Line Profile Analysis
for
Engineering Materials Science

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Software Workshop on Engineering Diffraction
for ESS Materials Engineering Beamline, 14-15 June, Copenhagen, Denmark

Convolutional Multiple Whole Profile (CMWP) software package

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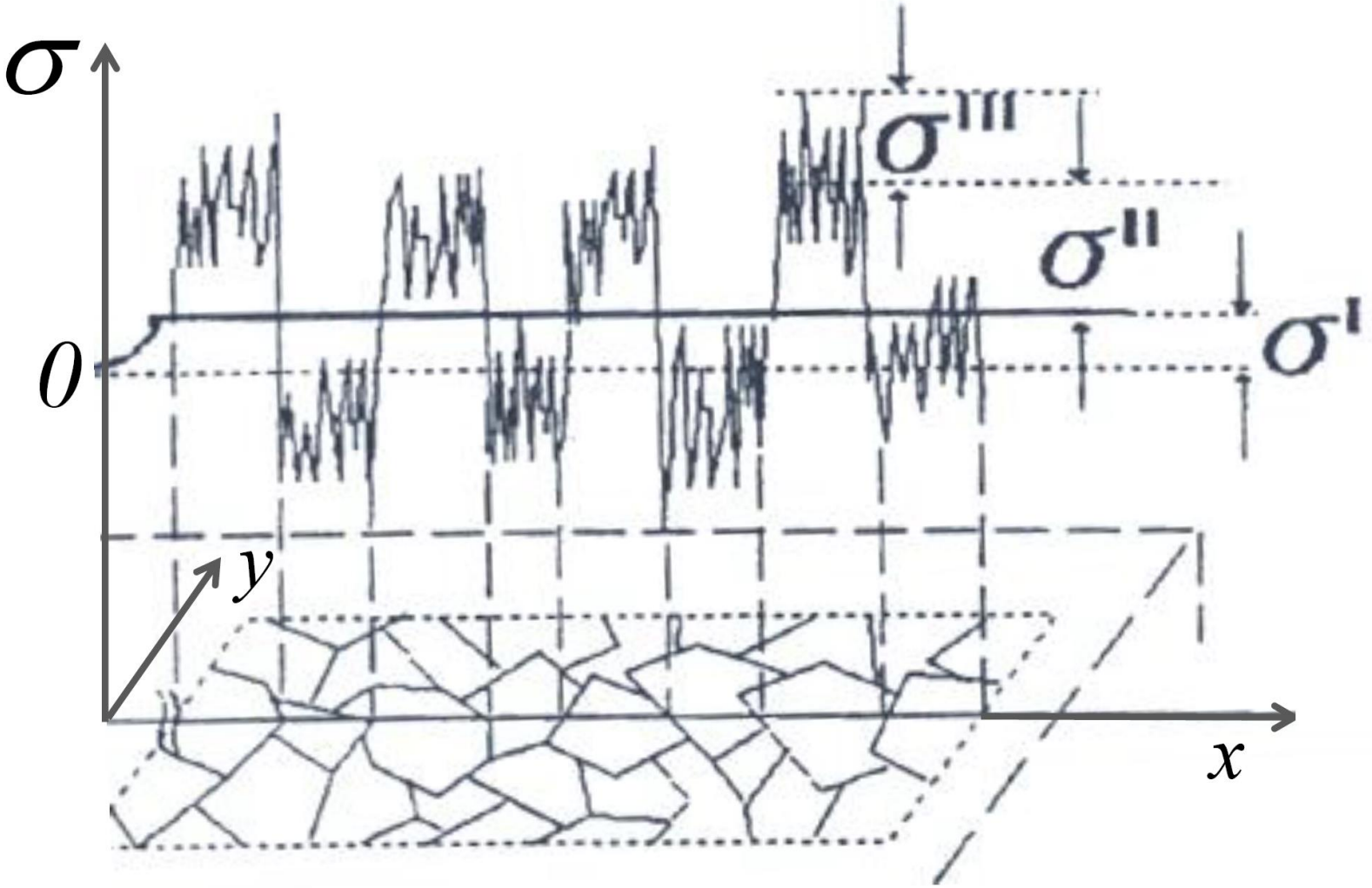
Macherauch, E. (~1965)
schematic classification of **internal stresses**



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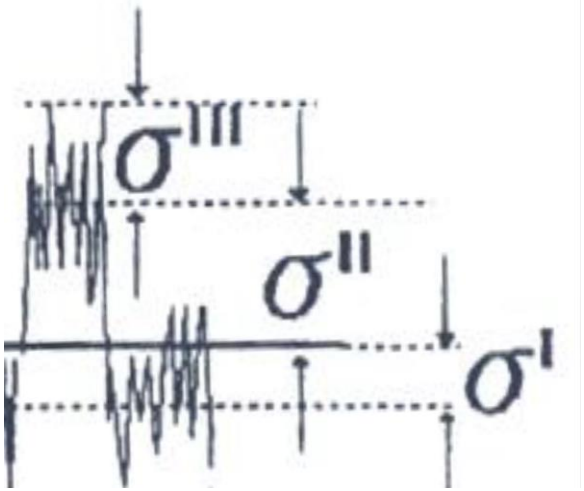


σ_I: ***macro-stress***
averaged over many grains

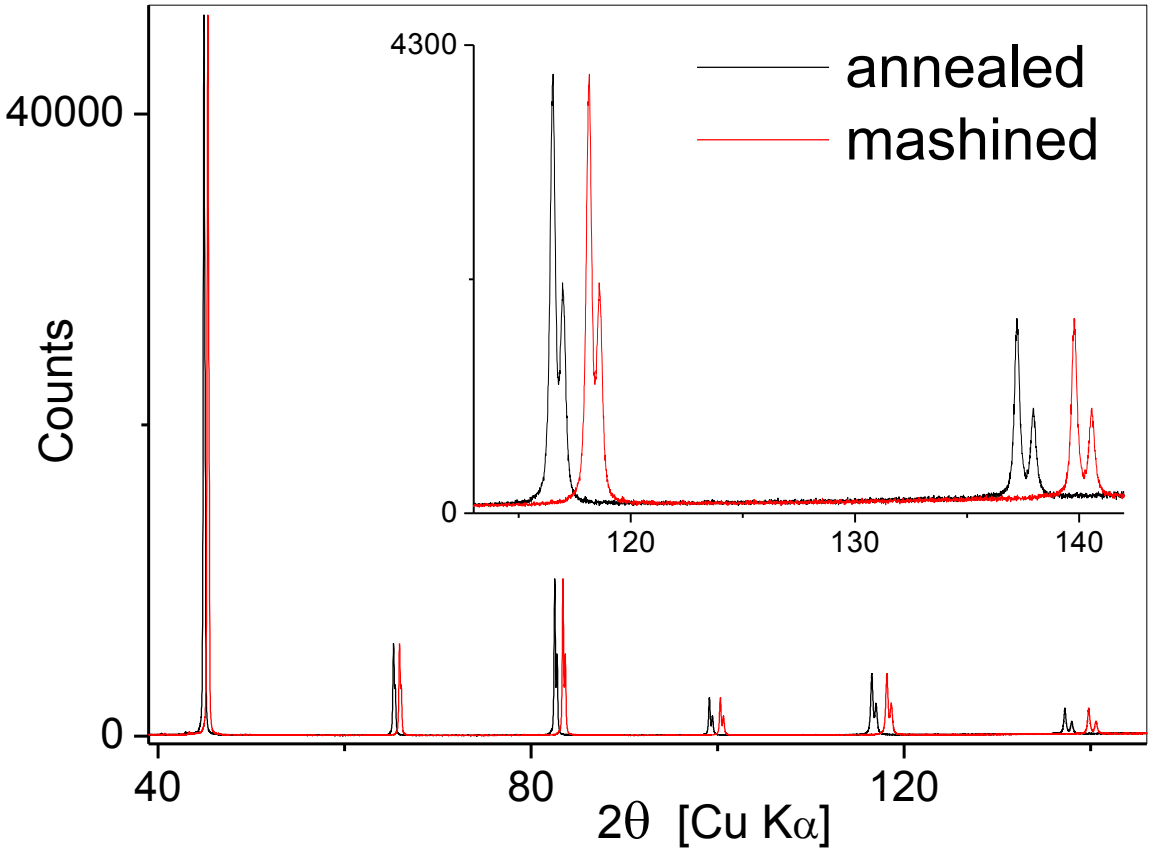
σ_{II}: ***intergranular-stresses***
averaged over individual grains

σ_{III}: ***micro-strains*** **(**or ***stresses*****)**
produced (mainly) by ***dislocations***

