

Line Profile Analysis for Engineering Materials Science

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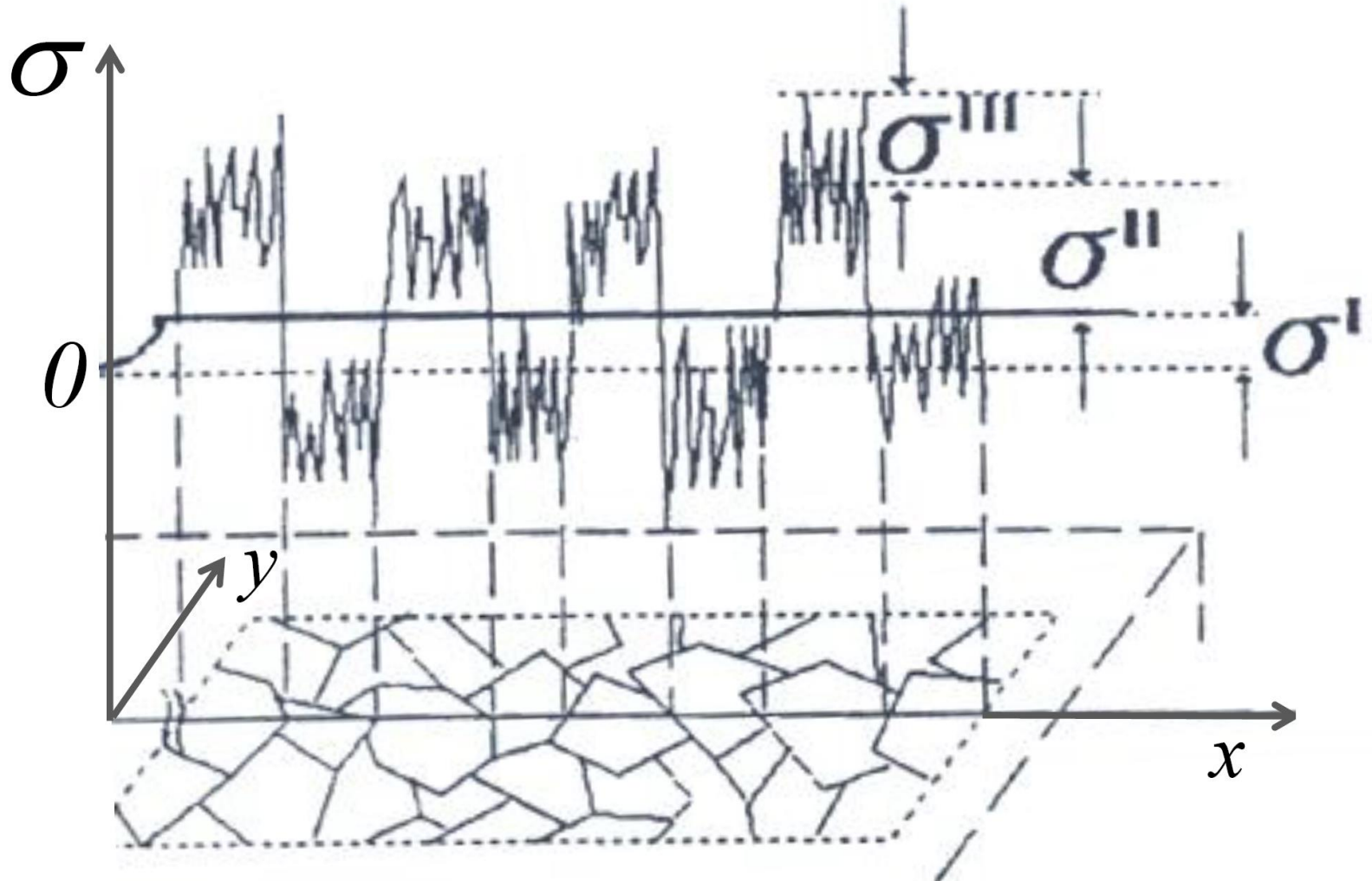
Software Workshop on Engineering Diffraction
for ESS Materials Engineering Beamline, 14-15 June, Copenhagen, Denmark

Convolutional Multiple Whole Profile (CMWP)
software package

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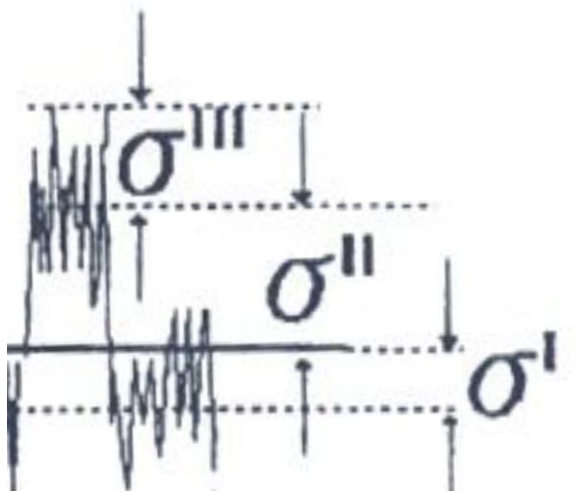
Macherauch, E. (~1965)

schematic classification of **internal stresses**



Macherauch, E. (~1965)

schematic classification of internal stresses



σ_{I} : *macro-stress*

averaged over many grains

σ_{II} : *intergranular-stresses*

averaged over individual grains

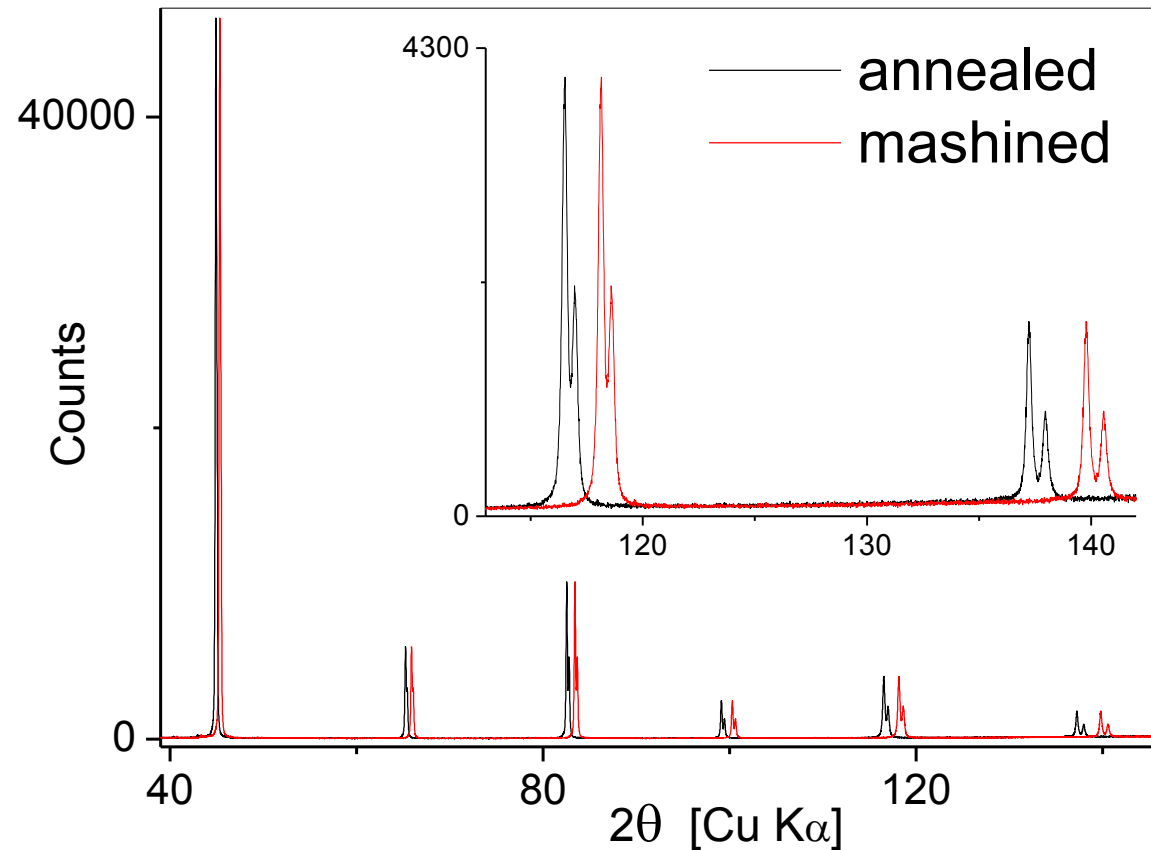
σ_{III} : *micro-strains* (or *stresses*)

