X-ray diffraction and reflectometry in studies of crystals

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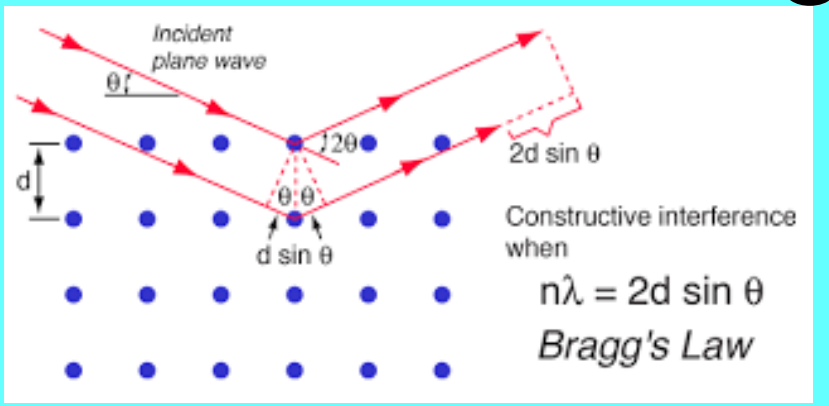
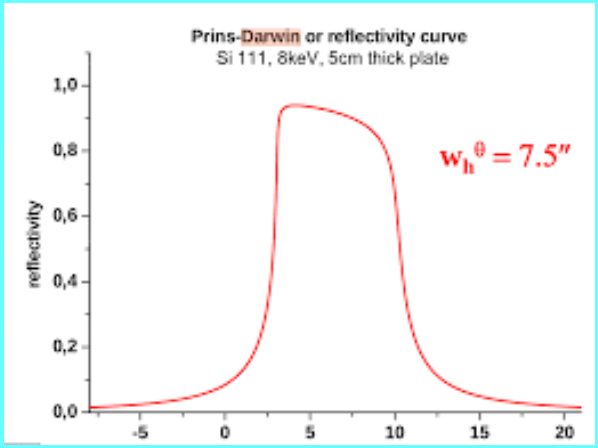
Instytut Wysokich Ciśnień i TopGaN

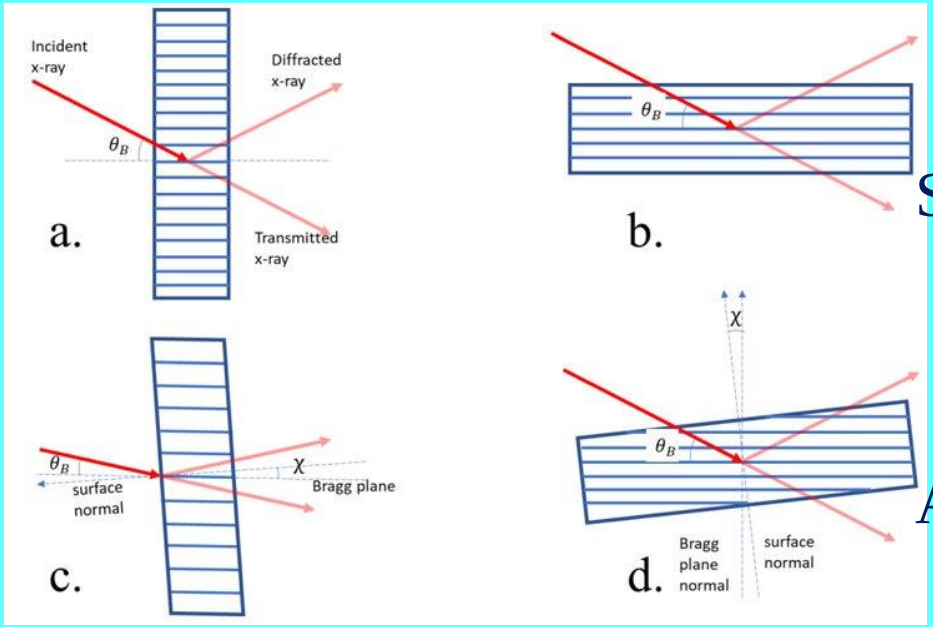
Lecture 17 May 2023

Bragg's law



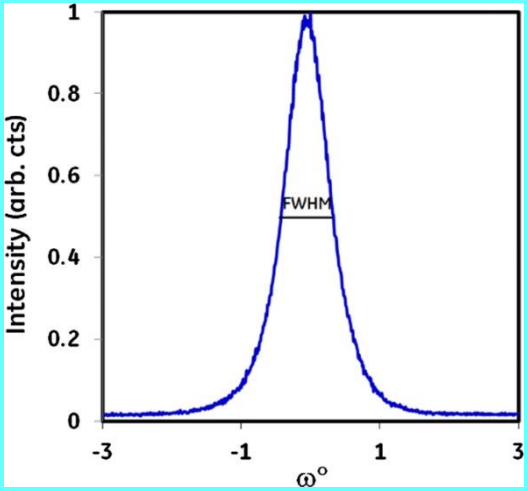


Bragg case



Symmetric

Asymmetric

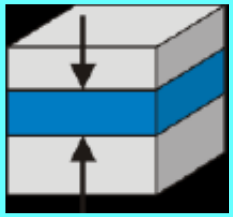


Laue case

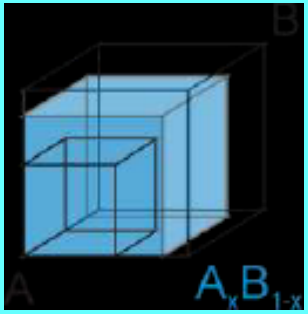
Transmission
(Bragg case)

Reflection
(Laue case)

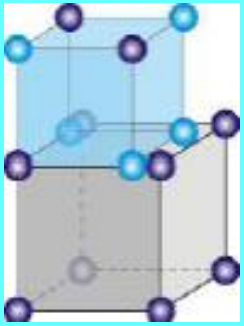
Analytical tasks



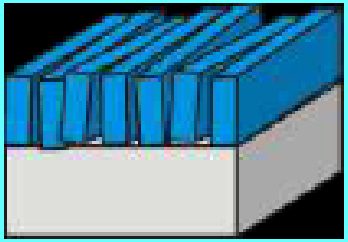
Layer thickness



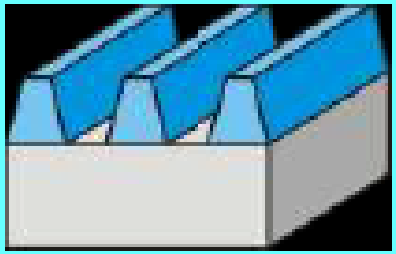
Chemical composition



Lattice relaxation

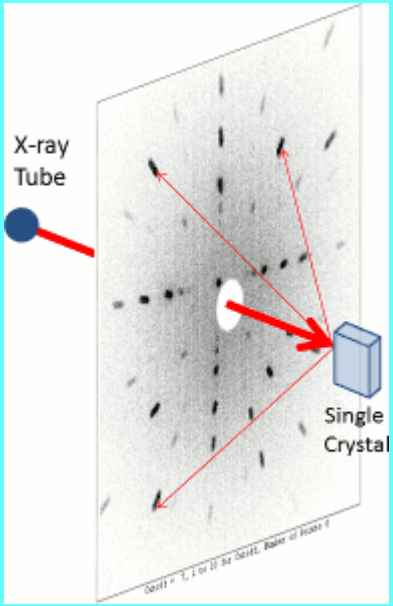


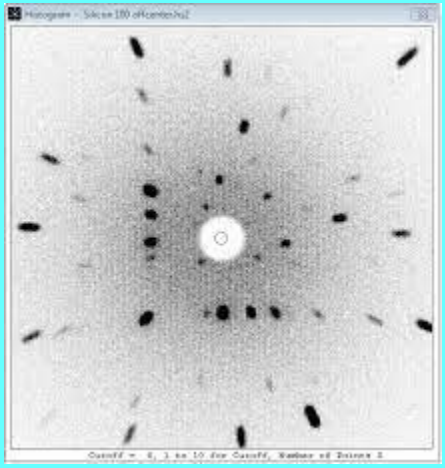
Defects and crystal size

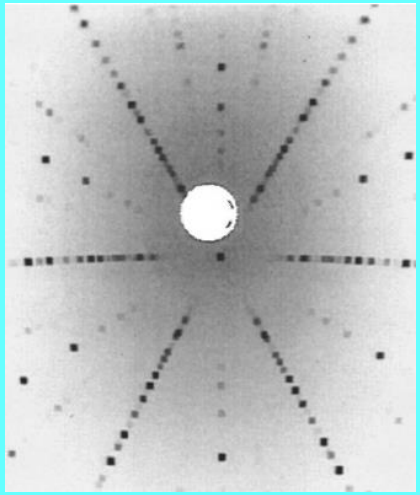


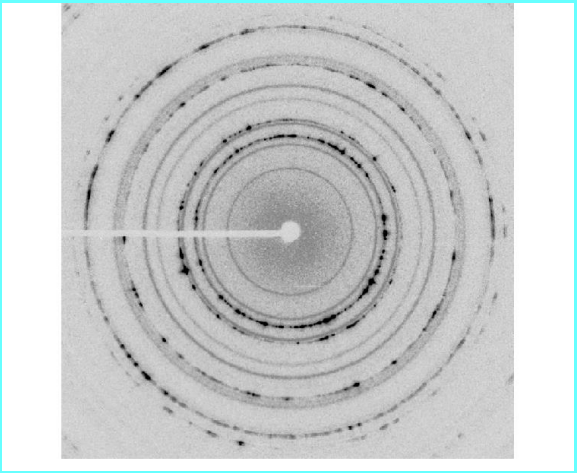
Lateral structures

Laue camera (white beam)

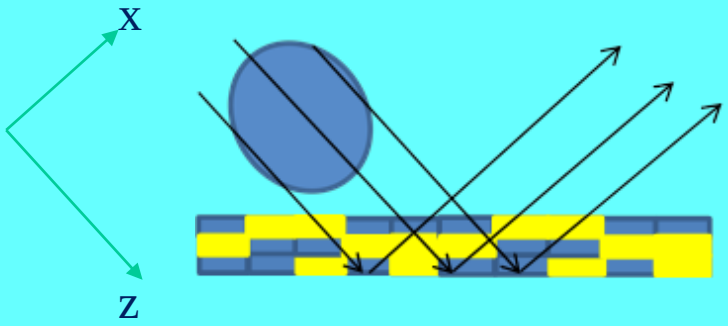


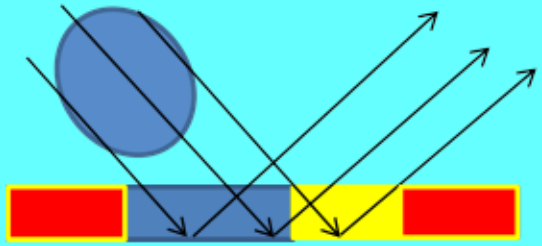






Photon coherence





We add amplitudes $(\sin+\sin+\sin+\ldots)^2$

We add intensities $(\sin^2+\sin^2+\sin^2+\ldots)$

Lz 1.5 μm.
Ly 0.5-5 μm
Ly 10-100 nm!!!

History

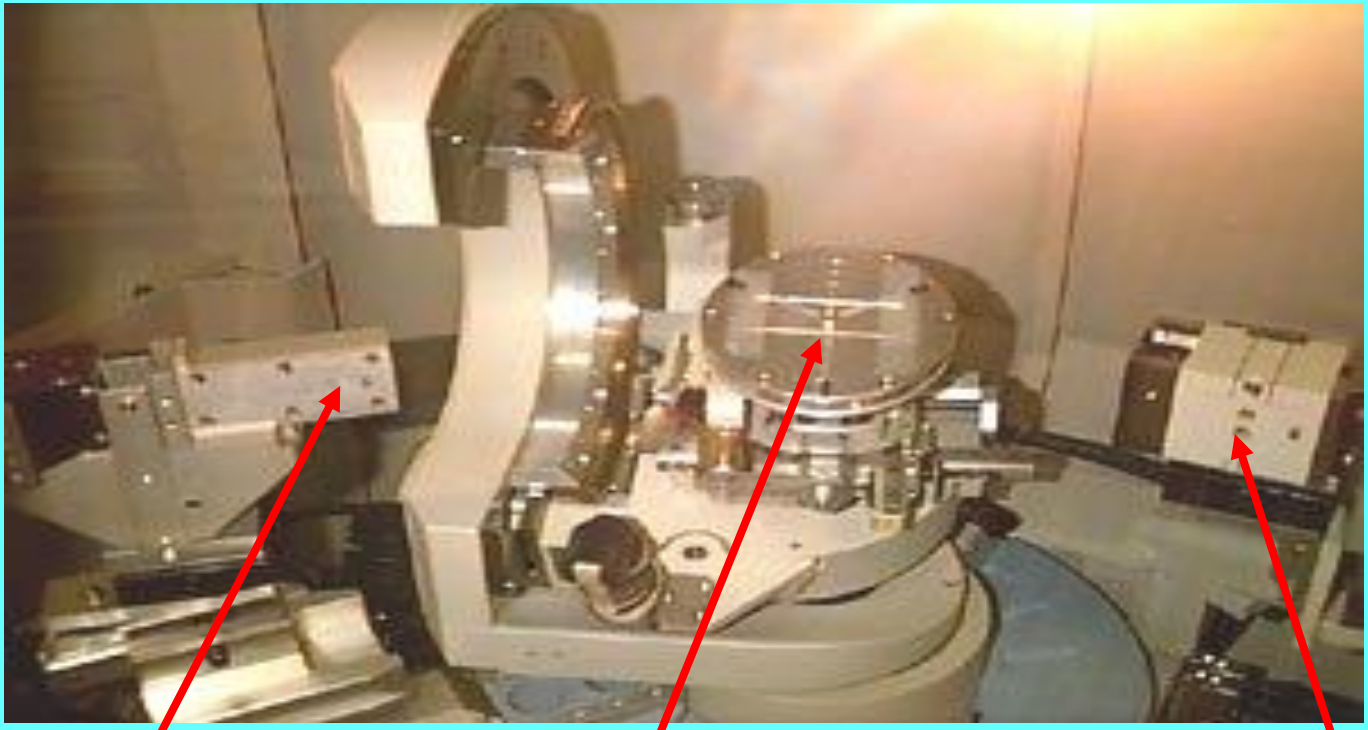
1912- first observation of XRD: Max von Laue

1912- 1940- diffraction theory: W.L. Bragg, W.H. Bragg, R.W. James

1948- first diffractometer: Philips Anal.

