

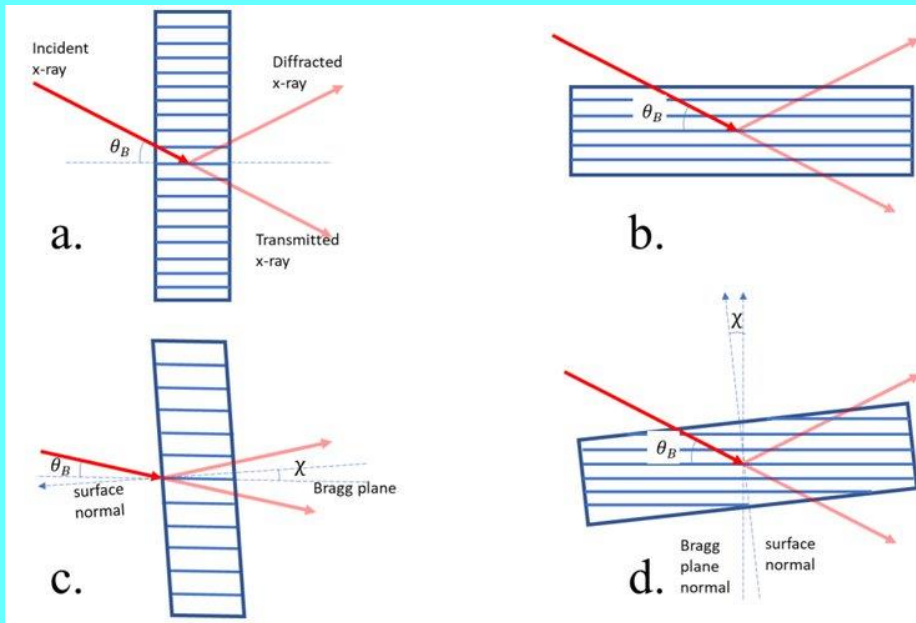
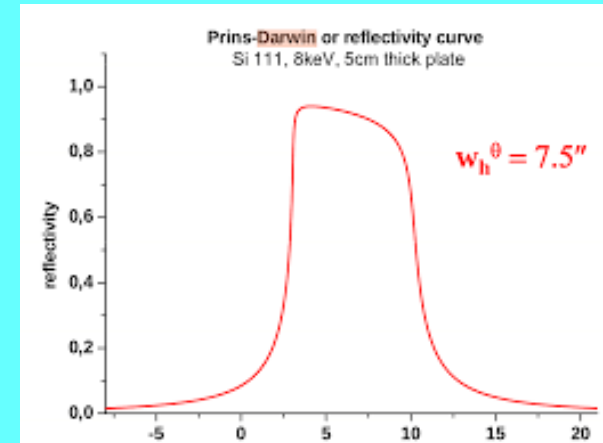
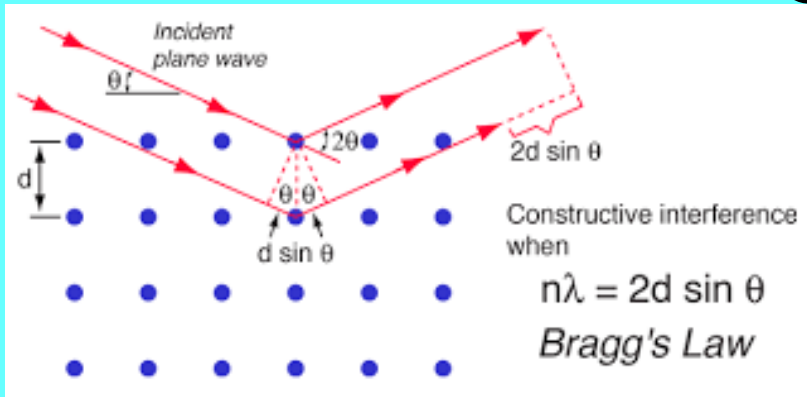
X-ray diffraction and reflectometry in studies of crystals

Michał Leszczyński

Instytut Wysokich Ciśnień i TopGaN

Lecture 22 Apr 2022

Bragg's law



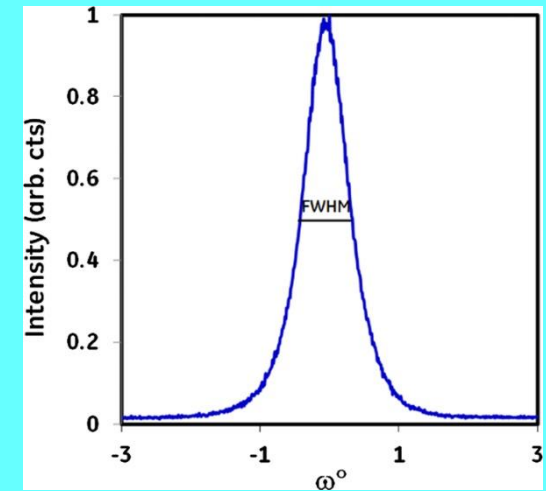
Symmetric

Asymmetric

Transmission
(Bragg case)

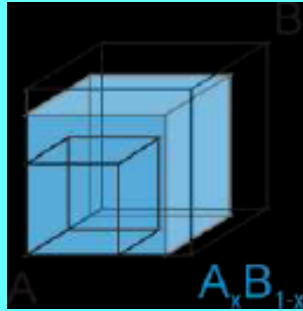
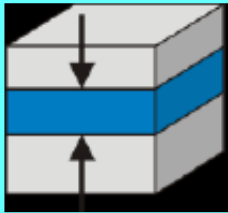
Reflection
(Laue case)

Bragg case



Laue case

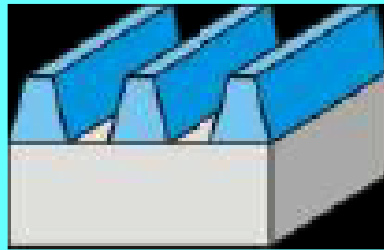
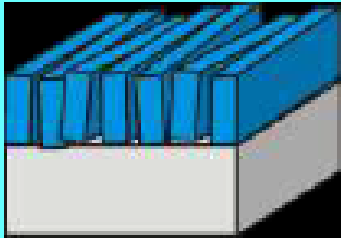
Analytical tasks



Layer thickness

Chemical composition

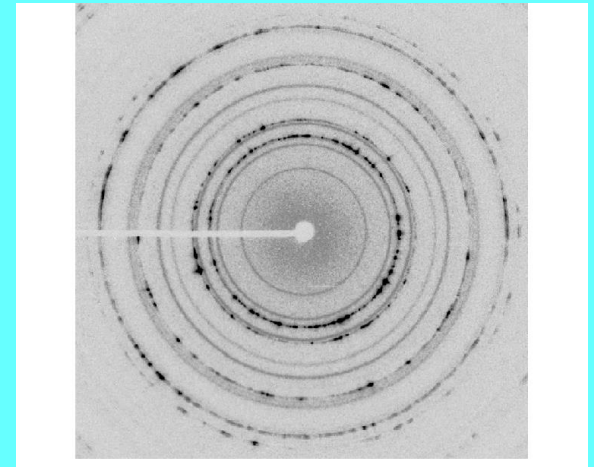
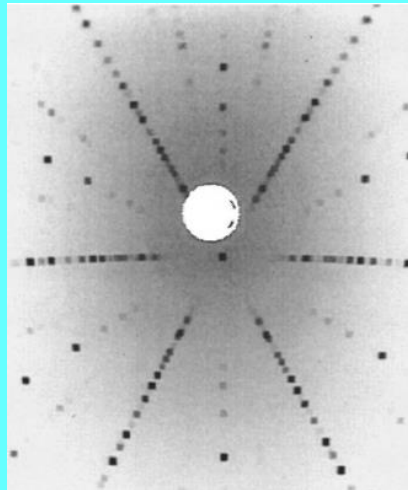
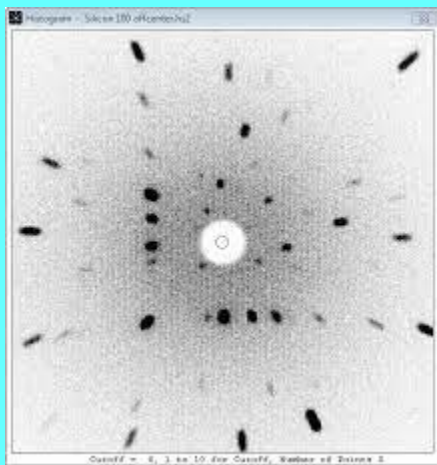
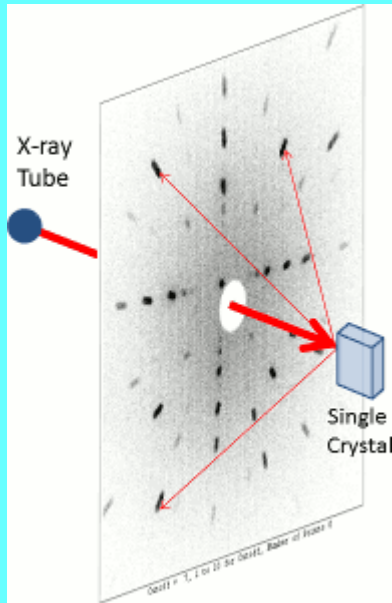
Lattice relaxation



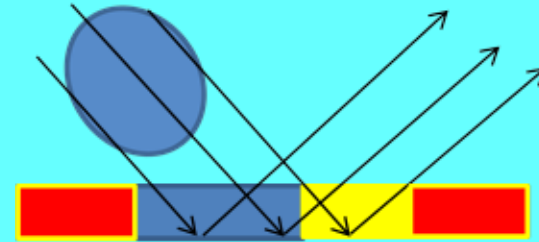
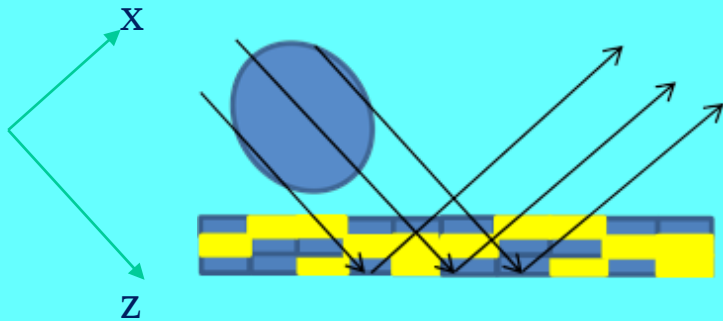
Defects and crystal size

Lateral structures

Laue camera (white beam)



Photon coherence



We add amplitudes $(\sin + \sin + \sin + \dots)^2$ We add intensities $(\sin^2 + \sin^2 + \sin^2 + \dots)$

Lz $1.5 \mu\text{m}$.

Ly $0.5\text{-}5 \mu\text{m}$

Ly $10\text{-}100 \text{ nm}!!!$

History

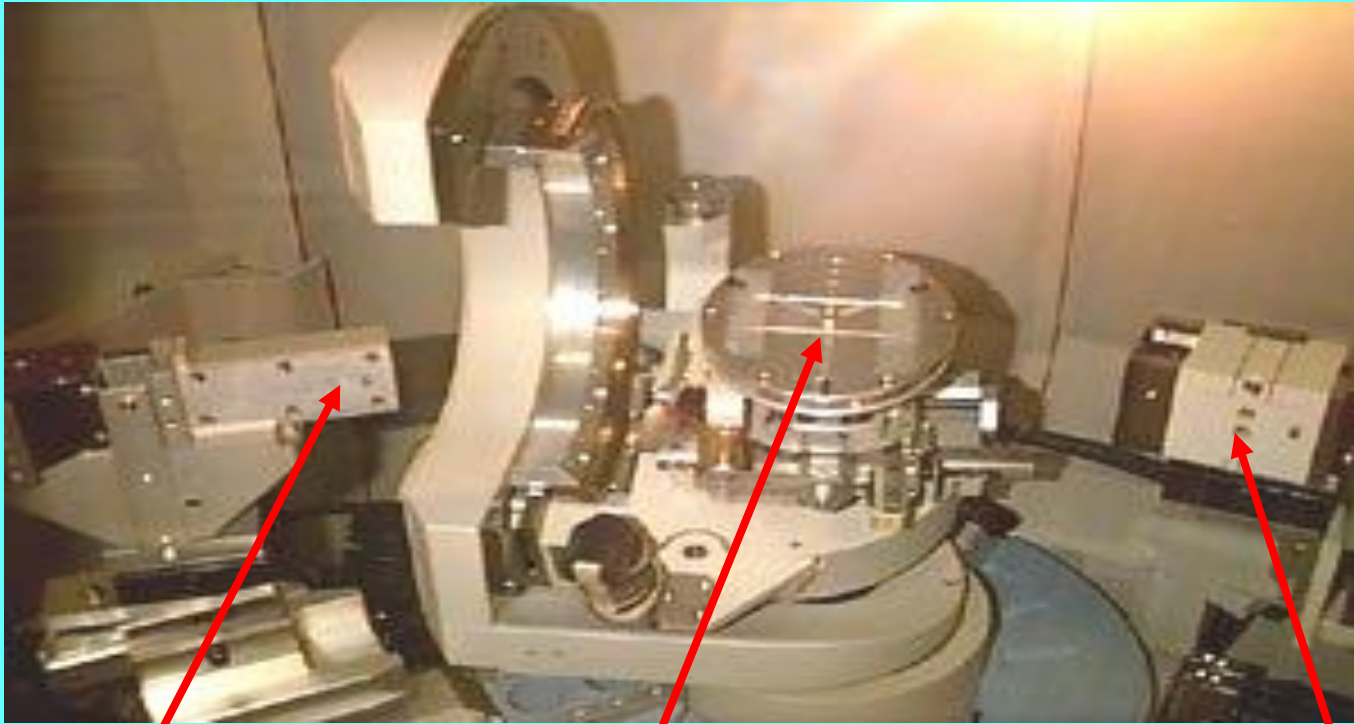
1912- first observation of XRD: Max von Laue

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

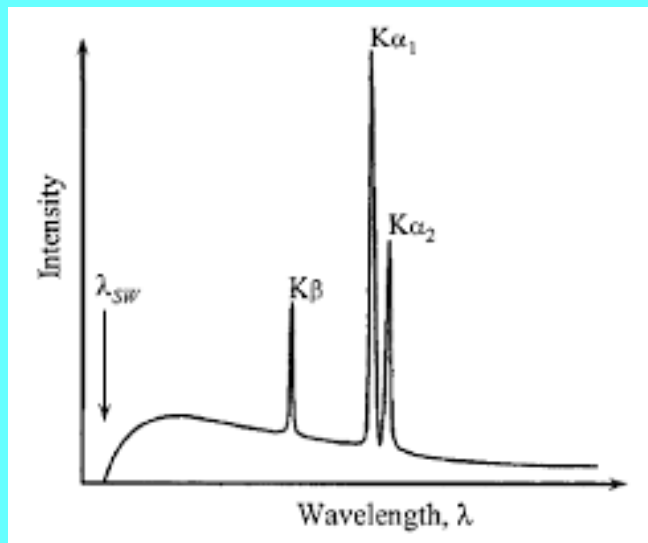
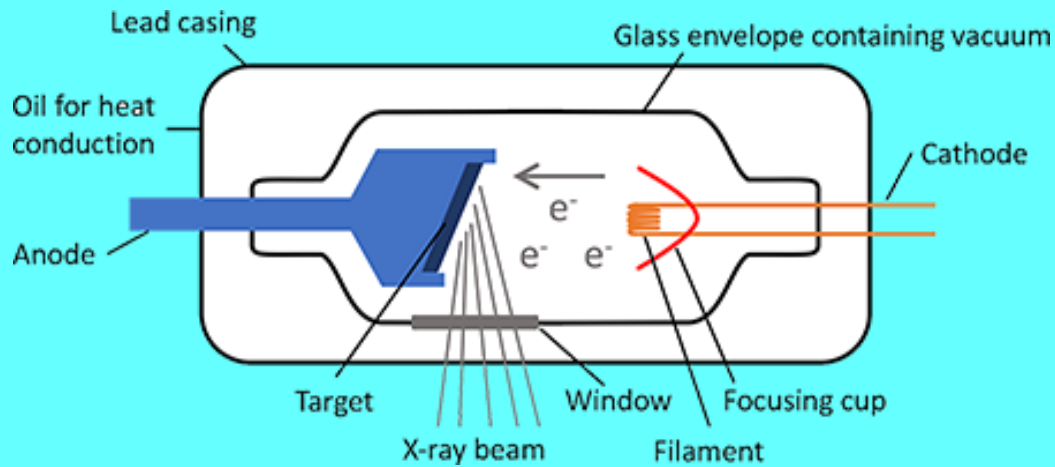