produced (mainly) by *dislocations*

Diffraction experiments

σ_I : *macro-stress*

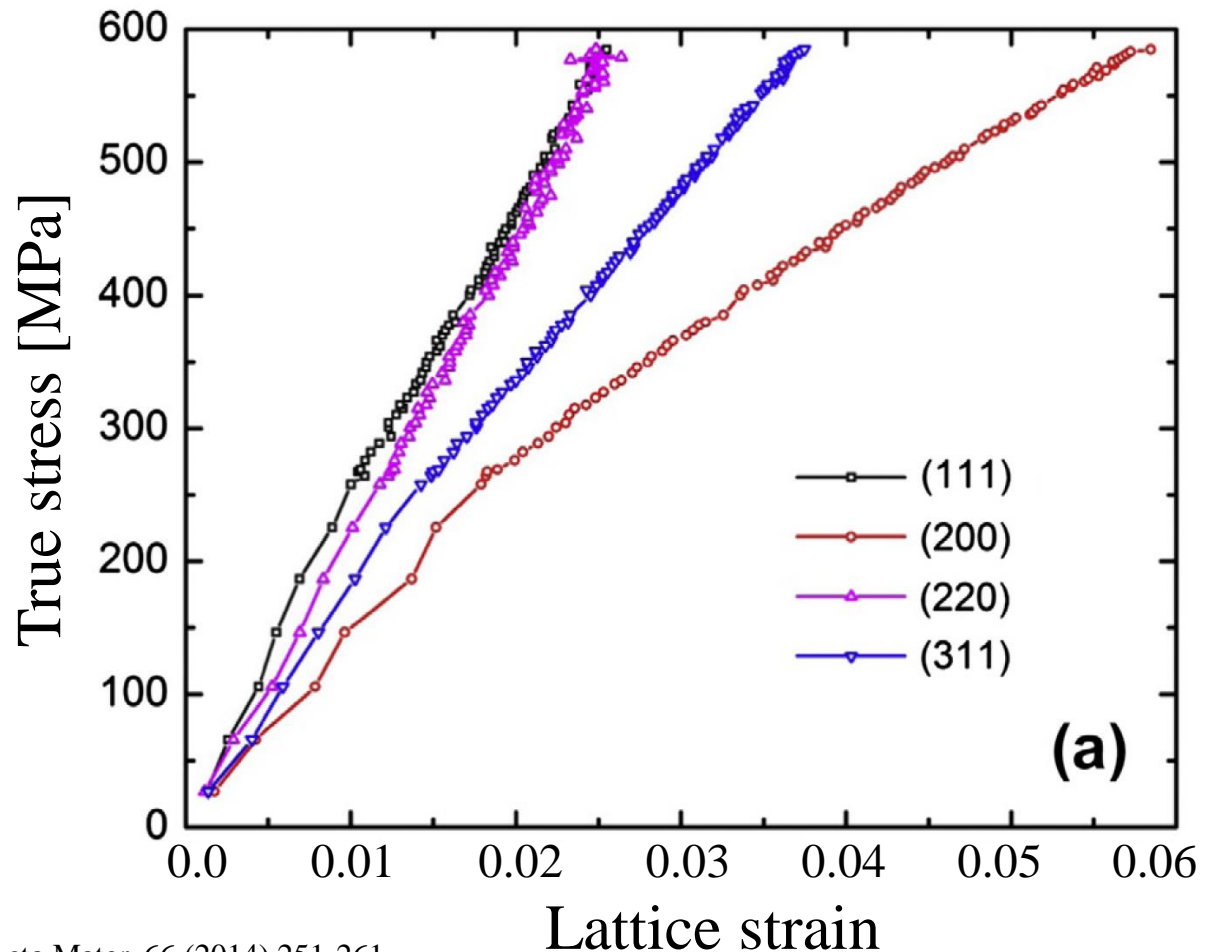
all peaks shift in the *same direction*



Diffraction experiments

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

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

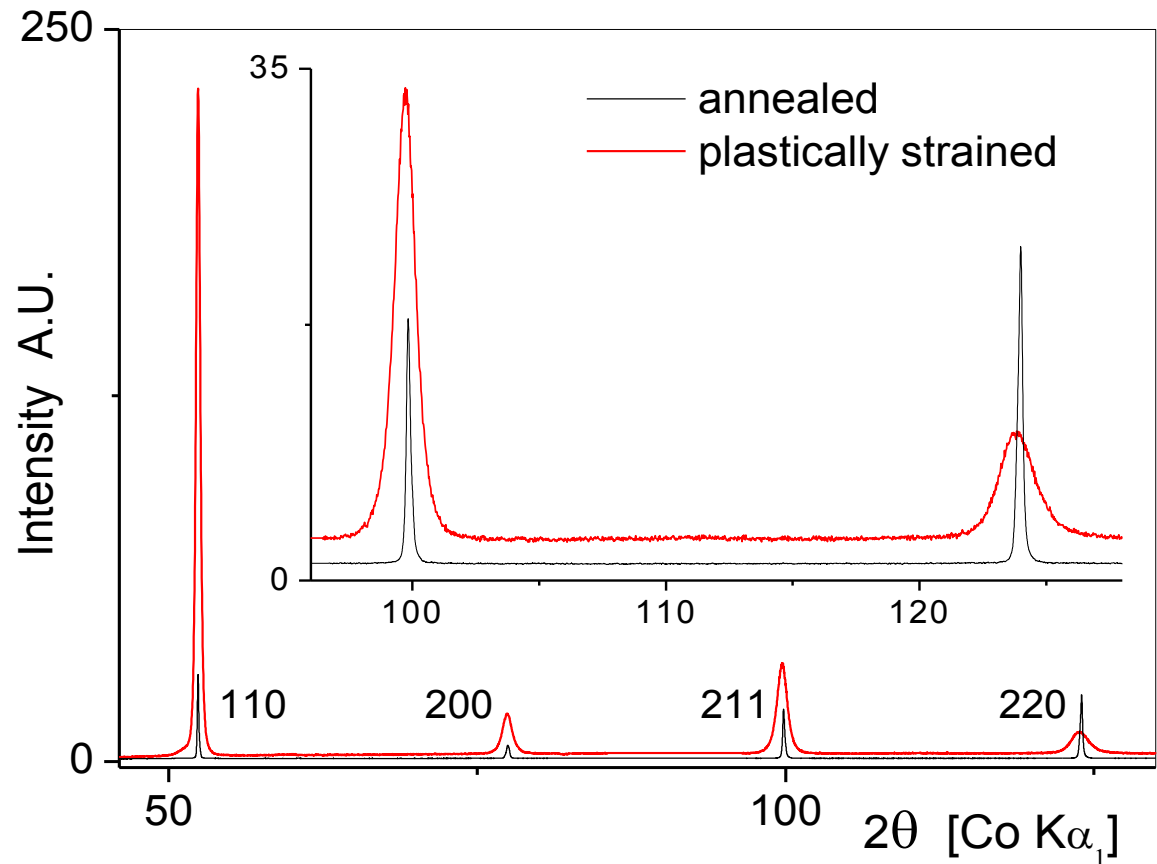


316 stainless steel

Diffraction experiments

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

are produced by *dislocations*: peaks *broaden*