1976- first personal computer: S. Wozniak and S. Jobs

Diffractometer

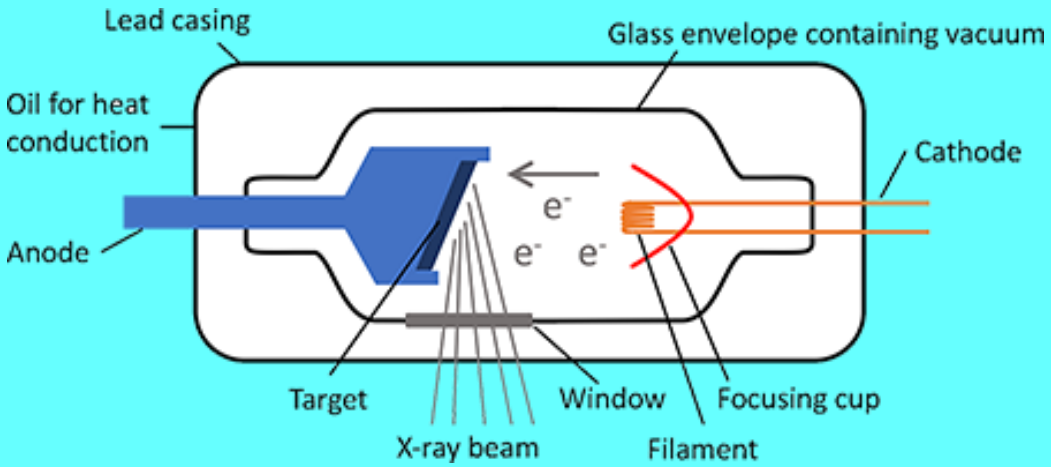


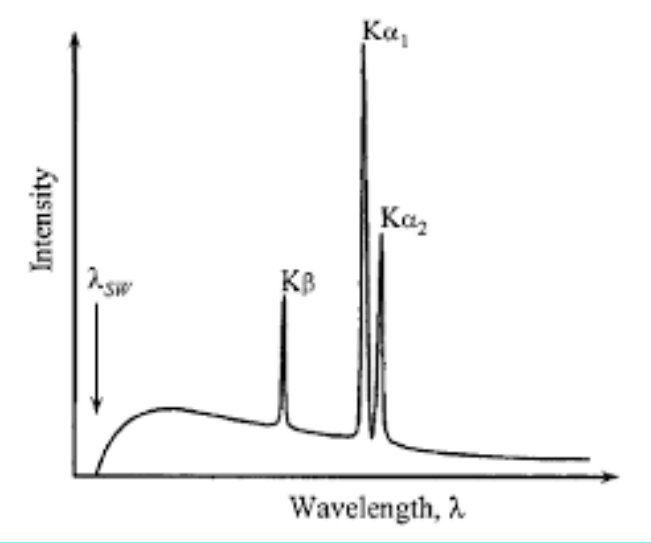
Primary beam

Goniometer head

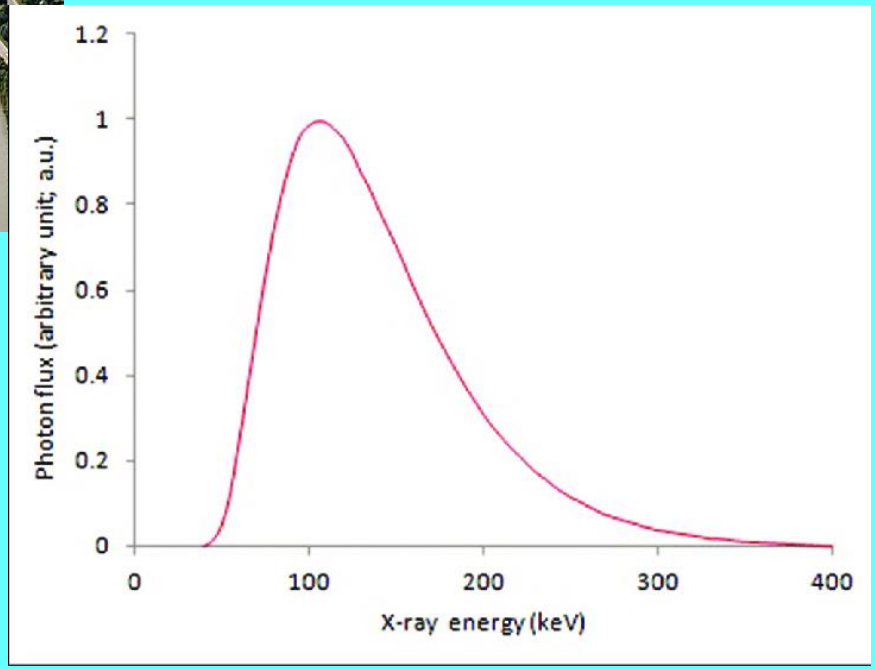
Reflected beam

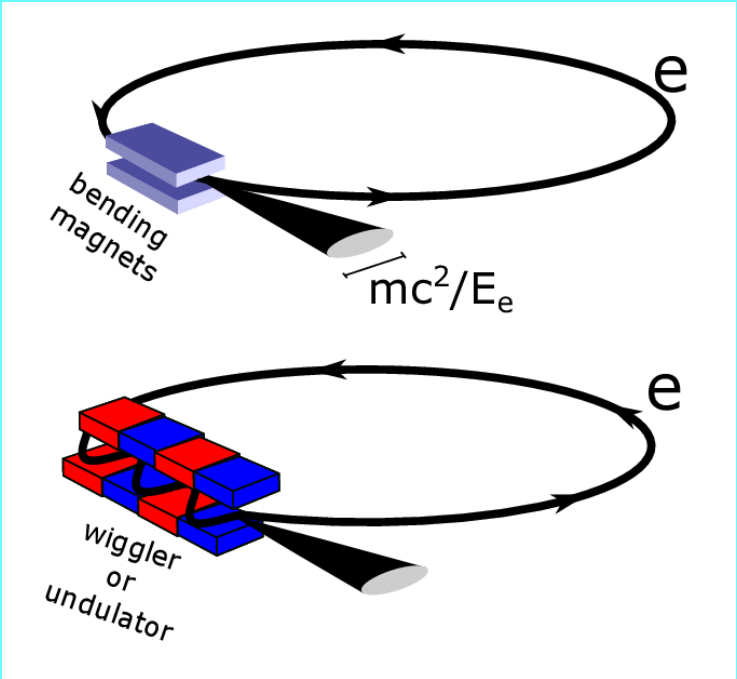
X-ray tube


Lead casing
Glass envelope containing vacuum
Oil for heat conduction
Cathode
Anode
e⁻
e⁻
e⁻
Target
Window
Focusing cup
X-ray beam
Filament

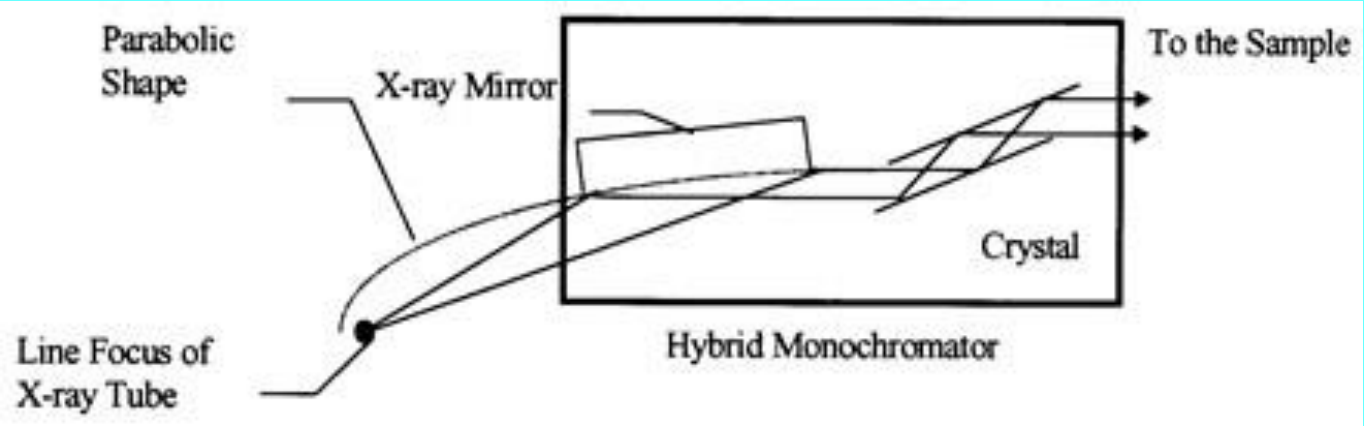

Kα1
Kα2
Intensity
λSW
Kβ
Wavelength, λ

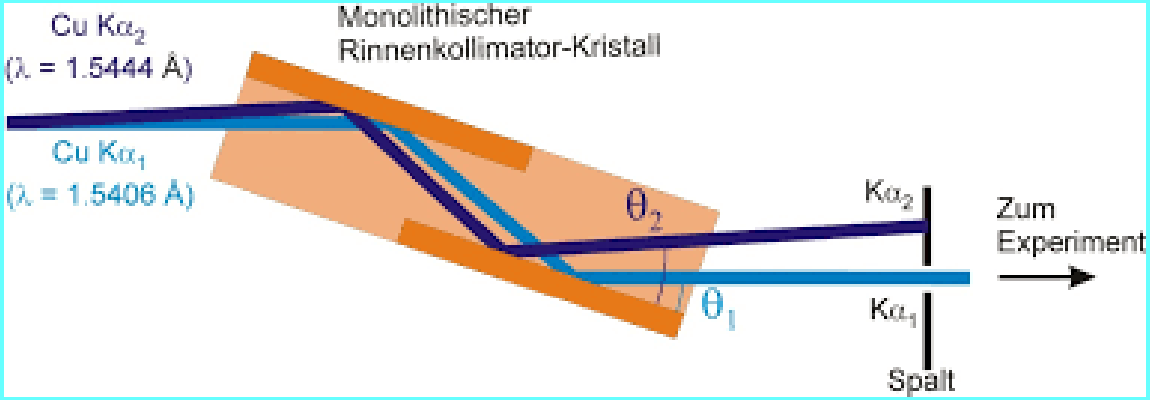
Synchrotron radiation



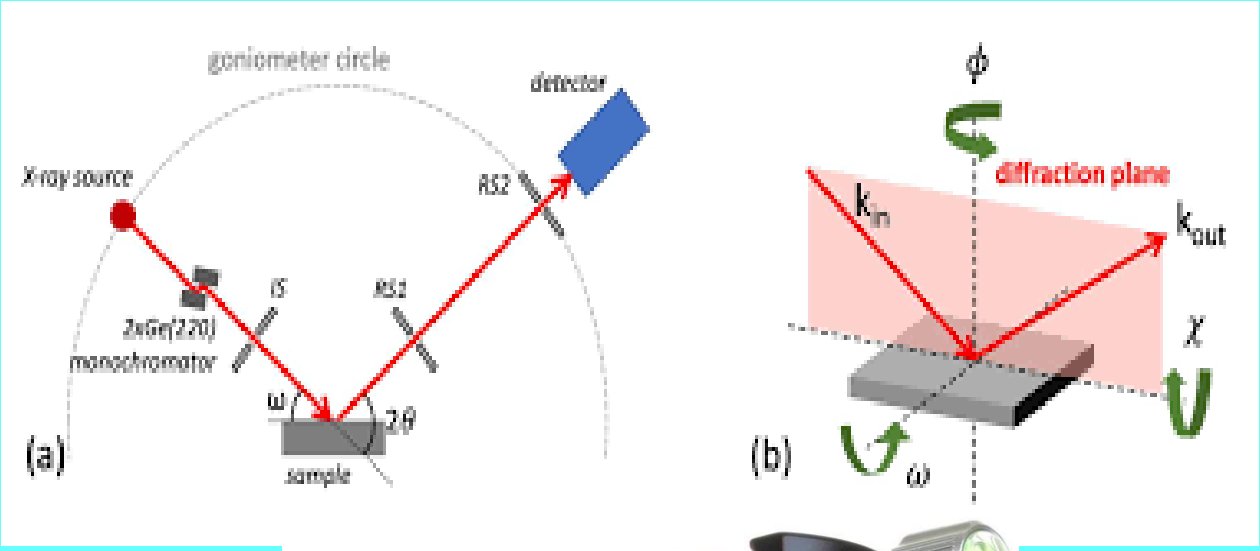


Monochromator

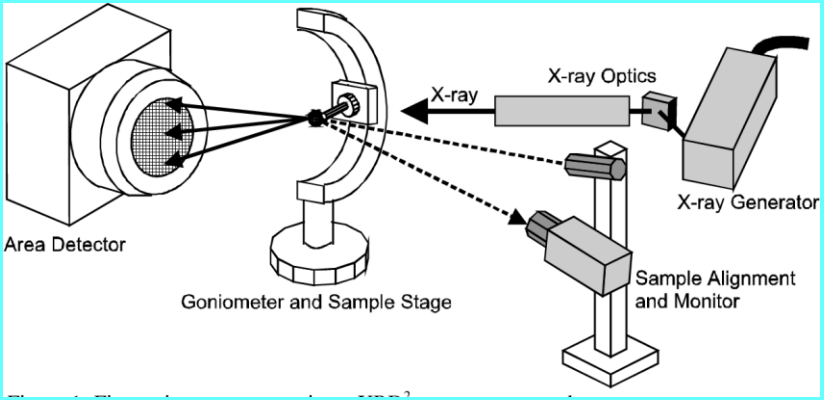


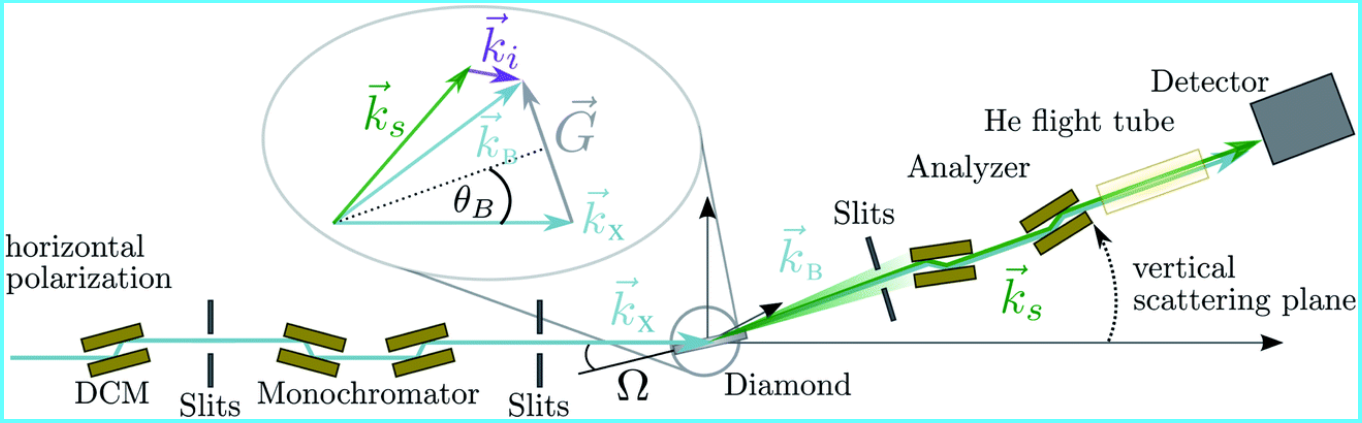


Goniometer head



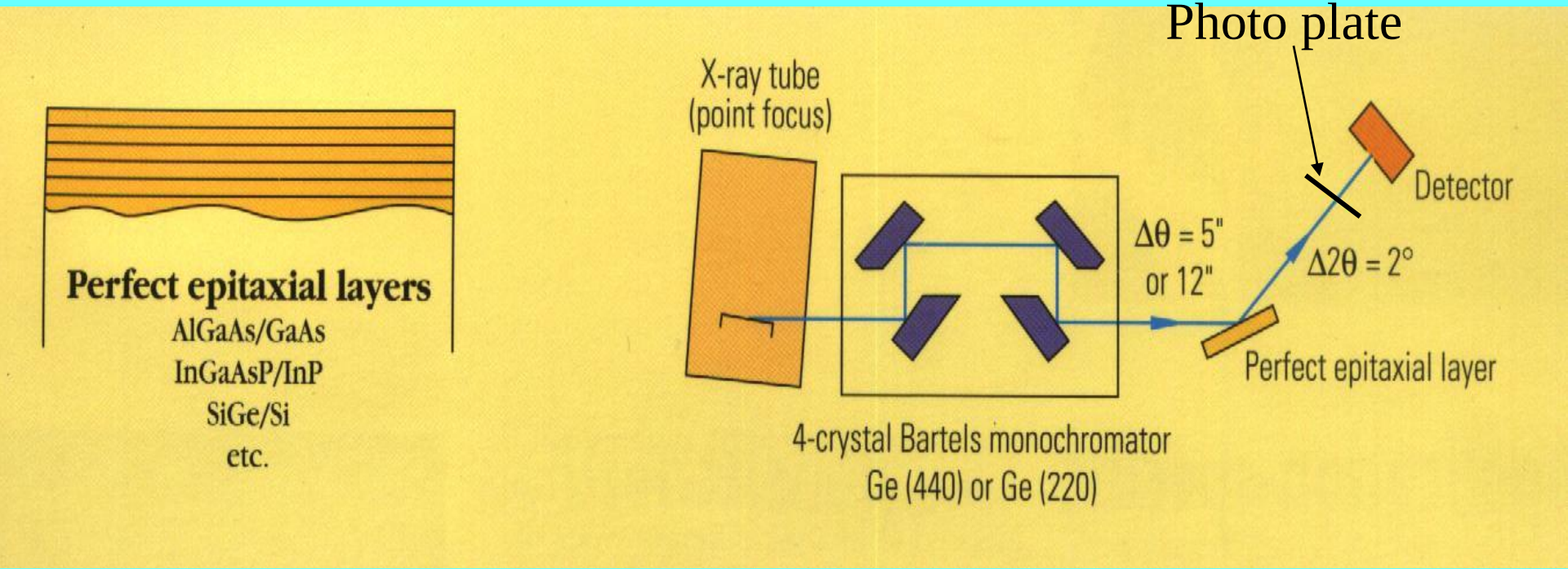
Analyzers and detectors



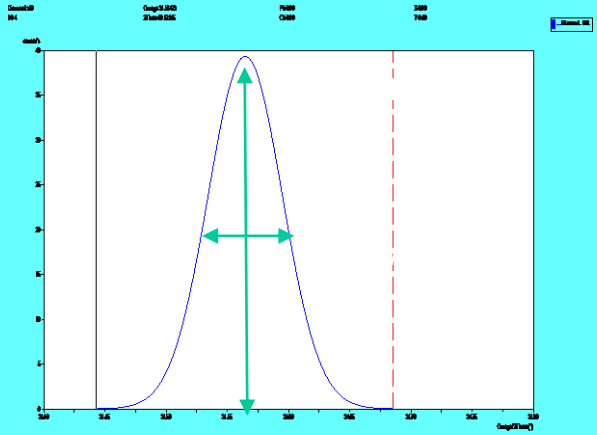


Double axis, double crystal, rocking curve configuration

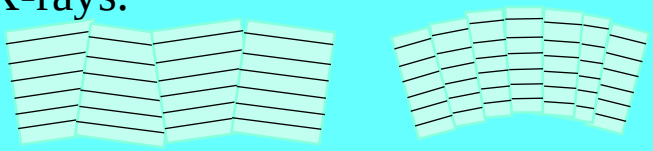
Sensitive to mosaicity and lattice parameters variations



