Microstructural parameters from ***Multiple Whole Profile (MWP)*** or ***Convolutional Multiple Whole Profile (CMWP)*** computer programs

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Line Profile Analysis Workshop

Tutorial outline:

1) Brief theoretical background,
2) Microstructural parameters,
3) Determining the dislocations types,
    1) cubic crystal system,
    2) hexagonal crystal system,
4) MWP (**M**ultiple **W**hole **P**rofile **F**itting),
5) CMWP (**C**onvolutional **M**ultiple **W**hole **P**rofile **F**itting),
6) ANIZC – program,
7) Examples,
8) Summary.

MWP and CMWP-fit

MWP and **CMWP**-fit are computer programs developed by ***Prof. Ungar Tamas*** and ***Dr. Ribarik Gabor*** (Diffraction Laboratory, Department of General Physics at Eotvos Science University in Budapest, Hungary) for the determining the microstructural parameters from X-ray diffraction patterns of materials with cubic or hexagonal crystal lattice.

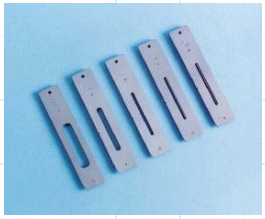
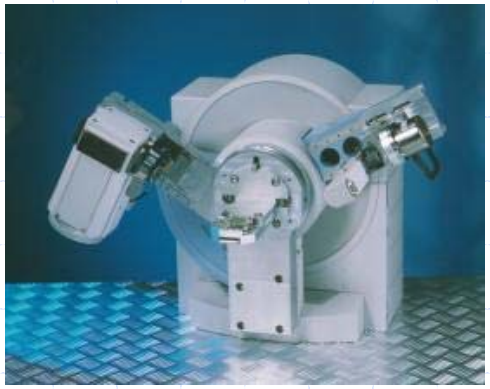
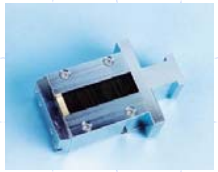
The programs are available for on-line usage at:

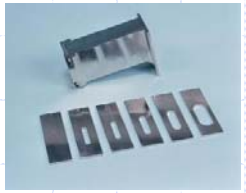
http://www.renyi.hu/cmwp/
http://www.renyi.hu/mwp/

Sources of X-ray Line Broadening:

Instrumental sources: broadening caused by ***diffractometer imperfections***

(Non ideal optics, Wavelength Dispersion, Sample Transparency, Axial Divergence, Detector resolution)

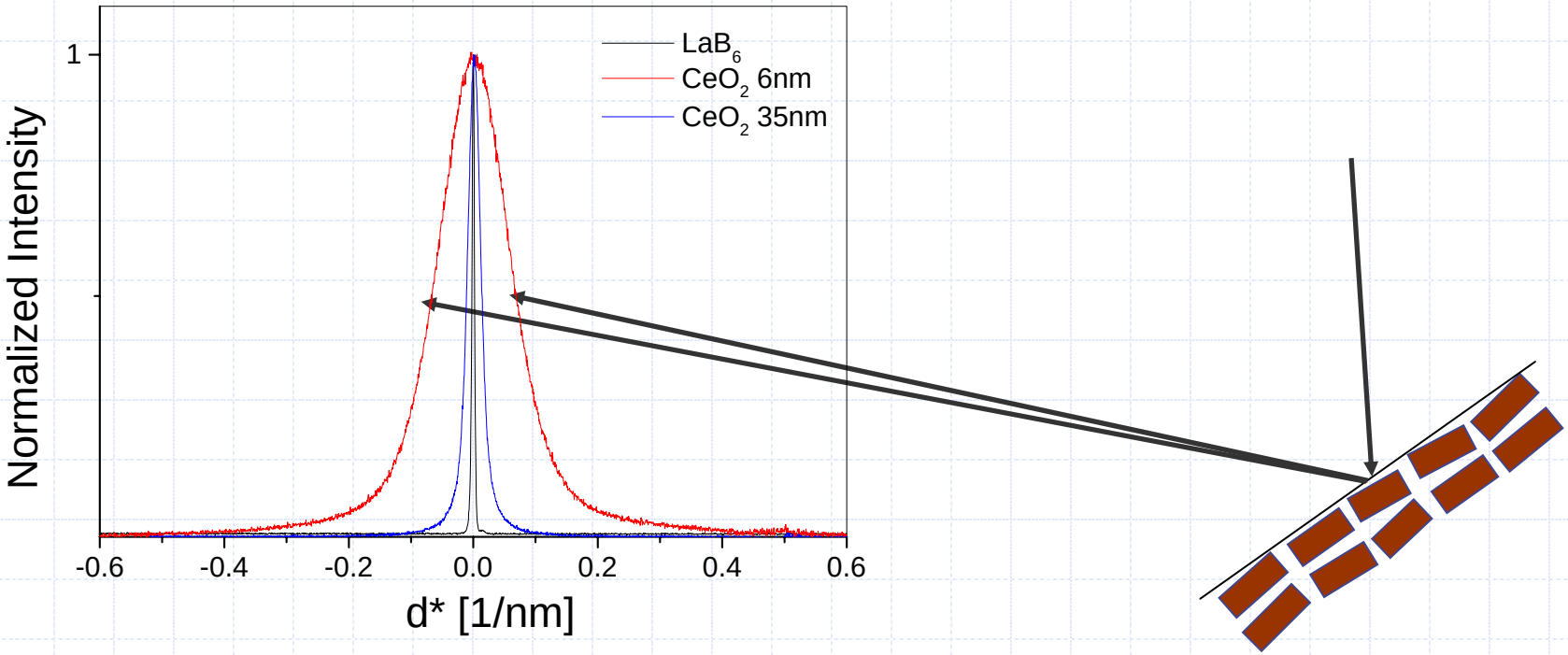


Standards for instrumental broadening: **SRM660a (LaB_6) , SRM640c (Si) - NIST**

Standard specimen preparation: **Berkum, J. G. M. van, Sprong, G. J. M., Keijser, Th. H. de, Delhez, R. and Sonneveld, E. J.** *(1995), Powder Diffr. 10, 129-139*

Physical sources X-ray line profile broadening :

- broadening effect due to crystallite smallness (**coherent domain).**



Coherent domain X-ray line broadening may include effects of stacking- and twin faults and sub-grain structures (for instance small-angle grain boundaries).

Physical sources X-ray line profile broadening :

- broadening effect due to micro-strain:

  — dislocations,

  — stacking faults,

  — precipitates of second phase particles,

  — concentration gradients in solid solutions,

  — severely distorted grain boundaries in nano-crystalline materials,

  — different types of internal stresses or strains which may be heterogeneous.

X-ray Line Broadening

$$I_m = I_{instrumental} * I_{size} * I_{distorsion} * I_{other}$$

MWP and CMWP-fit - theoretical background

Assumptions:

1) X-ray diffraction line is broadened due to:

   - small coherent domains (usually smaller then 1 μm),

   - lattice distortion.

2) The crystallites are spherical or ellipsoidal,

3) The crystallite size distribution is lognormal.

4) The lattice distortion is assumed to be caused by dislocations

MWP and CMWP-fit - theoretical background

- ***Physical*** profile functions - $I_m = I_{size} * I_{distorsion}$

Size effect:

Assumption → log-normal size distribution and spherical crystallites:

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

where *m* is the median,σ is the variance of the log-normal size distribution and erfc is the complementary error function.

MWP-fit - theoretical background

- ***Physical*** profile functions - $I_m = I_{size} * I_{distorsion}$

$A(L) = A^{size}(L)\, A^{distorsion}(L)$

Strain effect - dislocations:

Based on the Wilkens model (1970) the Fourier transform of the strain profiles are written as:

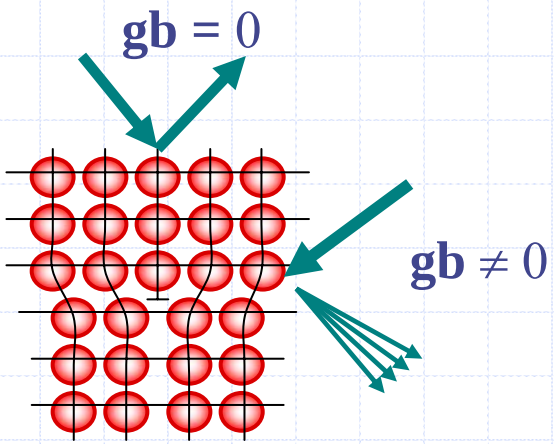
$$A^D(L) = \exp\{-2\pi^2 g^2 L^2 [(b/2\pi)^2 \pi\rho\overline{C} f(L/R_e^*)]\}$$

where g is the absolute value of the diffraction vector; L is the Fourier length, b is the absolute value of the Burgers vector, ρ is the dislocation density; $\overline{C}$ is the average contrast factor of dislocation; R_e^* is the effective outer cut-off radius and f is the Wilkens function.

Strain effect - dislocations

Strain anisotropy

Similar to TEM



MWP - fit:

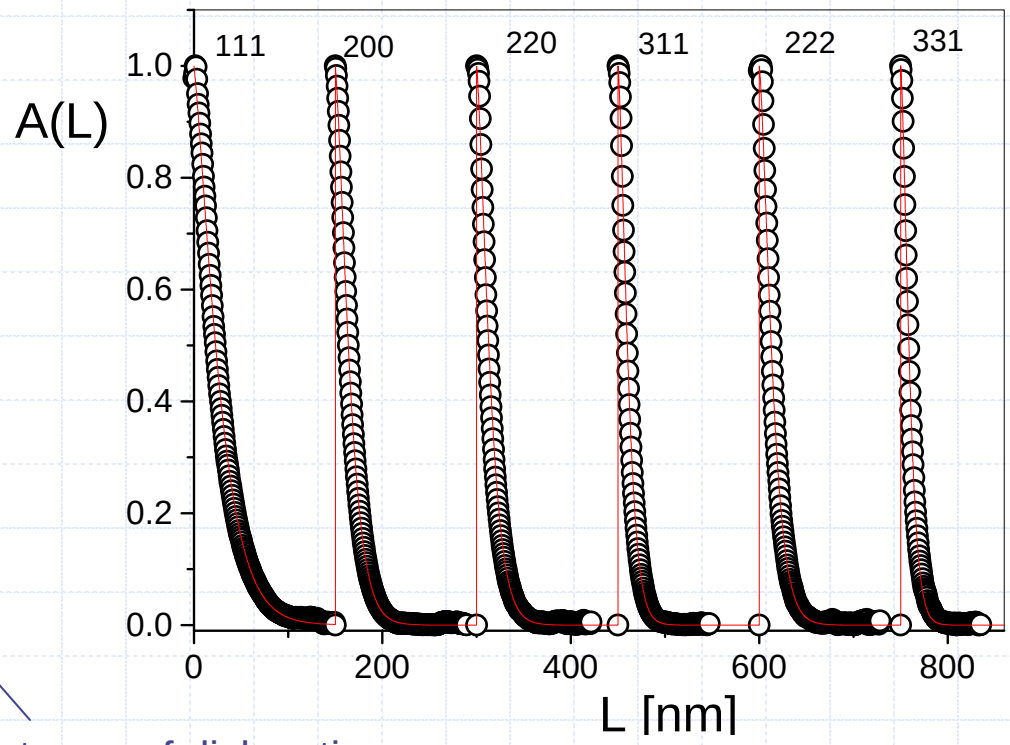
The Fourier transforms of the measured profiles are fitted all at once with:

$$A(L) = \left[A^S(L) / A^S(0)\right] \exp\{-2\pi^2 g^2 L^2 [(b/2\pi)^2 \pi \rho \overline{C} f(L/R_e^*)]\}$$

MWP - fit:

$$A_L = A_L^S \, A_L^D(hkl)$$

log-normal size distribution: $\Rightarrow$ **m** and σ



strain in terms of dislocations $\Rightarrow \rho$ and **M**

$\Rightarrow$Strain anisotropy: C_{av} (**q**, $\mathbf{c_{ij}}$)

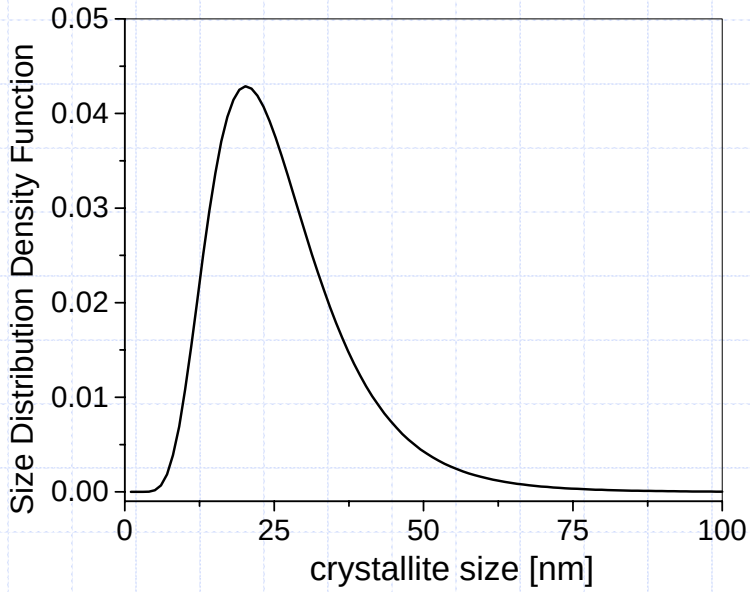
Nonlinear least squares method:

$$(A^{(meas)} - A^{(calc)})^2 = \min.$$

Microstructural parameters obtained from MWP and CMWP procedures – m and σ

Crystallite size distribution:

The procedures assumes lognormal size distribution with spherical or ellipsoidal crystallite shape.



spherical crystallites:

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

Strain anisotropy parameter, *q* :

Average contrast factors of dislocations for cubic crystal systems:

$$\overline{C} = \overline{C_{h00}}(1 - q \cdot H^2)$$

where $H^2=(h^2k^2+ h^2l^2+ k^2l^2) / (h^2+k^2+l^2)^2$

q depends on the a) studied material elastic proprieties,

b) dislocation geometry/character.

Reference: Ungár, T., Tichy, G. The effect of dislocation contrast on X-ray line profiles in untextured polycrystals. Phys. Stat. Sol. 1999;171:425434.

Microstructural parameters obtained from MWP and CMWP procedures:

Strain anisotropy parameter, ***q*** : $\overline{C} = \overline{C_{h00}}(1 - q \cdot H^2)$

1) If the dislocation structure is known from other techniques, such as TEM, than the C_{h00} and q can be numerically calculated for the given dislocation structure and inputted as a fix parameters in the MWP or CMWP procedure.

2) If the dislocation structure is unknown, than in the MWP or CMWP procedures C_{h00} is fixed while the q is refined. From the value of q the dislocations type/population can determined and the real C_{h00} can be obtained.

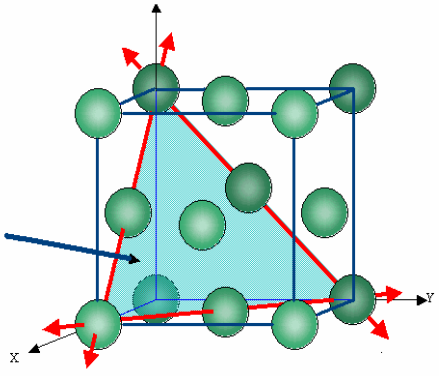
Microstructural parameters obtained from MWP and CMWP procedures:

Strain anisotropy parameter, *q* :

determining the dislocations character

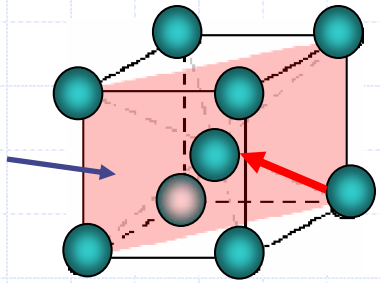
FCC: *a*/2 <110>{111}

{111}



BCC: *a*/2 <111>{110}
a/2 <111>{112}

{110}



Theoretical individual and average contrast factor of dislocations can be found in:

Ungár, T., Dragomir, I., Révész, Á., and Borbély, A.: “The Contrast Factors of Dislocations in Cubic Crystals: the Dislocation Model of Strain Anisotropy in Practice”, J. Appl. Cryst., 32, 992-1002, (1999).

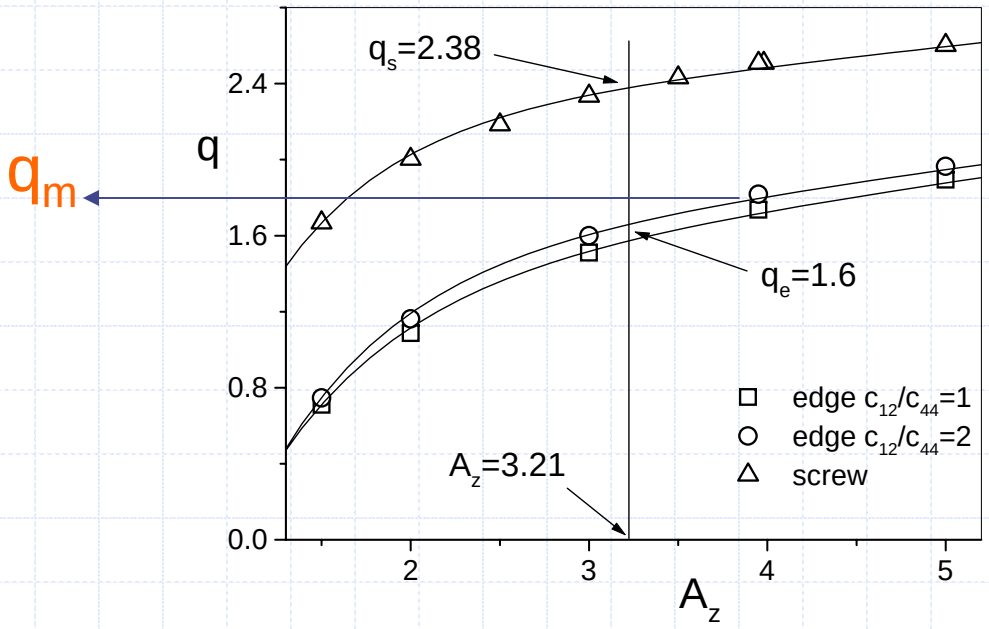
Dragomir I. C. and Ungár T.: “The dislocations contrast factors of cubic crystals in the Zener constant range between zero and unity” in Powder Diffraction Journal, 17, 104-111, (2002).