Primary beam

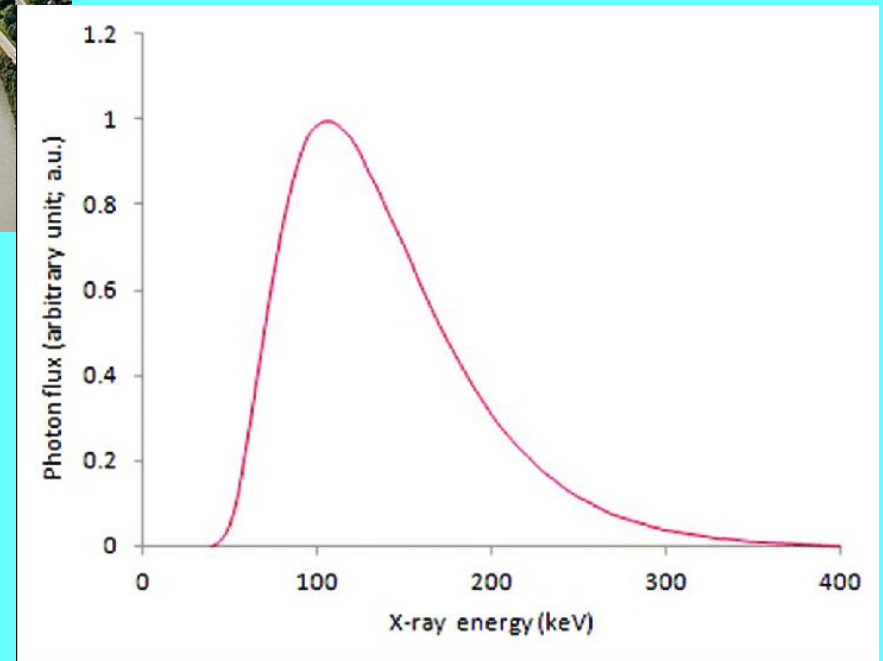
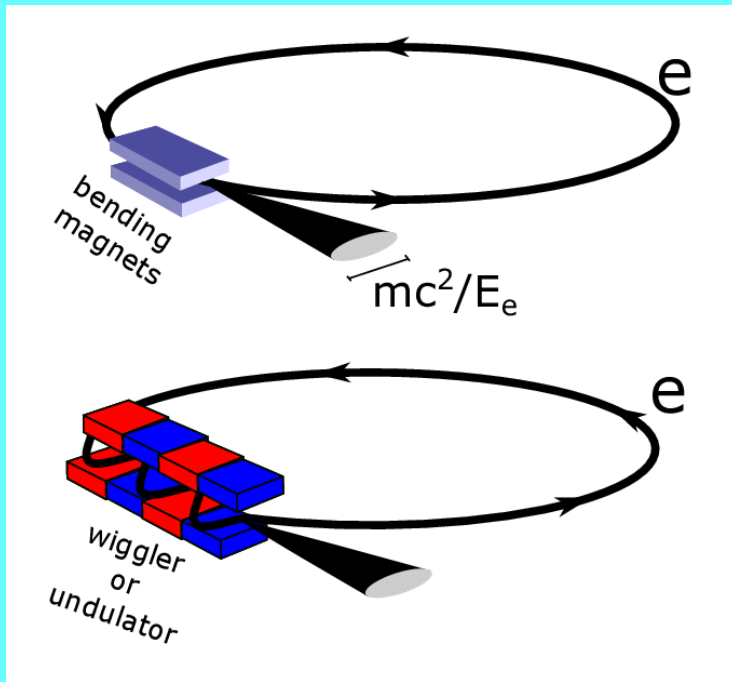
**Goniometer
head**

Reflected beam

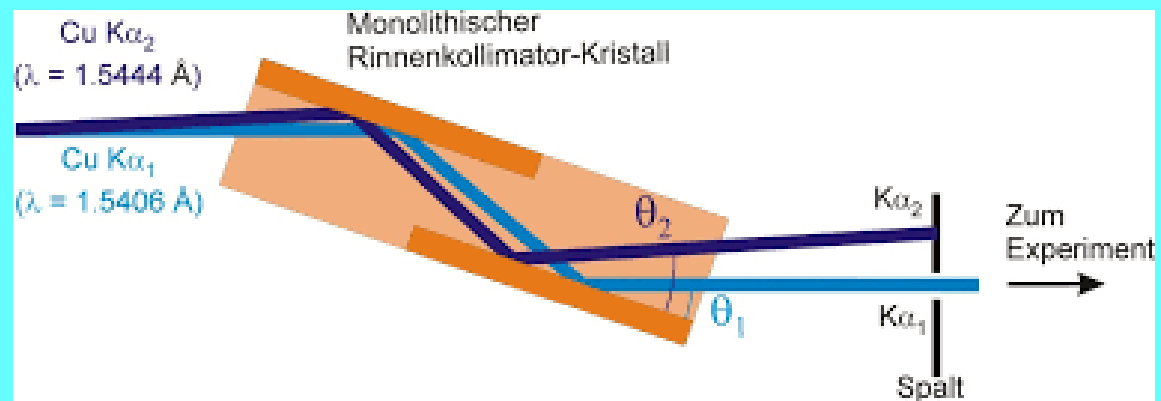
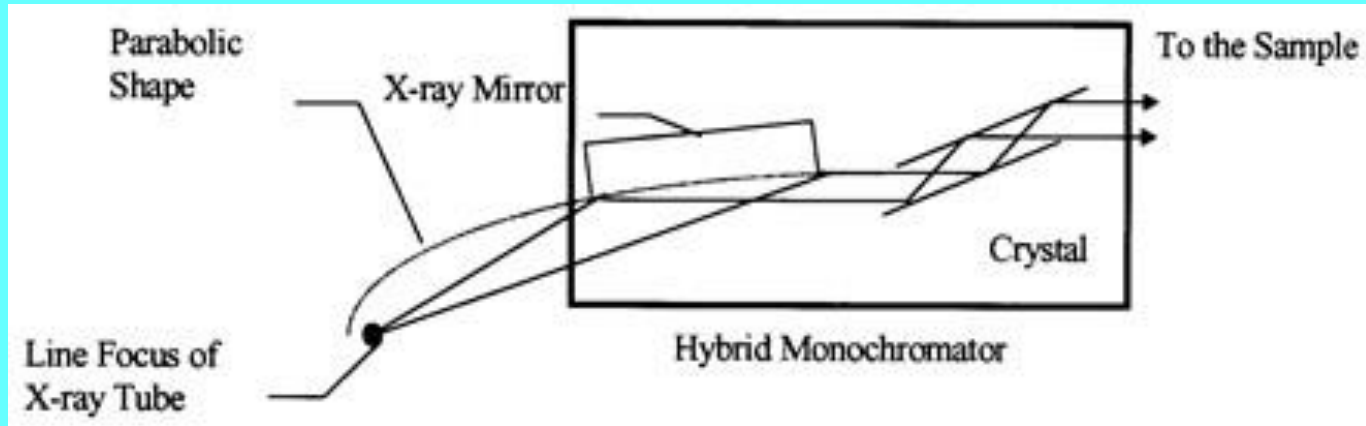
X-ray tube



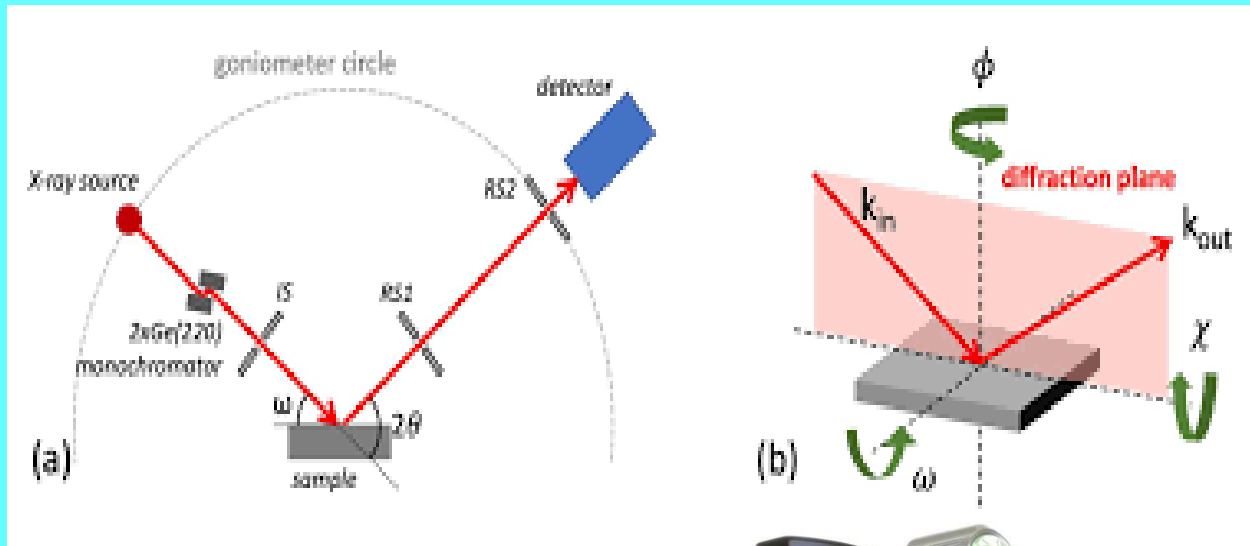
Synchrotron radiation



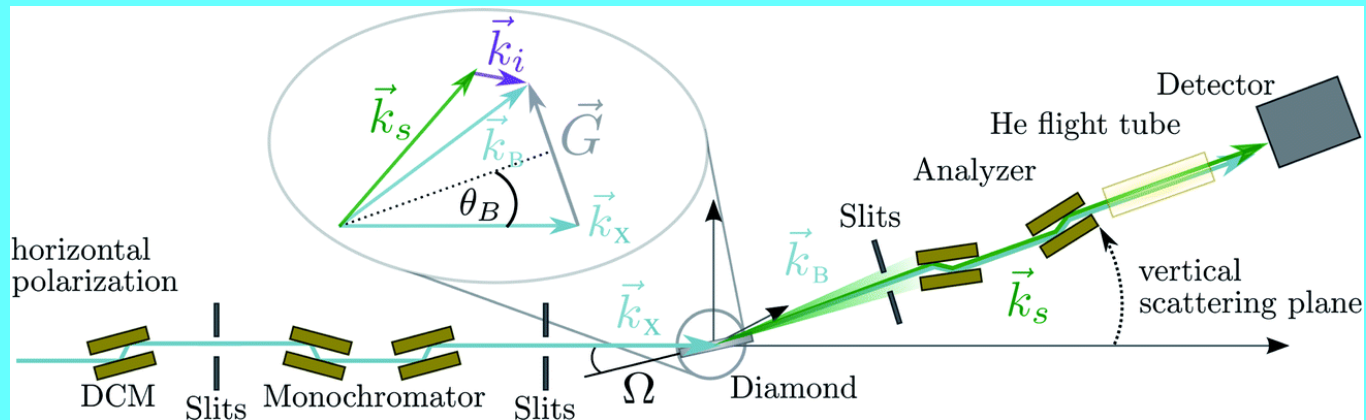
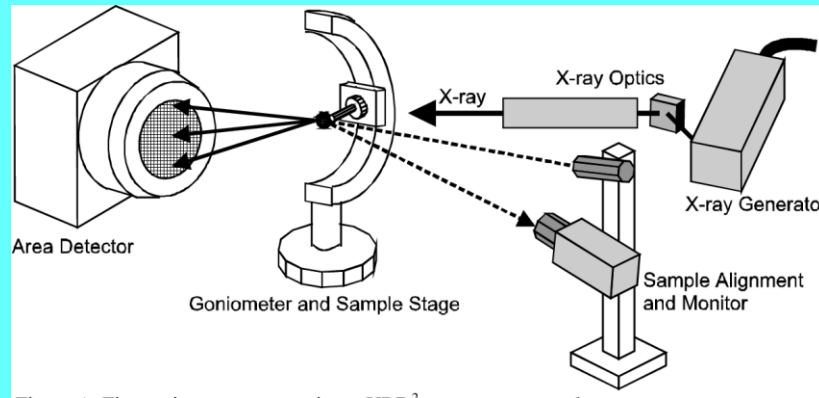
Monochromator



Goniometer head

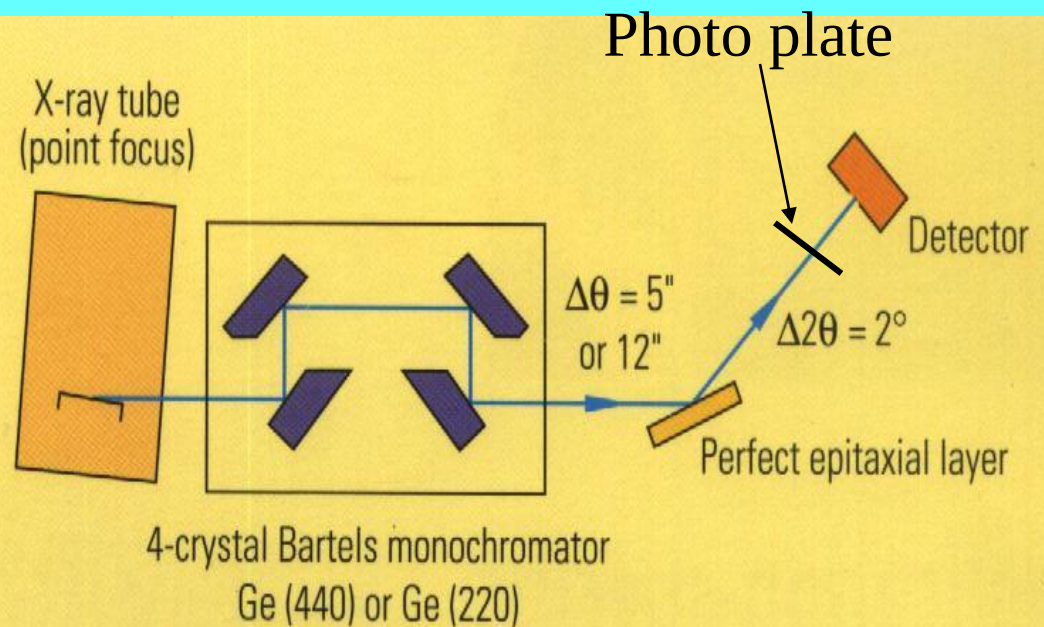
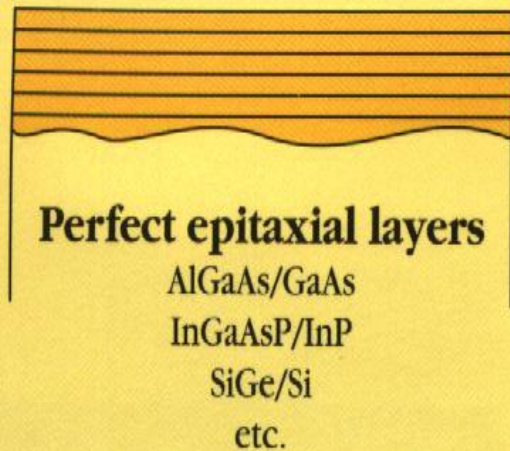


Analyzers and detectors

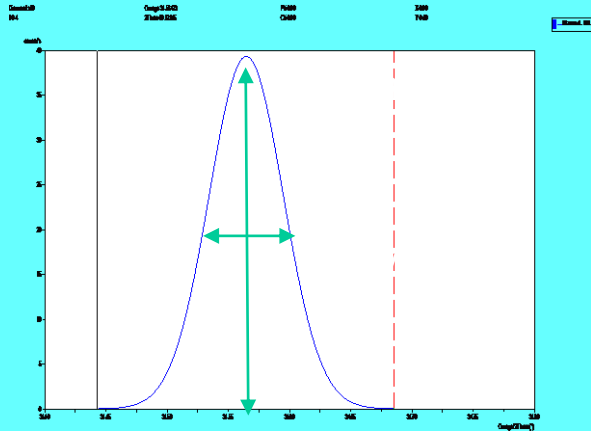


Double axis, double crystal, rocking curve configuration

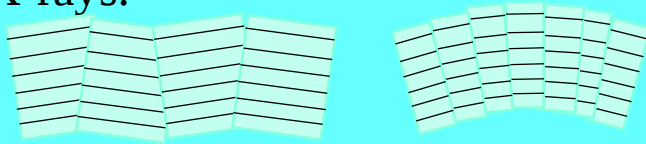
Sensitive to mosaicity and lattice parameters variations



