

# SGTools manual

# The Latest Version 2.02 (2021-08-17)

Update: The released version 2.02 develops new functions of polarization correction, creating mask, estimating standard error of the scattering intensity. This release also fixes the known issues reported in 1.0: the inability to show the image with nonlinear coordinate. The definition of the input and output format are added in the software document.

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## **SGTools manual**

#### 1, Introduction

SGTools is a new and open-source SAXS and GISAXS data processing and analysis suite based on the Matlab platform. The software contains functions of instrument parameter calibration, two-dimensional graphic output, background subtraction, data reduction, and dynamic animation generation (Movie, GIF), intensity mapping, and several analysis modules are also developed, such as order degree analysis based on both paracrystal model and Williamson-Hall theory, and interface correlation analysis for multilayer structure. Besides, the batch processing function is developed for large data sets produced from the in-situ x-ray scattering experiment, which can realize fast processing of the huge amount of data. Besides, some other tools including X-ray reflectivity data combination, file format transformation and penetrate depth calculation are also implanted in the tool kit. In the future, the modules for even more in-situ x-ray scattering experiments such as fiber stretching process, charging, discharging processes of battery, and the construction of threedimensional reciprocal image can also be envisaged. In addition, the using of parallel computing by GPU and multicore parameters to accelerate the data processing should be introduced for high-performance computing. The open-source code allows the users to integrate new features directly to the tools for personalized application.

The user manual of SGTools mainly introduces the software framework, data processing workflow, basic theory, features of the software. Several examples are demonstrated to show how the use of SGTools for dealing with the SAXS and GISAXS data. In addition, several videos are recorded to introduce the operation of SGTools. More tutorials about the functions in the program will be gradually improved in the updated manual. The software installation packages and video tutorials are provided on the below website.

(http://www.sgtools.cc, English); (http://www.nickelblog.online, Chinese)

## 2. Small-angle X-ray scattering



## 2.1 Theory of Small-angle X-ray scattering

Figure 1 The Interaction between X-ray and atoms

According to the X-ray theory, the incident X-ray mainly interacts with electrons in a material, in the form of absorption (produces fluorescence, which mainly occurs near the absorption edge), transmission, and scattering as shown in Figure 1. Among them, scattering is divided into inelastic scattering (wavelength change, outer electron interaction) and elastic scattering (wavelength does not change, inner layer binds electron), However, the interaction between X-ray and electron is mainly elastic scattering, the inelastic scattering of electrons in an atom is relatively weak. Elastic Xrays scattered by different electrons have a coherent effect due to the spatial density distribution. Therefore, the information of the electron density distribution in the materials can be analyzed according to the spatial distribution of the elastic X-rays[1].

Small-angle X-ray scattering (Figure 2) refers to the scattering of x-rays at small angles as the x-rays interact with the matters which is sensitive to the electron density difference within the materials. Based on the theory, the SAXS technique has become an important tool for structure characterization in the scale of nanometers of the materials. According to the size of the X-ray scattering angle, the experimental method can be divided into small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS). Small-angle scattering corresponds to large-scale structures in material (1nm-100nm), wide-angle scattering corresponds to small-sized structures



#### Figure 2 Schematic diagram of the experimental method of SAXS

(less than 1nm, atomic-scale structures), and the grazing incidence method is used for obtaining nano-scale structural information in the film system[2, 3].

Another important experimental method is the anomalous small angle X-ray scattering which could obtain contrast variation in a multicomponent system[4]. It is based on the fact that for X-rays whose energy is close to the absorption edge of an element, the atomic scattering factor of that element is reduced by a few electrons from its value far from the edge, then a contrast variation can be obtained by simply varying the energy of the incident X-ray photon. In recent years, the simultaneous SAXS-WAXS is widely used to realize the multiscale structure characterization from the atom to the nanoscale. Table 1 shows the detection scale range and typical application system of the main SAXS experimental methods when the energy of the incident X-ray is 10 Kev. The samples of TSAXS can be in various forms such as solutions, powders, and blocks. WAXS is mainly used for the analysis of crystal structures such as powders,

blocks, and fibers. GISAXS is mainly used for nano-scale structure analysis of thin films.

Experiment al method	2θ range	q(nm <sup>-1</sup> )@10 Kev	Size range	Application system
TSAXS	0.1°-8°	0.00628-0.628	1nm-100nm	Colloidal solution, Polymer,liquid crystal, fiber, Mesoporous
WAXS	8°-120°	7.06-87.72	0.01nm-1nm	Analysis of crystal structure of powder, block, and fiber
GISAXS	0.1°-8°	0.00628-0.628	1nm-100nm	Quantum dot film, block copolymer film, liquid crystal film, mesoporous film

Table 1.SAXS main experimental methods detection scale and application system

## **2.2 Data processing process**



Figure 3 Data processing workflow of small-angle X-ray scattering

In the small-angle X-ray scattering experiment, the detector system obtains a two-

dimensional(area detector) or one-dimensional(linear detector) matrix. As shown in Figure 3, the data processing and analysis flow in SAXS is data calibration, data process, and data analysis. Firstly, the instrument parameters need to be calibrated by standard samples, and space transformation of the scatter pattern into the coordinate of scattering angle or scattering vector could be realized. Due to background scattering from the beam slit, the container, and the air is also recorded by the detector. Therefore, it is necessary to perform a background correction according to the features of the sample system. To obtain the quantitative structure information of the sample, data reduction requires converting the two-dimensional data into one-dimensional data, commonly in the forms of arc or rectangular integration which are selected according to the symmetrical characteristics of the system. At last, the theory model is developed for obtaining the structure information through fitting to the experimental data, and the results error is estimated by fitting algorithm.

