Convolutional Multiple Whole Profile Fitting

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Introduction:

- extracting microstructure using X-ray line profile analysis
- modeling size and strain broadening
- the MWP method
- the CMWP method
- CMWP application to ball milled Al-Mg alloys
- the CMWP program
These methods are

- in fact: Whole Profile fitting, or Whole Powder Pattern fitting methods

- microstructural methods: the unit cell is NOT refined

The aim is microstructure in terms of:

- size

- strain
Theory

The Fourier coefficients of the line profiles (Warren & Averbach, 1952):

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

this means that the observed profile is the convolution of size and strain profiles.

If more physical effects and instrumental effects are simultaneously present:

$$A(L) = A_{instr.}(L)A_{size}(L)A_{disl.}(L)A_{pl.\, faults}(L) \cdots,$$

$$I(2\Theta) = I_{instr.}(2\Theta) \ast I_{size}(2\Theta) \ast I_{disl.}(2\Theta) \ast I_{pl.\, faults}(2\Theta) \ast \cdots$$
The size effect

If we suppose:

- spherical crystallites
- lognormal $f(x)$ size distribution density function:

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

($m$ and $\sigma$ are the two parameters of the distribution).
The size effect

The size intensity profile (Gubicza et al, 2000):

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

where \( \text{erfc} \) is the complementary error function:

\[ \text{erfc}(x) = \frac{2}{\sqrt{\pi}} \int_x^\infty e^{-t^2} \, dt. \]
The size effect

If we suppose (Ribárik et al, 2001):

- ellipsoidal crystallite shape
- lognormal size distribution density function

$I^S(s)$ has the same form than in the spherical case but $m$ depends on the indices of reflection:

$$m_{hkl} = \frac{m_a}{\sqrt{1 + \left(\frac{1}{\varepsilon^2} - 1\right) \cos^2 \alpha}},$$

($m_a$: the $m$ parameter of the size distribution in the direction $a$, $\varepsilon$: ellipticity, $\alpha$: the angle between the axis of revolution and the diffraction vector).
The size effect

If the relative orientations of the crystallographic directions to the axis of revolution are known, $\cos \alpha$ can be expressed by the indices of reflection.

For cubic systems:

$$\cos \alpha = \frac{l}{\sqrt{h^2 + k^2 + l^2}}$$

For hexagonal systems:

$$\cos \alpha = \frac{l}{\sqrt{\frac{4}{3} \frac{c^2}{a^2} (h^2 + hk + k^2) + l^2}}$$
The Size Fourier Transform:

It can be expressed in an almost closed form which is suitable for fast numeric evaluation (Ribárik et al, 2001):

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

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

\[
\frac{|L|^3}{6} \text{erfc} \left[ \frac{\log \left( \frac{|L|}{m} \right)}{\sqrt{2}\sigma} \right].
\]
The strain effect

The distortion Fourier coefficients (Warren & Averbach, 1952):

\[ A^D(L) = \exp \left( -2\pi^2 g^2 L^2 \langle \varepsilon^2_L \rangle \right) , \]

where

- \( g \) is the absolute value of the diffraction vector,
- \( \langle \varepsilon^2_L \rangle \) is the mean square strain.

The most important models for \( \langle \varepsilon^2_L \rangle \):

- Warren & Averbach (1952)
- Krivoglaz & Ryaboshapka (1963)
- Wilkens (1970)
The Wilkens dislocation theory

Wilkens introduced the effective outer cut off radius of dislocations, $R_e^*$, instead of the crystal diameter. Assuming infinitely long parallel screw dislocations with restrictedly random distribution (Wilkens, 1970):

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

where $f^*$ is the Wilkens strain function (Wilkens, 1970). Kamminga and Delhez (2000) have shown that this strain function is also valid for edge- and curved dislocations.

