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ATREX Powder

Program Description & Quick Start Guide

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License Agreement

Introduction

ATREX Powder is program for the conversion of two-dimensional powder diffraction data from area detectors to standard one-dimensional diffraction patterns, suitable for the use in existing Rietveld refinement programs. The key feature of the program is the statistical analysis of the intensity distribution of the binned diffraction data. This provides errors (σ) for the measured intensity values that can be used in the subsequent Rietveld refinements to determine a proper weighting scheme. Furthermore, the determined errors can be used as a criterion for the rejection of outliers such as damaged pixels on the detector, the shadow of the beam-stop or contributions of window material from sample environments *etc*. ATREX powder uses the user defined multiple of the errors (σ) of the bin intensity as criterion to create an automatic mask for the two dimensional diffraction data and to exclude all intensities outside of this region from the rebinning process.

Statistical Analysis of two-dimensional diffraction data

Nowadays, area detectors are the widely used in synchrotron and in-house powder diffraction experiments at ambient and non-ambient conditions. These detectors provide a large dynamic range (14-16 bit), a high time resolution and the advantage to collect full Debye-Scherrer rings from powder samples. However, most modern program used in the rebinning process of the 2D diffraction data into standard one-dimensional diffraction patterns are not propagating the errors for the rebinned intensity values. This can lead to problems in subsequent refinement of the diffraction data due to the application of a wrong weighting scheme for the measured intensities [1, 2]. This leads to unrealistically low errors for refined unit cell and structural parameters and meaningless values for fitting criteria such as the goodness-of-fit χ^2 and weighted residual value R_{wp} [1, 2]. The use of high-resolution powder diffraction data and the proper weighting of diffraction intensity in the least-

squares refinement are essential for obtaining accurate structural parameters in Rietveld refinement [2]. However, diffraction experiments at extreme environmental conditions have generally comparatively low count rates and a reduced peak-to-background ratio due to the properties of the sample environments. Therefore, a careful analysis of the errors is essential for robust and reliable structure determination and structure refinement from powder diffraction data collected from samples at extreme conditions.

Standard Counting Statistics and Intensity Error Estimation

The formalism for the determination of the intensity errors off 2D diffraction data is described in detail in reference [1]. Here we briefly repeat the main points of the underlying formalism for the intensity error estimation of 2D diffraction data. Since two dimensional detectors are pixelated the diffraction intensities at a given scattering angle are measured multiple times. The intensity distribution can then be described as a Poisson distribution

$$P(\bar{I}) = \frac{\lambda^I}{I!\, e^{-\lambda}}$$

with $\bar{I} = \lambda$ and standard deviation $\sigma = \sqrt{\lambda}$. For large mean intensity values $\bar{I}, i.e. \bar{I} > 20$ the distribution can be approximated by Gaussian distribution

$$P(\bar{I}) = \frac{1}{(\lambda\sqrt{2\pi})e^{-\frac{(I-\lambda)^2}{2\lambda}}}$$

with the condition $\lambda = \bar{I} = \sigma^2$. In this case the standard deviation of the distribution can be used as a measure of the uncertainty of the measured intensity values, for a single intensity measurement. Since in this case the mean intensity value of the distribution $\bar{I}$ is unknown, the measured intensity *I* is used to estimate the error:

$$\sigma = \sqrt{I}$$

With 67% probability the mean intensity is within the thus calculated error interval of the measured intensity. Obviously, the percentage error of the measured intensity decreases with increasing intensity; for a 1% error at a 99% confidence level an intensity of 67000 counts is required [1].

As mentioned before, the intensities for each diffraction angle are multiply measured in a diffraction experiment employing a two dimensional detector and grouped for a small diffraction angle interval (rebinned), their distribution is directly accessible and can be analyzed.

The intensity uncertainty of a 2θ bin is given as

$$\sigma_I = \sqrt{\bar{I}/_N}$$

with $\bar{I}$ being the mean intensity and *N* the number of contributing pixels to this bin. This simple analysis of the diffraction intensities contributing to each 2θ provides us with error for our measured intensities that can be used in to identify the proper weighting scheme in the subsequent Rietveld refinement of the data.

Automated Masking

Experiments at extreme environmental conditions are a number of obstacles into the collection of the 2D diffraction data. The sample environment usually contributes to the recorded diffraction signal on the 2D detector in form of single crystal spots, secondary excitations and other intensity stemming from the sample cell. These non-sample contributions to the diffraction data need to be rejected from further analysis, since they are reducing the overall quality of integrated diffraction patterns. Currently, the identification of these intensities regions is performed visually and the actual masking is done manually. Generally, this process is rather laborious and not very precise. However, using the fact that the frequency of intensities on a Debye-Scherrer ring of constant diffraction angle 2θ is Poisson distributed, such pixels can be identified by computing the difference of the pixel intensity to the mean intensity on a ring. In other terms the width of the intensity distribution $\sigma_{\bar{I}}$ around the mean intensity provides us with a measure to identify parasitic scattering and to reject it automatically from the rebinning process. ARTEX powder allows the user to define window around the mean intensity value $\bar{I}$ of the bin; all intensities outside of this window will be excluded from the following rebinning process. The width of the window is defined by multiples (m) of the standard deviation $\sigma_{\bar{I}}$ of the mean intensity $\bar{I}$ of each 2 θ bin.