#### 2.2.1 Instrument parameters calibration

The detectors used in the X-ray scattering experiment are mainly two-dimensional planar detectors, one-dimensional linear detectors, one-dimensional arc detectors, and point detectors. Although there are different kinds of detectors, scattering data collected in the detector is the intensity distribution of the pixel position. In this manual, we mainly discussed the method of calibrating the two-dimensional planar detector. The data obtained from the planar detector is usually saved in a two-dimensional matrix file, and the value of each unit in the matrix represents the intensity of scattered X-rays, and the row and column values of the matrix unit represent the position of each unit in the detector.

The scattering angle is defined as the angle between the incident beam and the scattered beam. Therefore, it is first necessary to determine the pixel position of the incident beam and the scattering beam in the two-dimensional matrix, Then the matrix could be converted into the coordinate system of the scattering angle or scattering vector. The conversion formula is as follows

$$2\theta = \tan^{-1}(\frac{\sqrt{(P_x - P_{xc})^2 + (P_y - P_{yc})^2}}{D})$$
 (1)

$$q = \frac{4\pi}{\lambda} \sin \theta \tag{2}$$

Among them,  $P_{xc}$ ,  $P_{yc}$  are the row and column values of the incident beam in the two-dimensional data respectively, D is the distance from the sample to the detector, and  $\lambda$  is the wavelength of the X-ray. The above parameters are usually calibrated by standard samples such as animal collagen materials (beef tendons, chicken tendons, etc.), organic chemical synthetic materials (silver behenate, etc.). For the WAXS method, the inorganic compounds (LaB<sub>6</sub>, CeO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, etc.) are usually used in the calibration. The two-dimensional scattering pattern of standard samples is a scattering arc or a scattering ring, then the center position of the incident beam is the center of the scattering ring. Then the center position of the incident beam is obtained by fitting the scattering ring to the circle equation. The distance D from the sample to the detector could be calculated by the position of the scattering peak of the standard sample.



#### 2.2.2 Data reduction of X-ray scattering data

Figure 4 (a) System with spherical symmetry (b) System with axial symmetry

The basic method of conversion two-dimensional scattering pattern into onedimensional intensity distribution is data integration or projection. According to the type of structure symmetry, the scattering system can be divided into an anisotropic scattering system, an axisymmetric system, and a specific orientation scattering system. For an isotropic scattering system, the scattering in all directions is symmetry about the beam center (Figure 4a), so ring or sector integration is usually used to obtain the scattering intensity as a function of the scattering vector. For the scattering system with axial symmetry (Figure 4b), the method of data reduction is polarization averaging and projection methods. For a specific orientation of the scattering system, the commonly used data preprocessing method is the cutting method. The integral formula show below corresponding to these three methods[5]:

$$I(q) = \int_0^{2\pi} I(q,\varphi) \, d\varphi \tag{3}$$

$$I(q_3) = 2\pi \int_0^\infty q_{12} I(q_{12}, q_3) dq_{12}$$
(4)

$$I(q) = I(q, \varphi)|_{\varphi=0\sim 2\pi}$$
(5)

To estimate the the uncertainty of the reduced data, standard deviation is calculated through equation (6). This is useful for determining the error of followed data analysis such as model fitting. And the error data is saved to the reduced output data file.

$$S = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} |I_i - \mu|^2}$$
(6)

Where S is the standard error of scattering intensity, N is the number of the intensity data. I<sub>i</sub> is the intensity data,  $\mu$  is the mean of the scattering intensity I<sub>i</sub>.

#### 2.2.3 Background correction

The scattered X-rays obtained by the detector include not only the scattering from the nanostructure in the sample but the background scattering from the instrument in the scattering experiment. Meanwhile, the scattered X-rays are partially absorbed after penetrating the sample. In order to obtain the real scattering intensity, the scattered X-rays need to be corrected for absorption and background scattering. The correction of the scattering intensity is different for the transmission and grazing-incidence experimental method.

For the transmission experimental method, according to the characteristics of the scattering system, it can be divided into four categories to correct the scattering data respectively[6]:

a) Solid and powder sample system:

$$I_c = I_{st} \exp\left(\frac{-\mu t}{\cos(2\theta)}\right) - I_b \tag{7}$$

b) Dilute solution system:

$$I_c = I_{st} \exp\left(\frac{-\mu t}{\cos(2\theta)}\right) - c * I_{sov} - I_b$$
(8)

c) Thick solution system:

$$I_c = I_{st} \exp\left(\frac{-\mu t}{\cos(2\theta)}\right) - I_{sov}$$
(9)

d) Protein and nucleic acid solution:

$$I_c = I_{st} \exp\left(\frac{-\mu t}{\cos(2\theta)}\right) - p * I_{sov}$$
(10)

Among them,  $I_c$  is the corrected scattering intensity,  $I_{st}$  is the sample scattering data collected by the detector,  $I_b$  is the scattering data of the blank sample,  $I_{sov}$  is the pure solvent scattering intensity of the solution sample. Absorption factor  $\exp\left(\frac{-\mu t}{\cos(2\theta)}\right)$  represents the absorption of scattered X-rays in  $2\theta$  scattering direction after penetrating a sample with a thickness of t, which can be approximated as  $\exp(-\mu t)$ , calculated by the ionization chamber before and after the sample:

$$\exp\left(-\mu t\right) = \frac{I_{2,s}I_{1,0}}{I_{2,0}I_{1,s}} \tag{11}$$

Among them,  $I_{1,0}$ ,  $I_{1,s}$  are the pre-ionization readings of the blank sample, and  $I_{2,0}$ ,  $I_{2,s}$  are the post-ionization readings of the test sample.