Diffraction experiments

σ_I: ***macro-stress***

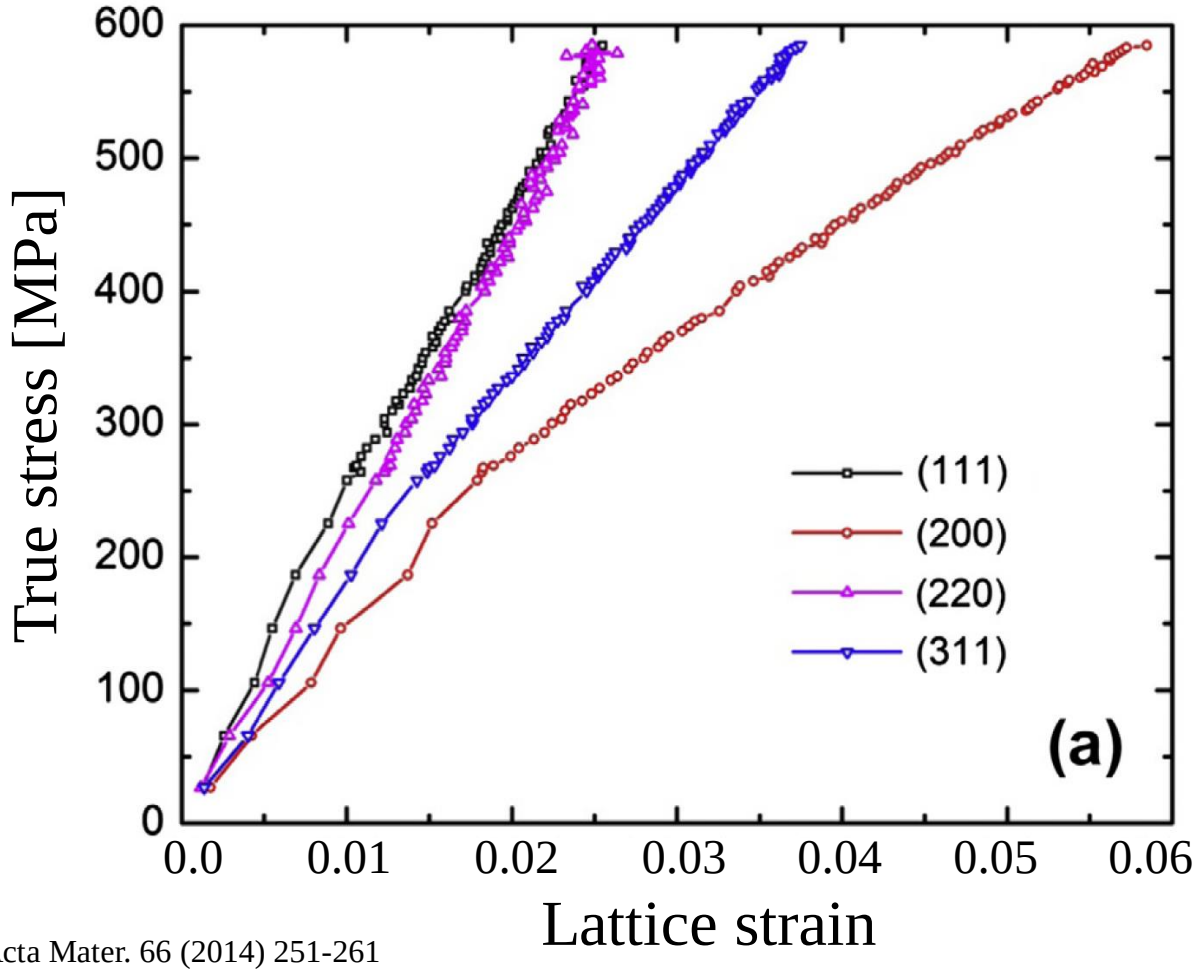
all peaks shift in the ***same direction***



Diffraction experiments

σ_{II}: ***intergranular-***strains/stresses

peak-positions corresponding to ***individual grains:*** **lattice-strain**



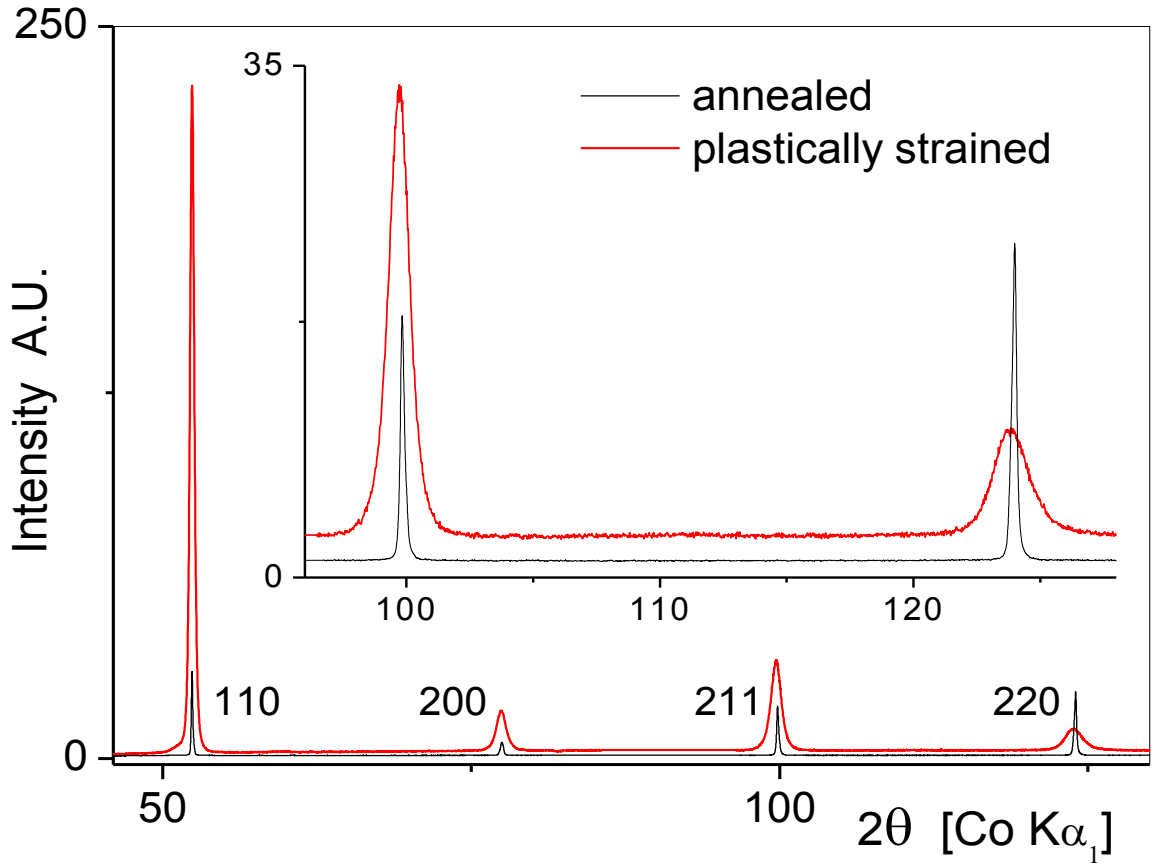
316 stainless steel

Ungár T., Stoica A.D., Tichy G., Wang X.-L. Acta Mater. 66 (2014) 251-261

Diffraction experiments

σ_{III}: ***micro-strains*** (or ***stresses***)

