Using the R-Axis Spider to run thin films

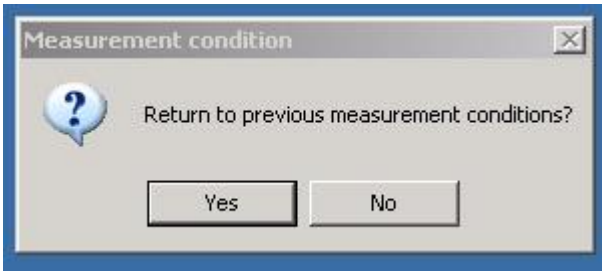
In addition to loose powder samples, it is possible to run thin film samples on the R-Axis Spider. Use the zero background plate holder for your thin film sample. A small amount a double sided tape will keep the thin film mount in place. The thin film mount needs to be kept smaller than xxx mm to keep from bumping into the collimator. Substrates composed of glass slides and Si wafers are easily accommodated. Thicker FTO plates may require some adjustments.

Instead of using Crystal_Clear, use RINT_XRD XG for powder samples. Before starting the program, it is useful to create the data directory first. You will be asked for a data at the start.

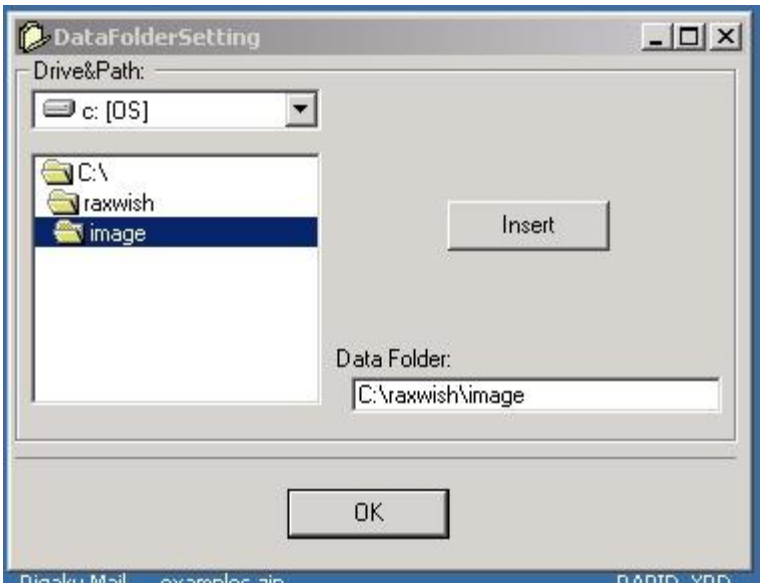
Double-click 

to start.

You will be asked if you wish to return to the previous data collection parameters.