The distortion Fourier–transform in the Wilkens model:

$$A^D(L) = \exp \left[ -\frac{\pi b^2}{2} (g^2 C') \rho L^2 f^* \left( \frac{L}{R_e^*} \right) \right].$$
The Wilkens strain function

\[ f^*(\eta) = -\log \eta + \left( \frac{7}{4} - \log 2 \right) + \frac{512}{90\pi} \frac{1}{\eta} + \]

\[ \frac{2}{\pi} \left[ 1 - \frac{1}{4\eta^2} \right] \int_0^\eta \frac{\arcsin V}{V} dV - \]

\[ \frac{1}{\pi} \left[ \frac{769}{180} \frac{1}{\eta} + \frac{41}{90} \eta + \frac{2}{90} \eta^3 \right] \sqrt{1 - \eta^2} - \]

\[ \frac{1}{\pi} \left[ \frac{11}{12} \frac{1}{\theta^2} + \frac{7}{2} + \frac{1}{3} \eta^2 \right] \arcsin \eta + \frac{1}{6} \eta^2, \quad \text{if } \eta \leq 1, \]

\[ f^*(\eta) = \frac{512}{90\pi} \frac{1}{\eta} - \left[ \frac{11}{24} + \frac{1}{4} \log 2n \right] \frac{1}{\eta^2}, \quad \text{if } \eta \geq 1, \]

where \( f \left( \frac{L}{R_e^*} \right) = f^*(\eta) \) and \( \eta = \frac{1}{2} \exp \left( -\frac{1}{4} \right) \frac{L}{R_e^*} \).
The Wilkens strain function

The Wilkens function and its approximations: \(- \log \eta + \left( \frac{7}{4} - \log 2 \right)\) and \(\frac{512}{90\pi} \frac{1}{\eta}\).
The dislocation arrangement parameter

Wilkens introduced $M^*$, a dimensionless parameter:

$$M^* = R_e^* \sqrt{\rho}$$

The $M^*$ parameter characterizes the dislocation arrangement:

- if the value of $M^*$ is small, the correlation between the dislocations is strong
- if the value of $M^*$ is large, the dislocations are distributed randomly in the crystallite
\[ R_e^* << \frac{1}{\sqrt{\rho}} \]

\[ M^* << 1 \]

\[ R_e^* >> \frac{1}{\sqrt{\rho}} \]

\[ M^* >> 1 \]
The strain profile for fixed $\rho$ and variable $M^*$ values:
The shape of the strain profile for fixed $\rho$ and variable $M^*$ values:
Strain anisotropy

According to (Ungár & Tichy, 1999), the average contrast factors of dislocations can be expressed in the following form for cubic crystals:

\[ C = C_{h00}(1 - qH^2), \]

where

\[ H^2 = \frac{h^2k^2 + h^2l^2 + k^2l^2}{(h^2 + k^2 + l^2)^2}. \]
For hexagonal crystals:

\[ C = C_{hk0}(1 + a_1 H_1^2 + a_2 H_2^2), \]

where

\[ H_1^2 = \frac{[h^2 + k^2 + (h + k)^2] l^2}{[h^2 + k^2 + (h + k)^2 + \frac{3}{2}(\frac{a}{c})^2 l^2]^2}, \]

\[ H_2^2 = \frac{l^4}{[h^2 + k^2 + (h + k)^2 + \frac{3}{2}(\frac{a}{c})^2 l^2]^2}, \]

and \( \frac{a}{c} \) is the ratio of the two lattice constants.
For orthorombic crystals:

$$
\overline{C}_{hkl} = C_{h00} \left( H_0^2 + a_1 H_1^2 + a_2 H_2^2 + a_3 H_3^2 + a_4 H_4^2 + a_5 H_5^2 \right),
$$

where:

$$
H_0^2 = \frac{h^4}{a^4} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2
$$

$$
H_1^2 = \frac{k^4}{b^4} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2
$$

$$
H_2^2 = \frac{l^4}{c^4} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2
$$
\[ H_3^2 = \frac{h^2k^2}{a^2b^2} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2 \]

\[ H_4^2 = \frac{l^2h^2}{c^2a^2} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2 \]

\[ H_5^2 = \frac{k^2l^2}{b^2c^2} \left( \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)^2 \]

and \( a, b, c \) are the lattice constants.

The constants \( C_{h00} \) and \( C_{hk0} \) are calculated from the elastic constants of the crystal (Ungár et al, 1999).
Planar and twin faults:

The peak profile is the sum of a delta function and shifted and broadened Lorentzian profile functions.

- The FWHM and shift value of the Lorentzians depend on the density of faults.