Rocking curve as a measure of GaN crystallographic quality



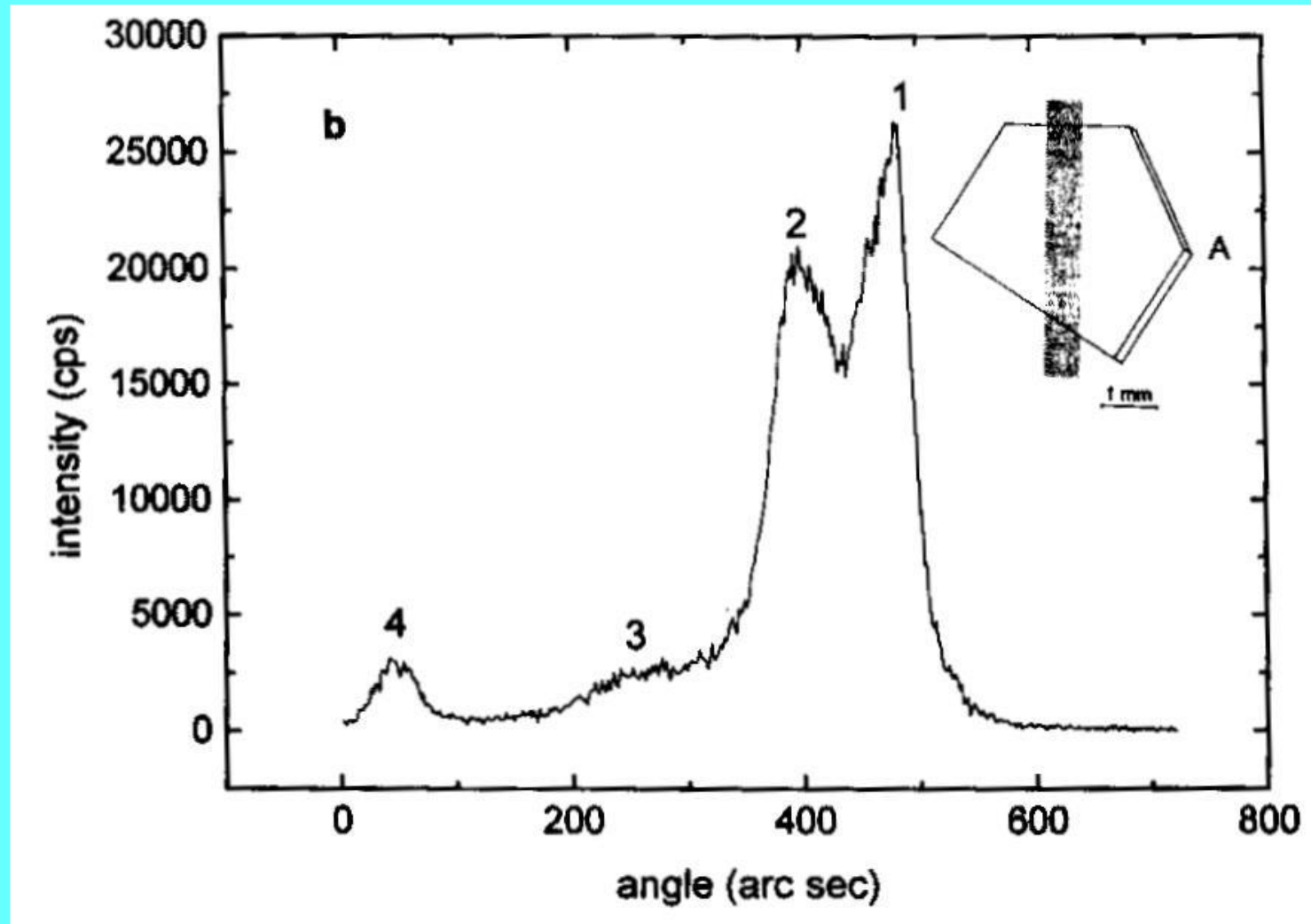
Always compare FWHM and intensities for various reflections (also asymmetrical ones), as well as different area illuminated with X-rays.



Small size of crystallites- additional peak broadening!!!

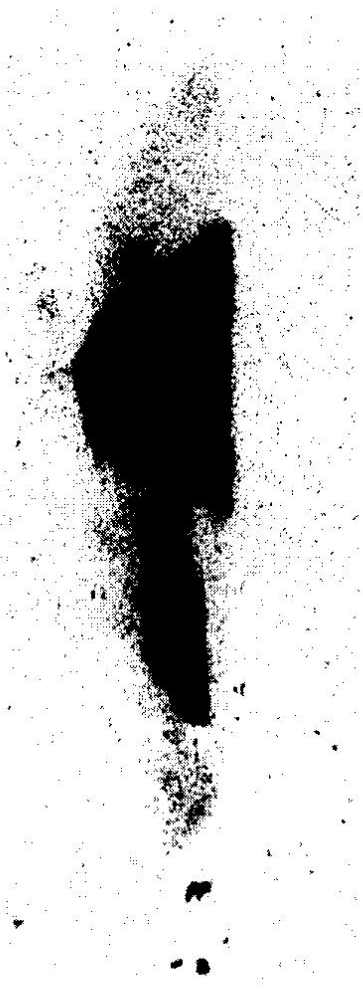
GaN crystal (dislocation density cm ⁻²)	FWHM 00.2 (arc deg)	Intensity 00.2 (Mcps)
A 2x10exp8	0.087	28
B 8x10exp6	0.030	29
C 1x10exp6	0.038	30
D 5x10exp4	0.015	32
GaN crystal	FWHM 00.4 (arc deg)	Intensity 00.4 (Mcps)
A	0.081	4
B	0.029	12
C	0.023	15
D	0.015	21

Mosaic structure of HP GaN crystals

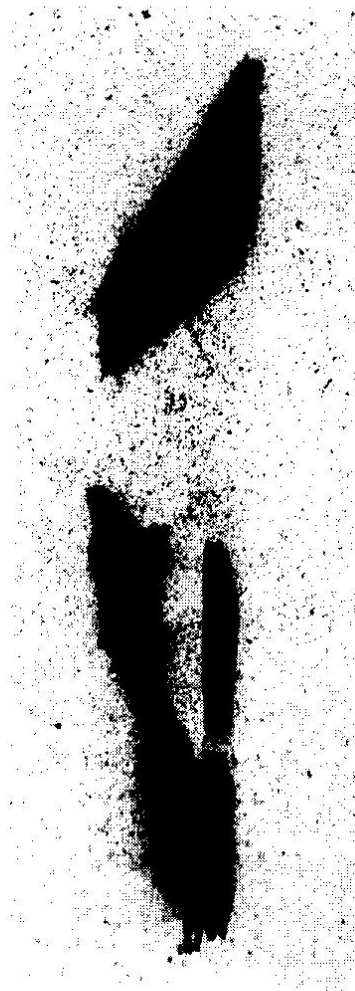


Topografia kryształu GaN

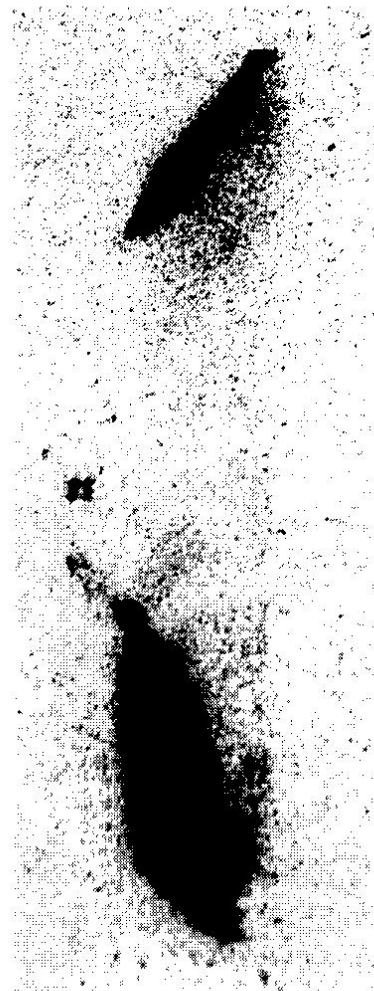
1



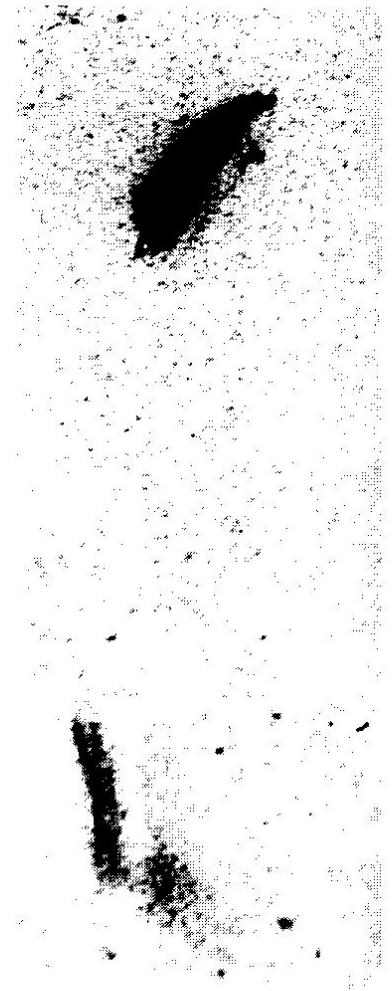
2



3



4



Information from the rocking curves and topography

- Bulk crystals(GaAs, Si, InP, i in.):
 - i) mosaicity (dislocation density higher than 10^6 cm^{-2}),
 - ii) bowing,
 - iii) off-orientation
- Epi wafers:
 - i) thickness (+/- 2-5 Å)
 - ii) chemical composition of ternary compounds (+/-1%)

EL2-like defects

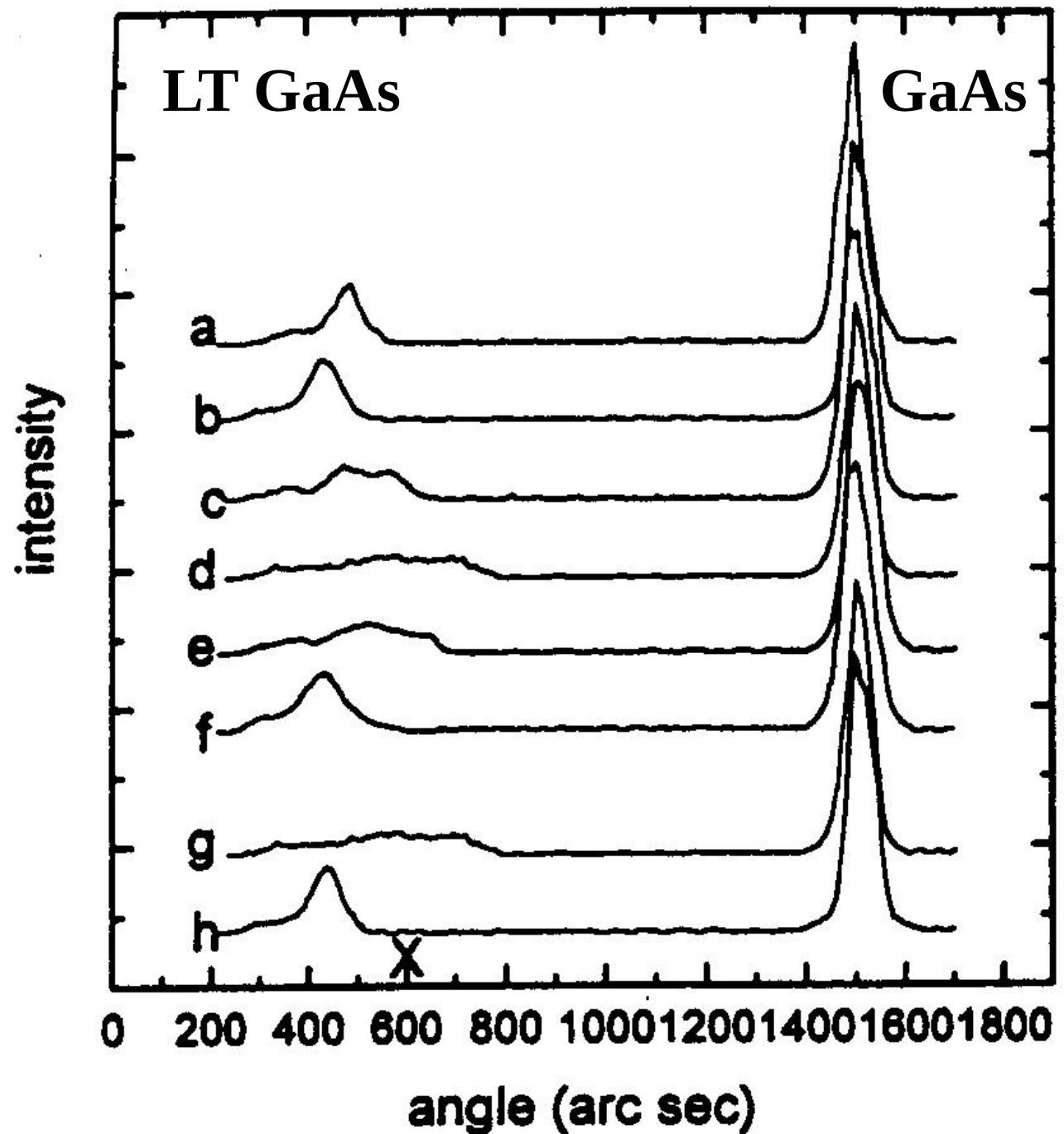
300 K

77 K dark

77 K + 900 nm

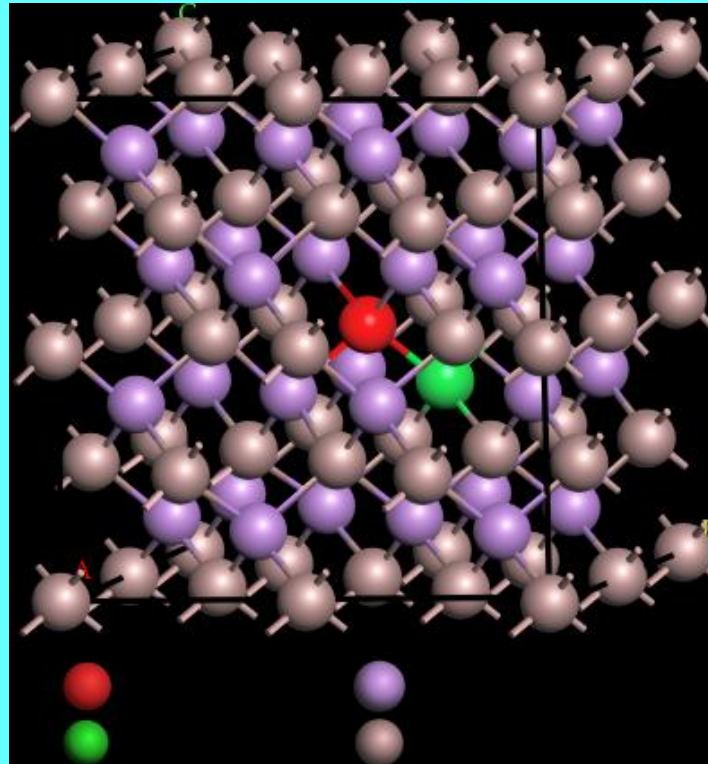
+1350 nm

Or +140 K





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



Theory

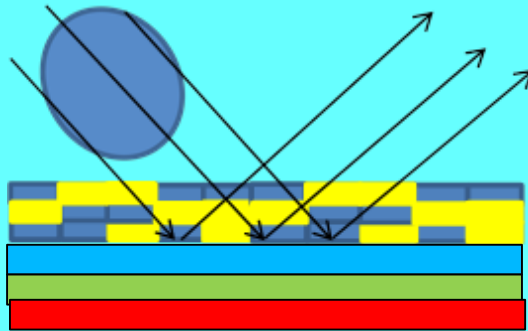
Kinematical theory

No rescattered radiation is taken into account

Dynamical theory

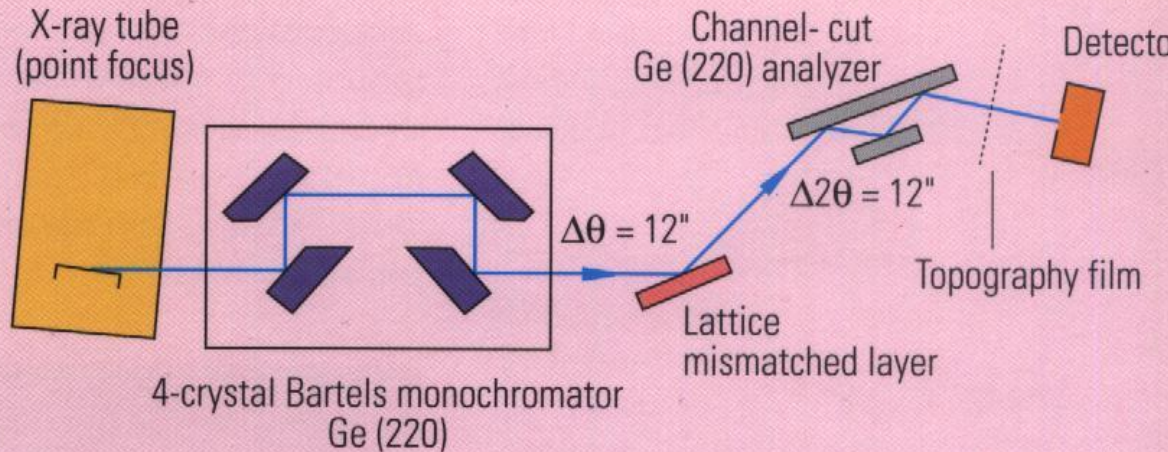
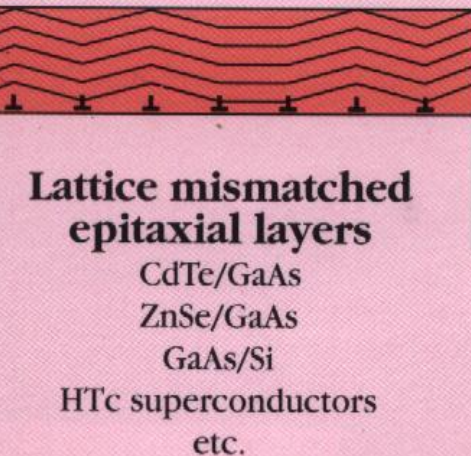
Based on Maxwell radiation