Microstructural parameters obtained from MWP and CMWP procedures:

Cubic crystal systems – theoretical average dialocation contrast factor:

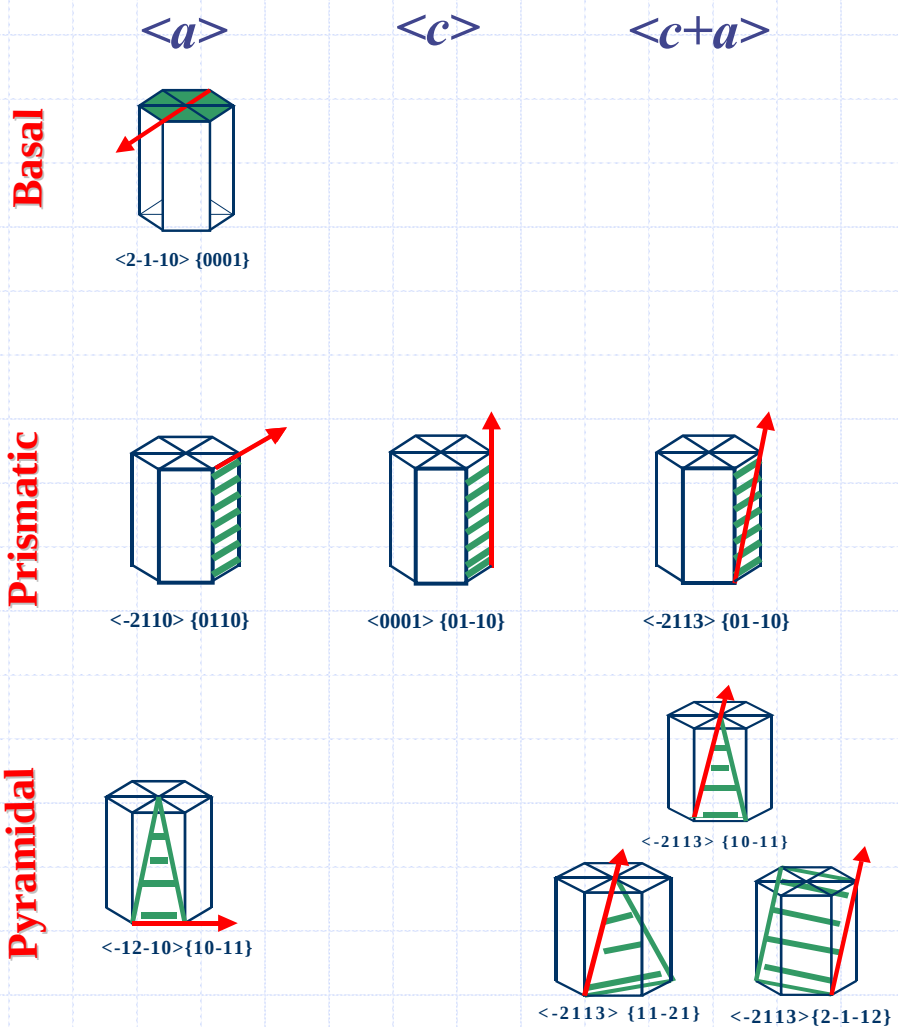
FCC system - dislocation slip systems: 1/2<110>{111} type

$$q_m = f\, q_e + (1-f)\, q_s$$



Average dislocation contrast factor for hexagonal crystal systems:

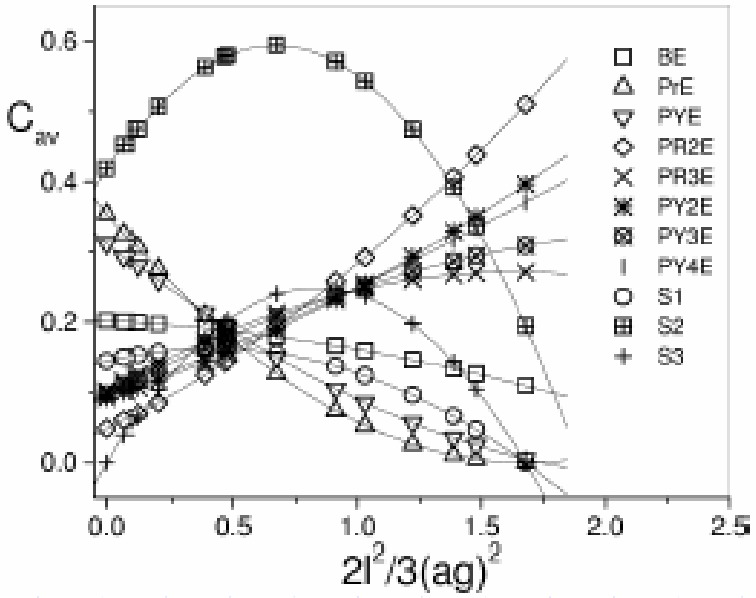
Dislocation slip systems



Microstructural parameters obtained from MWP and CMWP procedures:

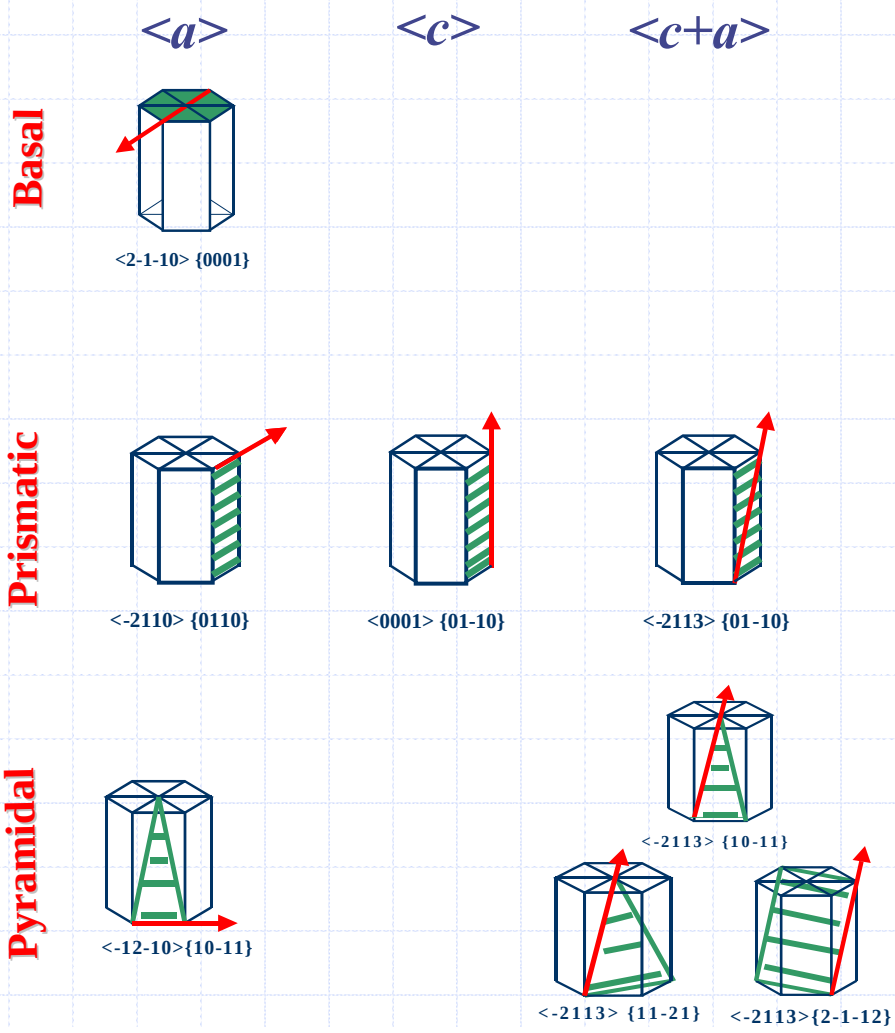
Average contrast factor for hexagonal crystal systems, q_1 and q_2:

$$\overline{C}_{hk.l} = \overline{C}_{hk.0} \left[1 + q_1 x + q_2 x^2\right],$$



Dragomir I. C. and Ungár T.: "The contrast factors of dislocations in the hexagonal crystal system" in J. Appl. Cryst., 35, 556-564, (2002).

To many unknowns – assumptions are needed



Microstructural parameters obtained from MWP and CMWP procedures:

Hexagonal crystal systems, q_1 and q_2:

Measured and Theoretical average contrast factor of dislocations:

$C_m = f_1C_1 + f_2C_2 + f_3C_3$

$f_1 + f_2 + f_3 = 1$

$\rightarrow$

$$q_1^{(m)} = \frac{1}{P}\sum_{i=1}^{3} h_i \overline{C}_{hk.0}^{(i)} b_i^2 q_1^{(i)} \quad ,$$

$$q_2^{(m)} = \frac{1}{P}\sum_{i=1}^{3} h_i \overline{C}_{hk.0}^{(i)} b_i^2 q_2^{(i)} \quad ,$$

$$\sum_{i=1}^{3} h_i = 1 \quad ,$$

where $$P = \sum_{i=1}^{3} h_i \overline{C}_{hk.0}^{(i)} b_i^2 = \overline{b^2 C_{hk.0}}^{(m)} \quad .$$

Microstructural parameters obtained from MWP and CMWP procedures – dislocation density

Evaluation of real dislocation density, ρ:

Cubic: $$....\rho\overline{C} = ...\rho\overline{C_{h00}}(1 - q \cdot H^2)$$

$$\rho^* \cdot \overline{C}_{h00}^{(input)} = \rho \cdot \overline{C}_{h00}^{(real)}$$

Hexagonal: $$....\rho b^2 \overline{C} = ...\rho \overline{b^2 C_{hk0}}(1 + q_1 x + q_2 x^2)$$

$$b^2 \overline{C_{hk.0}}^{(input)} \cdot \rho^* = \overline{b^2 C_{hk.0}}^{(real)} \cdot \rho$$

Microstructural parameters obtained from MWP and CMWP procedures – arrangement parameter

Dislocation arrangement parameter, *M* (Wilkens, 1970):

M is defined by Wilkens as the dislocation arrangement parameter.

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

M >> 1 week screening of dislocations fields

M ~ 1 quasi-homogeneous dislocation arrangement

M << 1 strong screening of dislocations filed, dipole configurations

MWP-fit:

Before using MWP the following profile corrections are necessary:

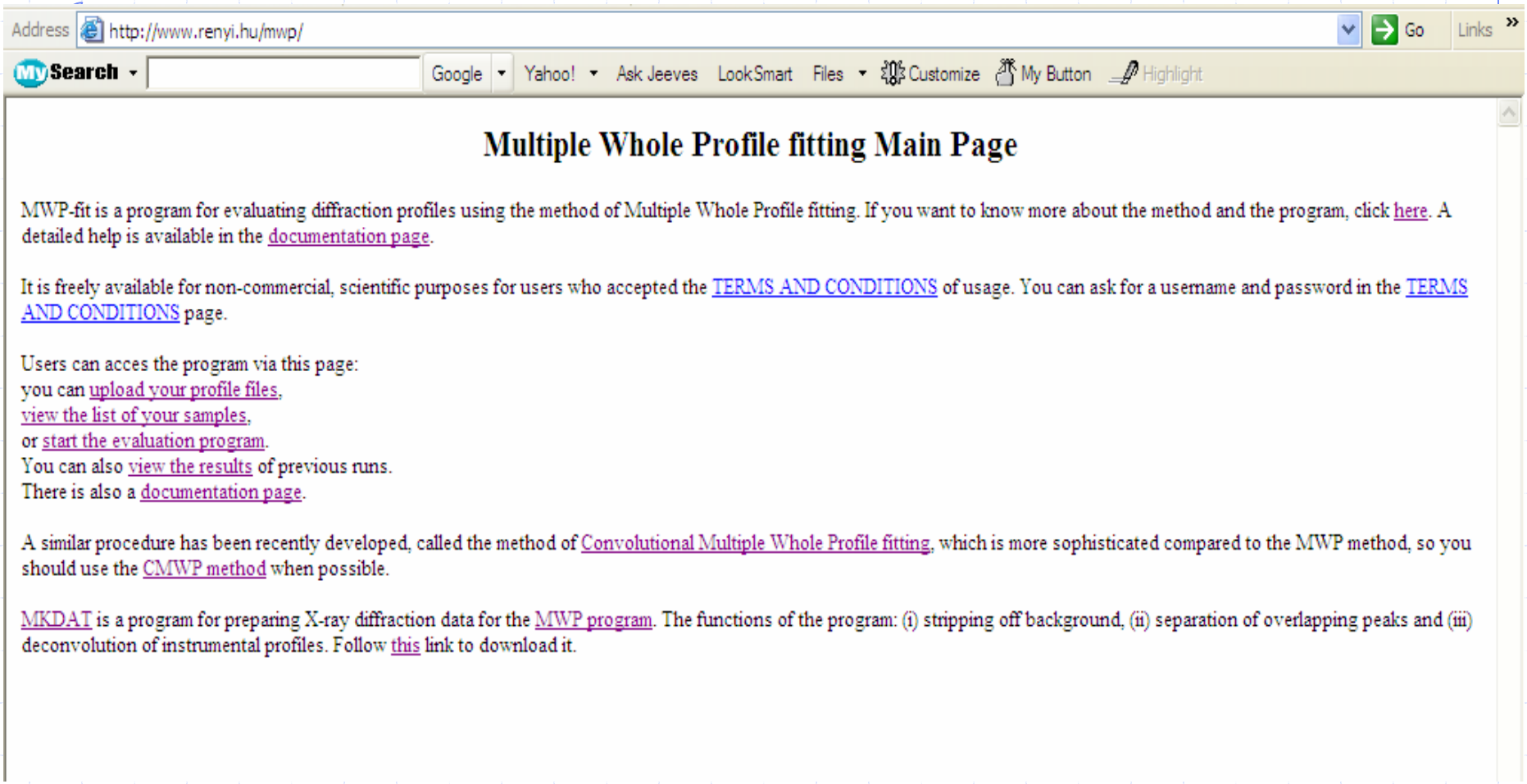
i) background
ii) overlapping peaks
iii) $K\alpha_2$
iv) instrumental effect

Free software: MKDAT at http://www.renyi.hu/mkdat

Other free software that can be used for corrections can be found at:
http://www.ccp14.ac.uk/

Also commercial software as Jade, Topas, High Score Plus, etc.

MWP – fit front-end:



MWP - fit - Input files

Format: 2-column ASCII type of files for every *hkl* diffraction profile

1) The first column contains :

$$d^* = 1/d = 2\sin(\theta - \theta_0)/\lambda$$

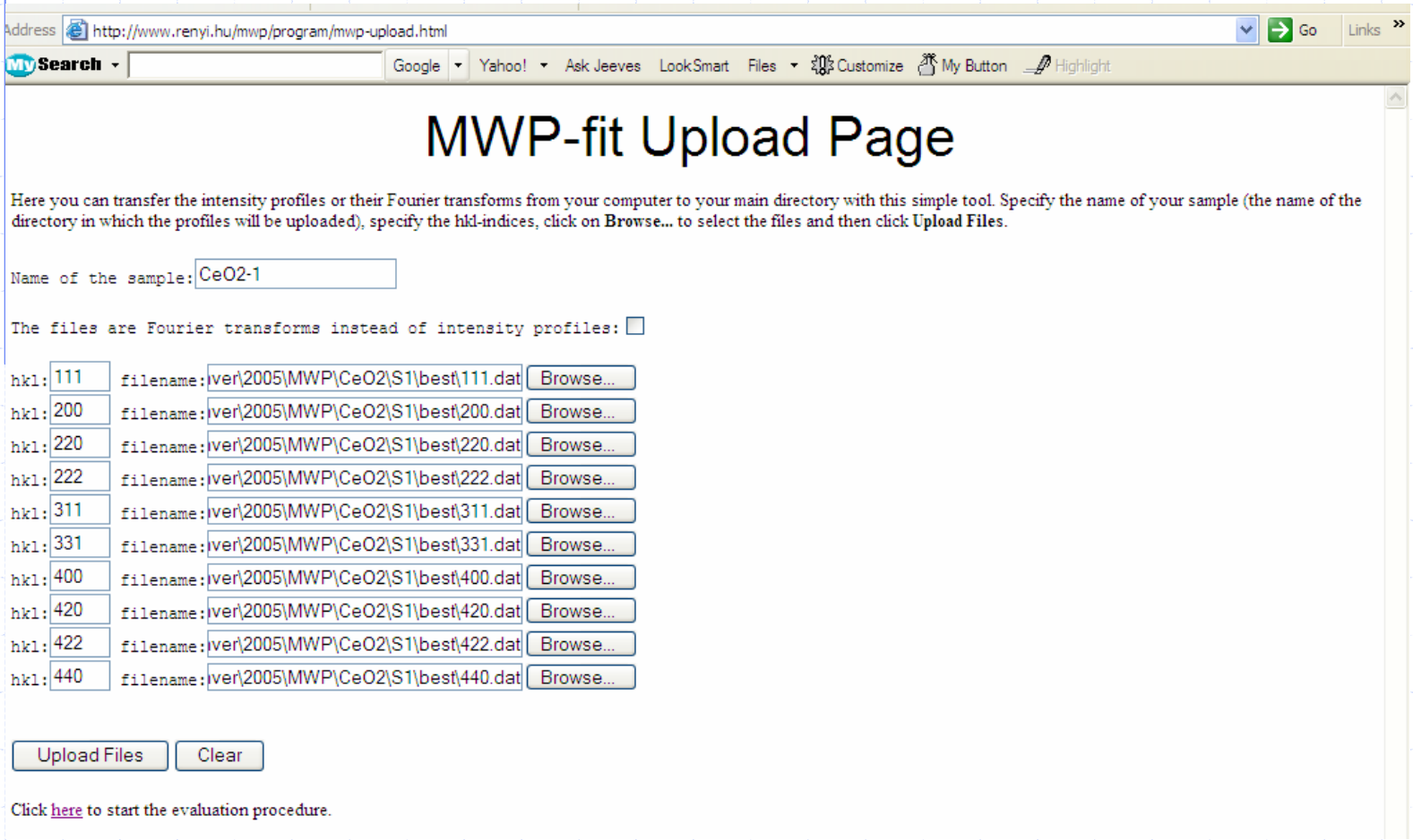
2) The second column contains the normalized intensity.