#### 2.2.4 Polarization correction

After the background subtraction, the polarization correction function in SGTools can be applied to correct the reduction of scattering intensity due to the polarization of the incident radiation. It is commonly important for increasing the accuracy of wide-angle scattering data. But, for the small angle scattering data, the polarization of X-ray source is often considered negligible. The correction factor for 2D detector images is given by Hura et al as show in equation (12) [7].

$$I_{j,cor} = I_j [P_i (1 - (\sin(\psi)\sin(2\theta))^2) + (1 - P_i)(1 - (\cos(\psi)\sin(2\theta))^2)]$$
(12)

Where  $\psi$  is the azimuthal angle on the detector surface(defined here clockwise, 0 at 12 o'clock), 2  $\theta$  is the scattering angle, and Pi is the fraction of incident radiation polarized in the horizontal plane (azimuthal angle of 90°), the Pi factor of most synchrotron beam lines may be about 0.95 or so.

#### 2.3 Data analysis model

At present, the data analysis model including in the program mainly focuses on the structural analysis of thin films. The models are divided into three modules. The first module is a typical self-assembled film system which is prepared by the bottomup thin film deposition process. In order to quantitatively characterize the relative length of the surface and the interface, the critical correlation length is determined by analyzing the diffuse scattering peak. The second module is used for analyzing the order degree of structure unit in thin films, such as block copolymer self-assembly film and superlattice quantum dots. The WH model and paracrystal model are both used to obtain the structure information through analyzing the position and width of the scattering peaks. As the program is still under development, more theoretical analysis models will gradually be implemented in the software.

#### 2.3.1 Correlated roughness

The surface structure of the film prepared by the bottom-up deposition method is usually controlled by the structure at the substrate interface, but the influence of the substrate interface structure on the surface is gradually reduced as the thickness increases. The critical cut-off length of surface-to-interface structure replication is defined as  $R_c$  which is used for characterization of the correlation between the surface and interface show in Figure 5. This correlation is one of the important factors for structure control and performance optimization of thin films such as nano-multilayer film, polymer film deposition, polyelectrolyte self-assembly film, LB film, and thin films.



Figure 5 Schematic diagram of surface correlation analysis

The phenomenon of scattering intensity oscillation between the critical angle of the film surface and the reflection angle of the substrate is considered as resonant diffuse X-ray scattering, which is formed by the coherence between the X-rays reflected on the film surface and the X-rays reflected by the interface. Therefore, the correlation between surface and interface can be obtained by analyzing the resonant diffuse peak[8, 9]. The period of the intensity oscillation in the vertical direction can reflect the thickness information of the film surface, and the decay extent of the scattering peak in the horizontal direction can reflect the correlation between the surface and the interface structure. Then the critical cut-off length of surface-to-interface structure replication defined as R<sub>c</sub> can be calculated by the following formula:

$$R_c = \frac{2\pi}{\Delta q_{coor}} \tag{13}$$

Among them,  $\Delta q_{coor}$  is the transverse (along  $q_y$ ) propagation length of the modulation in the reciprocal space.

#### 2.3.2 Order degree analysis

The fabrication of Ordered structure in nanoscale is an essential process for functional nanodevices such as waveguides, photonic crystals, and microelectronics. The order degree is usually an important factor for device performance. The application of SAXS technology is one of the effective methods for quantitative characterization of ordered structures. At present, the commonly used analysis models are WH model and quasi-crystalline model. The WH model is suitable for systems with high-order scattering peaks, and the quasi-crystalline model is suitable for systems with low order scattering peak.

#### (1) Williamson-Hall model analysis

According to the WH theory[10, 11], the integral width of the scattering peak is mainly composed of size broadening and strain broadening. The size broadening does not depend on the order of the scattering peak, and the strain broadening increases with the increase of the order of the scattering peak. Therefore, in order to separate the size broadening and the strain broadening, it is necessary to analyze the multi-level scattering peaks through the WH equation to obtain the size and strain structure information.

WH equation: 
$$\beta_i^* \equiv \frac{(\beta_i \cos \theta_i)}{\lambda} = \frac{2\pi}{D_V} + 2\varepsilon q_i$$
 (14)

Among them,  $\beta_i^*$  is the integral width of the scattering peak in the reciprocal space,  $D_v$  is the volume-averaged ordered structure area size,  $\varepsilon$  is the strain factor parameter, and  $q_i$  is the position of the scattering peak in the reciprocal space. It can be seen from the equation that the region size and strain factor parameters can be obtained by fitting the slope and intercept of the linear equation to the multi-stage scattering peak. (2) Paracrystal model

Based on the GISAXS theory, it can be known that the scattering intensity distribution is determined by the shape factor and the structure factor. Its general expression is [3]:

$$I_{sc}(2\theta_f, \alpha_f) = \iint |F(q)|^2 P(R) S(q) dR$$
(15)

