X-ray line profile analysis

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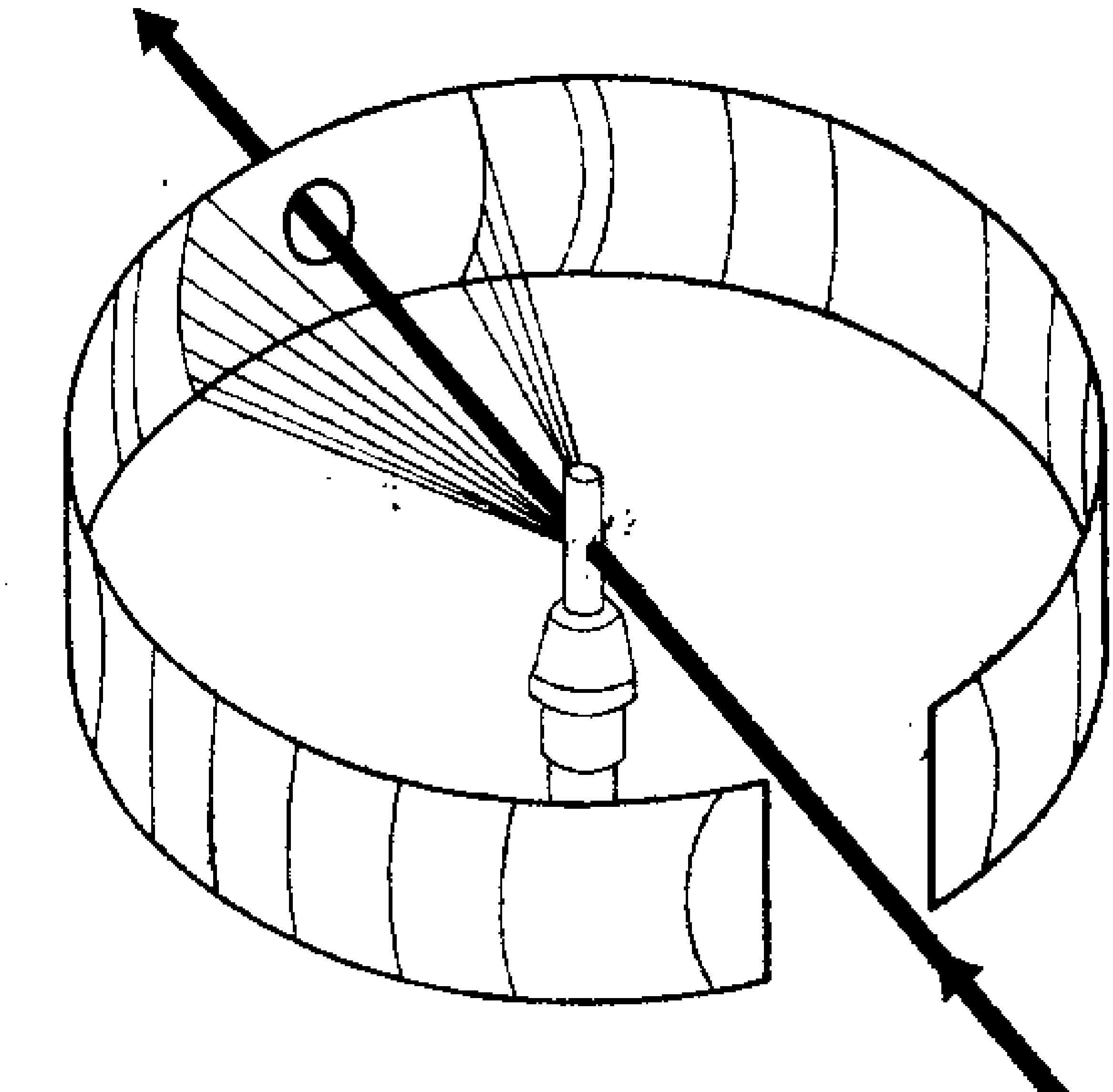
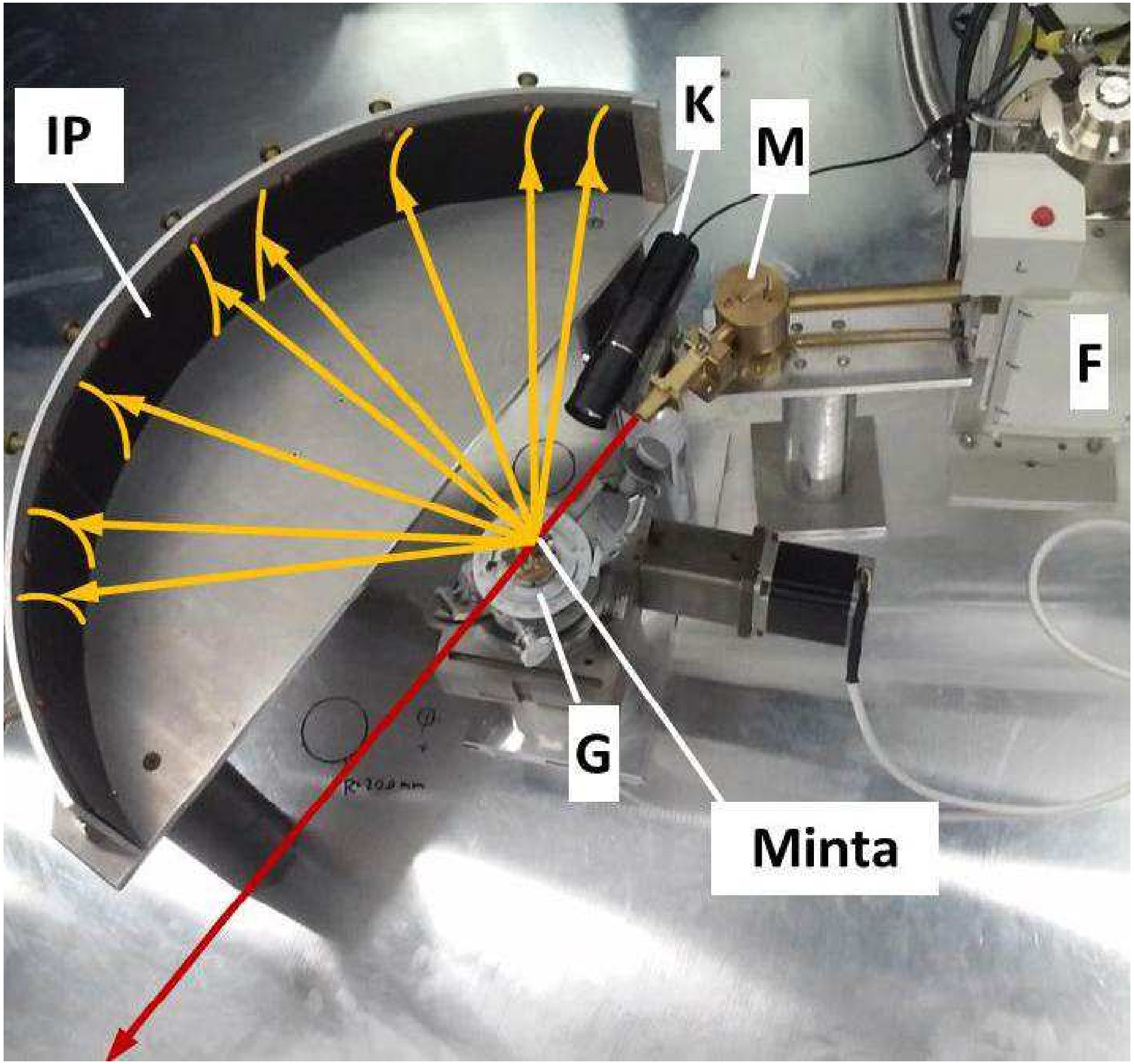
Introduction:

- XLPA measurements
- Information content of Diffraction profiles
- Microstructure determination methods
- Momentum method
- Convolutional Multiple Whole Profile fitting (CMWP) method