are produced by ***dislocations***: peaks ***broaden***

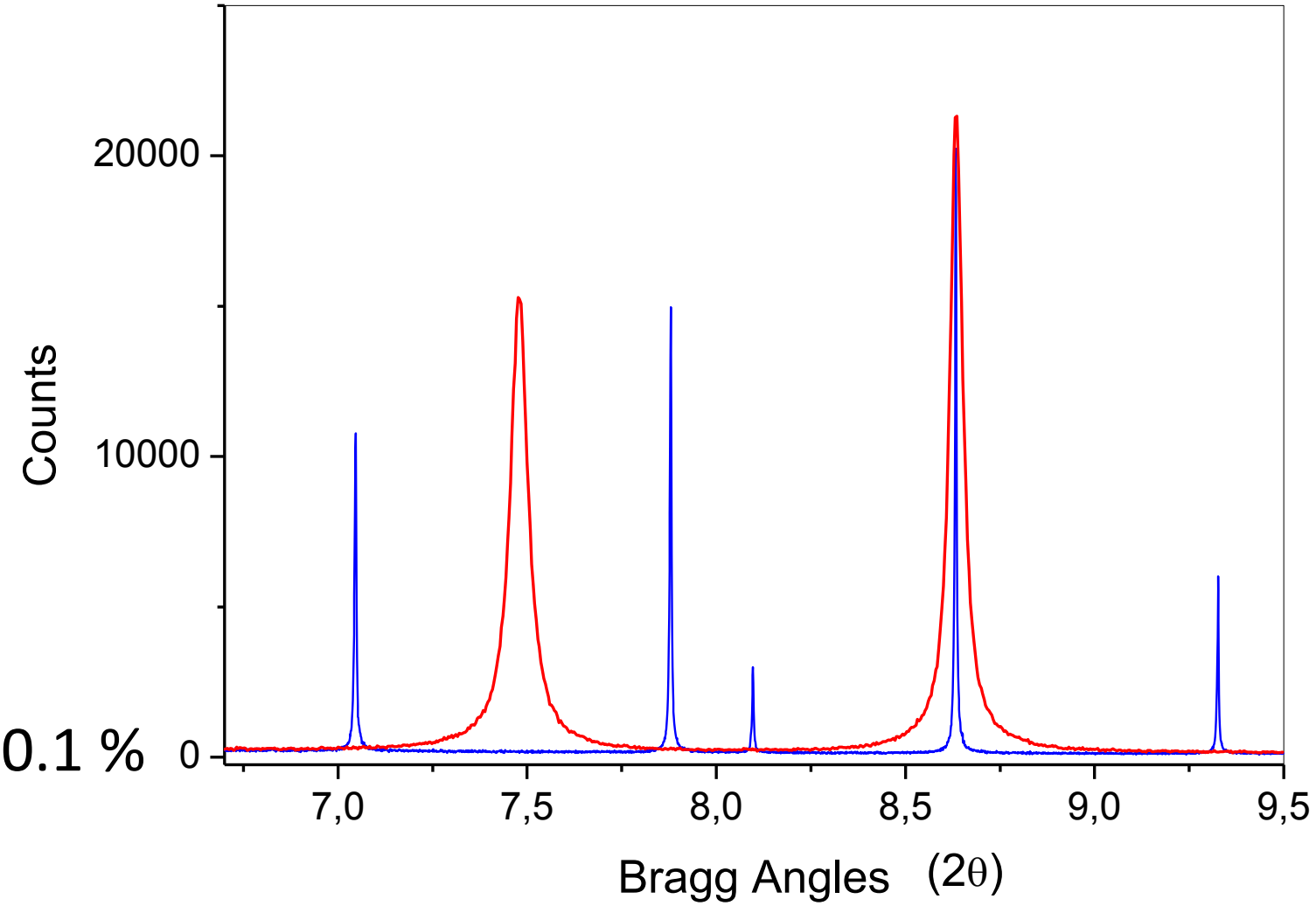


ESRF:

$\Delta K/K \cong 0.1\ \%$

Instrumental effects can be substantial, but can be measured

X-ray diffraction:

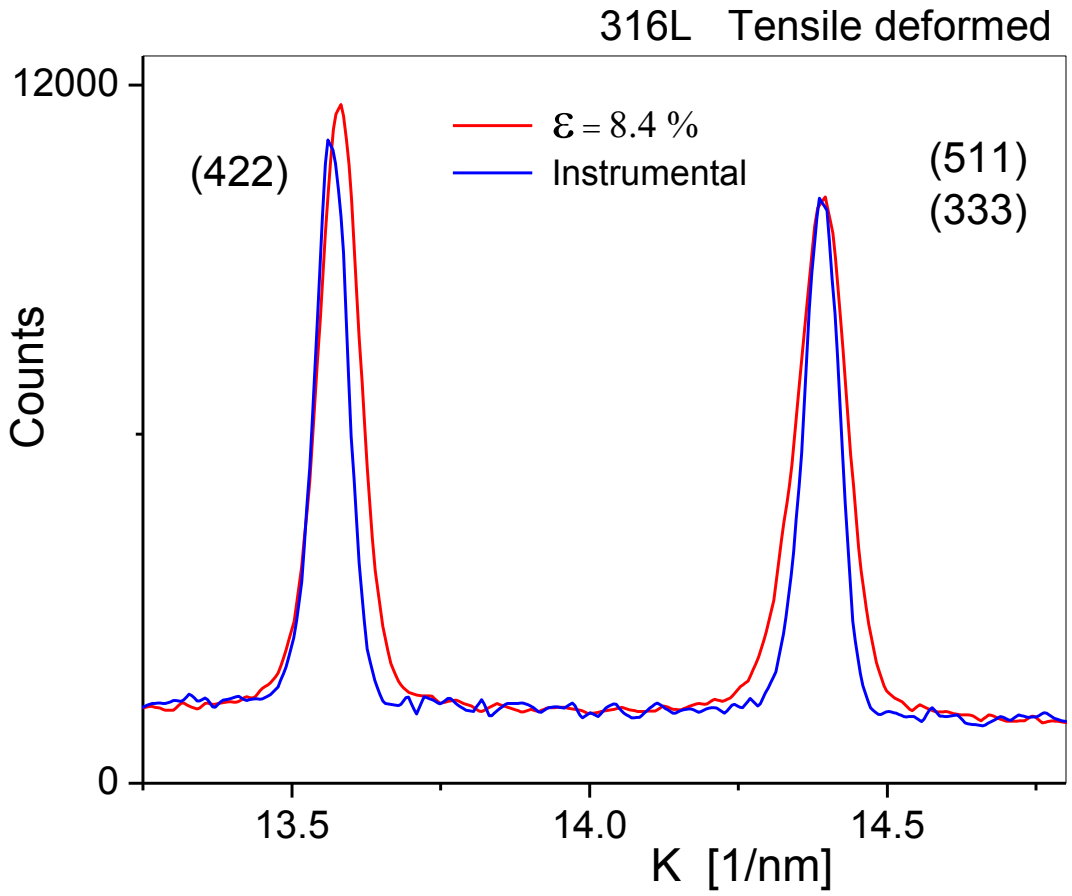


ESRF:

$\Delta K/K \cong 0.1\ \%$

Instrumental effects can be substantial, but can be measured

neutron diffraction: SNS-VULCAN

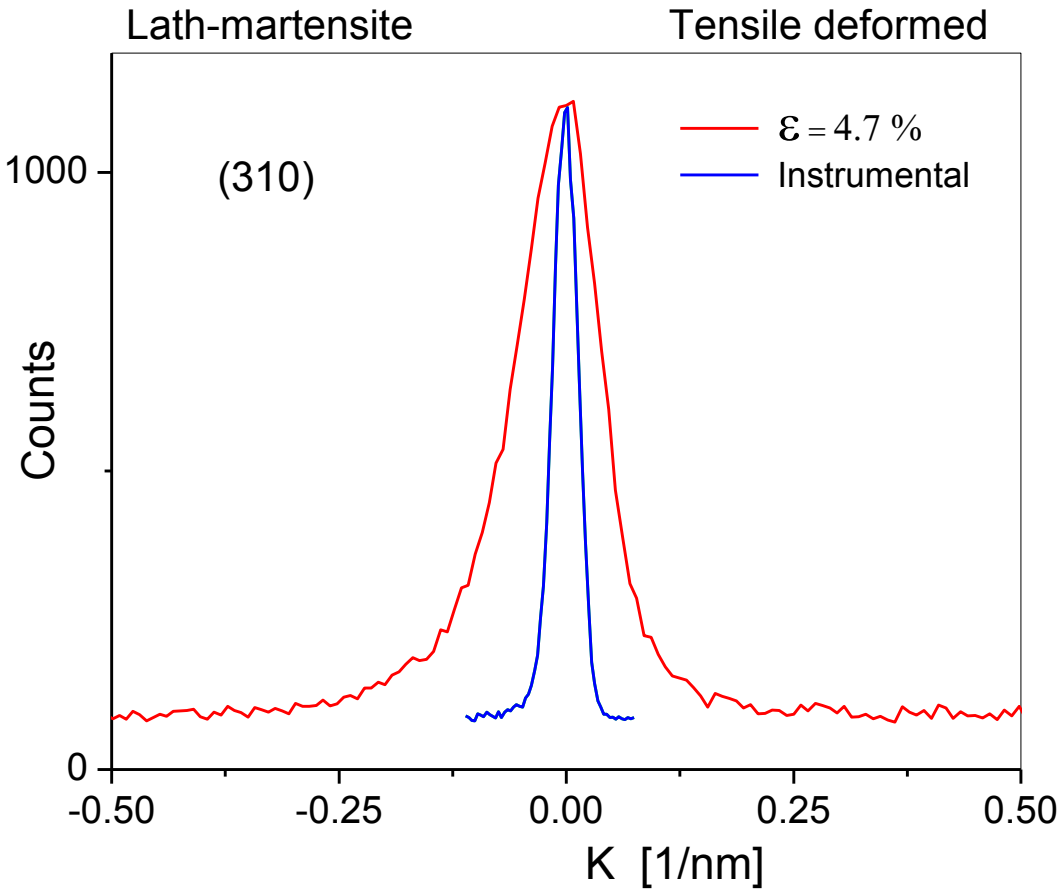


$\Delta K/K \cong 0.5\ \%$

Ungár T., Stoica A.D., Tichy G., Wang X.-L. Acta Mater. 66 (2014) 251-261

Instrumental effects can be substantial, but can be measured

neutron diffraction: TAKUMI J-PARC



$\Delta K/K \cong 0.3\ \%$

peak asymmetry is genuine

Ungár T., S. Harjo, T. Kawasaki, Y. Tomota, G. Ribárik, Z. Shi, Met. Mater. Transact. A48 (2017) 159-167.