The parameters were systematically calculated for each fundamental types of planar faults by Dr. Levente Balogh (see: L. Balogh, PhD thesis, Eötvös University, 2009).
Microstructural parameters

(C)MWP-fit provides:

- size: \(m, \sigma, \varepsilon\)
- dislocations: \(\rho, M, q\)
- planar and twin faults: \(\alpha, \beta\)
Multiple Whole Profile (MWP) fitting

The method (Ribárik et al, 2001) is:

- a Whole Profile fitting method using ab-initio theoretical profile functions
- a Fourier method, which works on multiple profiles simultaneously

The data must be prepared before applying the method:

- the profiles should be separated
- the instrumental broadening is corrected for by deconvolution using the Stokes method
- the separated and instrumental-free profiles are Fourier-transformed
Example for the profile separation in the case of the strong overlapping peaks of a carbon black sample.
MWP: instrumental correction

Example for the instrumental deconvolution in the case of an Al-6Mg sample. The $A(L)$ Fourier transforms of the measured profile, the instrumental profile and the corrected profile are plotted as a function of $L$. 
Example for the instrumental deconvolution in the case of an Al-6Mg sample. The raw measured and the corrected intensity profile are plotted as a function of $K$. 

MWP: instrumental correction
(i) Multiple Whole Profile fitting of the Fourier–transforms.

- the measured intensity profiles are Fourier–transformed and normalized,
- they are fitted simultaneously by the normalized theoretical Fourier–transform:

\[
A(L) = \frac{A^S(L)}{A^S(0)} \exp \left[ -\frac{\pi b^2}{2} (g^2 C) \rho L^2 f \left( \frac{L}{R^*_e} \right) \right],
\]
MWP-fit

(ii) **Multiple Whole Profile fitting of the intensity profiles.** In this procedure first the measured intensity profiles are normalized. Then all of them are fitted simultaneously by the normalized theoretical intensity function:

\[
I(s) = \frac{F_c(s)}{F_c(0)},
\]

where \( F_c \) is the Cosine Fourier–transform of \((??)\):

\[
F_c(s) = 2 \int_{0}^{\infty} A(L) \cos(2\pi Ls) \, dL.
\]

In both cases [(i) and (ii)] all profiles are fitted simultaneously using a nonlinear least-squares algorithm.
MWP application to Cu sample

The measured (solid lines) and theoretical fitted (dashed lines) Fourier transforms for copper sample deformed by ECAP (equal-channel angular pressing) as a function of the Fourier Frequency, $L$. The difference plot is also given.

Results of the MWP fit:

$m = 62 \text{nm}$

$\sigma = 0.53$

$\rho = 1.7 \cdot 10^{15} \text{ m}^{-2}$

$M = \Re \sqrt{\rho} = 1.7$

$q = 1.84$

$\rho = 1.7 \cdot 10^{15} \text{ m}^{-2}$
MWP application to PbS sample

The measured (solid lines) and theoretical fitted (dashed lines) Fourier–transforms for PbS sample as a function of the Fourier variable (Frequency), $L$, plotted by the program evaluate. The difference plot is also given in the bottom of the figure.
The measured (solid lines) and theoretical fitted (dashed lines) intensity profiles for PbS sample plotted by the program evaluate. The difference plot is also given in the bottom of the figure. The indices of reflections are also indicated.
Comparison with TEM

The size distribution density function corresponding to the parameters of the MWP fit and the size distribution obtained by TEM for an ECA pressed copper sample.
Comparison with TEM

The TEM micrograph (a) and the size distribution functions (b) measured by TEM and X-ray line profile analysis for nanoncrystalline $Si_3N_4$ particles.
Comparison with TEM

(a) High resolution TEM image of nanocrystalline titanium sample (b) Fourier-filtered image from the white frame in (a), showing the dislocation arrangement in the grain boundary.
Convolutional MWP (CMWP) fitting

When the MWP fitting procedure is used:

- the deconvolution of the physical and the instrumental profiles introduces noise,
- the selection of the analytical function used in the peak separation influences the shape of the individual profile.

When the CMWP method (Ribárik et al, 2004) is used:

- the whole measured powder diffraction pattern is fitted by the sum of a background polynomial and profile functions.
- the profile functions are calculated as the convolution of the theoretical functions for physical broadening and the instrumental profiles.