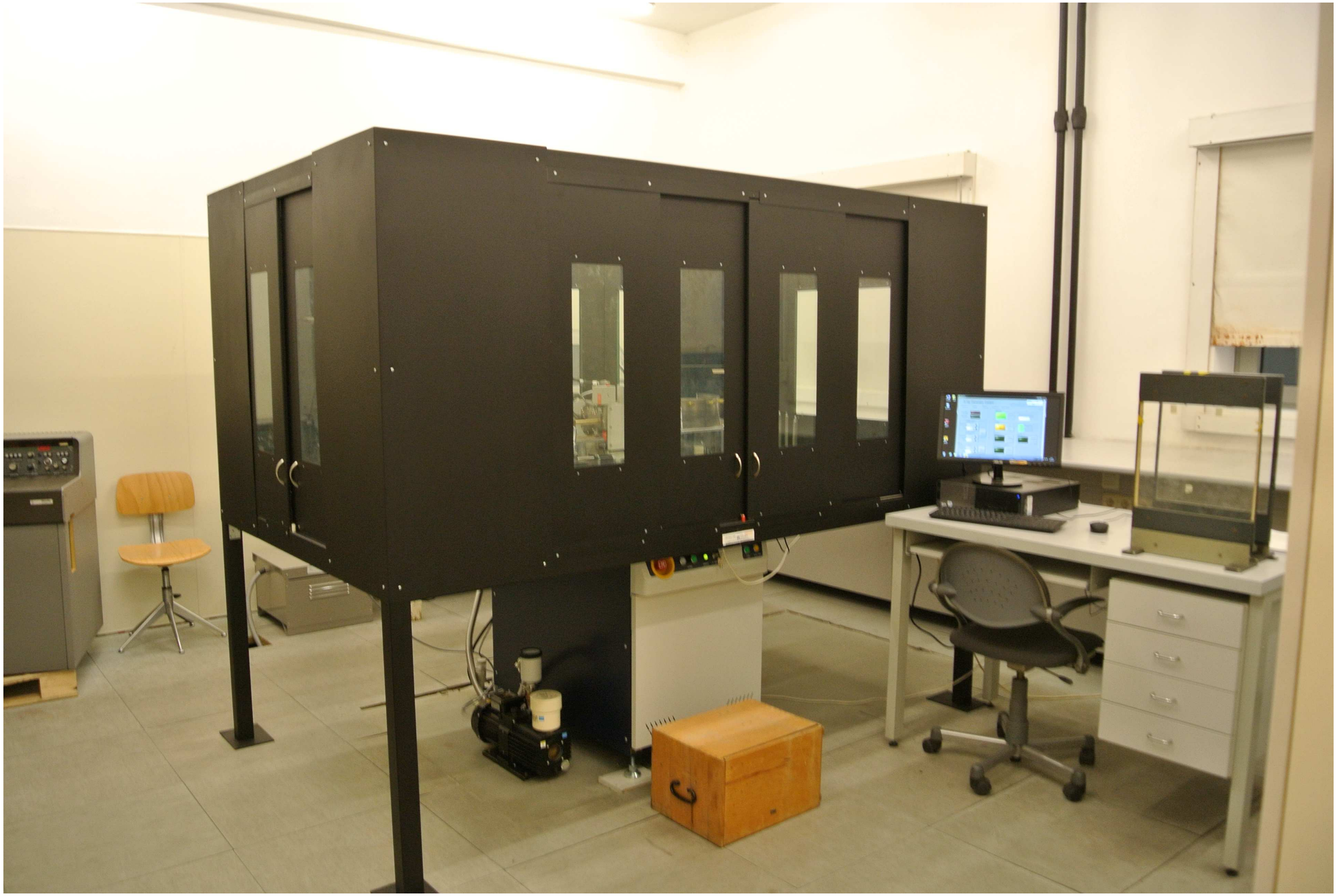
Debye-Scherrer geometry



Diffraction geometry



Rotating anode diffractometer



Geometry for Image Plate measurements

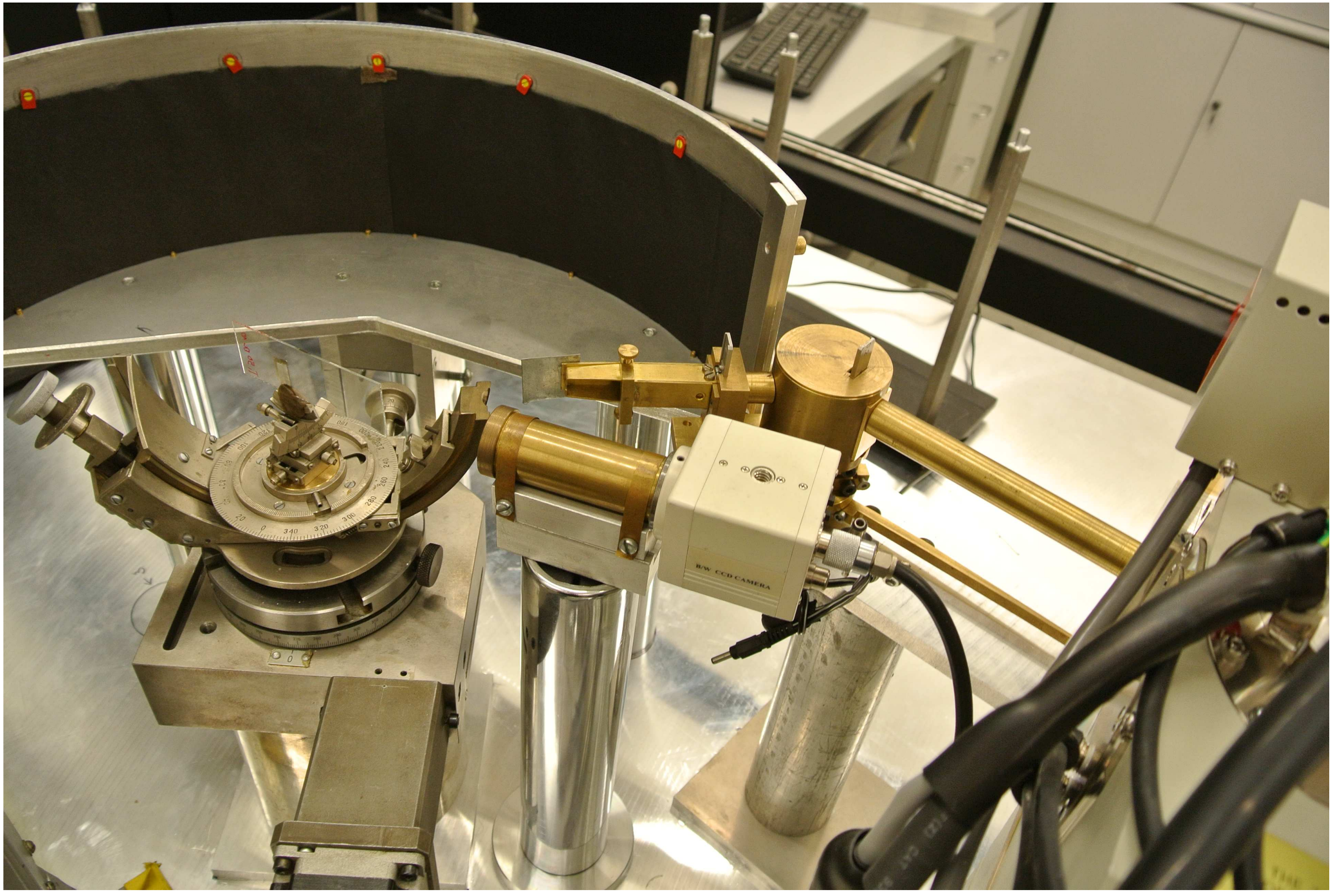


Image Plate reader

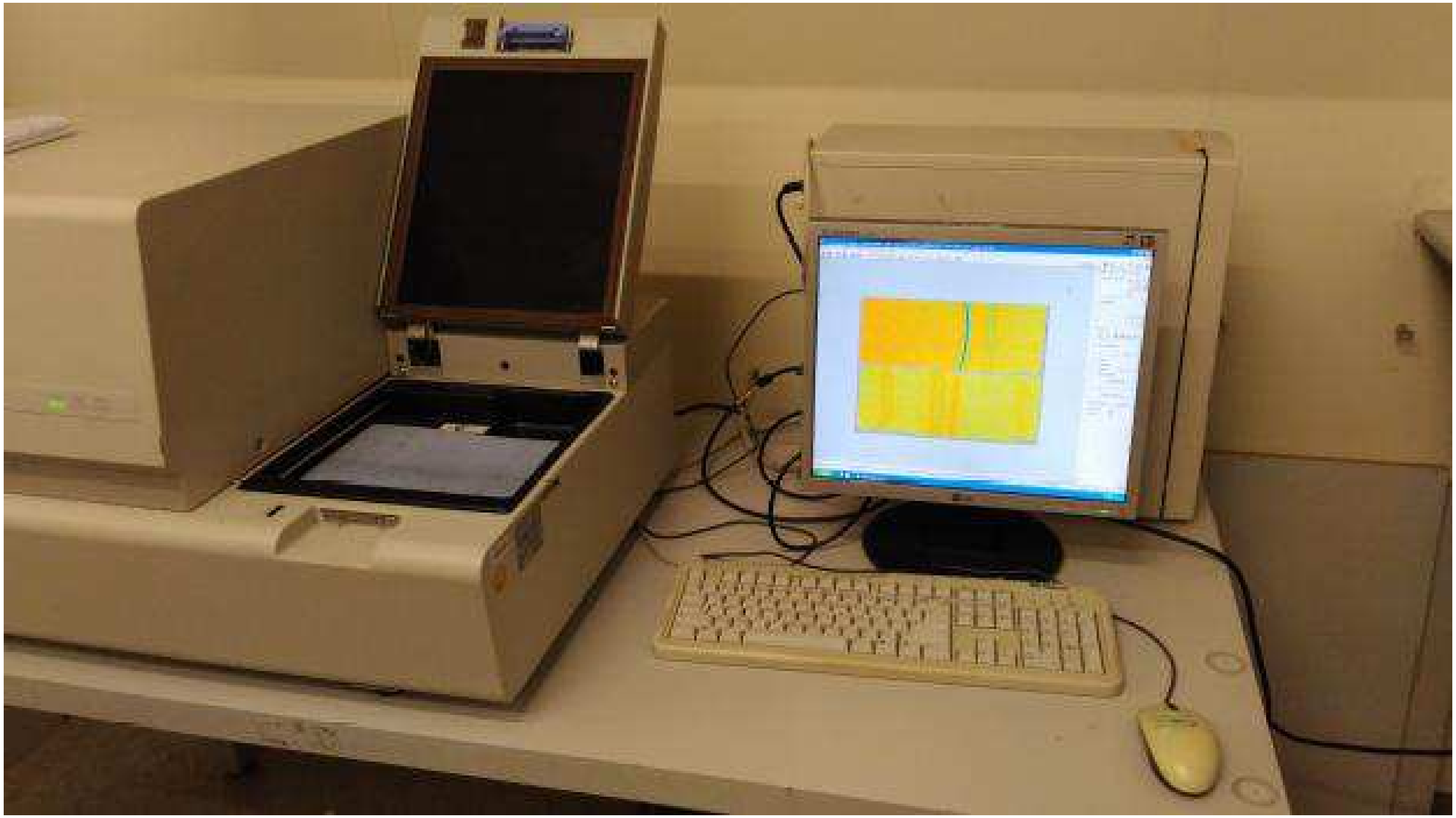
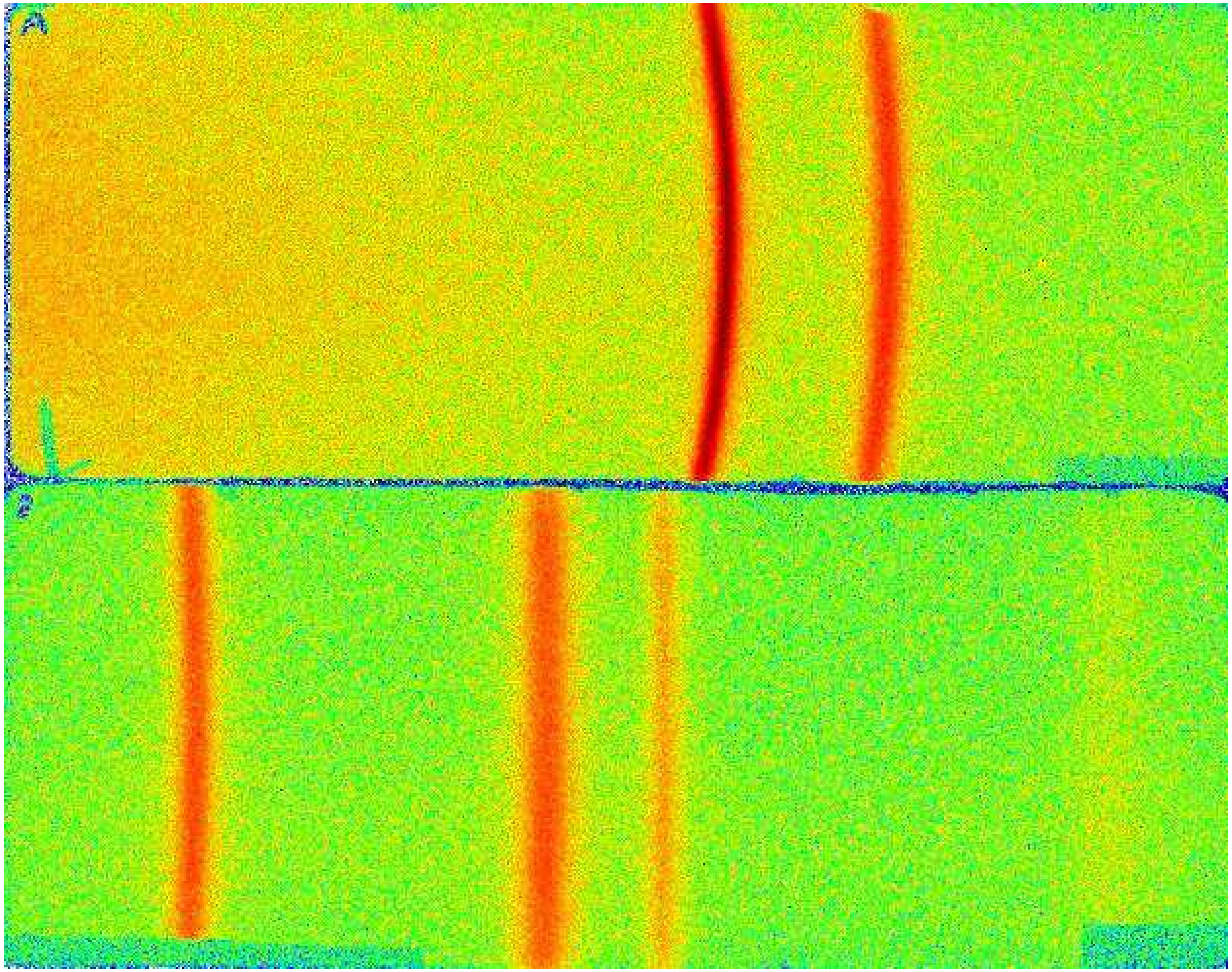