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

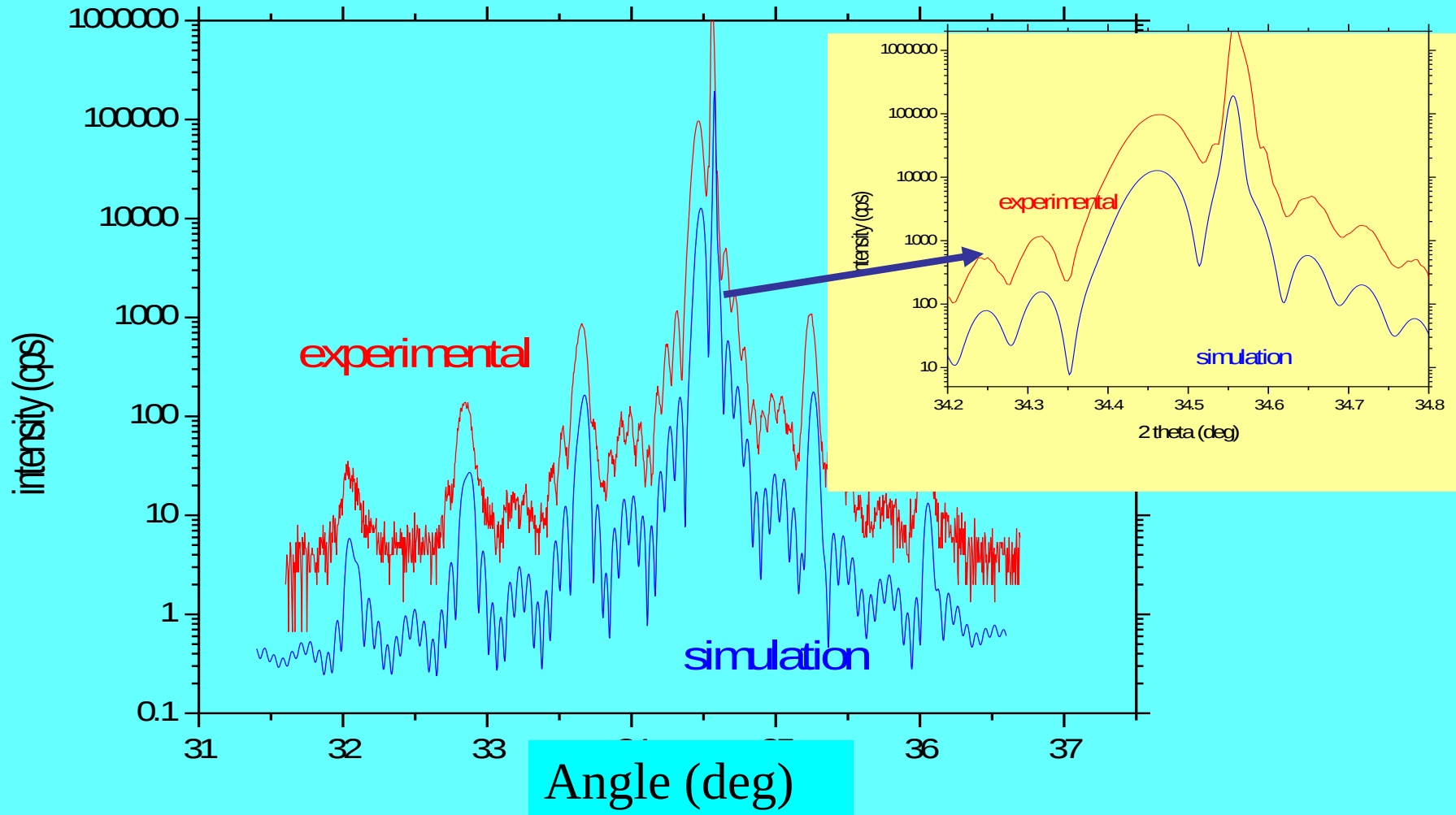


Triple axis

Sensitive to lattice parameters variations

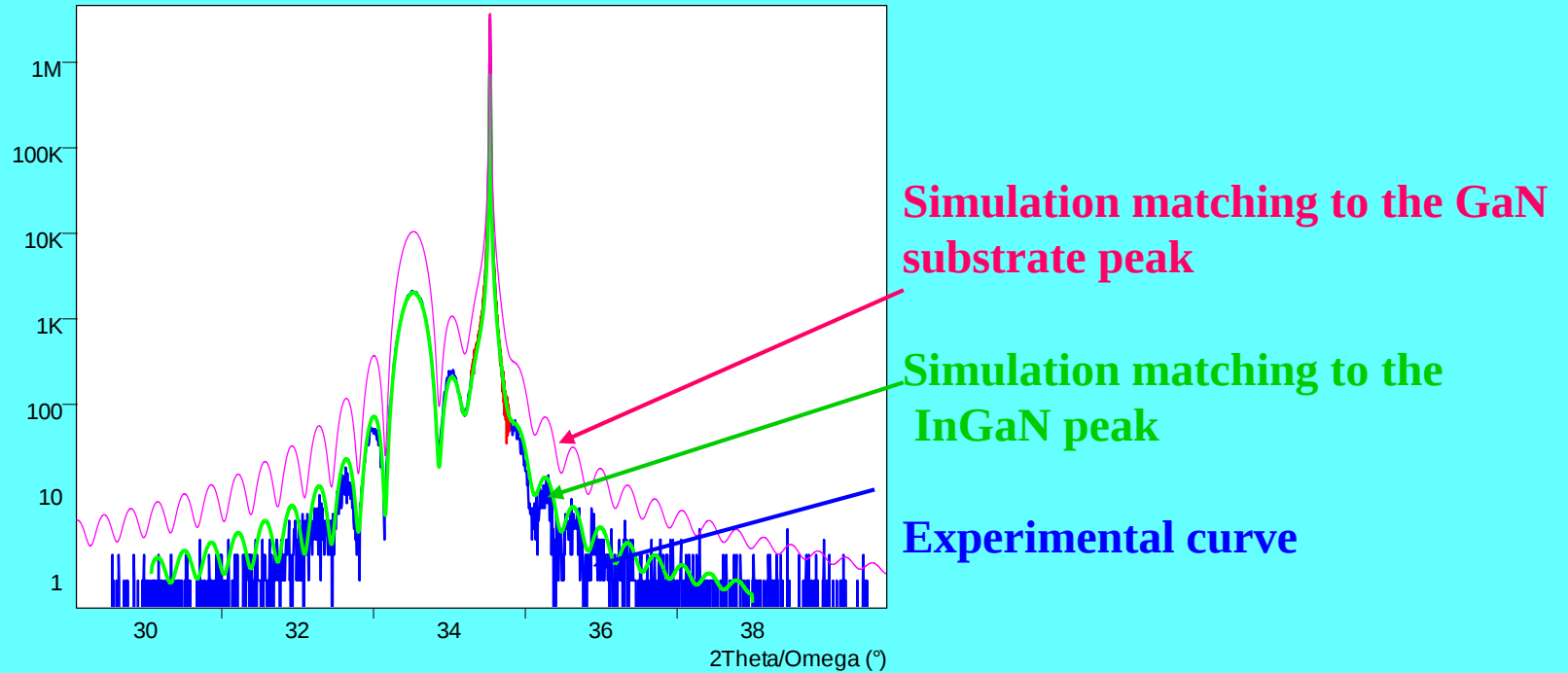


10-fold InGaN/GaN

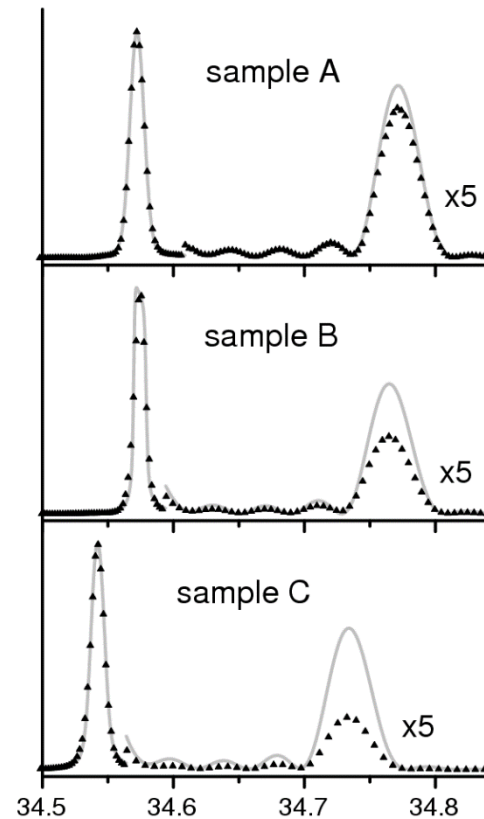
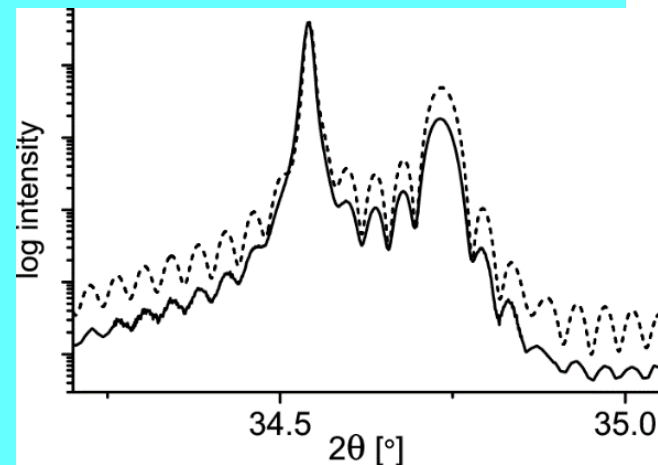


$d(\text{well})=3.2 \text{ nm}$, $d(\text{barrier})=7.1 \text{ nm}$, $x_{\text{average}}=3.2\%$

2theta/omega for InGaN layer on GaN/sapphire



AlGaN layers on different substrates

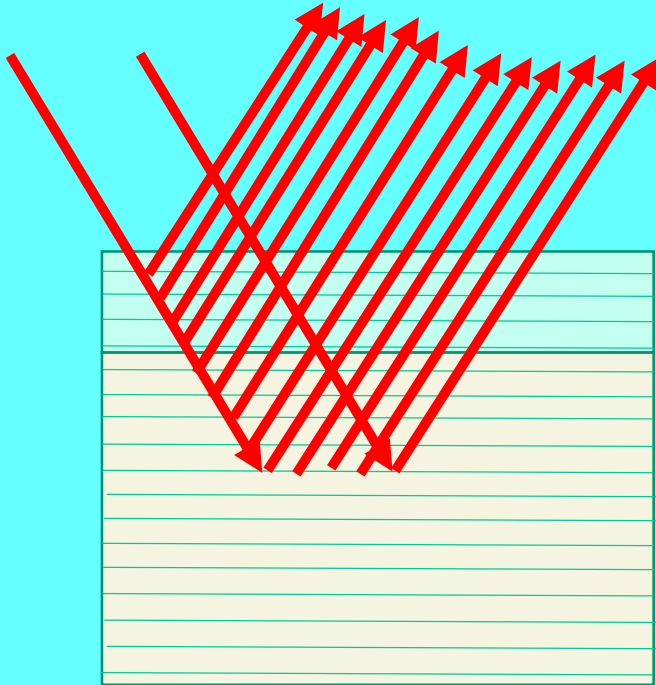


Ammono GaN substrate
EPD $10^4/\text{cm}^2$
87% of AlGaN peak intensity

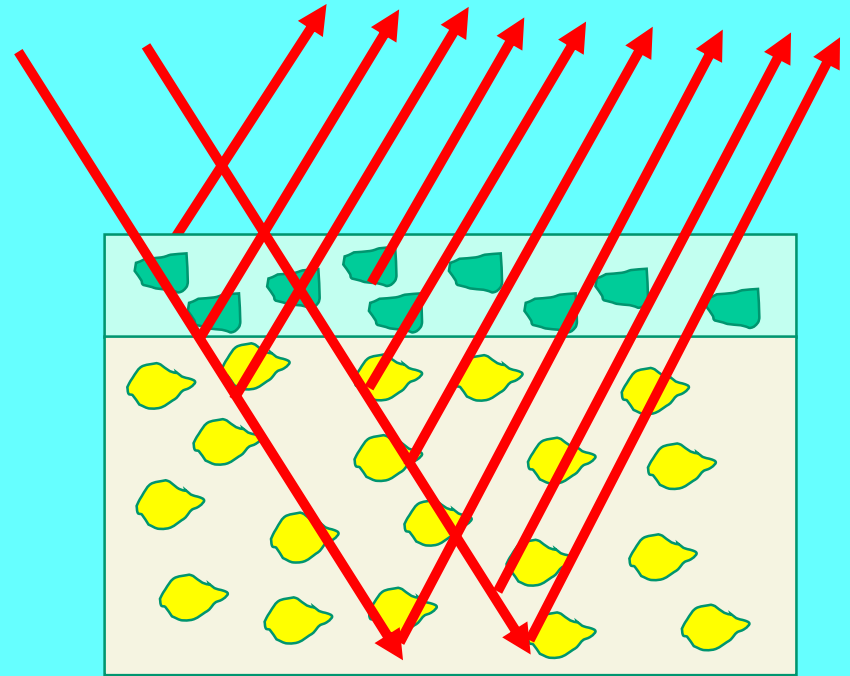
HVPE GaN substrate
EPD $10^7/\text{cm}^2$
60%