Therefore neither the separation of the peaks nor the deconvolution is needed.
The theoretical intensity pattern

\[ I_{\text{theoretical}} = BG(2\Theta) + \sum_{hkl} I_{\text{MAX}}^{hkl} I^{hkl}(2\Theta - 2\Theta_0^{hkl}), \]

where:

\[ I^{hkl} = I^{hkl}_{\text{instr.}} \ast I^{hkl}_{\text{size}} \ast I^{hkl}_{\text{disl.}} \ast I^{hkl}_{\text{pl. faults}}, \]

\[ I^{hkl}_{\text{instr.}} : \text{measured instrumental profile which is directly used} \]

The measured and theoretical patterns are compared using a nonlinear least-squares algorithm, the fitted parameters are the microstructural parameters (no individual profile parameters are used).
Instrumental pattern of LaB$_6$
Instrumental pattern of LaB$_6$
Al-3Mg ball milled 3 h.
Al-6Mg ball milled 6 h.

Results of the CMWP fit:

\( m = 21 \text{nm} \)

\( \sigma = 0.36 \)

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

\( M = Re \sqrt{\rho} = 1.3 \)

\( q = 1.3 \)
Al-20Mg ball milled 32 h.
Microstructure of ball milled Al-20Mg

(Révész et al., J. of Metast. and Nanocr. Mat., 2005)
Microstructure of ball milled Al-20Mg

(Révész et al., J. of Metast. and Nanocr. Mat., 2005)
CMWP features

- supports cubic, hexagonal or orthorombic crystal system
- it supports (practically) unlimited number of phases
- the lognormal size distribution and spherical or ellipsoidal crystallite shape can be used
- planar faults effect (intrinsic, extrinsic faults or twins) can be included
- the Wilkens or the Groma-Csikor strain function can be used
- it supports the fitting of the contrast factor parameters or individual contrast factors can be used
- it is using the measured instrumental profiles directly
- the peak positions and intensities can be refined
CMWP features

- each parameter value can be fixed, $d \cdot e$ can be fixed too
- weighting and parameter scaling is implemented
- it has a command line interface: evaluate which can be used interactively from a terminal window or by setting the parameter values and options in the ini files it can be automatically invoked without interaction (e.g. using it in a cycle or running it as a cron/at job)
- it has a graphical JAVA interface: every parameter value and option can be set in a JAVA panel and all the functions of the program can be reached
- it can be run using the WWW frontend: in this case only a working Web browser is needed, the input files can be uploaded to the server and the parameter values and options can be set by the frontend
File formats

ASCII input files:

- powder pattern file
- instrumental profiles
- indexing file
- background spline’s base points file
- ini files
Powder pattern file

2-column file, contains: $2\Theta$, $I$.

Example:

<table>
<thead>
<tr>
<th>$2\Theta$</th>
<th>$I$</th>
</tr>
</thead>
<tbody>
<tr>
<td>100.7800</td>
<td>335</td>
</tr>
<tr>
<td>100.7950</td>
<td>331</td>
</tr>
<tr>
<td>100.8100</td>
<td>335</td>
</tr>
<tr>
<td>100.8250</td>
<td>331</td>
</tr>
<tr>
<td>100.8400</td>
<td>342</td>
</tr>
<tr>
<td>100.8550</td>
<td>335</td>
</tr>
</tbody>
</table>
Instrumental profiles

2-column files, containing: $K - K_0$, $I$.

- $K = 2 \frac{\sin(\Theta)}{\lambda}$

- $K_0$: the K value at the peak center.

Example:

-0.0023 0.945234
-0.00112 0.955681
0 0.985527
0.00111 0.983823
0.00222 0.97676
Indexing file

3-column file, contains: $2\Theta_0$, $I_{MAX}$, $hkl$, $phase$.

Example:

<table>
<thead>
<tr>
<th>$2\Theta_0$</th>
<th>$I_{MAX}$</th>
<th>$hkl$</th>
<th>$phase$</th>
</tr>
</thead>
<tbody>
<tr>
<td>38.2887</td>
<td>13826</td>
<td>111</td>
<td>0</td>
</tr>
<tr>
<td>44.4726</td>
<td>5828</td>
<td>200</td>
<td>0</td>
</tr>
<tr>
<td>64.6108</td>
<td>2544</td>
<td>220</td>
<td>0</td>
</tr>
<tr>
<td>77.5747</td>
<td>2143</td>
<td>311</td>
<td>0</td>
</tr>
<tr>
<td>81.7108</td>
<td>579</td>
<td>222</td>
<td>0</td>
</tr>
<tr>
<td>98.1789</td>
<td>205</td>
<td>400</td>
<td>0</td>
</tr>
<tr>
<td>110.714</td>
<td>566</td>
<td>331</td>
<td>0</td>
</tr>
<tr>
<td>115.119</td>
<td>488</td>
<td>420</td>
<td>0</td>
</tr>
</tbody>
</table>

...
Spline background

2-column file, contains: $2\Theta$, $I$.