Example:

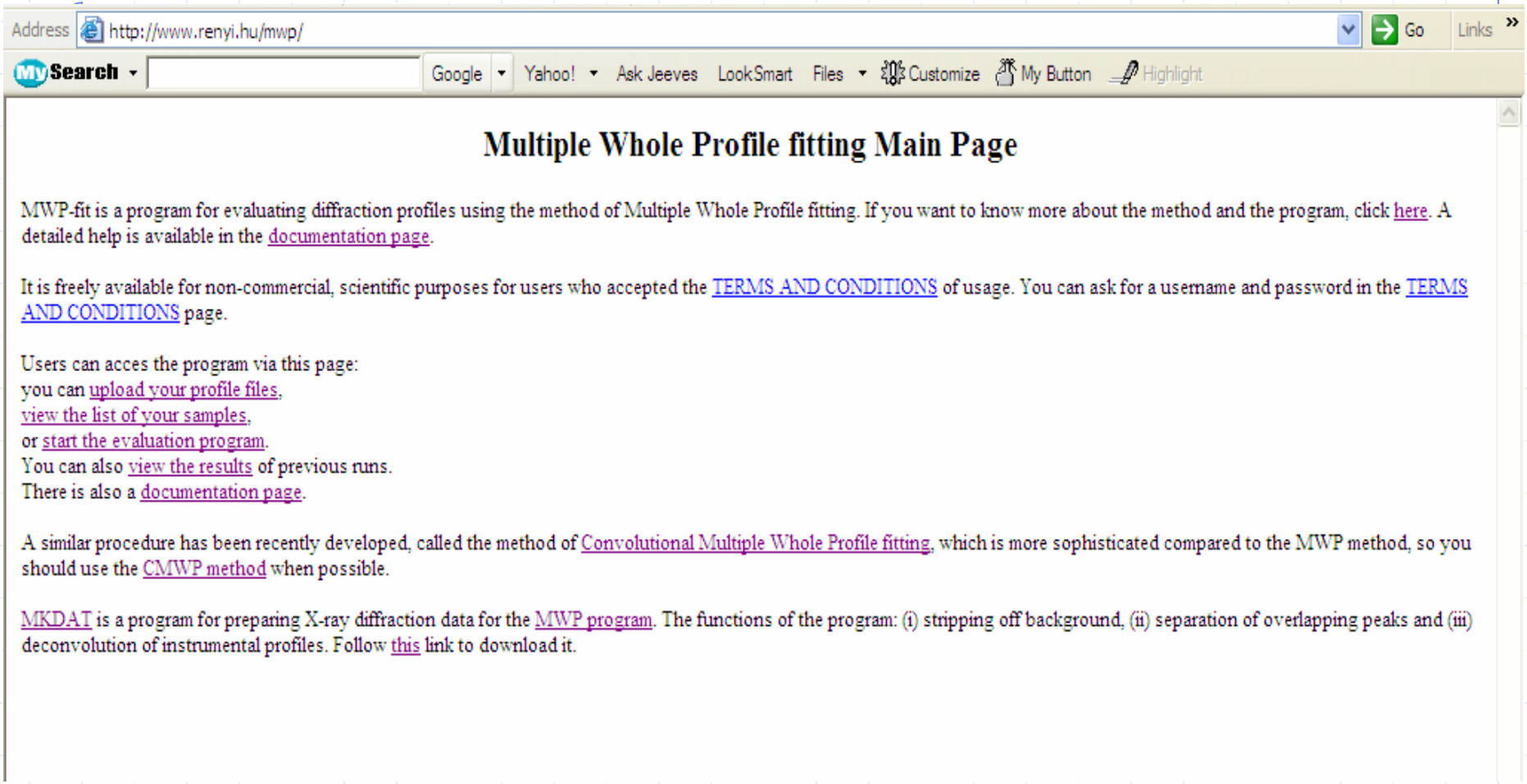
-0.0023	0.981756
-0.0018	0.989999
-0.0013	0.995834
-0.0009	0.999185
-0.0004	0.998253
0	1
0.0004	0.993943
0.0009	0.9871
0.0013	0.977779
0.0018	0.966061
0.0023	0.952054

MWP procedure:

Input files - examples

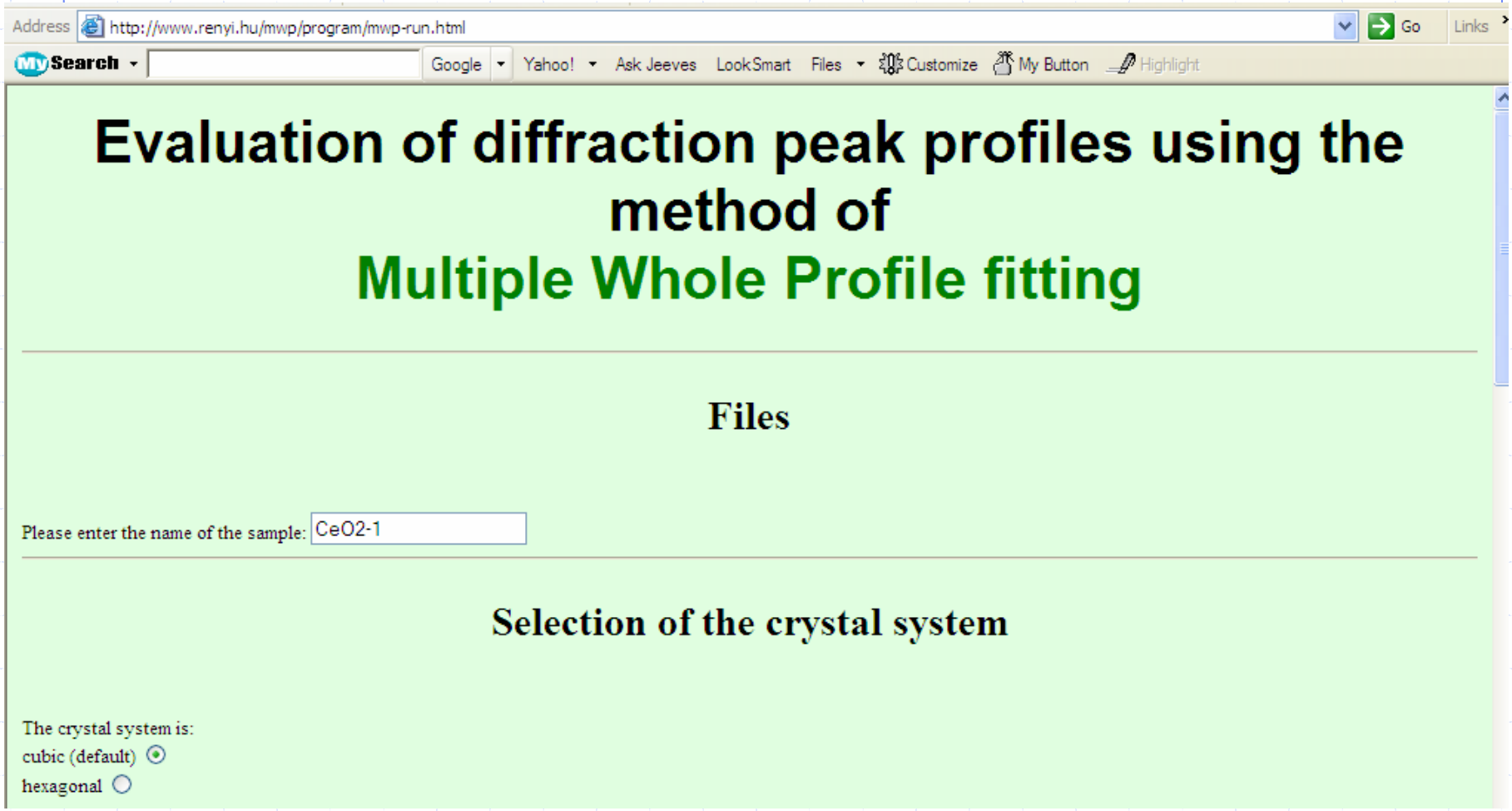


MWP – fit front-end:



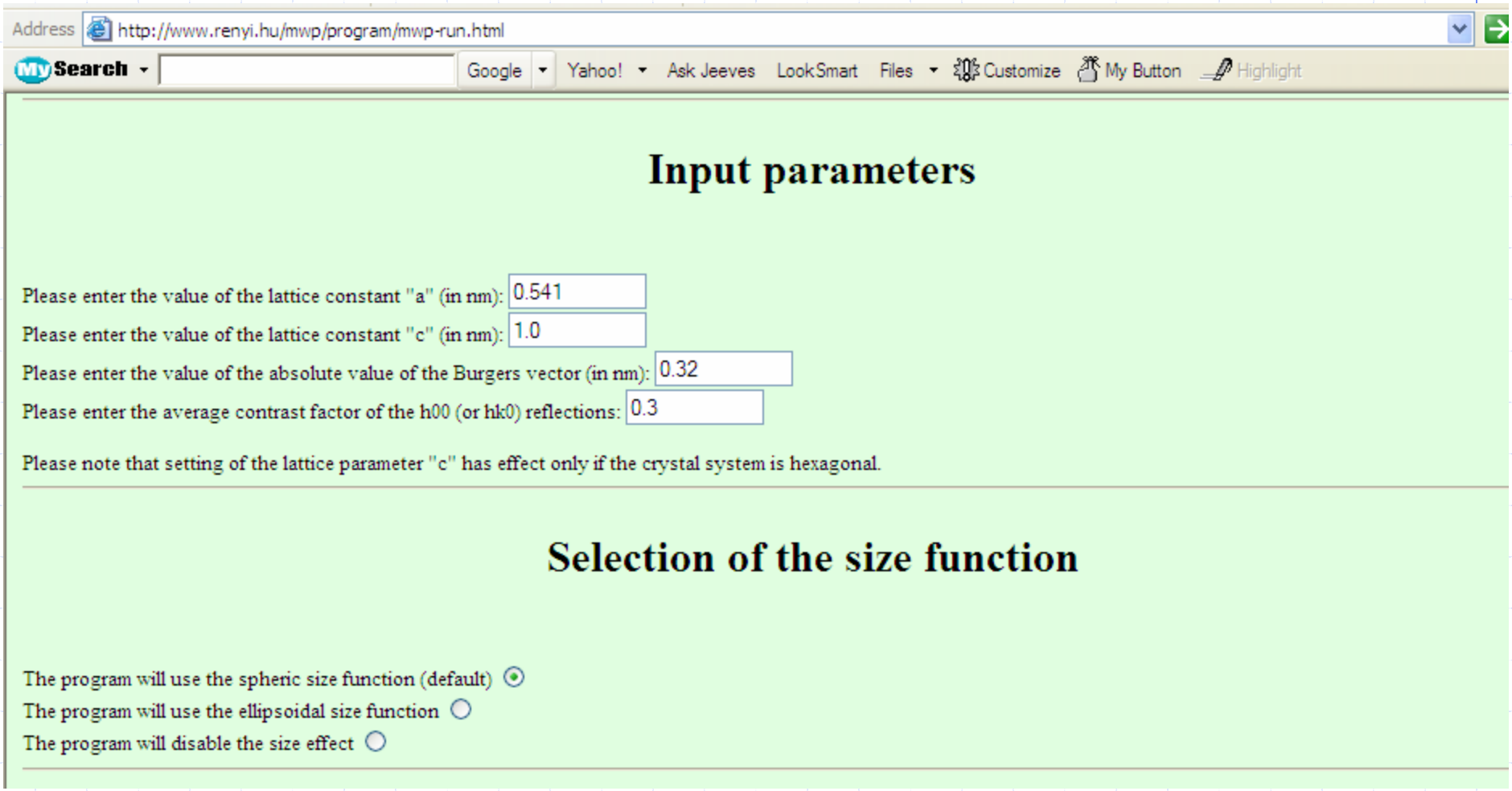
MWP procedure:

- *crystal system selection*



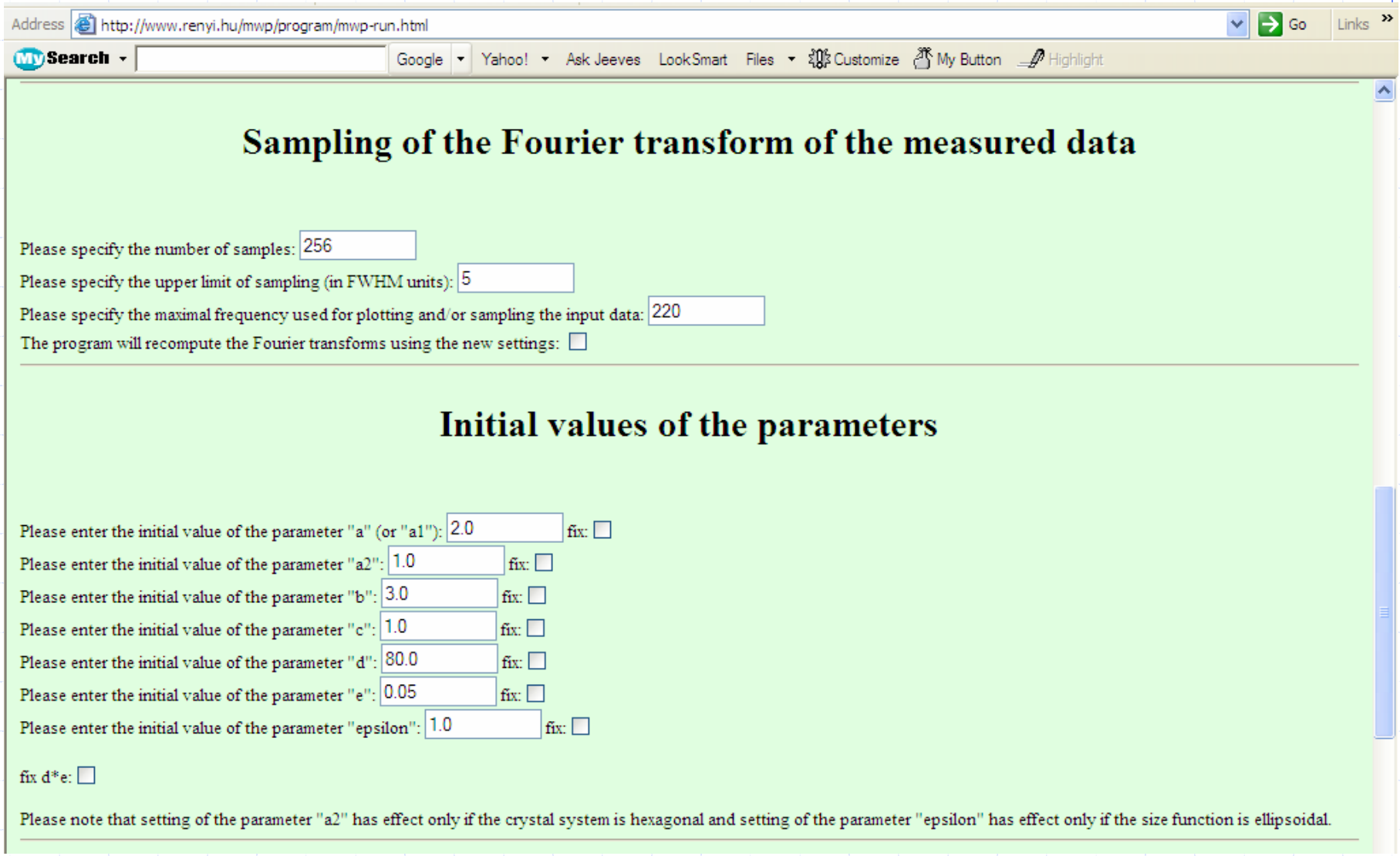
MWP procedure:

- *input parameters*

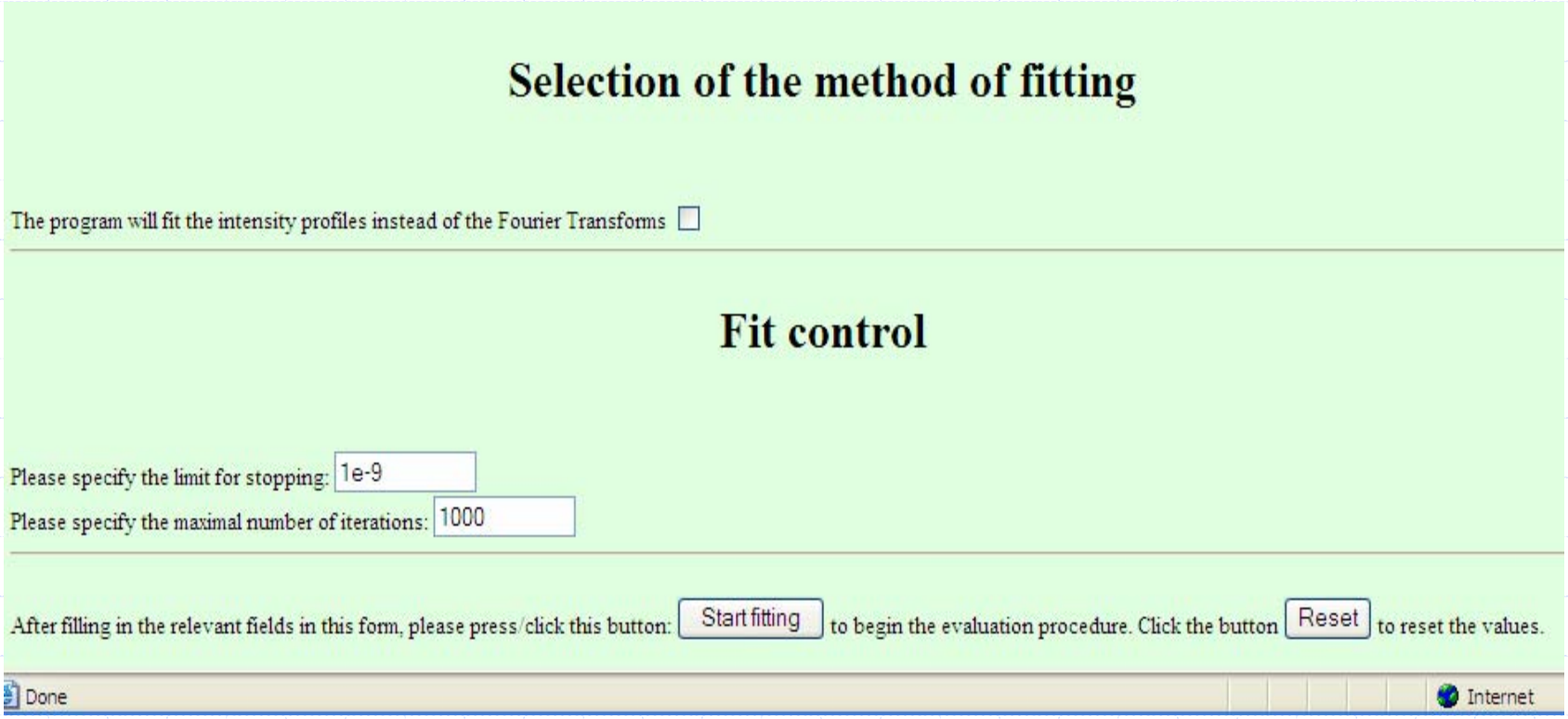


MWP procedure:

-settings



MWP procedure:



Address http://www.renyi.hu/cgi-bin/post-mwp?SAMPLENAME=CeO2-1&HEXAGONAL_CRYSTAL=n&la=0.3604&lc=1.0&bb=0.254841&C0=0.3040&N=256&FOURIER_FWH

Multiple Whole Profile fitting

Connecting the server for evaluation .....
The evaluation is started.
Please wait until this page loads to get the results.