GaN on sapphire
EPD $10^8/\text{cm}^2$
35%

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

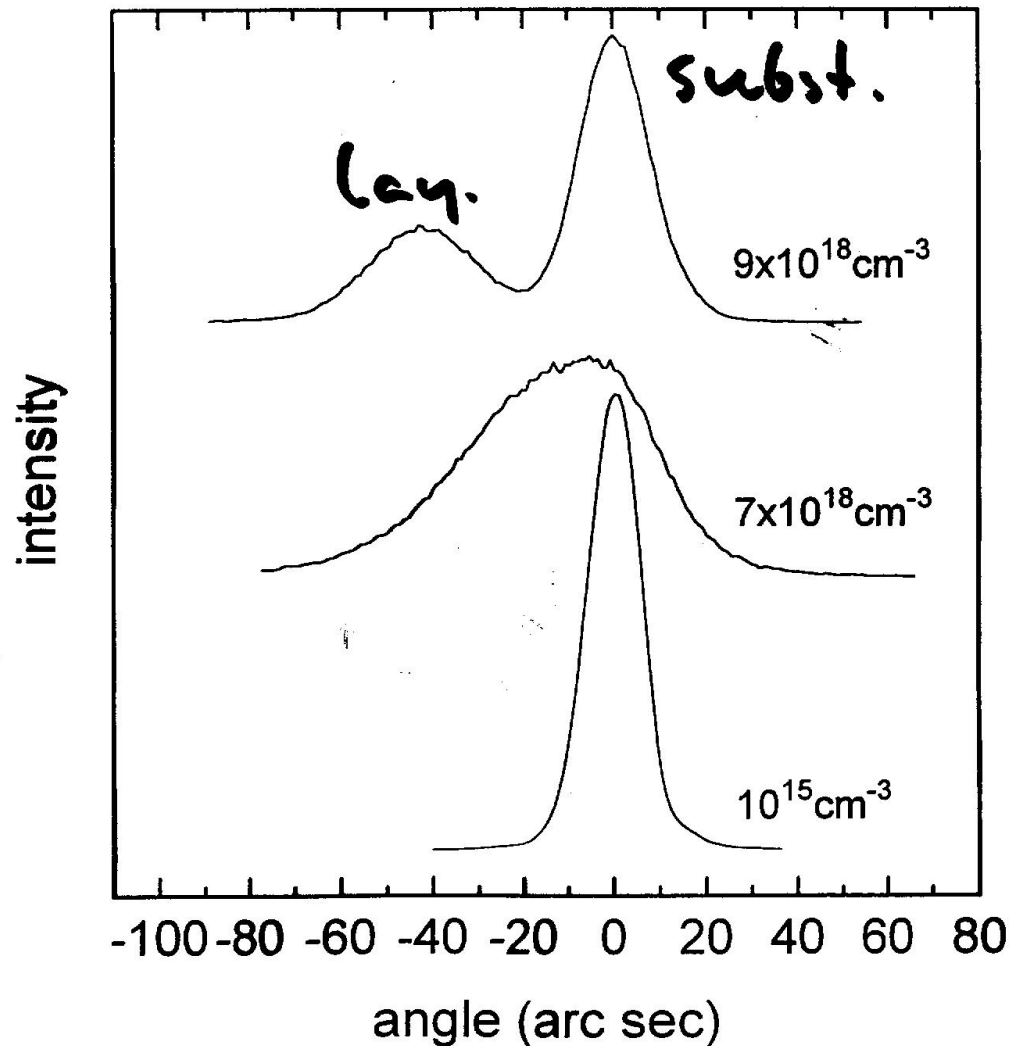


Perfect crystal

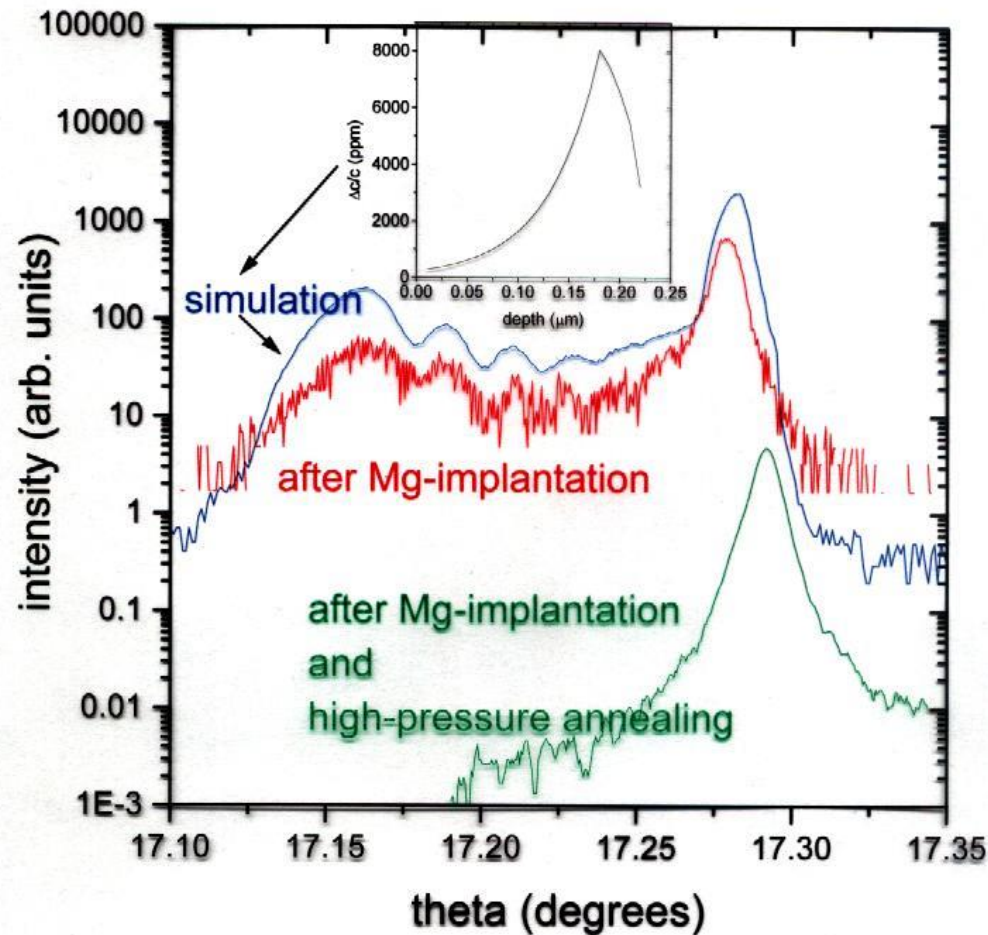


**Only small regions fulfill Bragg condition
Thin layers have much smaller intensity**

Example: Lattice expansion by free-electrons GaAs



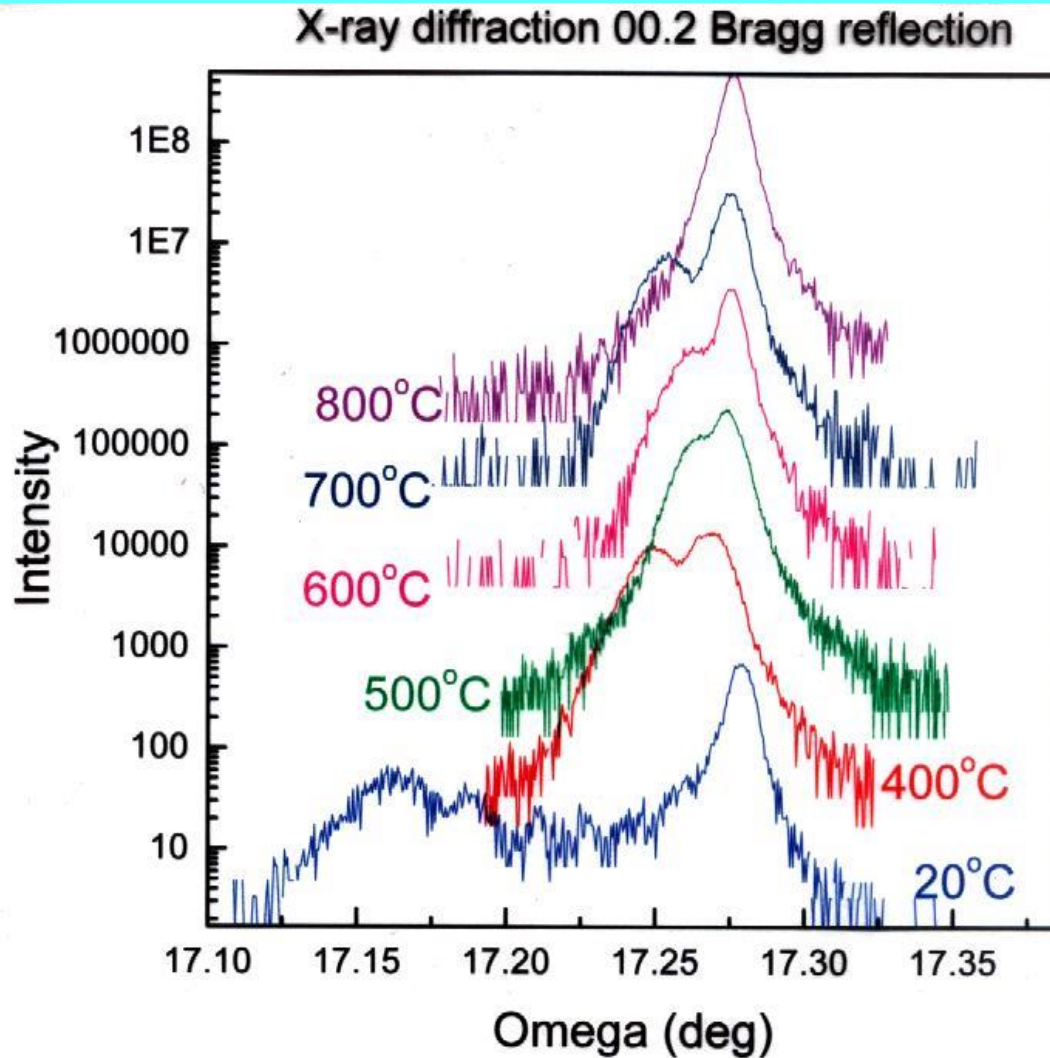
Example: Effect of implantation



1200oC
necessary

X-ray diffraction
GaN/sapphire layer

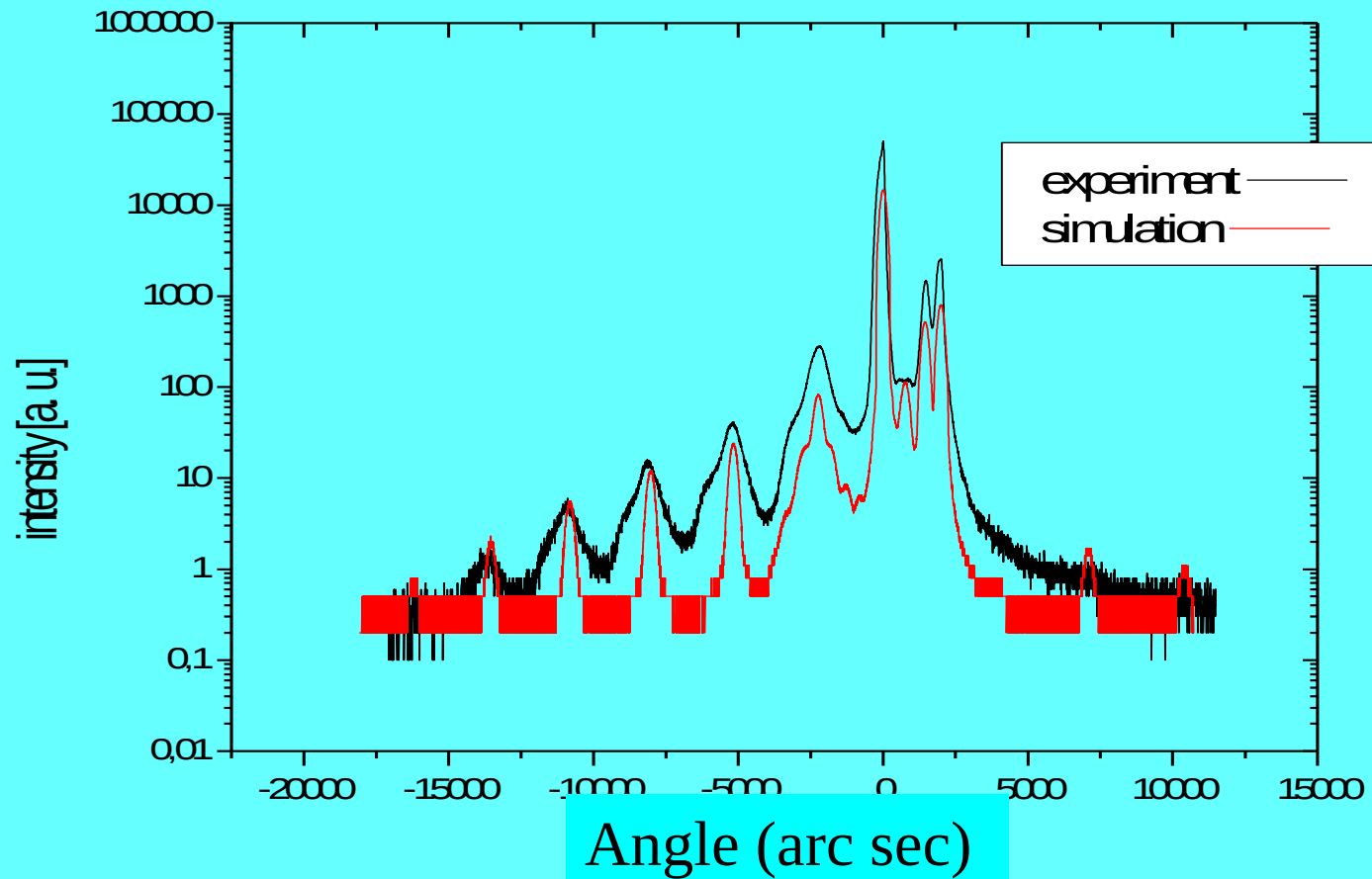
Implantation at high temperature



GaN layers on sapphire implanted
with Mg at different temperatures

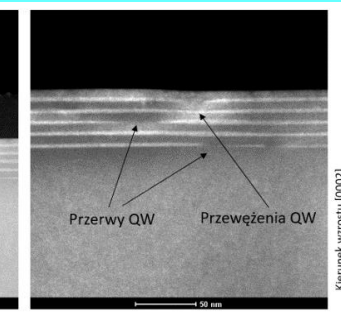
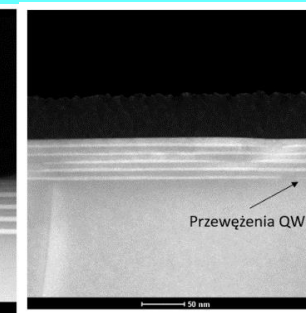
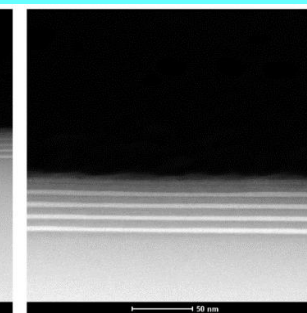
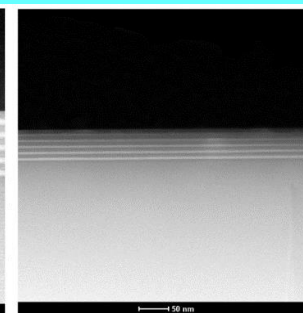
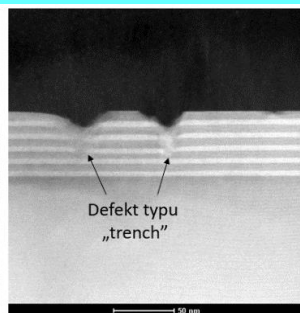
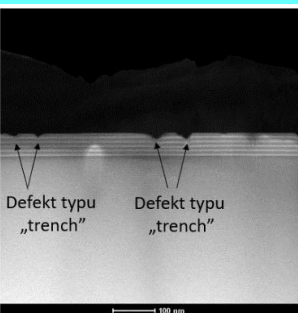
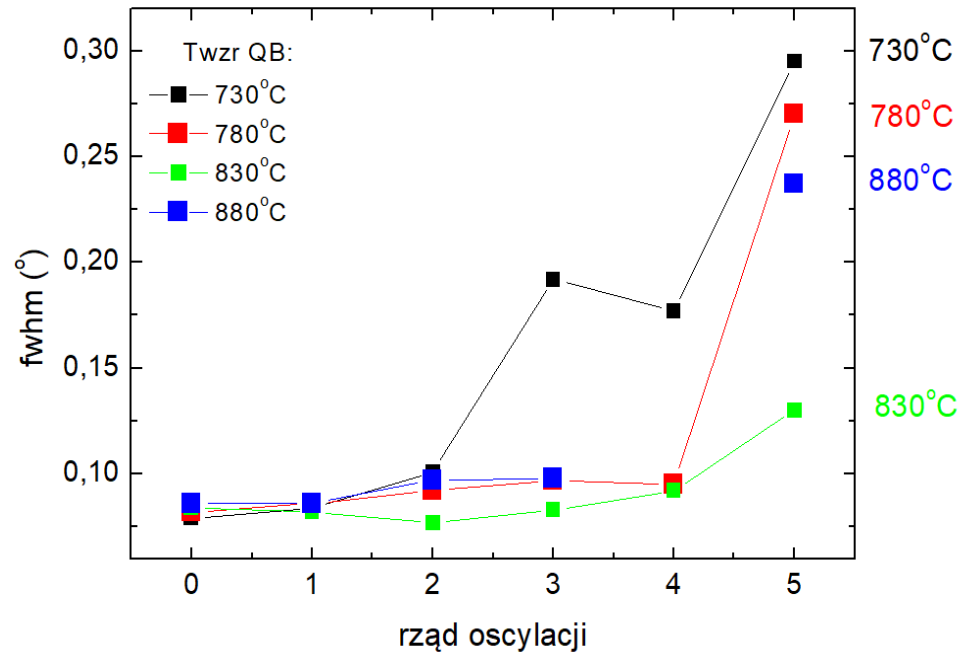
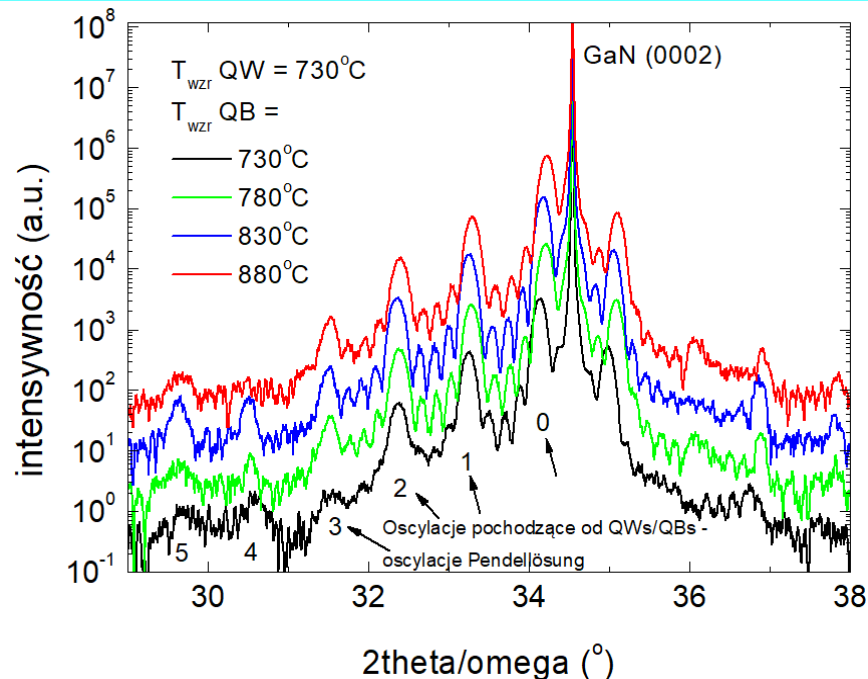
Only
800°C
necessary

