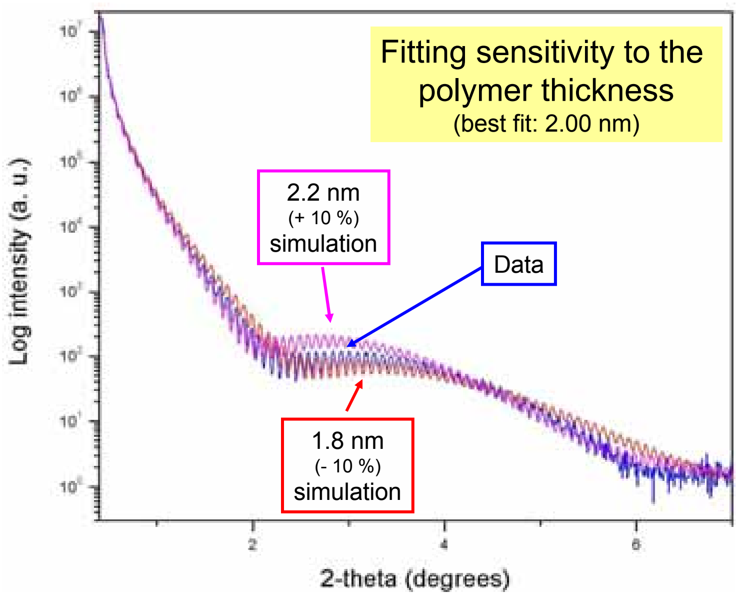
X-ray reflectivity data fitting



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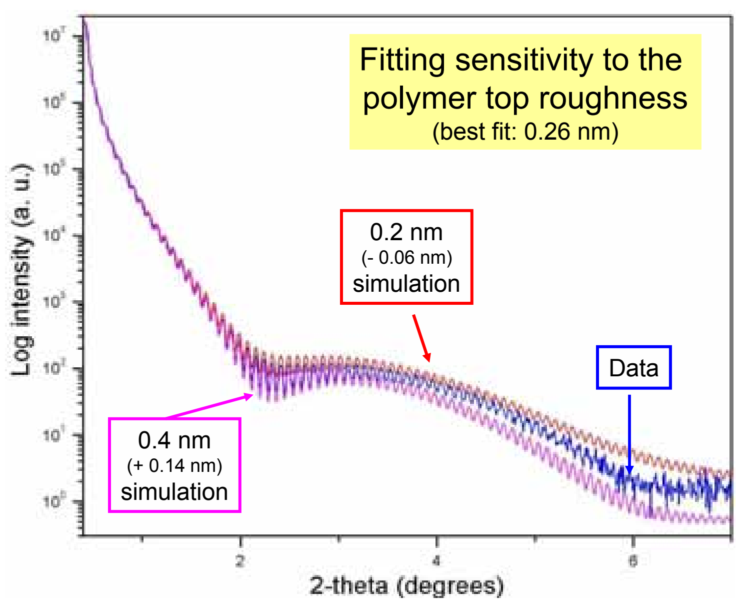
X-ray reflectivity data fitting



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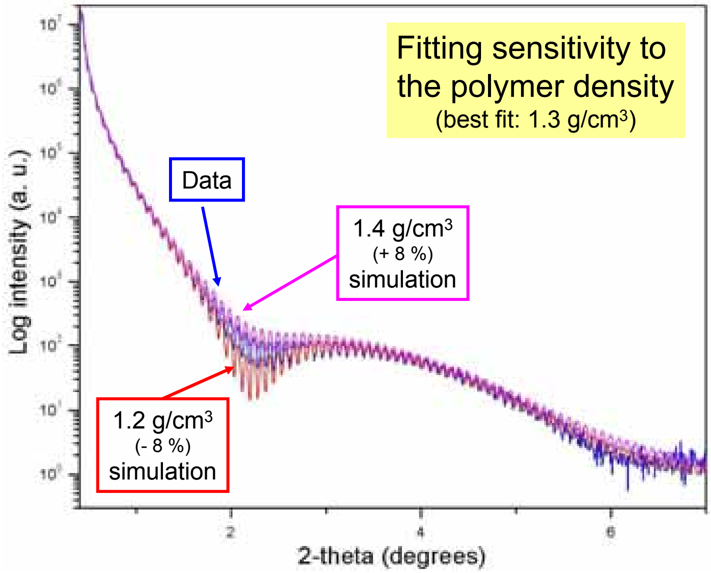
X-ray reflectivity data fitting



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X-ray reflectivity: summary

* Non destructive method
* Applicable to whole wafers (wafer mapping option)
* Fast method (in most cases)
* Do not depend on crystalline quality of the films (can also be used in amorphous layers).