Note that the results will be stored in http://www.renyi.hu/mwp/results/dragomir/CeO2-1-2005-08-02-05-46-33. .....

Address http://www.renyi.hu/cgi-bin/post-mwp?SAMPLENAME=CeO2-1&HEXAGONAL_CRYSTAL=n&la=0.3604&lc=1.0&bb=0.254841&C0=0.3040&N

Multiple Whole Profile fitting

Connecting the server for evaluation .....
The evaluation is started.
Please wait until this page loads to get the results.

Note that the results will be stored in http://www.renyi.hu/mwp/results/dragomir/CeO2-1-2005-08-02-05-46-33.

.....

The fitting is done, evaluate's output follows:

```
This is MWP-fit, a program for Multiple Whole Profile Fitting using
theoretical line profile functions.
Copyright (C) Gábor Ribárik and Tamás Ungár, 1998-2000. All rights reserved.
For permission to use, copy, modify this program or any of its components,
ask the authors. Redistributing this software is not allowed.

Date: 2005-08-02 05:46:34

Data directory: dragomir/CeO2-1

The value of the lattice constant (in nm): 0.3604
The absolute value of the Burgers vector (in nm): 0.254841
The average contrast factor of the h00 reflections: 0.3040
The definition of the Size Function:
sf_spheric(k,b,c)=Int(0.5*x*erfc((log(x)-b)/c)*(sin(Pi*k*x)/(Pi*k))**2,x=0..Inf)

Common characteristics of the Fourier Spectrums:
The number of samples: 256
The upper limit of sampling (in FWHM units): 5
The maximal frequency used for sampling and/or plotting: 220

*** FIT ***
```

Done

Address http://www.renyi.hu/cgi-bin/post-mwp?SAMPLENAME=CeO2-1&HEXAGONAL_CRYSTAL=n&la=0.3604&lc=1.0&bb=0.2548

```
*** FIT ***
Calculating a,b,c,d,e to fit the Fourier Spectrums with:
(fct(x)/fct(0))*exp(-(K^2*C*x^2/d^2)*wf(e*x)), where fct(x) is the Fourier
Transform of sf_spheric(x,b,c), K^2=(h^2+k^2+l^2)/a^2, C=Ch00*(1-a*H^2),
H^2=(h^2*k^2+h^2*l^2+k^2*l^2)/(h^2+k^2+l^2)^2 and wf(x)=f^*(x) as defined
by M. Wilkens in:
Proc. Conf. Fund. Aspects of Disl. Theory, National Bureau of Standards, 1969.

The initial values of the parameters:
C0=0.3040
a = 2.0
b = 3.0
c = 1.0
d = 80.0
e = 0.05
epsilon = 1.0 # FIXED

The solutions:
a=2.32832
b=3.27845
c=0.55576
d=11.089
e=44.5776

m=exp(b)=26.5346
sigma=c/sqrt(2)=0.392982
D=41.255847401345nm
d=34.168nm
L0=26.0254nm

q=a=2.32832
rho=2/(Pi*(0.254841nm*d)**2)=0.0797181(1/nm)^2
Re^*=exp(-1/4)/(2*e)=0.00873534nm
M^*=(Re^*)*sqrt(rho)=0.00246637

After 732 iterations the fit converged.
final sum of squares of residuals : 5.29228
rel. change during last iteration : -4.48066e-10
```

Done

Address http://www.renyi.hu/cgi-bin/post-mwp?SAMPLENAME=CeO2-1&HEXAGONAL_CRYSTAL=n&la=0.3604&lc=1.0&bb=0.254841&C0=0.3040&N=256&FOURIER_FV

```
e=44.5776

m=exp(b)=26.5346
sigma=c/sqrt(2)=0.392982
D=41.255847401345nm
d=34.168nm
L0=26.0254nm

q=a=2.32832
rho=2/(Pi*(0.254841nm*d)**2)=0.0797181(1/nm)^2
Re^*=exp(-1/4)/(2*e)=0.00873534nm
M^*=(Re^*)*sqrt(rho)=0.00246637

After 732 iterations the fit converged.
final sum of squares of residuals : 5.29228
rel. change during last iteration : -4.48066e-10
Final set of parameters            Asymptotic Standard Error
=======================            ==========================

a               = 2.32832          +/- 0.1804       (7.748%)
b               = 3.27845          +/- 0.03636      (1.109%)
c               = 0.55576          +/- 0.01891      (3.403%)
d               = 11.089           +/- 248.3        (2239%)
e               = 44.5776          +/- 1999         (4485%)

For more information about correlation matrix and confidence intervals,
please read the file dragomir/CeO2-1.fourier.sol.


*** END OF FITTING ***
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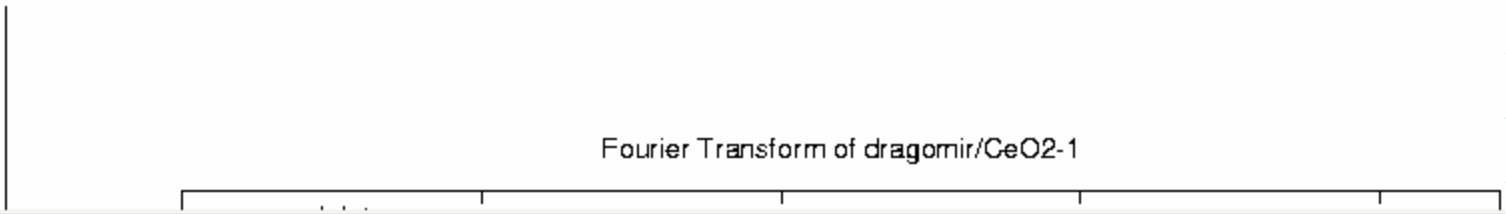

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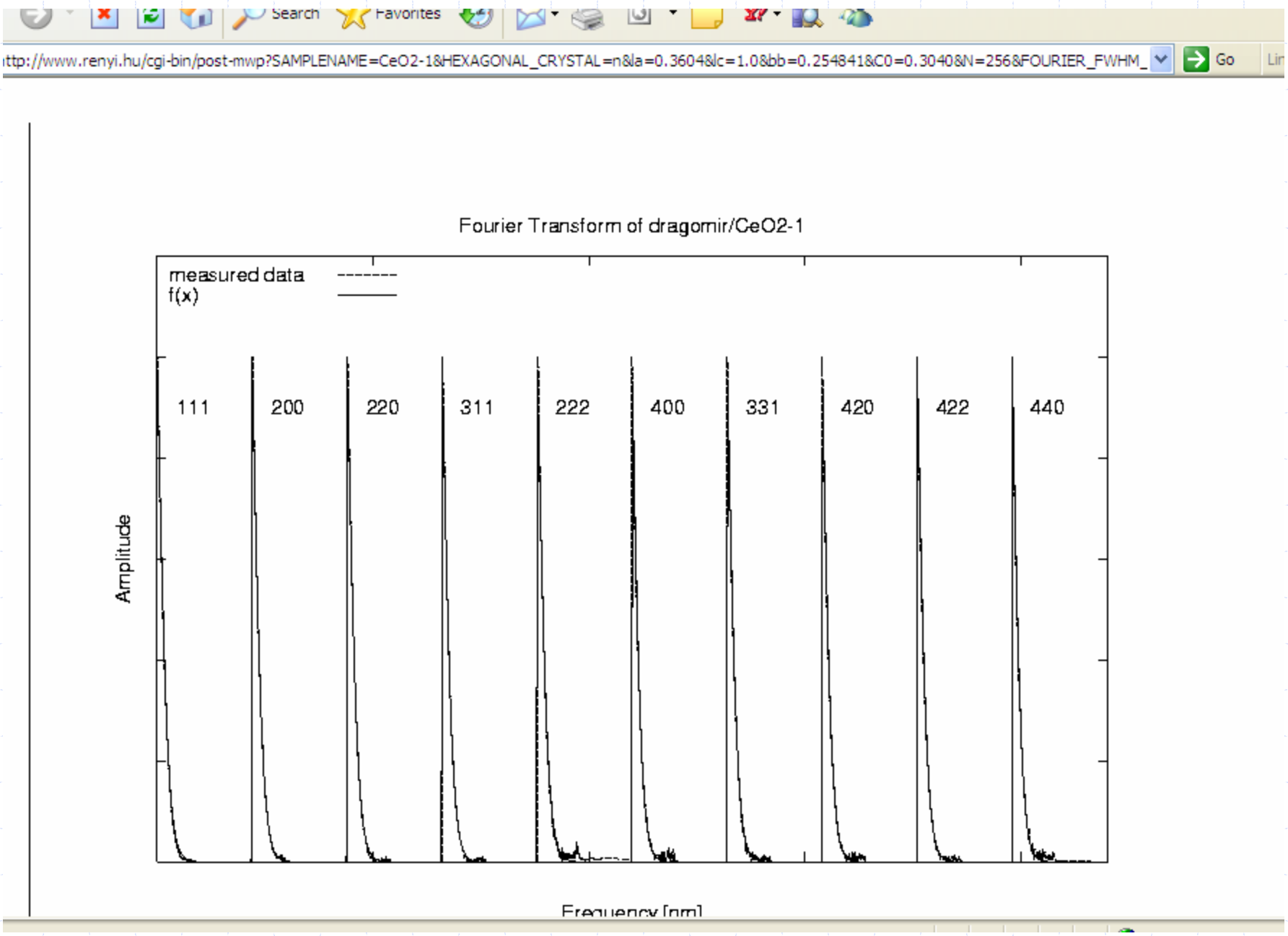
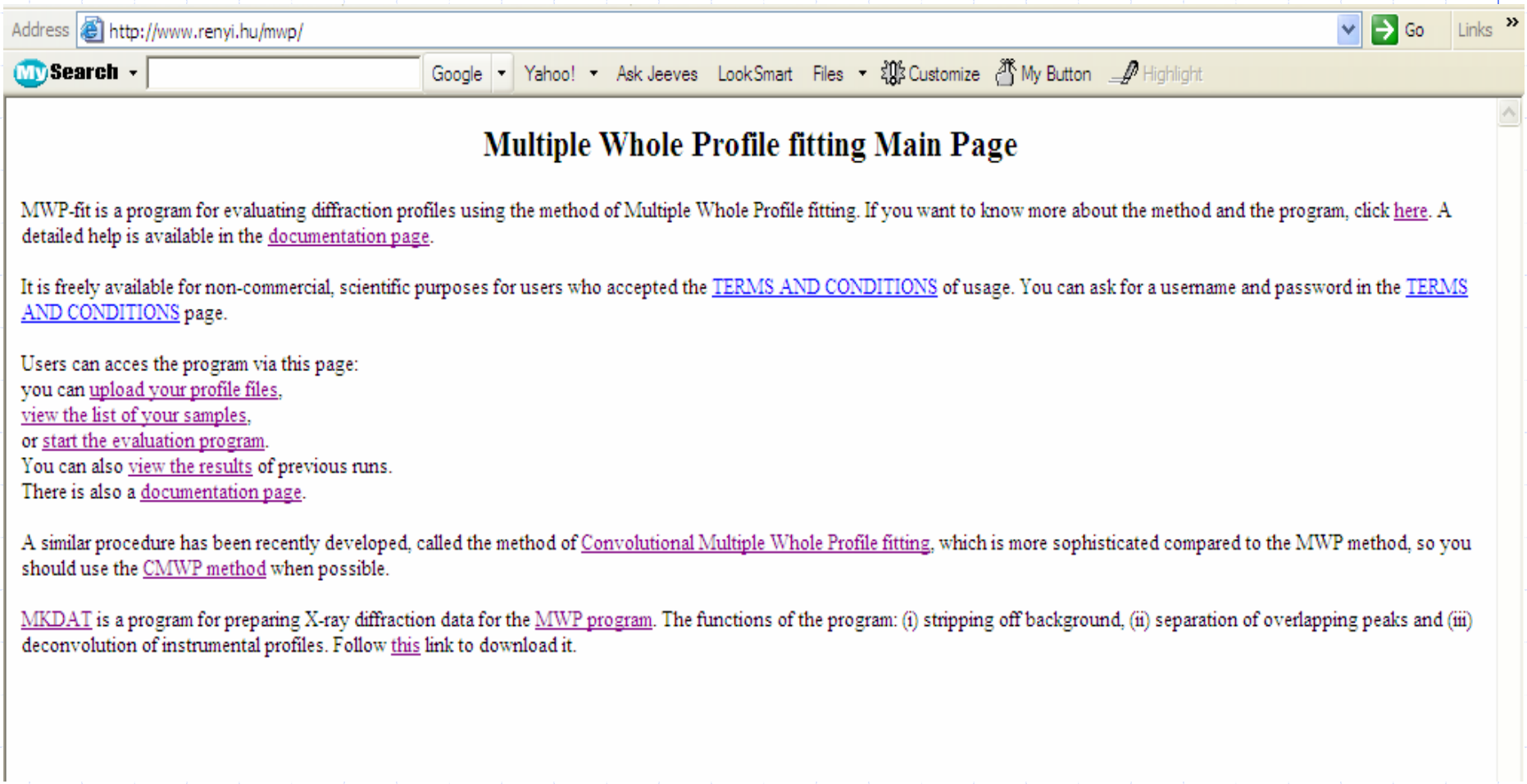
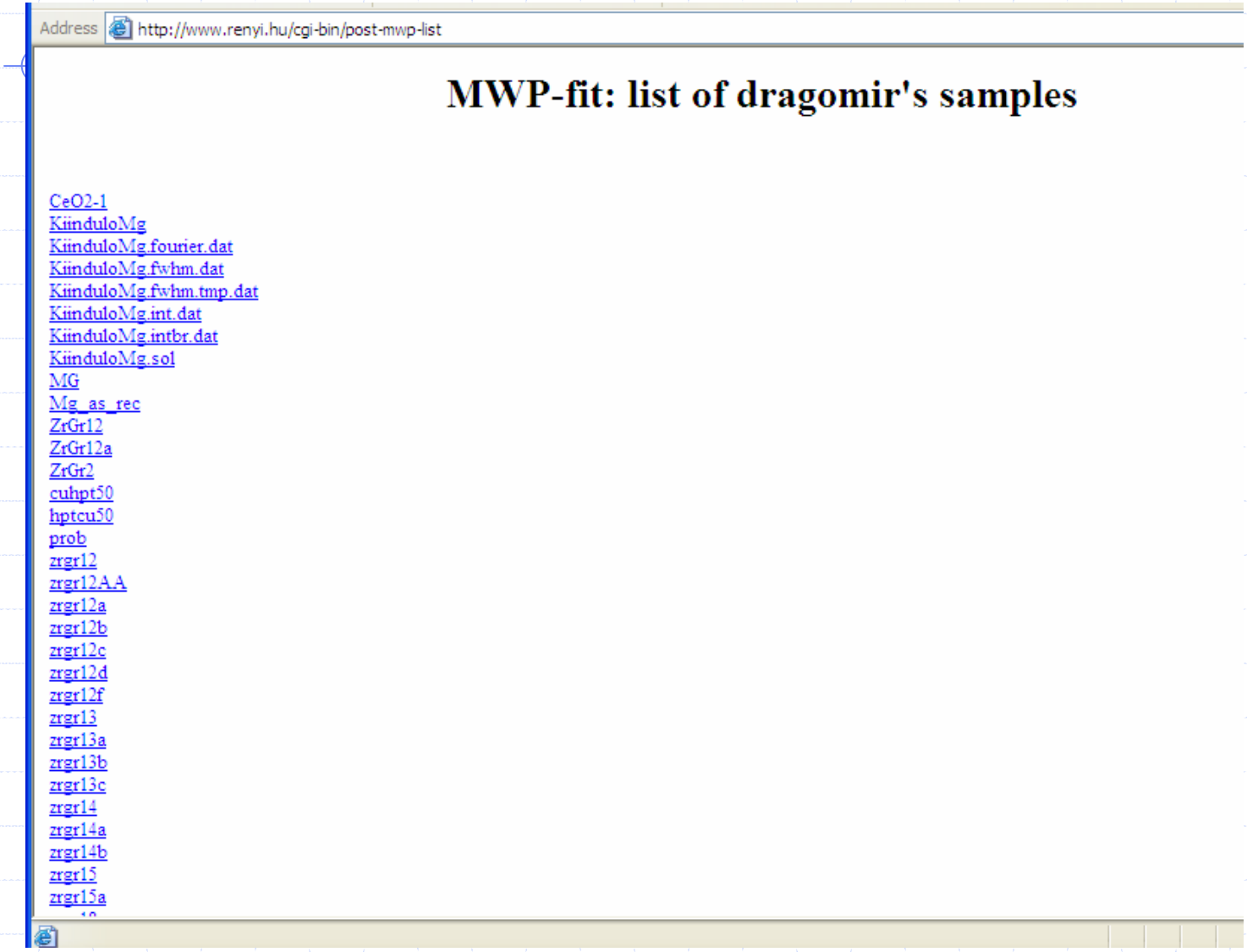

Search
Favorites
http://www.renyi.hu/cgi-bin/post-mwp?SAMPLENAME=CeO2-1&HEXAGONAL_CRYSTAL=n&la=0.3604&lc=1.0&bb=0.254841&C0=0.3040&N=256&FOURIER_FWHM_
Go
Fourier Transform of dragomir/CeO2-1
measured data
f(x)
111
200
220
311
222
400
331
420
422
440
Amplitude
Frequency [nm]

MWP procedure:



MWP output files:



MWP output files:

.../comp.sol

```
The solutions:
a=-2.42784
b=3.18557
c=0.596521
d=291.947
e=0.0219218

m=exp(b)=24.1811
sigma=c/sqrt(2)=0.421804
D=41.4270197228895nm
d=33.8048nm
L0=25.1511nm

q=a=-2.42784
rho=2/(Pi*(0.382545nm*d)**2)=5.10396e-05(1/nm)^2
Re^*=exp(-1/4)/(2*e)=17.7632nm
M^*=(Re^*)*sqrt(rho)=0.126904

After 17 iterations the fit converged.
final sum of squares of residuals : 1.24613
rel. change during last iteration : 0
Final set of parameters            Asymptotic Standard Error
=======================            ==========================