Rocking curve as a measure of GaN crystallographic quality



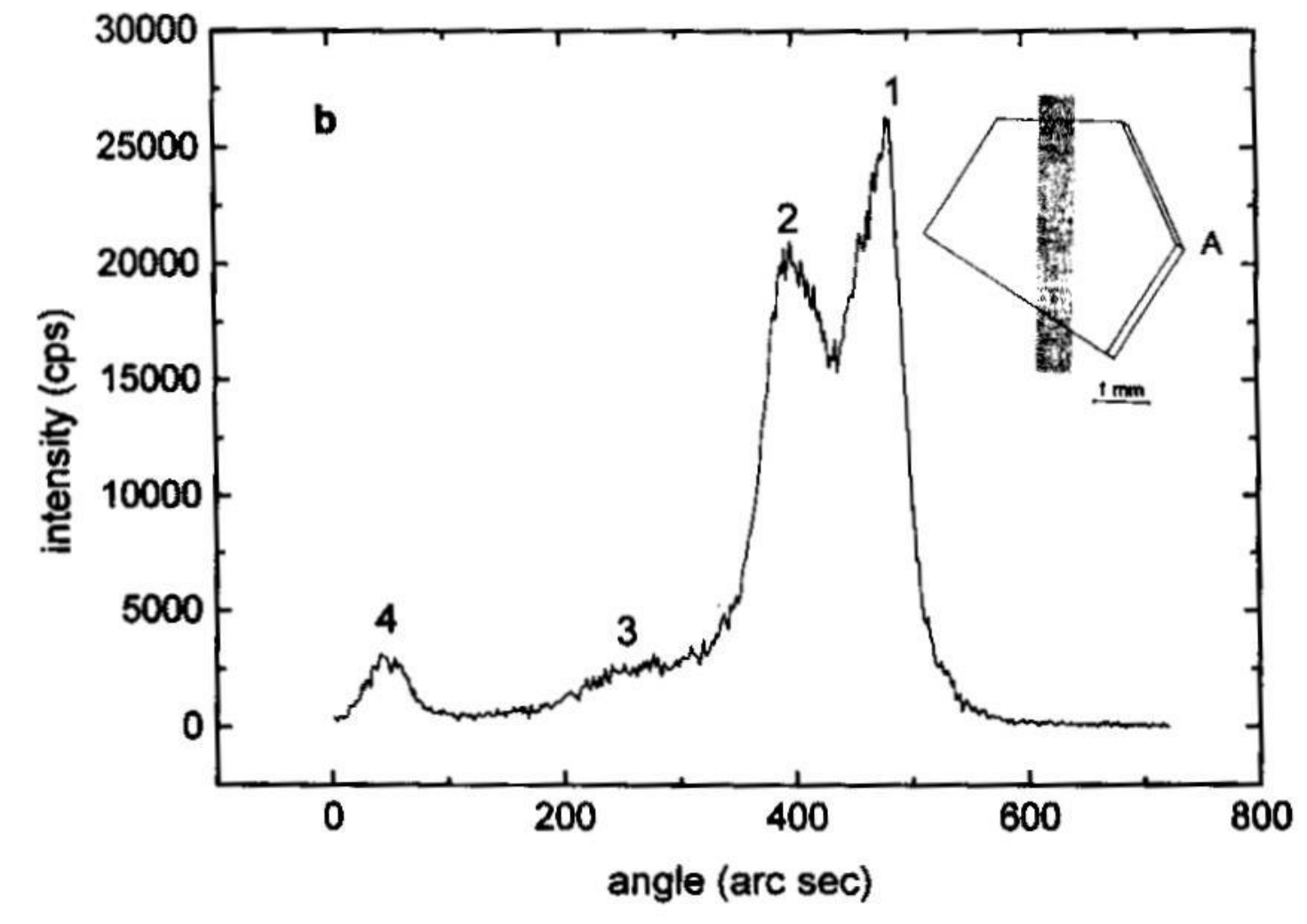
Always compare FWHM and intensities for various reflections (also asymmetrical ones), as well as different area illuminated with X-rays.



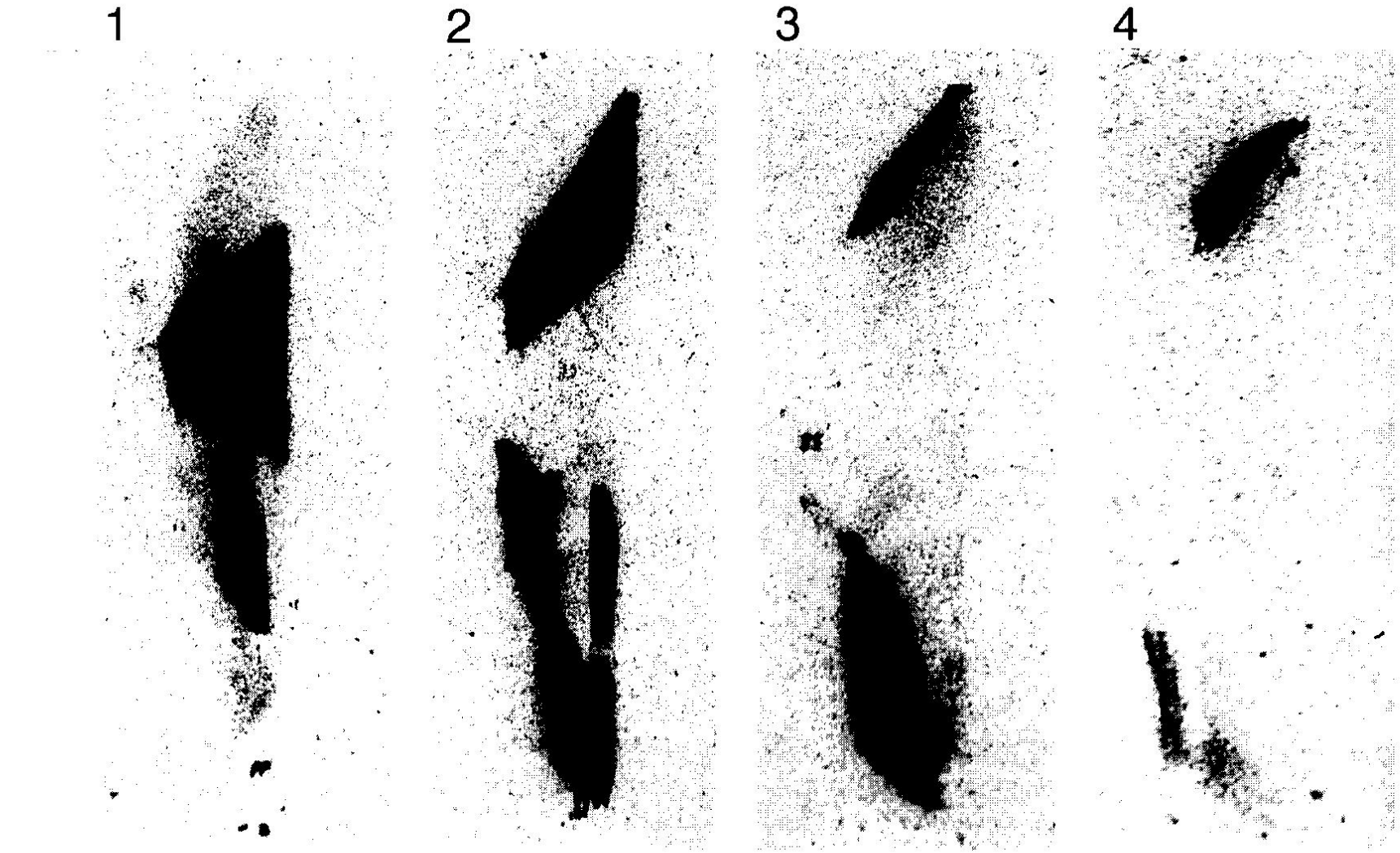
GaN crystal (dislocation density cm-2)	FWHM 00.2 (arc deg)	Intensity 00.2 (Mcps)
A 2x10exp8	0.087	28
B 8x10exp6	0.030	29
C 1x10exp6	0.038	30
D 5x10exp4	0.015	32
GaN crystal	**FWHM 00.4 (arc deg)**	**Intensity 00.4 (Mcps)**
A	0.081	4
B	0.029	12
C	0.023	15
D	0.015	21

Small size of crystallites- additional peak broadening!!!

Mosaic structure of HP GaN crystals



Topografia kryształu GaN



Information from the rocking curves and topography

- Bu lk crystals(GaAs, Si, InP, i in.):

i) mosaicity (dislocation density higher than10^6 cm^{-2}),

ii) bowing,

iii) off-orientation

- Epi wafers:

i) thickness (+/- 2-5 A)

ii) chemical composition of ternary compounds (+/-1%)

EL2-like defects

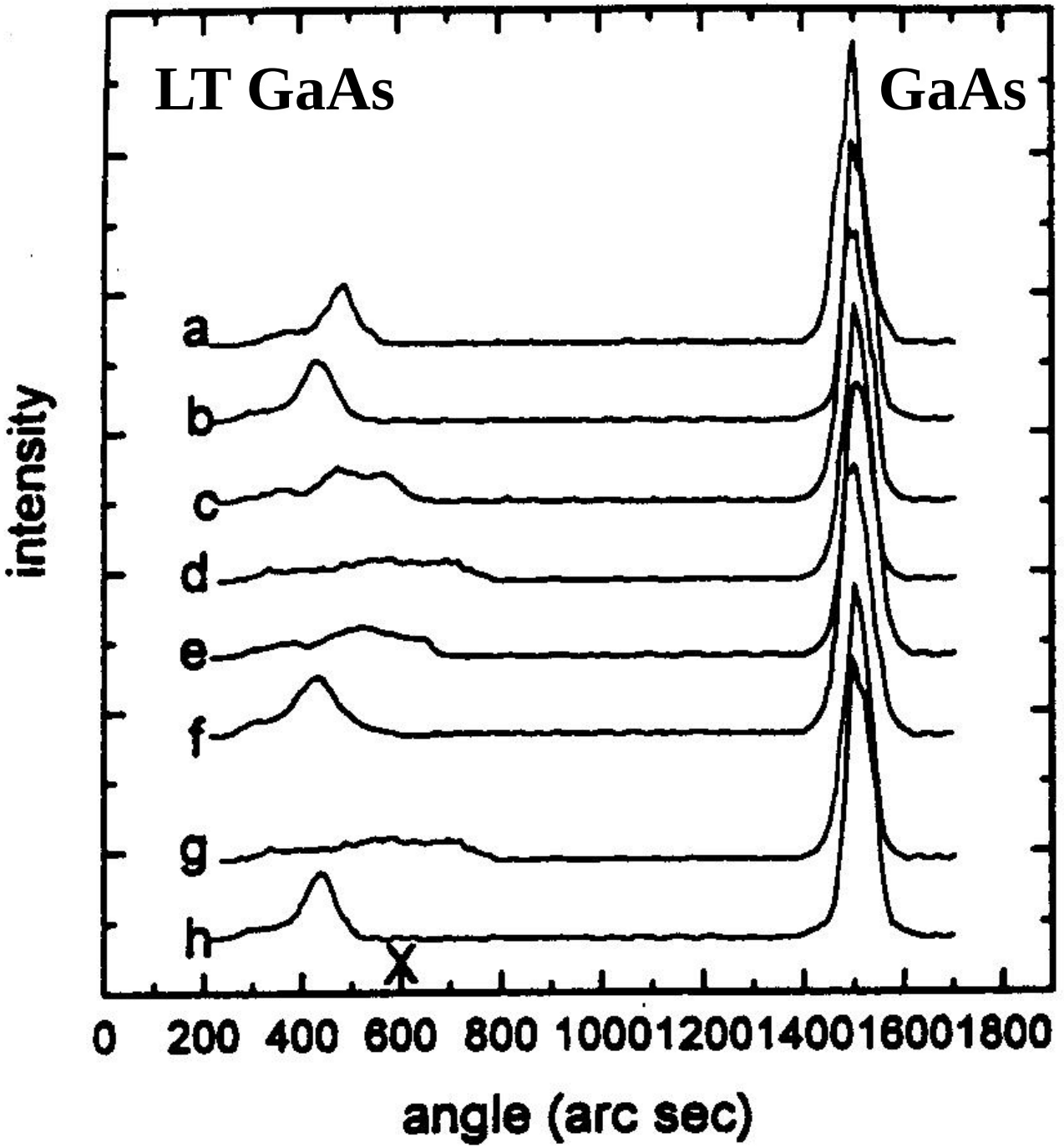
300 K

77 K dark

77 K + 900 nm

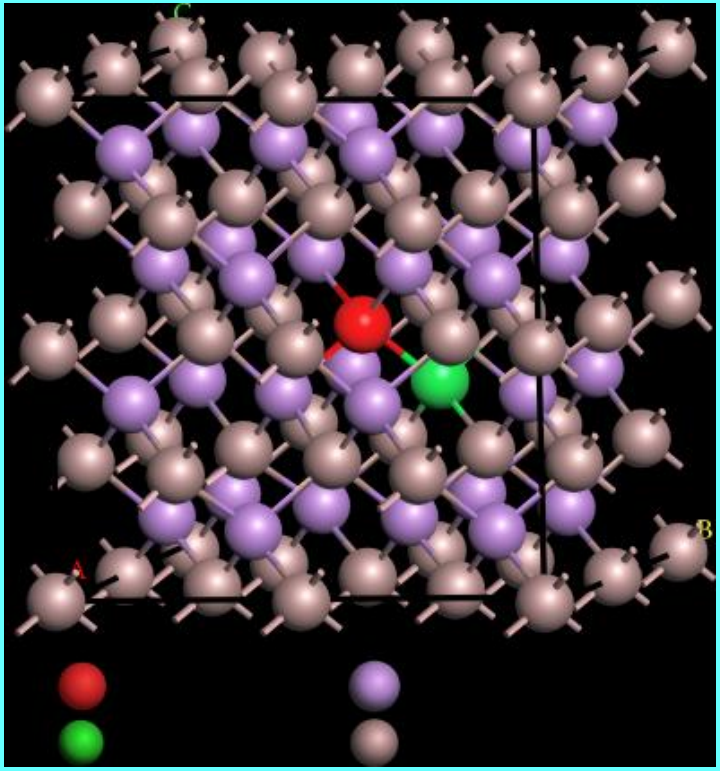
+1350 nm

Or +140 K



582186.fig.003a.svg

We are not able to see point defects in XRD- only their inhomogeneities (scale 0.1-1 μm)