Example: 10-fold GaN/InGaN with indium fluctuations



Broadening of fringes

InGaN QWs, QBs grown at different temperatures

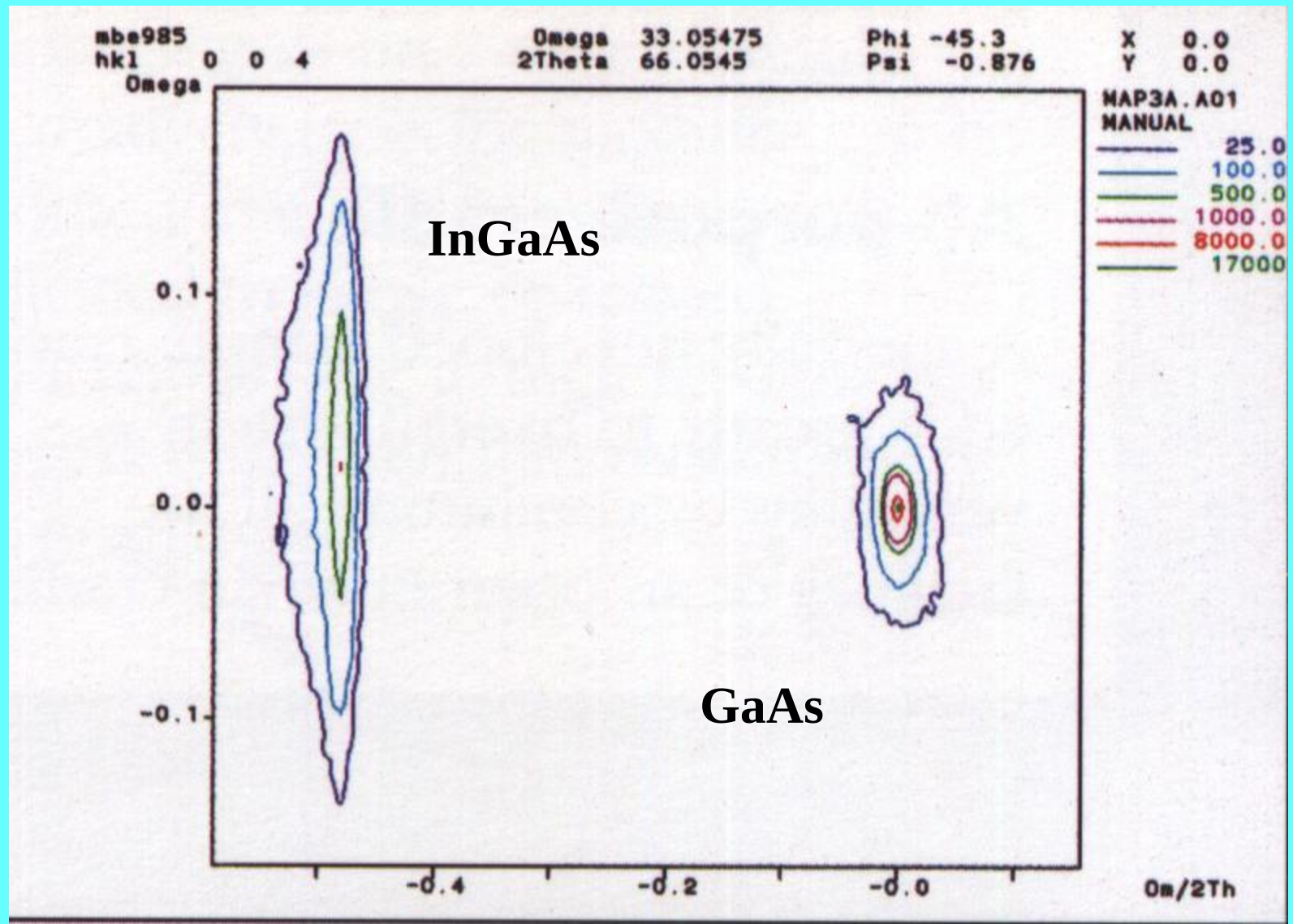


730

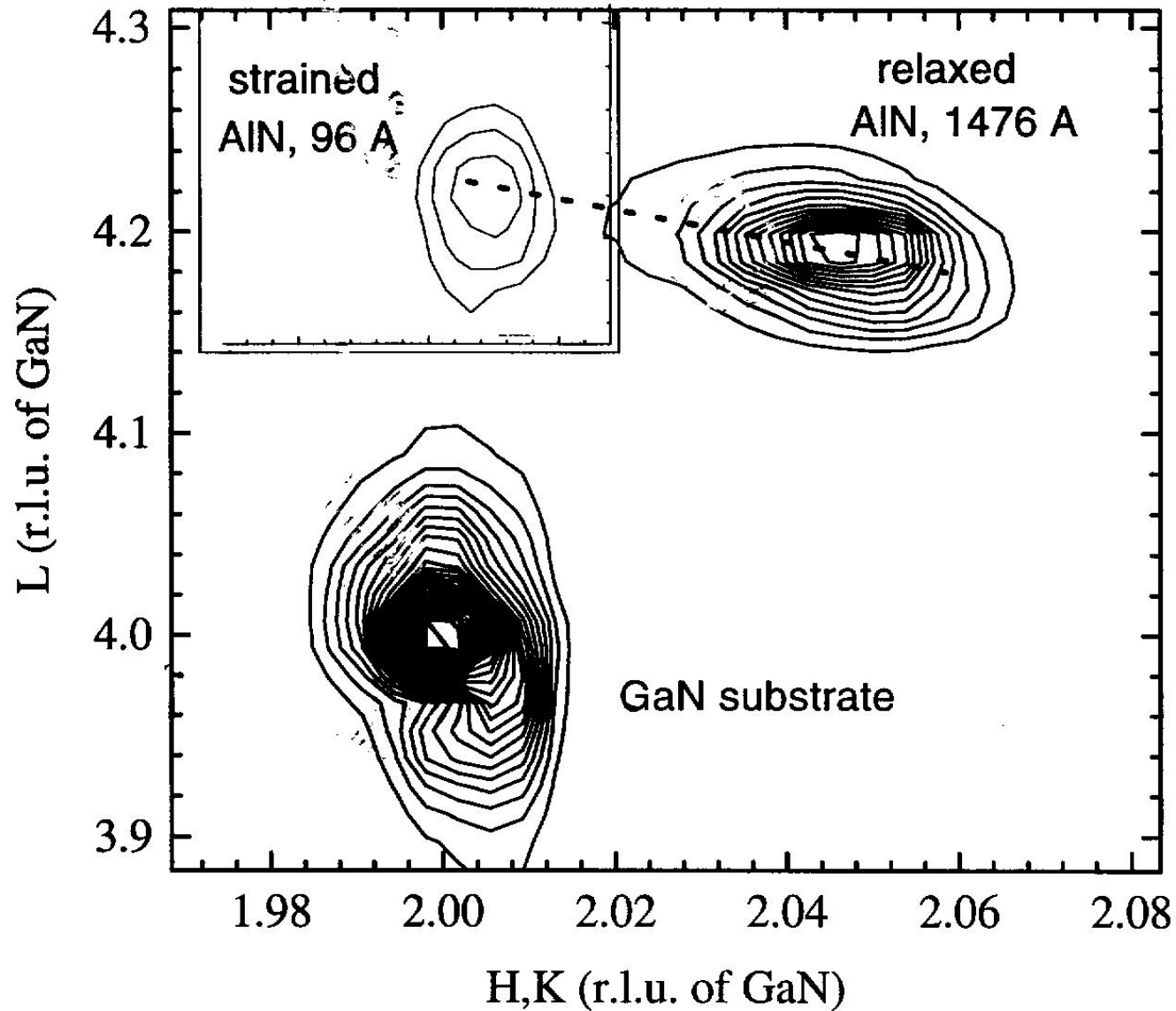
830

880

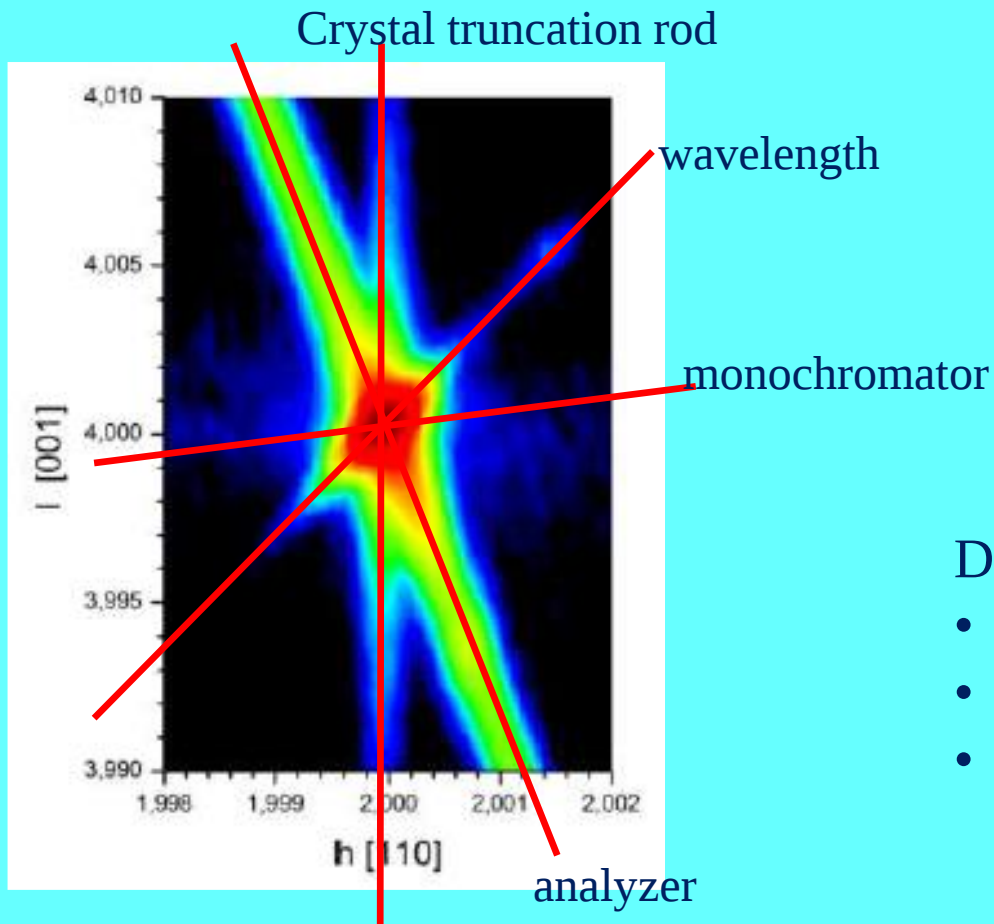
Reciprocal lattice mapping



Reciprocal lattice maps



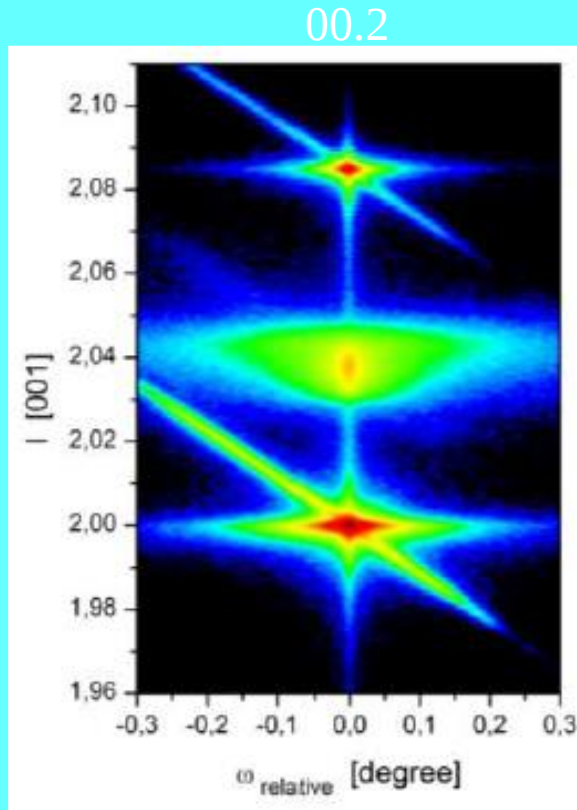
Streaks in reciprocal lattice mapping



Differ for:

- Different reflections
- Diffractometer set up
- Sample

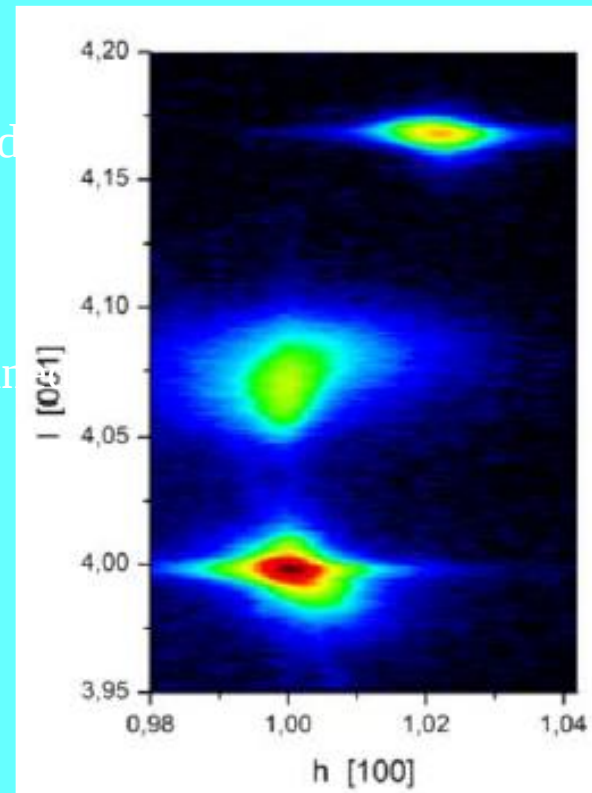
Reciprocal lattice mapping



AlN relaxed

AlInN strained
to GaN

GaN

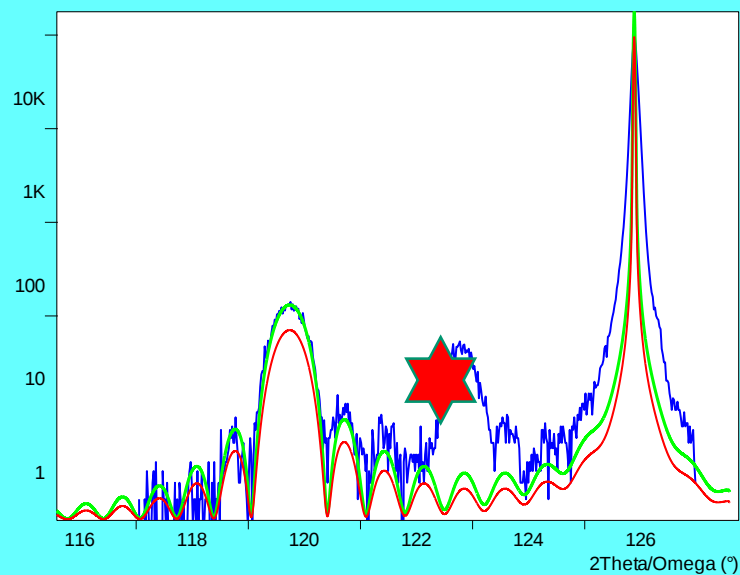
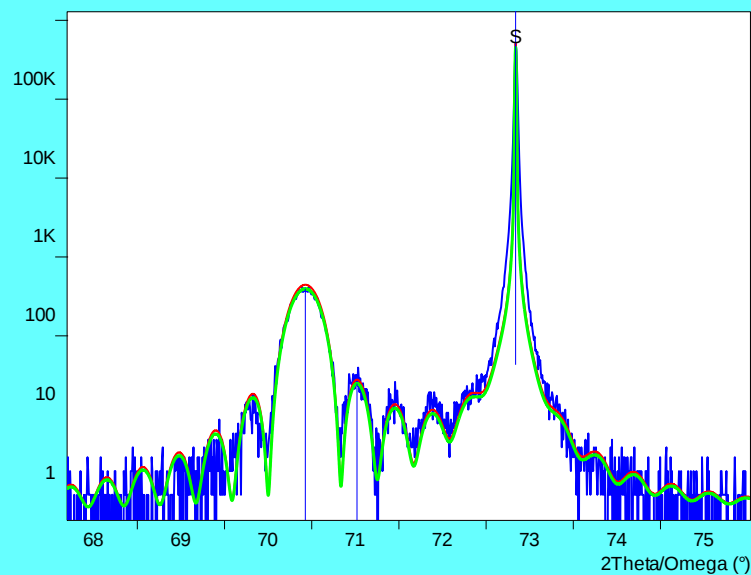