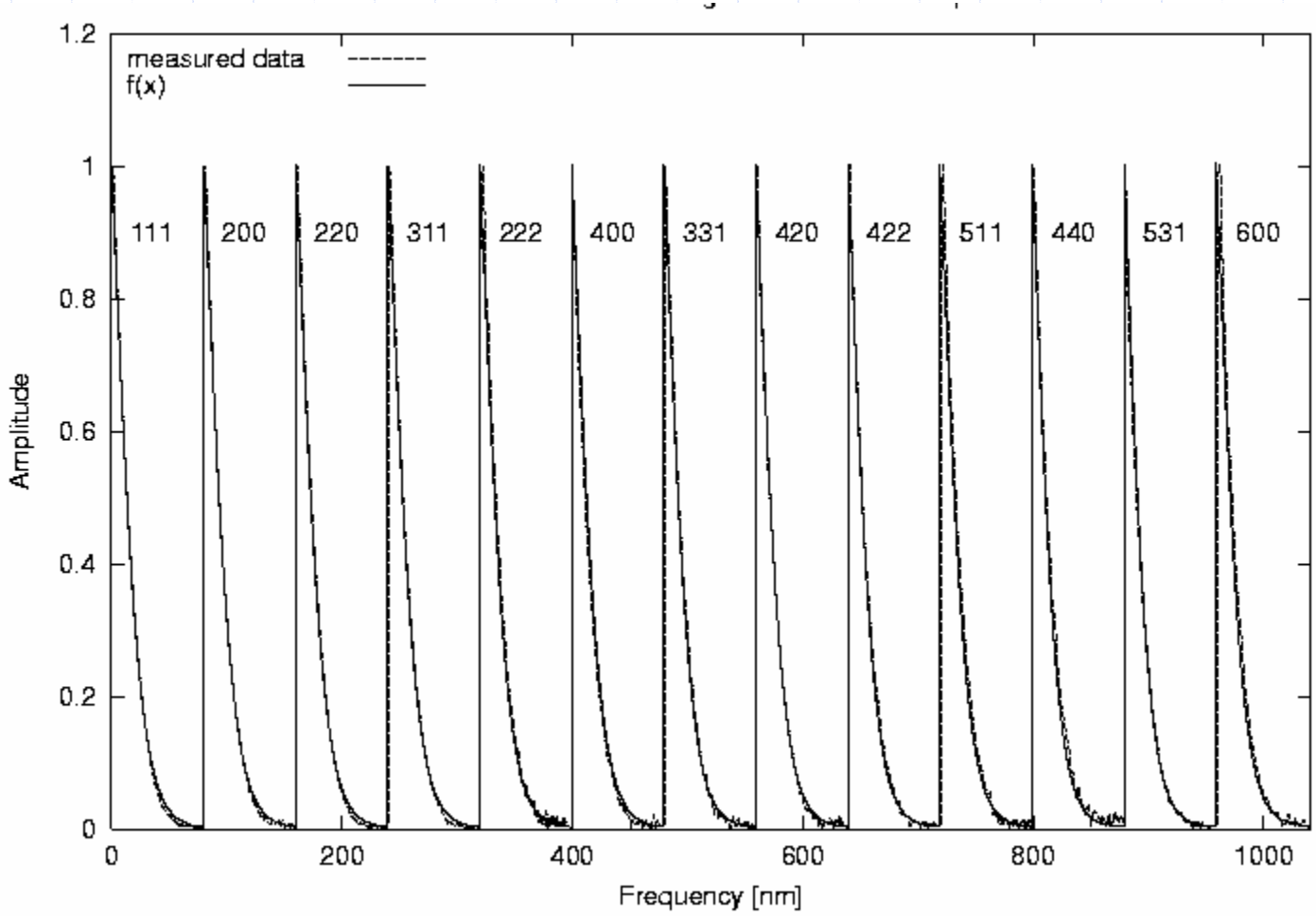
a                 = -2.42784         +/- 0.3039       (12.52%)
b                 = 3.18557          +/- 0.009763     (0.3065%)
c                 = 0.596521         +/- 0.00484      (0.8114%)
d                 = 291.947          +/- 7.676        (2.629%)

For more information about correlation matrix and confidence intervals,
please read the file gatech/CeO2/S1/comp.fourier.sol.


