RootProf

TUTORIAL 5

Size analysis

Contents

Chapter 1

The data set

Unidimensional patterns from X-ray diffraction measurements on polycrystalline samples compose our dataset. All profiles have been acquire by the same diffractometer. Experimental samples have been produced by synthesis processes of magnetite nanocrystal. The composition of the dataset is reported in Table 1. The last file is a NIST standard, named Lab6, which is used to determine the intrinsic broadening of peaks due to instrumental effects. The corresponding files are included as demo files. They are formed by two columns, the first containing the 2θ values, the second the corresponding values of diffracted intensity.

Table 1: Samples used for size analysis.

Nsample	Code
0	AADA-8A
1	AADA-4B
2	sample-2
3	AADA-1A
4	AADA-6A
5	AADA-5A
6	AADA-7A
7	lab6-54

Chapter 2

Average size determination

Motivation

Assessing the average size of crystalline domains in powder samples, determining the broadening of definite peaks in their X-ray powder diffraction spectrum.

The command file

The list of commands is the following.

```
whichanalysis 6
figpaper 1
dataType 2
range 20 50
preprocess 0 2 100
theta0 35.5
toltheta0 1.5
theta0st 30.35
lambda 1.54056
file  AADA-8A.TXT.extract
file  AADA-4B.TXT.extract
file sample-2.TXT.extract
file  AADA-6A.TXT.extract
file  AADA-5A.TXT.extract
file  AADA-7A.TXT.extract
file lab6-54.TXT.extract
nstandard
```

The commands have been included in the demo file named *fileInputSize*. See the user guide for an explanation of their meaning.

Running RootProf

Start ROOT by clicking on his icon, or by typing "root" on a terminal window. Then write the root command:

Root> .x RootProf.C("fileInputSize")

or

Root> .> outputSize

.x RootProf.C("fileInputSize")

.>

After some seconds, graphic windows will start appearing on your screen, while text output will appear on the terminal window, or redirected in the file named *outputSize*. When the run ends, the

root prompt will appear again on the ROOT terminal, and you will be able to edit each single graphic window and read the output file by your text editor.

The graphic output

Windows showing the result of the fitting procedure applied to all input profiles are produced as graphic output (Figs. 1-6). In each window, the fitted Gaussian function (red, dashed line) is superimposed on the pre-processed spectrum (black, full line). The center of the fitting range, and its size, is determined by the commands *theta0* and *toltheta0*. Toltheta0, in particular, should be chosen with care, since it highly influence the outcome of the fit. The fitting range should contain only the peak to be fitted. In this case the peak (311) characteristic of magnetite crystals has been chosen, which occurs at 2θ=35.5°. The peak width is calculated from the standard deviation parameter of the fitted gaussan. It is then used to estimate the average size of crystallites (in nm) through the Scherrer equation:

$$\langle D \rangle = \frac{k\lambda}{\beta Cos(\theta_0)},$$

where K=0.94 is the Scherrer constant, λ is the X-ray wavelength in Angstroms (given in input through the command *wavelength*), θ_0 is the Bragg angle of a given peak of the diffraction profile (given in input through the command *theta0*), and β is the full-with at half maximum of the same peak, in radiants, estimated by the fitted standard deviation parameter σ as $\beta = 2\sqrt{2\lg 2}\sigma$. The instrumental broadening is accounted for by considering the Lab6 NIST standard and the following equation $\beta = \sqrt{\beta_{Magnetite}^2 - \beta_{Lab6}^2}$. The error on the size was estimated by propagating the error on the fitted standard deviation through the Sherrer equation.

A plot of the average crystallite size and their respective errors obtained for all the input profiles is given in Fig.8.