Example:

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>120</td>
</tr>
<tr>
<td>40</td>
<td>420</td>
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<tr>
<td>60</td>
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<tr>
<td>65</td>
<td>520</td>
</tr>
<tr>
<td>70</td>
<td>580</td>
</tr>
<tr>
<td>93</td>
<td>670</td>
</tr>
</tbody>
</table>
The CMWP webpage

Available at:

http://www.renyi.hu/cmwp

After an easy Registration you get access to:

- the WWW frontend of the CMWP and MWP programs: you can evaluate online your samples, only a Web browser is needed

- the complete program packages: you can download and install it on your own Linux computer

Documentation page:

http://www.renyi.hu/cmwp/doc
The JAVA frontend of CMWP

![JAVA frontend interface for CMWP](image)

- **CMWP fit control**
  - **data/Al6Mg6**

- **Controls and Settings**
  - **CUBIC**: ✔️
  - **HEXAGONAL**: ➥
  - **ORTHOROMIC**: ➥

- **Unit Parameters**
  - **lat_a (CUB):** 0.405
  - **lat_b (ORT):** 0.15406
  - **Wavelength (nm):** 0.286378
  - **Ch00 or Ch0k**: 0.15

- **Additional Options**
  - **Include St. Faults effect**: ✔️
  - **Use ellipsoidal size func**: ✔️
  - **Stacking.dat file**: Use individual C factors
  - **Disable single count code**: ✔️
  - **Profile cutting parameter**: 6.0

- **Fit Data Parameters**
  - **FT limit of no instr. eff**: 1024
  - **Min 2*theta/K**: 50
  - **FIT limit**: 1e-9

- **Initialization Parameters**
  - **init_a (CUB):** 1.5
  - **init_a1 (HEX):**
  - **init_a2 (ORT):**
  - **init_a3 (ORT):**
  - **init_a4 (ORT):**
  - **init_a5 (ORT):**

  - **init_b**: 3.0
  - **init_c**: 1.0
  - **init_d**: 80
  - **init_e**: 0.05

- **Scaling Parameters**
  - **scale_a**: 1.0
  - **scale_b**: 1.0
  - **scale_c**: 1.0
  - **scale_d**: 1.0
  - **scale_e**: 1.0

- **Button Controls**
  - **Call MKSpline**
  - **Call MKSpline2**
  - **Index peaks**
  - **Set Individ. C values**
  - **Clone INI files**
  - **Save INI files**
  - **Start FIT**
  - **Stop FIT**
  - **Update Params**
  - **View Solutions**
  - **View FIT**
  - **Exit**
The MKSPLINE program of CMWP
The peak indexing program of CMWP
Setting the individual C values
The WWW frontend of CMWP

Convolutional Multiple Whole Profile fitting Main Page

CMWP-fit is a program for evaluating diffraction profiles using the method of Convolutional Multiple Whole Profile fitting. If you want to know more about the method and the program, click [here](http://www.renyi.hu/cmwp/). A detailed help is available in the [documentation page](http://www.renyi.hu/cmwp/).

It is freely available for non-commercial, scientific purposes for users who accepted the [TERMS AND CONDITIONS](http://www.renyi.hu/cmwp/) of usage. You can ask for a username and password in the [TERMS AND CONDITIONS](http://www.renyi.hu/cmwp/) page.

Users can access the program via this page:
you can [upload your patterns](http://www.renyi.hu/cmwp/),
[upload your instrumental profile files](http://www.renyi.hu/cmwp/),
[view the list of your samples](http://www.renyi.hu/cmwp/),
or [start the evaluation program](http://www.renyi.hu/cmwp/).
You can also [view the results](http://www.renyi.hu/cmwp/) of previous runs.
There is also a [documentation page](http://www.renyi.hu/cmwp/).

This procedure is similar to the method of [Multiple Whole Profile fitting](http://www.renyi.hu/cmwp/), however the CMWP method is more sophisticated, so you should use the CMWP method when possible.