*** END OF FITTING ***
```

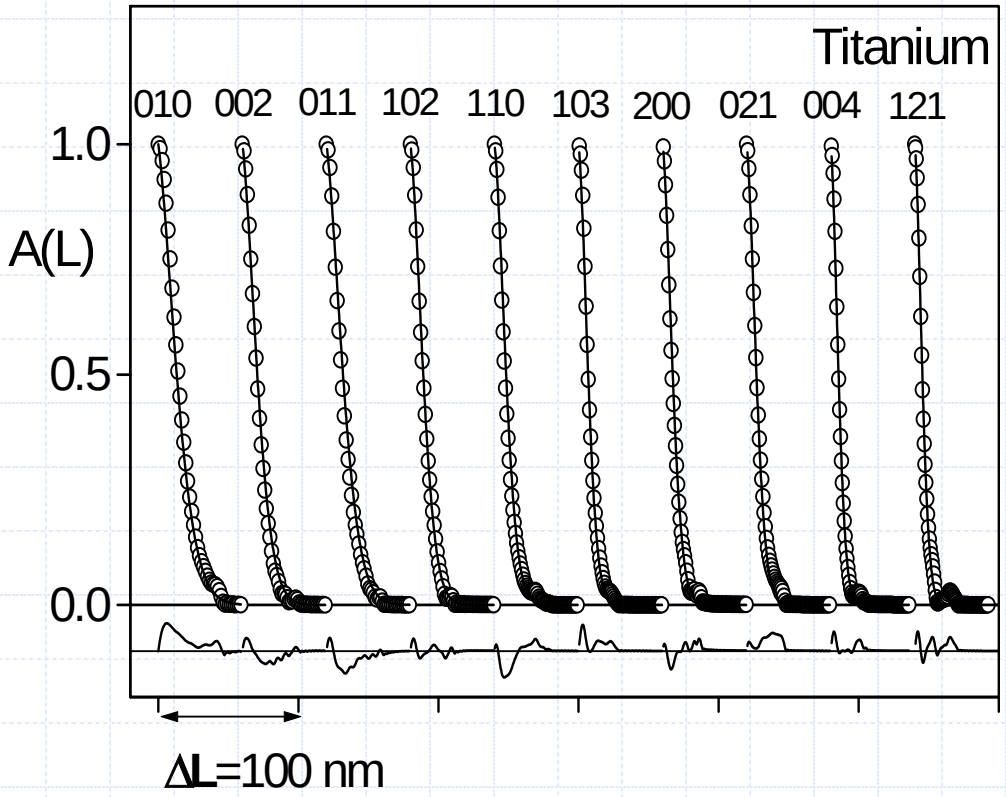
MWP output files:

.../S2MWP.Fourier.gif



MWP output files:

.../comp.fourier.th.dat & .../comp.fourier.m.dat



MWP References:

• Ungár, T., Gubicza, J., Ribárik, G. and Borbély, A.: Crystallite size-distribution and dislocation structure determined by diffraction profile analysis: principles and practical application to cubic and hexagonal crystals, J. Appl. Cryst. 34, 298-310, 2001

• Ribárik, G., Ungár, T. and Gubicza, J.: MWP-fit: a program for Multiple Whole Profile fitting of diffraction peak profiles by ab-initio theoretical functions, J. Appl. Cryst. 34, 669-676, 2001

CMWP - fit:

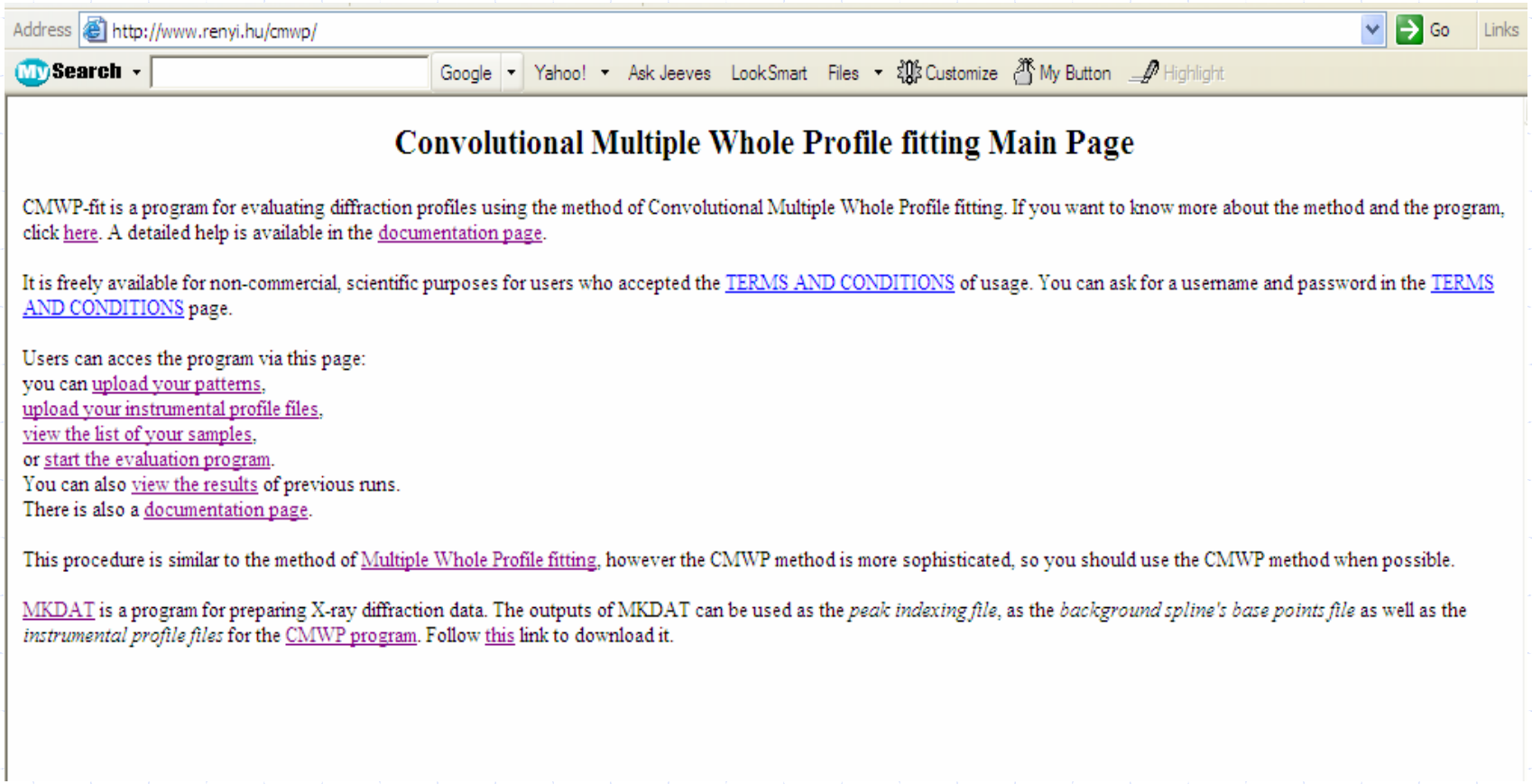
In order to avoid the uncertainties and the errors introduced by correction procedures in CMWP the measured pattern is directly fitted by the background plus the theoretical function of the pattern convoluted with the instrumental effect.

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

$$I^{hkl} = I_{instr.}^{hkl} * I_{size}^{hkl} * I_{disl.}^{hkl}$$

$(\boldsymbol{I}^{(meas)} - \boldsymbol{I}^{(th)})^2 = \min.$ Nonlinear least squares method

CMWP - fit:


Address
http://www.renyi.hu/cmwp/
Go
Links
MySearch
Google
Yahoo!
Ask Jeeves
LookSmart
Files
Customize
My Button
Highlight
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. A detailed help is available in the documentation page.
It is freely available for non-commercial, scientific purposes for users who accepted the TERMS AND CONDITIONS of usage. You can ask for a username and password in the TERMS AND CONDITIONS page.
Users can acces the program via this page:
you can upload your patterns,
upload your instrumental profile files,
view the list of your samples,
or start the evaluation program.
You can also view the results of previous runs.
There is also a documentation page.
This procedure is similar to the method of Multiple Whole Profile fitting, however the CMWP method is more sophisticated, so you should use the CMWP method when possible.
MKDAT 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 link to download it.

CMWP - fit – input files format:

1) measurement file -This is a two-column ASCII file. The first column should contain *Two Theta* and the second column contains the intensity values.

2) index file

2θ	I_{max}	*hkl*
28.5943	5371.97	111
33.1285	1425.45	200
47.5281	2708.36	220
56.3854	2023.35	311
59.1299	381.12	222
69.4534	327.03	400
76.7388	703.47	331
79.1124	432.52	420
88.4608	577.85	422
95.4312	463.26	511
107.2531	117.04	440
114.7488	551.35	531
117.3341	250.49	600

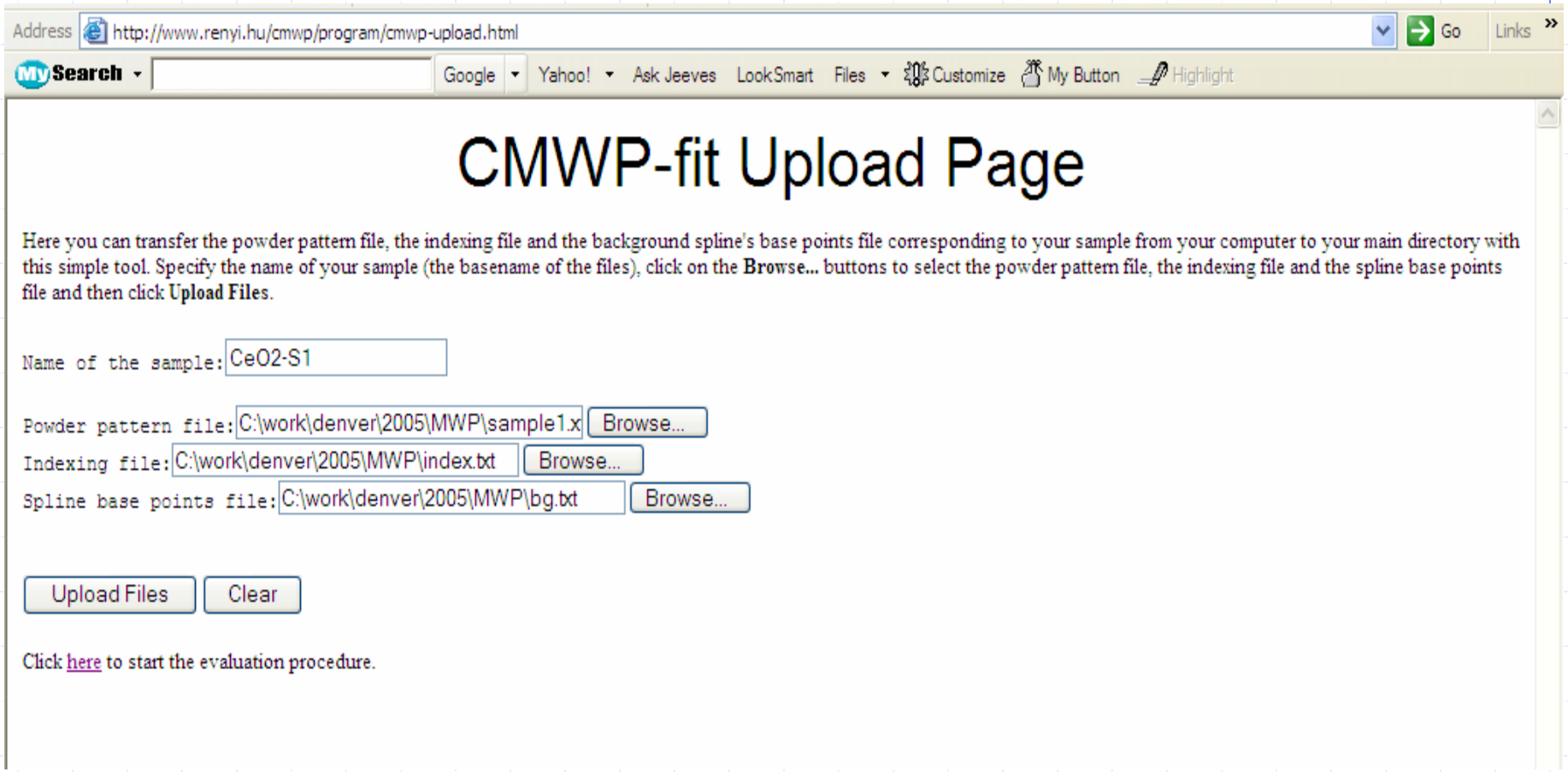
3) background file

2θ	I
28.5943	118
33.1285	106
47.5281	95
56.3854	94
59.1299	94
69.4534	91
76.7388	89
79.1124	88
88.4608	88
95.4312	89
107.2531	93
114.7488	99
117.3341	100

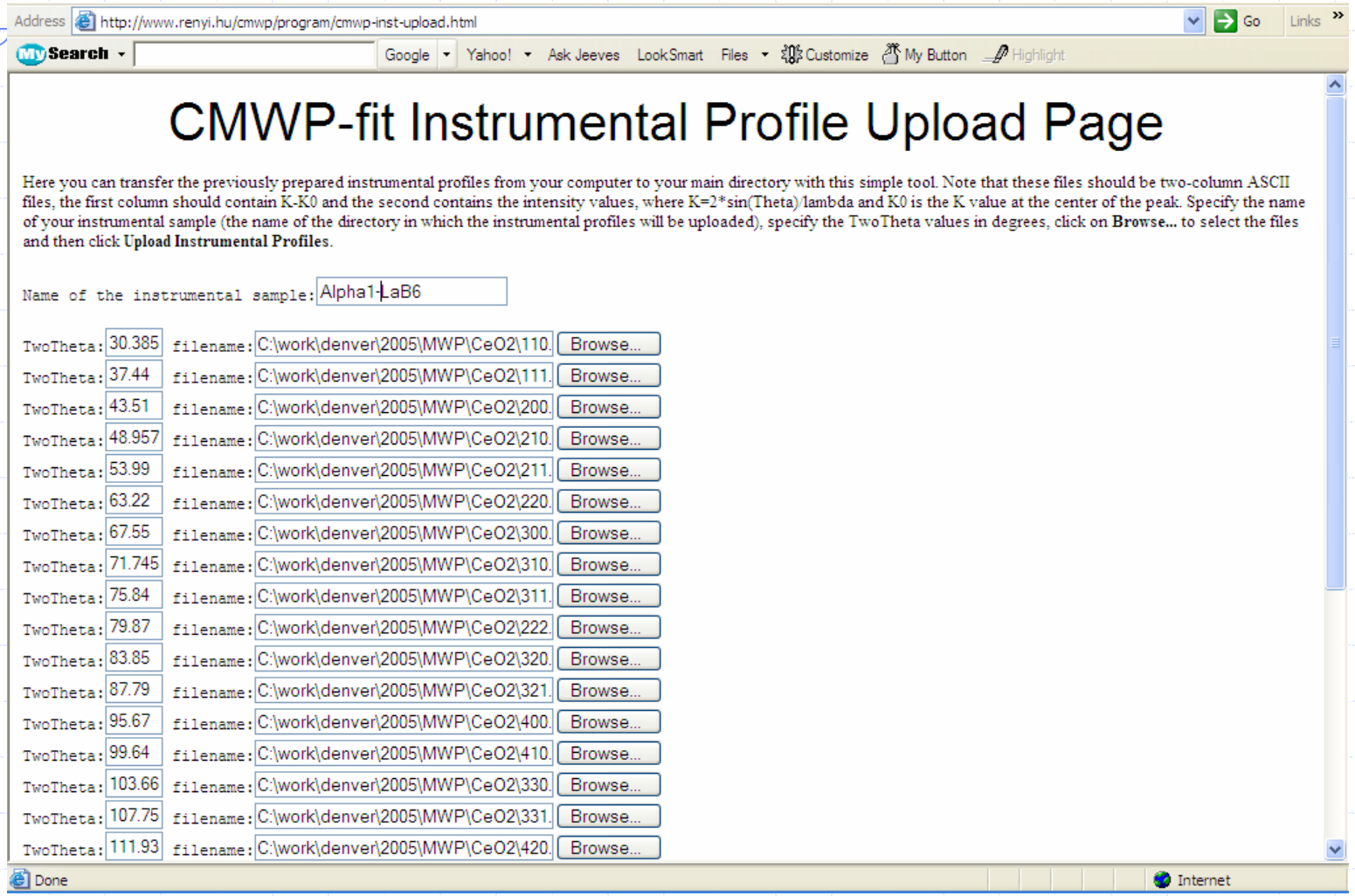
4) instrumental profiles file

d*	I/I_{max}
-0.00394	0.203045
-0.0035	0.26193
-0.00306	0.330089
-0.00262	0.411947
-0.00219	0.515034
-0.00175	0.643361
-0.00131	0.757919
-0.00087	0.879088
-0.00044	0.978878
0	1
0.00044	0.96513
0.00088	0.867677
0.00131	0.730476
0.00175	0.583633
0.00219	0.437497
0.00263	0.313898
0.00306	0.219254
0.0035	0.148984
0.00394	0.0989103

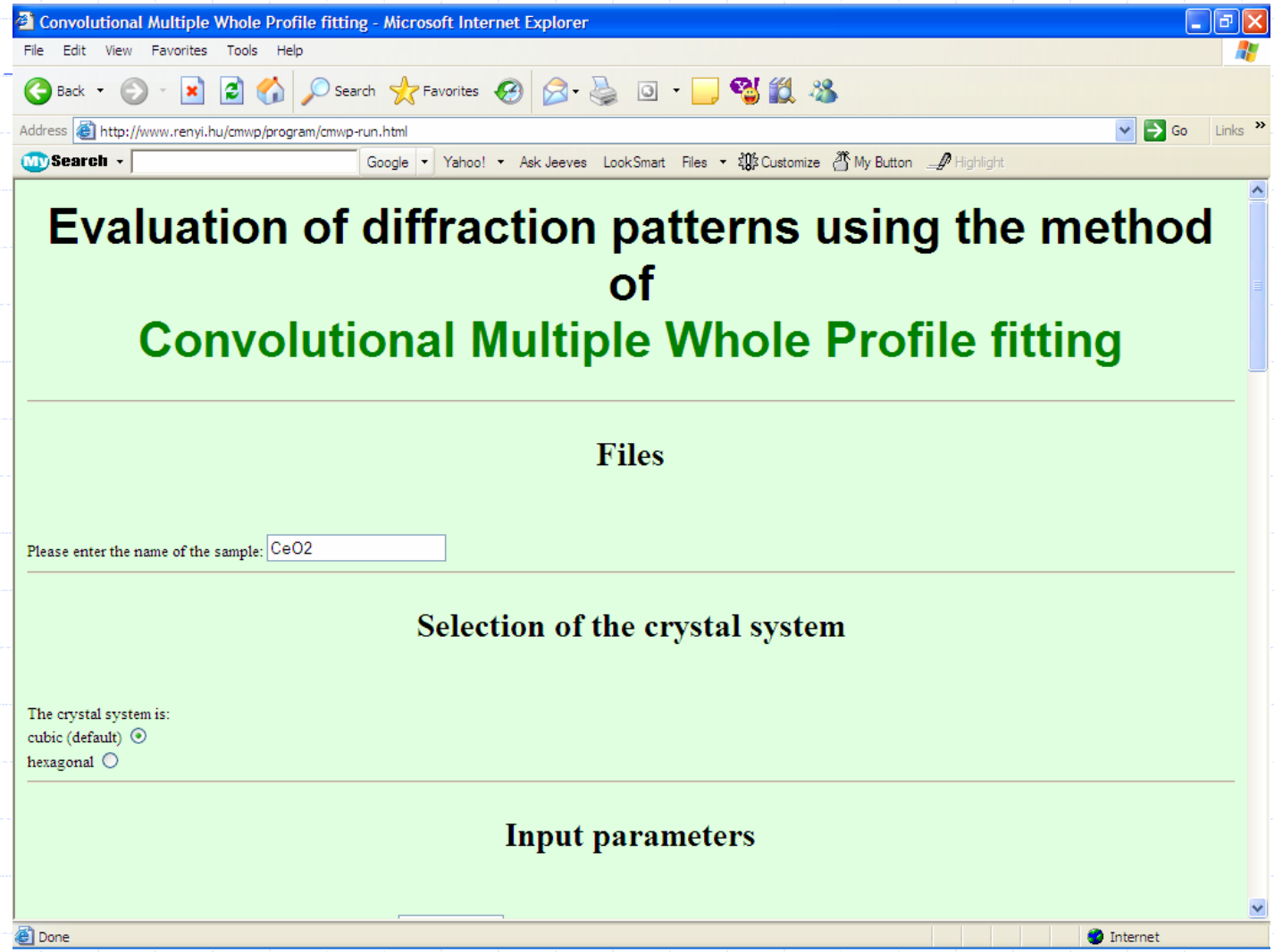
CMWP - fit - upload:



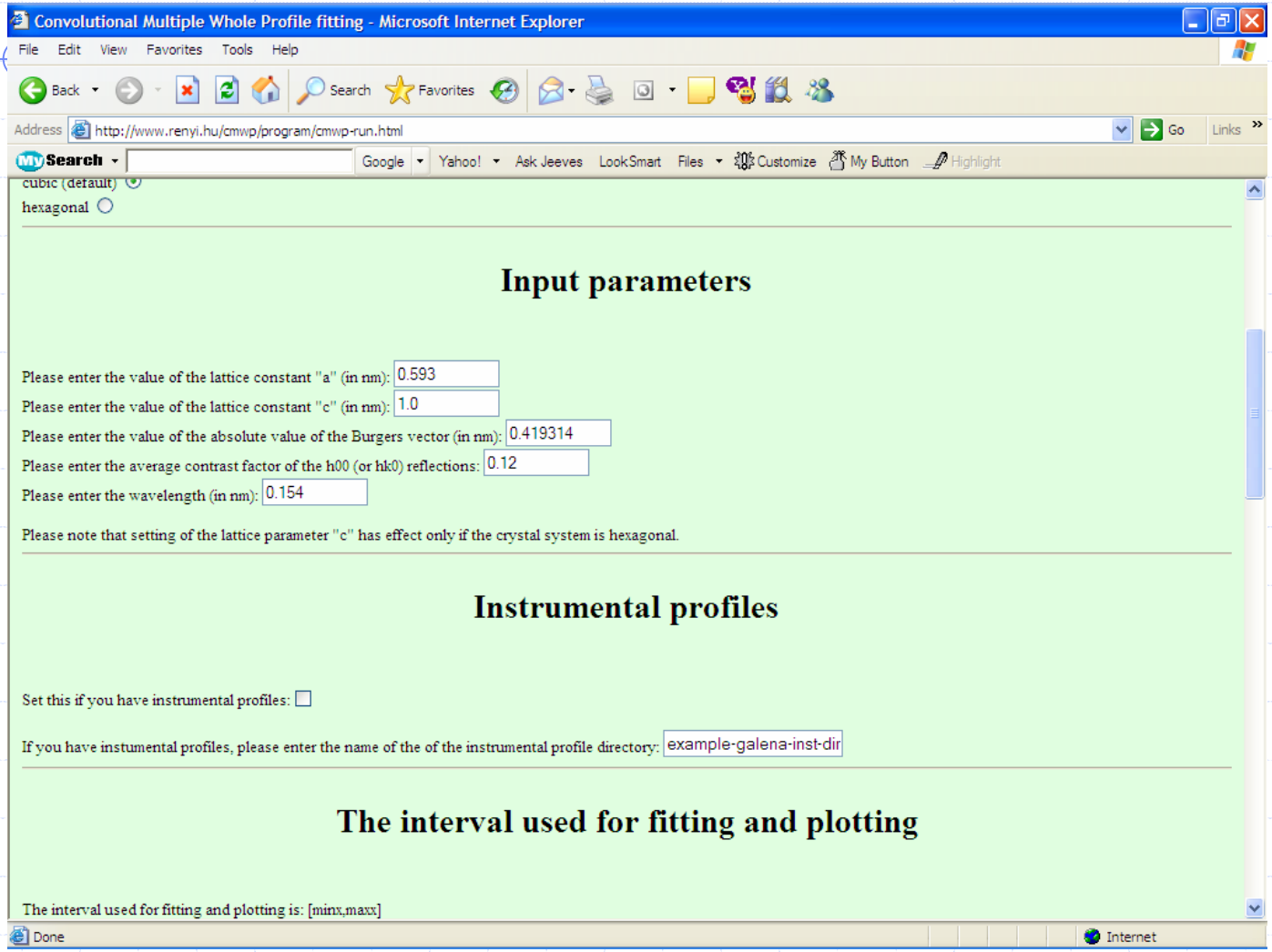
CMWP - fit:


Address http://www.renyi.hu/cmwp/program/cmwp-inst-upload.html
Go
Links
MySearch
Google
Yahoo!
Ask Jeeves
LookSmart
Files
Customize
My Button
Highlight
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: Alpha1-LaB6
TwoTheta: 30.385 filename: C:\work\denver\2005\MWP\CeO2\110. Browse...
TwoTheta: 37.44 filename: C:\work\denver\2005\MWP\CeO2\111. Browse...
TwoTheta: 43.51 filename: C:\work\denver\2005\MWP\CeO2\200. Browse...
TwoTheta: 48.957 filename: C:\work\denver\2005\MWP\CeO2\210. Browse...
TwoTheta: 53.99 filename: C:\work\denver\2005\MWP\CeO2\211. Browse...
TwoTheta: 63.22 filename: C:\work\denver\2005\MWP\CeO2\220. Browse...
TwoTheta: 67.55 filename: C:\work\denver\2005\MWP\CeO2\300. Browse...
TwoTheta: 71.745 filename: C:\work\denver\2005\MWP\CeO2\310. Browse...
TwoTheta: 75.84 filename: C:\work\denver\2005\MWP\CeO2\311. Browse...
TwoTheta: 79.87 filename: C:\work\denver\2005\MWP\CeO2\222. Browse...
TwoTheta: 83.85 filename: C:\work\denver\2005\MWP\CeO2\320. Browse...
TwoTheta: 87.79 filename: C:\work\denver\2005\MWP\CeO2\321. Browse...
TwoTheta: 95.67 filename: C:\work\denver\2005\MWP\CeO2\400. Browse...
TwoTheta: 99.64 filename: C:\work\denver\2005\MWP\CeO2\410. Browse...
TwoTheta: 103.66 filename: C:\work\denver\2005\MWP\CeO2\330. Browse...
TwoTheta: 107.75 filename: C:\work\denver\2005\MWP\CeO2\331. Browse...
TwoTheta: 111.93 filename: C:\work\denver\2005\MWP\CeO2\420. Browse...
Done
Internet

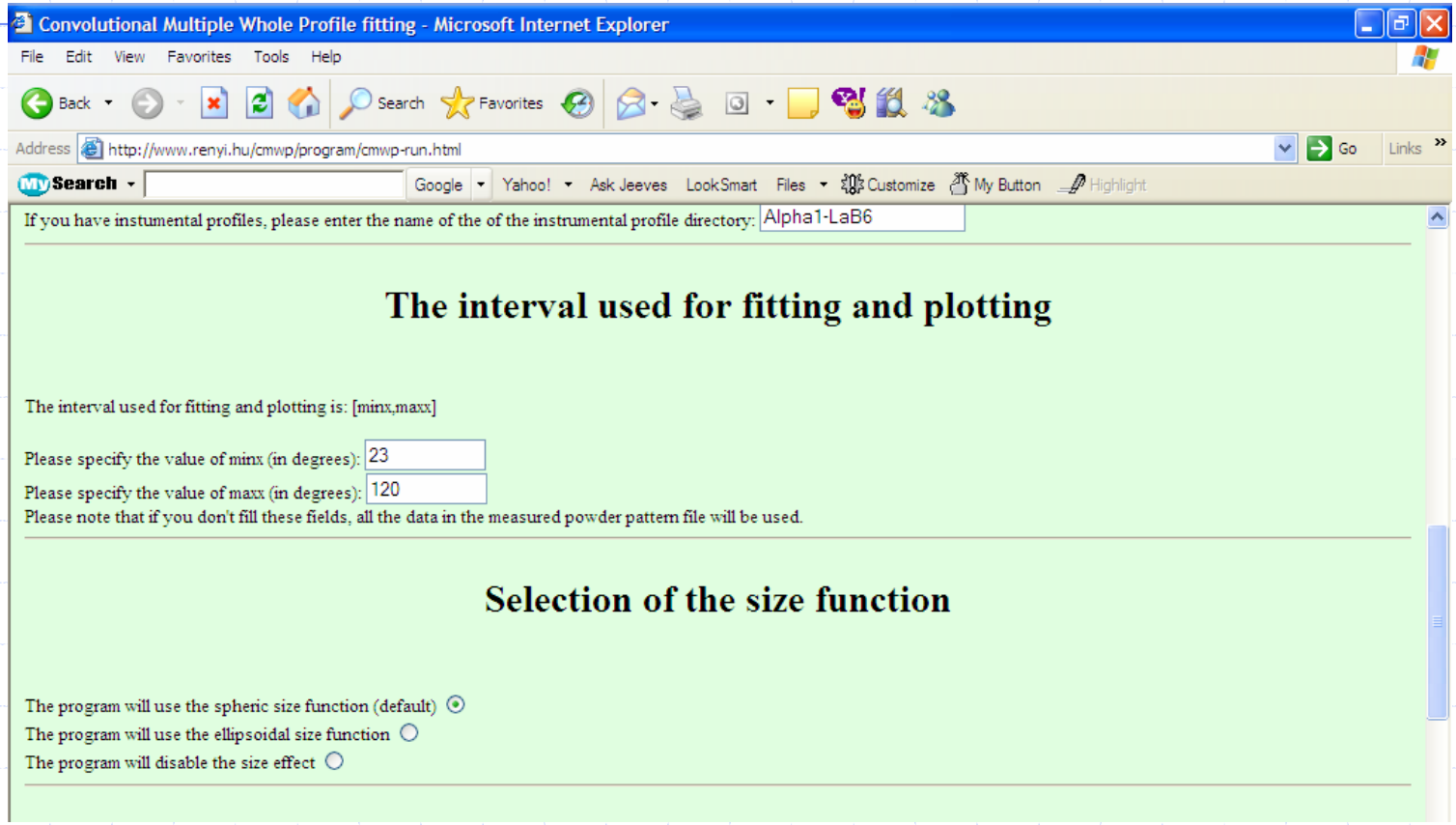
CMWP - fit – crystal system:



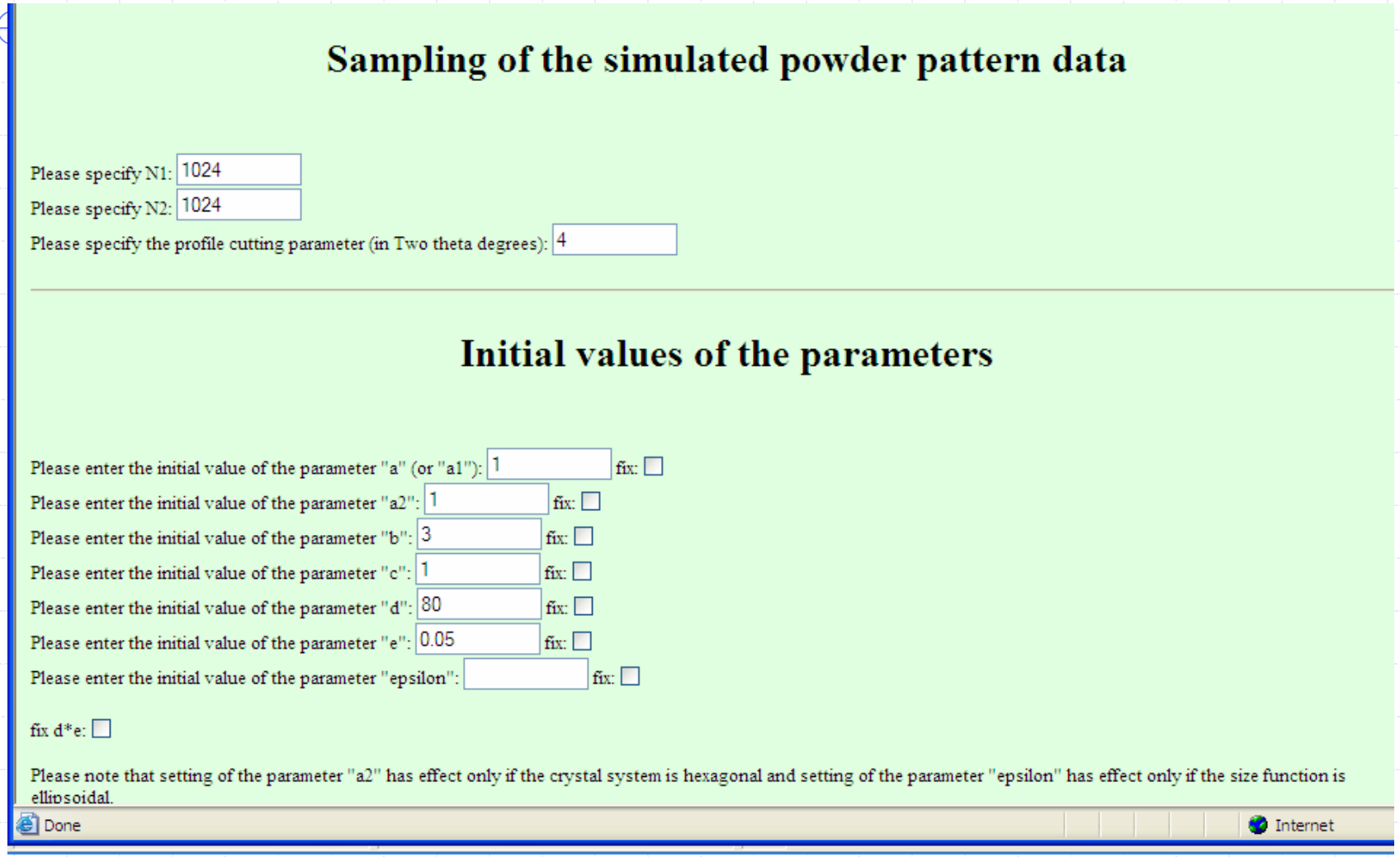
CMWP - fit – input parameters:



CMWP - fit - fitting interval:



CMWP - fit – settings/initial values:



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

Initial values of the parameters

Please enter the initial value of the parameter "a" (or "a1"): 1 fix: ☐

Please enter the initial value of the parameter "a2": 1 fix: ☐

Please enter the initial value of the parameter "b": 3 fix: ☐

Please enter the initial value of the parameter "c": 1 fix: ☐

Please enter the initial value of the parameter "d": 80 fix: ☐

Please enter the initial value of the parameter "e": 0.05 fix: ☐

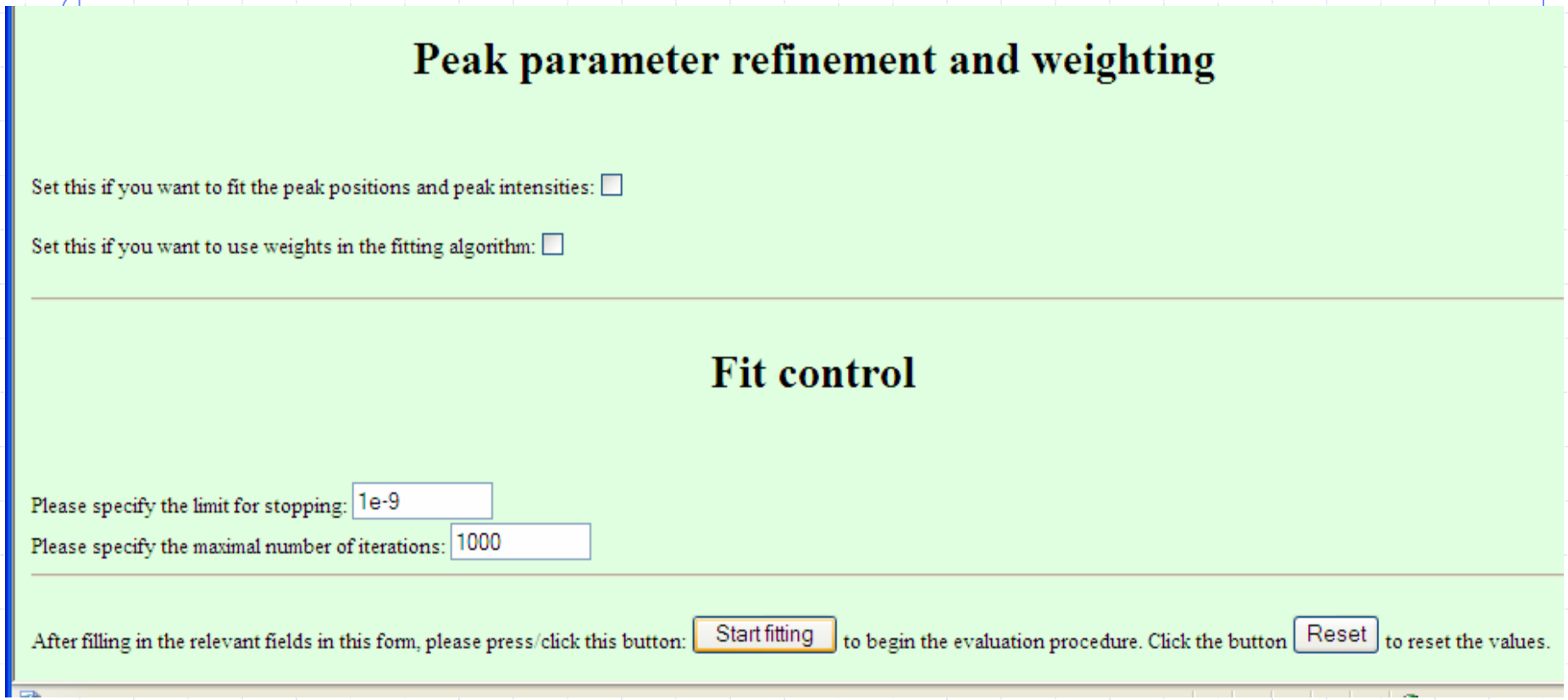
Please enter the initial value of the parameter "epsilon": fix: ☐

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.

Done Internet

CMWP - fit – refinement/fit control:



CMWP - fit –fitting.....:

Address http://www.renyi.hu/cgi-bin/post-cmwp?SAMPLENAME=CeO2-S1&HEXAGONAL_CRYSTAL=n&la=0.541&lc=0&bb=0.32&C0=0.3&wavelength=0.154&have_inst_profi

Convolutional Multiple Whole Profile fitting

Connecting the server for evaluation .....
The evaluation is started.
Please wait until this page loads to get the results.

Note that the results will be stored in http://www.renyi.hu/cmwp/results/dragomir/CeO2-S1-2005-08-02-07-03-55.

The fitting is done, evaluate's output follows:

```
This is CMWP-fit, a program for evaluation of diffraction patterns
using the method of Convolutional Multiple Whole Profile fitting.
Copyright (C) Gábor Ribárik, 1998-2004. All rights reserved.
For permission to use, copy, modify this program or any of its components,
ask the authors. Redistributing this software is not allowed.

Date: 2005-08-02 07:04:00

Data file: dragomir/CeO2-S1/CeO2-S1.dat

The value of the lattice constant (in nm): 0.541
The absolute value of the Burgers vector (in nm): 0.32
The average contrast factor of the h00 reflections: 0.3
The wavelength is: 0.154 nm.
The definition of the Size Function:
sf_spheric(k,b,c)=Int(0.5*x*erfc((log(x)-b)/c)*(sin(Pi*k*x)/(Pi*k))**2,x=0..Inf)

Found peak at 28.5943, intensity: 5371.97.
hkl=111
H2=0.333333
g^2=10.2501
```

Address http://www.renyi.hu/cgi-bin/post-cmwp?SAMPLENAME=CeO2-S1&HEXAGONAL_CRYSTAL=n&la=0.541&lc=0&bb=0.32&C0=0.3&wav

```
*** FIT ***
Calculating a,b,c,d,e to fit the measured powder diffraction pattern
with the corresponding theoretical function.
The initial values of the parameters:
C0=0.3
a = 1
b = 3
c = 1
d = 80
e = 0.05
epsilon = 1.0 # FIXED

The solutions:
a=0.288428
b=3.17207
c=0.59686
d=13.858
e=18.7037

m=exp(b)=23.8568
sigma=c/sqrt(2)=0.422044
D=gnuplot>nm
d=33.3751nm
L0=24.8264nm

q=a=0.288428
rho=2/(Pi*(0.32nm*d)**2)=0.0323727(1/nm)^2
Re^*=exp(-1/4)/(2*e)=0.0208194nm
M^*=(Re^*)*sqrt(rho)=0.00374591

After 334 iterations the fit converged.
final sum of squares of residuals : 5.84622e+06
rel. change during last iteration : -9.99023e-10
Final set of parameters            Asymptotic Standard Error
=======================            ==========================

a               = 0.288428         +/- 0.1331       (46.15%)
```

Address http://www.renyi.hu/cgi-bin/post-cmwp?SAMPLENAME=CeO2-S1&HEXAGONAL_CRYSTAL=n&la=0.541&lc=0&bb=0.32&C0=0.3&wavelength=0.

```
The solutions:
a=0.288428
b=3.17207
c=0.59686
d=13.858
e=18.7037

m=exp(b)=23.8568
sigma=c/sqrt(2)=0.422044
D=gnuplot>nm
d=33.3751nm
L0=24.8264nm

q=a=0.288428
rho=2/(Pi*(0.32nm*d)**2)=0.0323727(1/nm)^2
Re^*=exp(-1/4)/(2*e)=0.0208194nm
M^*=(Re^*)*sqrt(rho)=0.00374591

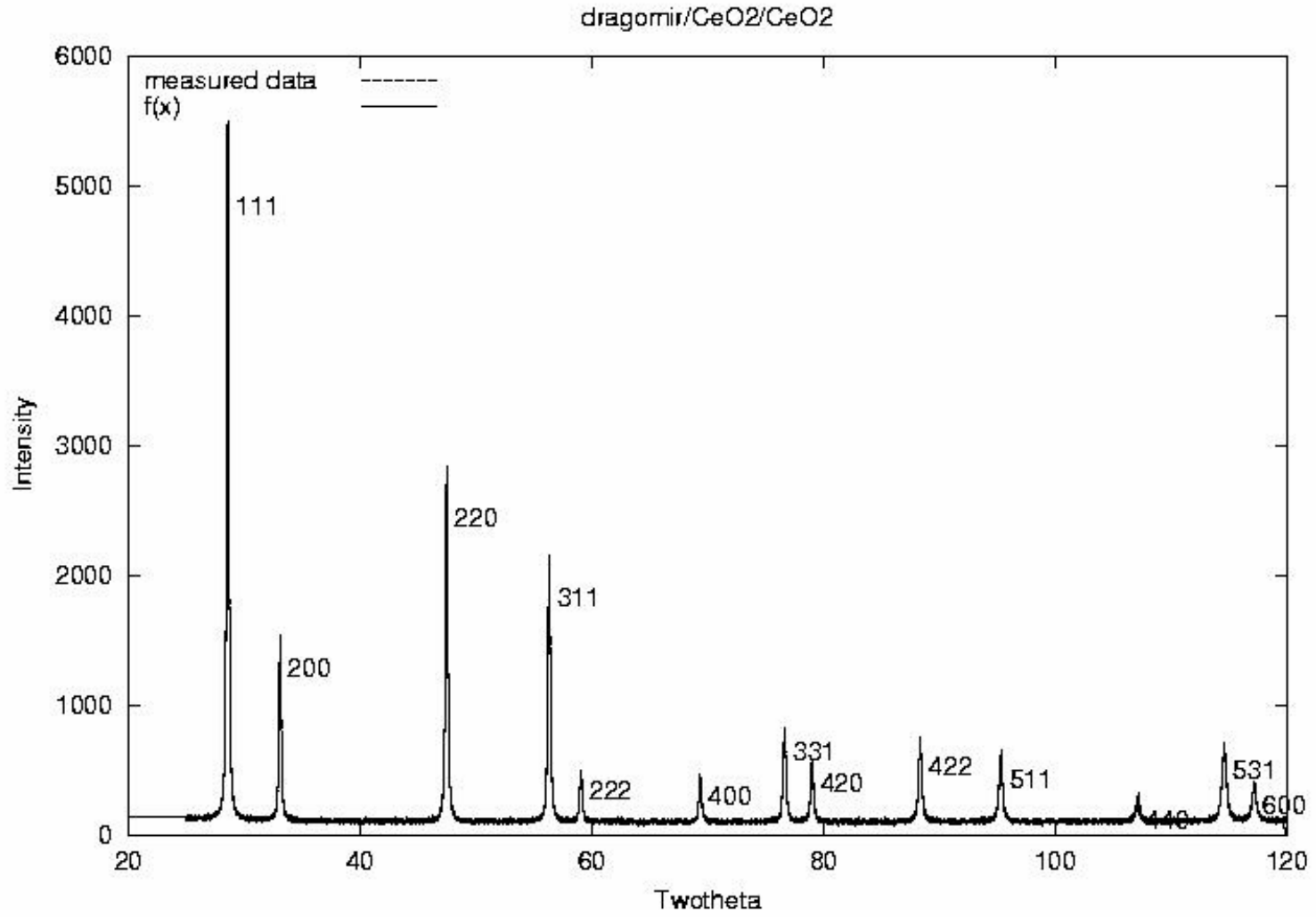
After 334 iterations the fit converged.
final sum of squares of residuals : 5.84622e+06
rel. change during last iteration : -9.99023e-10
Final set of parameters            Asymptotic Standard Error
=======================            ==========================

a               = 0.288428         +/- 0.1331       (46.15%)
b               = 3.17207          +/- 0.004389     (0.1384%)
c               = 0.59686          +/- 0.001963     (0.3288%)
d               = 13.858           +/- 48.26        (348.3%)
e               = 18.7037          +/- 130.5        (697.9%)

For more information about correlation matrix and confidence intervals,
please read the file dragomir/CeO2-S1/CeO2-S1.int.sol.