Theory

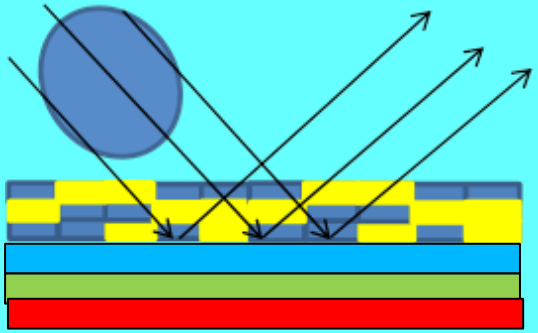
Kinematical theory

No rescattered radiation is taken into account

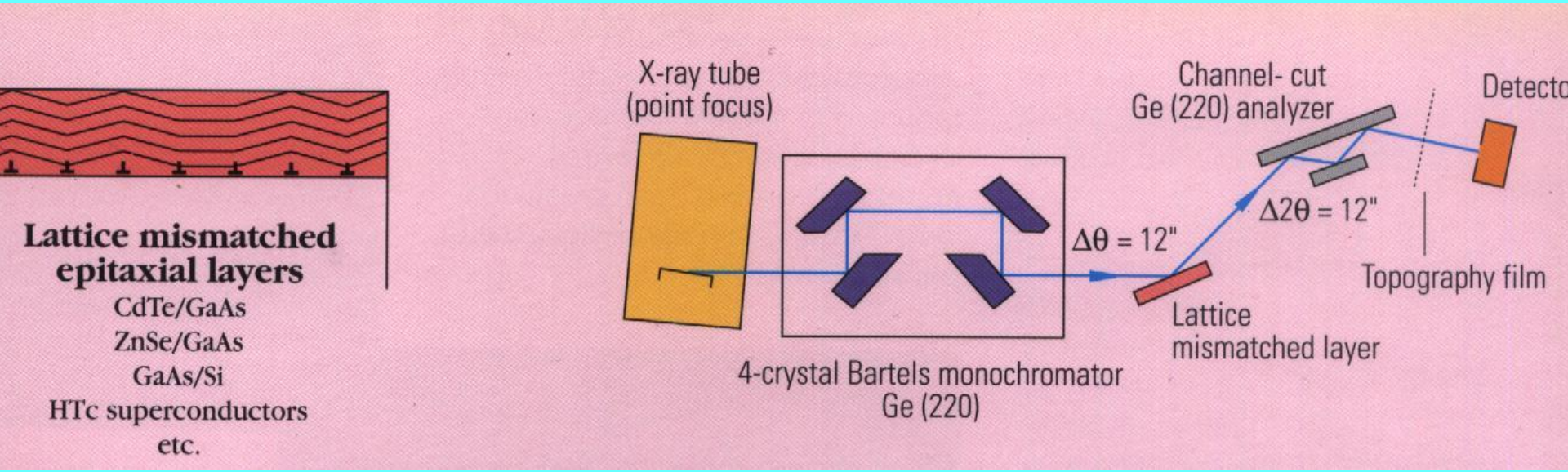
Dynamical theory

Based on Maxwell radiation

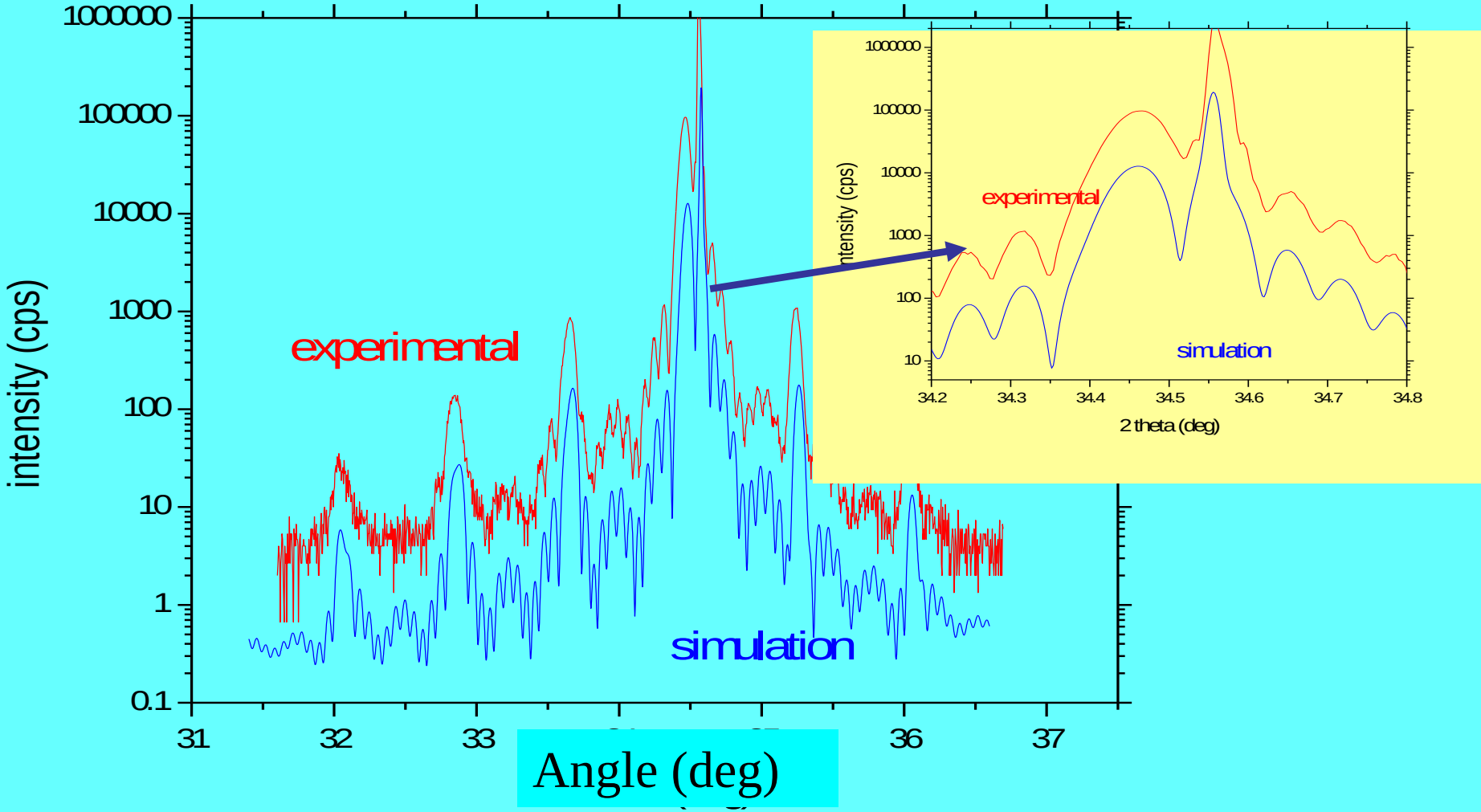
Commercial programs (for example, Epitaxy) for pefect epi-structures (only vertical changes)



Triple axis
Sensitive to lattice parameters variations

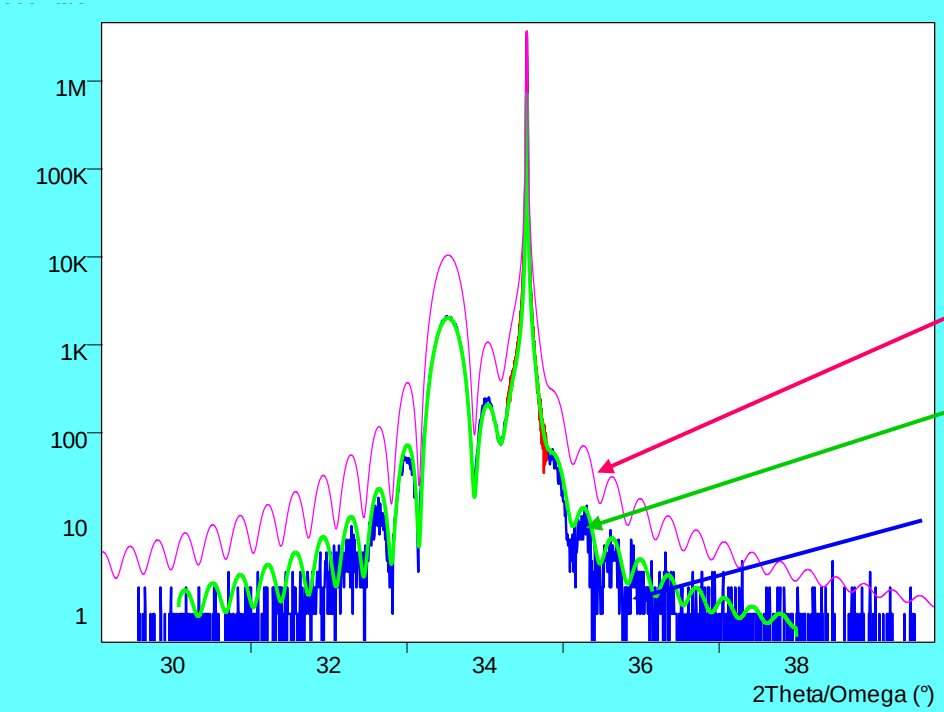


10-fold InGaN/GaN



d(well)=3.2 nm, d(barrier)=7.1 nm, $x_{average}$= 3.2%

2theta/omega for InGaN layer on GaN/sapphire

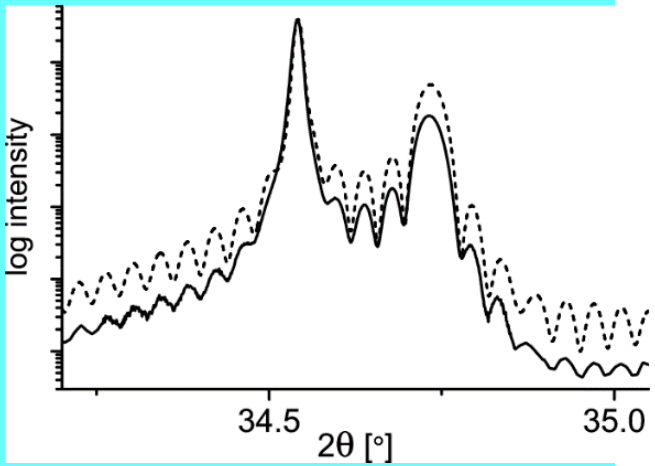


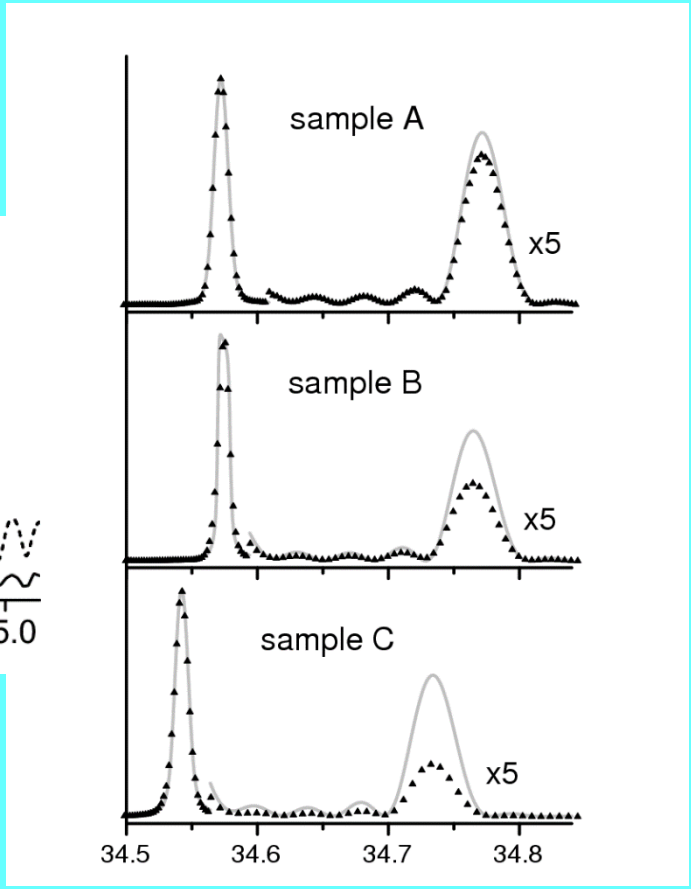
Simulation matching to the GaN substrate peak

Simulation matching to the InGaN peak

Experimental curve

AlGaN layers on different substrates



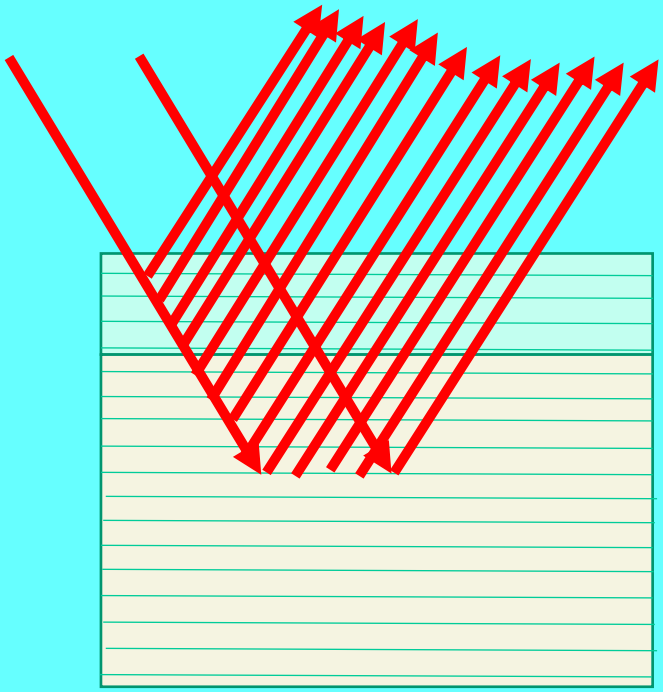


Ammono GaN substrate
EPD 10^4/ cm^2
87% of AlGaN peak intensity

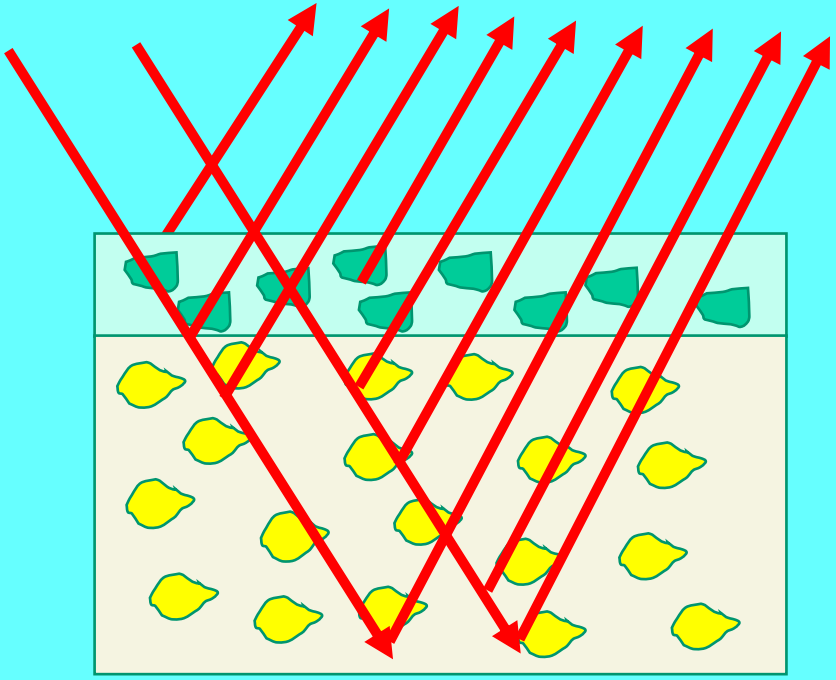
HVPE GaN substrate
EPD 10^7/ cm^2
60%

GaN on sapphire
EPD 10^8/ cm^2
35%

Qualitative explanation why peaks from thin layers depend very strongly on crystallographic quality