The NIST standard compound is identified by the standard command. Its spectrum can contain a different number of points with respect to the sample profiles. The center of the peak to be considered in the standard is determined by the command *theta0St*. The fitting of the standard is shown in Fig.7, and this sample is not included in Fig.8.
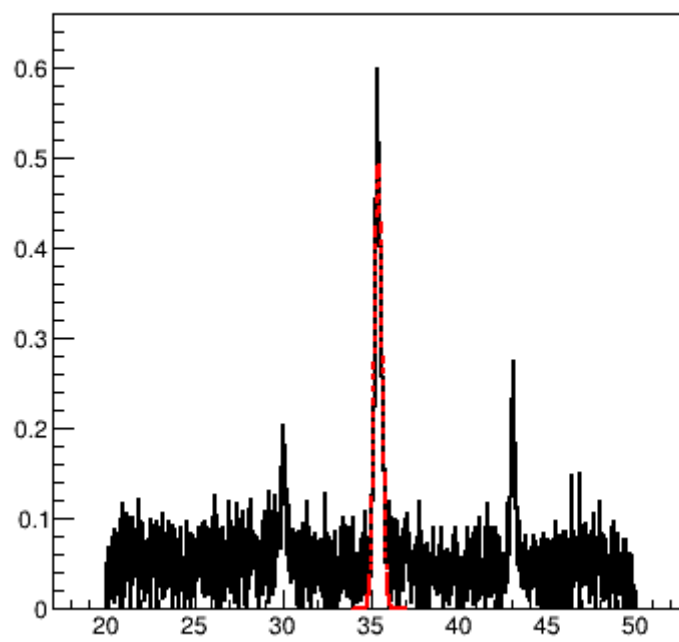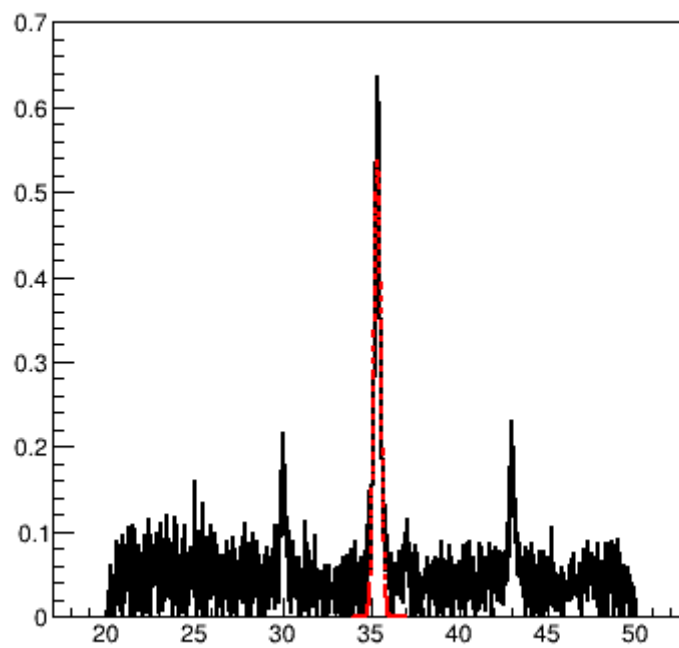
Fig.1 Size Analysis on Sample 0
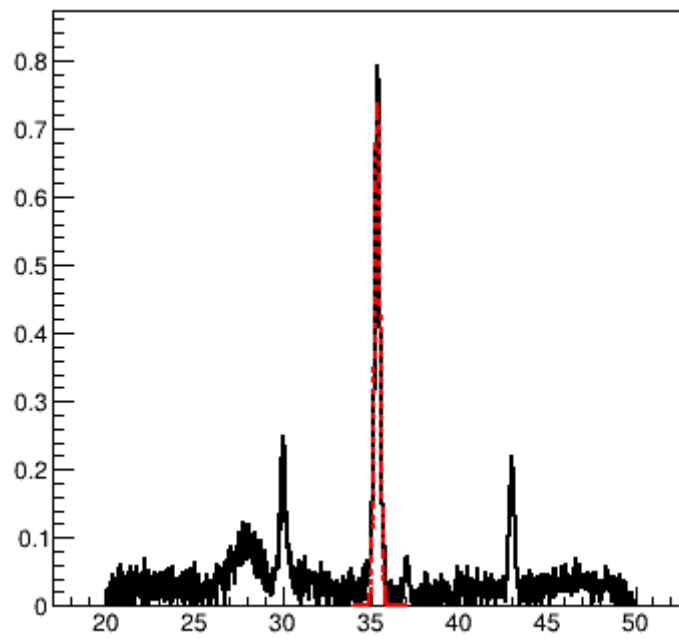