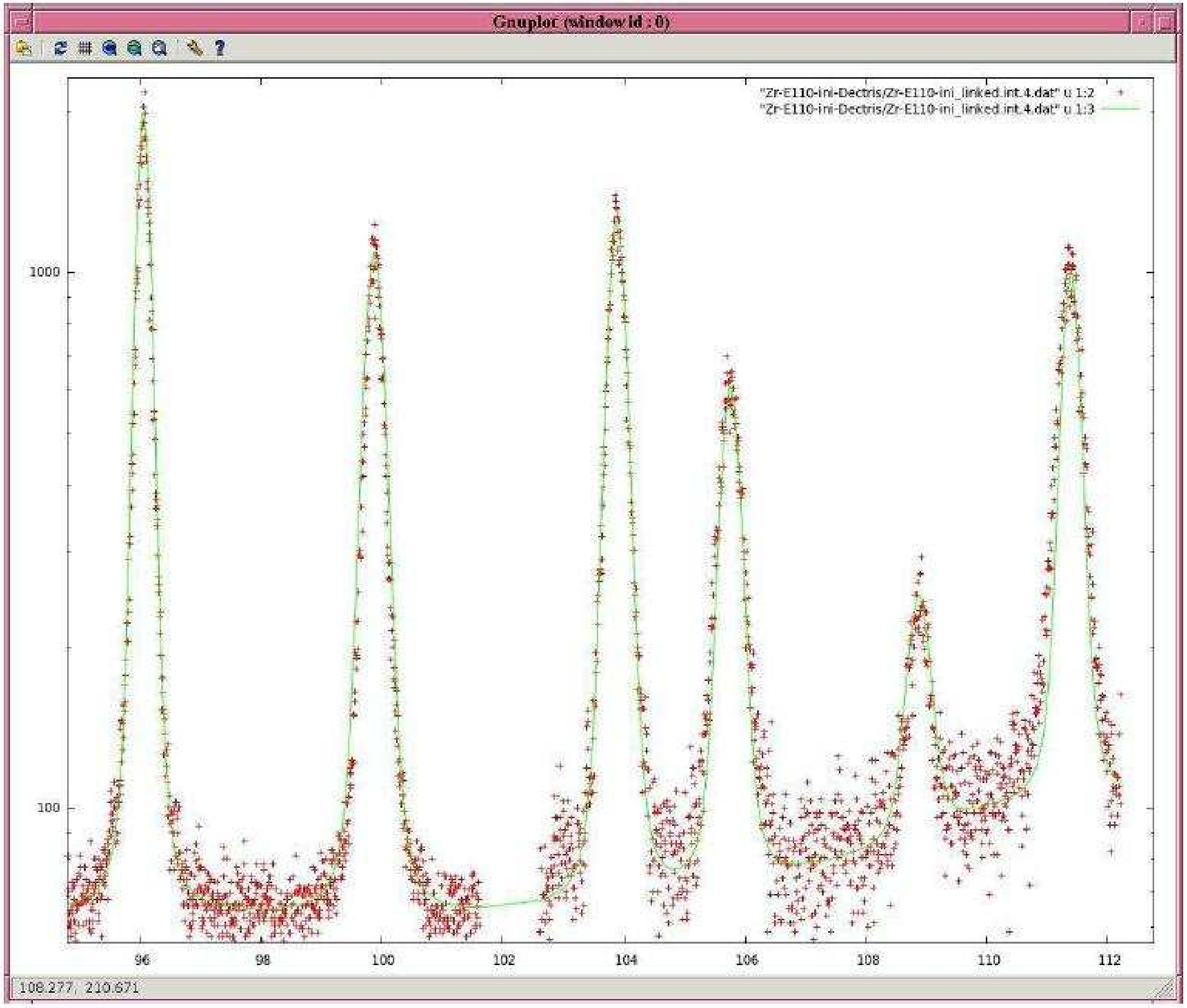
Image Plate exposition



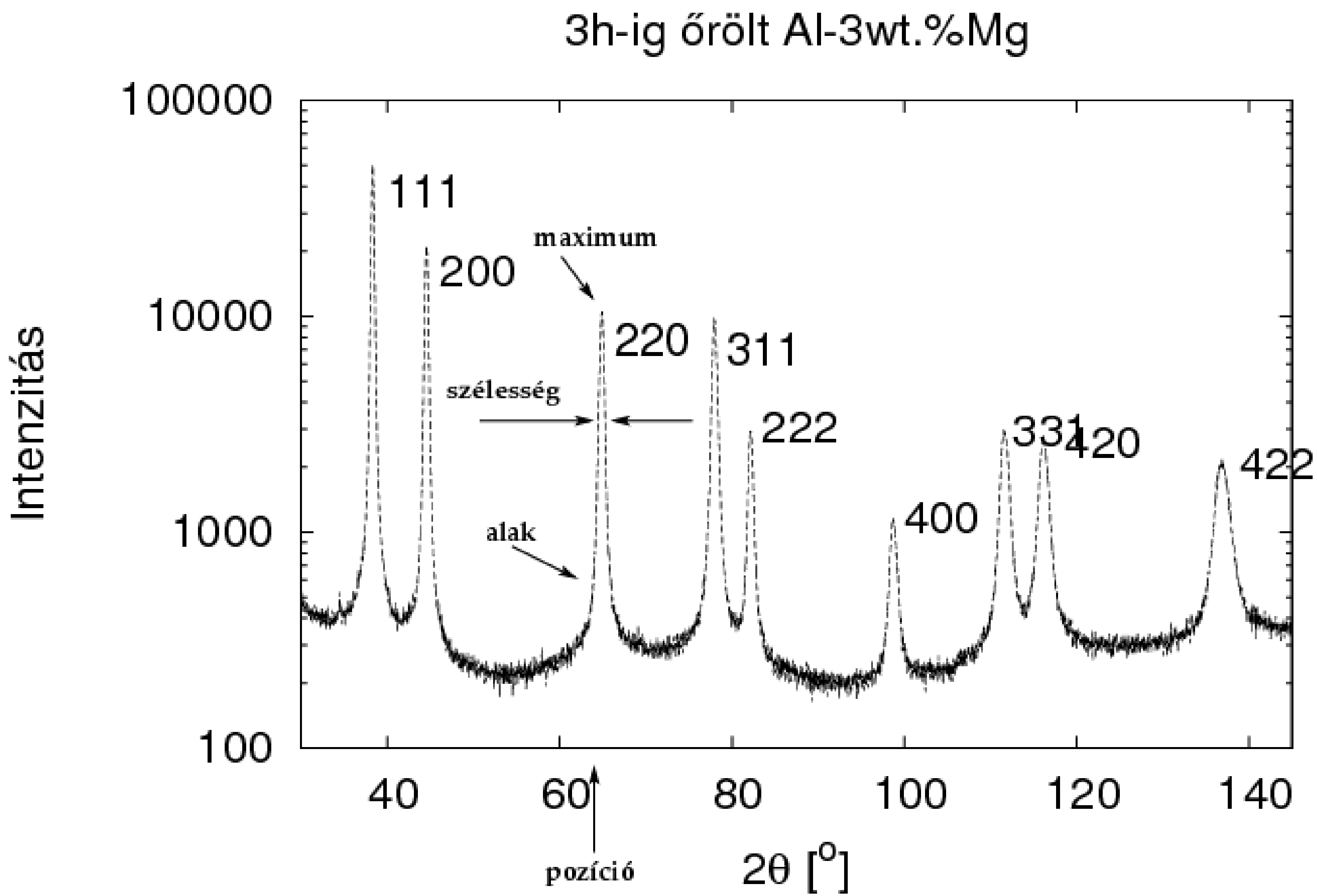
Geometry for Detector measurements

Geometry for Detector measurements

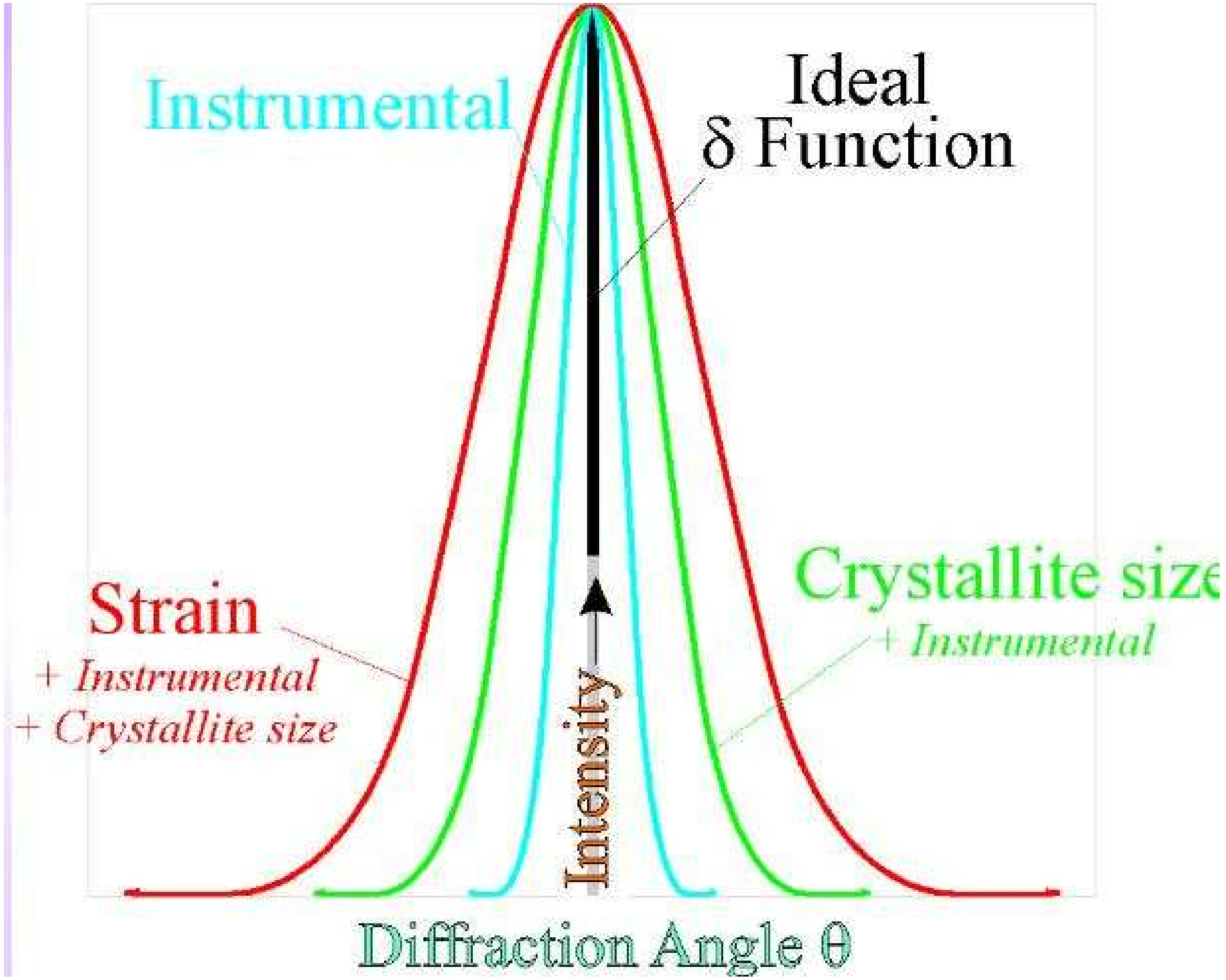
Detector measurement (Zr E110 ini)



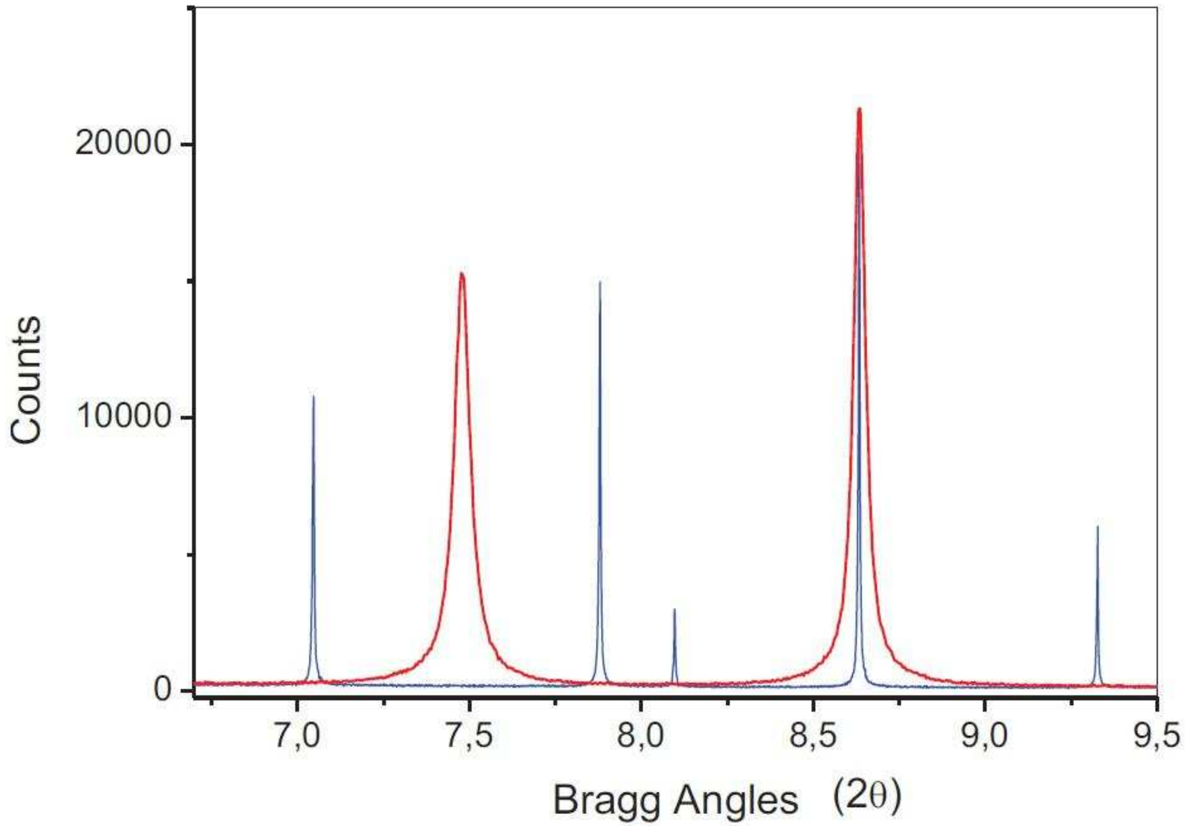
Typical diffraction pattern



Composition of line profiles



Ieasurement of a sample and instrumental



Information content of X-ray patterns

Information about the crystal structure:

- position of Bragg reflexions
- intensity of Bragg reflexions

Information about the microstructure:

- broadening of Bragg reflexions
- shape of Bragg reflexions

Effect of microstructure

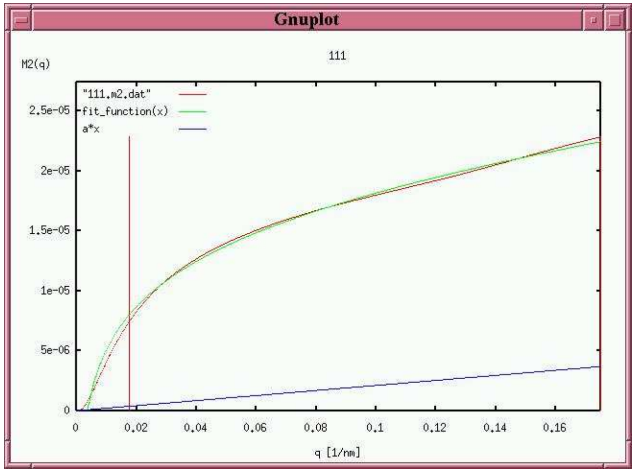
Microstructure components causing line broadening:

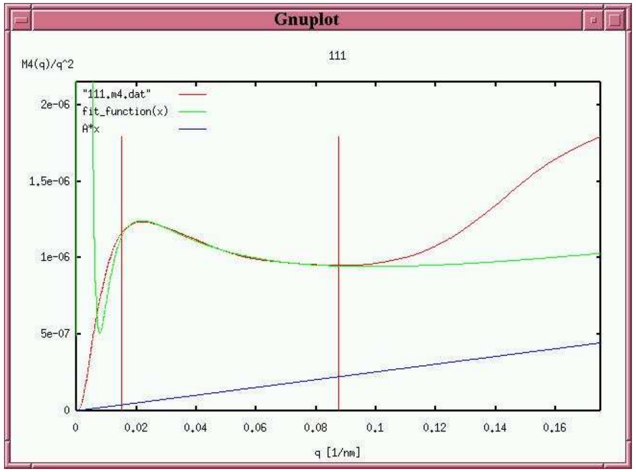
- finite particle, crystallite, grain or subgrain size (coherent domain size)
- crystal defects: dislocations (density and type of dislocations)