[MDKDAT](http://www.renyi.hu/cmwp/) is a program for preparing X-ray diffraction data. The outputs of MKDAT can be used as the *peak indexing file*, as the *background spline's base points file* as well as the *instrumental profile files* for the CMWP program. Follow [this](http://www.renyi.hu/cmwp/) link to download it.
The CMWP upload page

Here you can transfer the powder pattern file, the indexing file and the background spline's base points file corresponding to your sample from your computer to your main directory with this simple tool. Specify the name of your sample (the basename of the files), click on the Browse... buttons to select the powder pattern file, the indexing file and the spline base points file and then click Upload Files.

Name of the sample: Al-20Mg32

Powder pattern file: /home/ribarik/Al-Mg/almg32.dat
Indexing file: /home/ribarik/Al-Mg/almg32.peak-index.dat
Spline base points file: /home/ribarik/Al-Mg/almg32.bg-spline.dat

Upload Files  Clear

Click here to start the evaluation procedure.
The CMWP instrumental upload page

CMWP-fit Instrumental Profile Upload Page

Here you can transfer the previously prepared instrumental profiles from your computer to your main directory with this simple tool. Note that these files should be two-column ASCII files, the first column should contain K-K0 and the second contains the intensity values, where K=2*sin(Theta)/lambda and K0 is the K value at the center of the peak. Specify the name of your instrumental sample (the name of the directory in which the instrumental profiles will be uploaded), specify the TwoTheta values in degrees, click on **Browse...** to select the files and then click **Upload Instrumental Profiles**.

Name of the instrumental sample: **LaB6-Xpert**

<table>
<thead>
<tr>
<th>TwoTheta</th>
<th>filename</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>21.34</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/21.34</td>
<td>Browse...</td>
</tr>
<tr>
<td>30.37</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/30.37</td>
<td>Browse...</td>
</tr>
<tr>
<td>37.42</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/37.42</td>
<td>Browse...</td>
</tr>
<tr>
<td>43.5</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/43.5</td>
<td>Browse...</td>
</tr>
<tr>
<td>48.96</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/48.96</td>
<td>Browse...</td>
</tr>
<tr>
<td>53.98</td>
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<td>63.22</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/63.22</td>
<td>Browse...</td>
</tr>
<tr>
<td>67.56</td>
<td>/home/ribarik/LaB6-xpert-inst-dat/67.56</td>
<td>Browse...</td>
</tr>
</tbody>
</table>
The CMWP sample listing page

CMWP-fit: list of ribarik's samples

CdF-inst-dat
CdF30
SrF40
proba
Evaluation of diffraction patterns using the method of Convolutional Multiple Whole Profile fitting

Files

Please enter the name of the sample: example-galena

Selection of the crystal system

The crystal system is:
- cubic (default)
- hexagonal
Input parameters

Please enter the value of the lattice constant "a" (in nm): 0.593
Please enter the value of the lattice constant "c" (in nm): 1.0
Please enter the value of the absolute value of the Burgers vector (in nm): 0.419314
Please enter the average contrast factor of the h00 (or hk0) reflections: 0.12
Please enter the wavelength (in nm): 0.154

Please note that setting of the lattice parameter "c" has effect only if the crystal system is hexagonal.

Instrumental profiles

Set this if you have instrumental profiles:

If you have instrumental profiles, please enter the name of the of the instrumental profile directory: example-galena-inst-dir
The interval used for fitting and plotting

The interval used for fitting and plotting is: [minx,maxx]

Please specify the value of minx (in degrees): 
Please specify the value of maxx (in degrees): 
Please note that if you don't fill these fields, all the data in the measured powder pattern file will be used.

Selection of the size function

The program will use the spheric size function (default) 
The program will use the ellipsoidal size function 
The program will disable the size effect
The CMWP evaluation page IV.

Sampling of the theoretical Fourier transforms

Please specify the intensity limit of the normalized Fourier transforms: $1 \times 10^{-7}$

Please note that you need to specify this limit only in case if you don't have instrumental profiles.