Quantification of:

* Layer thickness in thin films and superlattices: 1 nm ~ 1 μm (± 0.5-1%).
* Layer density and porosity (± 1-2%).
* Interface roughness: 0.1 – 10 nm (model dependent; reproducibility ~ 3%).
* Layer density gradients (variations > 2%).
* Interface roughness correlation in superlattices and multilayers.

Alternative techniques:

* Thickness: optical methods (TEM, SEM) poor contrast issues.
* Density: RBS (issues for ultra thin layers).
* Interface roughness: AFM (surface only – not buried interfaces).

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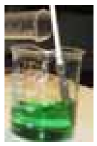
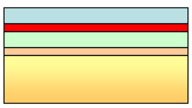
X-ray fluorescence methods

Bulk materials:

Liquids:



Multilayered systems:



Which elements are present down to *ppm* levels?

What is the elemental composition (%) ?

Fast, accurate.

Liquids, solids, amorphous, crystalline materials.

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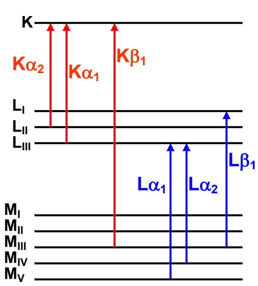
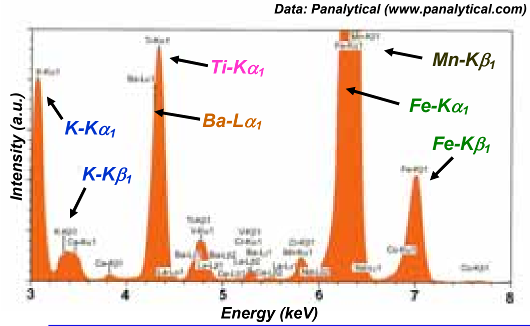
X-ray fluorescence (XRF)



Data: Panalytical (www.panalytical.com)



Transition notation:

IUPAC:
<element><"hole" shell><"originating" shell>
Ex.: *Cr*-KL_{III}

Siegbahn: <element><"hole" shell><α,β,γ, etc.>
Ex.: *Cr*-$K\alpha_1$

(for L_{III} to K transition in Cr)

- **Typical plot: Intensity *vs.* Energy.**
- **Line position associated to element and specific transition**
- **Line positions: fingerprint of the element.**
- **Element identification *Na-U* or Be-U.**
- **Peak assignment uses database and search/match.**
- **Peak area/height: composition (sub ppm to 100%).**
- **Peak overlap requires deconvolution.**
- **Composition determination requires standards, calibration.**
- **Fast data acquisition (seconds to ½ hr).**
- **Solids, liquids, powders, thin films,…**
- **Minimum or no sample preparation required.**

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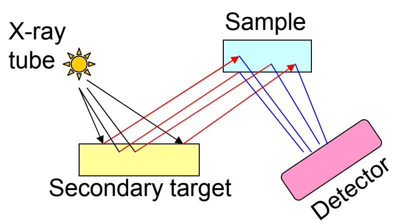
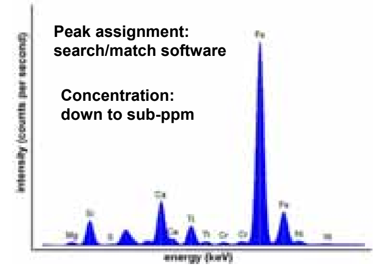
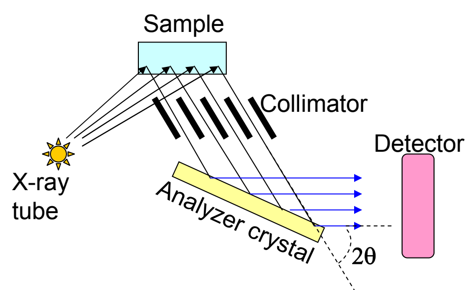
EDXRF and WDXRF

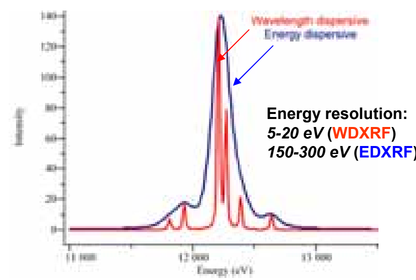
Energy Dispersive XRF (EDXRF)