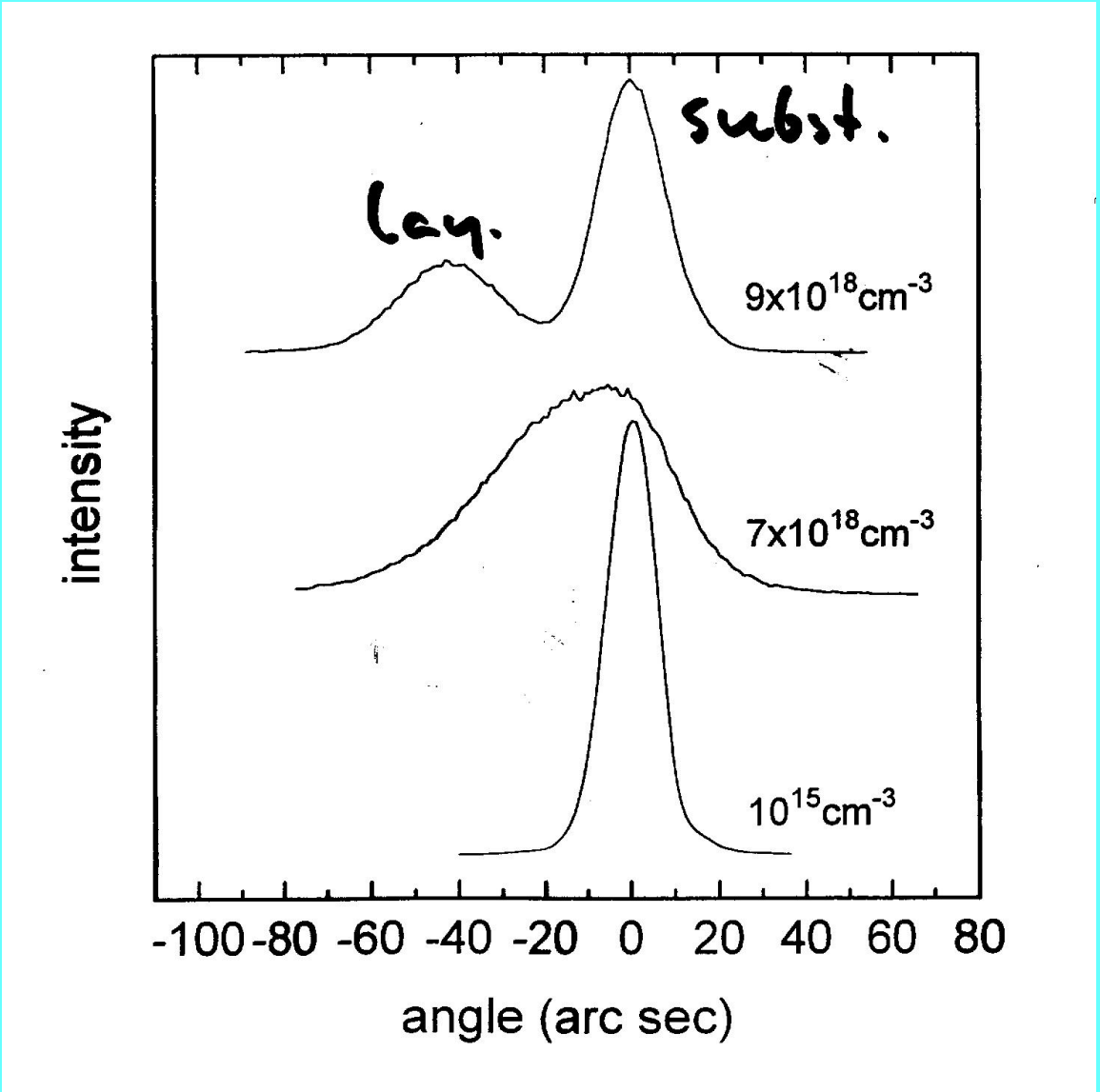


Perfect crystal

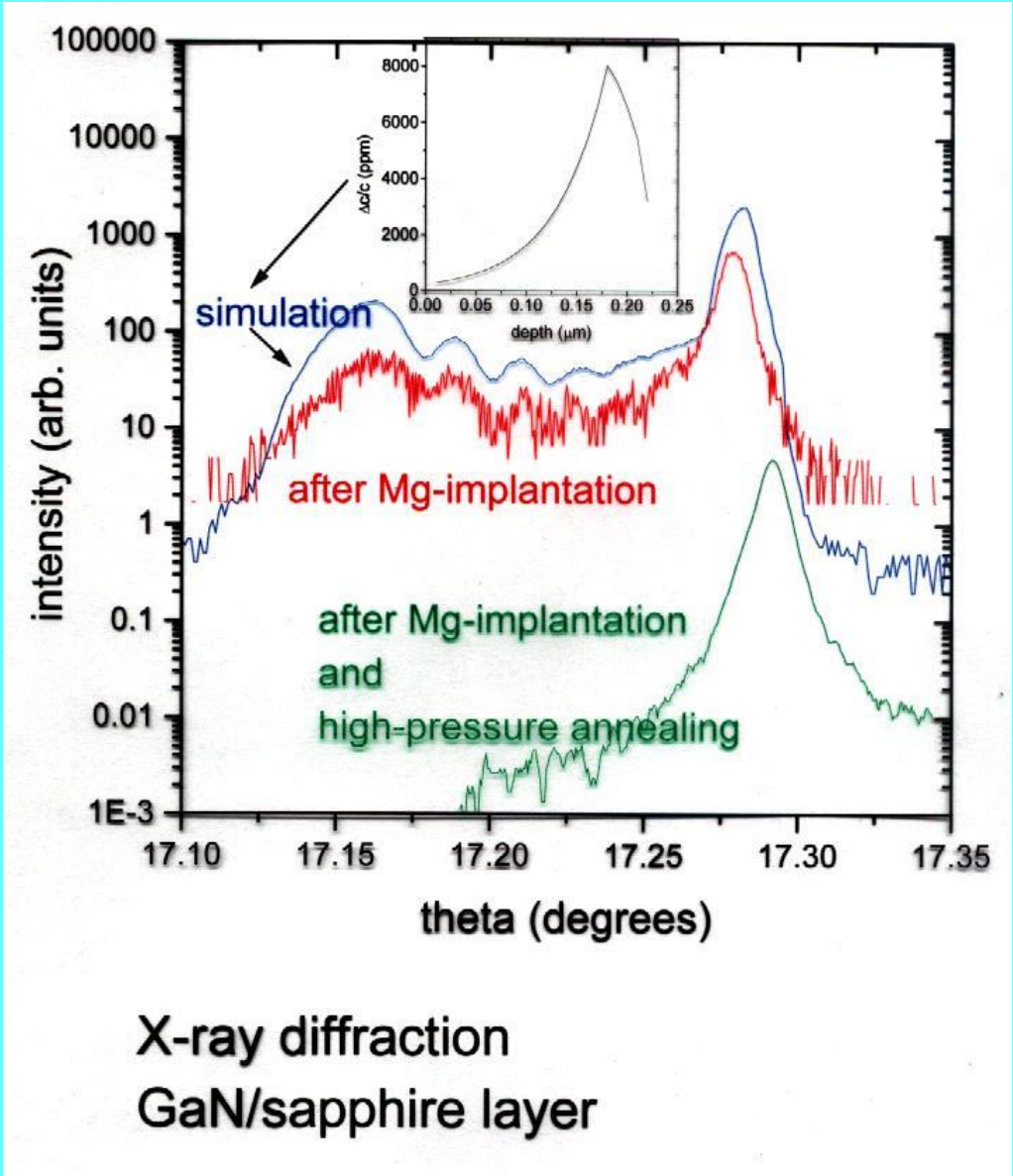


Only small regions fulfill Bragg condition
Thin layers have much smaller intensity

Example: Lattice expansion by free-electrons GaAs



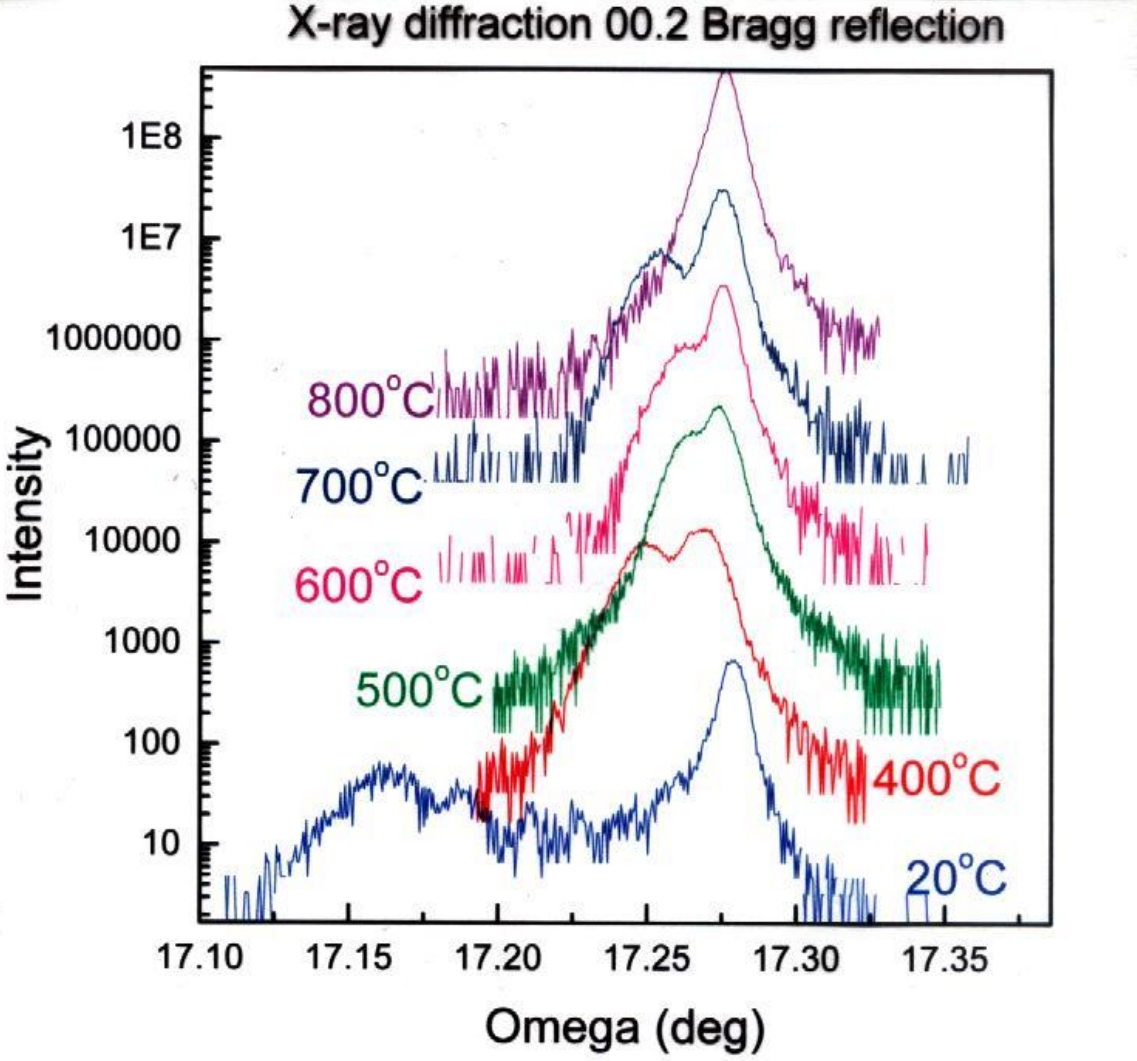
Example: Effect of implantation



X-ray diffraction
GaN/sapphire layer

1200oC
necessary

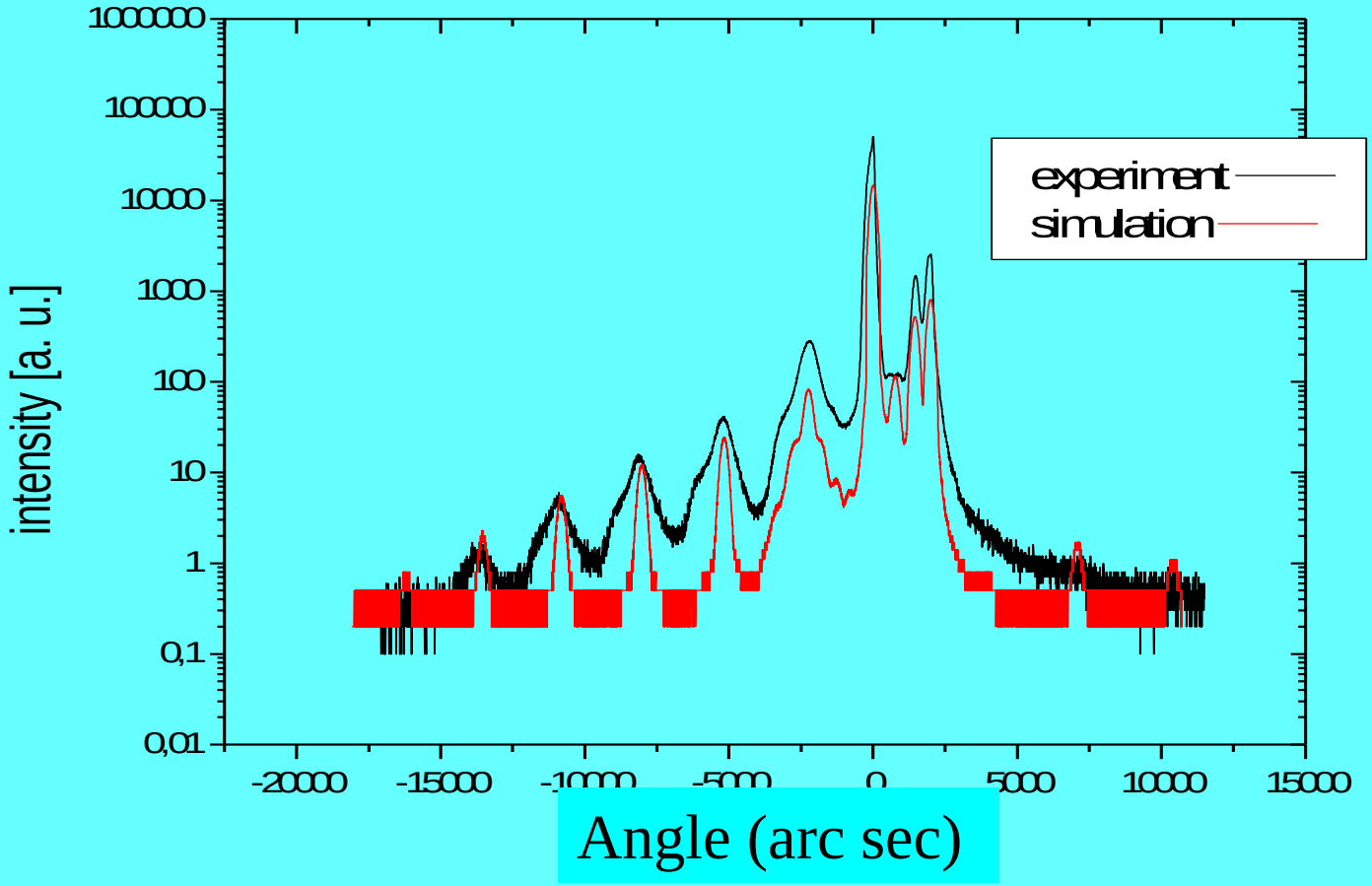
Implantation at high temperature



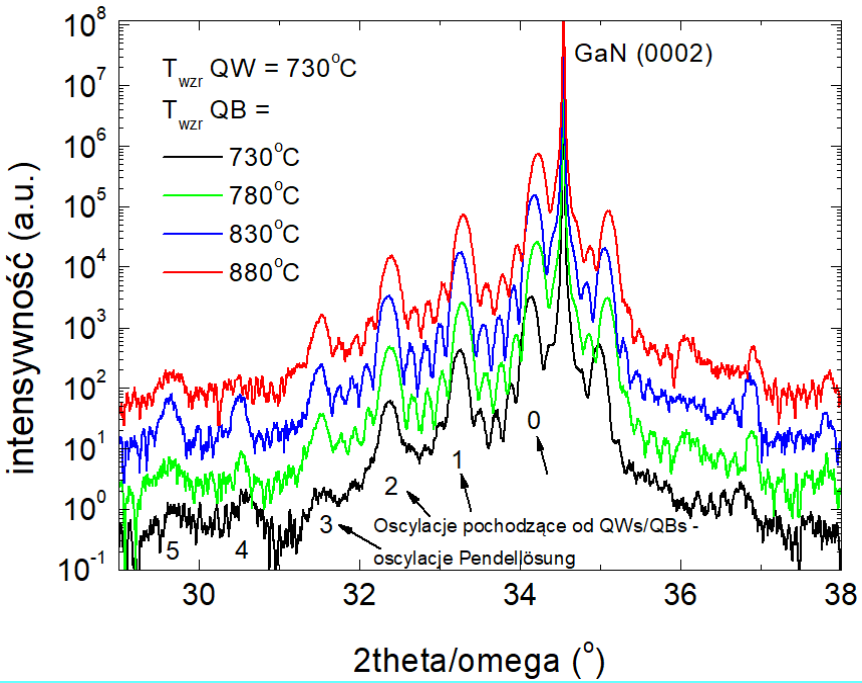
GaN layers on sapphire implante
with Mg at different temperatures

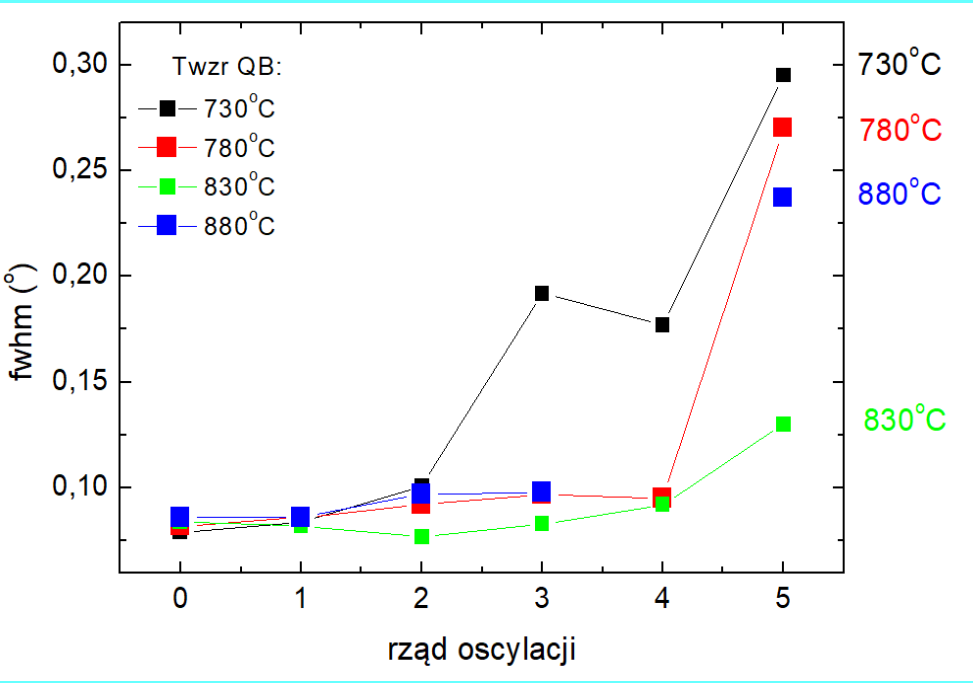
Only 800oC necessary

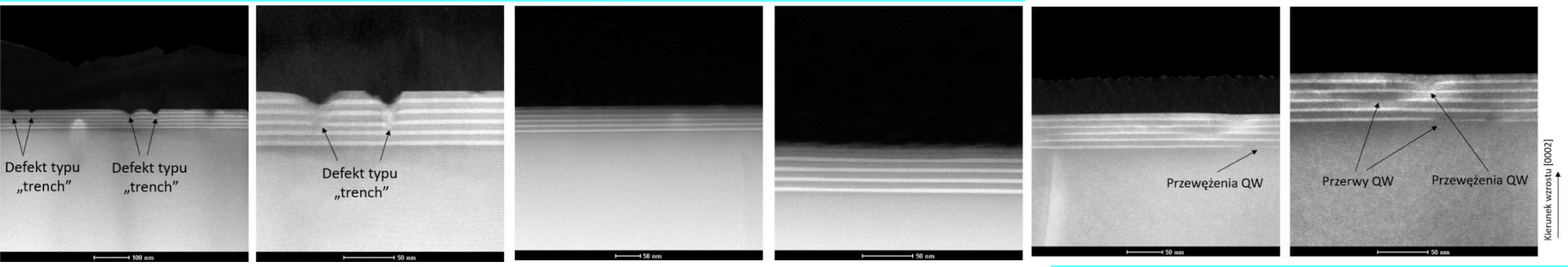
Example: 10-fold GaN/InGaN with indium fluctuations



