Tutorial and Brief manual of

GISAXShop

Version 4.X

Byeongdu Lee

Advanced Photon Source
Argonne National Laboratory
blee@aps.anl.gov
1-630-252-0395

Contents

Note on the use of GISAXShop

References for definitions of the scattering vector & angles

References for GISAXS

Installing GISAXShop

Symbols and Terms

Displaying GISAXS image data

Background subtraction

Toolboxes

Note on the use of GISAXShop

GISAXShop has been developed for a personal use purpose. It might still have lots of bugs, inconveniencies, and some mistakes. Error report would be appreciated.

Some of NIST codes (http://www.ncnr.nist.gov/programs/sans/data/red_anal.html) are used with or without modification. Dr. Dong Ryeol Lee provides valuable comments at the beginning stage of software development.

GISAXShop is not for the use of commercial purposes. Free for academic and research purposes. While user can modify and redistribute GISAXShop, it is recommended to send a modified version to B. Lee to keep a consistent version.

References for definitions of angles and uses of GISAXShop.

B. Lee, C.-T. Lo, P. Thiyagarajan, D.R.Lee, Z. Niu, Q. Wang "Structural characterization using the multiple scattering effects in grazing incidence small-anlge X-ray scattering", *J. Appl. Cryst*. Accepted (2007).

B. Lee, I. Park, H. Park, C.-T. Lo, T. Chang, R. E. Winans "Electron density map using multiple scattering in grazing-incidence small-angle X-ray scattering", *J. Appl. Cryst*. **40**, 496-504 (2007)

B. Lee, C.-T. Lo, S. Seifert, R. E. Winans, "Silver behenate as a calibration standard of grazing-incidence small-angle x-ray scattering", *J. Appl. Cryst*. **39**(5), 749-751 (2006)

B. Lee, S. Seifert, S. J. Riley, G. Tikhonov, N. A. Tomczyk, S. Vajda, and R. E. Winans, "Anomalous grazing incidence small-angle x-ray scattering studies of platinum nanoparticles formed by cluster deposition", *J. Chem. Phys*. **123**(7), 074701-074707 (2005)

B. Lee, I. Park, S. Park, J. Yoon, J. Kim, K.-W. Kim, T. Chang, and M. Ree, "Structural Analysis of Block Copolymer Thin films with Grazing Incidence Small Angle X-ray Scattering", *Macromolecules* **38**(10), 4311-4323 (2005)

B. Lee, J. Yoon, W. Oh, Y. Hwang, K. Heo, K. S. Jin, J. Kim, K.-W. Kim, and M. Ree, "In-situ Grazing Incidence Small Angle X-ray Scattering Studies on Nanopore Evolution in Low-k Organosilicate Dielectric Thin Films", *Macromolecules* **38**(8), 3395-3405 (2005)

Some references for GISAXS

Book/Reviews – may be good for metal/metal oxide/alloy researcher:
R. Lazzari, Manual of IsGISAXS
(http://www.insp.upmc.fr/axe2/Oxydes/IsGISAXS/isgisaxs.htm)

U. Pietsch, V. Holy, and T. Baumbach, "High-resolution x-ray scattering; from thin film to lateral nanostructures", Springer, 2004.

M. Schmidbauer, "X-ray diffuse scattering from self-organized mesoscopic semiconductor structures" Springer, 2004.

Theories:
Vineyard, G. H. *Phys. Rev. B* **26**, 4146 (1982)

S. Sinha *et al. Phys. Rev. B* **38**, 2297 (1988)

M. Rauscher *et al. Phys. Rev. B* **52**, 16855 (1995)

B. Lee *et al*. *J. Appl. Cryst*. **40**, 496-504 (2007) – Dynamic diffraction theory/phase determination of diffraction peaks

Metallic particles:
G. Renaud *et al*. *Science* **30**, 300, 1416 – 1419 (2003)/Phys. Rev. B 69, 035411 (2004)

M. Rauscher *et al*. *J. Appl. Phys*. **86**(12), 6763 (1999)

D. Babonneau *et al*. *Phys. Rev. B* **71**, 035430 (2005)

Nanostructures in films:
- Nanoparticles in or on a film
D. Babonneau *et al*. *J. Appl. Cryst*. **32**, 226-233 (1999) – Read to know whether your scatterer is on a film or in a film.

S. Narayanan *et al*. *Phys. Rev. Lett*. **94**, 145504 (2005) – particle dynamics in a film/standing wave effects

- Pores in a film
B. Lee *et al*. *Nature Mater*. **4**(2)**,** 147–150 (2005) – Pores in a film on a substrate

Y. Wang et al. *J. of Phys. Chem. B* **2007**, DOI 10.1021/jp0679212, ASAP article – scatterings from the surface of a film or structures in the film.

- Block copolymer
Detlef-M. Smilgies – Decent summary and introduction of GISAXS
http://staff.chess.cornell.edu/~smilgies/gisaxs/GISAXS.php

B. Lee *et al*. *Macromolecules* **38**(10), 4311-4323 (2005) – Intuitive explanation on the meaning/use of DWBA / Qualitative/quantitative application of GISAXS on thin films of a block copolymer.

P. Busch *et al*. *Macromolecules* **40**(3) 630-640 (2007) – block copolymer film (Lamellar morphology)

G. E. Stein *et al*. *Macromolecules* **40**(7) 2457-2460 (2007) – Block copolymer film (Spherical morphology) / full calculations of GISAXS patterns of various crystal packings of nano-spheres.

Installing GISAXShop

1. Make a directory (“GISAXShop”) under “..\Igor Pro Folder\Igor Procedures\”.

2. Make a directory (“GISAXShop”) under “..\Igor Pro Folder\Igor User Procedures\”.

3. Unzip the downloaded file, copy files in “Igor Procedures” procedures to “..\Igor Pro Folder\Igor Procedures\GISAXShop” and those in “Igor User Procedures” procedures to “..\Igor Pro Folder\Igor User Procedures\GISAXShop”.

Symbols or terms

DBx and DBy : Coordinates of a direct beam X and Y
Waveln : the wave length
Q : Capital Q's in any place always mean total q, which is $\sqrt{(q_x^2 + q_y^2 + q_z^2)}$

$\mathbf{q_t}$: the scattering vector q defined by the direct beam without any refraction, $\mathbf{q_t}$ =(q_x, q_y, $q_z = q_{t,z}$), where $q_{t,z} = k_{z,f} - k_{z,i}$
$\mathbf{q_r}$: the scattering vector q defined by the reflected beam, $\mathbf{q_r}$ =(q_x, q_y, $q_z = q_{r,z}$),where $q_{r,z} = k_{z,f} + k_{z,i}$

$q_{t,z}$ for the embedded : refraction corrected $q_{t,z}$.
$q_{r,z}$ for the embedded : refraction corrected $q_{t,z}$.

Displaying GISAXS image data

When GISAXShop is installed properly, new menu “BESSRC” will show up on the main menu line of Igor as shown in Fig. 1

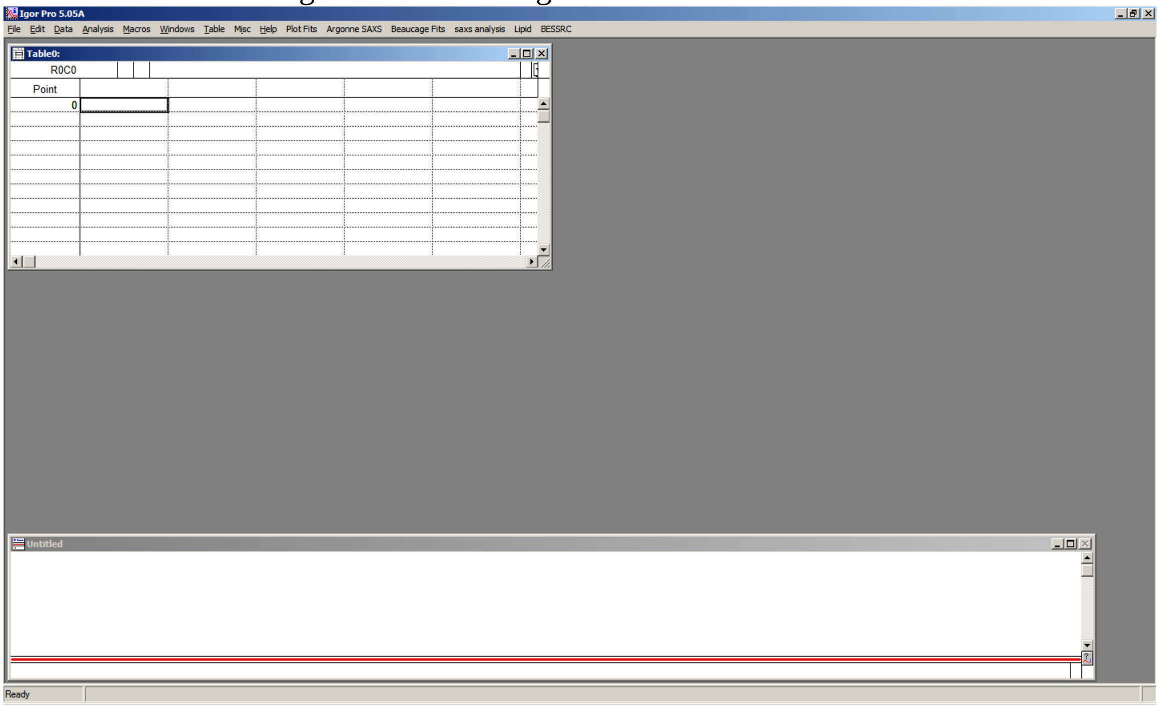

Fig.1 Starting screen

To start GISAXShop, click “BESSRC” and “Load GISAXS” under “BESSRC” as shown Fig. 2