Two different approaches to line-profile-analysis:

Top-down: diffraction patterns are

fitted by **analytical** profile-functions

usually pseudo-Voight or Pearson-VII

Bottom-up:

profile-functions are

determined for **actual lattice defects**

based on **well-established physical principles**

diffraction patterns are fitted by theoretically calculated

defect-related profile-functions

For many good reasons CMWP follows the

bottom-up approach

The hierarchy of lattice defects

Krivoglaz: the spatial dependence of strain: $\varepsilon(r)$

---

0 dimensional:

point-defects, point-defect-type, e.g. GP-zones, small particles

$$\varepsilon(r) \sim 1/r^2$$

---

1 dimensional: **dislocations**

non-equilibrium triple-junctions, linear-type defects

$$\varepsilon(r) \sim 1/r$$

---

2 dimensional: planar defects, e.g.

stacking-faults, twin-boundaries, grain-boundaries, etc.

$$\varepsilon(r) \sim \text{constant}$$

crystal-space *versus* **reciprocal-space**

short distance [m] → **long** distance [1/m]

long distance [m] → **short** distance [1/m]

The hierarchy of lattice defects

Krivoglaz: the spatial dependence of strain: $\varepsilon(r)$

---

0 dimensional: $\boldsymbol{\varepsilon(r) \sim 1/r^2}$ point-defects, GP-zones

diffuse scattering **below** the Bragg reflections

---

1 dimensional: $\boldsymbol{\varepsilon(r) \sim 1/r}$ **dislocations**

broadening **around** the Bragg reflection

---

2 dimensional: $\boldsymbol{\varepsilon(r) \sim}$ **constant** planar defects

peak shifts

Fundamental equation for line-broadening: Warren [1958]:

$$A_L(g) \cong A_L^S \exp\{-2\pi^2 L^2 g^2 \langle \varepsilon_{g,L}^2 \rangle\}$$

Strain Fourier coefficients

Dislocation-model of $\langle \varepsilon_{g,L}^2 \rangle$: **Krivoglaz-Wilkens** [1963-1970]:

$$\langle \varepsilon_{g,L}^2 \rangle = \frac{\rho \cdot C \cdot b^2}{4\pi} f(\eta)$$