Among them, F(q) is the shape factor, S(q) is the structure factor, and P(R) is the size distribution. According to the theory of x-ray crystallography, finite size and position dislocation were the major factors to broaden the Bragg peaks. The paracrystalline theory[12, 13] popularized by Hosemann and co-workers were usually used to associate these two effects with the peak shape. In our research, the two-dimensional paracrystal lattice model was decomposed into one-dimensional term due to the symmetry around the  $q_z$  axis. The position of the particles were basically described by a one-dimensional Markov chain where the loss of long range order was introduced in a cumulative way from point to point through the knowledge of the distance probability p(x) between two neighboring nodes. The node-node autocorrelation function  $g_+(x)$  for positive value  $x \ge 0$  reads:

$$\mathbf{g}_{+}(\mathbf{x}) = \mathbf{p}(\mathbf{x}) + (\mathbf{p} \otimes \mathbf{p})(\mathbf{x}) + (\mathbf{p} \otimes \mathbf{p} \otimes \mathbf{p})(\mathbf{x}) + \cdots$$
(16)

Where  $\otimes$  is the folding product of p(x) by itself. Each term corresponds to the distance of the nth neighbours to the origin. When x < 0  $g_{-}(x)$  reads  $g_{-}(x) = g_{+}(-x)$ . Once added the contribution of the origin  $\delta(x)$  and  $g_{-}(x)$ , the interference

function(structure factor) is obtained by Fourier transform of the total pair correlation function g(x)

$$S(q) = 1 + \mathcal{P}(q) + \mathcal{P}(q) \cdot \mathcal{P}(q) + \mathcal{P}(q) \cdot \mathcal{P}(q) + \cdots$$
(17)

Where  $\mathcal{P}(q)$  is the Fourier transform of the probability law p(x). Finite size effects can be easily accounted by restricting the sum to a given number of nodes. Thus for normally distributed distances,

$$p_{(x)} = \frac{1}{\sigma\sqrt{2\pi}} e^{-\frac{(x-D)^2}{2\sigma^2}}$$
(18)

The interference function with finite size effect can be given by

$$S(q) = \left(\frac{1}{N} \frac{1 - \psi(q)^2}{1 + \psi(q)^2 - 2\psi(q)\cos qD} - \frac{1}{N^2} \frac{2\psi(q)(\cos(qD) - 2\psi(q) + \psi(q)^2\cos(qD))}{(1 + \psi(q)^2 - 2\psi(q)\cos qD)^2}\right)$$
(19a)

$$\psi(q) = e^{\pi q^2 \sigma^2} \tag{17b}$$

Where D is the average distance between the neighbor nodes,  $\sigma$  is the full width at maximum distance distribution, N is the number of subunits in a coherent region.

## 3、Software Installation and instruction

### **3.1 SGTools installation**

SGTools is developed based on the Matlab platform and made available as compiled packages, the users do not require a Matlab license. But the Matlab runtime needs to be installed before SGTools start. Two methods are provided for the Matlab runtime: standalone version and web version. For the standalone version, the Matlab runtime is embedded into the install packages. For the web network version, the Matlab runtime is automatically downloaded from the website. The installation of the SGTools through the standalone and web method is described below.

#### 3.1.1 Offline installation

Open the **SGTools\_alone** folder, right-click **MyAppInstaller<u>mcr</u>** and run as administrator, wait for a while and follow the prompts to install(Please close 360 and other software before installation. If the blocking interface pops up, please allow this operation.). After installation, you can start the SGTools through the desktop icon.

#### 3.1.2 Online installation

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Figure 6 The installation package file of the SGTools

Open the **SGTools\_web** folder, right-click **MyAppInstaller\_web** in Figure 6 and run as administrator (If the blocking interface pops up, please allow this operation). Then wait for a while and follow the prompts to install, the install package will

automatically detect the Matlab runtime, if it has not existed in the system, the package downloads the runtime and install. After installation, you can start the SGTools through the desktop icon.

SGTools	- 🗆 X
SAXS/GISAXS Tools	
SAXS/GISAXS data treatment	Description of SG Tools
Ins Calibrate BPSAXS view 1D BG Cor	A brief description of the SG tools for dealing and analyzing
Ins Mapping	SAXS/GISAXS data
SAXS/GISAXS data analysis	
Order degree cal 1D paracrystal model	
Other tools	
Rel cur com File formatTR PD-Caculator	•
Developed by Nie Zhao, 278560798@qq.com; Chunming Yang, yangchunming@zjlab.org.cn;	

## **3.2 Instruction of SGTools**

#### Figure 7 Interface of the SGTools for starting tools

The data processing and analysis of SAXS data are varied due to the experimental methods used in characterizing the sample. The SGTools software compiled the data processing modules into several standalone tools. Figure 7 shows the quick start interface of the SGTools including the instrument parameter correction module (Ins Calibrate), two-dimensional data processing (BPSAXS view), one-dimensional data background correction (1D BG Cor), order degree calculation (order degree cal), one-dimensional quasi-crystal model (1D paracrystal model), reflectivity data combine (Rel cur com), File format transformation (File format TR), penetration depth calculation (PD calculator), etc. More data processing and analysis modules will be added to the program. This document demonstrates the main functional modules in the software.

#### 3.2.1 Instrument parameter calibration



Figure 8 Main interface of the instrument parameter calibration tool

The program uses the standard sample to calibrate the instrument parameters including beam center and sample to detector distance. First, open the program's main interface SGTools (version 1.0); click the instrument calibration program **Ins Calibrate**, then enter the program's interface shown in Figure 8, and click ① import image data to import the scattering data of the standard sample.



1. Calibration of Beam center:

Figure 9 Fitting of the beam center

Fitting the beam center through the standard circle equation: right-click (2) beam center in Figure 9; click the lower-left corner (3) F1; select six points (small circles) on the scattering ring evenly, and then click (3) F1 again. The points are fitted with standard circle equations to obtain the center coordinates, and the system updates the results to the parameter (4) center of the incident beam.