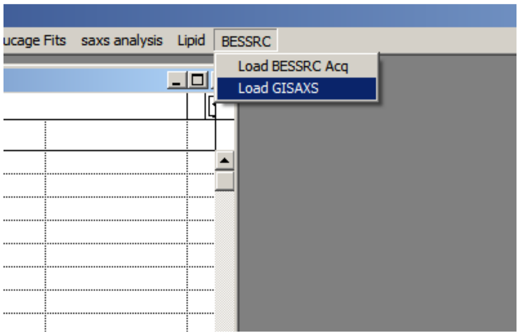

Figure 2. Menu list on “BESSRC”

Once “Load GISAXS” run, Igor will load all GISAXShop procedures located under “.. > User Procedures > GISAXShop” directory into Igor memory and generate new menus, two sub-menus, “Unload GISAXS” and “Start GISAXS shop”, under “Load GISAXS” and one main-menu, “Image”, next to “BESSRC”. “Image” is one of built-in Igor software packages, which might be good to be explored by User.

GISAXShop is utilizing the “image” package to make a line cut, which is why the menu is shown up automatically. “Unload GISAXS” will unload the loaded GISAXShop procedures and “Start GISAXS shop” will start GISAXShop as expected.

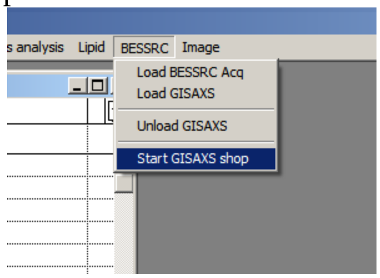

Figure 4. Starting GISAXShop

Fig. 5 is the starting screen of GISAXShop, of which base frame is adopted from old version of NIST SAXS software. If user is using the SANS software currently, some macros might interfere with those in GISAXShop, which can be avoided by unloading SANS software while GISAXShop is running or vise versa.

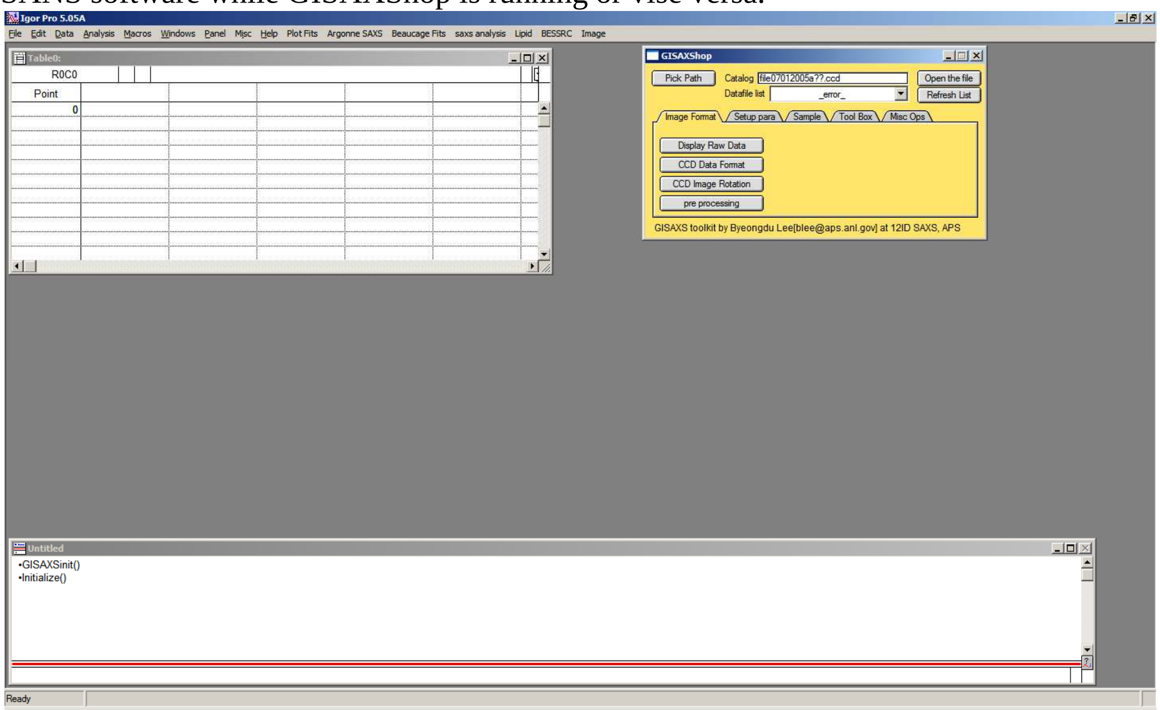

Figure 5. Starting screen of GISAXShop

The first step of using GISAXShop is to select “CCD data format” on “Image Format” tab. Currently, gold CCD [BESSRC-CAT/APS], BESSRC MarCCD [Mar 165, BESSRC-CAT/APS], Princeton CCD [*.spe, Roper Scientific], and TIFF [typical MarCCD format] are image formats included in GISAXShop. While some other specific formats like EDF (from D. Babonneau’s code, david.babonneau@lmp.univ-poitiers.fr), IMM, Mar image plate (from Jan Ilavsky’s code, ilavsky@aps.anl.gov) are included, they are not fully served yet. Users can load their images of arbitrary formats with “User” format: The binary image format has to be known priorly.

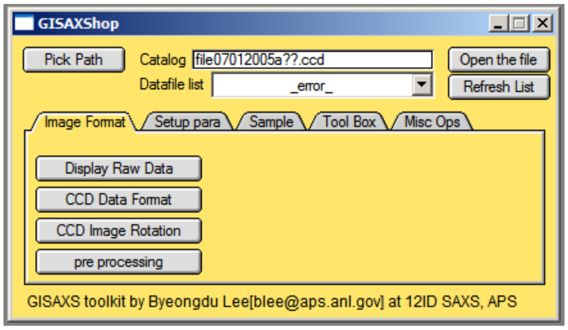

Figure 6. Main panel

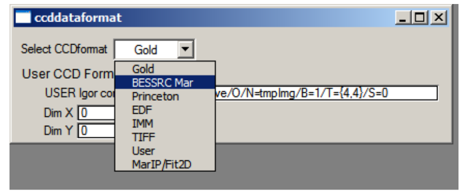

Figure 7. CCD data format

Main panel of GISAXShop has 5 tabs with some buttons on each tab. While the buttons on tabs are for conditioning and reducing data, the other buttons on the main panel, "*Pick Path*", "*Open the file*", and "*Refresh List*" are for loading series of image data. Before trying these buttons, let's click "*Display Raw Data*" button on "*Image format*" tab. Then, you will see typical GUI for selecting a file.

Let us assume you have image files under "C:\data\" and select a file named "Ag2000VVac6cAlO.00249.008" [at 12ID-C at APS, Ag2000VVac6cAlO, 249, and 8 are called the *file name*, *file index* and *file extension*, respectively]. New window "*RAW*" displaying the image will pop up with its full filename on it. If you click "*File->Catalog*" button, the filename will be transferred to "*Catalog*" list box on the main panel. The next button is "*isLin*", which change linear scale image to log one and itself to "*isLog*". "*isLog*" will do reverse. User can go back and forth with this button. The button "*Aspect 1:1*" makes the aspect ratio of an image 1 to 1. Please note that GISAXShop is assuming the length X and Y of a pixel equal, or a square pixel. The rectangular pixel is not considered. The button ">" and "<" is for quick data searching and will be presented later.