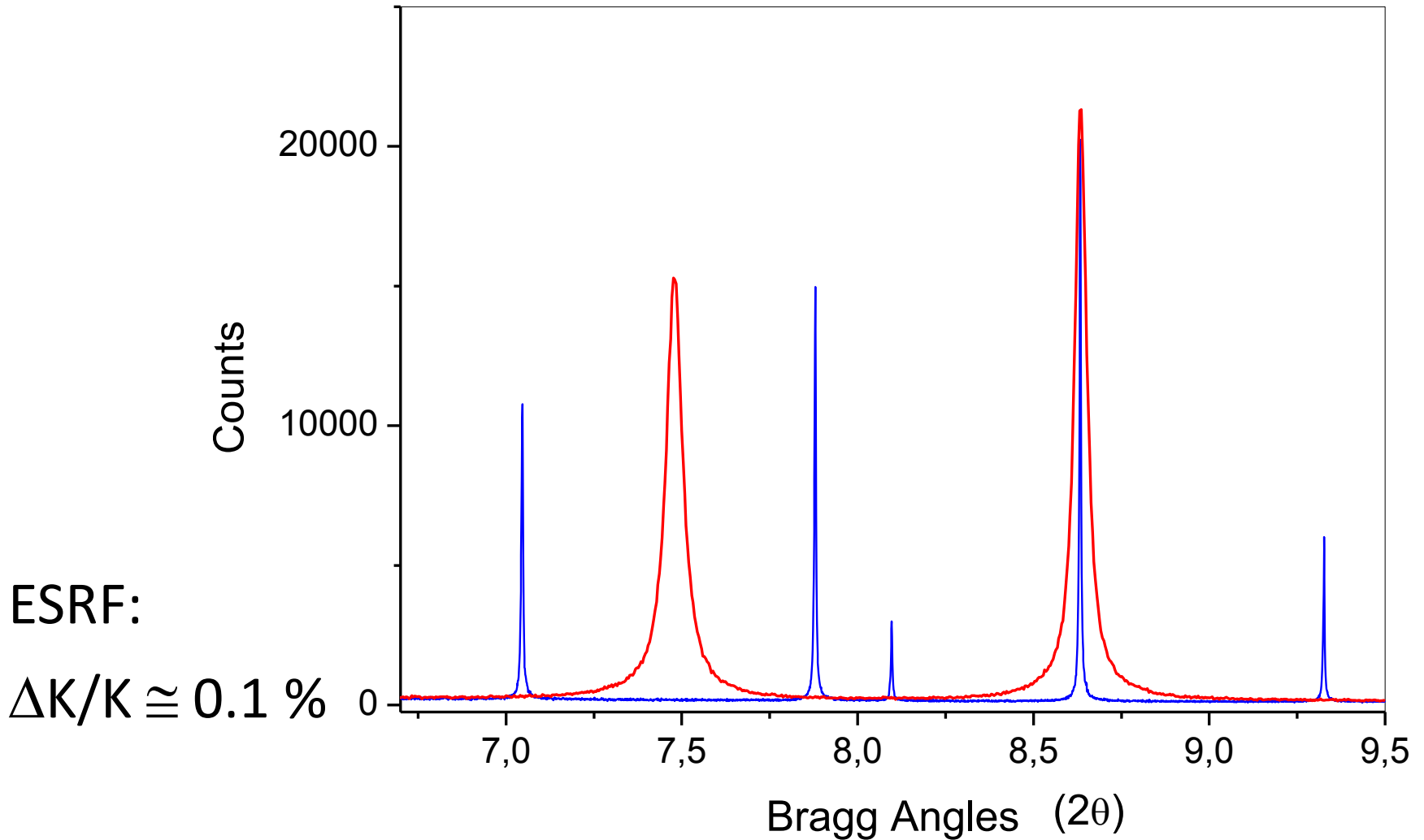


ESRF:

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

Instrumental effects can be substantial, but can be measured

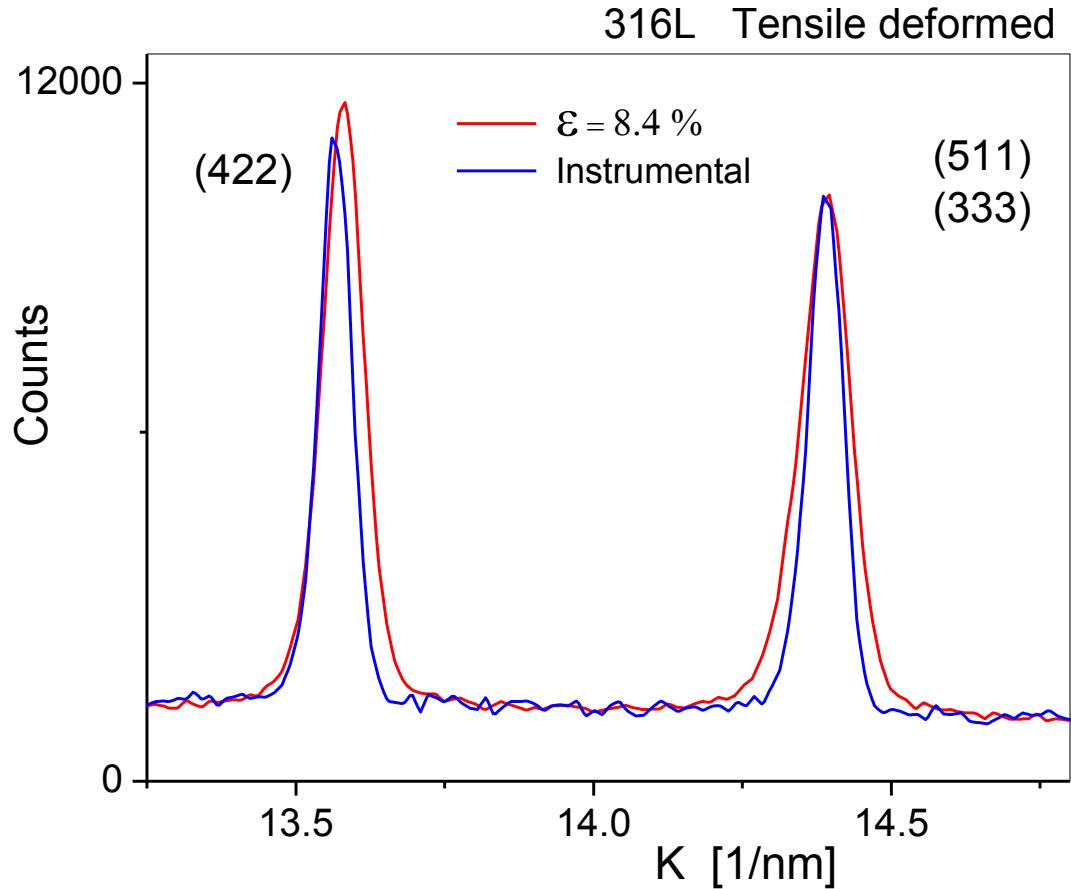
X-ray diffraction:



Instrumental effects can be substantial, but can be measured

neutron diffraction:

SNS-VULCAN

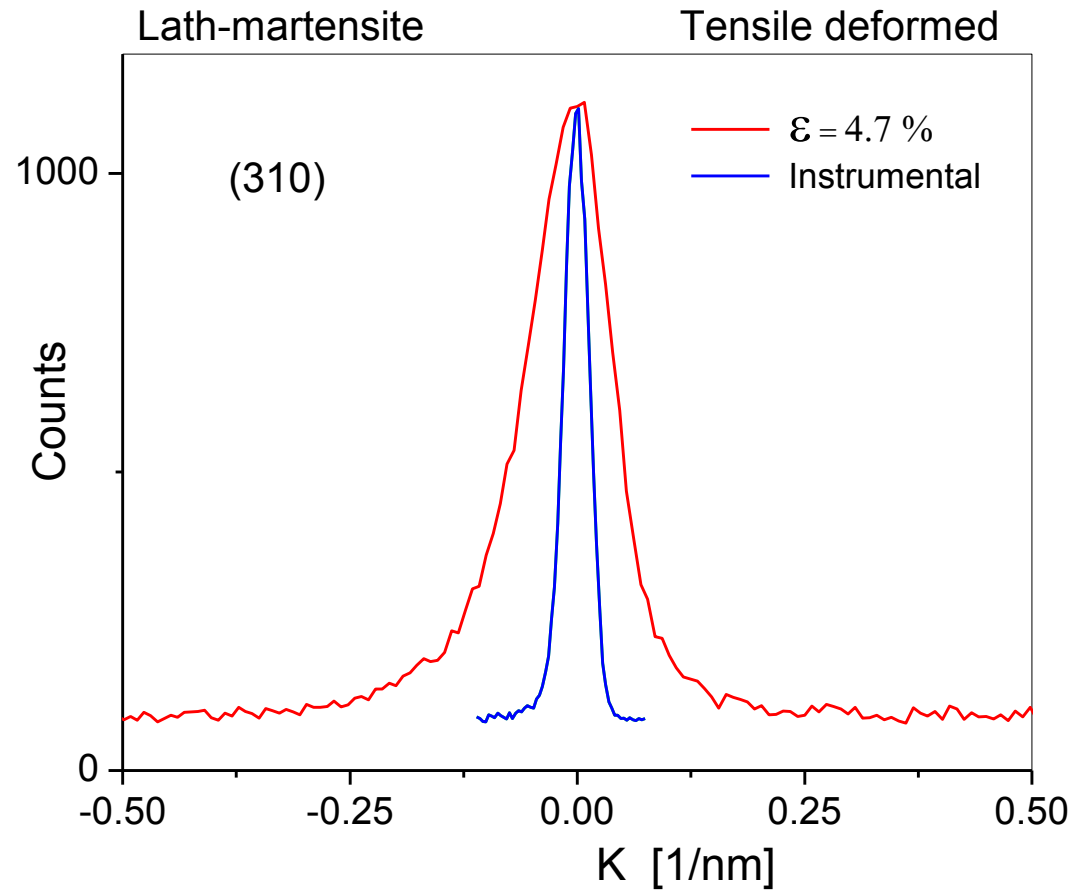


$$\Delta K/K \cong 0.5\%$$

Instrumental effects can be substantial, but can be measured

neutron diffraction:

TAKUMI J-PARC



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

peak asymmetry is genuine

Two different approaches to line-profile-analysis:

Top-down: diffraction patterns are
fitted by **analytical** profile-functions
usually **pseudo-Voigt** 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: $\epsilon(r)$

0 dimensional:

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

$$\epsilon(\mathbf{r}) \sim 1/r^2$$

1 dimensional: **dislocations**

non-equilibrium triple-junctions, linear-type defects

$$\epsilon(\mathbf{r}) \sim 1/r$$

2 dimensional: planar defects, e.g.

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

$$\epsilon(\mathbf{r}) \sim \text{constant}$$

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: $\epsilon(r)$

0 dimensional: $\epsilon(\mathbf{r}) \sim 1/r^2$ point-defects, GP-zones
diffuse scattering **below** the Bragg reflections

1 dimensional: $\epsilon(\mathbf{r}) \sim 1/r$ **dislocations**
broadening **around** the Bragg reflection

2 dimensional: $\epsilon(\mathbf{r}) \sim \text{constant}$ 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-Wilkins** [1963-1970]:

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

b : Burgers vector

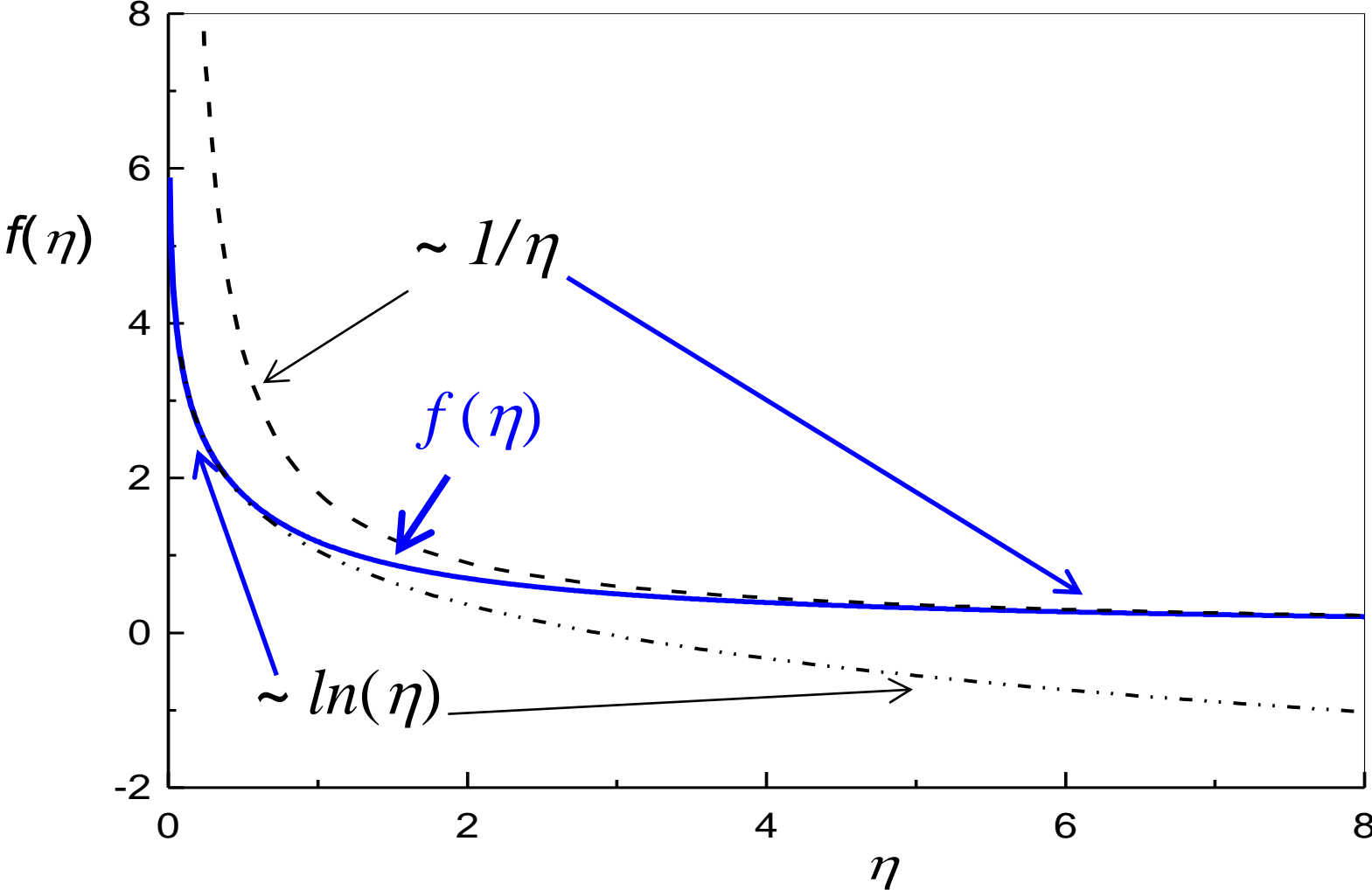
ρ : dislocation density

C : Contrast factor of dislocations

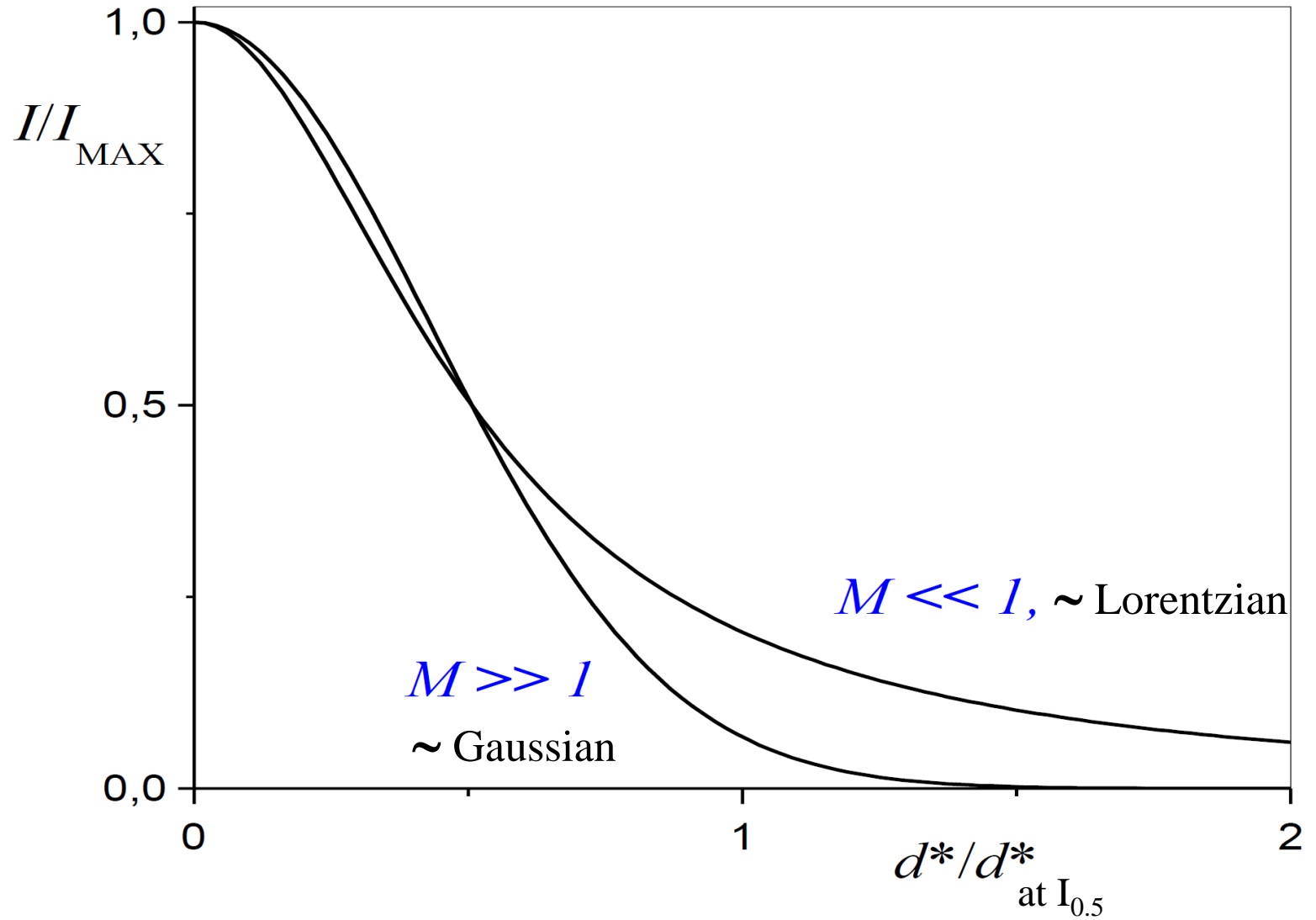
$f(\eta)$: **Wilkins function** [1970]