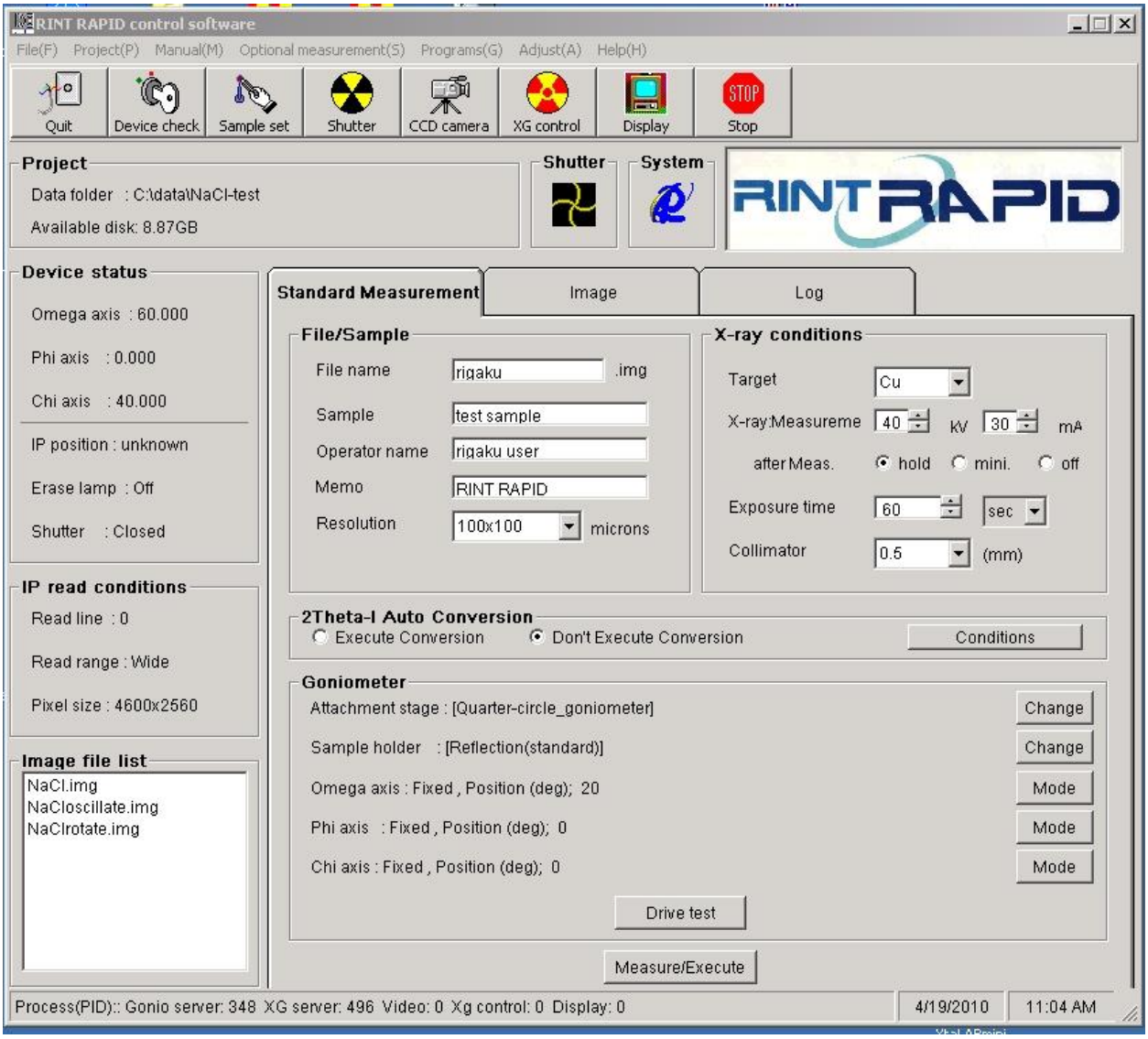


If you answer yes, you experimental setup will be the same as the one run just before you. If you answer no, the default experimental conditions will be set. The default setup uses an incorrect setting for KV and mA, which should be set to 40/40.



After selecting the proper directory, you must hit the Insert button. The selected directory will be displayed in the Data Folder entry. If you forget to hit Insert, you will go to the default directory, C:\raxwish\image.

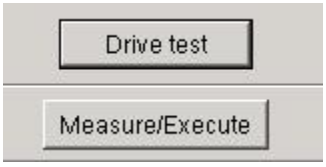
The program control panel will appear as below.



You should change the File name to something that makes sense to you. Under Sample, you can enter some additional useful information about the sample.

On the Spider, the target radiation is Cu. Change the X-ray measurement settings to 40 KV and 40 mA. A sample exposure time of 10 minutes is usually sufficient.

As with a single crystal, you have to align the sample. Set omega axis, fixed position to 70°, set chi axis, fixed position to 0° and the phi axis, fixed position to 0°. Hit the drive_test button



on the lower middle portion of the control stream. The angles should

drive to the assigned location. Hit the CCD-camera icon, 

to view the sample using the RAXVideo routine as in Crystal_Clear. Alignment is achieved by setting the height of the film along the horizontal axis in the microscope cross-hairs. Unfortunately, the focal point of the alignment microscope is set for single crystals and loose powders. With much larger thin film, the view will be blurred but a reasonably accurate height adjustment can be achieved. No additional alignment is necessary.