$\boldsymbol{b}$: Burgers vector

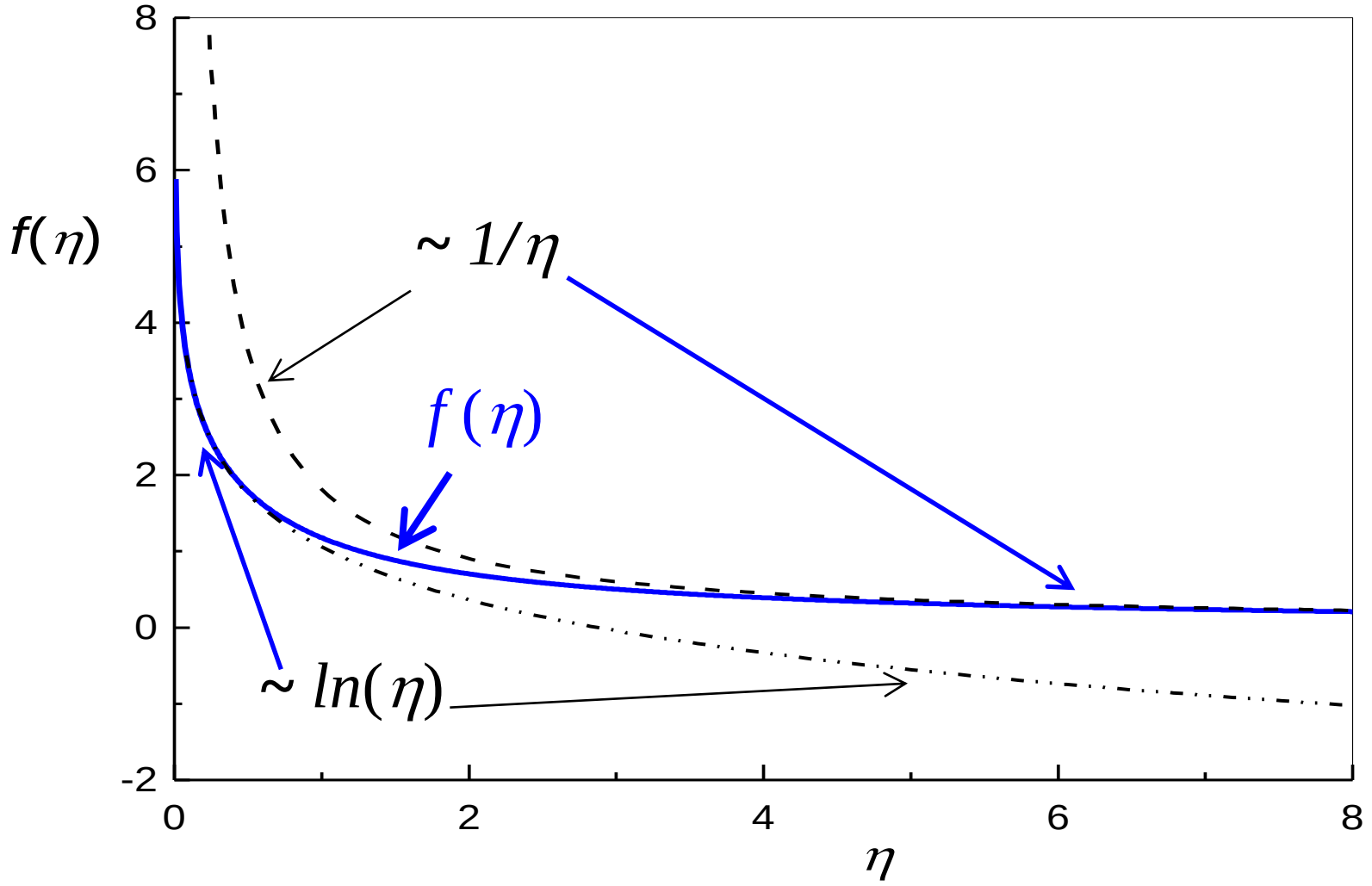
ρ : dislocation density

C : Contrast factor of dislocations

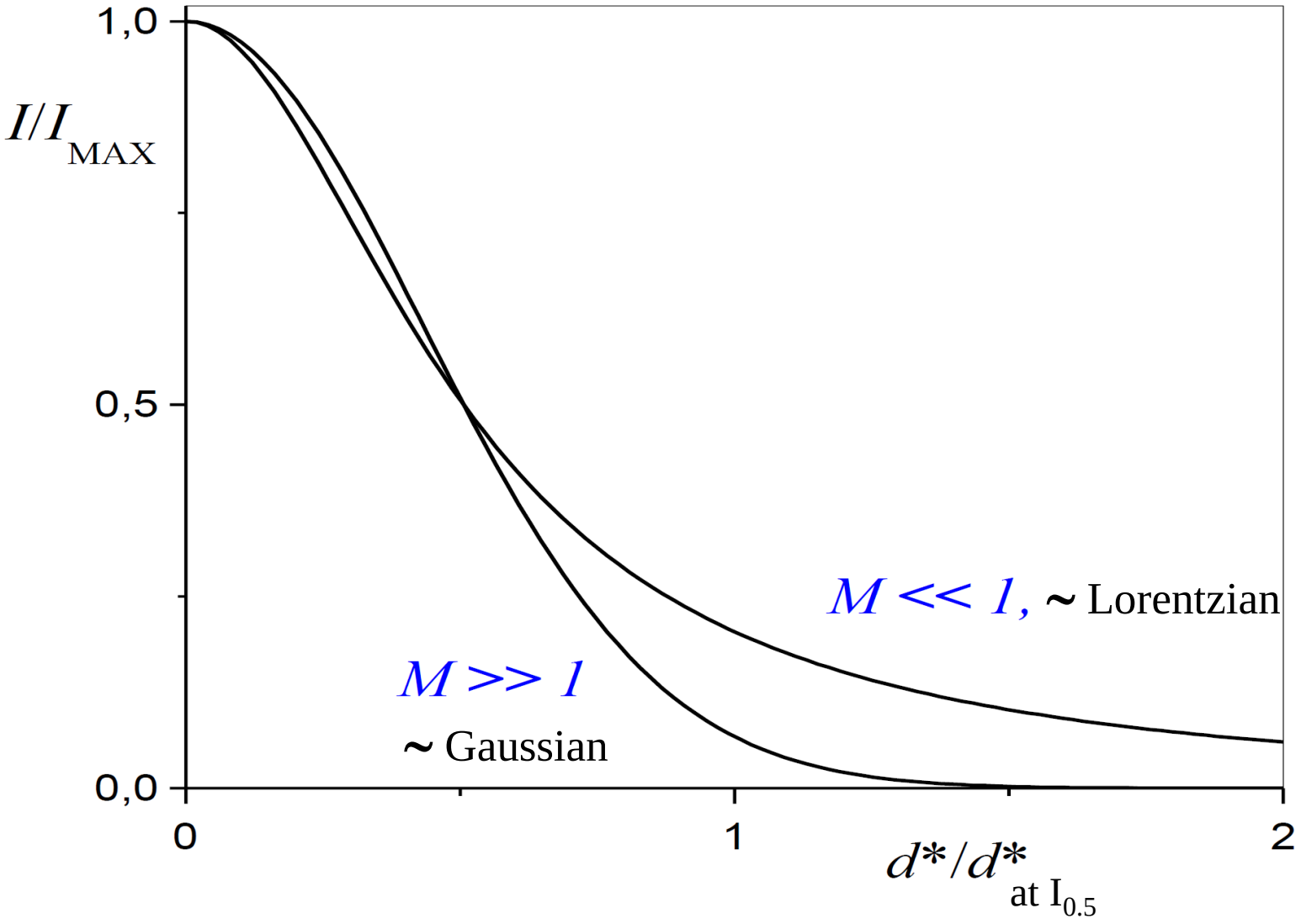
$f(\eta)$: Wilkens function [1970]

$$\eta = L/\boldsymbol{R}_e$$

The Krivoglaz-Wilkens function [1963-1970]

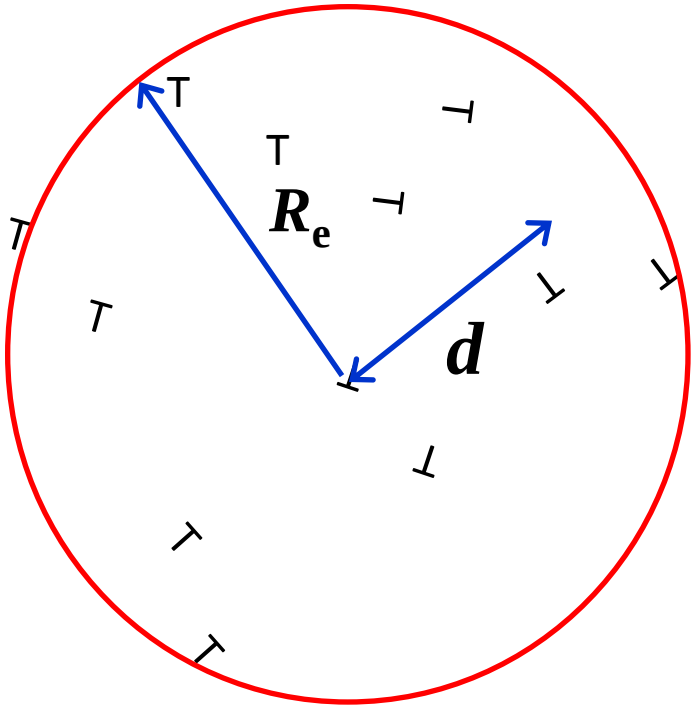


shape of strain-profiles



M arrangement parameter, **dipole character** of dislocations

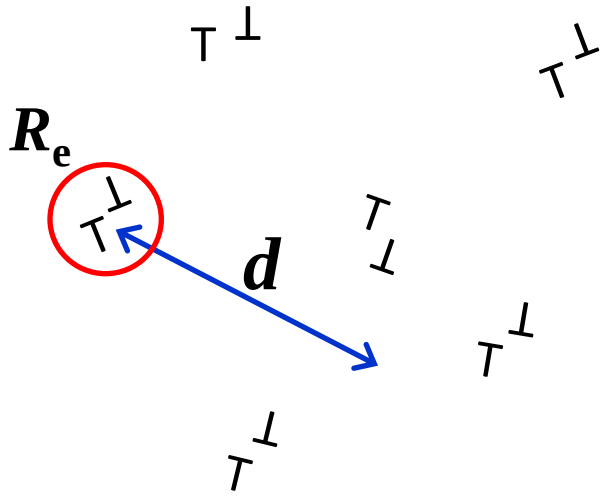
weak dipole character



$R_e > d$

$M = R_e/d = R_e\sqrt{\rho} > 1$

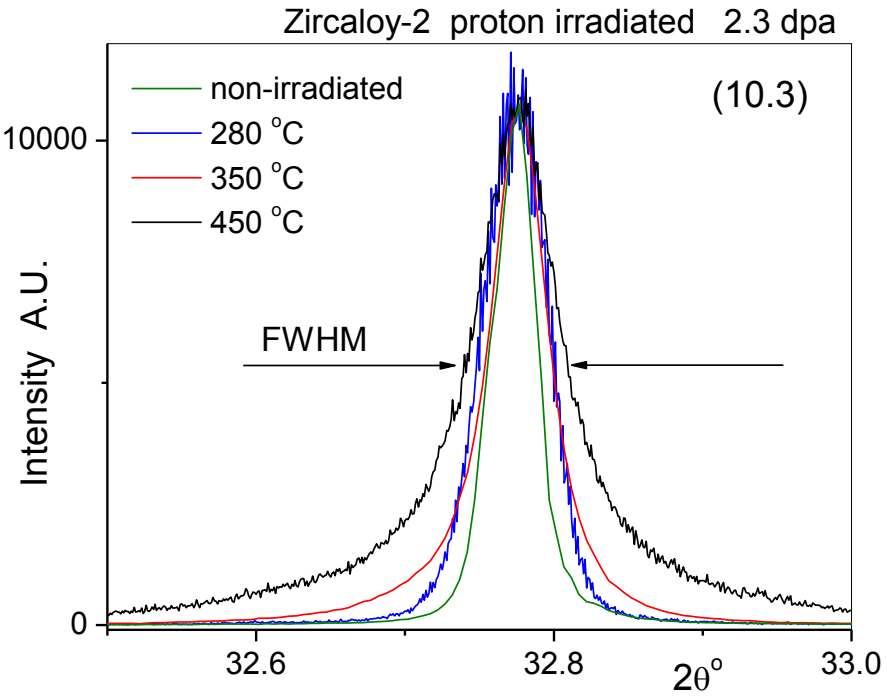
strong dipole character



$R_e < d$

$M = R_e/d = R_e\sqrt{\rho} < 1$

dislocation loops can have strong dipole character



irradiation at:

280 °C

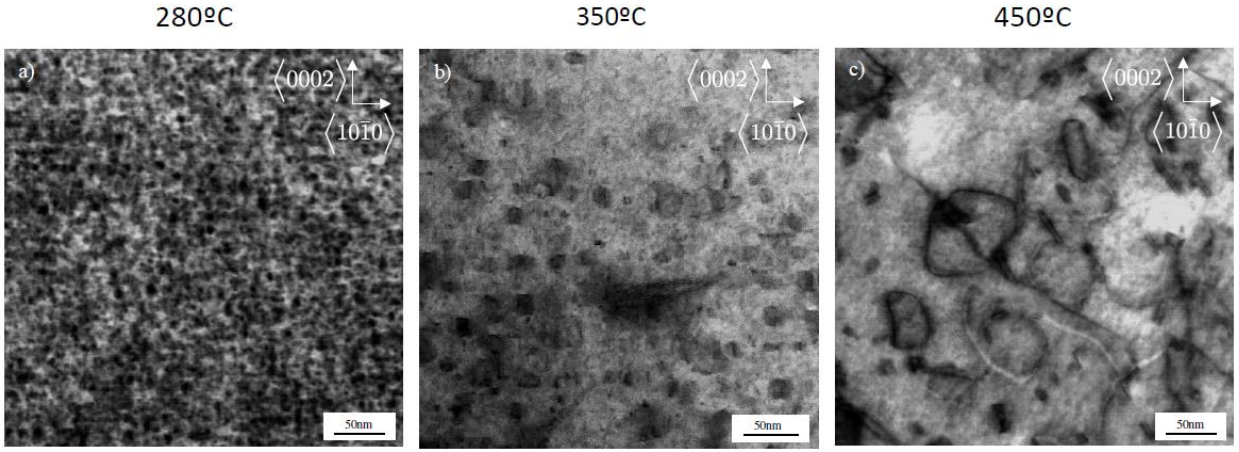
small loops, with large density

350 °C

small loops, with medium density

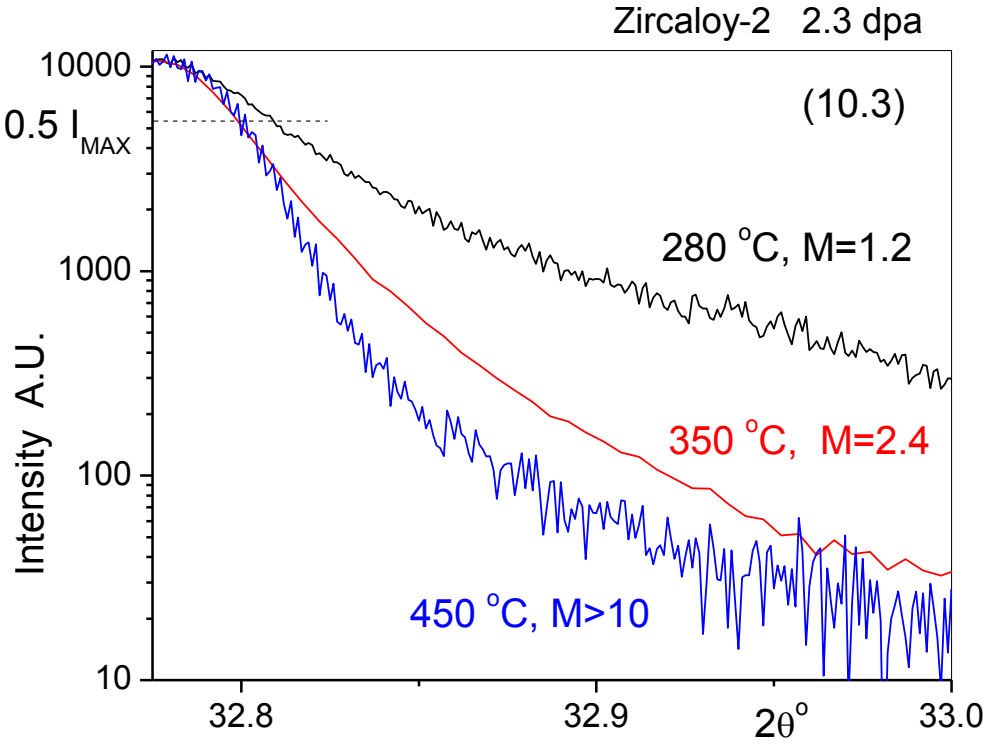
450 °C

large loops, with smaller density



M. Topping et al. *Acta Mater, accepted*

dislocation loops can have strong dipole character



irradiation at:

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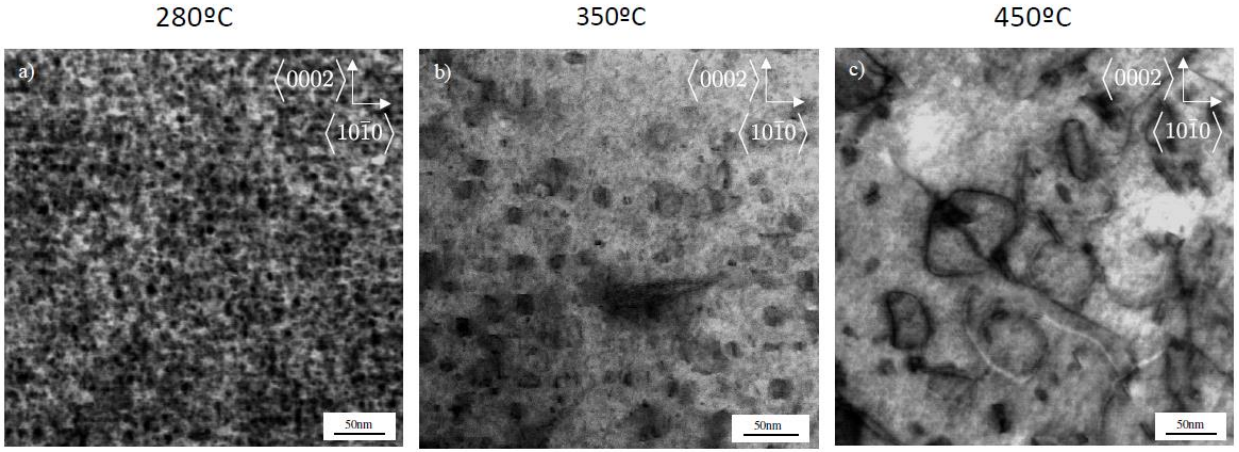
small loops, with medium density

450 °C
large loops, with smaller density



M. Topping et al. *Acta Mater, accepted*

Strain profile:

Fourier transform of the *strain Fourier coefficients*

$$I^D(s) = \int \exp\{ - 2\pi^2 L^2 g^2 \langle \varepsilon_{L,\boldsymbol{g}}{}^2 \rangle \} \exp(2\pi i L s) \mathrm{d}L$$

where: $\langle \varepsilon_{g,L}^2 \rangle = \dfrac{\rho \cdot C \cdot b^2}{4\pi} f(\eta)$

dislocation - density : ρ

- arrangement parameter: M

contrast factor - strain anisotropy: $q_1 (q_2)$

Size profile: I^S

assuming **log-normal** size distribution:

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

m: median

σ: variance

$$I^S(s) = \int_0^\infty \mu \, \frac{\sin^2(\mu\pi s)}{(\pi s)^2} \, \mathrm{erfc}\left[\frac{\log(\mu/m)}{2^{1/2}\sigma}\right] \mathrm{d}\mu$$

CMWP method (Convolutional Multiple Whole Profile)

Philosophy of evaluation

Measured pattern ***Physically modeled*** ***Experimentally determined***

Combined

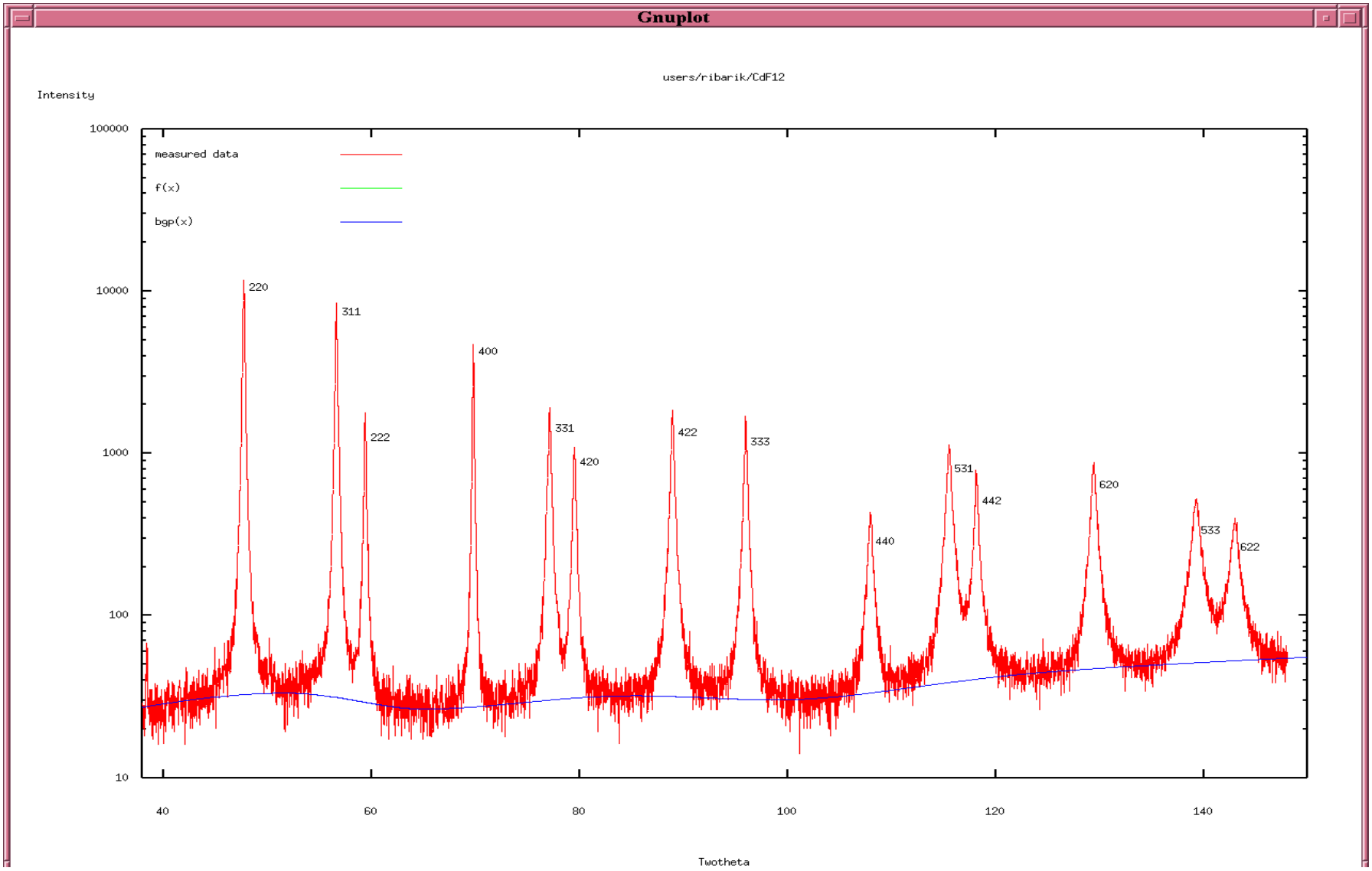
Levenberg–Marquardt and Monte-Carlo algorithms