Evaluation of microstructure

- single profile methods (Momentum method: I. Groma)

$$M_k(q) = \frac{\int_{-q}^{q} x^k I(x) \mathrm{d}x}{\int_{-\infty}^{\infty} I(x) \mathrm{d}x}$$





- multi profile methods (CMWP method: T. Ungár and G. Ribárik)

Separation of mikrostructural effects

In case of single profile (momentum) method:

- Crystallite size effect: $1/q^2$ decaying
- Strain effect: $1/q^3$ decaying

In case of multi profil methods:

- Crystallite size effect: the broadening is independent of K ($K = \frac{2\sin(\theta)}{\lambda}$)
- Strain effect:
  - the broadening globally increases with K
  - typical anisotropy
- Planar defects: anisotropy, asymmetric broadening

Momentum method

Theoretical background:

- Size profile: Lorentz-function, asymptotic form:

$$I(q) = \frac{1}{\pi^2 d} \frac{1}{q^2}$$

- Asymptotic decaying of dislocation profile (Groma, 1998):

$$I(q) = \frac{\Lambda}{4\pi^2} \langle \rho \rangle \frac{1}{q^3}$$

- Asymptotic decaying if both effects are present:

$$I(q) = \frac{1}{\pi^2 d} \frac{1}{q^2} + \frac{\Lambda}{4\pi^2} \langle \rho \rangle \frac{1}{q^3}$$

Momentum method

$$M_k(q) = \frac{\int_{-q}^{q} x^k I(x) \mathrm{d}x}{\int_{-\infty}^{\infty} I(x) \mathrm{d}x}$$

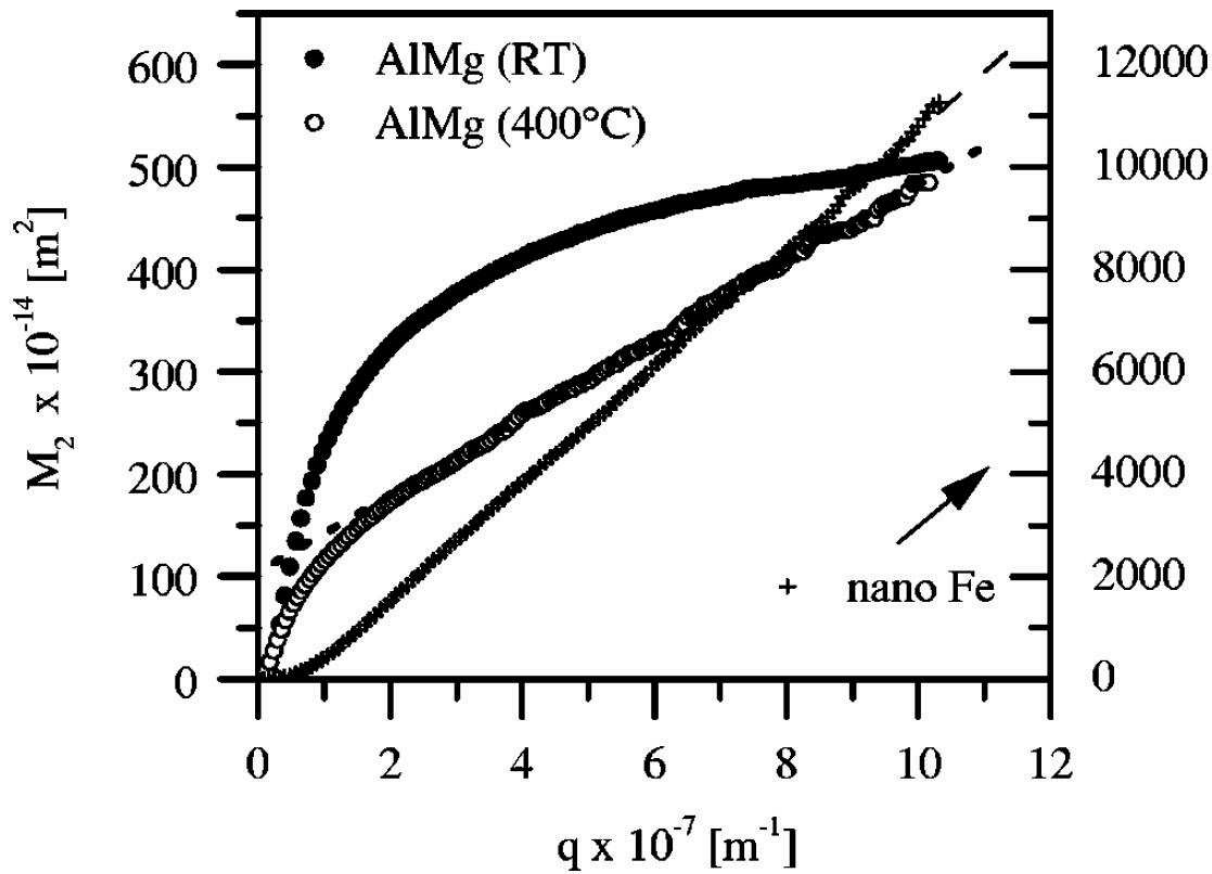
- Asymptotic form of second moment ($\Lambda = \frac{\pi}{2} g^2 b^2 C$):

$$M_2(q) = \frac{2}{\pi^2 d} q + \frac{\Lambda < \rho >}{2\pi^2} \log\left(\frac{q}{q_0}\right),$$

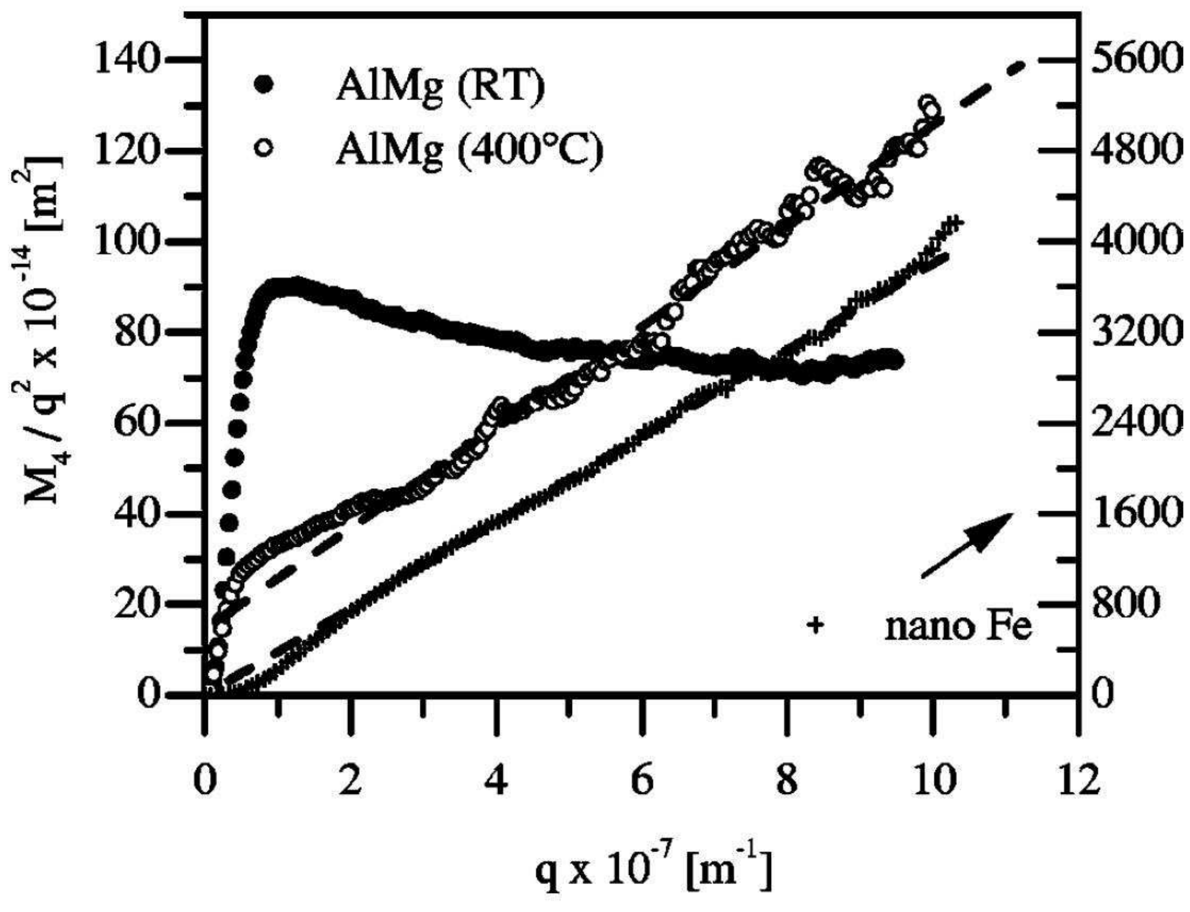
- Asymptotic form of fourth moment (divided by q^2):