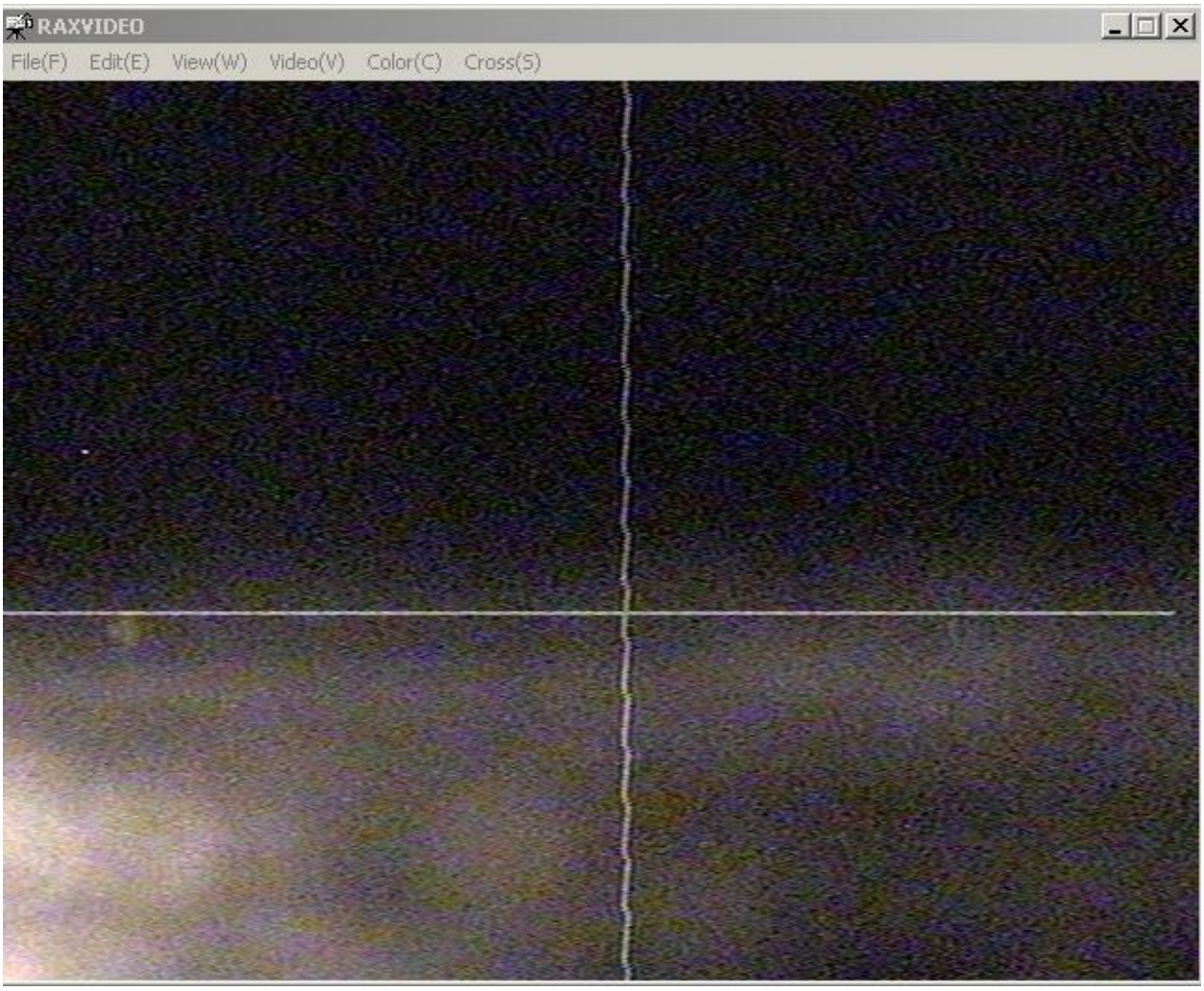
After each drive instruction is issued, you must hit the stop button 

before you can enter another drive instruction. Note the top portion of the control menu.