**Correcting the beam center by line profile**: Click on the lower right corner of Figure 10 <sup>(5)</sup> FD, the program will automatically make an intensity profile along the line starting from the <sup>(6)</sup> beam center to the mouse cursor. The program will automatically find the peak within the defining range and displaying the peak order on the right panel. Fill in the number of the line profile in **line num** below the curve, and fill in the number of <sup>(9)</sup> scattering peak in **peak num**. Enter ENTER and select six line profiles in different directions to correct the beam center.



Figure 10 Correction of the beam center

#### 2. Calibration of the sample to detector distance SD:

**Basic parameter input**: Right-click **SD Cal**; click ① SP in Figure 11 to obtain the corrected beam center position; input energy ② E and ③ detector resolution **pixel** size, for example, MARCCD165 is 79 um, PILATUS is 172 um.



Figure 11 Instrument parameter calibration interface

Calculate SD by the standard d value: There are two methods to calculate SD. The first is to use a single standard peak. This requires to determine the order of the standard peaks. The coordinate position of the standard peak is also determined by the intensity curve from the line profile. Click ④ FD in Figure 12 to define the line profile direction and range, and fill in the line number below the curve in ⑤ line num, fill in the number of scattering peak in ⑥ peak num; input ENTER will update the position of the scattering peak to the textbox ⑦, fill in the interplanar distance of the standard sample into ⑧ d. click ⑨ CAL, the system will calculate the sample to detector distance ⑩ SD, click save to save the data, and complete the instrument parameter calibration.



Figure 12 Calibration of the Sample to detector distance

#### 3.2.2 Data processing

Open SG Tools (version 1.0); click on BP\_SAXS view to enter the BPSaxs\_view page.

### (1) Basic parameter setting

**Instrument parameter import:** Click ① **Set-up** option box in Figure 13 and select to enter the **Parameter-Set** panel. Click ②**Load** to import the parameter file obtained from the calibration module and ③**Set** to confirm the parameter settings. The related parameters can also be manually input. Click the ③ **set** button to confirm and save as a new parameter file with the ④ **save** button.



#### Figure 13 Interface of instrument parameter sets

**Data files import**: Click the <sup>(5)</sup> **Add** option in Figure 14 to import the data image which will be processed. Press and hold **ctrl** to select multiple files. After the files are imported, the **File list** will display the name of imported files, and the delete button is used to delete imported files from the file list. The supported input formats for 2-dimensional data are \*.tif as used by the MAR-CCD and pilatus detector, as well as some common formats like Bitmap (\*.bmp), 8-bit and 16-bit TIFF (\*.tif), and \*.png. The supported input data formats for 1-dimensional data are columnar ASCII in EXCEL format (\*.csv), Fit2D-format (\*.chi), raw data format (two columns without

header information, \*.txt), and reduced data format used in SGTools(\*.sect,\*.cake). Scattering patterns display: Click to select a file in the file list, and click <sup>(6)</sup> Plot to display the image. The <sup>(7)</sup>Previous and <sup>(8)</sup>Next options are used to plot the previous and next data file in the file list. The <sup>(9)</sup> PowerPoint button automatically displays the scattering data in the file list, There are five seconds pause between the displayed scattering data. Selecting the image range through mouse cursor for zoom. If the <sup>(10)</sup> keep zoom option is on, the next scattering pattern will be plotted in the defined range. In addition, the <sup>(11)</sup> Image Log option is the logarithmic output of the scattering pattern, and the <sup>(12)</sup>3D option is the three-dimensional display of the scattering



Figure 14 Data import and scattering pattern display

#### (2) Composite image of 2D scattering patterns

In Figure 15, click **Composite Image** in the ① **Multi-image** to create the graph for plotting the composite image. You can set the number of rows and columns through the parameter panel. The line spacing and column spacing can also be defined. After confirming the parameters and clicking OK, the **Figure** including multiple axes appears in the graph; click the ② **UP** icon in the **BPSaxs\_view** interface, and the scattering pattern shown in the main interface is copied into the ③ **Figure**, the scattering patterns are updated to the composite image in turn. Click ④ File option to save the image and



Figure 15 Interface of 2D scattering pattern output

define parameters such as graphic format and output resolution. The output format of composite image are standard graphic format(\*.tif,\*.bmp,\*.jpg,\*.pdf,\*eps). In addition, the Format\_TR tool are now developing to convert 1D reduced data format between different software for data analysis.



(3) Background correction

Figure 16 Interface of background scattering correction

According to the form of the sample, we can select the method of background

correction. A concentrated solution system is used to show how to use the background correction module. First of all, click the checkbox ① to turn on the background correction module in Figure 16, and then ② input the fore ionization parameter and back ionization parameter which could read from the ionization chamber file (\*.Ion). set the dark current of the detector into ③, and the scattering data of the blank sample is imported through the button ④, fore ionization parameter and back ionization parameter setting is finished, click ⑥ plot, the background will be automatically subtracted, and the scattering pattern after the deduction of the background will be displayed. The subsequent data reduction and analysis will



Figure 17 Output of background scattering image

use this background correcting scattering data. After clicking  $\bigcirc$ , the scattering pattern can be quickly saved, and the format and resolution of the pattern can be set in the textbox.