GISAXShop uses the Cartesian coordinate system to describe GISAXS image, while the Polar coordinate system is common in SAXS. This is because GISAXS appears distinct either along the out-of-plane or in-plane directions, which are orthogonal to each other. Direction of the in-plane is presumed to be parallel to X, which is a horizon in "*RAW*" window (if it is not, an image can be rotated so whenever it is loaded. See "*CCD Image Rotation*" button on "*Image Format*" tab on the main panel). Coordinates of mouse are presented by five different ways: Geometric coordinates [Geo. crd block : Pixels (X, Y) and angles (tth_f, a_f) from a center], Q coordinates based on the transmitted beam [Trans. Beam block] and the reflected beam [Refl. Beam block], and Refraction corrected Q [Refrac. q block], and finally CCD pixel itself [X, Y, counts at (X, Y)]. Q defined by the transmitted beam will be designated as $\mathbf{q_t}=(q_{xy}, q_{t,z})$ and that by the reflected one as $\mathbf{q_r}=(q_{xy}, q_{r,z})$. The refraction correction is only applied to z component of the scattering vector $\mathbf{q_t}$ and $\mathbf{q_r}$, and the corrected q_z components will be designated as q_{t,z_ref} and q_{r,z_ref}. The capital Q_t, Q_r , $Q_{t,ref}$, and $Q_{r,ref}$ stand for total q's, or $|\mathbf{q_t}|$, $|\mathbf{q_r}|$, $|\mathbf{q_{t,ref}}|$, and $|\mathbf{q_{r,ref}}|$, respectively. Note that *q* definitions in the "Trans. Beam" block are common definitions for general scattering. And, in GISAXShop, q_x is parallel to x-ray beam, q_z and q_y is normal to it, and q_z is normal to the substrate plane (see references, Byeongdu Lee's papers).

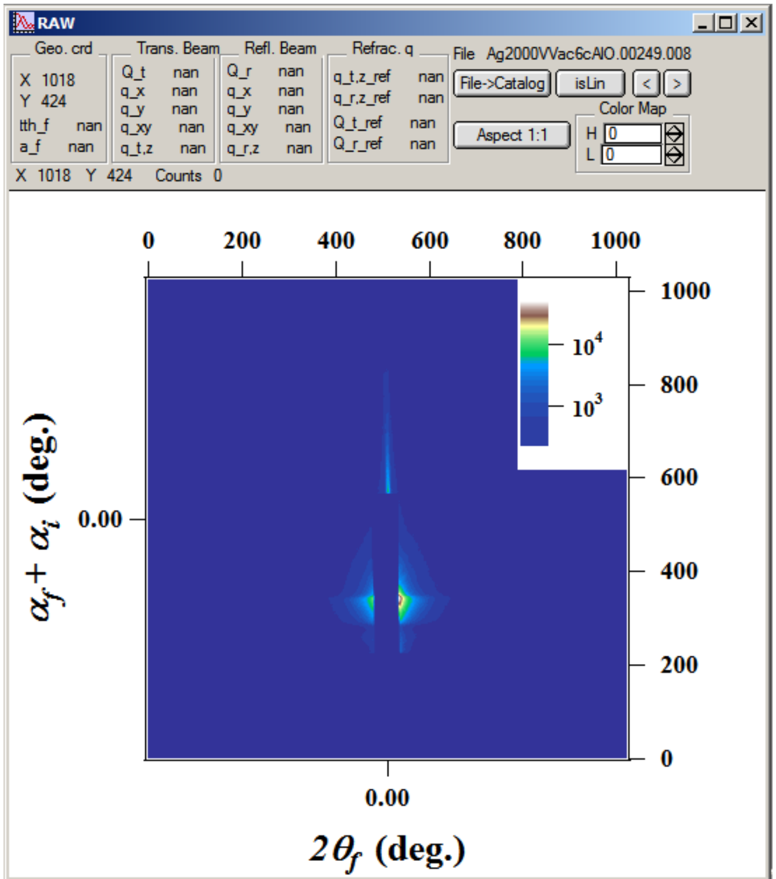

Figure 8. Window for displaying GISAXS image data.

Once a GISAXS image is displayed, user can precede next step: *Setting experimental parameters*. Click second tab in the main panel. Two ways of making the experimental parameters are available. Firstly, if you know setup parameters such as Pixel size of CCD and Sample to detector distance (SDD) exactly, click "*Q from Geometry*" button and put the values with X-ray energy, Beam center (DBx, DBy), the other parameters will be automatically calculated. Secondly, if you measured a standard sample like "silver behenate", one could click "*Q from Standard*" and then need to put following two values: 1) the position of first order diffraction ring of the standard sample, *pixel distance from a beam center*, and 2) Q value associated to the peak. There is a tool box to help to determine the pixel distance in GISAXShop (see Tool Box tab).

When GISAXS images are taken at 12ID BESSRC-CAT at APS, some parameters including an incident angle can be read directly from the header of images.

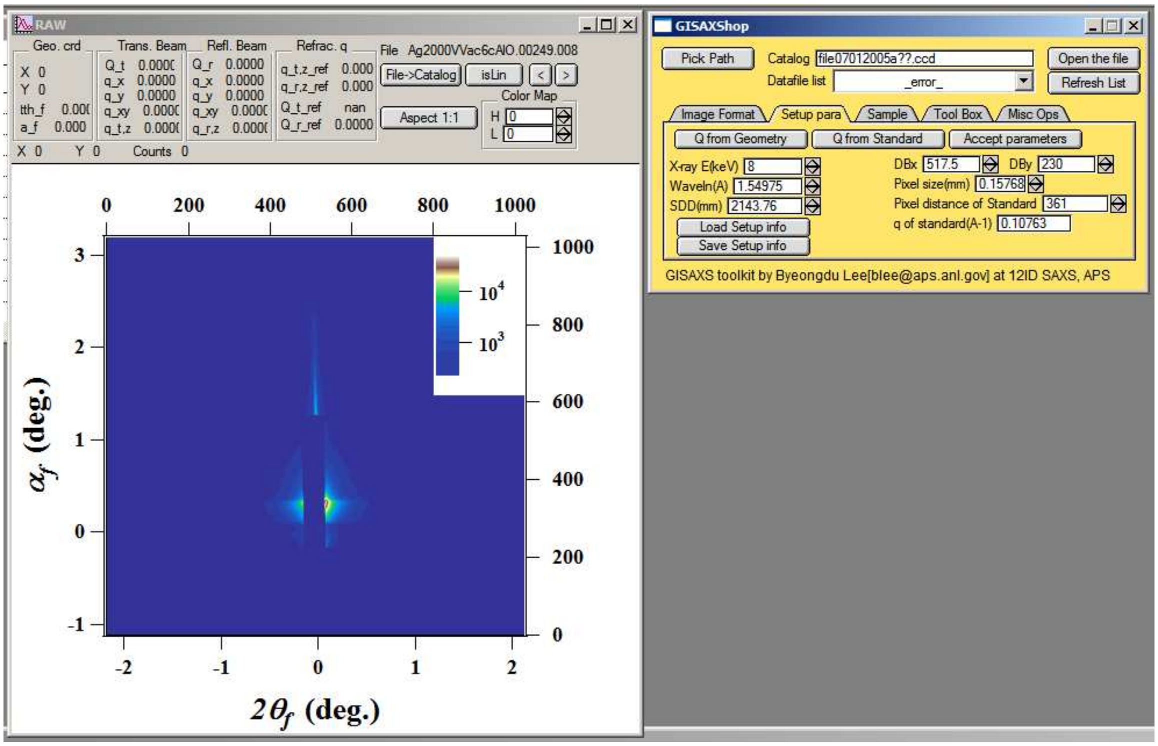

Figure 9. Experimental setup.

In the "*Sample*" tab, user can input some other parameters, which might be changed for each image data, such as the incident angles. When the sample is so-called 2D powder, tth_i should be set 0. When the scattering on a displayed image is generated from particles on a film or substrate, don't assume refraction of x-ray beam (practically, this is a good approximation, because x-ray will enter into the particle with various angles) and set "*E-density of film*" and "*beta of film*" 0. If you are studying the diffraction or scattering from structures embedded in a film or substrate, you may want to consider the refraction by the film or substrate and need to provide those values. Refraction corrected q_zs are designated as q_{t,z_ref} and q_{r,z_ref}. As were for a film, "*E-density of substrate*" is not really necessary to be entered. It is mainly for checking sample alignment, for example, you can compare the calculated position of Yoneda wing and the measured on your image. This will be mentioned later in details when "*Drawing toolbox*" is introduced.

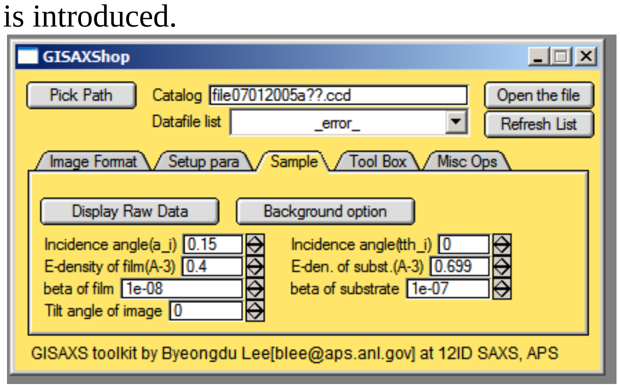

Figure 10. Sample tab.