Broadening of fringes
InGaN QWs, QBs grown at different temperatures

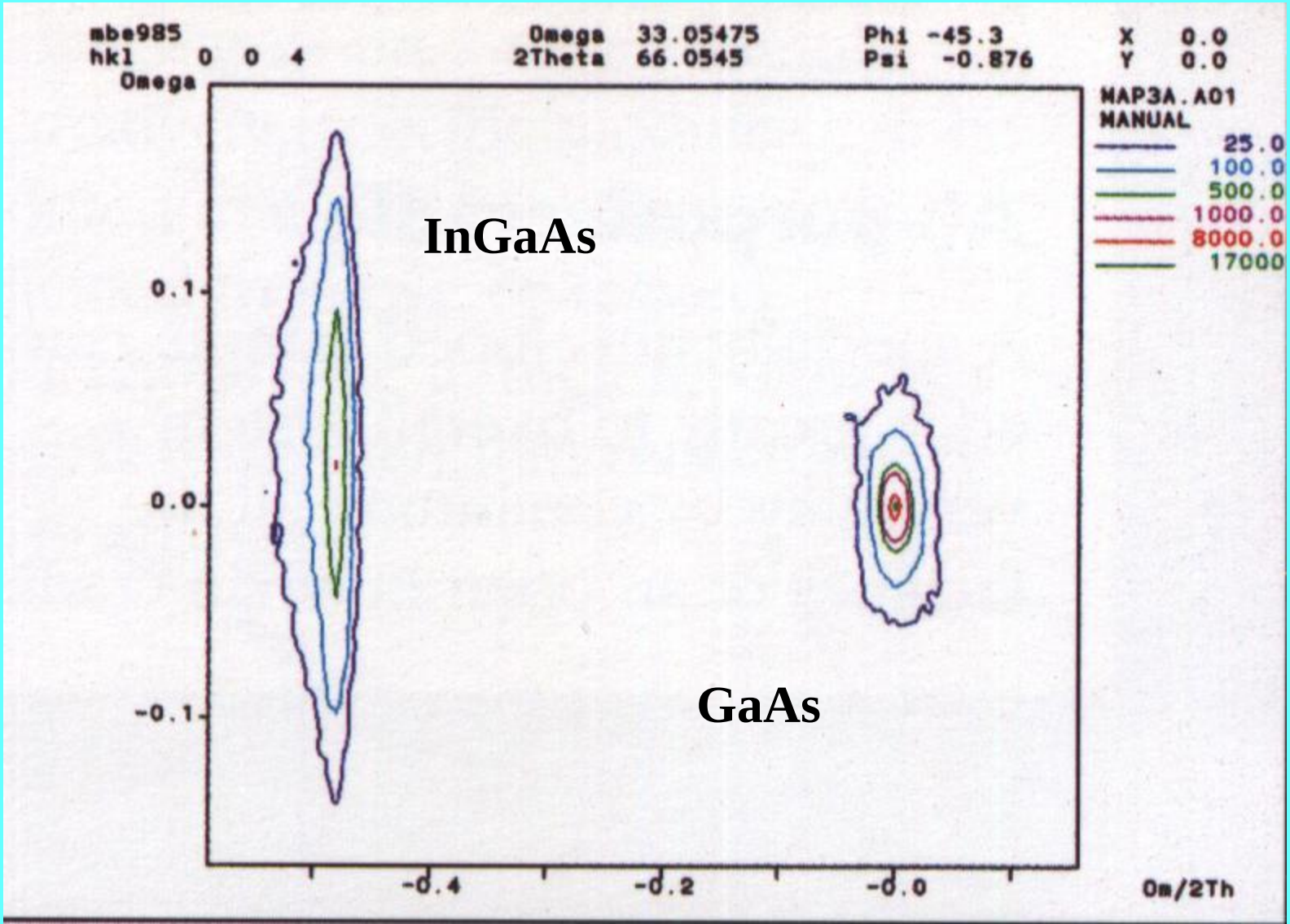




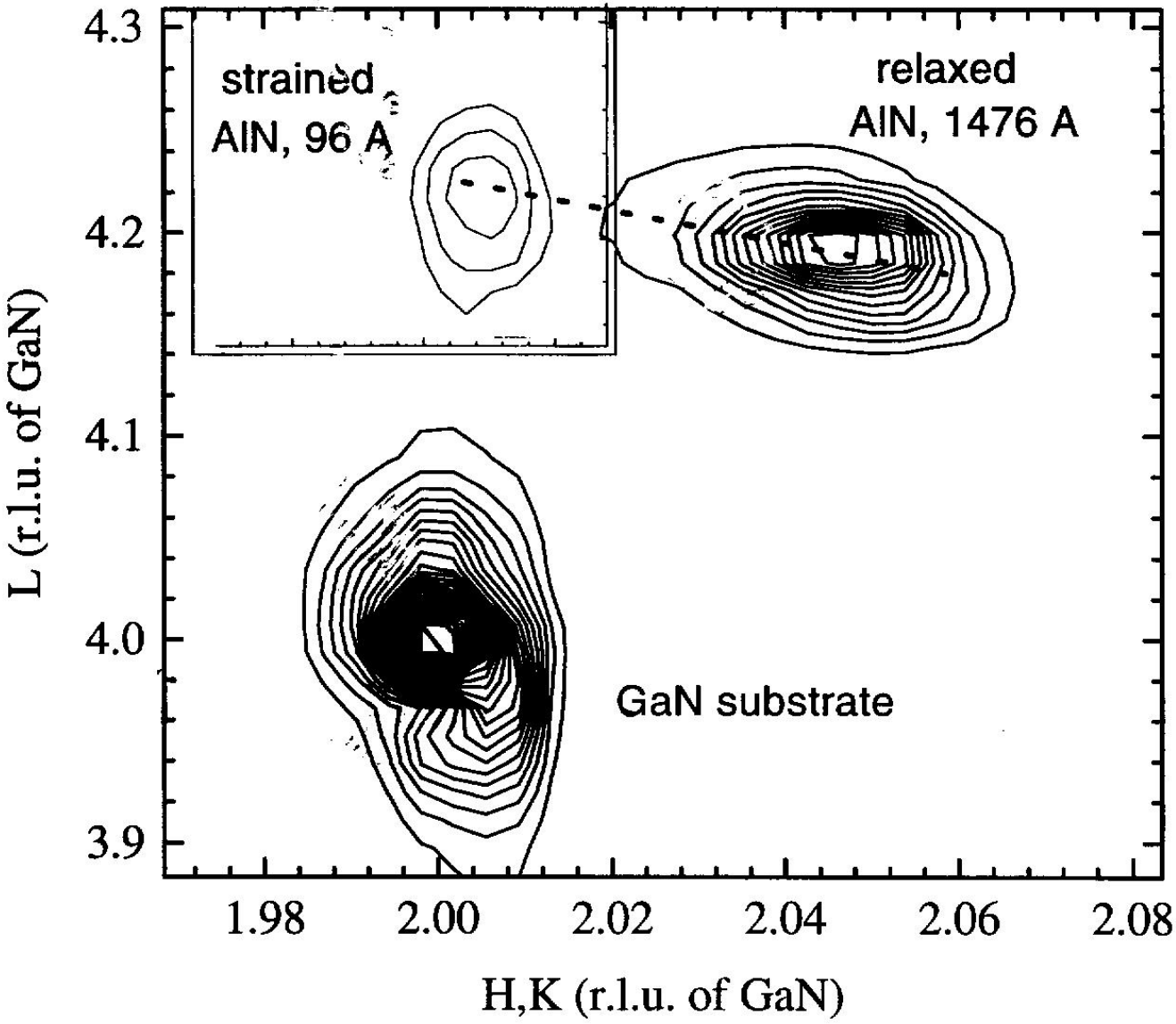


730 830 880

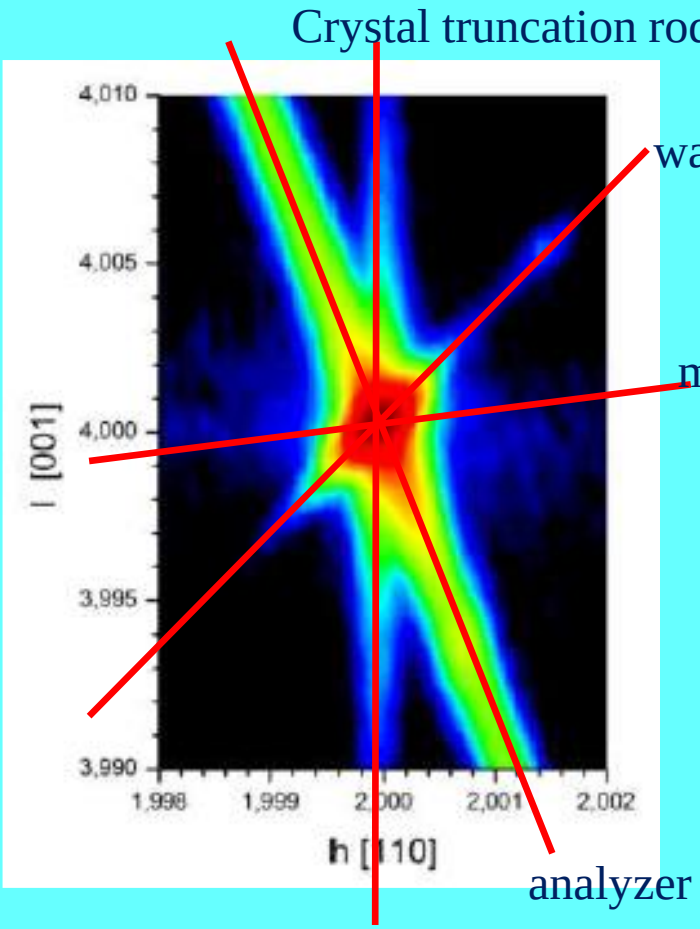
Reciprocal lattice mapping



Reciprocal lattice maps



Streaks in reciprocal lattice mapping

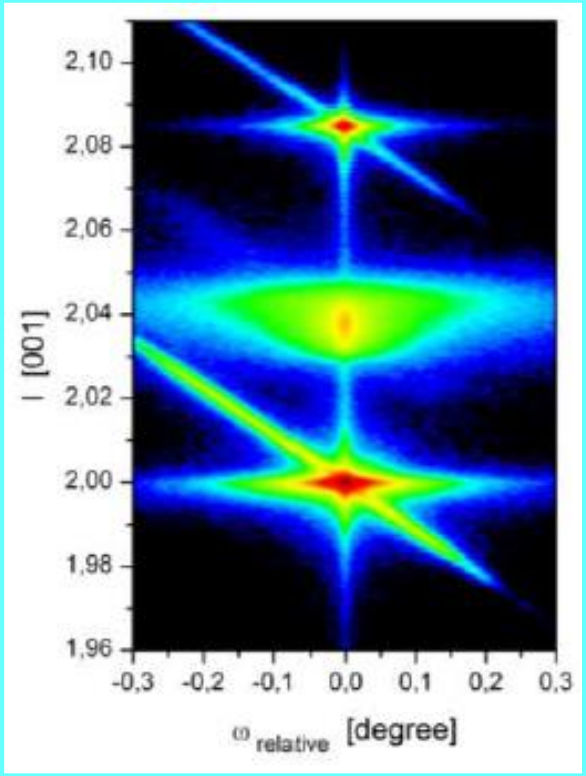


Differ for:

- Different reflections
- Diffractometer set up
- Sample

Reciprocal lattice mapping

00.2



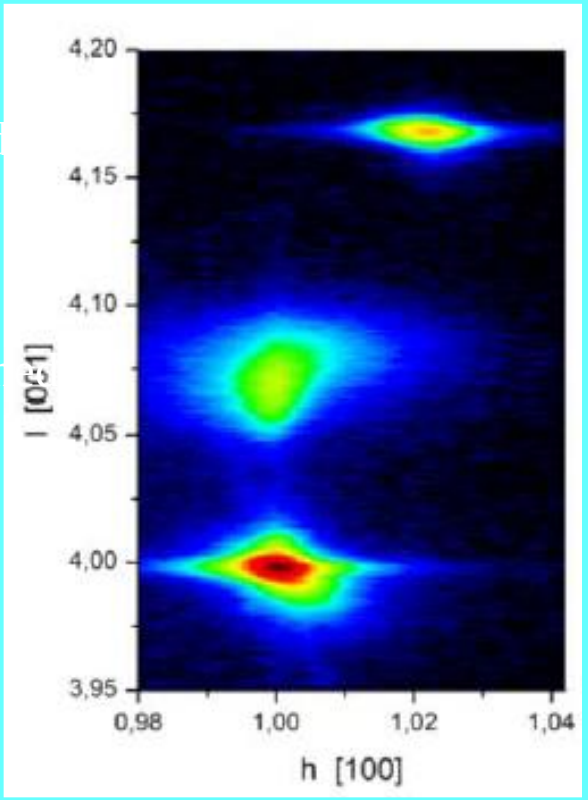
Example from Bruker webminar

AlN relaxed

AlInN strain
to GaN

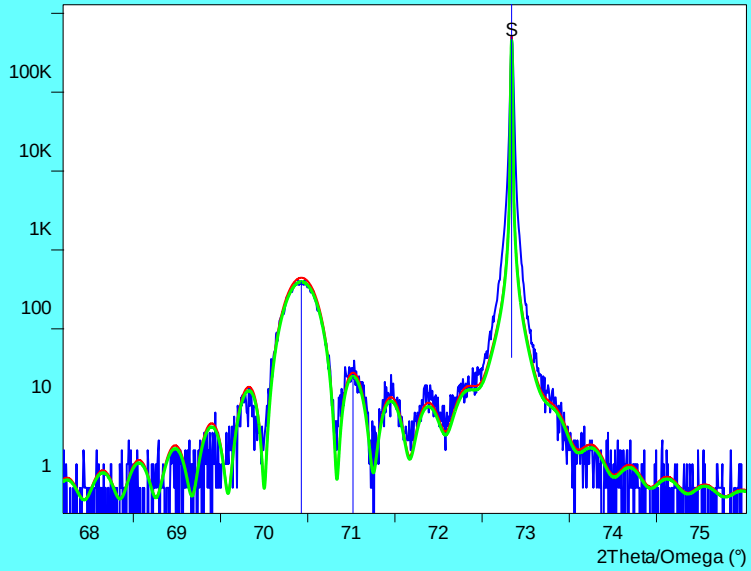
GaN

10.4

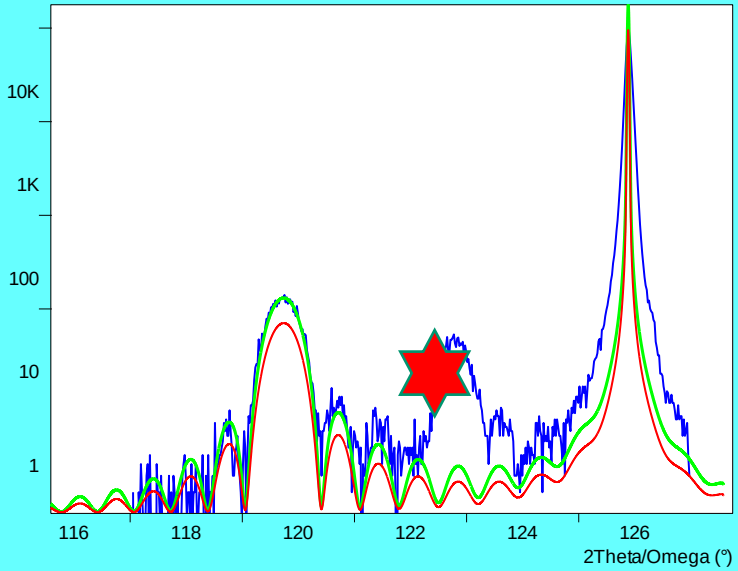


Look out for Domagala's peaks (hybrid peaks)