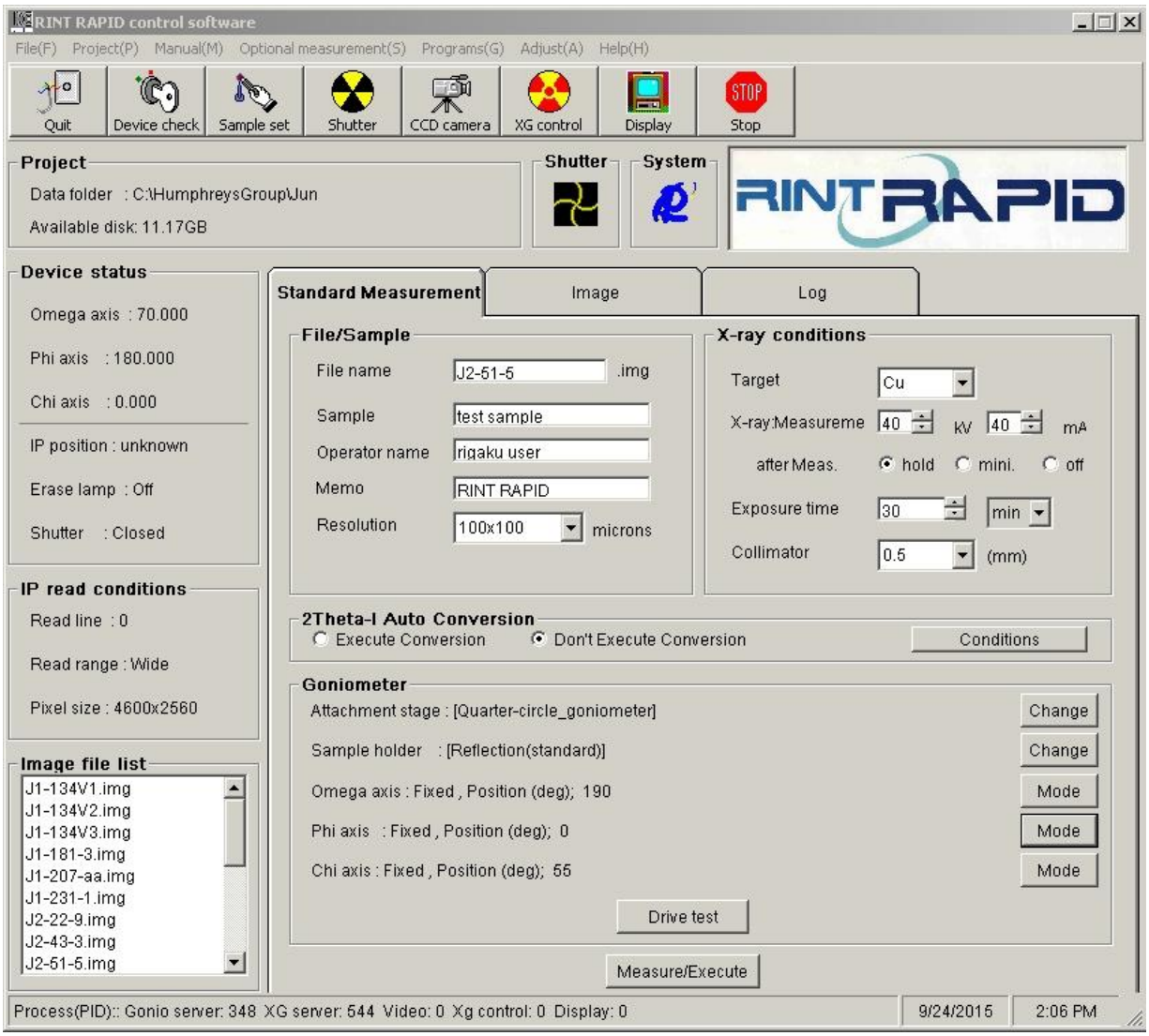
means that the program is ready to accept instructions.

means the program is busy. The program can be slow to respond depending on what it is doing so be patient.