“*Tilt angle of image*” is for compensating the image rotation along chi angles. In many times, GISAXS stage has only one circle, which is defined as alpha for the incident angle, and does not have a way to correct chi. Thus, Yoneda wing on image data is not parallel to the horizon and could be tilted by small amount. GISAXShop would not rotate image by the amount. Instead, it will rotate Cartesian coordinate.

Until now, image loading and parameter settings in GISAXShop have been explained. From now on, some convenient loading function will be introduced. Basically, it is to make a list of filenames of interest with multiple uses of wild card *.

First, click “*Pick Path*” in the main panel to select a working directory where images are achieved. Then, you can use wild card (*) to list filenames containing the word in the catalog box. You can use several wild cards, for example, “*Vac*.*.001”. After putting the template filename with wild cards, click “*Refresh List*” and then all names of files containing the template filename will be loaded on to the “*Datafile list*”. By selecting one of the filenames listed (see Fig. 13), you can pick, load, and display the file onto “*RAW*” window. Then, you can use “>” and “<” button to load next and previous data in the list.

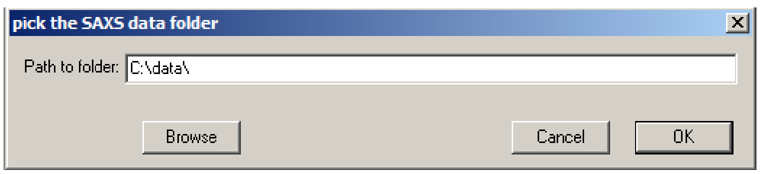

Figure 11. Pick Path

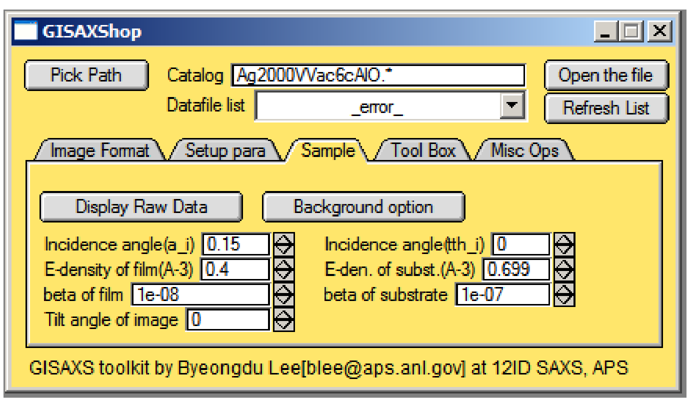

Figure 12. Use of the wild card *.

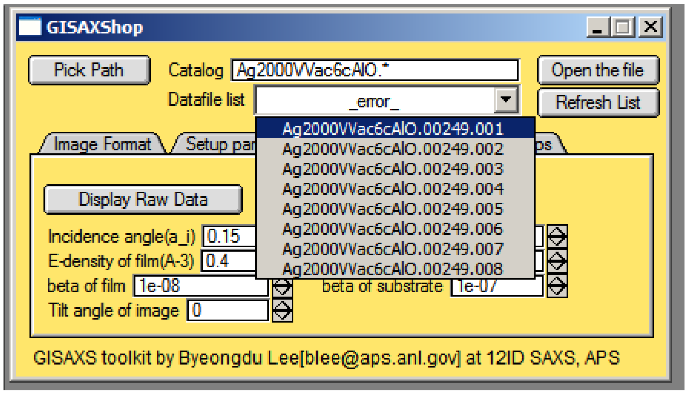

Figure 13. Datafile list

Background subtraction

There are several ways of background subtraction in GISAXShop. Let's first define what kinds of background are. First, dark current of a detector or an offset value can be considered the background. One, then, needs ways to subtract a certain value from a whole image or a dark current image in a pixel by pixel manner. Second, scattering originated from anything other than sample is the background in its typical definition. One can get the background pattern by measuring a pattern without any sample. In this case, it is assumed that changes of the direction of direct beam (caused by the reflection at the sample position: it might hit windows and a beam stop at different locations) do not cause any difference. This is a good approximation when the background is very diffuse and mostly coming from air or windows located upstream of a sample. In principles, there should be no background sources at downstream of a sample for a perfect background subtraction for GISAXS. One might want to use scattering from a substrate as a background for the particles on top of the substrate. In this case, one has to be careful to check whether the substrate scatterings vary sample by sample or condition by condition. (See ref. D. Babonneau et al. *J. Appl. Cryst*. **1999**, *32*, 226.; Y. Wang et al. *J. of Phys. Chem. B* **2007**, DOI 10.1021/jp0679212; B. Lee *et al. J. Chem. Phys.* **2005**, *123*, 074701)

To be able to subtract such backgrounds, GISAXShop provides two methods for subtraction and one for normalization: Constant value [Option 1] and image with certain scaling factors [Option 2] could be subtracted. After the subtraction, GISAXS image could be normalized by either I0 (counter 1) or Transmitted intensity (counter 2) [currently only for BESSRC data file format].

Click the button "*Background option*" in the sample tab on the main panel of GISAXShop to pop up the window shown in Fig. 14. It is straightforward to use of Option 1. To use Option 2, user has to define a background image prior to use. If user wants to use the offset subtracted image as a background, load the image with Option 1, click "*Copy Current image as Background Image*" button, and put the scaling factor. If the scale factor is a real value larger than 0, the background will be scaled by the value. Otherwise one can use -1, -2, and 0 for using count1, count2 values in MarCCD header (only for BESSRC-CAT data), and averaged value in the region of interest (ROI) that can be defined manually by putting pixel coordinates or using marquee tool on "*RAW*" window. The last method, defining ROI for determining a scale factor, is useful when GISAXS image contains sort of reference scattering, for example, circular ring from kapton windows. Move mouse on GISAXS image in "RAW" window, click left mouse button and drag to make a rectangular marquee, click right mouse button on the marquee, and choose "*PutROIforGISAXSBackground*". Then, the selected coordinate will be transferred to number boxes in the Option 2. If you like to see the averaged value in the ROI, click "*Calculate ROI average of BG*" button and find the displayed results on IGOR command window. Option 3 in GISAXShop is for the normalization (only for BESSRC-CAT data).

These options are sequentially applied. Following is a mathematical formula for this:

$$I_{sub} = ((I - I_c) - s*(I_b-I_c))/I_{v(x)}*I_m,$$

where I_c and s are the values in Option 1 and Option 2. if *s* (scale factor) is either -1 or -2, $s=I_{v(x)}/I_{v(x),b}$, where *x* is the number of a counter (1 or 2) and $I_{v(x)}$ denote the value of the counter x saved in the header of MarCCD. I_m is a constant factor to be multiplied. I, I_b, and I_{sub} are images of sample, background, and subtracted, respectively.