$$\eta = L/R_e$$

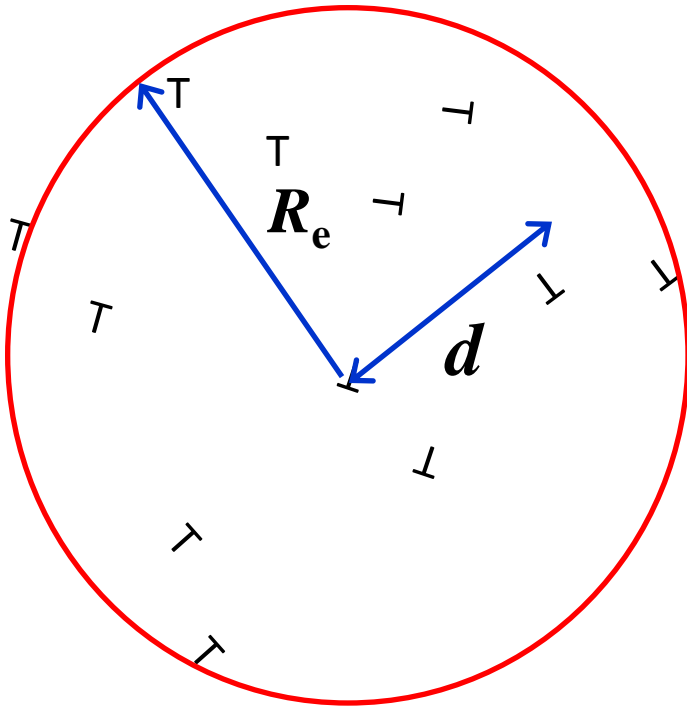
The Krivoglaz-Wilkins function [1963-1970]



shape of strain-profiles



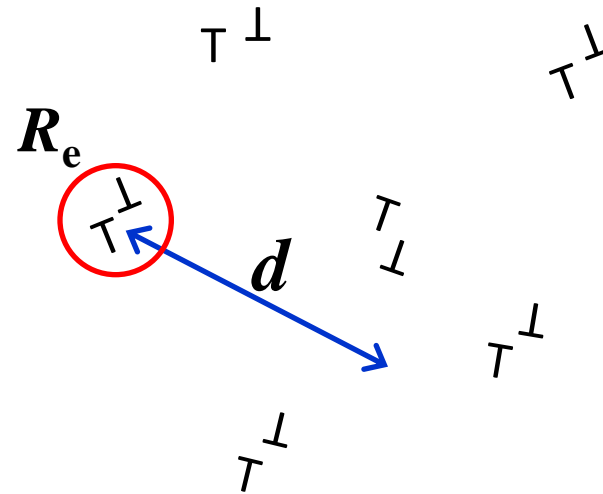
weak dipole character



$$R_e > d$$

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

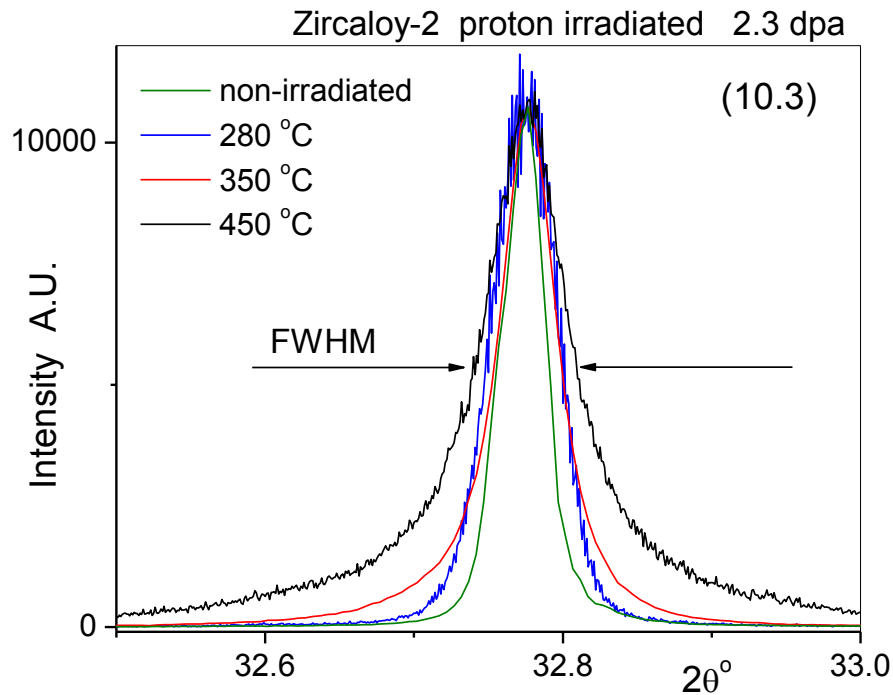
strong dipole character



$$R_e < d$$

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

dislocation loops can have strong dipole character



irradiation at:

280 °C

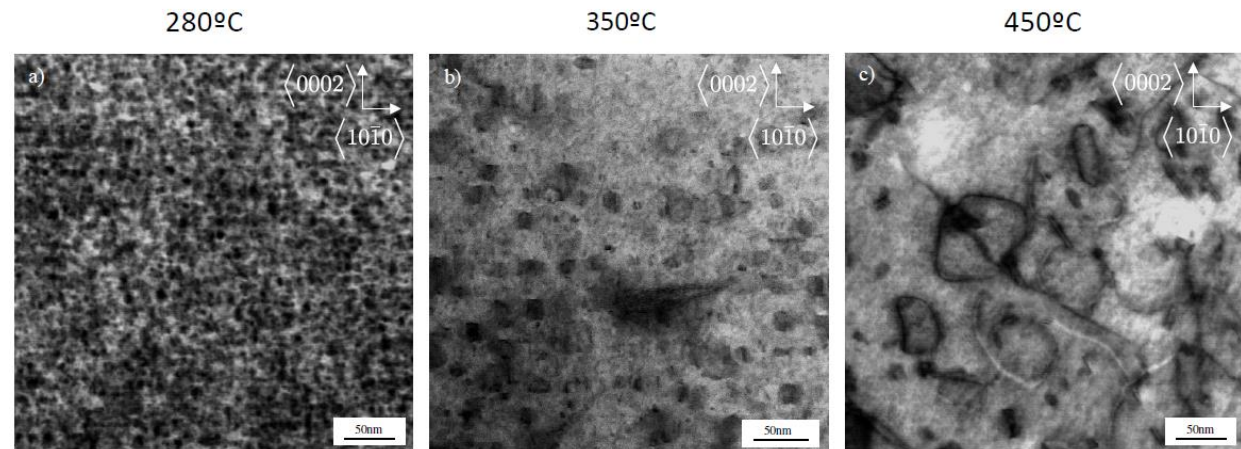
small loops, with large density

350 °C

small loops, with medium density

450 °C

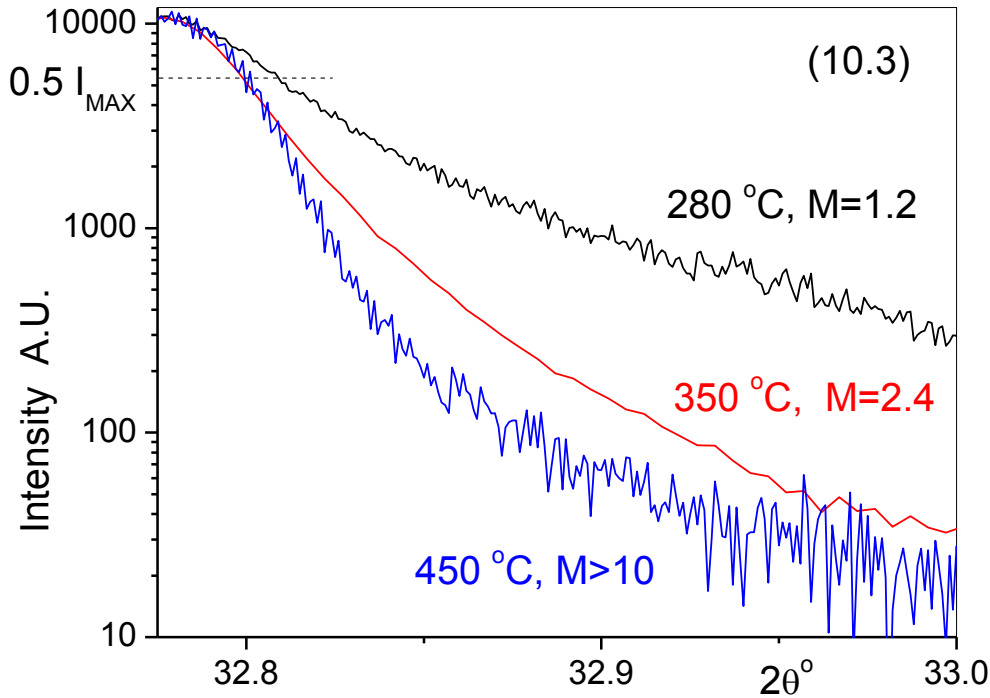
large loops, with smaller density



dislocation loops can have strong dipole character



Zircaloy-2 2.3 dpa



irradiation at:

280 °C

small loops, with large density

350 °C

small loops, with medium density

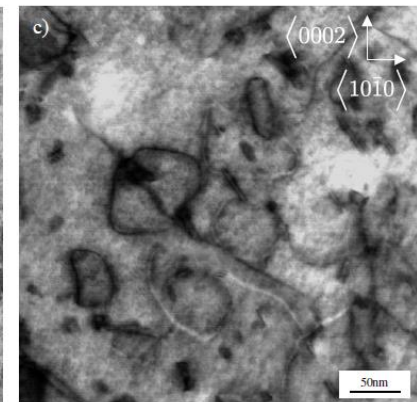
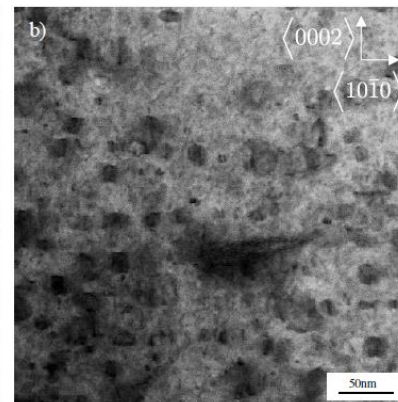
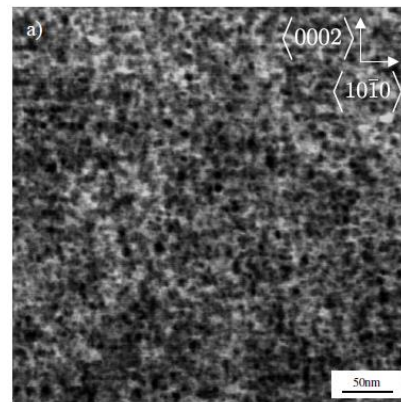
450 °C

large loops, with smaller density

280°C

350°C

450°C



Strain profile:

Fourier transform of the *strain Fourier coefficients*

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

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

dislocation - density : ρ
 - arrangement parameter: M
contrast factor - strain anisotropy: $q_1(q_2)$

Size profile: I^S

assuming log-normal size distribution:

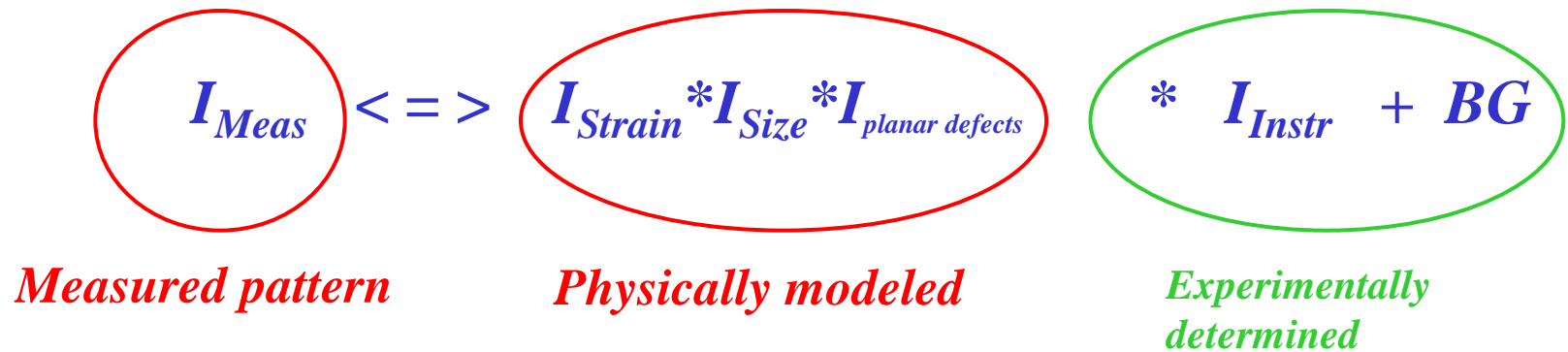
$$f(x) = \frac{1}{(2\pi)^{1/2}} \frac{1}{\sigma x} \exp\left\{-\frac{[\log(x/m)]^2}{2\sigma^2}\right\}$$