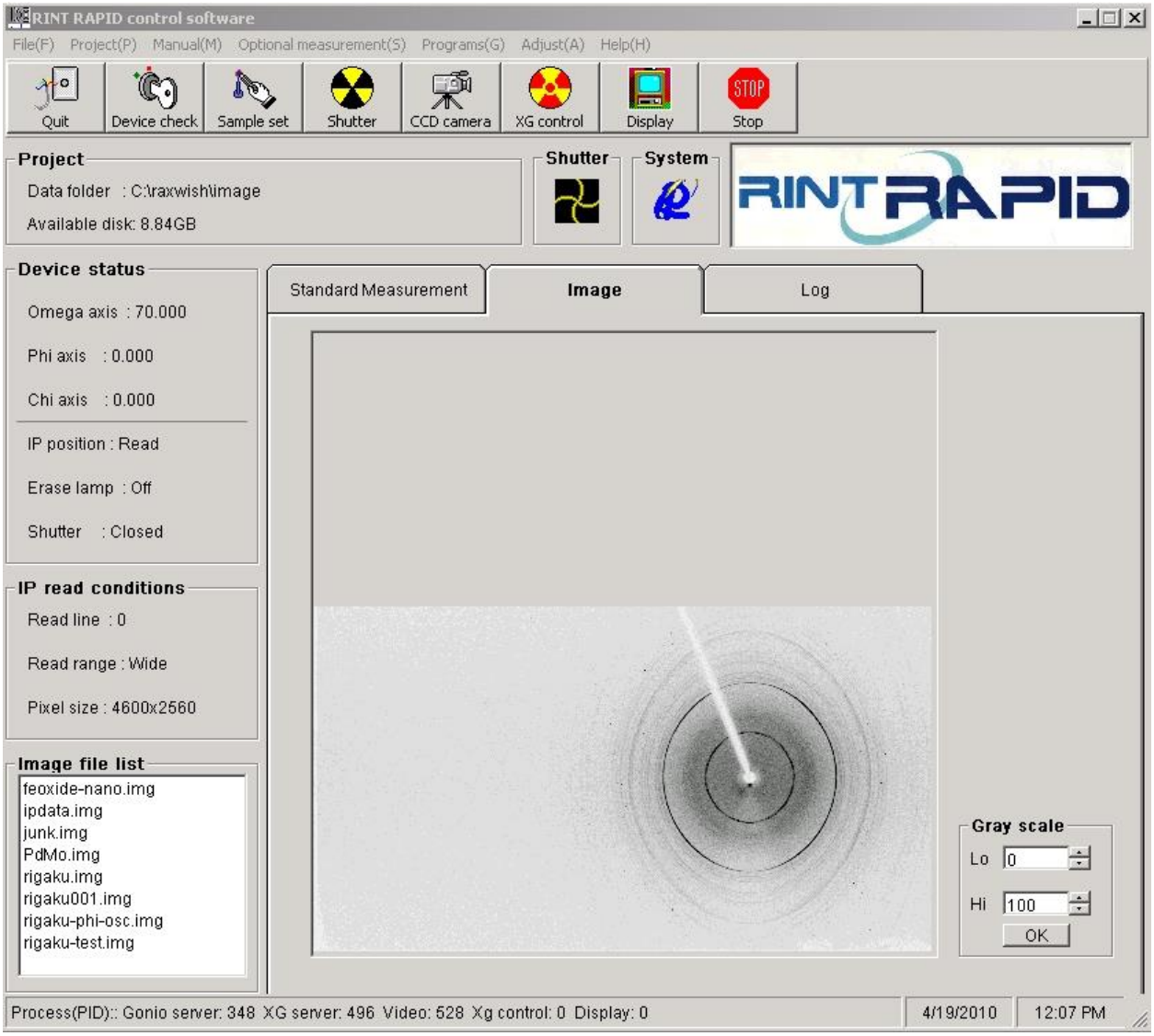
Below is a view of a film in the microscope. The actual view is a bit clearer but the idea is to set the surface of the film along the horizontal line in the viewer. Once that is set, you are ready to set up for data collection.



When you have aligned the sample, you can setup the data collection parameters. For thin films, set omega to 190° and chi to 55°. Compared to a conventional powder instrument where the set up for thin films has the X-ray beam is nearly parallel to the sample surface, the corresponding angles on the Spider would have an omega setting of 180° and a chi setting of 90°. The maximum chi setting for the Spider is only 55°. The omega setting of 190° is analogous to a grazing incidence angle of 10°. The omega angle setting can be experimented with but experience has shown that there is not much improvement using lower angles. A reasonable data collection time of 10 minutes with the generator set to 40 KV and 40 mA is usually sufficient. For very thin films or films without high atomic number elements, a longer exposure time may be necessary.