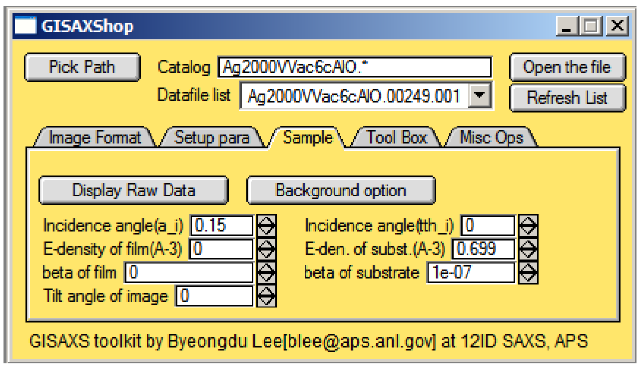

Figure 14. Background option

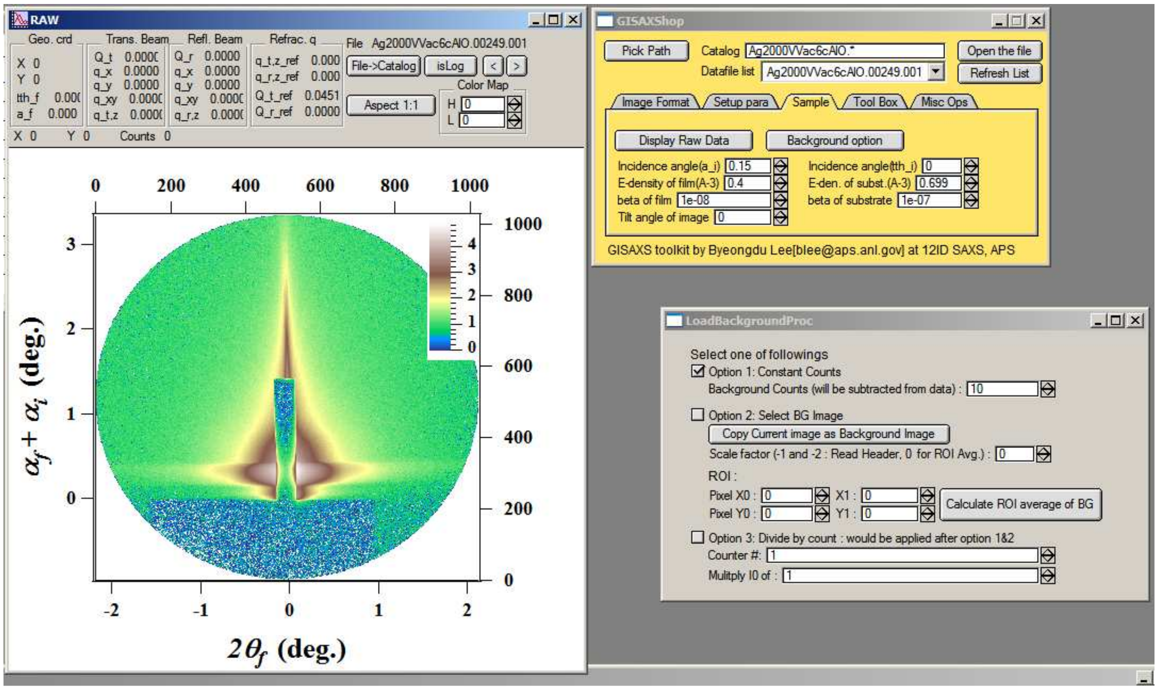

Figure 15. Background subtracted image.

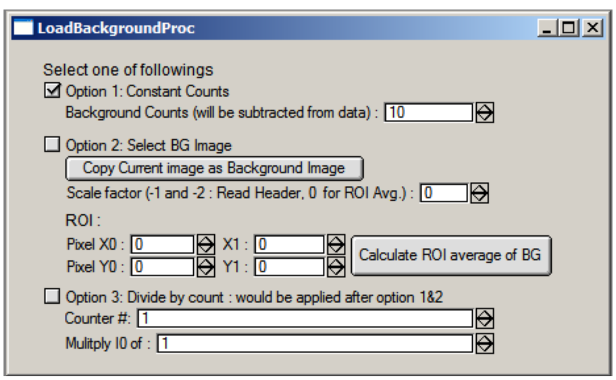

Figure 16. Background subtraction.

Toolboxes

Several toolboxes are available in GISAXShop, which are accessible from "*Tool box*" tab on the main panel.

1. Drawing toolbox

This toolbox is useful for seeing diffraction patterns (from samples like block copolymer, silver behenate … see Fig. 18) [see "*Draw a circle*"] and checking the critical angles of samples ["*Mark a_f = 0 line*", "*Mark specular reflection*", "*critical angle of film*", "*critical angle of substrate*"]

User can draw contour plots of **q** spaces (this can be quite slow at low performance machine): "*Q image Calc*" → "*Put Q contour*" (after select a *q* component to be plotted), "*Q image Remove*", "*Clear Drawing*".

"*Equi Q line*" button will draw a line, which is so-called Debye-Scherror ring. Rings of $|\mathbf{q_t}|$ and $|\mathbf{q_r}|$ will be plotted with different colors.

"*Put a spot*" is useful to point a diffraction peak. Try these two buttons and notice that incident angle should be correctly provided to use these functions.

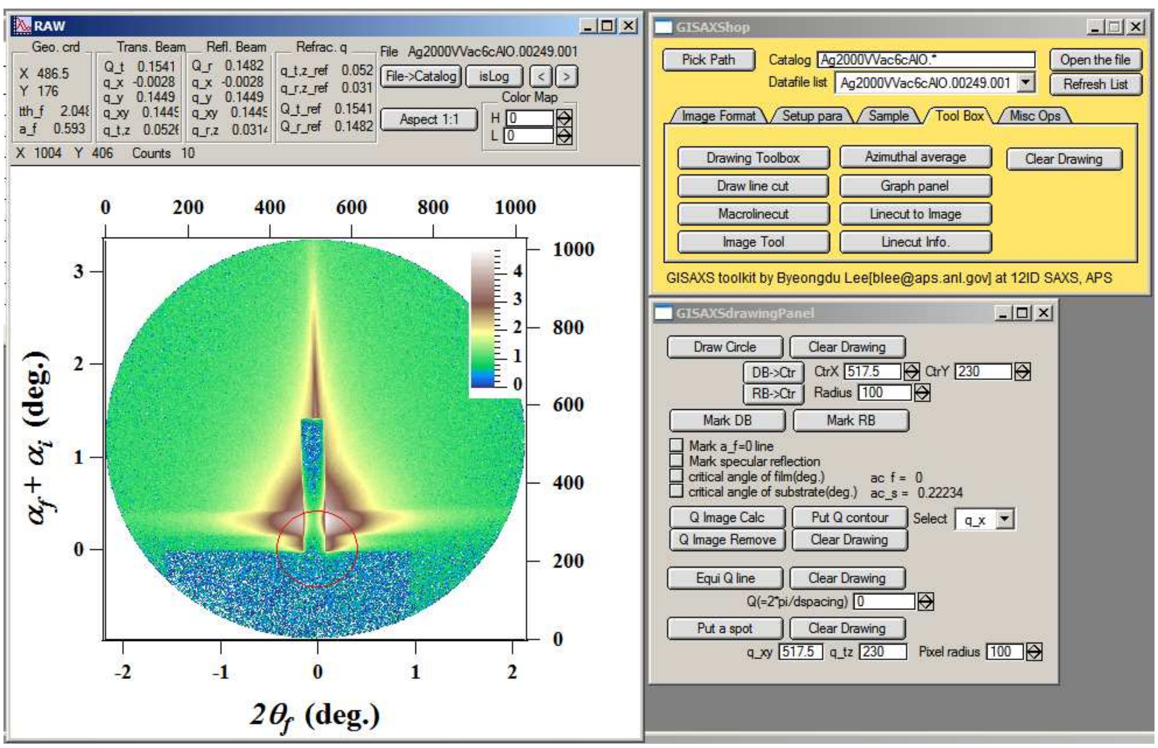

Figure 17. Drawing toolbox

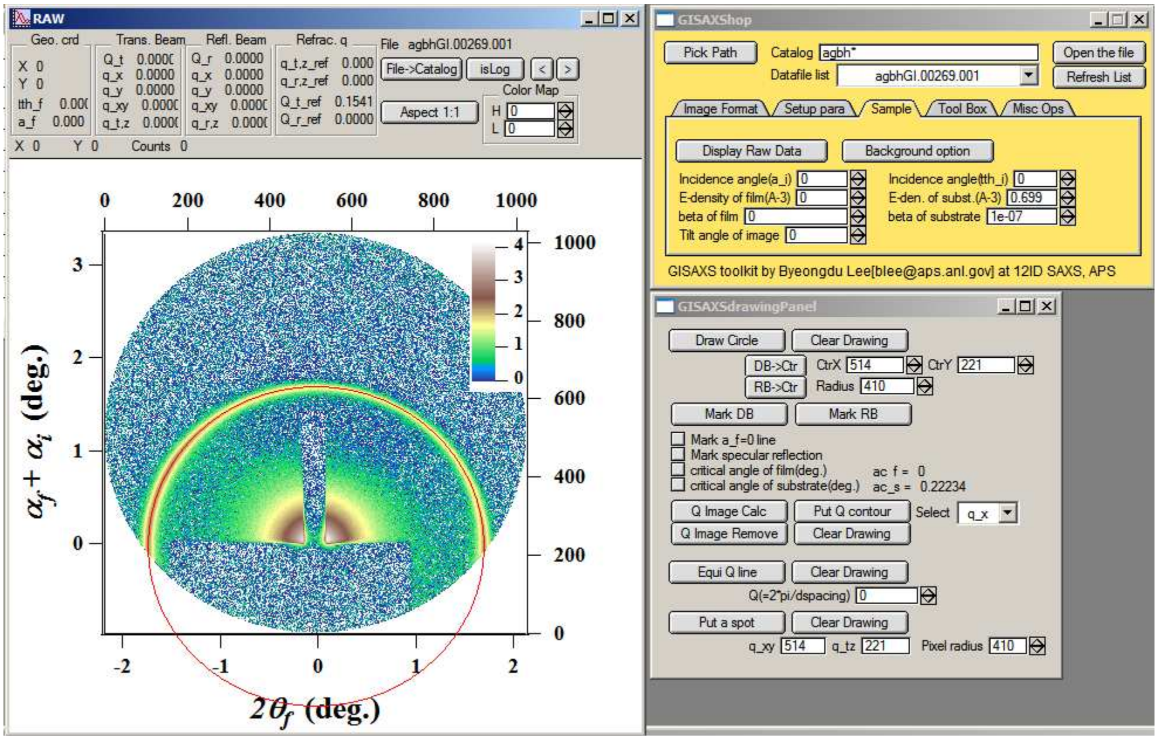

Figure 18. Drawing function with a silver behenate pattern.