$$\frac{M_4(q)}{q^2} = \frac{2}{3\pi^2 d} q + \frac{\Lambda < \rho >}{4\pi^2}$$

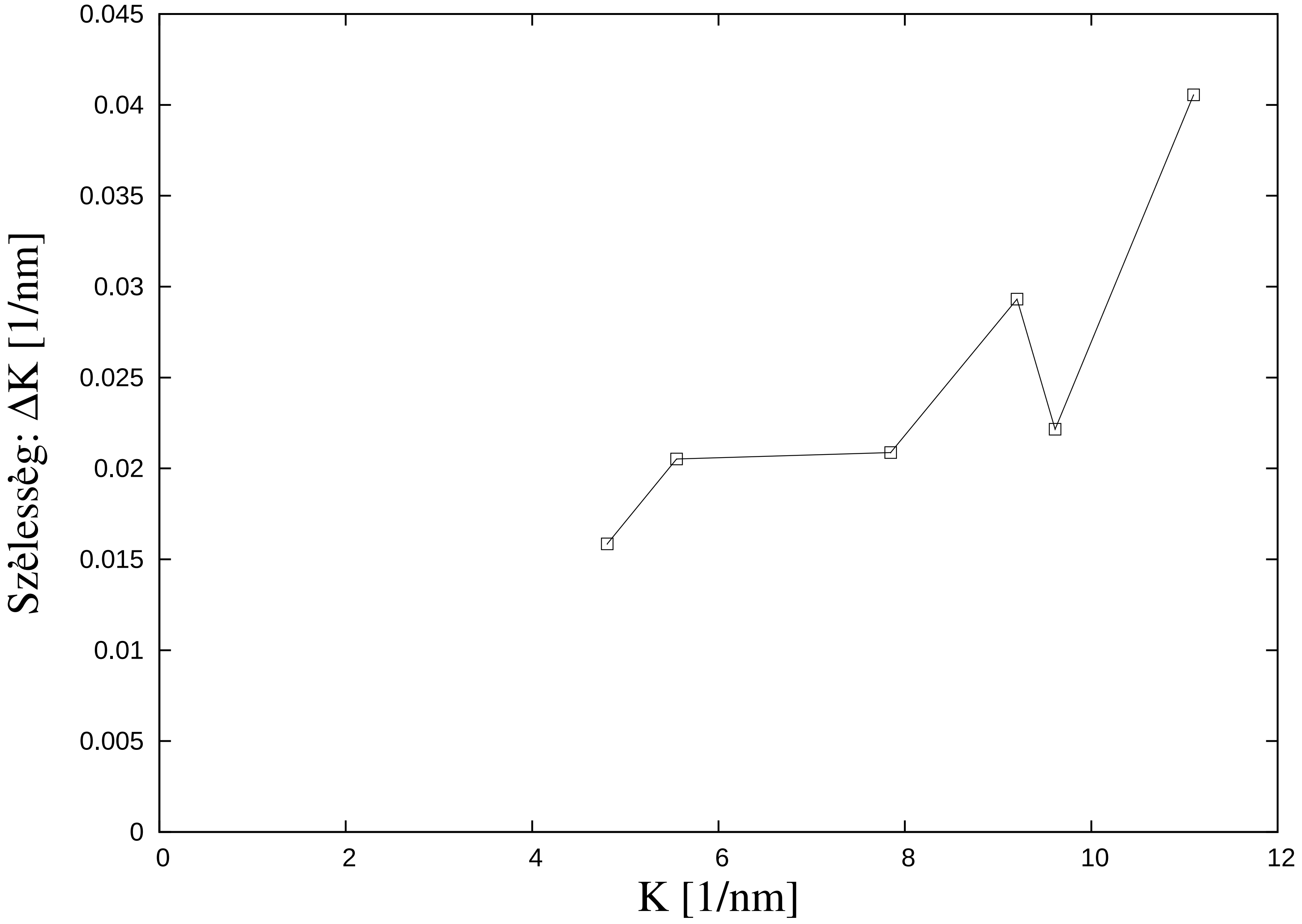
Moments (Borbély & Groma, 2001)



Moments (Borbély & Groma, 2001)

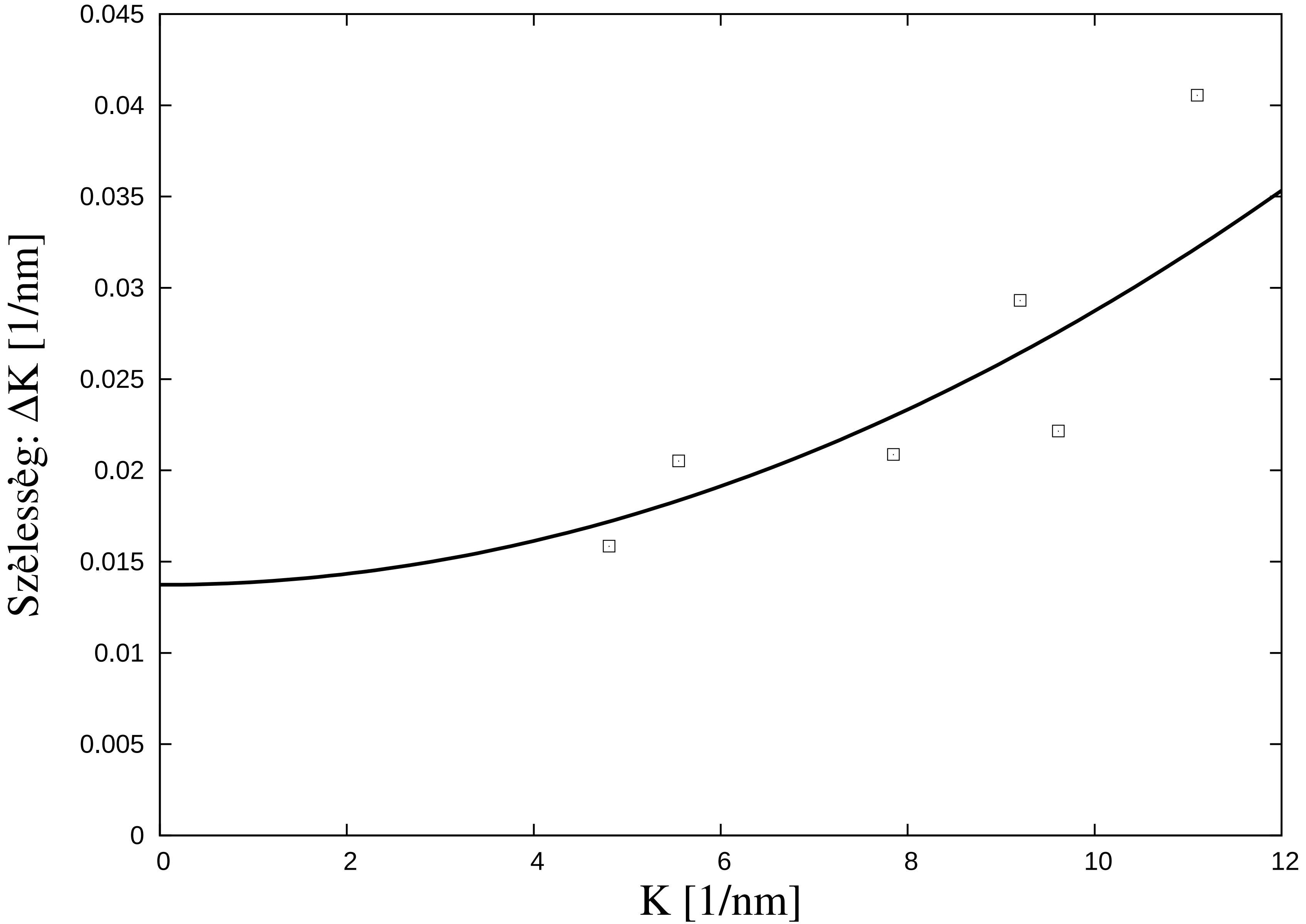


Deformation anisotropy



Williamson-Hall plot for FWHM (Cu sample).

Deformation anisotropy



Williamson-Hall plot for FWHM (Cu sample).

slocation model for deformation anisotropy

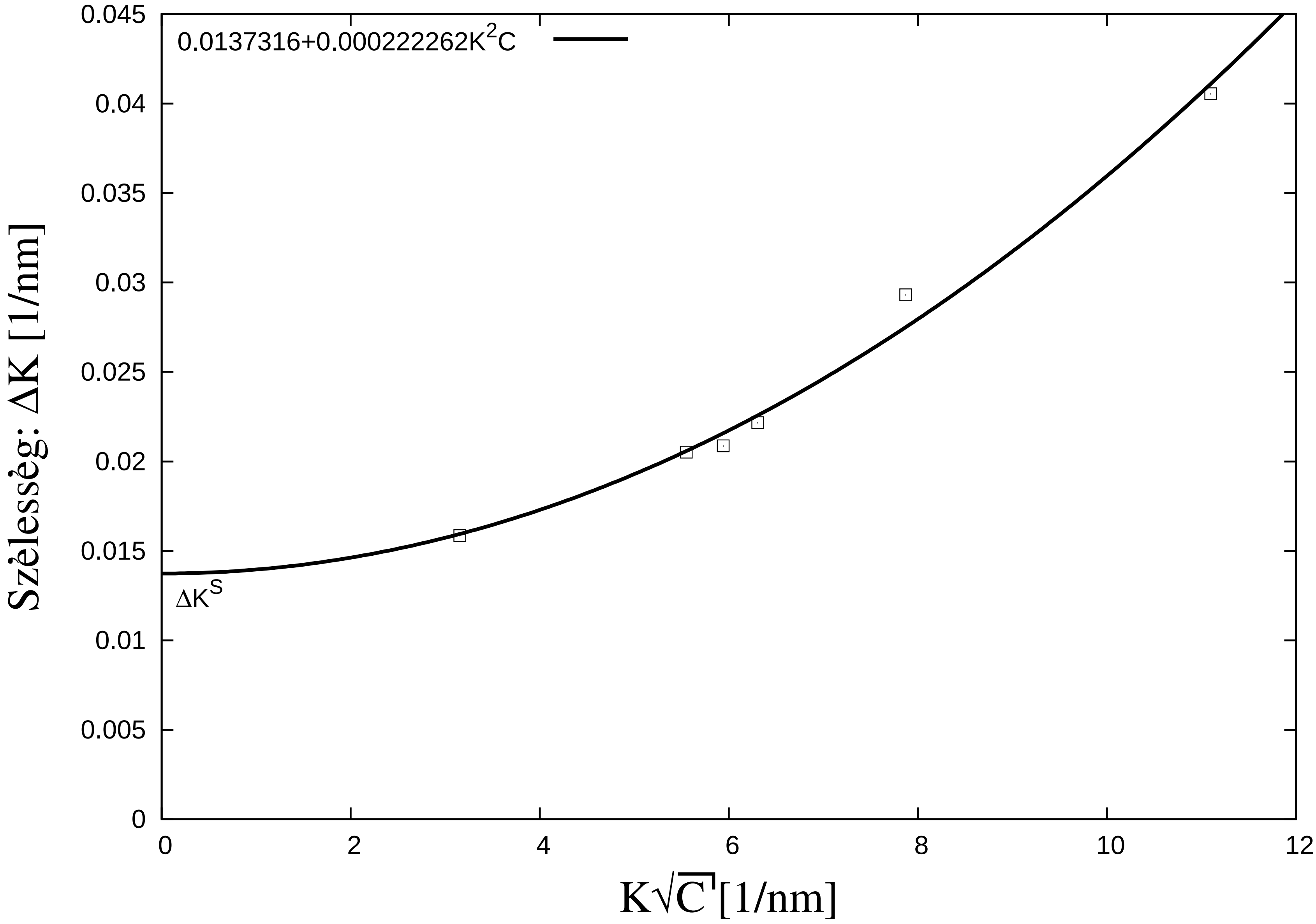
- Kuzel & Klimanek, 1998, JAC
- Ungár & Borbély, 1996, APL
- Ungár & Tichy, 1999, phys. stat. sol.