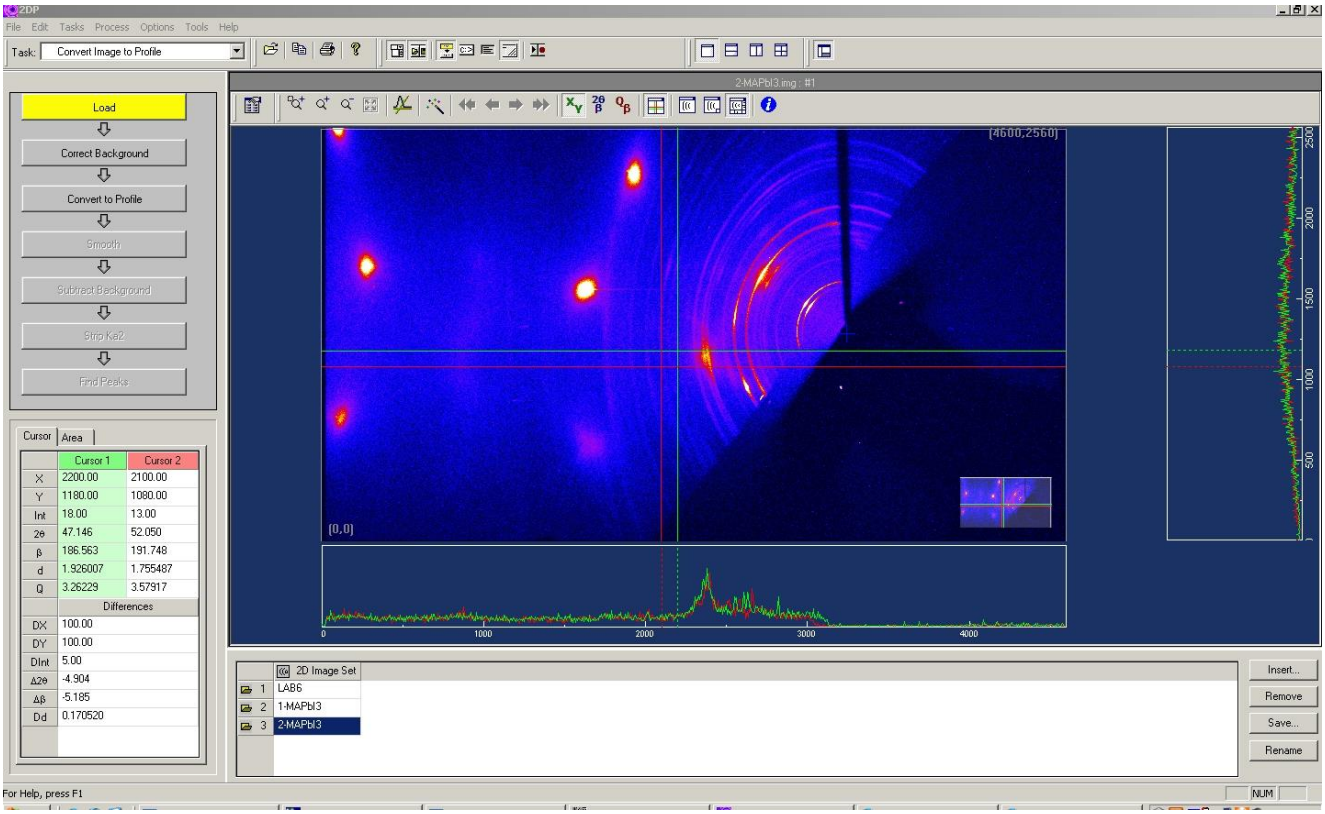


Hit Measure/Execute to start the data collection process. Be patient, the goniometer is slower than the others. In addition, the image plate detector may have to be erased before beginning. This process takes about 2 minutes as the detector is driven down to the read/erase heads in the bowels of the instrument. The yellow shutter open light will come on during data collection. When data is finished collecting, the image plate is driven to the read/erase heads and an image is created.