Fig.2 Size Analysis on Sample 1

Fig.3 Size Analysis on Sample 2
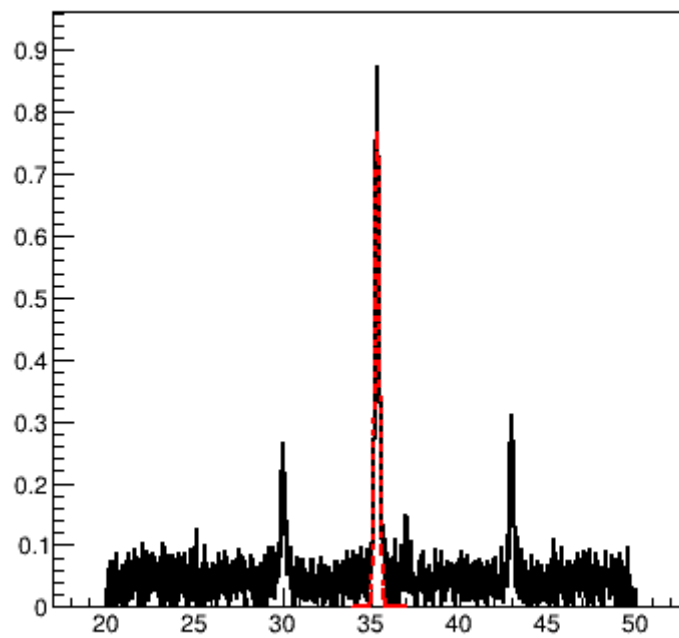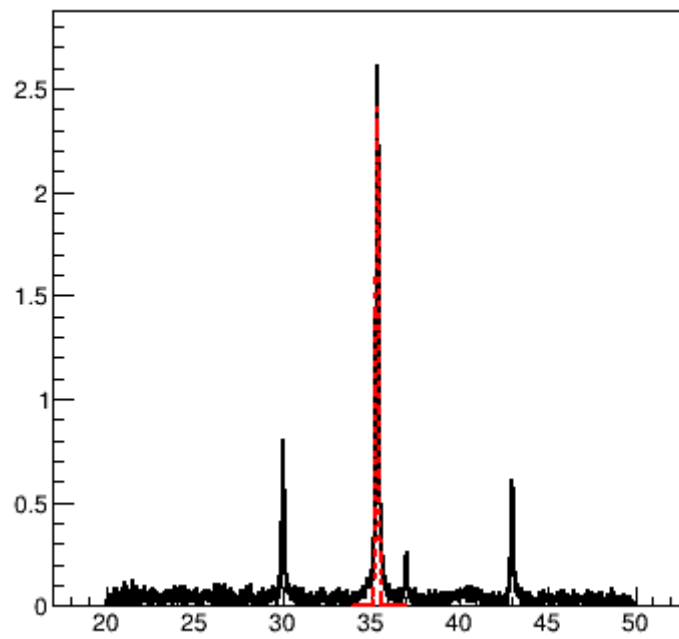