For cubic crystals:

$$C = C_{h00}(1 - qH^2),$$

$$H^2 = \frac{h^2k^2 + h^2l^2 + k^2l^2}{(h^2 + k^2 + l^2)^2}.$$

Deformation anisotropy



Modified Williamson-Hall plot for FWHM (Cu sample).

Modeling of strain effect

Warren & Averbach (1952):

$$A(L) = A^M(L)A^D(L),$$

$$A^D(L) = \exp(-2\pi^2 K^2 L^2 \langle \varepsilon_L^2 \rangle),$$

$\langle \varepsilon_L^2 \rangle$ mean square *strain*.

Modeling of strain effect

- Krivoglaz & Ryaboshapka (1963): randomly distributed dislocations

$$\langle \varepsilon_L^2 \rangle \approx \left(\frac{b}{2\pi}\right)^2 \pi \rho C \log\left(\frac{D}{L}\right),$$

D is the crystallite size

- Wilkens (1970): restrictedly random dislocation distribution

$$\langle \varepsilon_L^2 \rangle \approx \left(\frac{b}{2\pi}\right)^2 \pi \rho C \log\left(\frac{R_e}{L}\right),$$

R_e is the outer cut-off radius of dislocations.

The Wilkens strain profile

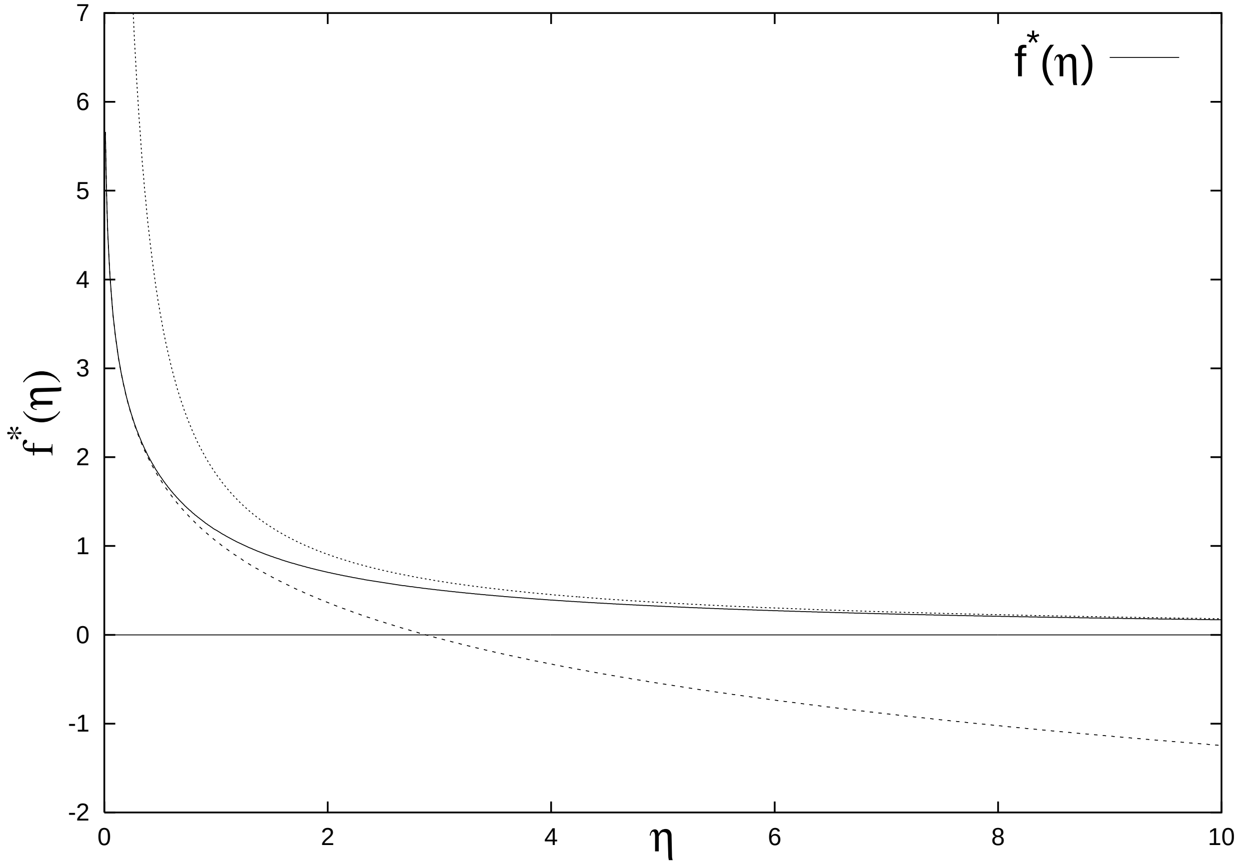
$$A^D(L) = \exp(-2\pi^2 K^2 L^2 \langle \varepsilon_L^2 \rangle),$$

$$\langle \varepsilon_L^2 \rangle = \left(\frac{b}{2\pi} \right)^2 \pi \rho C f^* \left(\frac{L}{R_e} \right),$$

f^* is the Wilkens strain function (Wilkens, 1970).

The Wilkens profile together with the model of strain anisotropy is implemented in the multi profile analysis method.

The Wilkens function



The Wilkens function as a function of $\eta \approx \frac{L}{R_e}$ and its approximations: $-\log\eta + \left(\frac{7}{4} - \log 2\right)$ és $\frac{512}{90\pi}\frac{1}{\eta}$.

Modeling of size effect

- size isotropy
- lognormal $f(x)$ size distribution:

$$f(x) = \frac{1}{\sqrt{2\pi}\sigma} \frac{1}{x} \exp\left[-\frac{\left(\log\left(\frac{x}{m}\right)\right)^2}{2\sigma^2} \right],$$

(m: mean, σ: variance).

The size profile:

$$I^M(s) = \int_0^\infty \mu \frac{\sin^2(\mu\pi s)}{(\pi s)^2} \operatorname{erfc}\left[\frac{\log\left(\frac{\mu}{m}\right)}{\sqrt{2}\sigma} \right] \mathrm{d}\mu,$$

Modeling of size effect

The Fourier transform of size profile:

$$A^M(L,m,\sigma) = \frac{m^3 \exp\left(\frac{9}{4}(\sqrt{2}\sigma)^2\right)}{3} \operatorname{erfc}\left[\frac{\log\left(\frac{|L|}{m}\right)}{\sqrt{2}\sigma} - \frac{3}{2}\sqrt{2}\sigma\right] -$$

$$\frac{m^2 \exp(\sqrt{2}\sigma)^2}{2} |L| \operatorname{erfc}\left[\frac{\log\left(\frac{|L|}{m}\right)}{\sqrt{2}\sigma} - \sqrt{2}\sigma\right] +$$

$$\frac{|L|^3}{6} \operatorname{erfc}\left[\frac{\log\left(\frac{|L|}{m}\right)}{\sqrt{2}\sigma}\right].$$

Microstructural parameters

Microstructural parameters for whole profile modeling:

- size: m, σ
- dislociations: ρ, M, q
- planar faults: α

The CMWP method

Properties of the CMWP method:

- the whole pattern is fitted
- convolutional method
- theoretical intensity pattern:

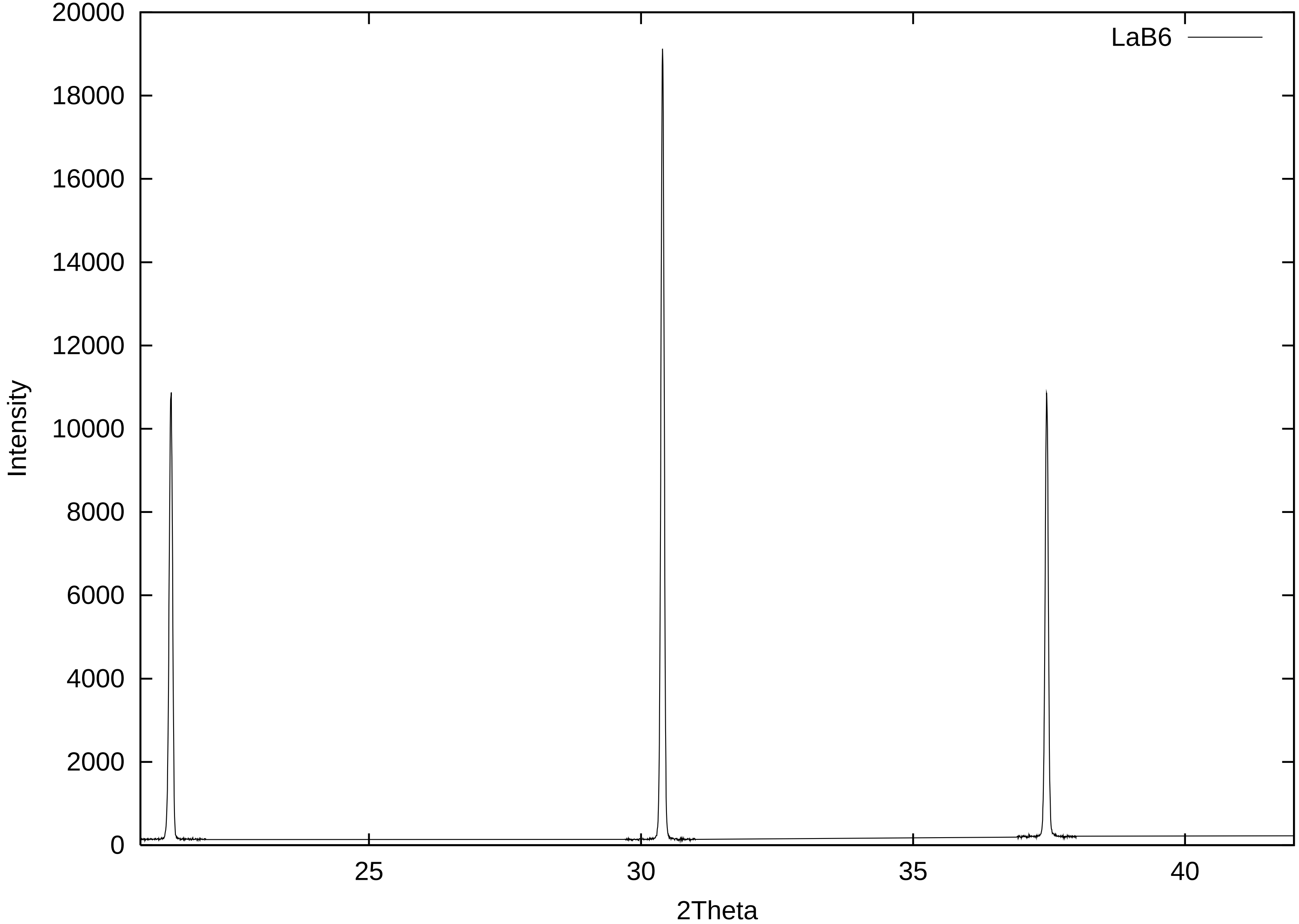
$$I(2\Theta) = BG(2\Theta) + \sum_{hkl} I_{hkl}^{MAX} I_{hkl}(2\Theta - 2\Theta_{hkl}^{0}),$$