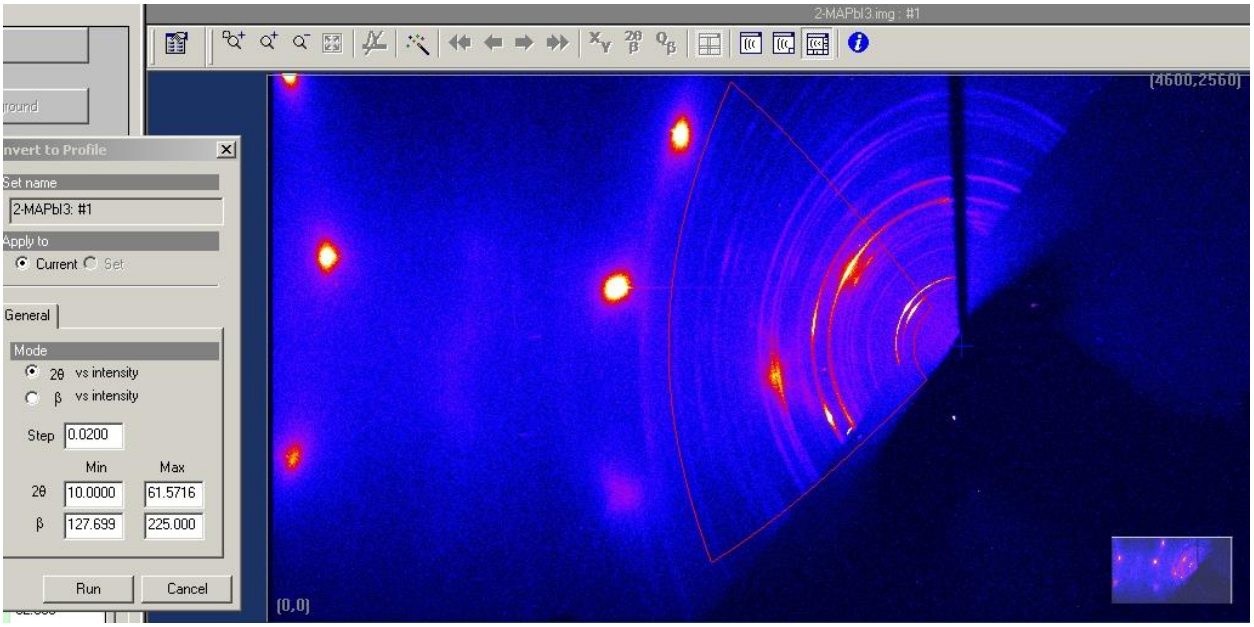


The 2D image can be converted into a 1D image suitable for data analysis in Jade or Bruker-AXS Eval using 2DP 

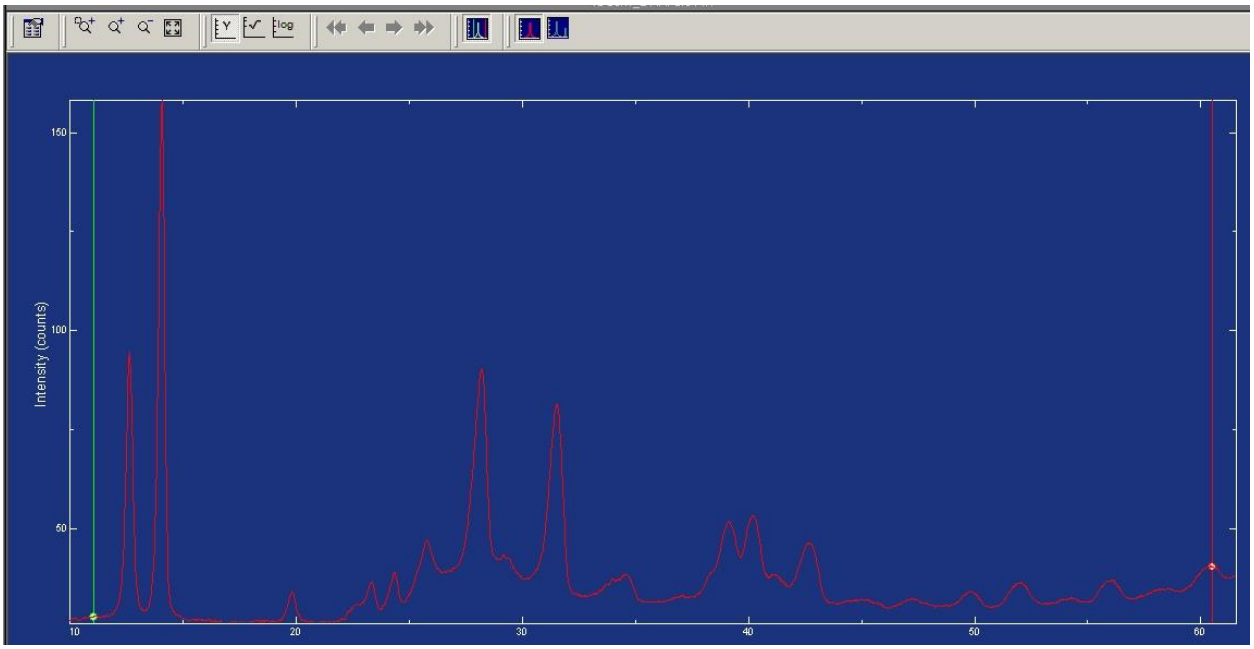
Load 2DP and select your image file for analysis. A 'feature' of 2DP is that if a binary file other than .img or .osc files are present in the working directory, 2DP will hang. There is no message output to explain why this is happening. The solution is to move the offending files to a different directory. So far, files of the type .raw (Bruker powder data files) and .jpg will cause 2DP to hang.