Wavelength Dispersive XRF (WDXRF)



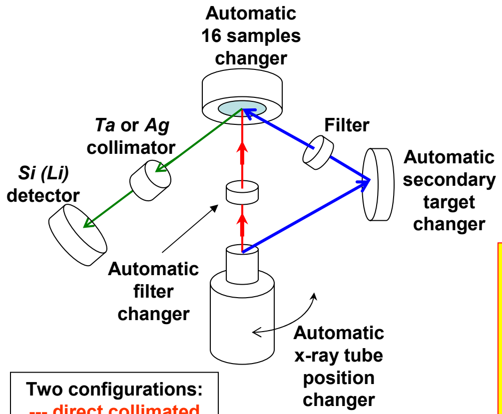


Data: Horiba Jobin Yvon
http://www.jyinc.com/Spain/esdivisions/XRay/tutorial.htm

EDXRF and WDXRF

	EDXRF	WDXRF
Dispersive system	Energy	Wavelength
Raw data	Intensity vs. Energy (keV)	Intensity vs. Detector angle (2θ).
Basic set up	• X-ray tube • (Secondary Target) • Sample • Detector	• X-ray tube • Sample • Collimator (for // beam) • Analyzer Crystal • Detector (+ goniometer)
Elemental Range	*Na – U*	*Be – U*
Detection limit	Good for heavier elements (less optimum for light elements)	Good for all range
Sensitivity	Good for heavier elements (less optimum for light elements)	Moderate for light elements. Good for heavy elements.
Resolution	Good for heavy elements (less optimum for light elements)	Good for light elements (less optimum for heavy elements)
Cost	Moderate	Relatively expensive
Measurement	Simultaneous	• Sequential (moving detector on goniometer) • Simultaneous (fixed detector)
Moving parts	No	Crystal, goniometer
Detector	Solid state detector	• Gas-filled (for *Be – Cu*) • Scintillation (for *Cu – U*).
Qualitative analysis	Peak area	Peak height

Basic EDXRF system configuration



Two configurations:
--- direct collimated
--- secondary target

CMM instrument: Kevex Analyst 770 XRF system

- * X-ray tube: *Rh* (side window) 3.3mA, 60kV.
- * Secondary targets: *Gd*, *Sn*, *Ag*, *Ge*, *Fe*, *Ti*.
- * Filter: *Gd* (6 mil), *Sn* (4 mil), *Ag* (2 mil).
- * Detector collimators: *Ag*, *Ta*.
- * Samples:
  - 16 samples up to 2'' diameter, or
  - 2'' square mounts, or
  - 1.25'' diameter (inserts).
- * Detector: *Si* (*Li*) 30mm^2 active area.
- * Energy resolution ~ 165 eV.

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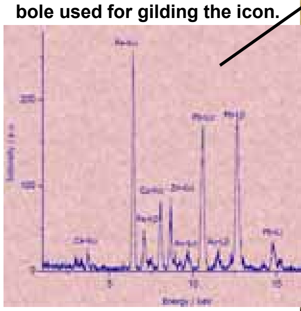
XRF application

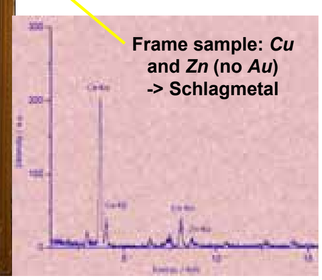
"Spectroscopy investigation of icons painted on canvas"
Lj. Damjanovic *et al*, Belgrade
http://www.srs.ac.uk/scienceandheritage/presentations/Olgica_Marjanovic.pdf

The Virgin and the Child, Unknown author
Museum of the Serbian Orthodox Church, Belgrade

Surface sample:
- Original made of *Au*.
- Restoration with *Au* imitation Schlagmetal (*Cu* and *Zn*).
- *Fe*: natural *Fe*-*Al*-silicate red bole used for gilding the icon.