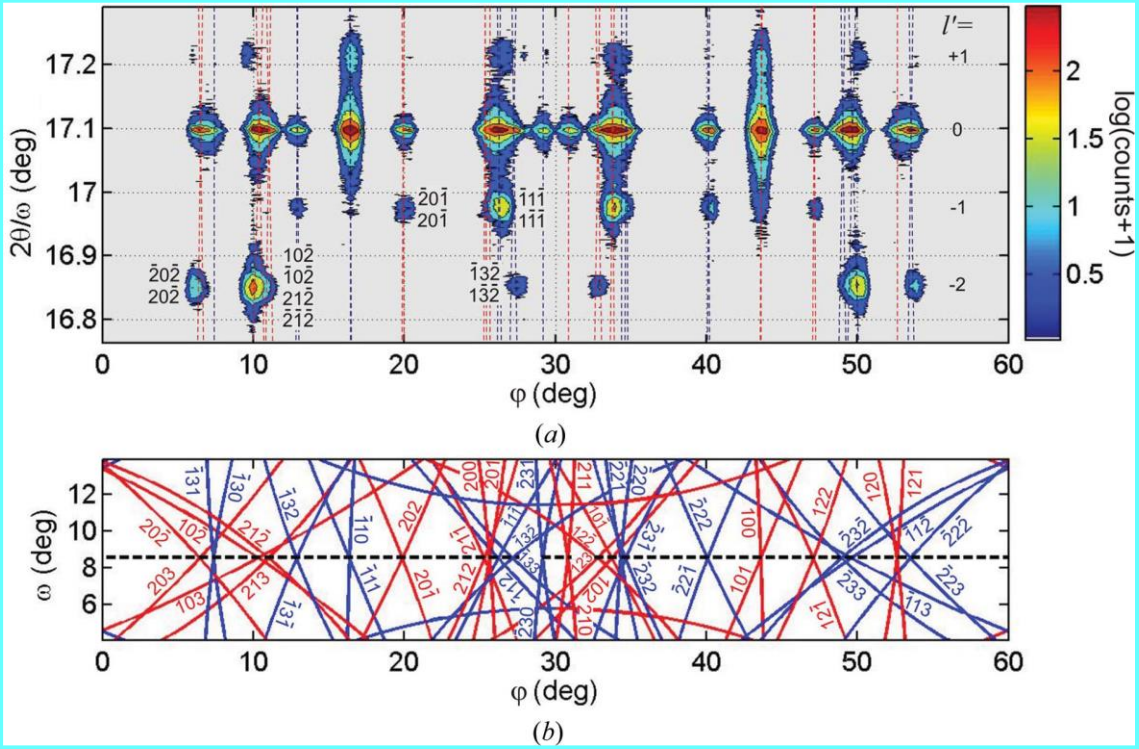
00.4

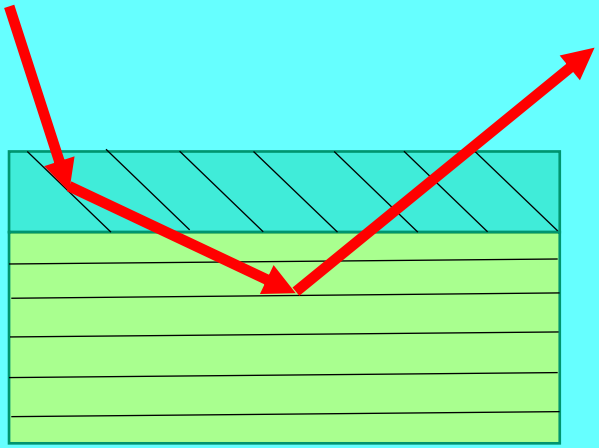


00.6

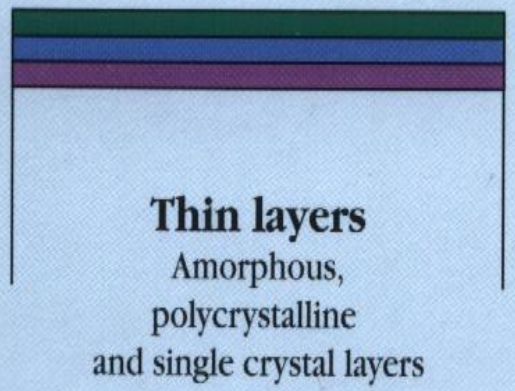


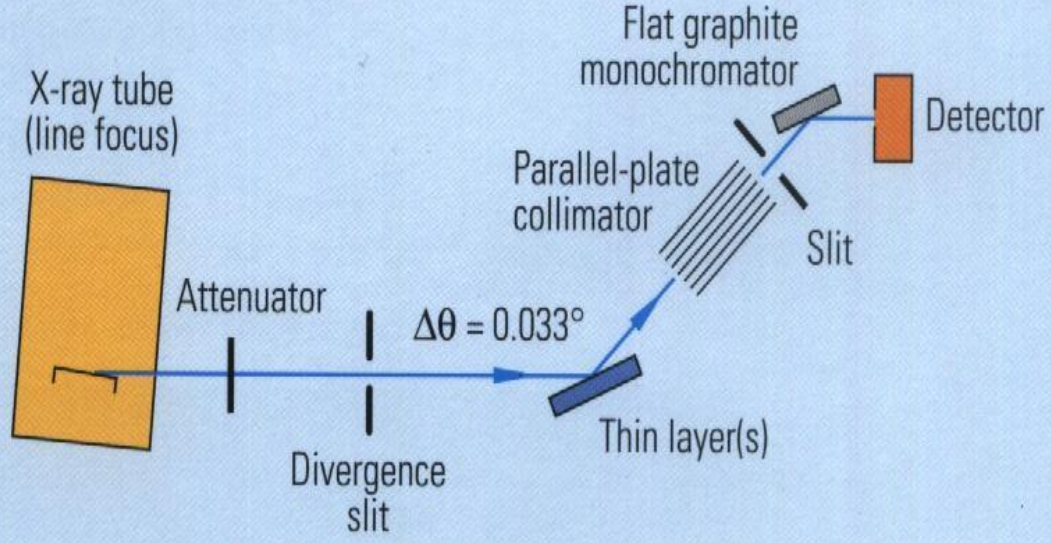
Hybrid reciprocal lattice





Thin layers

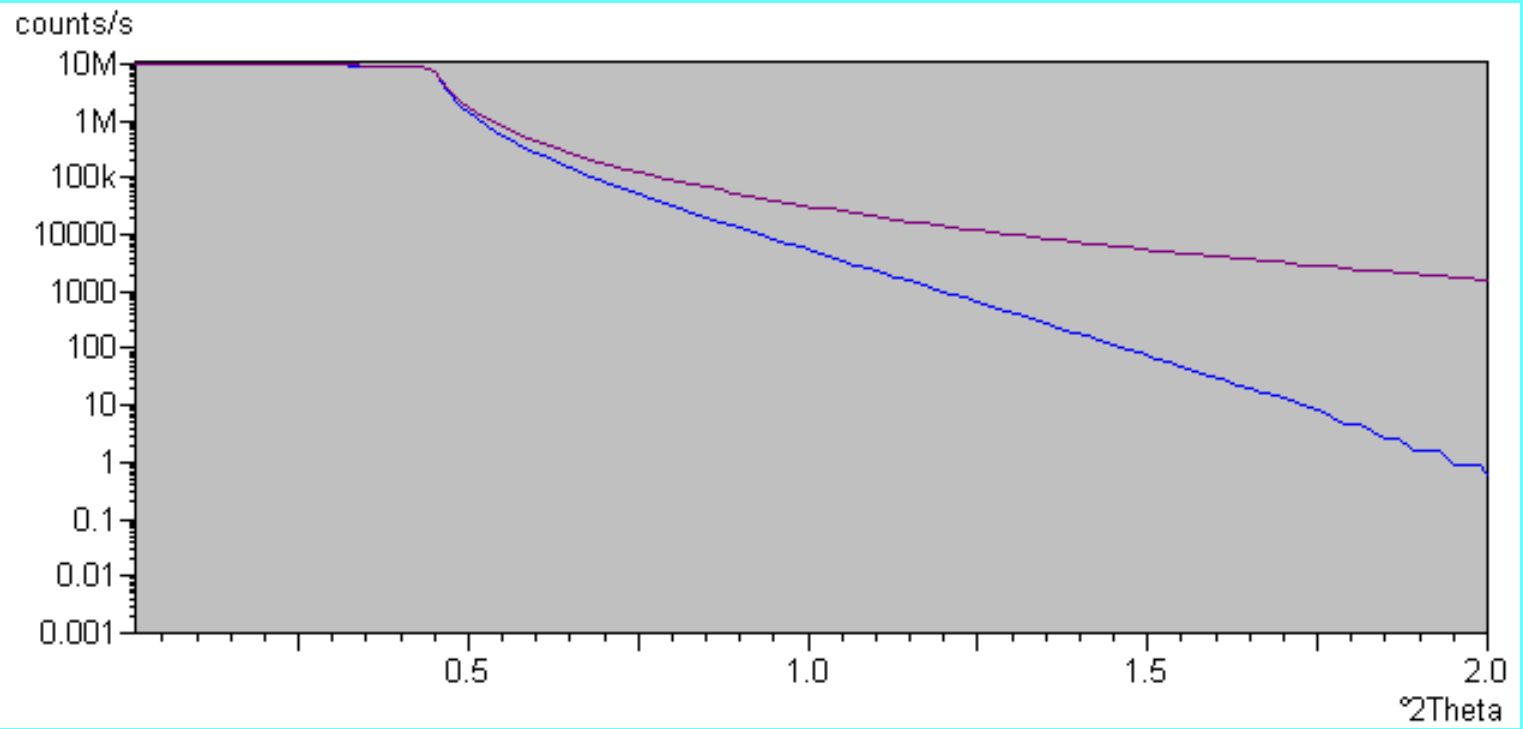




Diffraction

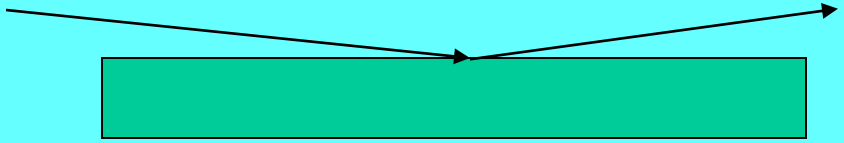
Reflectivity

Reflectivity- surface roughness

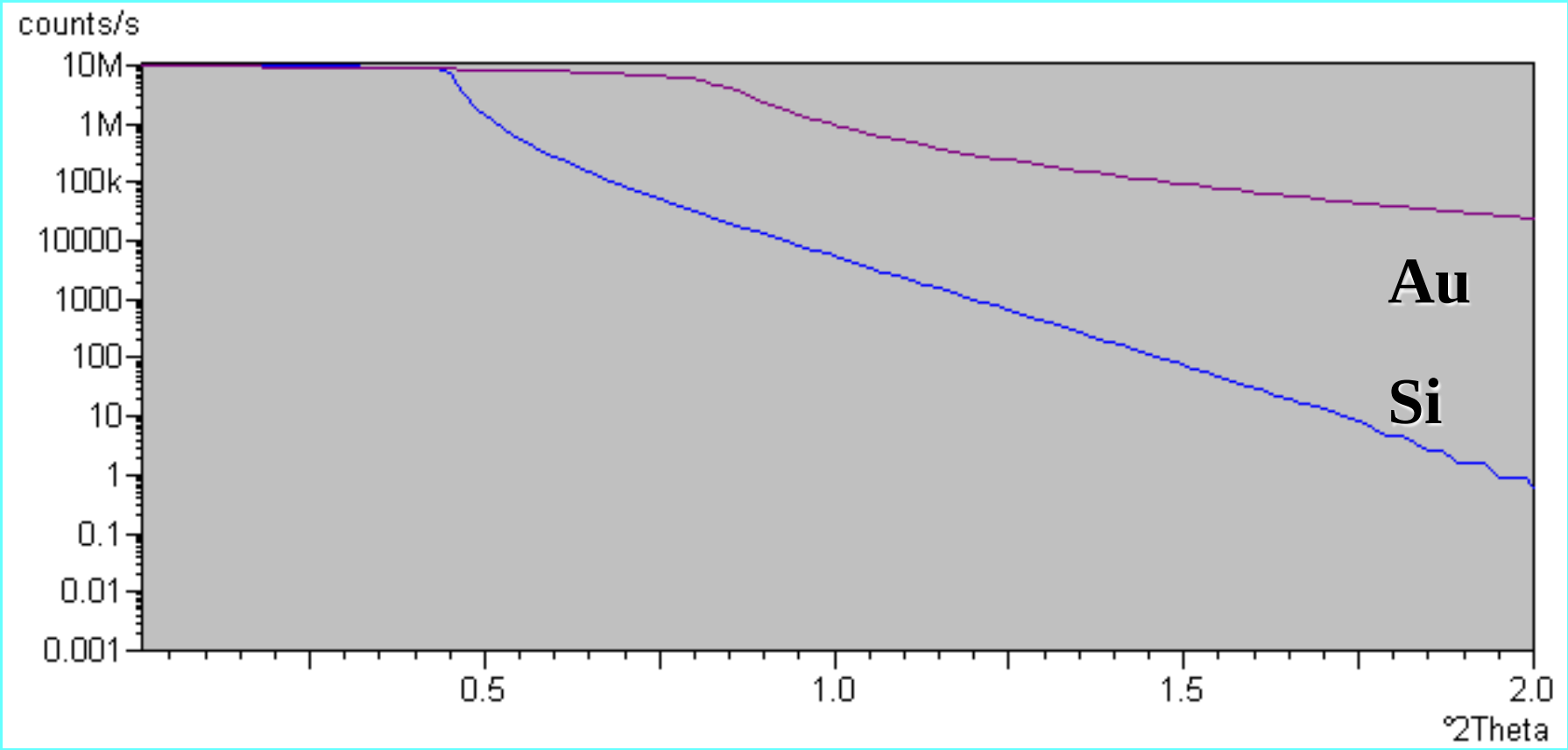


RMS 1A

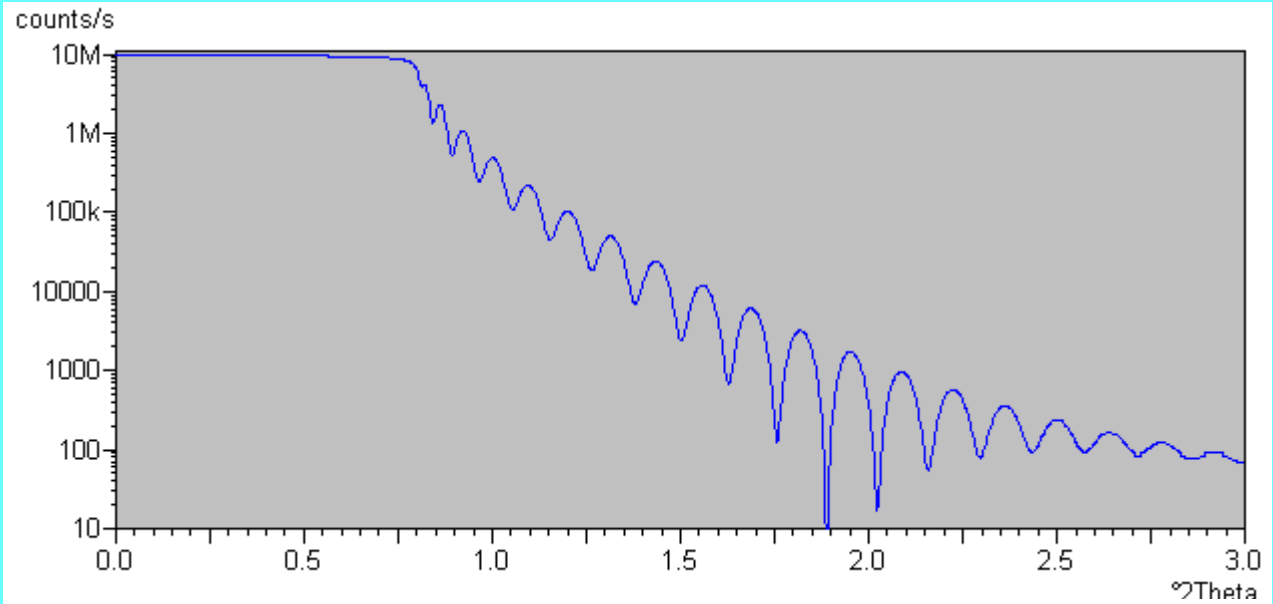
RMS 20A



Reflectivity- density

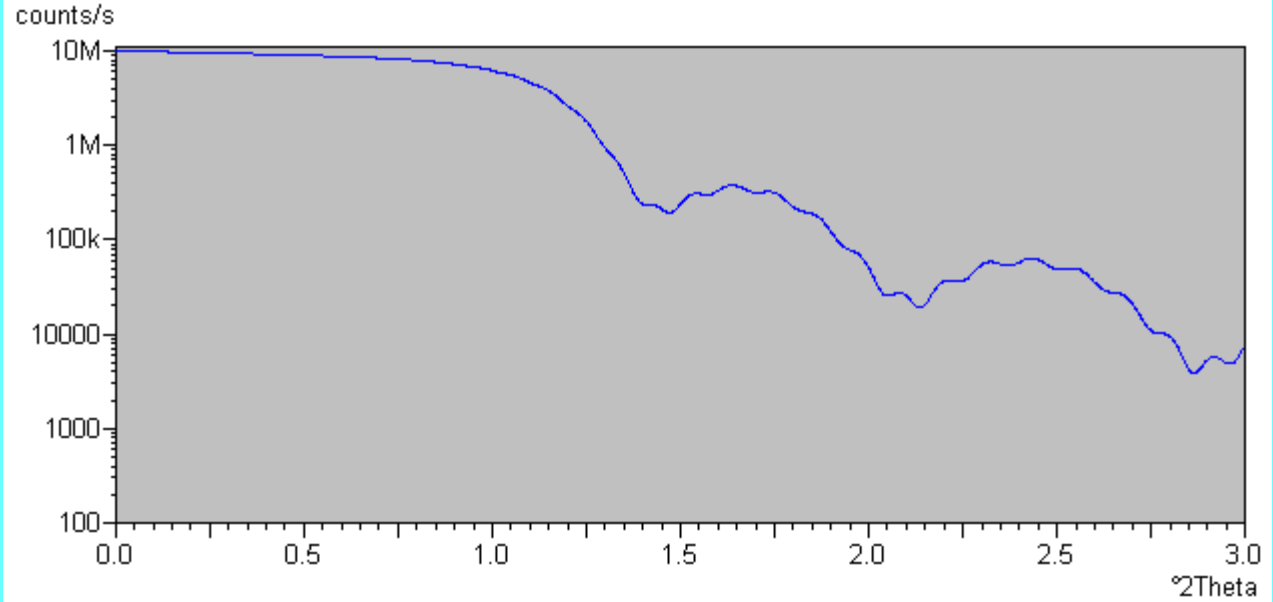


Reflectivity-layer thickness



60 nm Ni

on Si

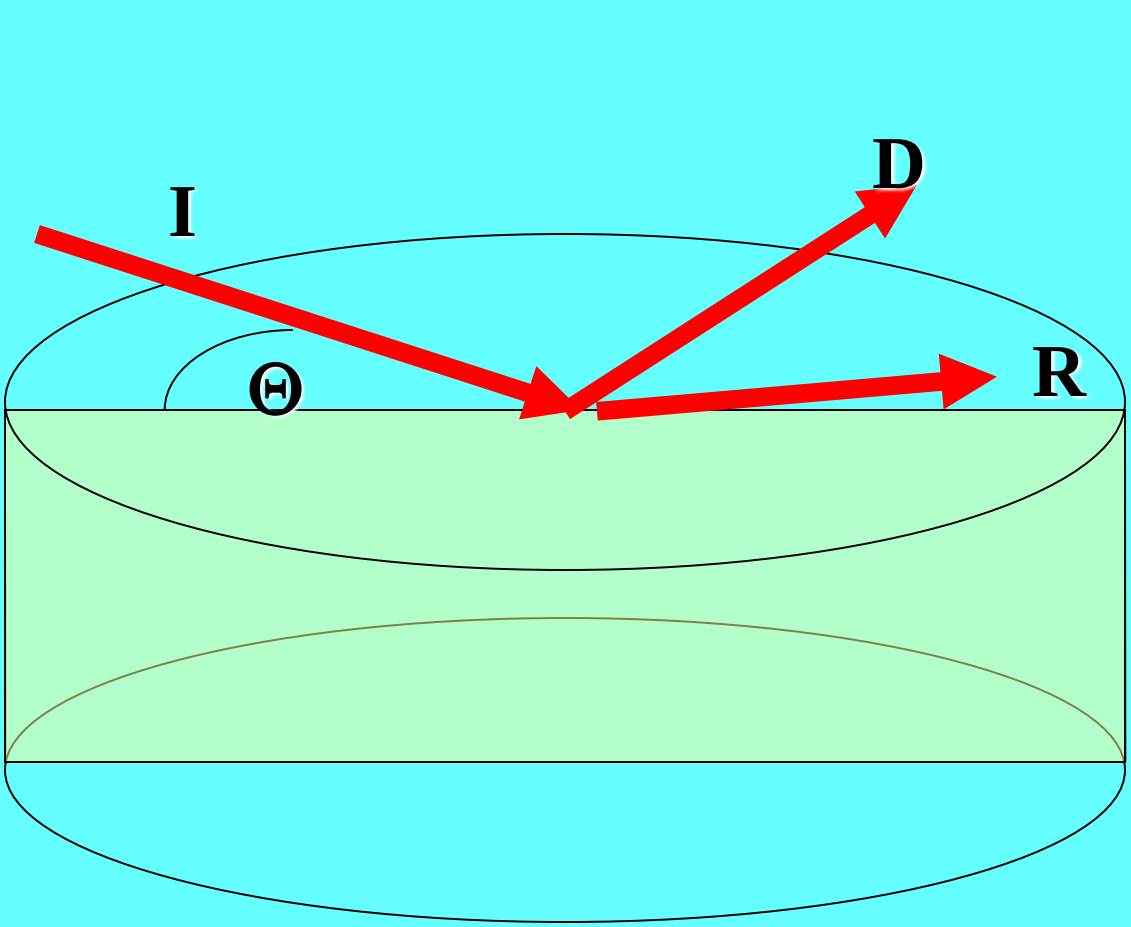


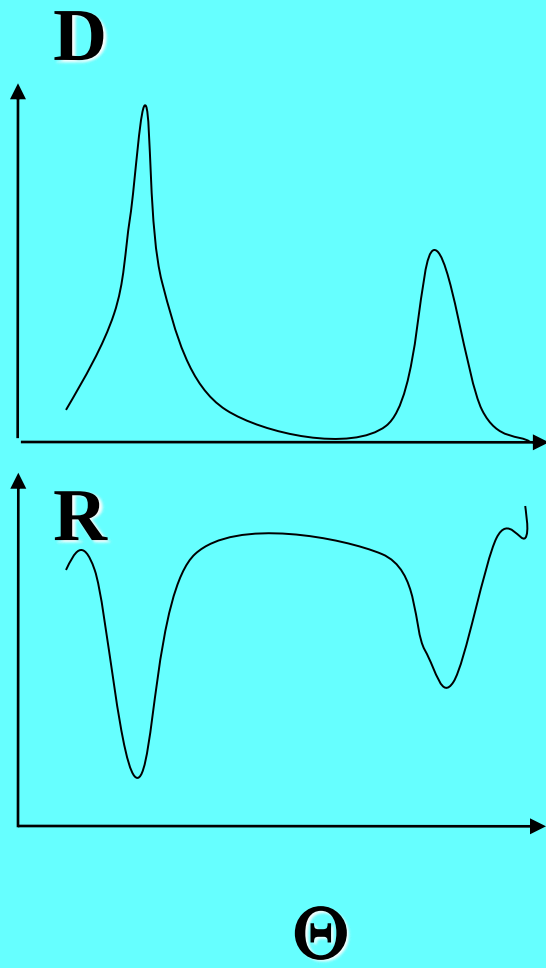
10 nm Au

60 nm Ni

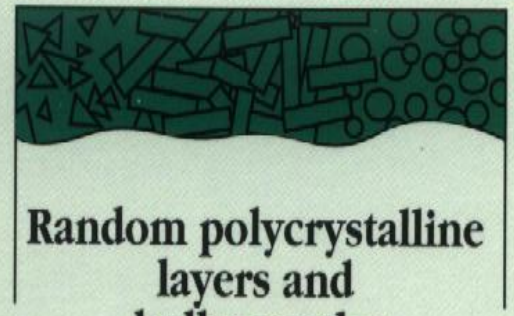
on Si

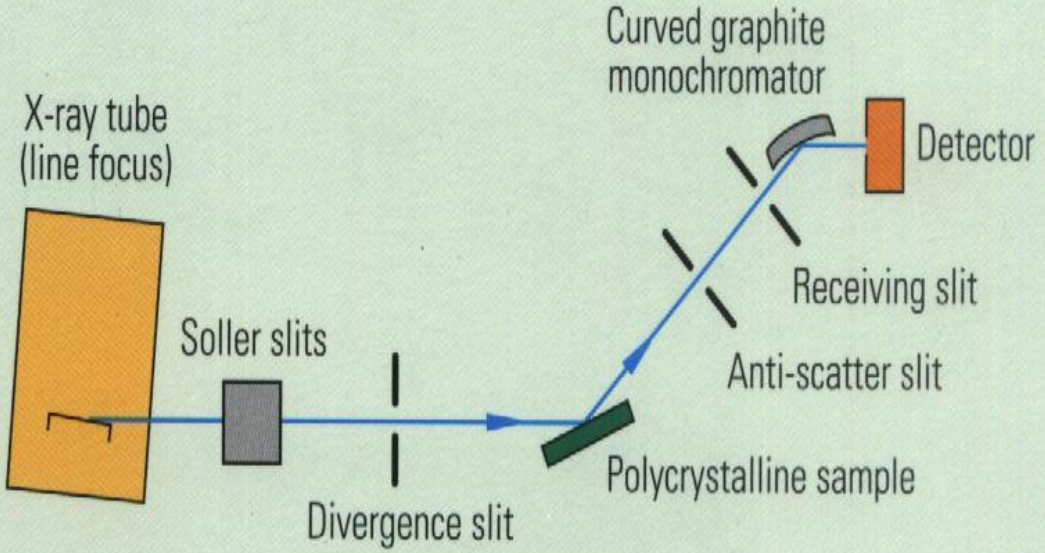
Surface diffraction (grazing incidence)