The slope image is due to the chi angle setting. The large reflections are single crystal reflections from the substrate. Select the Convert to Profile menu item to generate a 1-dimensional pattern from the 2-dimensional image collected.



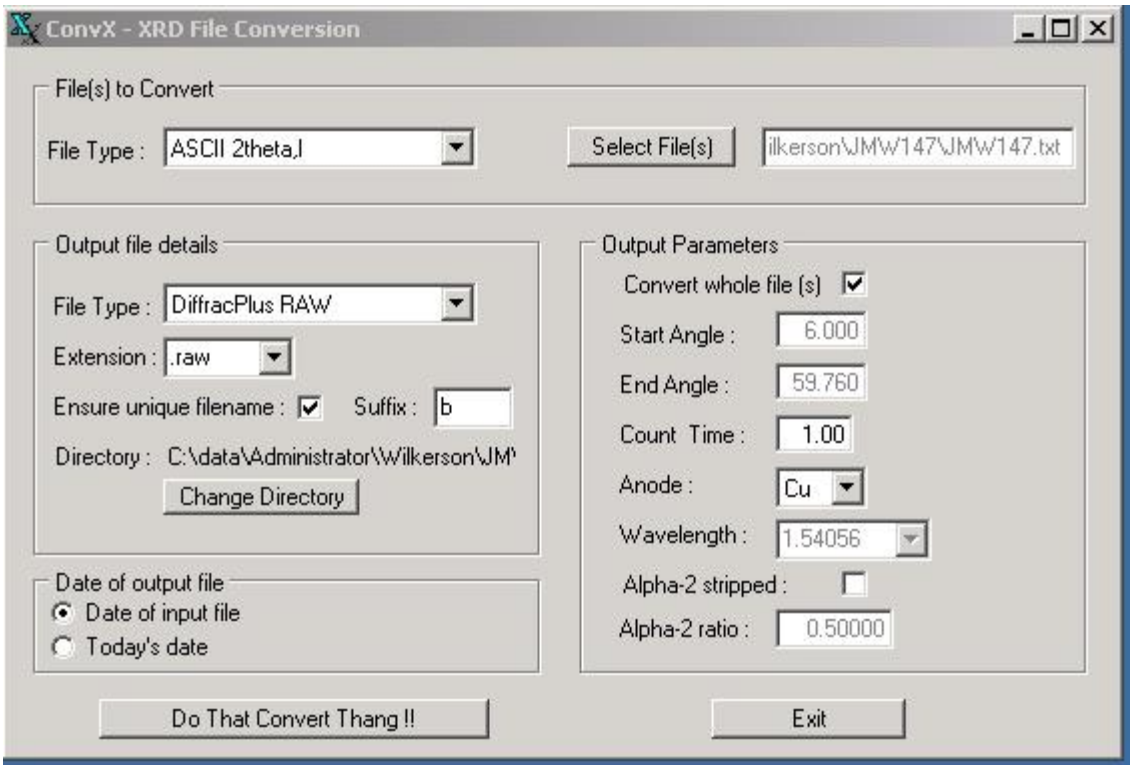
Use the mouse to control the size of the area to be converted. You will have to experiment to see what size area gives the best results. If you exceed the acceptable limits, the Run button will be grayed out. When you are satisfied with the area, hit run. You will see something like this:



You can save this file for analysis in either Jade or Eval. If you wish to use Jade, save the file as type .asc. If you plan on using Eval, save as type .txt. You will have to use the free-ware program ConvX.exe, 

to convert from the ascii format .txt to the Bruker Raw format.

The program menu for ConvX will appear as below.



The default file type for ASCII 2theta, I is .dat. You will have to select the appropriate .txt file. The output file type is DiffracPlus Raw. This is the Bruker format needed for Eval. Make sure the Anode type is set to Cu. The wavelength is set automatically. You can convert one or several files at once. The newly created .raw files will be stored in the same directory as the .txt files unless you have created a separate directory, which is recommended.

Note that there is an issue with 2DP when the directory containing the .img files also contains the Bruker .raw files that causes 2DP to hang. It is important to move the Bruker .raw files to a separate directory before using 2DP again.