#### (4) Polarization reduction

For the small ange X-ray scattering data, the influence on the patterns due to polarization of incident radiation is very small. So it is considered to be negligible in data treatment. But it is very important for increasing the accuracy of the wide angle



Figure 18 Interface of Polarization correction

X-ray scattering data. First of all, click the checkbox (1) to turn on the polarization correction module in Figure 18. Then (2) input the polarization factor, for most of the synchrotron radiation is more than 0.95. Lastly, click the plot button (3) to apply the correction.



## (5) Creat mask

Figure 19 Interface of the creating mask tool

In order to eliminate the unwanted regions for data processing, such as module-gap in Pilatus detector, X-ray beam-stop, and spurious data points, the create mask tool is developed for users to define the mask area. There are four types of region shape (ellipse, rectangle, polygon, freehand) for users to create the mask area in the image. It should be noted that the process of defining mask region is only available in the raw image plot mode. But as the mask is created, it can be effective in any other plot modes.

First, click the checkbox ① to turn on the Mask tool in Figure 19, select the shape for defining the mask region ②, four types of shape (Ellipse, Rectangle, Polygon, Freehand) were provided. If you want to delete the the current shape, you could Right click the shape, and select the delete item ⑧ to remove the shape. Click the Add button ③, move the mouse pointer to the image and select the area wanted to mask(note: keep the left key of the mouse down when defining the mask area). Then click the Create mask button ④ to create the mask, and the mask area was shown in the figure with red color. The defined mask can also be saved in a mask file (\*\_mask.mat) through click the save mask button ⑤. And click the load button⑦to load the mask file for avoiding creating mask repeatly in next time data processing. If you want to clear the current mask, click the reset button⑥.

#### (6) Data reduction

The main methods of data reduction for scattering patterns are data integration and projection. The data integration could be divided into three types according to the symmetrical characteristics of the scattering patterns: line integration, rectangular integration, and arc integration. For isotropic scattering systems, the scattering intensity is symmetrical around the incident X-ray beam center, the arc or sector integration is used to obtain the one-dimensional profile. For scattering systems with rotational symmetry, the commonly used data methods are polarization averaging or projection along the rotation axis. For scattering systems with a specific orientation, the available integrating method is the cutting of the selected interesting region. This is commonly used for GISAXS data integration. In the **Control Panel**, **Sect** is the line integration and rectangular integration modes, and **cake** is the arc integration mode.

## Line (Rectangle) mode:



Figure 20 Interface of data integration with the line method

Right-click on ① sect in Figure 20 to open the integration parameter setting interface. Tick ② Section to choose ③ Line (line integration) mode or ④ Rectangle (matrix integration) mode. In line integration mode, the⑥ Pixels-Intensity curve is obtained in the selected range which is defined by the starting and ending point of ⑤ line. The starting and ending coordinates of the integration line can also be adjusted precisely through the input box. In Rectangle mode, click the mouse to select the position of the left bottom and right upper for the rectangular range; the integration  $2 \theta$  (or q) -Intensity curve data can be obtained (show in the right panel). The data reduction results can be saved by the ⑦ save data button, and the directory for data saving is defined in the parameter Set interface.

#### Cake mode:

Arc integration is the main data reduction method for scattering patterns with central symmetrical characteristics. First, click the check box ① Cake integration function module. The integration range is defined by four parameters: inner diameter, outer diameter, starting angle, and ending angle. The parameters can also be modified in the ② text box, and the integration result is updated in real-time with the changing



of the parameters. The auto button in the panel is used to automatically realize data

Figure 21 Interface of data integration with cake method

integration according to parameters in the text box, the integration result is displayed in (4), and button (5) is used to save the integration result. When a large amount of data needs to be processed, the Bat-pro button (6) is used to batch process the data files in the list.

## 3.2.3 Data analysis



## (1) Correlation roughness analysis

#### Figure 22 Analysis interface of the correlation length

Resonance dispersion scattering is one of the important methods to analyze the correlation length of the surface interface. We can obtain critical correlation length by analyzing the attenuation of scattering peaks in the in-plane direction. First, click the

check box (1) to open the analysis module, and select the analysis area (2) by the left bottom and right upper position of the mouse cursor (3). Button (4) is used to automatically realize the data integration to the selected range of scattering patterns. The attenuation of the diffuse scattering peaks is analyzed by multiple integrations according to the defined number of integration steps and integration width. The correlation degree of the resonance scattering peak is displayed in (5). By selecting the  $q_y$  position of the intensity curve in which high-order scattering peaks is gradually disappeared, then apply button (6) to calculate the critical correlation length.

- (2) Structural order analysis
- (a). WH analysis



Figure 23 Fitting interface of the scattering peak

Based on the WH theory, the broadening of the scattering peak is determined by the dislocation factor and grain size. Therefore, it is not an effective method to obtain a dislocation factor just by analyzing the width of the scattering peak. The WH equation is an important characterization method for ordered structure materials by analyzing the multiple scattering peaks (high-order scattering peaks) in the scattering pattern, then dislocation factor and grain size parameter information are obtained at the same time. As shown in the figure, right-click ①PFIT to open the peak fitting module, click the

Fit button to import the fitting data and initialize the parameters, and baseline subtraction and peak shape selection are set through keyboard control (the ipf function used in this part comes from Matlab center, provided by Thomas C. O'Haver, etc., the detail description see Appendix 1), press Q to directly output the result to the text box ④, click button ⑥ to upload the fitting results to the table, The information of the processed data is shown in table⑦ after the fitting of scattering peaks are all finished in sequence.