$$I_{hkl} = I_{hkl}^{instr.} * \quad I_{hkl}^{M.} * \quad I_{hkl}^{D.} * \quad I_{hkl}^{R.},$$

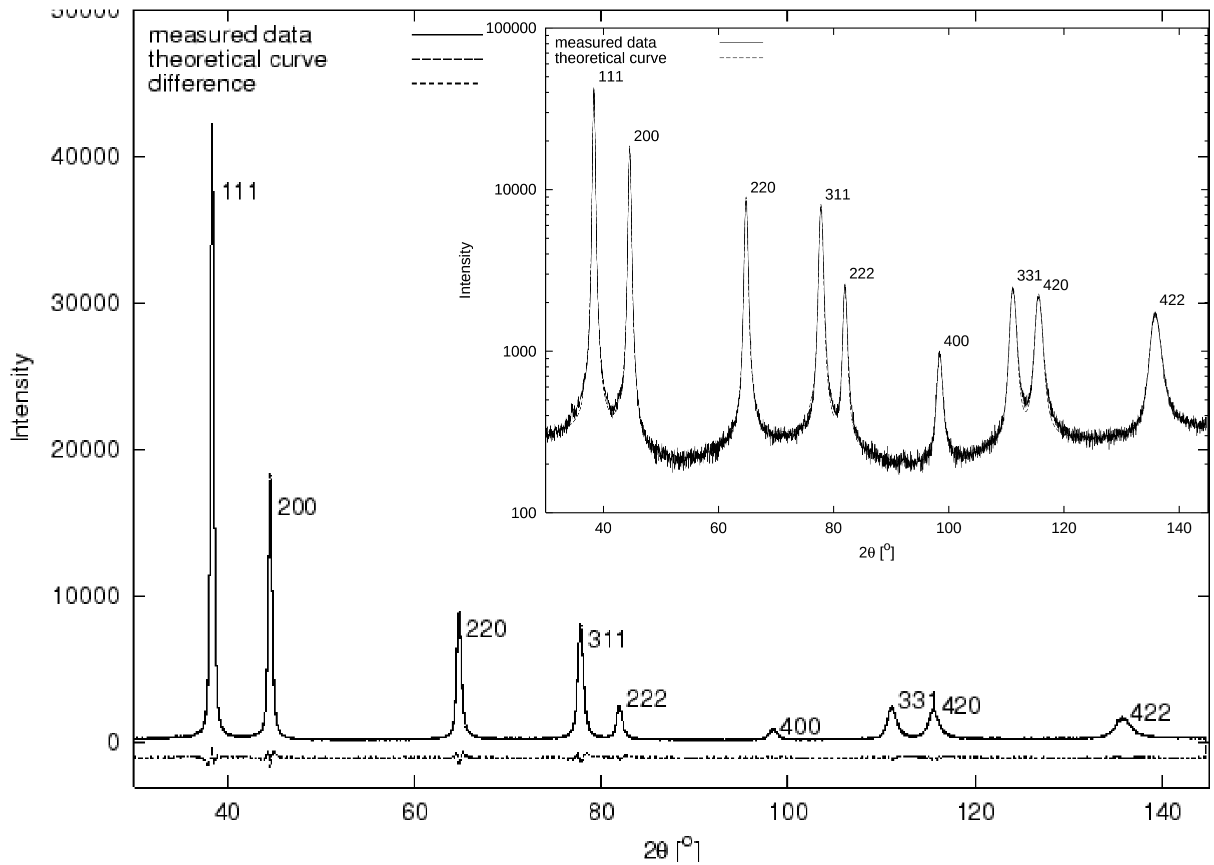
  BG: background spline
  $I_{hkl}^{instr.}$: measured instrumental profile

- microstructural method: the unit cell is NOT fitted.

Instrumental pattern of LaB_6



WP fitting for Al-6Mg ball milled for 6 hour



Results of CMWP fit:

$m = 21\text{nm}$

$\sigma = 0.36$

$\rho = 10^{16}\ \text{m}^{-2}$

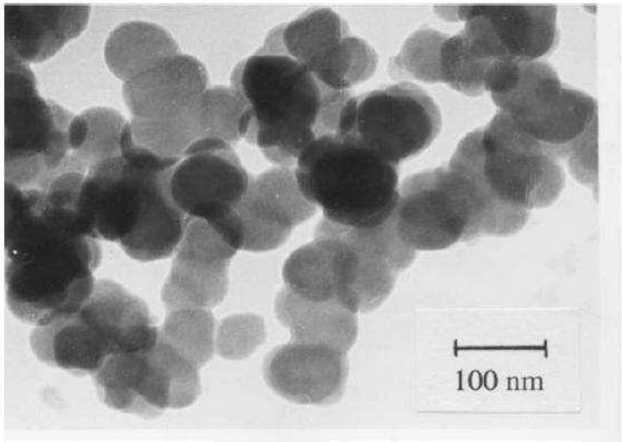
$M = R_e\sqrt{\rho} = 1.3$

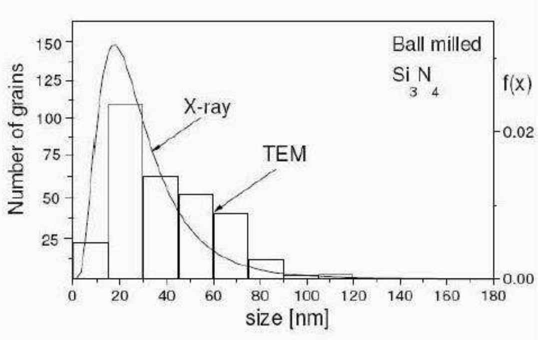
$q = 1.3$

The measures (solid line) and theoretical fitted (dashed line) patterns as a function of 2θ.

The difference plot is also given at the bottom.

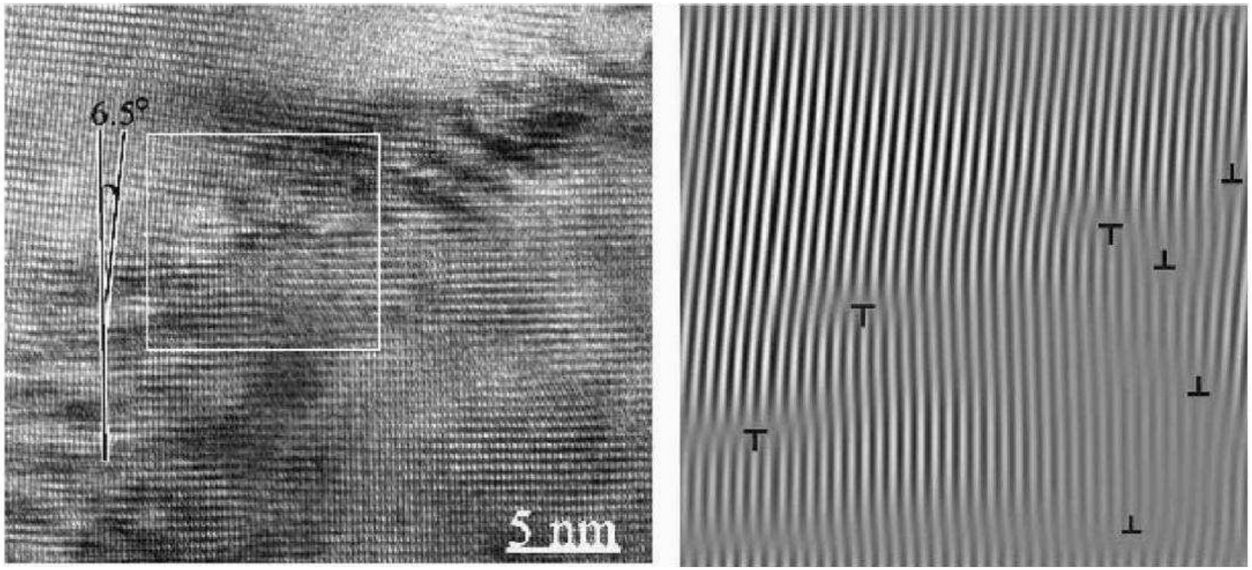
Comparing results with TEM


a)



b)

Comparing results with TEM



Thank you for your attention!