XRF and IR results show pigments and binders widely used in the XIX century confirming that the work is from late XIX to XX centuries



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X-ray analysis summary

Information contents	• % of crystallinity and amorphous contents • Identification and quantification of phases and mixtures • Chemical information (if crystalline) • Texture (type and strength) and fraction of random grains • Grain / crystallite size (> 2 nm) • Strain (> 10^{-5}) • Stress (type, direction and value) • Relative crystallographic orientation • Lattice constants and unit cell type • Structure determination • Thickness (1.5 nm – 3 mm) • Roughness (including buried interfaces) • Density and porosity • Relative fraction of domains • Defects and dislocation densities • Mosaic tilts and sizes • …
Detection limits	> 0.1 – 0.5 w%
Depth information	Up to 20 – 50 mm (typical) > 10 nm and variable with glancing angle XRD
Lateral resolution	~ a few cm (typical) 10 mm (microdiffraction) – 5 cm
Angular resolution	0.1° 2q (typical powder diffraction) 0.003° 2q (high resolution methods)
Sample requirement	Mostly non destructive Sample sizes from ~ 50 mm to many cm No vacuum compatibility required Solids, liquids, gels

* Semiconductors
* Coatings
* Pharmaceutical
Cements
* Metals
* Ceramics
* Geology
* Archeology
* Biosciences
* Forensics
* Medical applications
* Nanotechnology
* Polymers
* Food science
* Combustion
* Energy
* …

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Comparison with other techniques

	X-ray analysis methods	Other techniques
Sample preparation and vacuum compatibility	o No vacuum compatibility required (except XRF on vacuum). o "Any" sample size (depends on the goniometer size/weight capability). o Rough surfaces acceptable (parallel beam configuration). o No sample preparation required (prep recommended for the detection of unknown phases or elements in XRD/XRF).	o Surface analysis and electron microscopy techniques will require vacuum compatibility and in many cases sample preparation. o Optical techniques will do analysis on air.
Composition and impurity determination and quantification	➢ ~ 0.1 w % (XRF > ppm); may require standards. ➢ XRD: also phase information and % of crystallinity. ➢ Data averaged over large lateral area.	➢ XPS: > 0.01 – 0.1 at % (may require depth profiling). ➢ SIMS: > 1 ppm (requires sputtering depth profiling). ➢ EDS: > 0.1 – 1 w % over small volume 1μm^3. ➢ Little with phase information; averages over small lateral areas (< 100 μm).
Lattice constants	o Better than within 10^{-5}	o TEM: estimates ~ 10^{-3}
Thickness in thin films	➢ HR-XRD or XRR: direct measurement (no modeling for single or bi-layers). ➢ Requires flat interfaces.	➢ RBS: > 10 nm (requires modeling). ➢ Ellipsometry: requires modeling. ➢ TEM: requires visual contrast between layers.
Grain size	o Measures Crystallite Size. o Typically ~ 1-2 nm – microns, requires size/strain assumptions/ modeling. o "Volume average" size.	o SEM: grain size distribution averaged over small area. o TEM/SEM: "number average" size.

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Comparison with other techniques

	X-ray analysis methods	Other techniques
Texture	o Type and distribution averaged over large sample volume.	o EBSD: within grain sizes dimensions, better sensitivity at the surface.
Residual Stress	➢ 10 MPa, averaged over large sample volume (large number of grains). ➢ Needs crystallinity. ➢ Measures strain and obtains stress from Hooke's law. ➢ Averages macro and micro stresses over large area of a layer.	➢ Wafer curvature: No need for crystallinity. Direct measurement of stress, but only interlayer stress between film and substrate (macrostress).
Depth dependent information	➢ Phase, grain sizes, texture and stress "depth profiling" – requires x-ray information depth modeling	➢ Surface analysis depth profiling: compositional depth profiles.
Surface or Interface roughness	➢ XRR: interface roughness 0.01 – 5 nm, including buried interfaces	➢ SPM: top surface only; rsm~ 0.01-100 nm.
Defects	➢ Misfit dislocations (HR-XRD). ➢ Point defects (diffuse scattering with model). ➢ Extended defects (powder XRD with model). ➢ Average over larger sample area (> mm).	➢ TEM: accurate identification of defects and their densities; average over small sample area. Sample preparation may introduce artifacts.
Instrument cost	➢ Portable instruments ~ \$ 60 K. ➢ Average well-equipped: ~ \$ 200 – 300 K. ➢ Top of the line ~ \$ 500 K (including microdiffraction and 2D detectors).	➢ Surface analysis instruments > \$ 500 K. ➢ Electron microscopes ~ \$ 300 K – 1 M. ➢ RBS ~ \$ 2 M. ➢ Raman, ellipsometry > \$ 100 K.