*** END OF FITTING ***
```

CMWP procedure - output:



CMWP References:

G. Ribárik, J. Gubicza, T. Ungár: Correlation between strength and microstructure of ball milled Al-Mg alloys determined by X-ray diffraction, Mater. Sci. Eng. A 387-389 (2004) 343-347.

Common features:

- Both procedures uses the ***whole X-ray diffraction pattern.***
- The diffraction profiles are fitted by well established size and strain ***physical*** profile functions.

Differences:

- MWP fits all at once the Fourier transforms or the inverse Fourier transforms of the measured profiles after they were corrected for instrumental effect and background.
- CMWP fits the whole pattern with the convolution of the physical profiles and the instrumental effect plus the background.

Microstructural parameters obtained from MWP and CMWP procedures:

Cubic crystal systems:

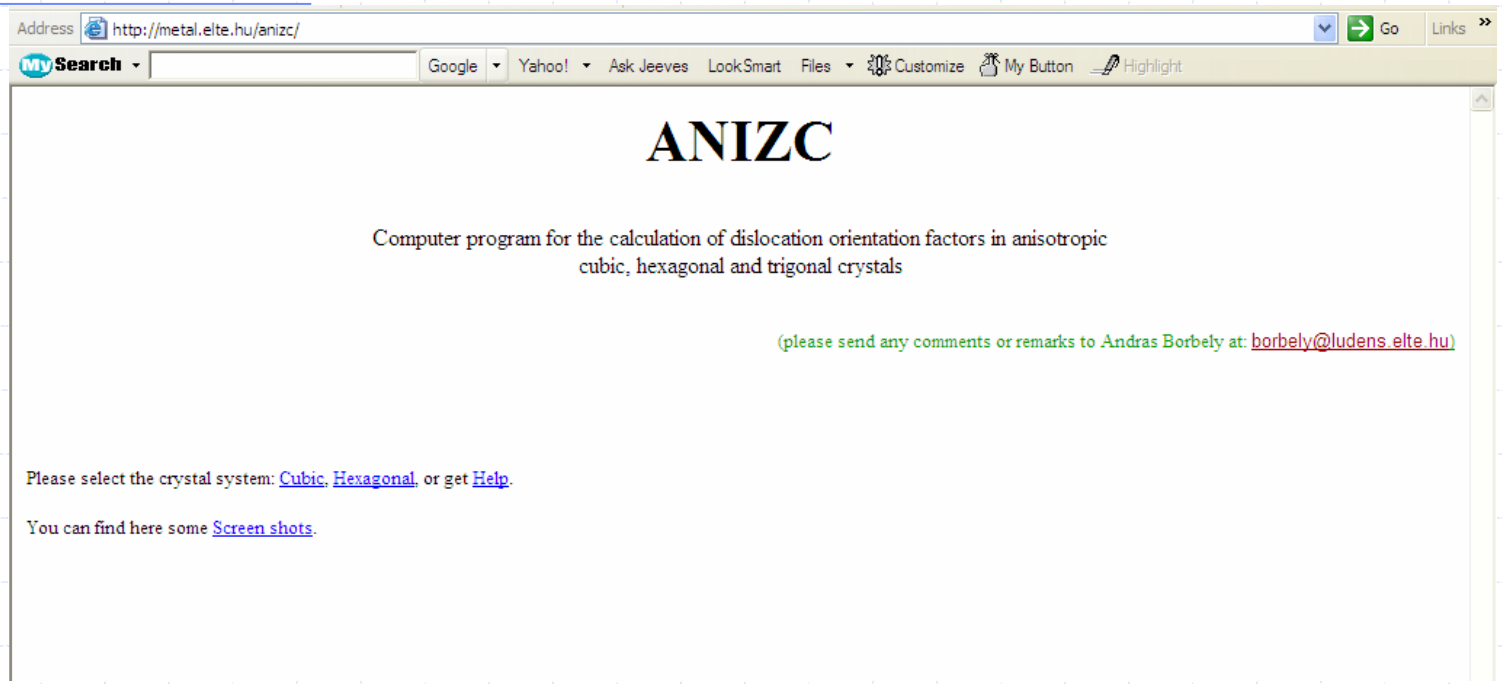
m, σ:	$\rightarrow$	size profile
q:	$\rightarrow$	strain anisotropy
ρ^*, M	$\rightarrow$	strain profile

Hexagonal crystal systems:

m, σ	$\rightarrow$	size profile
q_1 and q_2	$\rightarrow$	strain anisotropy
ρ^*, M	$\rightarrow$	strain profile

Theoretical individual and average contrast factor of dislocations

Or can be calculated using ANIZC software at http://metal.elte.hu/anizc/

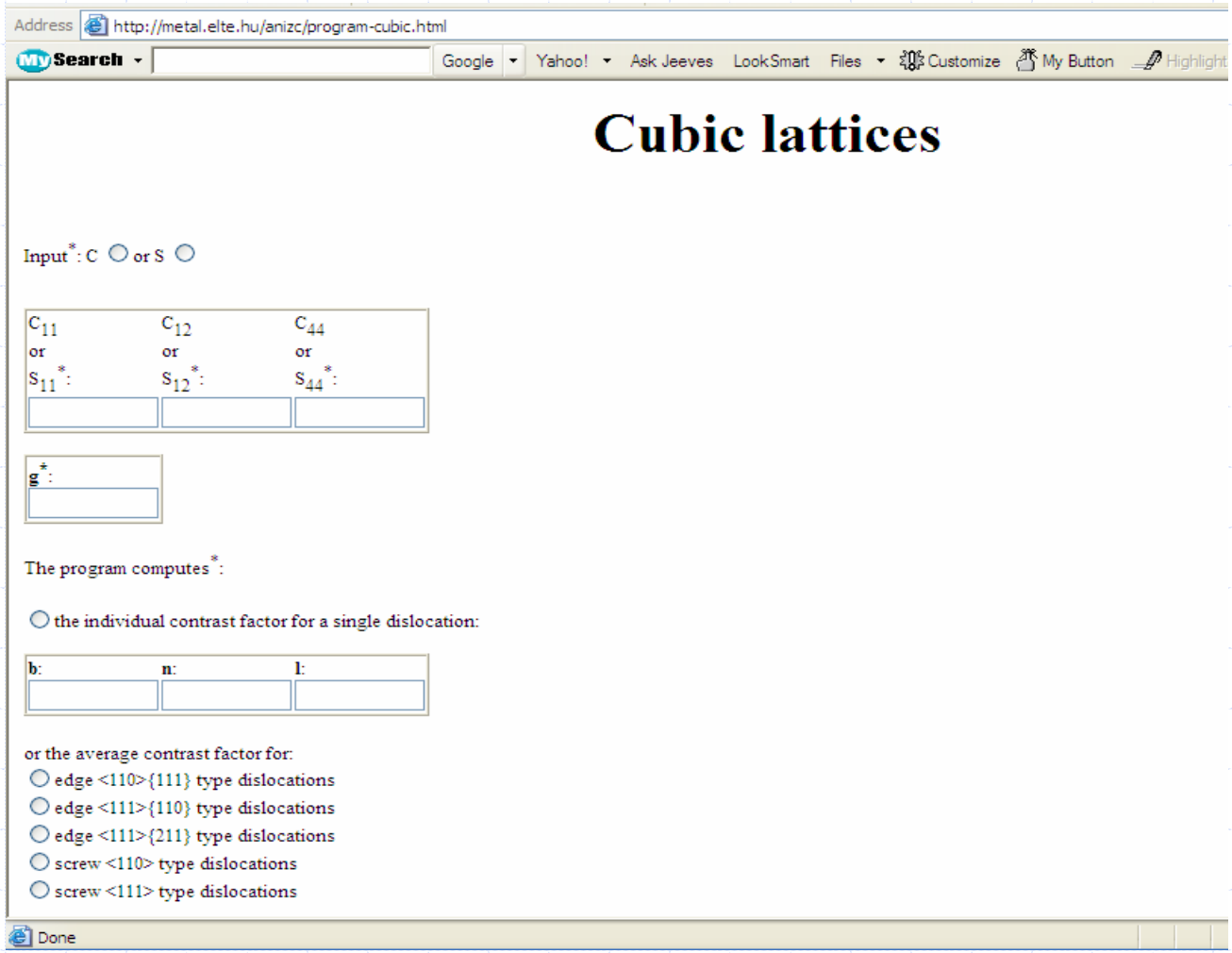


AnizC program was developed by Dr. Andras Borbely (Department of General Physics at Eotvos Science University in Budapest, Hungary).

Reference: Borbély A., Dragomir-Cernatescu I., Ribárik G. and Ungár T.: "Computer program ANIZC for the calculation of diffraction contrast factors of dislocations in elastically anisotropic cubic, hexagonal and trigonal crystals", in J. Appl. Cryst., 36, 160-162, (2003)

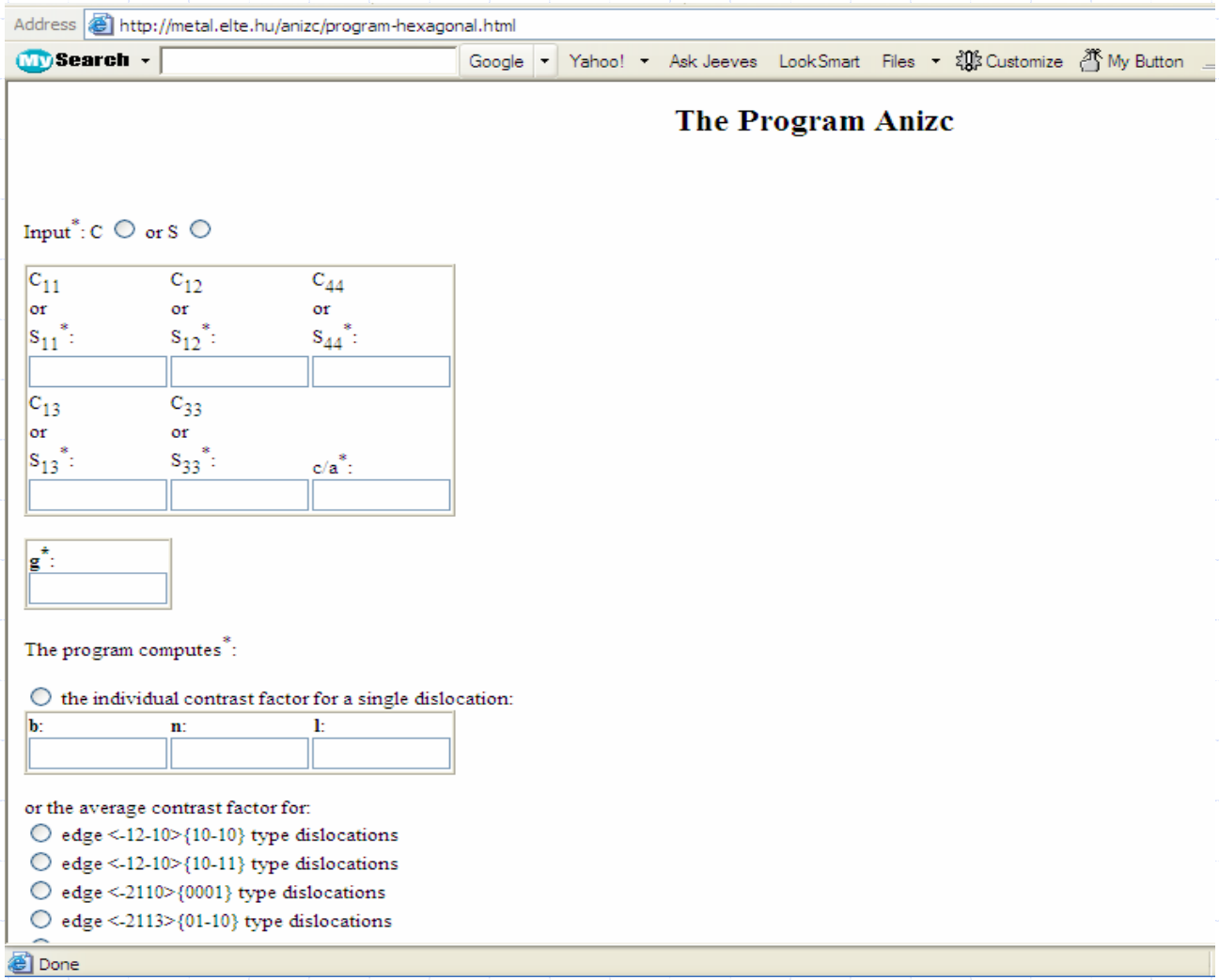
Theoretical individual and average contrast factor of dislocations

AnizC for cubic crystals



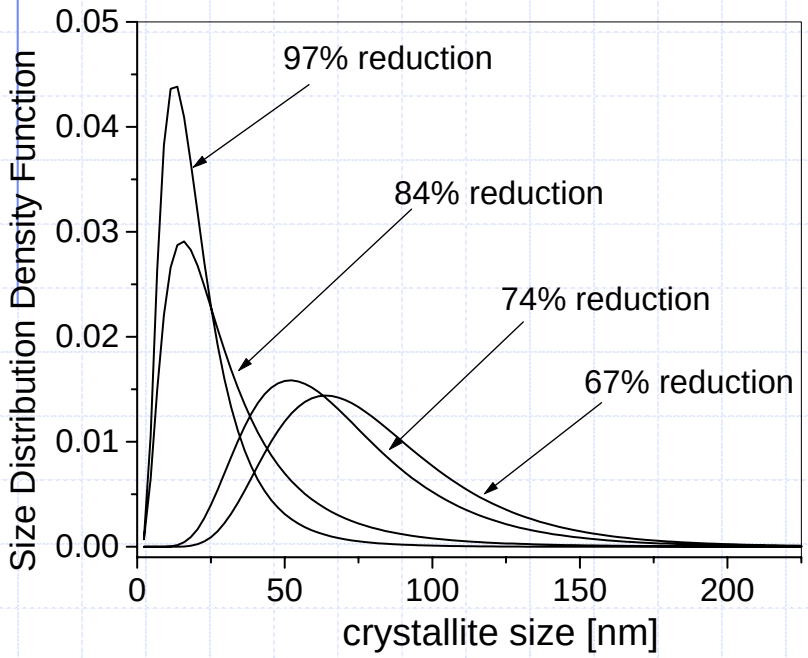
Theoretical individual and average contrast factor of dislocations

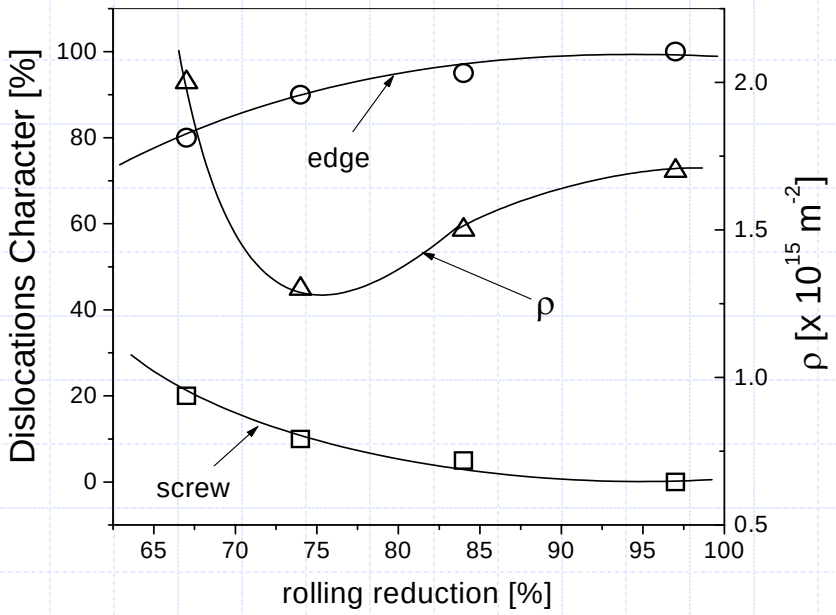
AnizC for hexagonal crystals



Applications:

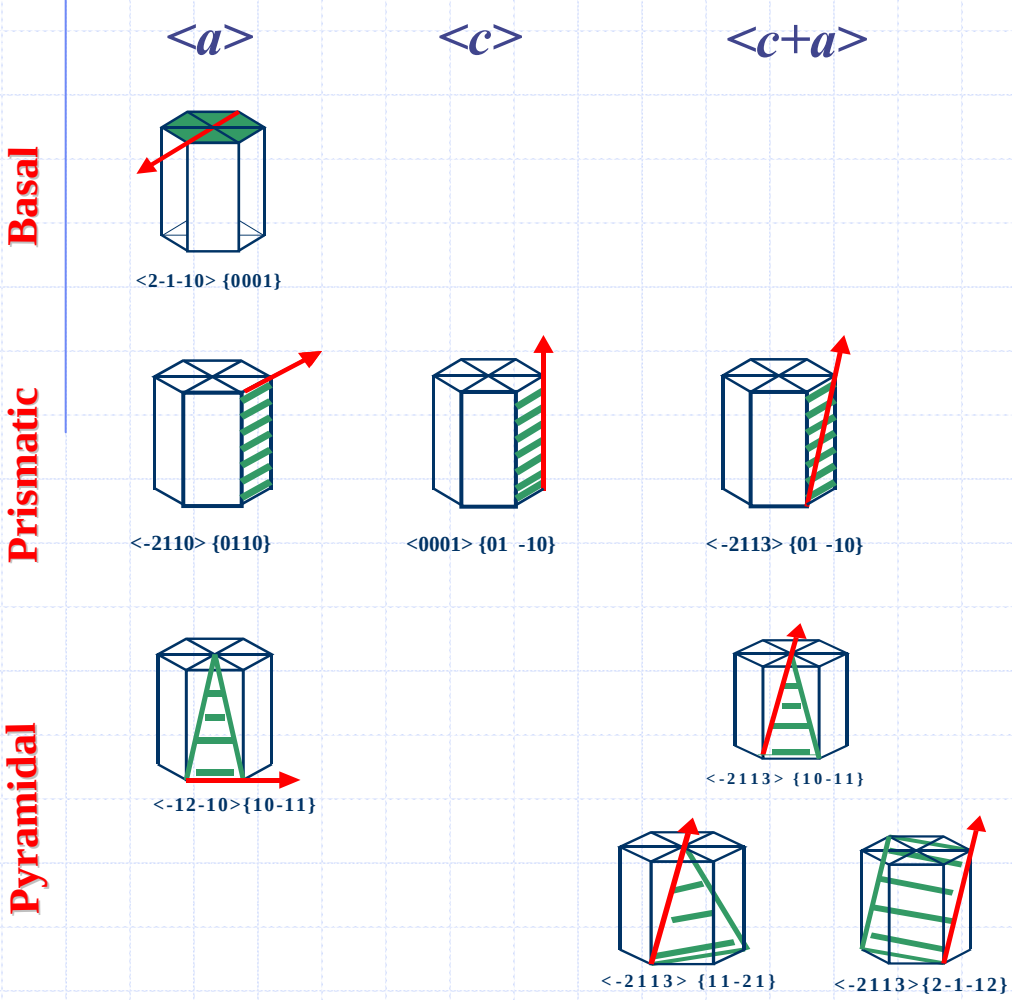
Crystallite size distribution and Dislocation density and type in nano-Cu as a function of deformation

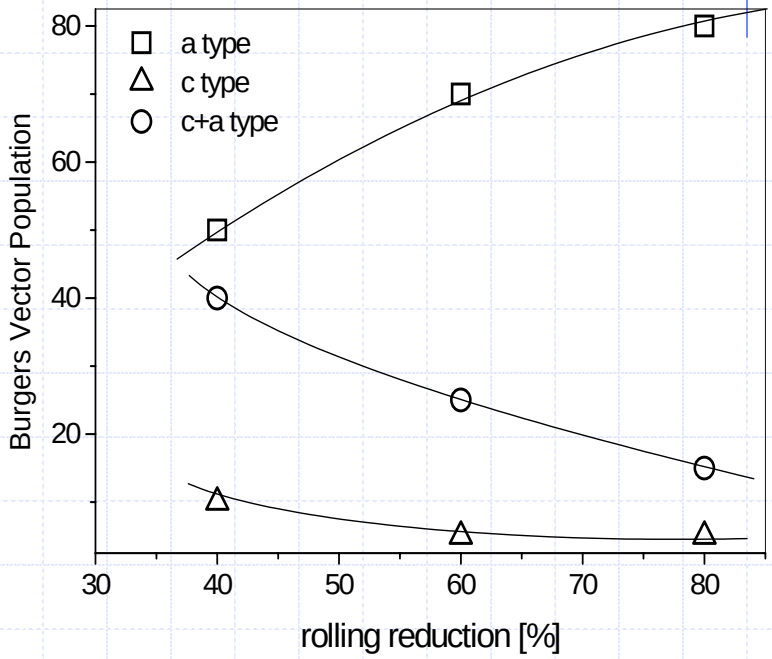




Applications:

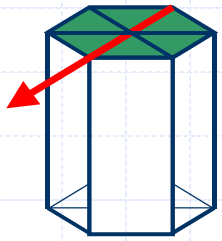
Burgers vector types in Titanium deformed by rolling at 500^0 F



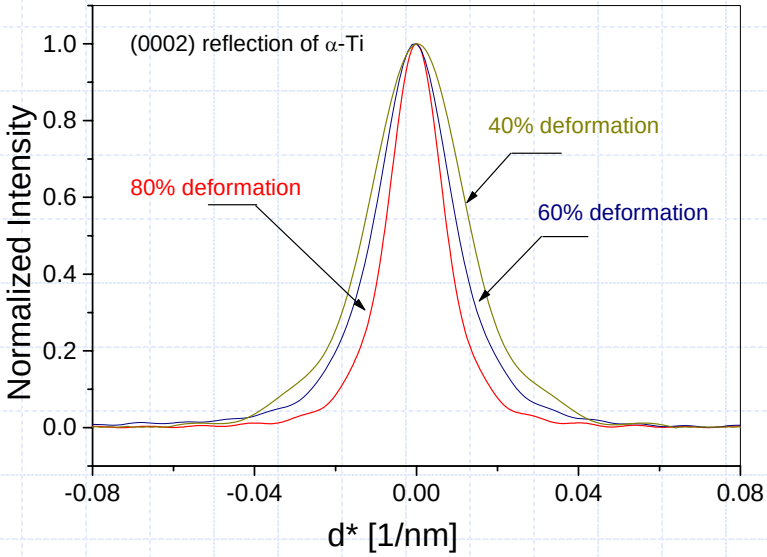


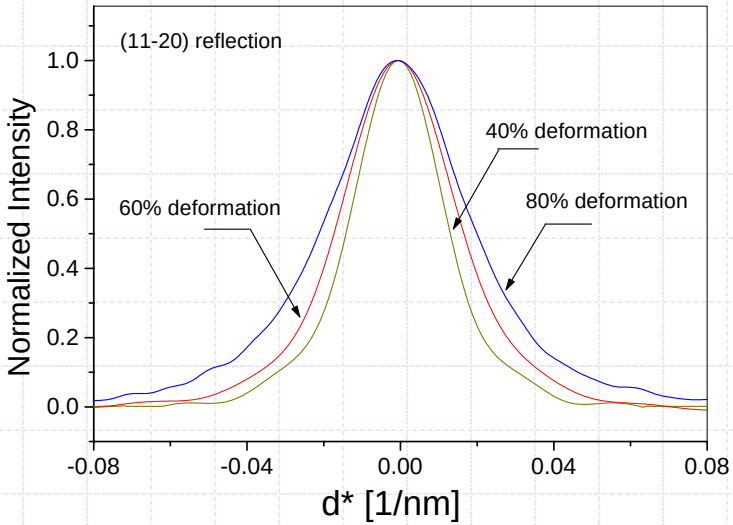
Applications:

Burgers vector types in Titanium deformed by rolling at 500^0 F



<2-1-10> {0001}





Summary

MWP or CMWP procedure enables the determination of the sample microstructure form the X-ray diffraction pattern in terms of:

1) crystallite size distribution
2) dislocation density
3) dislocations type
4) dislocations arrangement.

Acknowledgements are due to ***Prof. Ungar Tamas*** and ***Dr. Ribarik Gabor*** for continuous help provided in the preparation of the present tutorial.