m : median

σ : variance

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

Philosophy of evaluation



Combined

Levenberg–Marquardt and Monte-Carlo algorithms

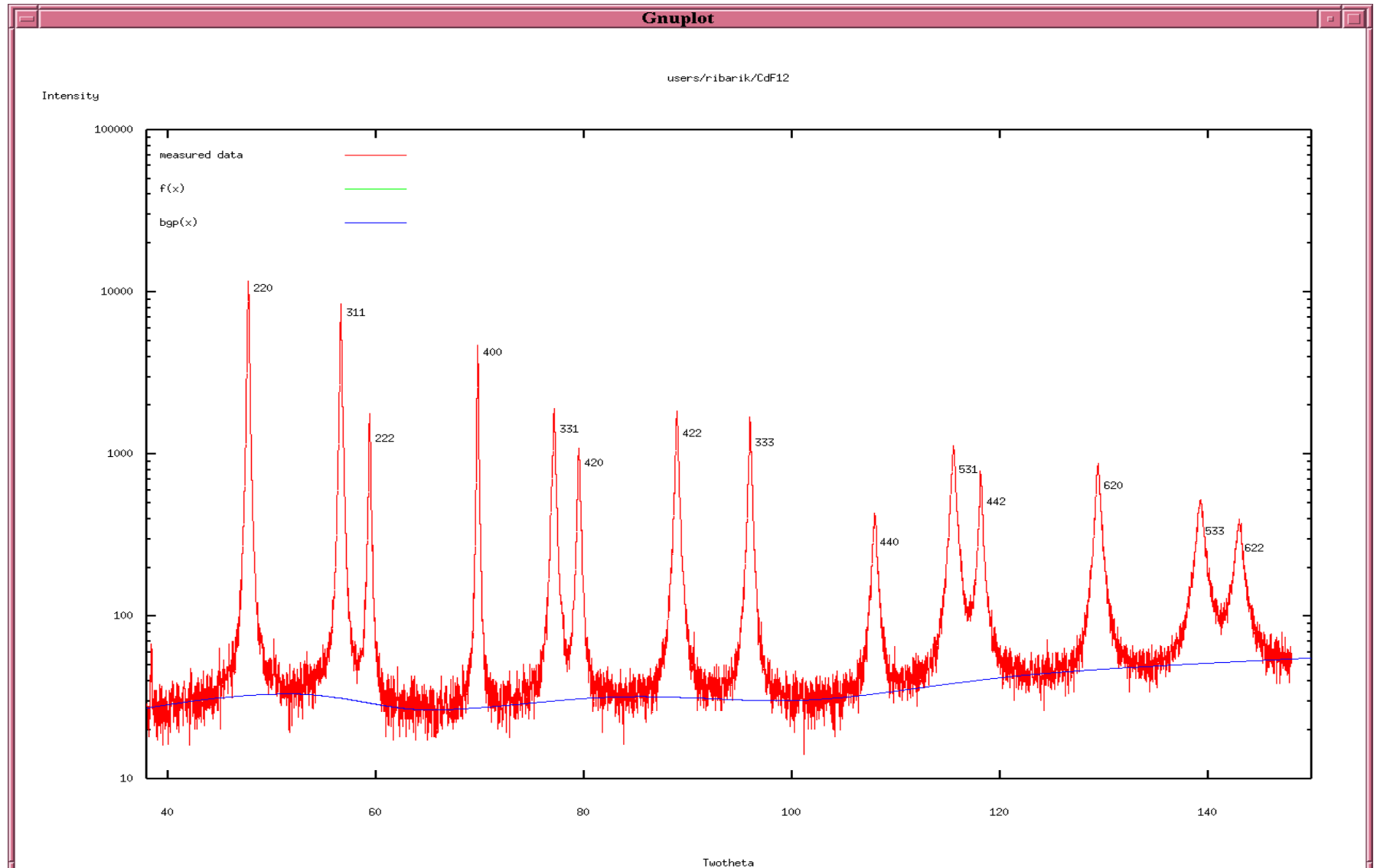
applied in a cyclically alternating sequence

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

CdF₂ ball milled for 12 min

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

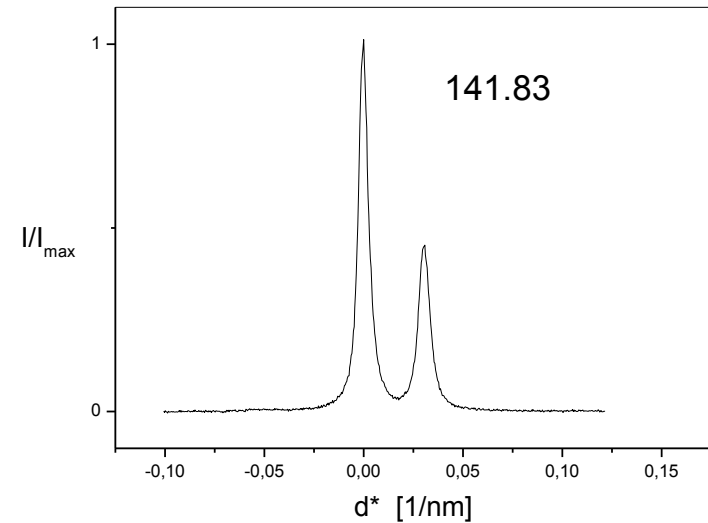
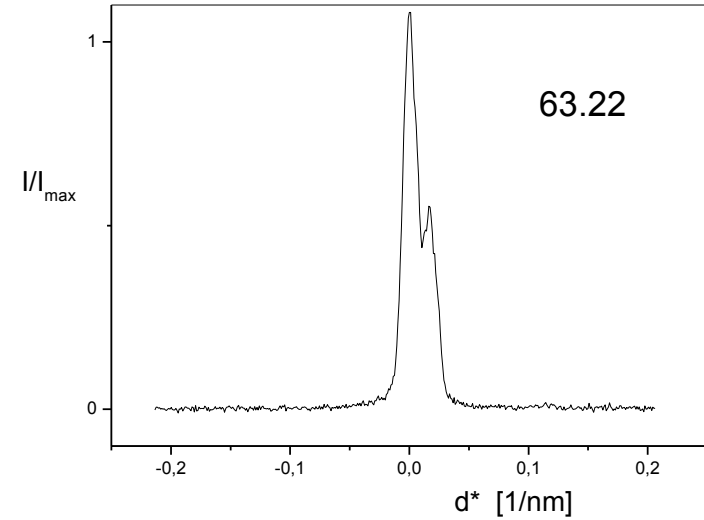
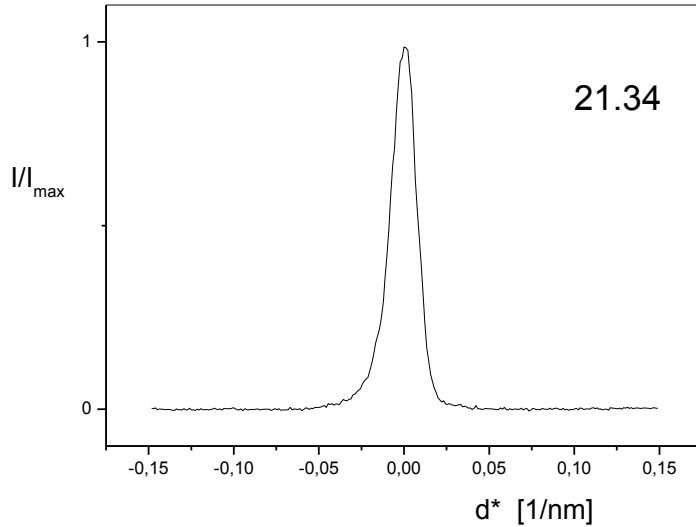


Powder pattern file

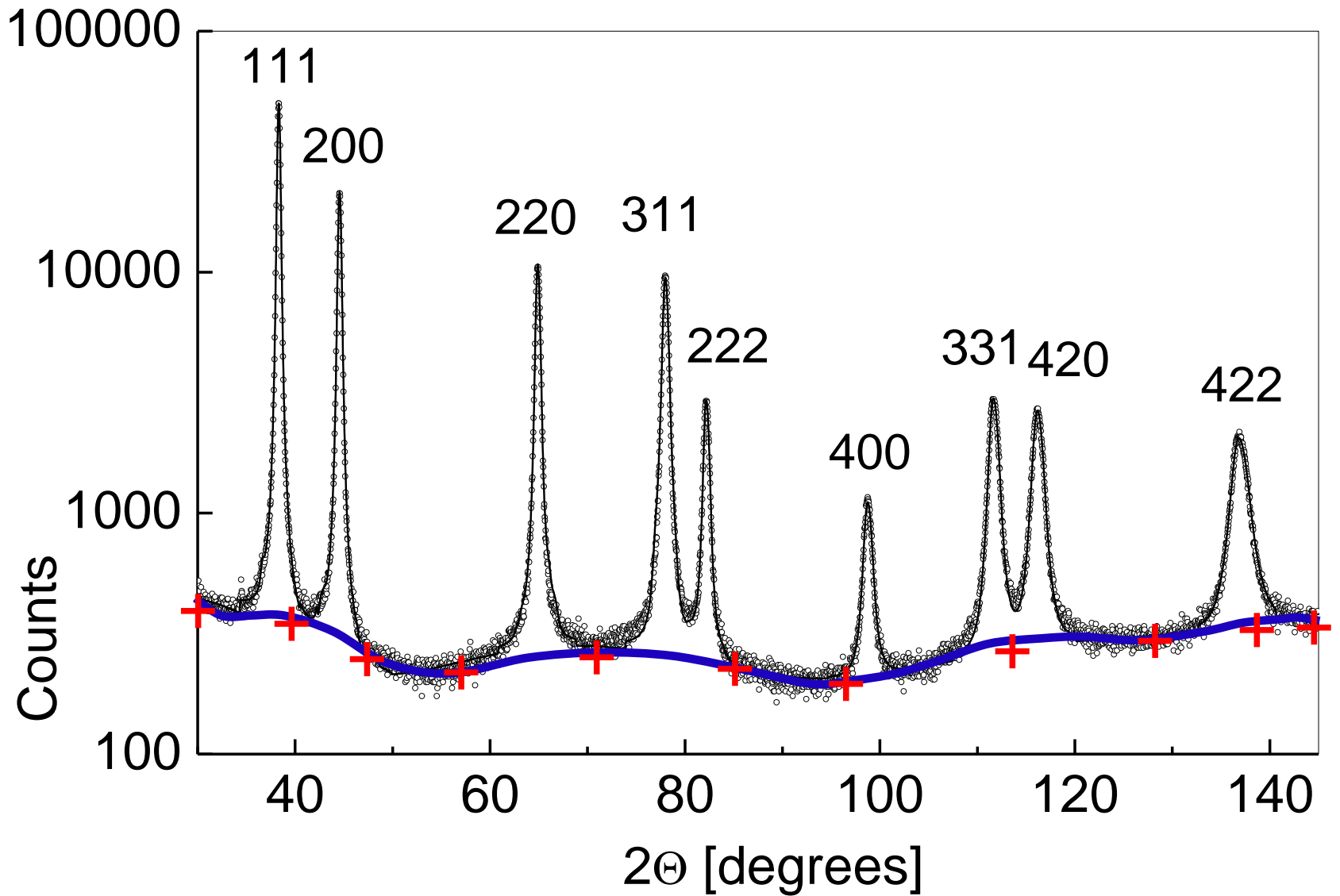
2-column file, contains: 2Θ , I or $d^*(1/\text{nm})$, I