Figure 24 WH fitting analysis interface

After completing the peak fitting, right-click the ORD to open the WH analysis module(Figure 24), select the scattering data used for WH analysis show in ①, input the analysis mode, peak order and x-rya energy applied to the analysis in ②, click the Fitting button to finish batch WH analysis of all scattering data. The results are shown in Figure ⑥ and undated into Table ①. Click button ④ to plot the dislocation factor and grain size as a function of the file number (shown in panel ③), and the range of the Y-axis can be set through textbox ⑤.

(b) Paracrystal model

The GUI interface is under development.

#### 3.2.4 Batch Processing function

The batch processing function is embedded into the program for several modules including data processing, display and output of two-dimensional graphics, background deduction, data integration, data storage, etc. The corresponding module has a Bat-pro option in the panel. More batch processing functions will be added in updated versions.

#### (1) Mapping of the intensity curve

Through analysis of the mapping of the scattering intensity as a function of time(temperature, strain), the dynamic change of the microstructure in a material can be effectively observed, which is useful for studying the mechanism of the dynamic process. The program imports the one-dimensional intensity curve obtained by the data reduction modules and then set the correlation between the intensity curve and time, and finally plot the one-dimensional scattering curve as a function of time.



Figure 25 Mapping of intensity curve as a function of time

As shown in the figure, click the Add button to add Import the list of data, then select the data to be imported, click ① load button to import the data, the data information is displayed in the list ③, including the data name, the q value range of the one-dimensional scattering data, etc., in ④ the method of data drawing can be selected, Mainly consists of waterfall, pcolor, Surface, multiline, etc., where the method of surface is a three-dimensional graphics display method. In ⑤, select the intensity to be linear or logarithmic. In ⑥, set the correlation between the intensity curve and

time(temperature, strain). If it is a linear change, you can customize the range of the time. If it is a non-linear change, you need to import the associated data. After completing the settings, click ⑦ plot to draw the mapping of the intensity curve, and the results will be displayed in <sup>(®)</sup>, and the graph can be saved as a variety of image format files.

(2) Create animation



Figure 26 Interface of animation creation

To observe dynamic structure information during an in-situ X-ray scattering experiment, the scattering patterns are synthesized into an animation. It is a favorable way to observe the evolution of structure in physical or chemical processes. First import the data file to list ①, select the data from the file list to be synthesized into animation, right-click the Movie button to switch to the animation synthesis window, and then click the check box ③ to open the parameter settings, animation name, frame name, frame rate, and other information, frame, click start button to proceed the animation generation, the synthesized is displayed dynamically in the graph of the main panel.

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## 5、Appendix

#### 5.1 Operation guide of the Ipf function % Keyboard Controls: % Pan signal left and right...Coarse pan: < and > % Fine pan: left and right cursor arrow keys Nudge: [] (one data point left and right) % % Zoom in and out.....Coarse zoom: / and ' % Fine zoom: up and down cursor arrow keys % Resets pan and zoom......ESC % Select entire signal......Crtl-A % Select # of peaks......Number keys 1-9, or press 0 key to enter manually % Complete peak shape menu... - (minus or hyphen), then type number and Enter % Select peak shape ......g Gaussian % Shift-G Fixed-width Gaussian % Shift-P Fixed-position Gaussians % h Equal-width Gaussian Shift-H bifurcated Gaussian (a,z keys adjust % shape) % 1 Lorentzian % ; Equal-width Lorentzian Shift-L Fixed-width Lorentzian % % Shift-[ Fixed-position Lorentzians Breit-Wigner-Fano (a,z keys adjust % Shift-B Fano factor) % o Lognormal distribution (See "S" for logistic function) % p Pearson (use a, z keys to adjust shape) % e exponentially-broadened Gaussian Shift-R ExpGaussian (var. tau) % % Shift-E Exponential-broadened Lorentzians j exponential-broadened equal-width Gaussian % % (a,z keys adjust broadening) % K doublegaussian peak % u exponential pUlse: y=exp(-tau1.\*x).\*(1-exp(tau2.\*x)) % Shift-U Alpha: y=(x-tau2)./tau1.\*exp(1-(xtau2)./tau1); % s Up Sigmoid (logistic function): y=.5+.5\*erf((x-tau1)/sqrt(2\*tau2)) Shift-D Down Sigmoid (logistic function): % y=.5-.5\*erf((x-tau1)/sqrt(2\*tau2))

%	~ Gauss/Lorentz blend (a/z keys adjust shape)
%	Shift-V Voigt profile (a/z adjusts shape)
%	Shift-T Triangular
%	Select other peak shapes by number: press - key to display menu.
%	Fitf perform single Fit from another start point.
%	Select BaselineMode modet selects one of 5 available baseline corrections
%	Monopolar/bipolar mode= + Flips between + peaks only and +/- peak mode
%	Toggle log y mode OFF/ONm Plot linear or log Y axis on lower graph
%	2-point Baselineb, then click left and right baseline
%	Set manual baselineBackspace, then click baseline at multiple points
%	Restore original baseline\ to cancel previous background subtraction
%	Click start positionsc, click on peak position for each peak
%	Type in start vectorC (Shift-C) Type or Paste start vector [p1 w1 p2 w2]
%	Enter value of 'extra'Shift-x, type value, press Enter.
%	Adjust 'extra' up/downa,z: 5% change; upper case A,Z: 0.5% change.
%	Print parameters & resultsq
%	Print fit results onlyr
%	Plot signal in figure 2y
%	save model to Diskd Save model to disk as SavedModel.mat.
%	Refine fitx Takes best of 10 trial fits (change in line 224)
%	Print peakfit functionw with all input arguments
%	Compute bootstrap statsv Estimates standard deViations of parameters.
%	Test effect of Noisen Test effect of Noise by fitting subset of points
%	Save Figure as png fileShift-S Saves as Figure1.png, Figure2.png
%	Display current settingsShift-? displays table of current values
%	Prints list of commandsk
%	Switch to iPeakShift-Ctrl-P transfer current signal to iPeak.m
%	Switch to iSignalShift-Ctrl-S transfer current signal to iSignal.m
%	Fit polynomial to segmentShift-o Asks for polynomial order
%	Enter minimum widthShift-W
%	Enter saturation maximumShift-M
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https://terpconnect.umd.edu/~toh/spectrum/ifpinstructions.html