Fig.4 Size Analysis on Sample 3
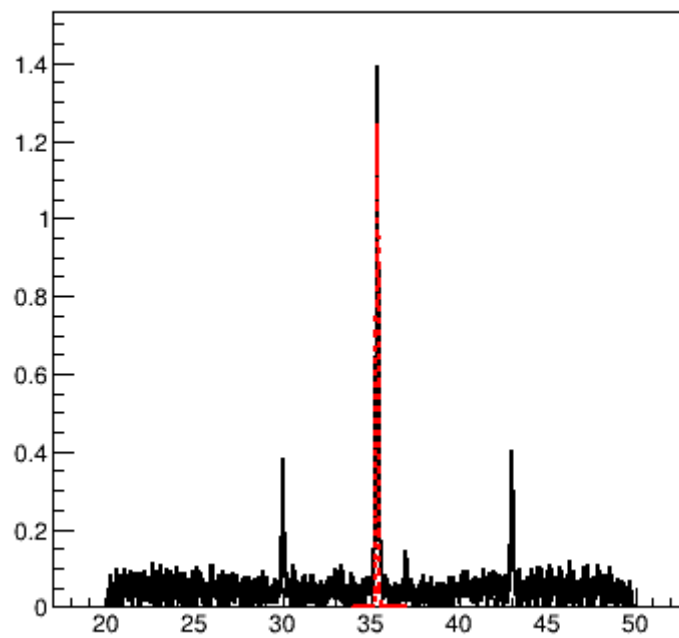
Fig.5 Size Analysis on Sample 4

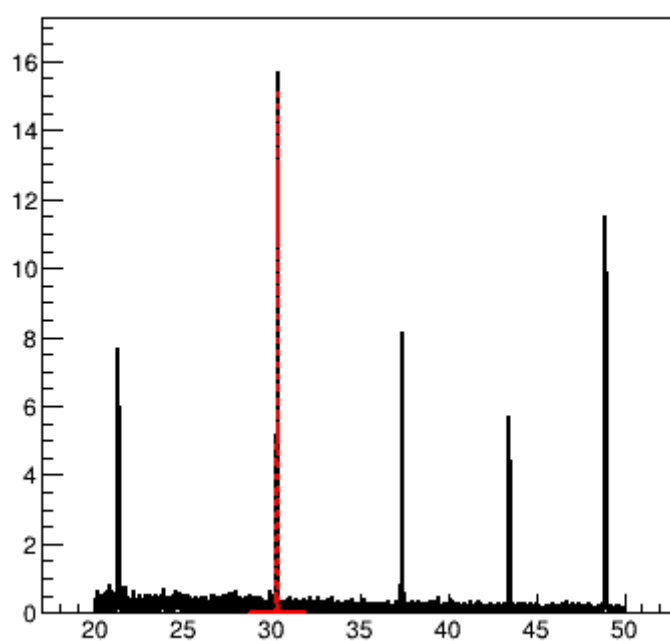Fig.6 Size Analysis on Sample 5
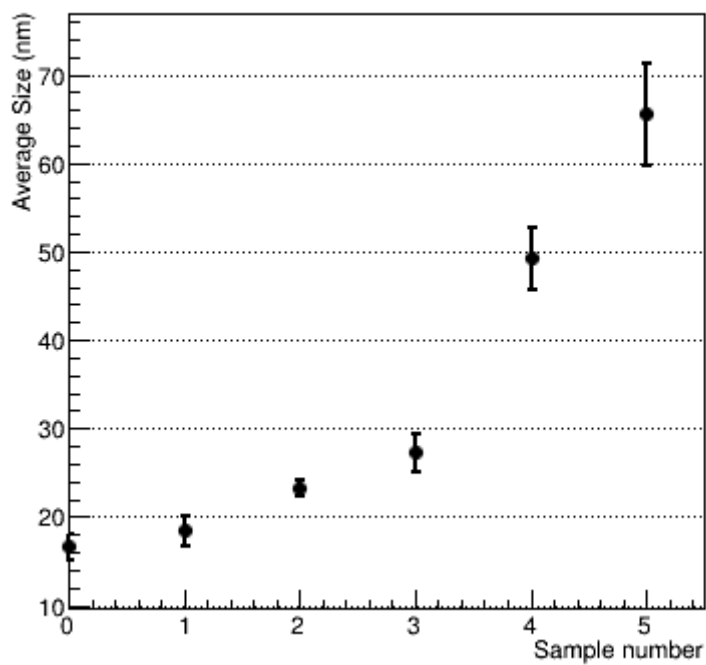
Fig.7 Size Analysis on Standard Compound