Installation Instructions

ARTEX powder has been developed using the eclipse 4.2.2 build of python for 64-bit windows systems (eclipse-SDK-4.2.2-win32-x86_64). The following package need to be installed for the use of ARTEX powder:

- cx_Freeze-4.3.4.win-amd64-py2.7.exe
- fabio-0.1.3.win-amd64-py2.7.exe
- h5py-2.5.0-cp27-none-win_amd64.whl
- lxml-3.4.2.win-amd64-py2.7.exe
- Mako-1.0.2-py2.py3-none-any.whl
- matplotlib-1.2.0.win-amd64-py2.7.exe
- Msvcp71.dll-And-Msvcr71.dll-files by harjot pawra.zip
- numpy-1.10.1+mkl-cp27-none-win_amd64.whl
- opencl_runtime_14.2_x86_setup.msi
- PIL-1.1.7.win-amd64-py2.7(1).exe
- pyFAI-0.9.4.win-amd64-py2.7.msi
- PyFFTW3-0.2.2.win-amd64-py2.7.exe
- pyopencl-2015.1-cp27-none-win_amd64.whl
- pyparsing-2.0.3.win-amd64-py2.7.exe
- PyQt4-4.11.3-gpl-Py2.7-Qt4.8.6-x64.exe
- pyqtgraph-0.9.10.win-amd64.exe
- python_dateutil-2.4.2-py2.py3-none-any.whl
- python-2.7.10.amd64.msi
- scikit-image-0.10.1.win-amd64-py2.7.exe
- scikits.umfpack-5.1.0.win-amd64-py2.7.exe
- scipy-0.11.0.win-amd64-py2.7.exe

- six-1.10.0-py2.py3-none-any.whl
- VCForPython27.msi

Notes: Alternative Scientific Python stacks like Enthought Python Distribution, Anaconda, Canopy, PythonXY or WinPython offer most of the scientific packages already installed and can make the installation of dependencies easier. However, they all offer different packaging systems and we can't support them all. Using a 64bit operating system is highly recommended since there are issues and limitations when using 32bit operating systems for dealing with large datasets.

Program Features

This module allows for:

- The .tif format is currently the only diffraction image format supported
- The number of pixels that run horizontally and vertically along an image can be specified.
- The adding or averaging of files and saves the summed or averaged image as a .tif, .edf or .txt file. The saved file(s) are located in: C:\Users\MainDirectoryName\workspace\PythonProjectFolder
- The subtraction of a dark image from a data image and saves the new image as a .tif or .edf file. The saved file(s) are located in: C:\Users\MainDirectoryName\workspace\PythonProjectFolder
- The calculation of a 2-theta file and saves the geometry information as a .tif, .edf or .txt file. The saved file(s) are located in: C:\Users\MainDirectoryName\workspace\PythonProjectFolder
- The usage of pyFAI and performs azimuthal integration to acquire a 1D diffraction pattern and it can produce a 2D diffraction pattern from the 1D data, while using matplotlib to save the image in a variety of formats and anywhere on the computer.
- The use of a statistical analysis method that automatically masks data that is outside a certain number of standard deviation away from the mean.
- Its output files to be loaded/used in other programs like FIT2D and DIOPTAS.

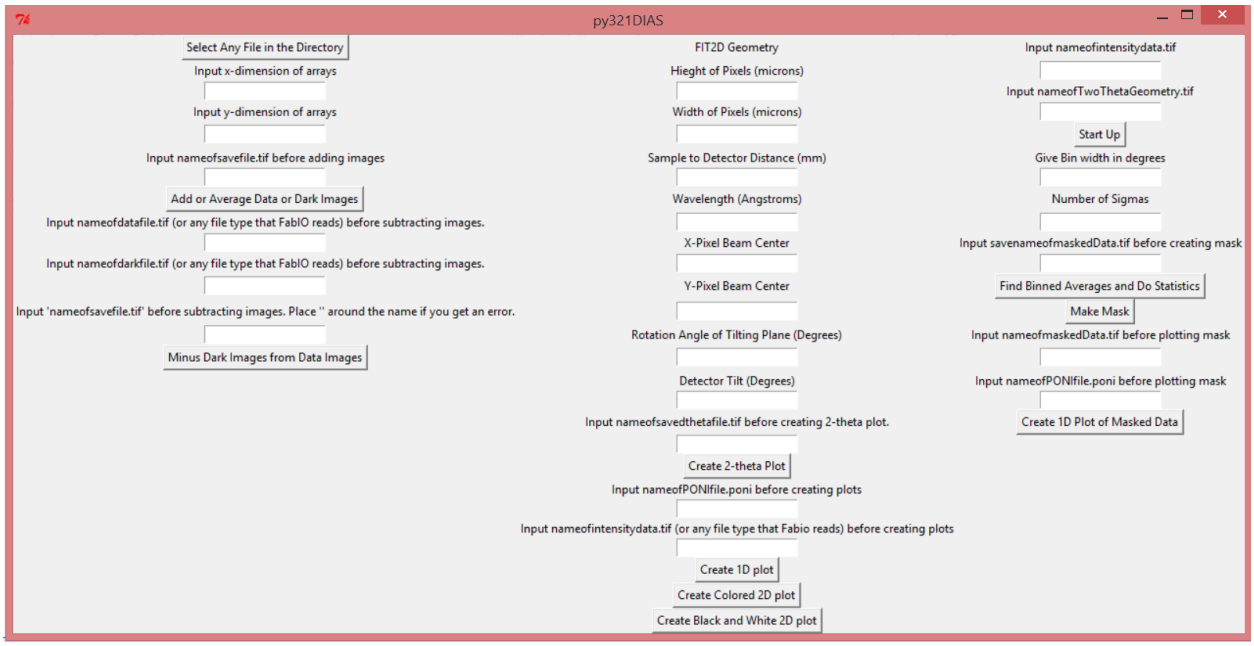

Figure 1 Graphical User Interface of ARTEX powder.

References

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[2] H. Toraya, Weighting Scheme for the Minimization Function in Rietveld Refinement, Journal of Applied Crystallography 31:333-343 (1998).

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