Example from Bruker webinar

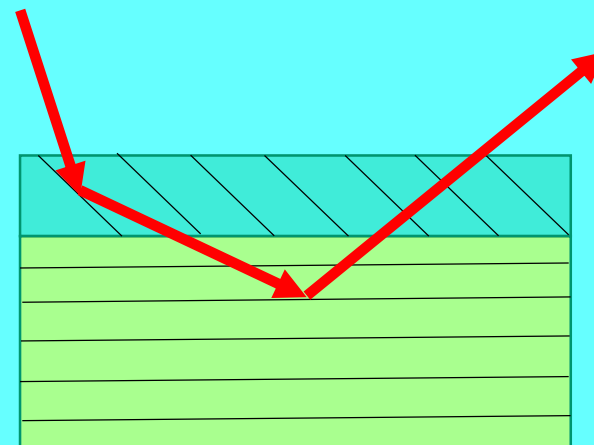
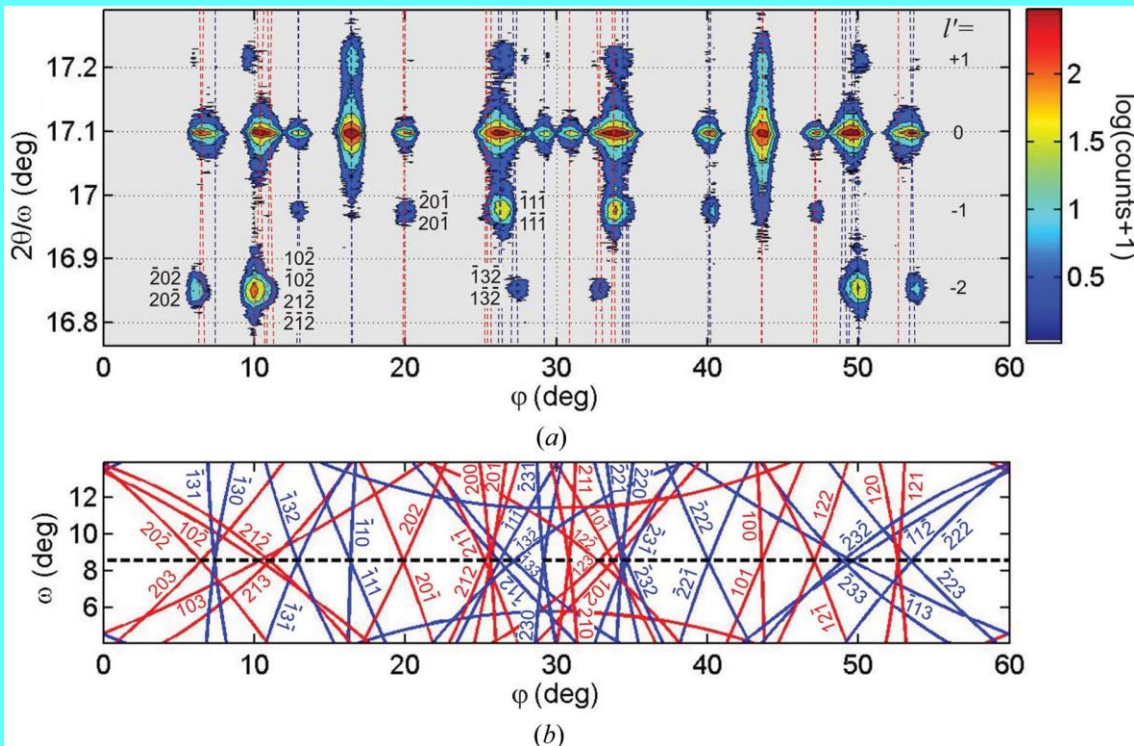
Look out for Domagala's peaks (hybrid peaks)

00.4

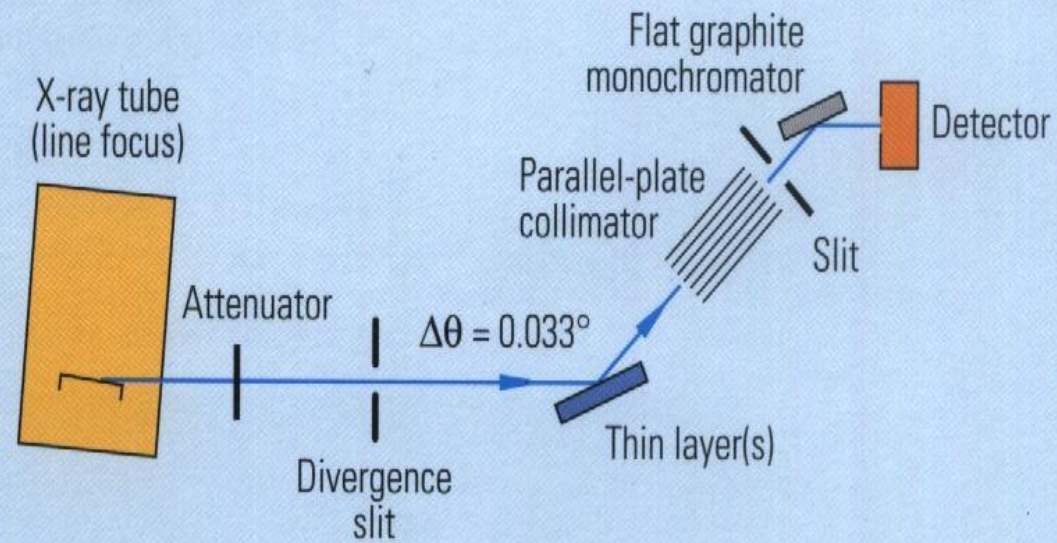
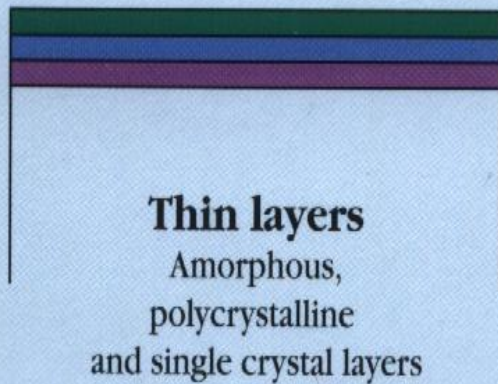
00.6