2. Linecut toolbox

There are about 4 ways of making a line cut: Horizontal, Vertical, Arbitrary direction, and Circular cut are those. User can put a tilt angle (azimuth): Fig. 19 shows an example of images tilted about 0.8 degree. If you need to cut data along 45 degree, set the angle 45 and do a horizontal cut for instance.

Arbitrary factor could be applied on the x-axis of a line cut for example to swap +/- direction by multiplying -1 or for some other reasons. "*Bandwidth of linecut*" will define the number of lines averaged. A line cut is composed of 4 waves (q, r, s, and t waves for x-axis, intensity, square root of |intensity|, and information for the line cut, respectively).

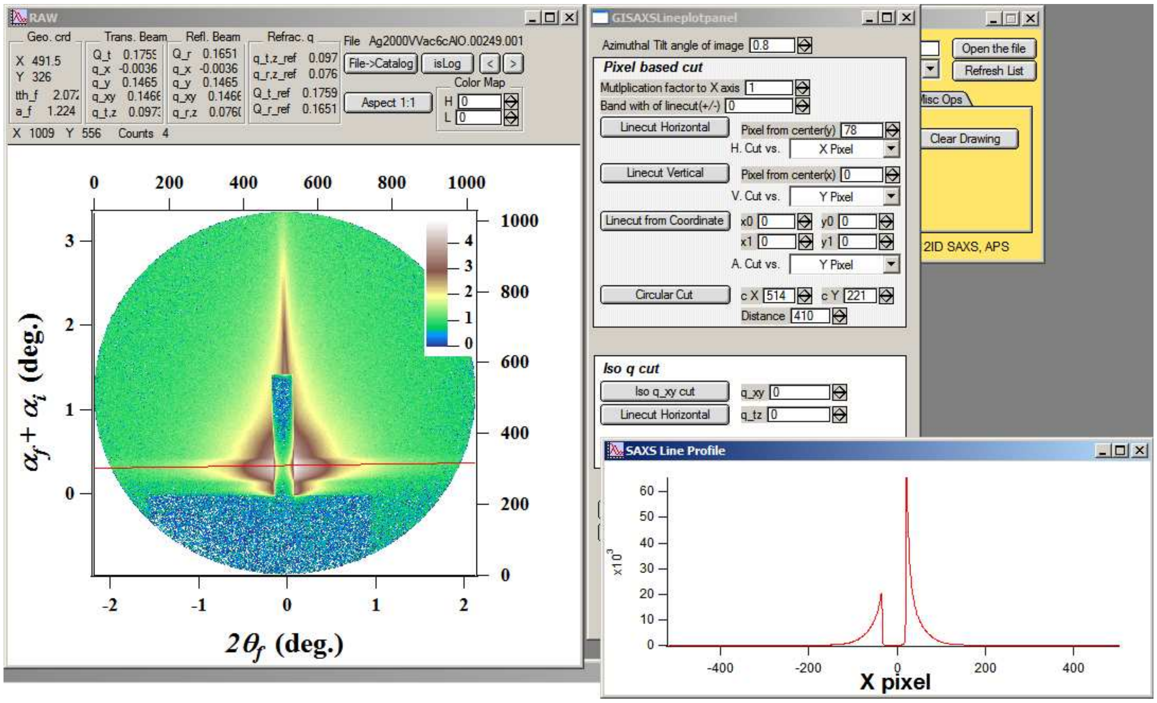

Figure 19. Line cut

As Fig. 20 shows, the number of options for the x-axis of a linecut are 17, four of them are refraction-corrected (electron density of a film should be correctly provided by user).

Once user made a linecut, it will be displayed on “*SAXS line profile*” window. But, the displayed wave will disappear if user makes another line cut.

To save or convert the linecut on the “*SAXS line profile*” window to a wave, user has to provide names for the cut. On behalf of users, GISAXShop will provide a file name as a part of the wavename. For example, user loads “test.00001.001”, makes a horizontal cut (by clicking “*Linecut Horizontal*” button), puts 1 for a cut number, and coverts the linecut to waves (by clicking “*Copy Linecut to root*:”). Then, four waves named “qH1_test.00001.001”, “rH1_test.00001.001”, “sH1_test.00001.001”, and “tHx_test.00001.001” will be generated under root: of Igor, where 1 and H stands for the cut number (user’s input) and horizontal cut (if it was vertical cut, it will be V).

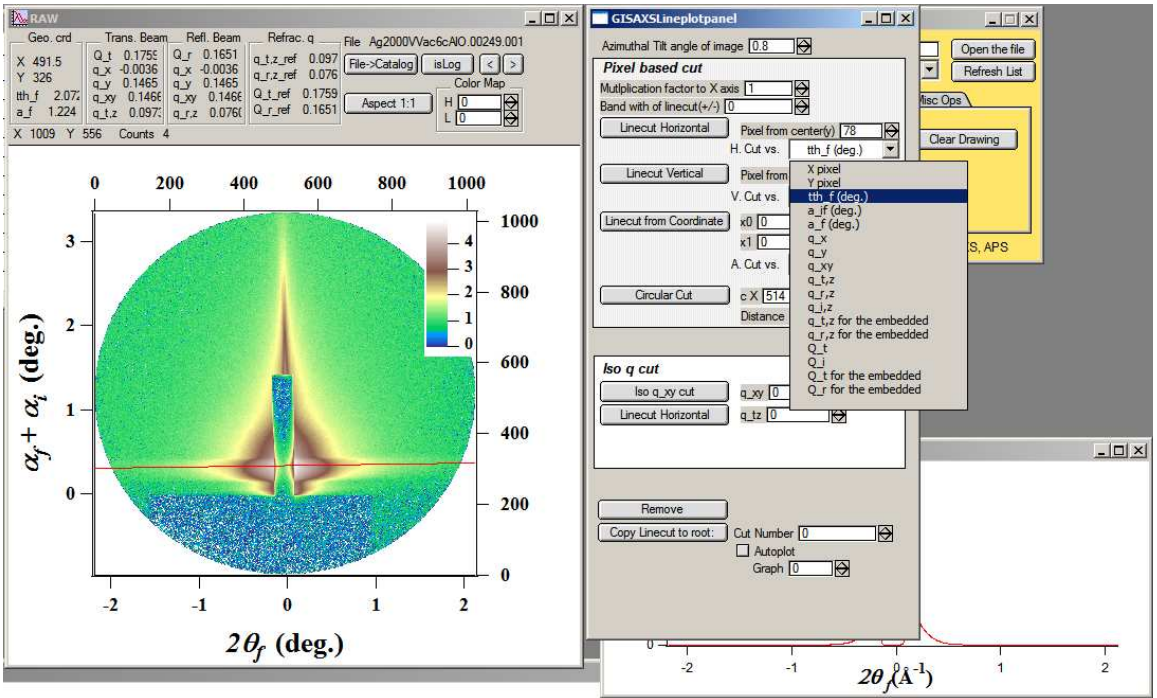

Figure 20. Line cut

If the check box “*Autoplot*” is checked prior to converting a linecut to waves, newly generated waves will be plotted on to the graph named by the number that user provide through a number box (see the number box below the “*Autoplot*” check box and next to the text “*Graph*” on the “*GISAXSlineplotpanel*” window).