2θ	I	or	$d^*(1/\text{nm})$	I
:			.	
100.7800	335		1.005	120
100.7950	331		1.010	122
100.8100	335		1.025	118
100.8250	331		1.030	130
100.8400	342		1.035	126
100.8550	335		.	
:			.	
:			.	

Typical instrumental profiles: laboratory X-rays



Background spline



CMWP frontend

CMWP fit control - All-users/Tomota-Yo-Japan/Test/310-stainless-steel-Tensile-40/s40

CUBIC:		HEXAGONAL:		ORTHOROMBIC:	
lat_a (CUB HEX ORT) [nm]:	0.359	lat_b (ORT) [nm]:		lat_c (HEX ORT) [nm]:	
Burgers vector [nm]:	0.254	Wavelength [nm]:	1.5	Ch00 or Chk0:	0.28
Don't include size effects:	<input type="checkbox"/>	Use ellipsoidal size func.:	<input type="checkbox"/>	Use individual C factors:	<input type="checkbox"/>
Include St. Faults effect:	<input checked="" type="checkbox"/>	stacking.dat file:		SP-dat-files/fcc/sf_cu_twin.dat	Browse
Use weights:	<input checked="" type="checkbox"/>	Weighting algorithm (1-4):	1	Disable coinc. g ² code:	<input checked="" type="checkbox"/>
Use Instrum. profiles:	<input checked="" type="checkbox"/>	Fit peak pos.:	<input checked="" type="checkbox"/>	Instrum. profiles dir.:	All-users/Tomota-Yo-Japan/LaB6_s Browse
Fit peak int.:	<input checked="" type="checkbox"/>	Clone peak-index.dat file:	<input type="checkbox"/>	Use 2 W.f. asymmetry:	<input type="checkbox"/>
Fit in K instead of 2*theta:	<input checked="" type="checkbox"/>	Use FFT:	<input type="checkbox"/>	Clone bg-spline.dat file:	<input type="checkbox"/>
FT limit (if no instr. eff.):		N1:	1024	FFT cutting parameter (0-1):	.25
Profile cutting parameter:	2	Max. 2*theta/K:		N2:	1024
Min. 2*theta/K:	3.3	init_a1 (HEX ORT):		init_a2 (HEX ORT):	
init_a (q, CUB):	2	a1_fixed:	<input type="checkbox"/>	a2_fixed:	<input type="checkbox"/>
a_fixed:	<input type="checkbox"/>	init_a4 (ORT):		init_a5 (ORT):	
init_a3 (ORT):		a4_fixed:	<input type="checkbox"/>	a5_fixed:	<input type="checkbox"/>
a3_fixed:	<input type="checkbox"/>	epsilon_fixed:	<input type="checkbox"/>		
init_epsilon:	1	b_fixed:	<input type="checkbox"/>		
init_b (m):	433.789	c_fixed:	<input type="checkbox"/>		
init_c (sigma):	1	d_fixed:	<input type="checkbox"/>		
init_d (Rho14):	1	e_fixed:	<input type="checkbox"/>		
init_e (M*):	0.387055	st_pr_fixed:	<input type="checkbox"/>		
init_st_pr:	0.65587	Fit ONLY phase No:			
Number of phases:					
Call MKSpline	Call MKSpline2	Index peaks	Set individ. C values	Clone INI files	Save INI files
(Re)Start FIT	Stop FIT	Update Params	View Solutions	View FIT	Exit

CMWP frontend

CMWP fit control - All-users/Tomota-Yo-Japan/Test/310-stainless-steel-Tensile-40/s40

CUBIC:		HEXAGONAL:		ORTHOROMBIC:	
lat_a (CUB HEX ORT) [nm]:	0.359	lat_b (ORT) [nm]:		lat_c (HEX ORT) [nm]:	
Burgers vector [nm]:	0.254	Wavelength [nm]:	1.5	Ch00 or Chk0:	0.28
Don't include size effects:	<input type="checkbox"/>	Use ellipsoidal size func.:	<input type="checkbox"/>	Use individual C factors:	<input type="checkbox"/>
Include St. Faults effect:	<input checked="" type="checkbox"/>	stacking.dat file:		SP-dat-files/fcc/sf_cu_twin.dat	Browse
Use weights:	<input checked="" type="checkbox"/>	Weighting algorithm (1-4):	1	Disable coinc. g ² code:	<input checked="" type="checkbox"/>
Use Instrum. profiles:	<input checked="" type="checkbox"/>	Fit peak pos.:	<input checked="" type="checkbox"/>	Instrum. profiles dir.:	All-users/Tomota-Yo-Japan/LaB6_s Browse
Fit peak int.:	<input checked="" type="checkbox"/>	Clone peak-index.dat file:	<input type="checkbox"/>	Use 2 W.f. asymmetry:	<input type="checkbox"/>
Fit in K instead of 2*theta:	<input checked="" type="checkbox"/>	Use FFT:	<input type="checkbox"/>	Clone bg-spline.dat file:	<input type="checkbox"/>
FT limit (if no instr. eff.):		N1:	1024	FFT cutting parameter (0-1):	.25
Profile cutting parameter:	2	Max. 2*theta/K:		N2:	1024
Min. 2*theta/K:	3.3				
init_a (q, CUB):	2	init_a1 (HEX ORT):		init_a2 (HEX ORT):	
a_fixed:	<input type="checkbox"/>	a1_fixed:	<input type="checkbox"/>	a2_fixed:	<input type="checkbox"/>
init_a3 (ORT):		init_a4 (ORT):		init_a5 (ORT):	
a3_fixed:	<input type="checkbox"/>	a4_fixed:	<input type="checkbox"/>	a5_fixed:	<input type="checkbox"/>
init_epsilon:	1	epsilon_fixed:	<input type="checkbox"/>		
init_b (m):	433.789	b_fixed:	<input type="checkbox"/>		
init_c (sigma):	1	c_fixed:	<input type="checkbox"/>		
init_d (Rho14):	1	d_fixed:	<input type="checkbox"/>		
init_e (M*):	0.387055	e_fixed:	<input type="checkbox"/>		
init_st_pr:	0.65587	st_pr_fixed:	<input type="checkbox"/>		
Number of phases:		Fit ONLY phase No:			
Call MKSpline	Call MKSpline2	Index peaks	Set individ. C values	Set fit parameters	Update peak-index.dat
(Re)Start FIT	Stop FIT	Update Params	View Solutions	Edit phase No:	Save INI files
				Clone INI files	Exit
				View FIT	

Thank you