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Amazing x-ray samples

Pieces from Egyptian mummy
Powder from Inca ruins
Corrosion in pipes from IL water supply
Rocks from Mississippi river
Mud from a cadaver's shoes
Dove chocolate
Corn starch
Train tracks
Heavy machinery valves
100 μm micro chips
10 μm superconductor single crystal
Next generation CPU processors
Micro extraction from Dutch paintings
Virus, bacteria, DNA, proteins
Powder from Mars terrain
Gasoline, electrical car battery
Bone implants
Pork tissue

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Amazing x-ray samples

W. C. Roetgen wife's hand (1895)

A. Bertha Roetgen
(1833-1919)

Wedding ring

W.C. Roetgen
(1845-1923)
1st Physics Nobel

It "frightened Bertha terribly" as a "premonition of death"
After Boston Globe 11/6/95

Quick guide to our x-ray analysis instruments (1)

X-ray analysis instrumentation available as user facility in the FS-MRL

Instrument	Set up	Applications
Panalytical X'pert (#1)	• Source: *Cu* Kα1, line or point focus. • High resolution configuration. • 4 or 2 reflection *Ge* monochromator (12 or 30 arc-sec parallel beam). • 3 reflection *Ge* analyzer crystal (12 arc-sec parallel beam). • Eulerian cradle. • Proportional detectors.	• High resolution XRD capabilities. • Rocking curve. • Single crystals, epitaxial systems. • Reciprocal space mapping. • Reflectivity. • Curvature, wafer mapping, miscut, diffuse scattering. • Topography. • Glancing angle. • Parallel beam applications. • Max sample size: 10 cm diameter x 2 cm thick.
Panalytical X'pert (#2)	• Source: *Cu* Kα1+Kα2, line or point focus. • Crossed slit collimator (variable aperture). • X-ray lens. • Programmable divergence slit. • Eulerian Cradle. • Parallel plate collimator and flat graphite monochromator • Programmable receive and scatter slits and graphite monochromator. • Proportional detectors.	•.Phase, size, strain, stress, texture, crystallinity •.Parallel beam applications •.Bragg-Brentano applications •.General thin film analysis •.Glancing/grazing angle •.Max sample size: 10 cm diameter x 2 cm thick

Quick guide to our x-ray analysis instruments (2)

X-ray analysis instrumentation available as user facility in the FS-MRL

Instrument	Set up	Applications
Rigaku D/Max b	• Source: *Cu* Kα1+Kα2, line focus. • Bragg-Brentano focusing configuration. • Theta/2theta goniometer. • Divergence, soller, scatter and receiving slits. • Curved graphite monochromator. • Scintillation detector.	• Phase, size, strain, crystallinity. • Bragg-Brentano applications. • Rietveld analysis. • Mostly for powder, bulks and thin film with small preferred orientation.
Rigaku Laue	• Source: *Mo* point focus. • Four circle sample stage (manual). • Polaroid film camera detection system.	• Single crystal orientation. • Miscut information. • Crystallographic alignment prior to crystal cutting.
Bruker / Siemens D5000 (Fall 2008)	• Source: *Cu* Kα1+Kα2, line focus. • Bragg-Brentano focusing configuration. • Theta / theta goniometer. • Horizontal sample load. • No sample movement required during analysis. • Divergence, scatter and receiving slits. • Scintillation detector.	• Ideal for powder and soft samples (horizontal load). • Phase, size, strain, crystallinity. • Bragg-Brentano applications. • Rietveld analysis. • Mostly for powder, bulks and thin film with small preferred orientation.
Kevex Analyst 700 XRF	•.Source: *Rh* (side window) 3.3mA, 60kV •.6 secondary targets, 2 detector collimators, 3 filters. •.*Si (Li)* solid state detector. •.Energy resolution 165 meV.	•.Elemental identification: *Na – U*. •.Liquids, solids, powder samples. •.Composition: > ppm (some standards available).

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Recommended literature

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"Quantitative x-ray fluorescence analysis theory and application", G.R. Lachance and F. Claisse, Willey, 1995.

Laue methods:
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Two-dimensional XRD (with areal detectors):
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Fundaments of x-ray scattering:
"X-ray diffraction", B.E. Warren, Addison-Wesley, 1962.
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"Elements of x-ray diffraction", B.D. Cullity, Addison-Wesley, 1978.
"Elements of Modern X-ray Physics", J. Als-Nielsen (Author), D. McMorrow, Wiley, 2001.
"Coherent X-Ray Optics (Oxford Series on Synchrotron Radiation)", D. Paganin, Oxford University Press, 2006.

Web resource: www.ccp14.ac.co.uk (free x-ray data analysis programs and tutorials)

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