Hybrid reciprocal lattice



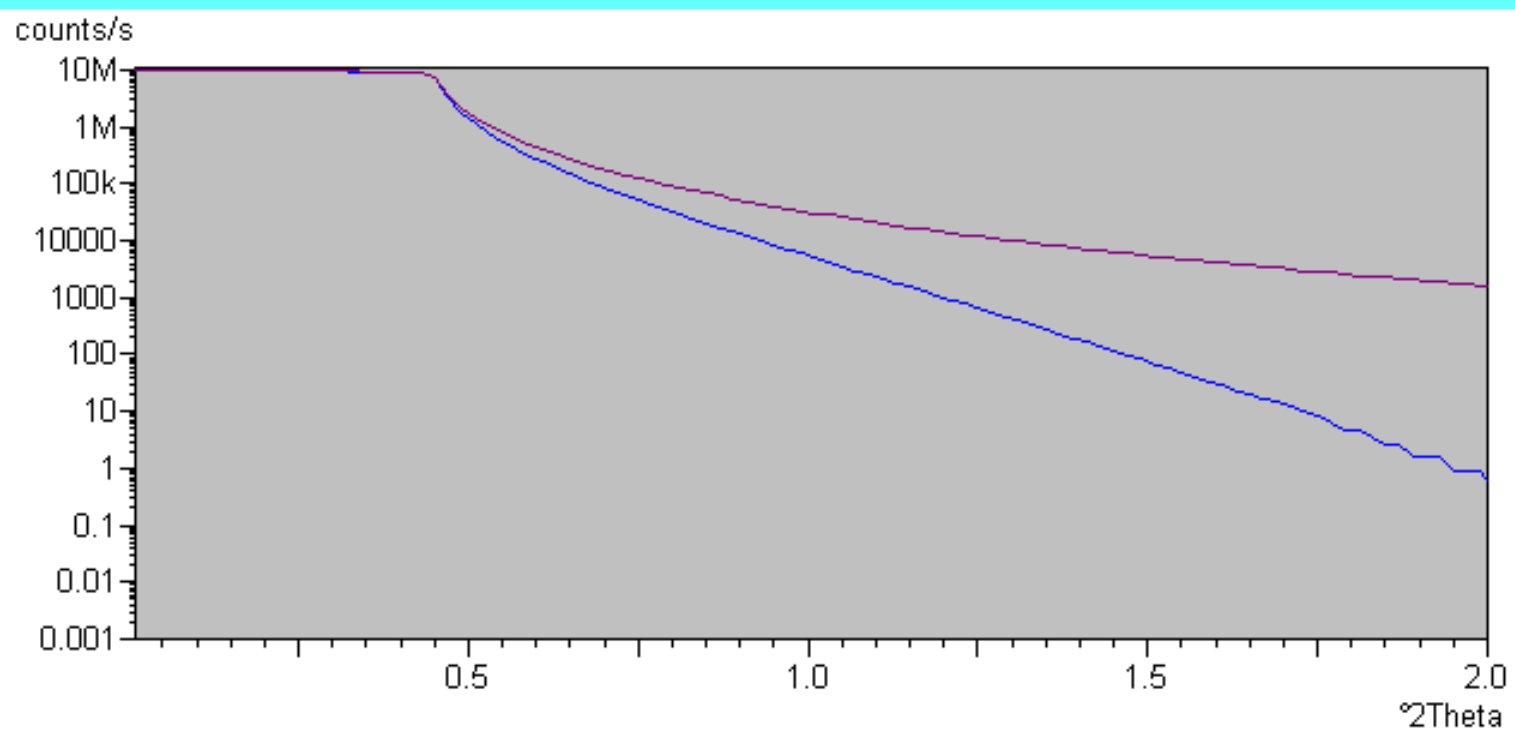
Thin layers



Diffraction

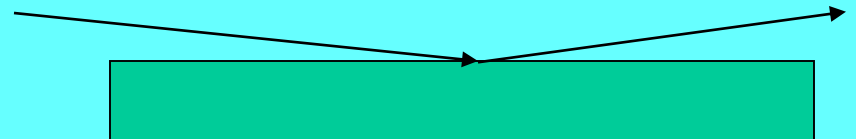
Reflectivity

Reflectivity- surface roughness

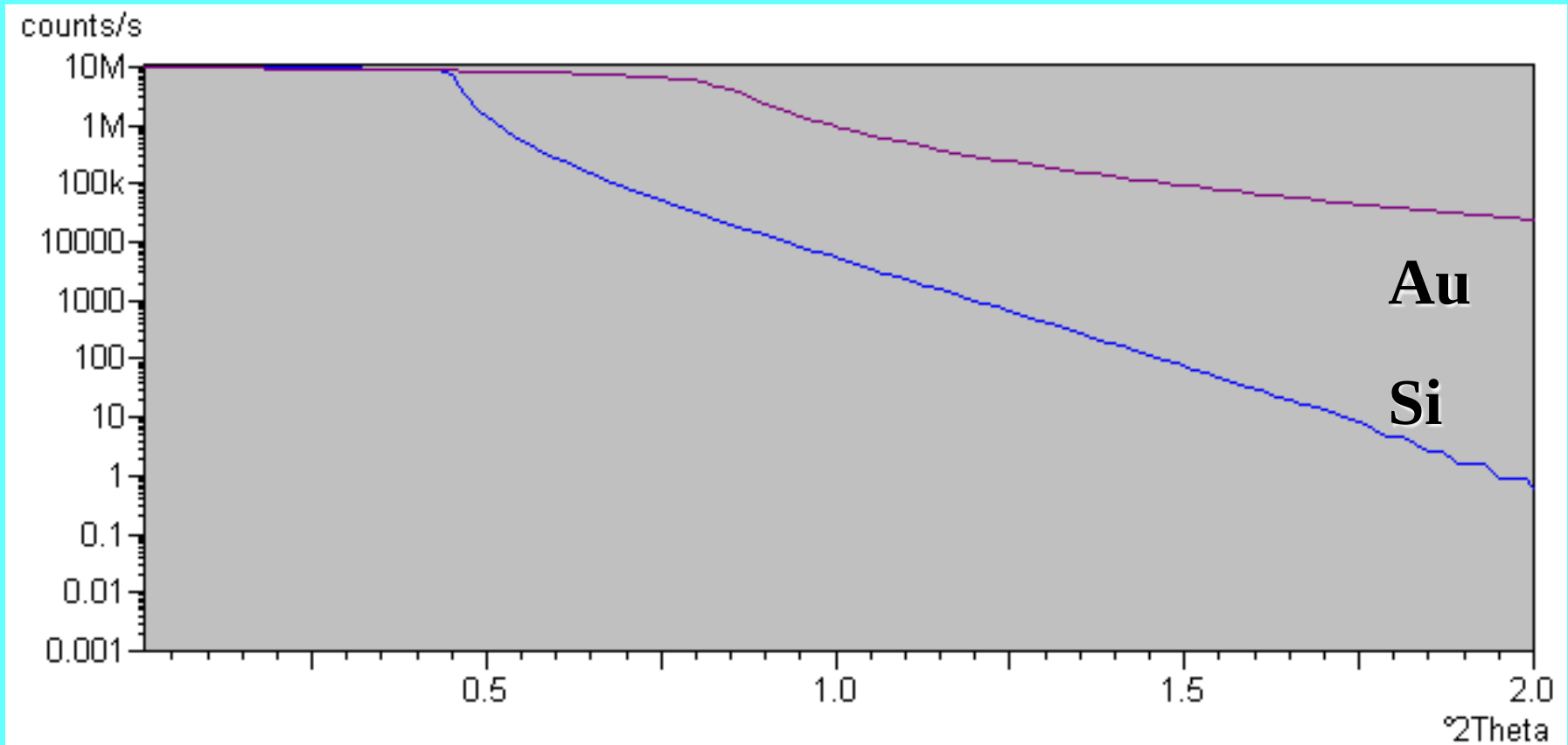


RMS 1A

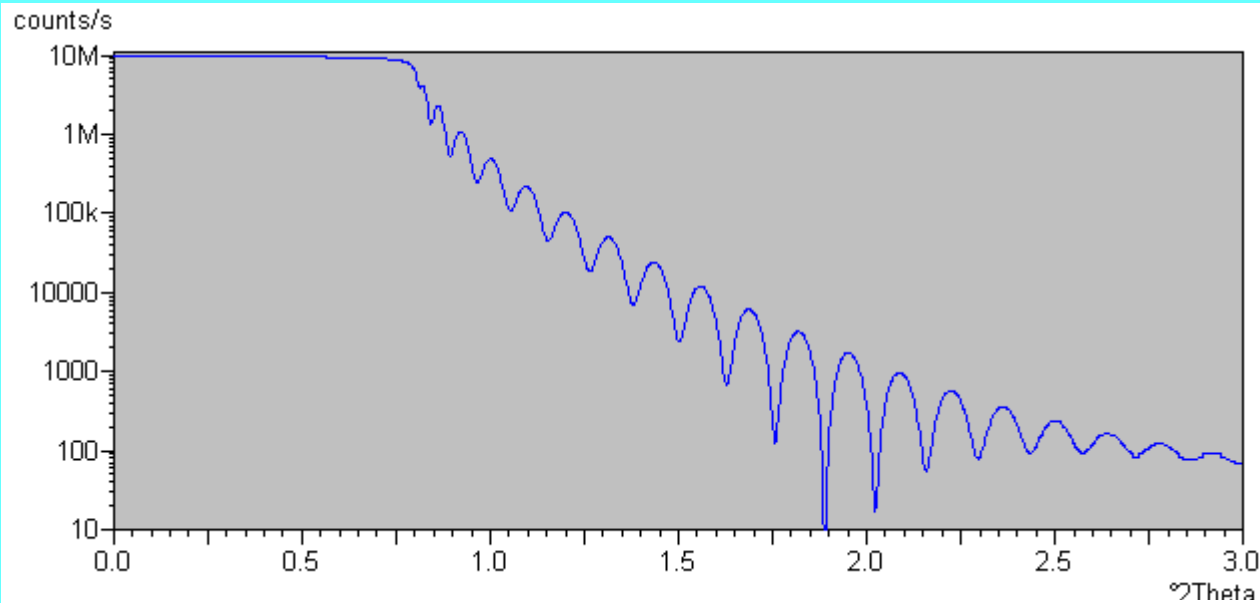
RMS 20A



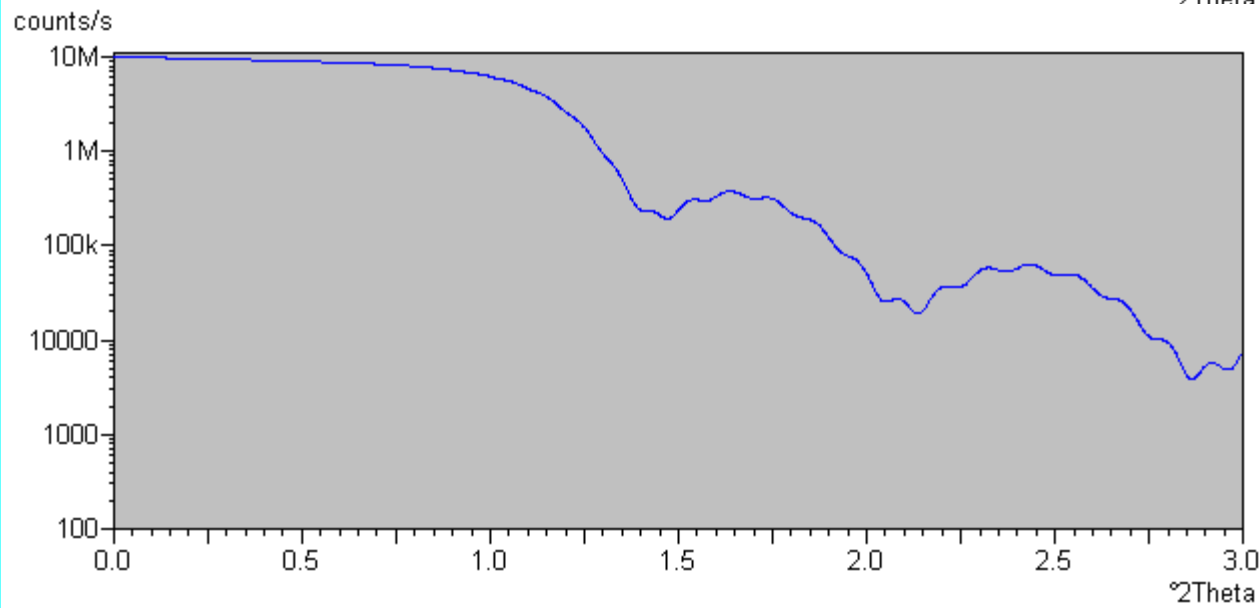
Reflectivity- density



Reflectivity-layer thickness

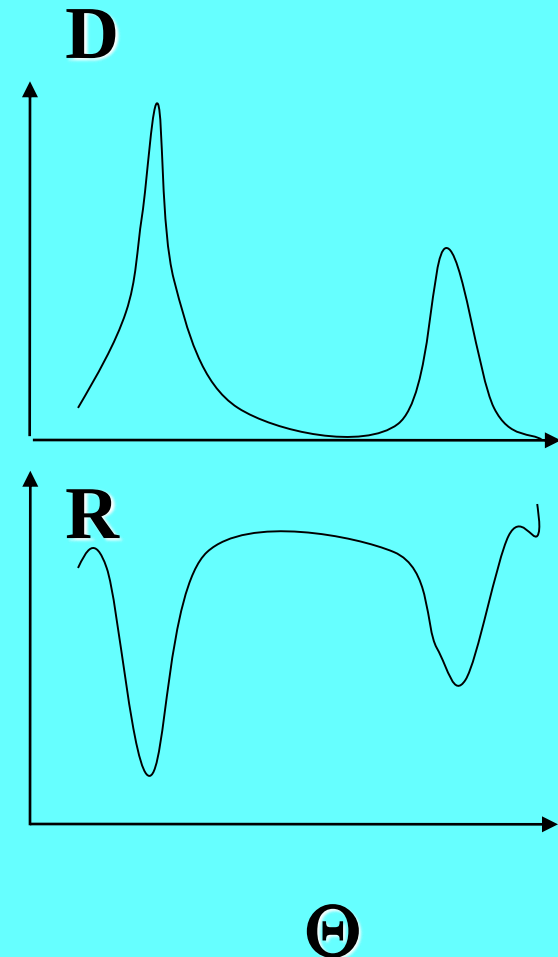
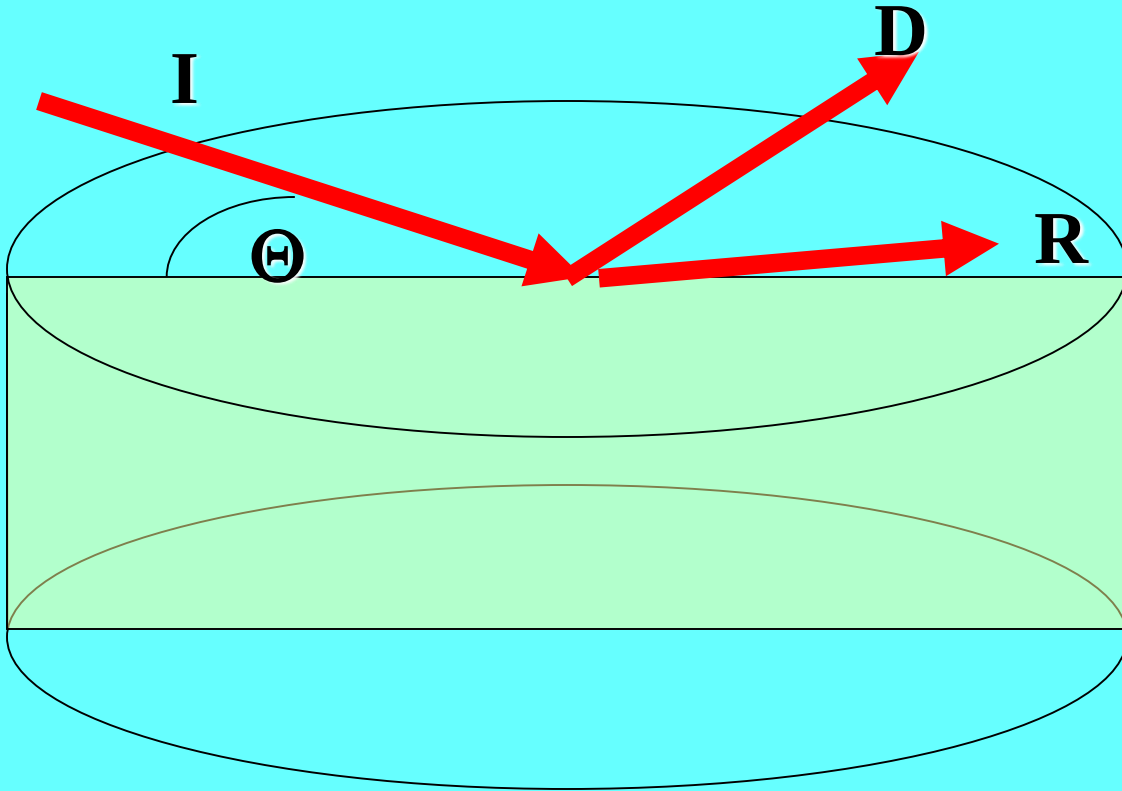


**60 nm Ni
on Si**

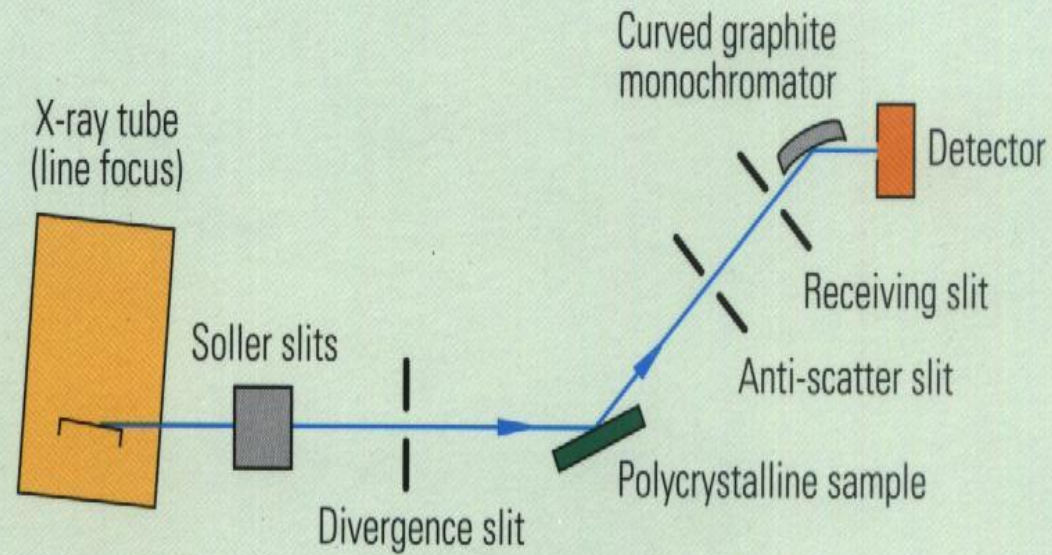
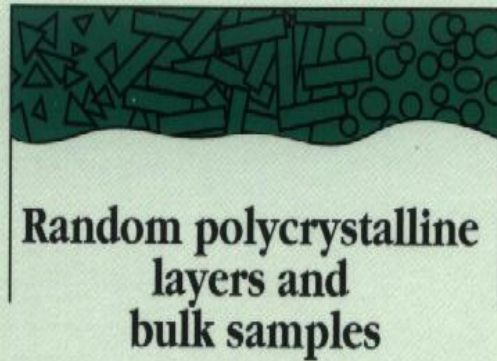


**10 nm Au
60 nm Ni
on Si**

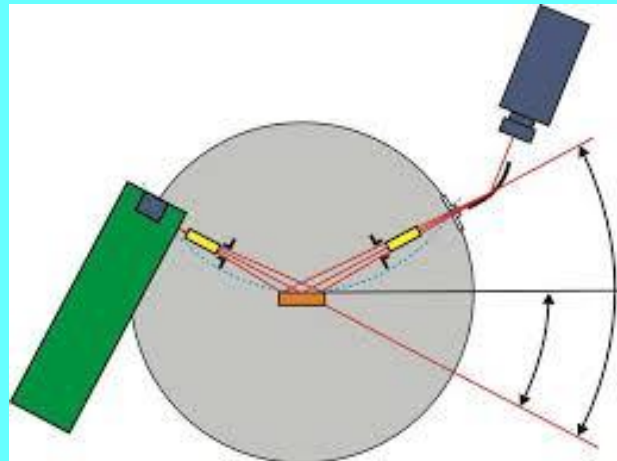
Surface diffraction (grazing incidence)



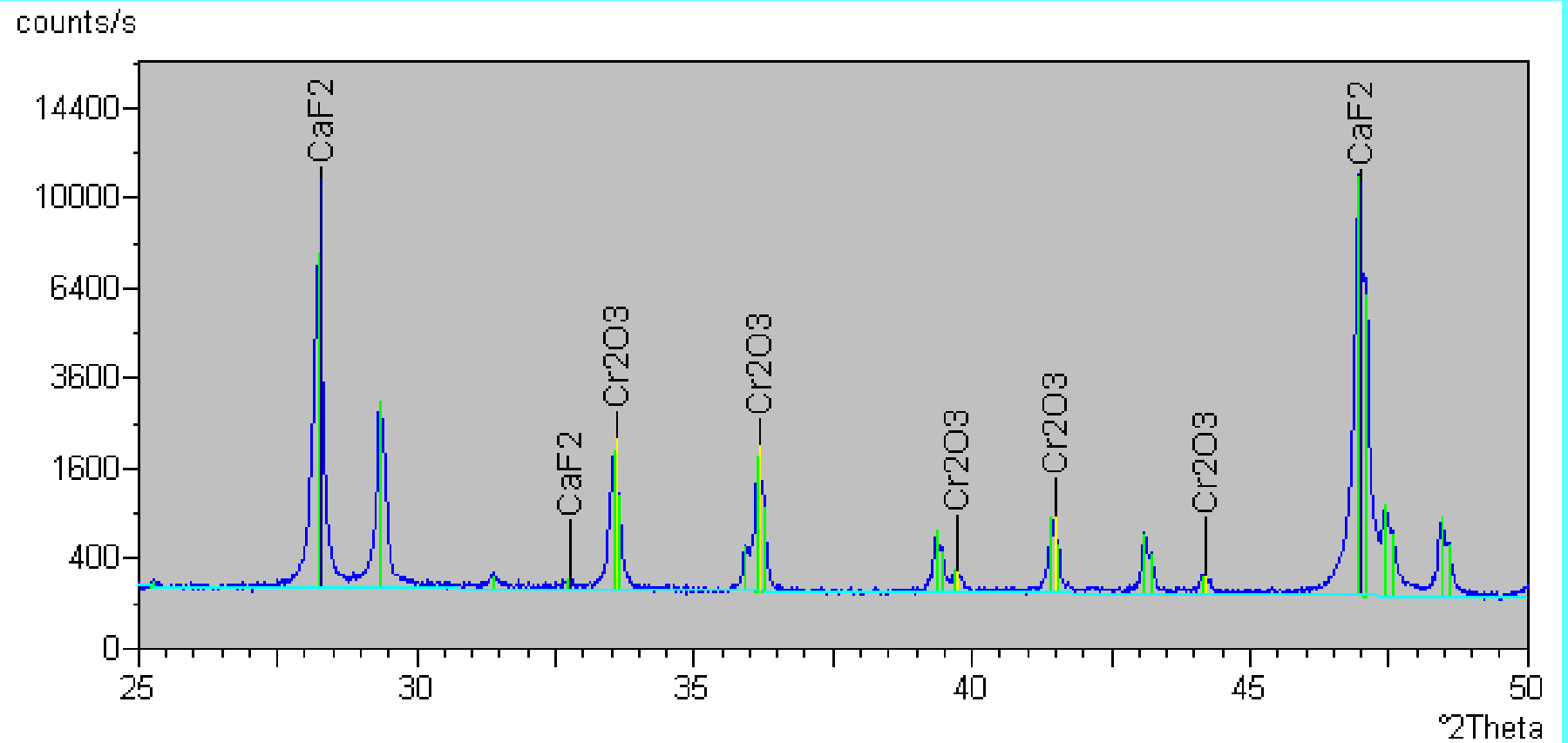
Polycrystalline materials



Bragg-Brentano configuration



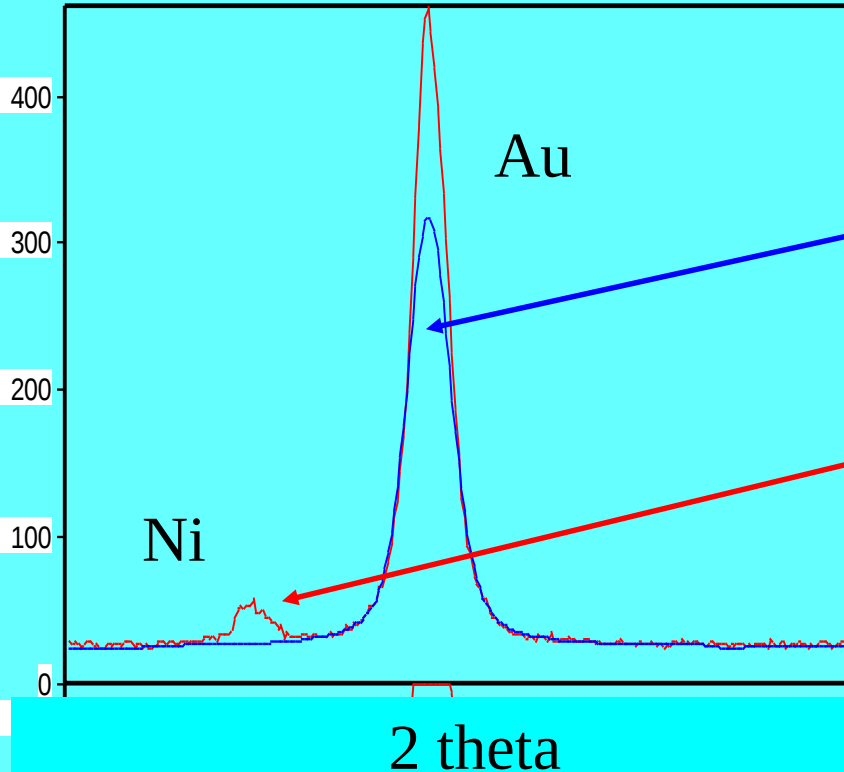
Powder diffractogram



Diffraction from polycrystalline thin layers

File name: NIT12.IDF, date and time: 15/11/2003 18:25:04

Counts

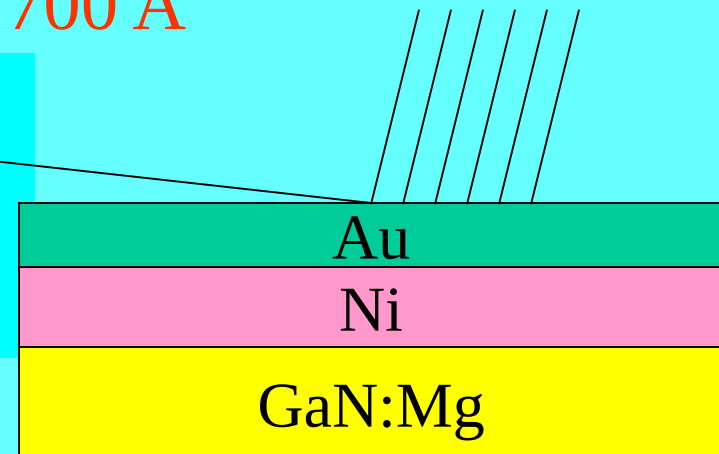


Incidence angle 0.2 deg

Penetration depth about 200 Å

Incidence angle 0.6 deg

Penetration depth about 700 Å



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 intravedere... visioni insospettite. 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