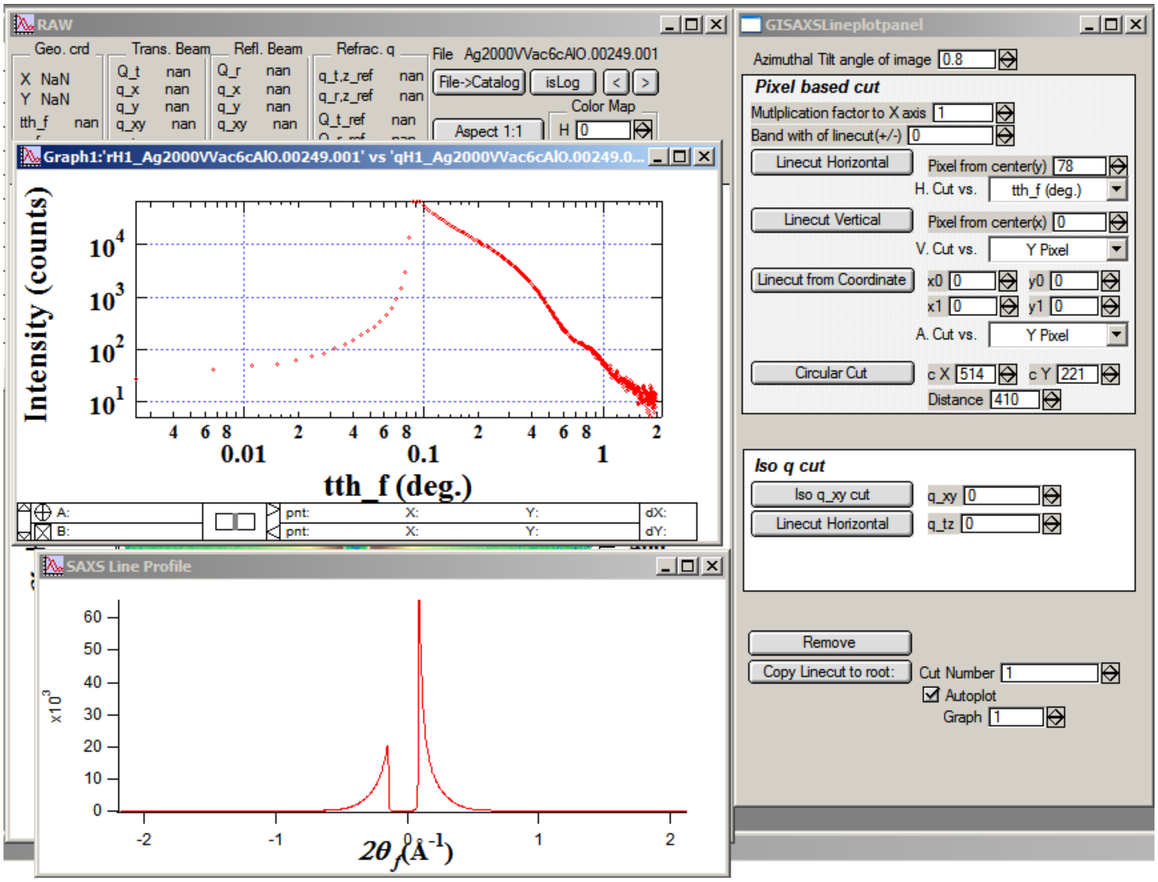

Figure 21. copy linecuts to Igor memory (or convert linecuts to Igor waves)

3. Macrolinecut

User can perform the line cut automatically for many different images using "*macrolinecut*" toolbox. Pop up "*Macrolinecut*" window. If the box "*body filename*" is empty, GISAXShop will make linecuts for all files listed in "*Datafile list*", where the linecut is the same with the latest one. User can make his/her own list of files with a combinations of "*Body filename*", "*starting number*", "*End number*", and "*Extension*".

Example :
Body filename = test.00
Starting number = 001
End number = 010
Extension =
Then, file list will be test.00001; test.00002; test.00003;…. ;test.00010;

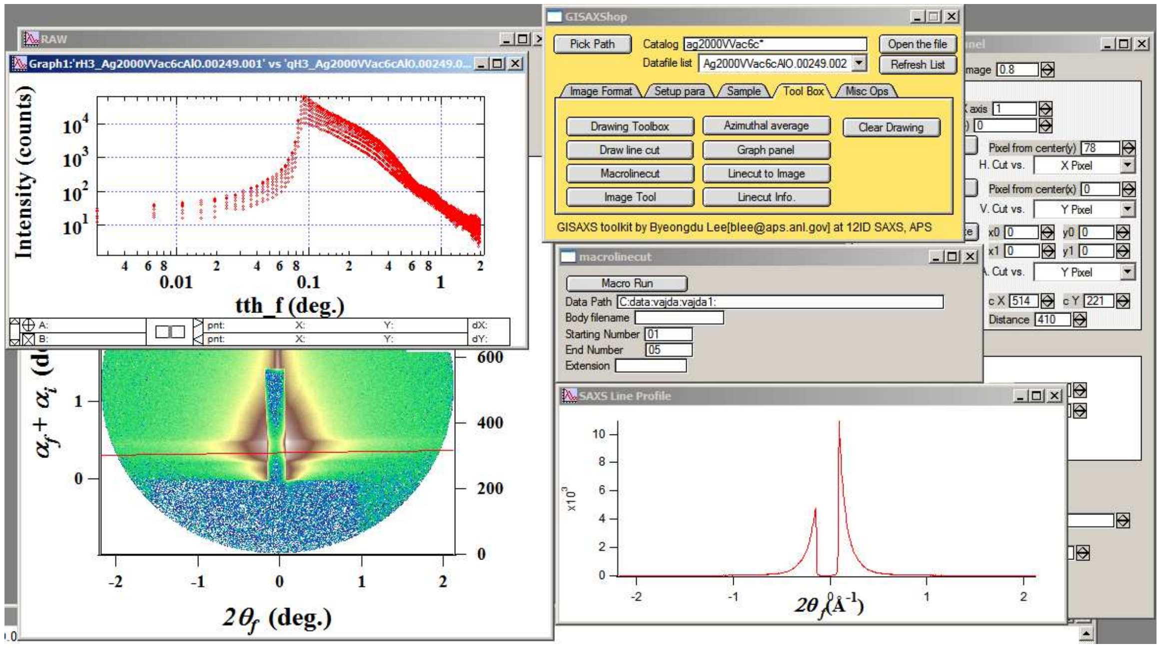

Figure 22. macrolinecut

4. Image tool

   One can change axes of a displayed image for a publication purpose. To keep the image in IGOR memory, user can provide a name for the wave storing the image (“aa” in Fig. 23) and copy it using “*copy Image*” button.

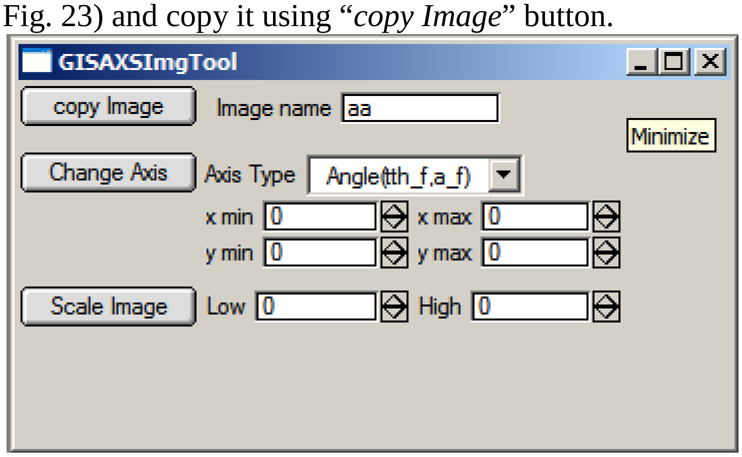

Figure 23. Change axes of images

To save the displayed image data as a image, use default IGOR functions: File>Save Graphics…

5. Lines to image tool

   There is a tool to make an image from linecuts. Pop up “*Line2Img_panel*”. Usage of this tool is similar with that of “*macrolinecut*”. It requires two more user inputs to produce the image by stacking linecuts: “*prefix to BF*” and “*Imgname*” are those. User can provide x-axis of the image. The default is “0;0.01;” which means a start from 0 with

0.01 step. If no idea what this means, put 1;1; in the box and click "*show~~*" button, and put 0;0.01; and do it again.

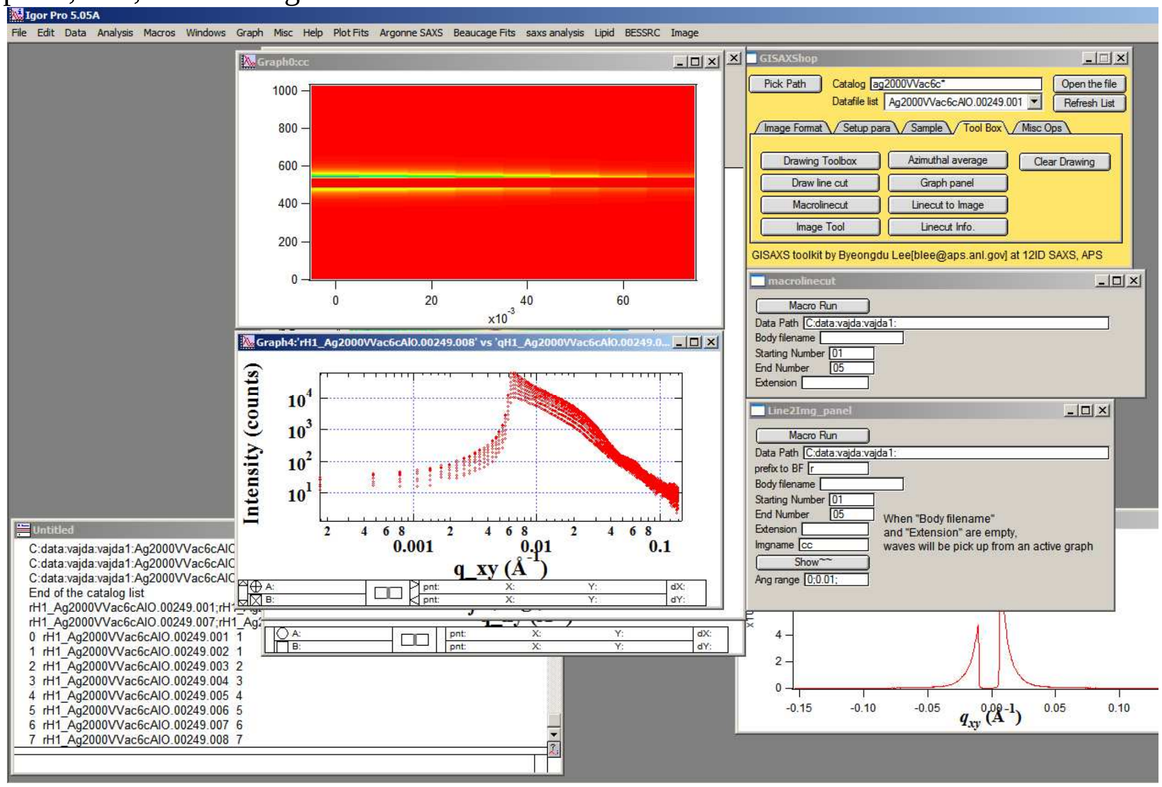

Figure 24. Make an image from linecuts.