Fig.8 Size Analysis

The output file

The content of the output file named *outputSize* is reported below, with comments added.

```
Input from file: fileInputSize
```

```
-----------------------------
  whichanalysis 6

  figpaper 1

  dataType 2

  range 20 50

  preprocess 0 2 100

  theta0 35.5

  toltheta0 1.5

  theta0st 30.35

  lambda 1.54056

  file  AADA-8A.TXT.extract

  file  AADA-4B.TXT.extract

  file sample-2.TXT.extract

  file  AADA-6A.TXT.extract

  file  AADA-5A.TXT.extract

  file  AADA-7A.TXT.extract

  file lab6-54.TXT.extract

  nstandard
```

The above section shows the commands read from the command file. It should be checked to ensure that they are interpreted correctly.

```
Reading input files:
 ----------------------
Sample 0 -> file AADA-8A.TXT.extract
        Found 1501  points
Sample 1 -> file AADA-4B.TXT.extract
        Found 1501  points
Sample 2 -> file sample-2.TXT.extract
        Found 1501  points
Sample 3 -> file AADA-6A.TXT.extract
        Found 1501  points
Sample 4 -> file AADA-5A.TXT.extract
        Found 1501  points
Sample 5 -> file AADA-7A.TXT.extract
        Found 1501  points
Sample 6 -> file lab6-54.TXT.extract
        Found 3001  points

Profiles have different binning
```

The above section reports the number of data points read within each input file. The program realizes that profiles have different binning, but no special procedure is undertaken, since in the size analysis each spectrum is treated separately.

```
 Starting Size analysis
```

```
FIT RESULTS:
 -----------------------
 Spectrum 0: AADA-8A.TXT.extract
 Chi-Square=3.73e-01, Reduced Chi-Square=2.52e-03, NDF=148
 FWHM=0.009 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 1: AADA-4B.TXT.extract
 Chi-Square=4.14e-01, Reduced Chi-Square=2.80e-03, NDF=148
 FWHM=0.008 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 2: sample-2.TXT.extract
 Chi-Square=1.69e-01, Reduced Chi-Square=1.14e-03, NDF=148
 FWHM=0.006 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 3: AADA-6A.TXT.extract
 Chi-Square=5.23e-01, Reduced Chi-Square=3.53e-03, NDF=148
 FWHM=0.005 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 4: AADA-5A.TXT.extract
 Chi-Square=1.88e+00, Reduced Chi-Square=1.27e-02, NDF=148
 FWHM=0.003 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 5: AADA-7A.TXT.extract
 Chi-Square=5.36e-01, Reduced Chi-Square=3.62e-03, NDF=148
 FWHM=0.002 +- 0.000
```

```
 FIT RESULTS:
 -----------------------
 Spectrum 6: lab6-54.TXT.extract
 Chi-Square=2.38e+01, Reduced Chi-Square=8.00e-02, NDF=298
 FWHM=0.001 +- 0.000
```

The above section reports the results of the fitting procedure applied to each input spectrum separately. Fit results include goodness-of-fit estimates (Chi Square and Reduced Chi Square), number of degrees of freedom (NDF), and the best fit estimates of the full with at half maximum (FWHM) of the peak considered for size analysis.

```
AVERAGE SIZE DETERMINATIONS:
 -----------------------
 sample=0  Size=16.6 +- 1.4 nm
 sample=1  Size=18.4 +- 1.7 nm
 sample=2  Size=23.3 +- 1.0 nm
```

```
sample=3   Size=27.3 +- 2.2 nm
sample=4   Size=49.3 +- 3.5 nm
sample=5   Size=65.6 +- 5.8 nm
```

The above section reports a summary of the average size determinations (in nm) for all the input profiles.