applied in a cyclically alternating sequence

Gábor Ribárik, Bertalan Jóni, Tamás Ungár
Global Minimum in the Convolutional-Multiple-Whole-Profile (CMWP) line profile analysis procedure by implementing Monte-Carlo optimization
J. App. Cryst. to be submitted

CdF_2 ball milled for 12 min

G. Ribárik, N. Audebrand, H. Palancher, T. Ungár and D. Louër, *J. Appl. Cryst.* (2005). 38, 912–926



Powder pattern file

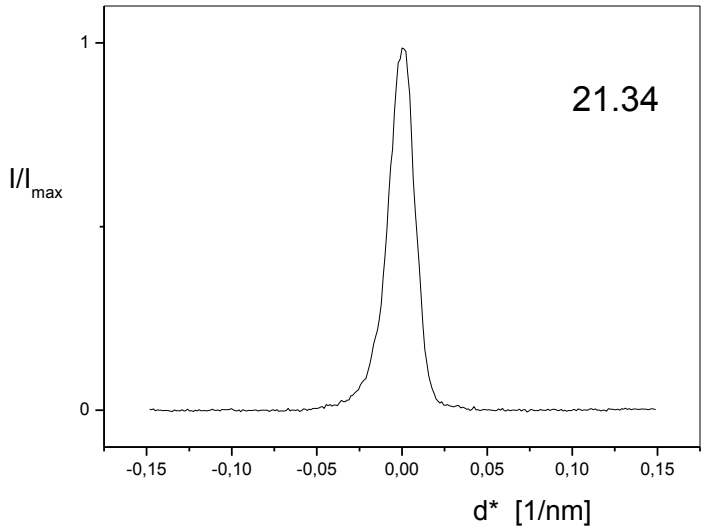
2-column file, contains: 2Θ, I or d*(1/nm, I)

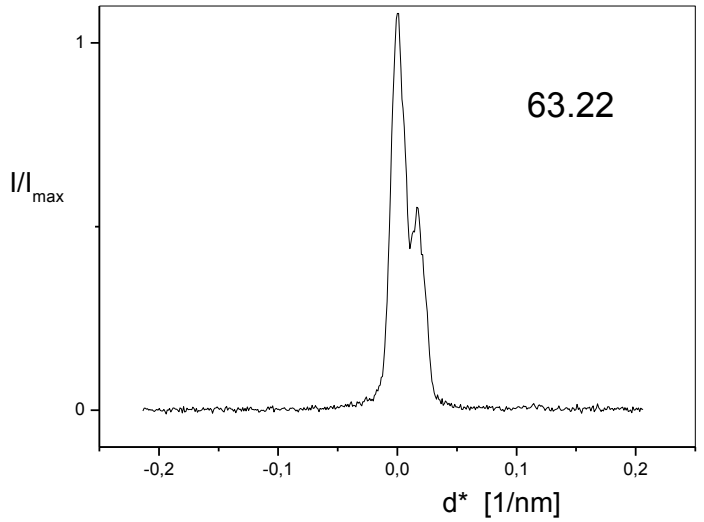
2θ	I
⋮	
100.7800	335
100.7950	331
100.8100	335
100.8250	331
100.8400	342
100.8550	335
⋮	

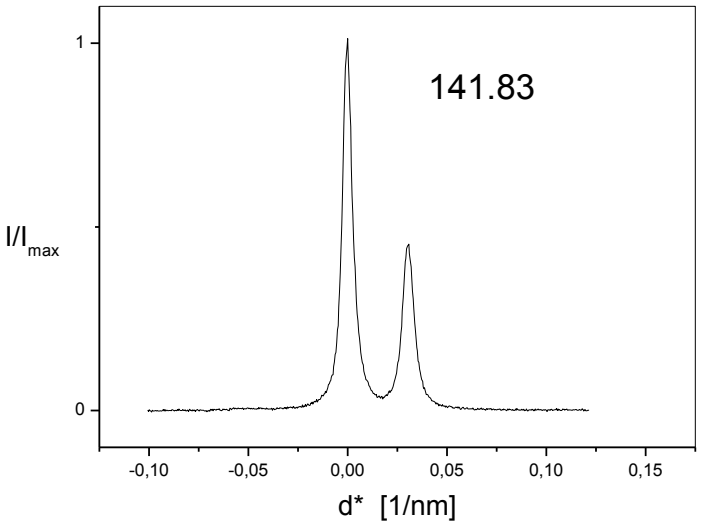
or

d*(1/nm)	I
.	
1.005	120
1.010	122
1.025	118
1.030	130
1.035	126
.	
.	
.	

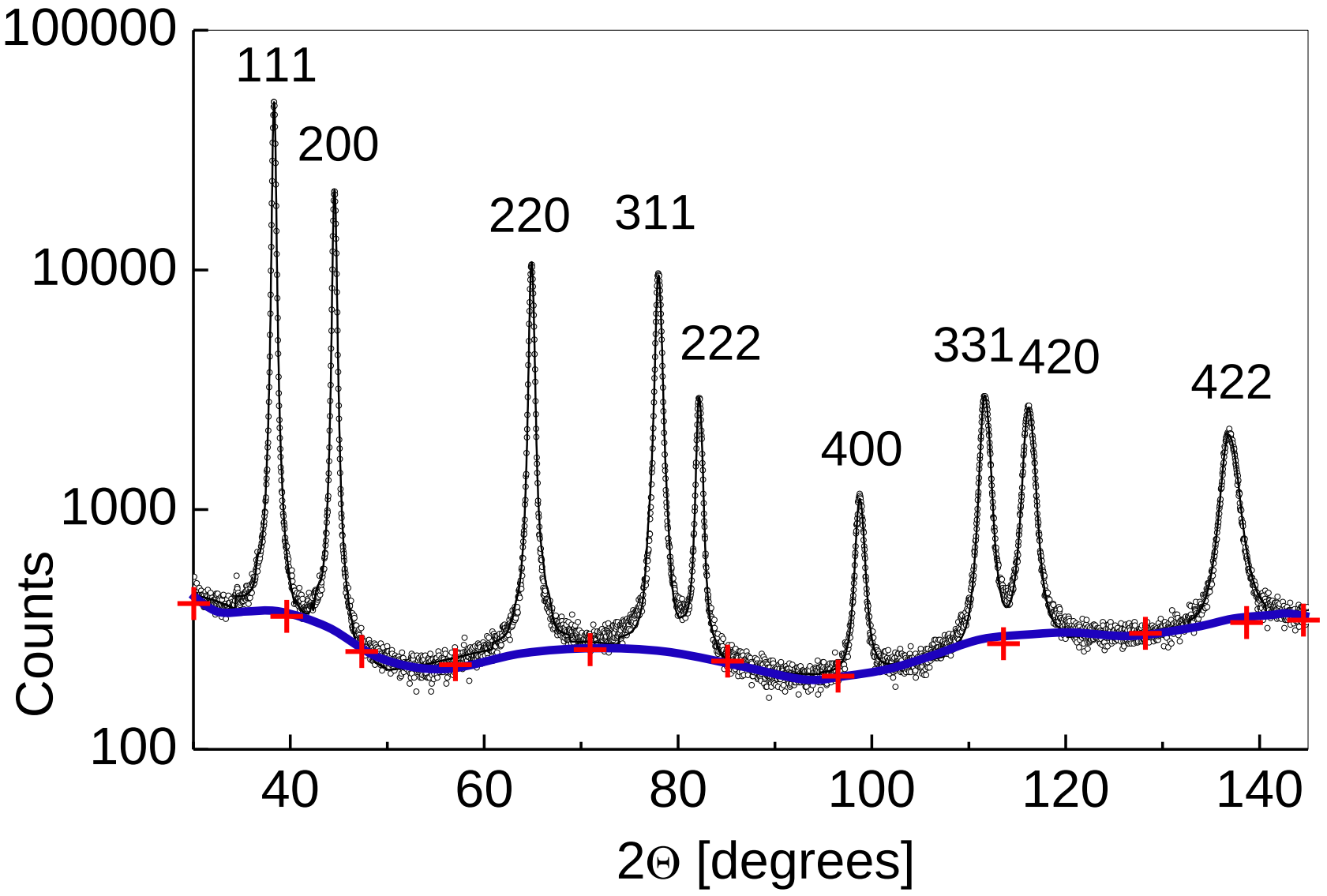
Typical instrumental profiles: laboratory X-rays