6. Linecut info

To see how the linecut was made for a certain wave, use "*Linecut Info*" tool in the main panel. Put cursor A to a wave of interest and click "*Show Linecut Info.*".

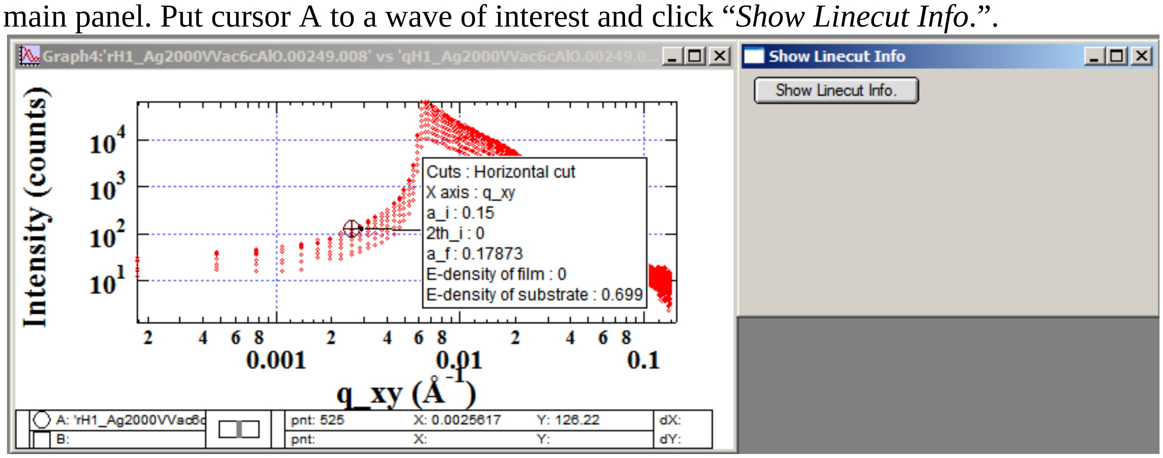

Figure 25. Display information of a linecut.

7. Azimuthal averaging

Click the button "*azimuthal average*" on the main yellow panel to show "*saxstoolkit*" below.

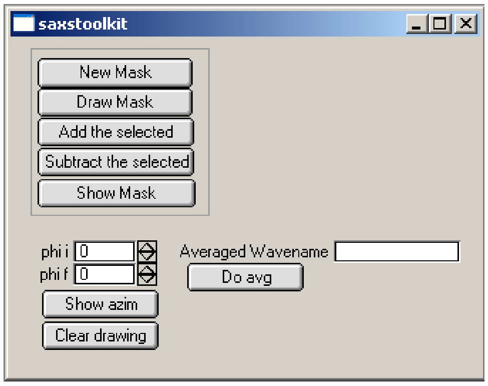

Figure 26. SAXStoolkit panel

You have to make a mask prior to averaging data. To produce a new mask, click "*New Mask*" button. By doing so, you may lose previous mask you might generage. Then, click "*Draw mask*", which will pop up editing tools on the data window "*RAW*". You may select whatever shapes of closed loop as shown in the next figure.

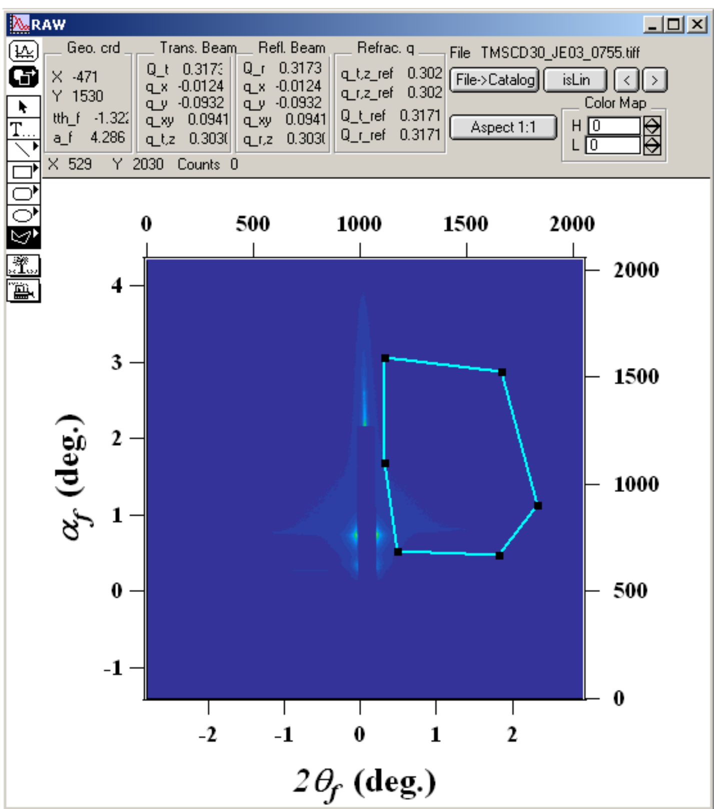

Figure 27. Mask editing.

If you like the selected mask, click either "*Add the selected*" or "*subtract the selected*". As you may guess, it will add or remove the selected area to or from the existing mask, respectively. The next button "*Show mask*" will display overall mask you generated. If you are happy with the mask, please proceed azimuthal averaging by clicking "*do avg*" button.

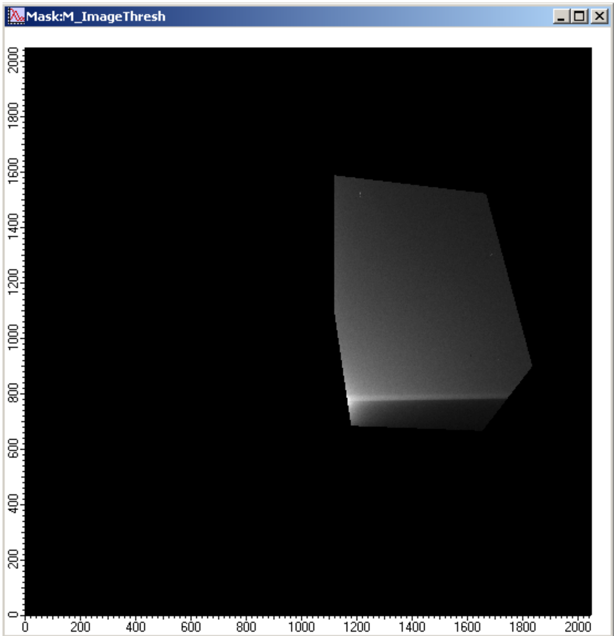

Figure 28. generated mask.

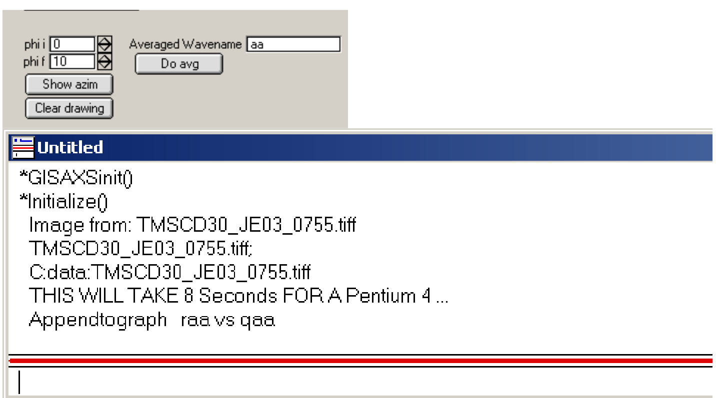

Figure 29. azimuthal averaging done.

When you put "aa" as a wavename for the averaged curve, we will see the result shown in Figrue 29, where the data has been averaged from azimuthal angle 0 to 10 degree. Resulting waves are then "qaa" for x-axis and "raa" for y-axis.