Sampling of the simulated powder pattern data

Please specify N1: 1024
Please specify N2: 1024
Please specify the profile cutting parameter (in Two theta degrees): 4.0
Initial values of the parameters

Please enter the initial value of the parameter "a" (or "a1"): -6.0
Please enter the initial value of the parameter "a2": 1.0
Please enter the initial value of the parameter "b": 3.0
Please enter the initial value of the parameter "c": 1.0
Please enter the initial value of the parameter "d": 80.0
Please enter the initial value of the parameter "e": 0.05
Please enter the initial value of the parameter "epsilon": 1.0

fix:  

fix d*e:  

Please note that setting of the parameter "a2" has effect only if the crystal system is hexagonal and setting of the parameter "epsilon" has effect only if the size function is ellipsoidal.
Peak parameter refinement and weighting

Set this if you want to fit the peak positions and peak intensities:

Set this if you want to use weights in the fitting algorithm:

Fit control

Please specify the limit for stopping: $1 \times 10^{-9}$

Please specify the maximal number of iterations: 200

After filling in the relevant fields in this form, please press/click this button: Start fitting to begin the evaluation procedure. Click the button Reset to reset the values.
The CMWP results page

CMWP-fit: ribarik's results

CdF30-2004-05-11-10-02-01
CdF30-2004-05-20-22-53-21
CdF30-2004-05-21-00-15-32
CdF30-2004-05-21-00-15-49
CdF30-2004-05-21-00-16-24
CdF30-2004-05-21-00-18-23
CdF30-2004-05-21-00-19-42
CdF30-2004-05-21-00-21-15
CdF30-2004-05-21-00-30-57
CdF30-2004-05-21-00-32-37
SrF40-2004-07-01-13-12-39
SrF40-2004-07-02-14-00-19
The solutions:
a=-0.829953
b=1.29038
c=1.32846
d=53.1719
e=0.0752277

m=\exp(b)=3.63417
\sigma=c/\sqrt{2}=0.939363
D=gnuplot>nm
d=59.8061nm
L0=21.9974nm

q=a=-0.829953
\rho=2/(\pi*(0.419314nm*d)**2)=0.00128067(1/nm)^2
Re^* = \exp(-1/4)/(2*e)=5.17629nm
M^*=(Re^*)*sqrt(\rho)=0.185241

After 23 iterations the fit converged.
final sum of squares of residuals: 1.99428e+07
rel. change during last iteration: -9.79374e-10
Final set of parameters
<table>
<thead>
<tr>
<th>Asymptotic Standard Error</th>
</tr>
</thead>
</table>
Example of a statistics file

final sum of squares of residuals : 1.99428e+07
rel. change during last iteration : -9.79374e-10

degrees of freedom (ndf) : 4146
rms of residuals (stdfit) = sqrt(WSSR/ndf) : 69.355
variance of residuals (reduced chisquare) = WSSR/ndf : 4810.12

Final set of parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Error</th>
<th>Error (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>-0.829953</td>
<td>+/- 0.1029</td>
<td>12.39%</td>
</tr>
<tr>
<td>b</td>
<td>1.29038</td>
<td>+/- 0.03568</td>
<td>2.765%</td>
</tr>
<tr>
<td>c</td>
<td>1.32846</td>
<td>+/- 0.008704</td>
<td>0.6552%</td>
</tr>
<tr>
<td>d</td>
<td>53.1719</td>
<td>+/- 0.7626</td>
<td>1.434%</td>
</tr>
<tr>
<td>L0</td>
<td>4.3159520070043</td>
<td>+/- 0.7626</td>
<td>1.434%</td>
</tr>
</tbody>
</table>

correlation matrix of the fit parameters:

<table>
<thead>
<tr>
<th></th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>1.000</td>
<td>0.460</td>
<td>-0.534</td>
<td>-0.874</td>
</tr>
<tr>
<td>b</td>
<td>0.460</td>
<td>1.000</td>
<td>-0.990</td>
<td>-0.421</td>
</tr>
<tr>
<td>c</td>
<td>-0.534</td>
<td>-0.990</td>
<td>1.000</td>
<td>0.437</td>
</tr>
<tr>
<td>d</td>
<td>-0.874</td>
<td>-0.421</td>
<td>0.437</td>
<td>1.000</td>
</tr>
</tbody>
</table>
Summary

The MWP method works on the separated profiles or their Fourier transforms and it’s a powerful method to extract microstructural parameters from X-ray peak profiles.

The CMWP method works in the direct space on the whole pattern, the instrumental effect is included in the model based pattern by using convolution and therefore neither the separation of the peaks nor the deconvolution is needed.

Using these methods several microstructural parameters can be determined for size, dislocations, and planar faults.