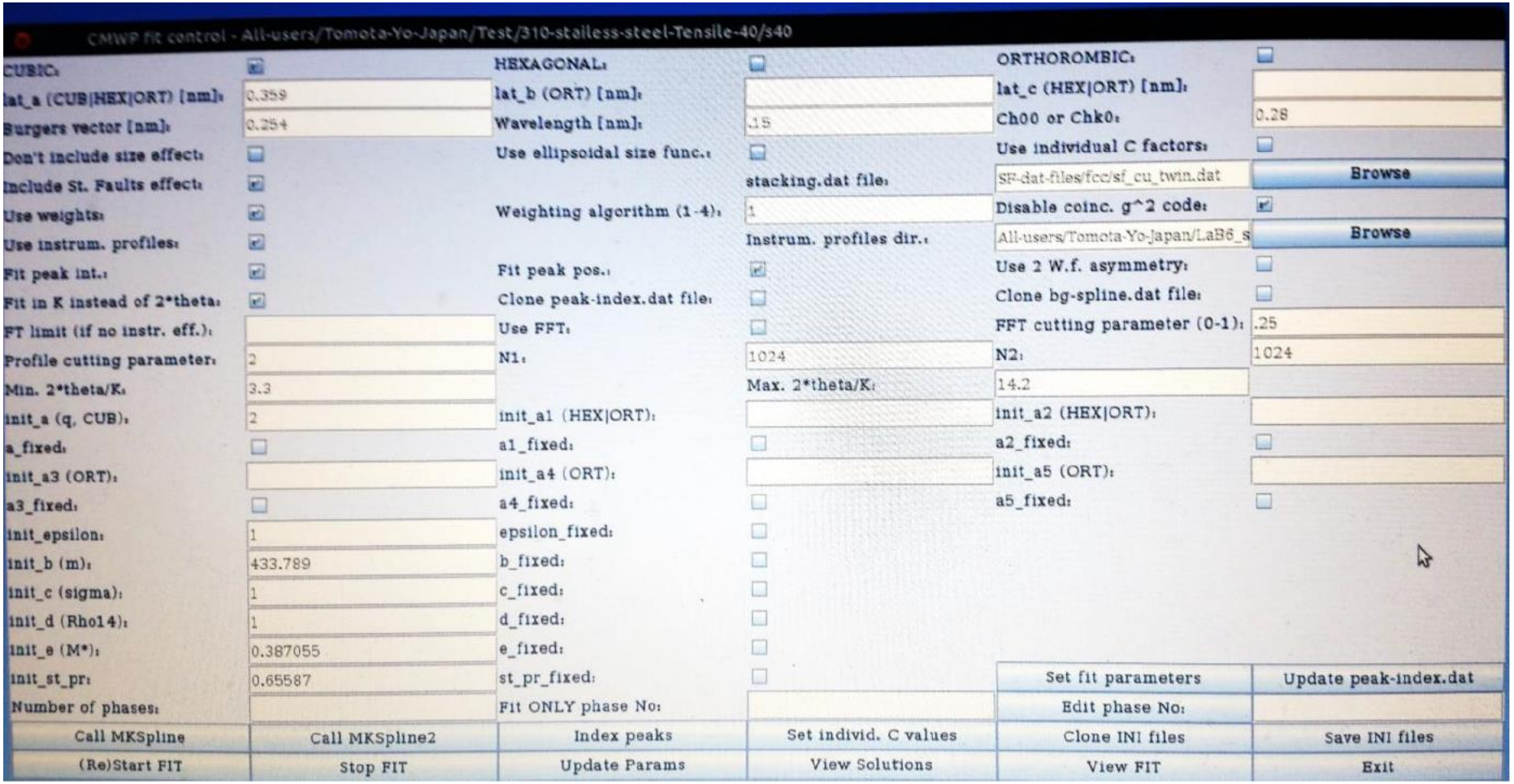




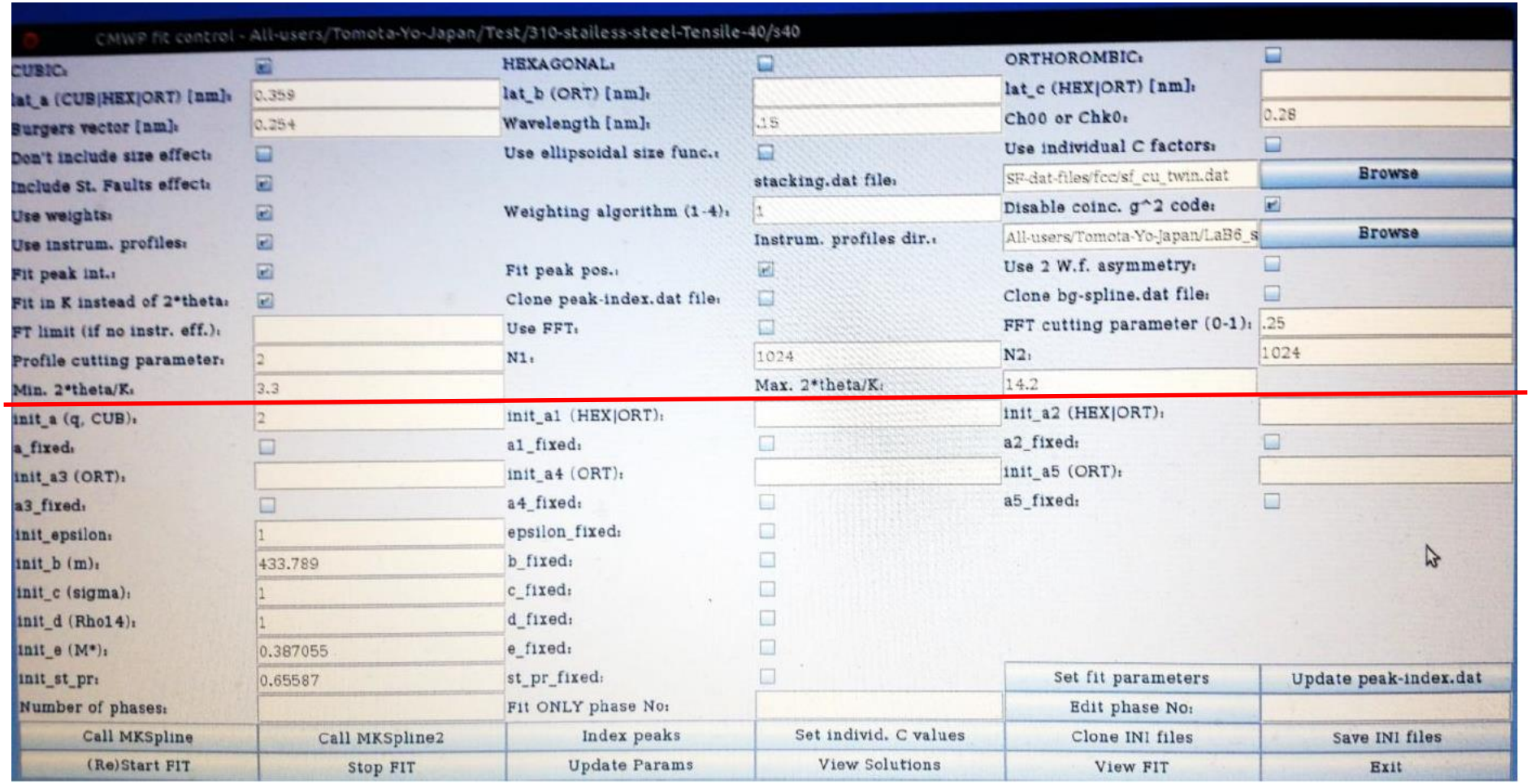
Background spline



CMWP frontend



CMWP frontend



Thank you