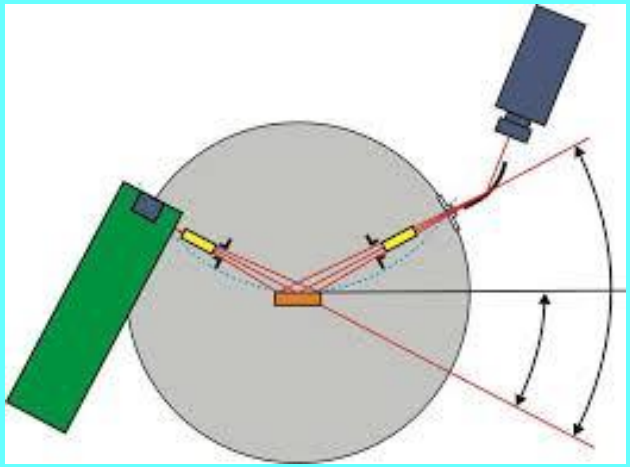


Polycrystalline materials

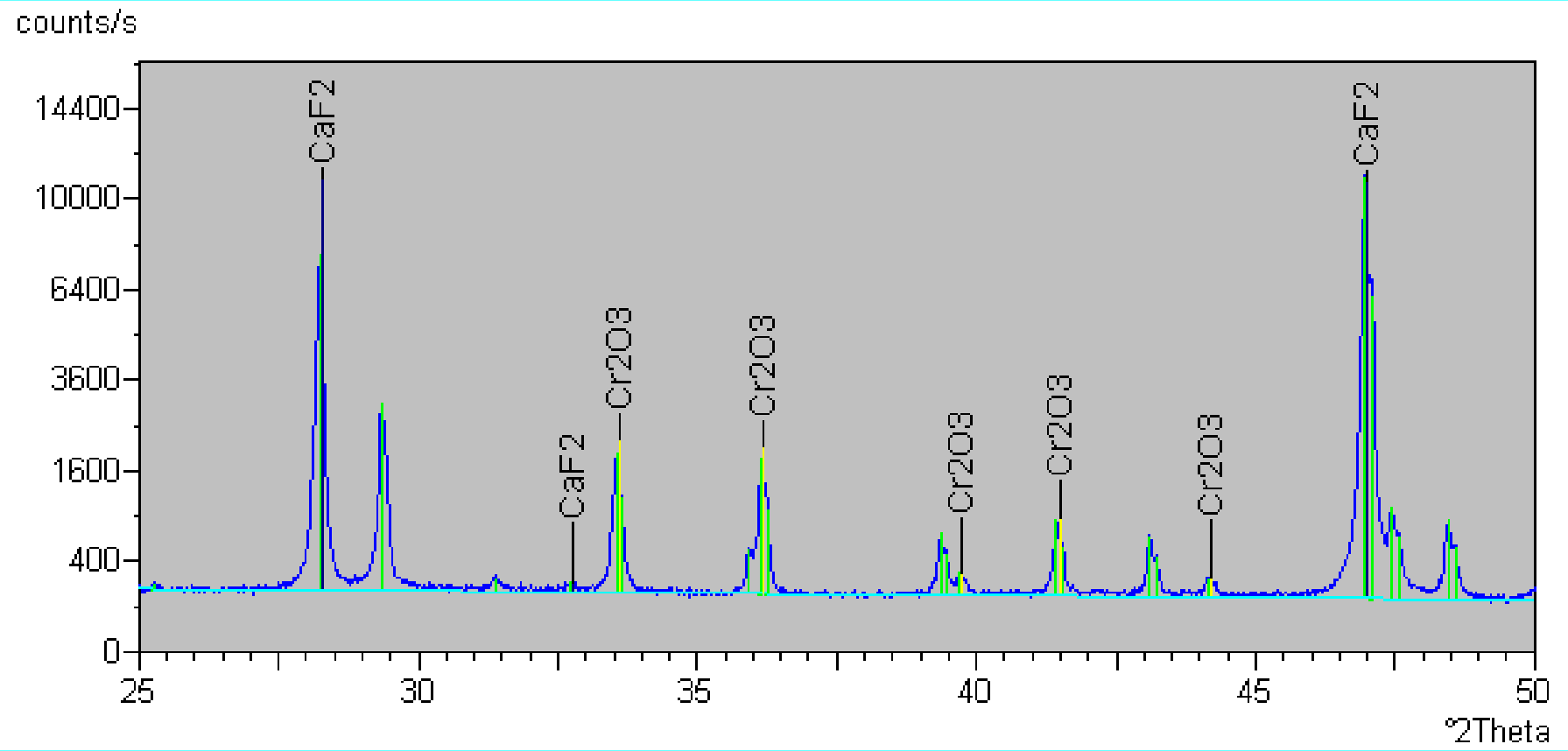




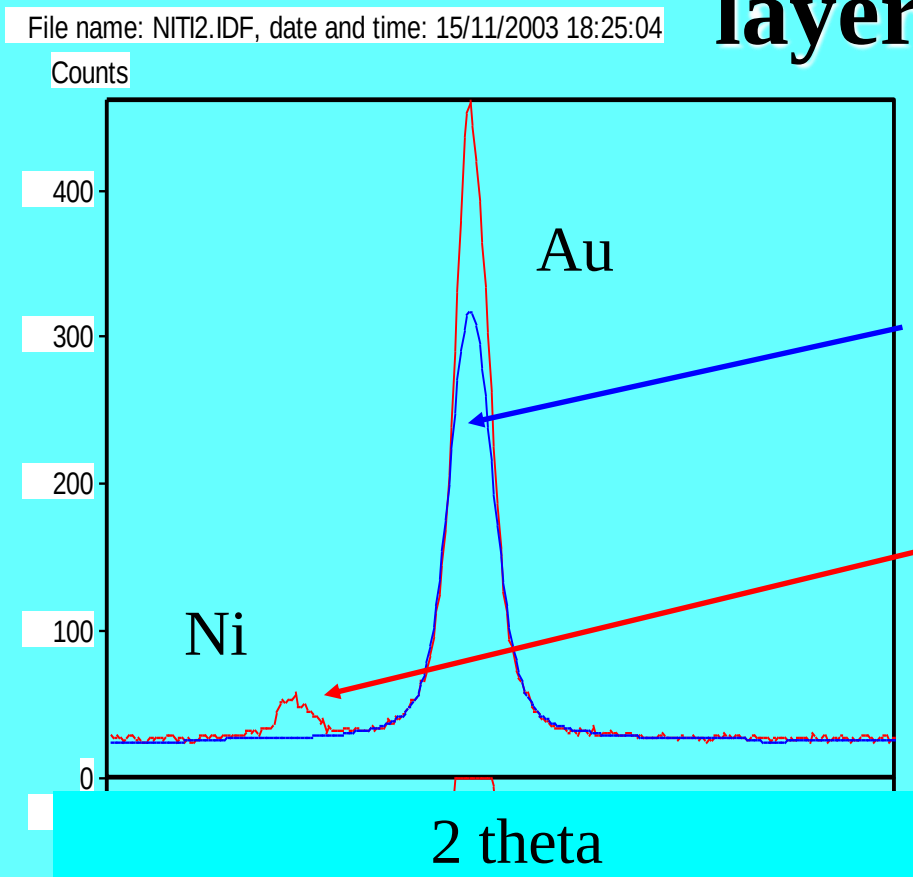
Bragg-Brentano configuration



Powder diffractogram



Diffraction from polycrystalline thin layers

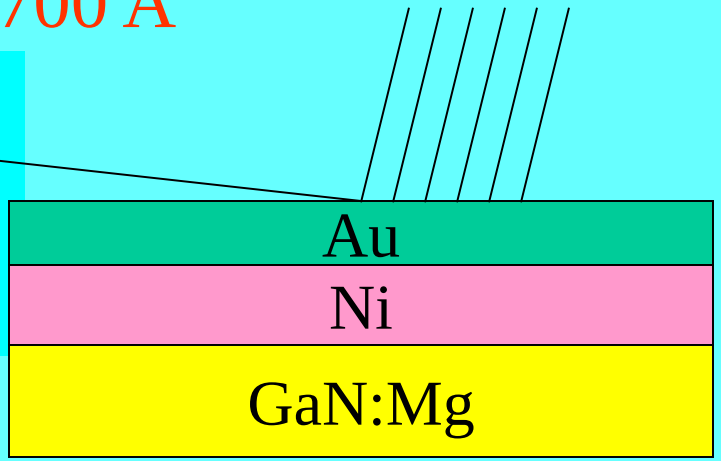


Incidence angle 0.2 deg

Penetration depth about 200 A

Incidence angle 0.6 deg

Penetration depth about 700 A



Information from powder diffractometry

- Phase analysis
- Quantitative analysis (with standards, standardless)
- Grain size
- Strains

OCCHIALI A RAGGI X

Guardando attraverso le particolari lenti, l'effetto ottico che ne risulta vi farà intravvedere... visioni insospettate. Guardandovi le mani ne vedrete lo scheletro, osservando una persona ne scoprirete le fattezze sotto gli abiti.

E045 - Occhiali a Raggi X ..... L. 7.900