# 5.2 Information of Standard samples

Calibranta	Longth goals
Calibrants	
silver behenate	d = 58.376  Å
silver stearate	d = 48.68  Å
rat collagen	Wet: 668 Å - 670 Å, Dry: 620 Å - 650 Å.
silica spheres	$278 \pm 4$ Å; 1360 Å
polystyrene spheres	$250 \pm 10$ Å; $1015 \pm 22$ Å

1. Periodic Calibrants for SAXS

## 2. Periodic Calibrants for WAXS

(1) LaB<sub>6</sub> (34-0427), E=10 Kev (1.24 Å for 20 plot)



Peak List	hkl			d (Å)	Ι
1	1	0	0	4.1577	54
2	1	1	0	2.9392	100
3	1	1	1	2.3998	41
4	2	0	0	2.078	22
5	2	1	0	1.8586	46
6	2	1	1	1.6969	24
7	2	2	0	1.4697	8
8	3	0	0	1.3853	23

9	3	1	0	1.3144	16
10	3	1	1	1.2533	10
11	2	2	2	1.2	2
12	3	2	0	1.1529	6
13	3	2	1	1.111	13
14	4	0	0	1.0393	2
15	4	1	0	1.0082	8
16	4	1	1	0.9798	7
17	3	3	1	0.9536	3
18	4	2	0	0.9295	4
19	4	2	1	0.9071	9
20	3	3	2	0.8862	3
21	4	2	2	0.8485	3
22	5	0	0	0.8314	3
23	5	1	0	0.8152	10

(2) CeO<sub>2</sub> (34-0394), E=10 Kev (1.24 Å for 2 $\theta$  plot)

Peak List	hkl			d (Å)	Ι
1	1	1	1	3.1234	100
2	2	0	0	2.7056	30
3	2	2	0	1.9134	52
4	3	1	1	1.6318	42
5	2	2	2	1.5622	8
6	4	0	0	1.3531	8
7	3	3	1	1.2415	14
8	4	2	0	1.2101	8
9	4	2	2	1.1048	14
10	5	1	1	1.0415	11
11	4	4	0	0.9566	4
12	5	3	1	0.9147	13
13	6	0	0	0.9019	6

14	6	2	0	0.8556	9
15	5	3	3	0.8252	6
16	6	2	2	0.8158	5

(3) Y<sub>2</sub>O<sub>3</sub> (89-5592), E=10 Kev (1.24 Å for  $2\theta$  plot)

1	2	e	)

Peak List	hkl			d (Å)	Ι
1	2	0	0	5.298	3
2	2	1	1	4.3258	130
3	2	2	0	3.7463	1
4	2	2	2	3.0588	999
5	1	2	3	2.8319	5
6	4	0	0	2.649	247
7	4	1	1	2.4975	49
8	4	2	0	2.3694	11
9	3	3	2	2.2591	55
10	4	2	2	2.1629	9
11	1	3	4	2.0781	79
12	1	2	5	1.9346	25
13	4	4	0	1.8731	386
14	4	3	3	1.8172	20
15	6	0	0	1.766	4
16	6	1	1	1.7189	47
17	0	2	6	1.6754	10
18	5	4	1	1.635	36
19	6	2	2	1.5974	251
20	6	3	1	1.5623	54
21	4	4	4	1.5294	41
22	5	4	3	1.4985	18
23	0	4	6	1.4694	11
24	7	2	1	1.4419	28
25	6	4	2	1.416	13

26	1	5	6	1.3457	17
27	8	0	0	1.3245	36
28	8	1	1	1.3043	30
29	8	2	0	1.285	16
30	6	5	3	1.2665	17
31	8	2	2	1.2488	11
32	8	3	1	1.2318	27
33	6	6	2	1.2155	59
34	7	5	2	1.1998	1
35	0	4	8	1.1847	38
36	8	3	3	1.1701	8
37	8	4	2	1.1561	9
38	9	2	1	1.1426	18
39	6	6	4	1.1295	1
40	1	5	8	1.1169	18
41	7	6	3	1.0929	19

## 5.3 Reduced data format(.rect, .cake, .slice )

Position:

19.4007, 76.3845 0.508906, 1.89851 Cake Type: Radial q(nm^(-1)) Intensity error 0.50891,197.53,8.3028 0.51095,193.22,8.1297 0.51299,191.23,8.3436 0.51503,188.07,8.8276 0.51707,184.03,8.5573 0.51911,181.38,8.5896 0.52115,179.13,8.4701 0.52319,176.68,8.626 0.52523,173.27,8.8043 0.52727,170.67,8.1858 0.52931,168.17,8.1153 0.53135,166.5,7.8701 0.53339,163.83,7.8107 0.53543,160.83,7.978 0.53747,158.55,7.6614 0.53951,157.16,8.0319 0.54155,154.32,8.0963