Simon Fraser University 8888 University Drive, Burnaby, B.C. V5A 1S6
E info@4dlabs.ca T 778.782.8158 F 778.782.3765 www.4dlabs.ca

Rigaku Rapid Axis XRD

Operation Procedure

Standard Operating Procedure

4D Labs Confidential

Revision: 1.2 — Last Updated: July 15, 2016

Revision History

#	Revised by:	Date	Modification
1.0	Nathanael Sieb	2009/10/26	Initial Release
1.1	Nathanael Sieb	2010/12/17	
1.2	Philip Kubik	2016/07/15	

Table of Contents

1 Purpose

This document describes how to measure x-ray diffraction using 4D Labs' Rigaku Rapid Axis X-Ray Diffractometer (XRD1). It is intended to be a reference for trained users of the system and is not a substitute for training by Nanofabrication Facility staff. In addition, to training in the operation of XRD1, users of XRD1 are required to have recent x-ray safety training. Readers of this document are expected to have a basic familiarity with x-ray diffraction.

2 Definitions

XRD: X-Ray Diffraction

3 Conventions

In this document, the following items are italicized.

- Hardware units
- Software menu items
- Software windows and panels
- Software box names

The following items are underlined.

- Hardware buttons and switches
- Software buttons
- Special keyboard keys, e.g., Enter.

Text to be typed is shown inside double quotation marks.

Hazard conventions:

- CAUTION indicates a hazard which may cause damage to equipment.
- WARNING indicates a hazard which may cause injury to personnel. It may cause damage to equipment as well.

4 References

XRD1 training video: https://www.youtube.com/watch?v=C1EY_LD-2qY

Cambridge Structural Database: http://cufts2.lib.sfu.ca/CRDB4/BVAS/resource/5639 [1]

Crystallography Open Database (COD): http://www.crystallography.net/cod/

Inorganic Crystal Structure Database (ICSD): https://www.fiz-karlsruhe.de/de/leistungen/kristallographie/icsd.html

International Centre for Diffraction Data (ICDD): http://www.icdd.com/

5 Contact

Questions or comments concerning this document or operation of the system should be directed to the current tool owner at 4D Labs Nanofabrication Facility, Simon Fraser University, Burnaby, BC, Canada. The current tool owner is listed on the web page for the tool on the 4D Labs Nanofabrication Facility web site.

[1] Available at standalone computer #2 on the third floor of SFU's Bennett library but restricted to SFU students, faculty and staff with an SFU computing ID and password.

6 Overview

The Rigaku Rapid Axis X-ray Diffractometer is capable of measuring x-ray diffraction patterns of small powder samples and inviscid liquids as well as rapid screening of crystallinity. It is equipped with a three-axis goniometer and an image plate detector.

7 Procedure

7.1 Start-up

1) Sign in to Log Book.

2) Create a ticket on the Nanofab user web site.
3) Ensure that all components of XRD1 are switched on and that the x-ray generator is on. Unless measurements are in progress, the x-ray generator should be in standby mode, i.e., 20 kV and 2 mA.

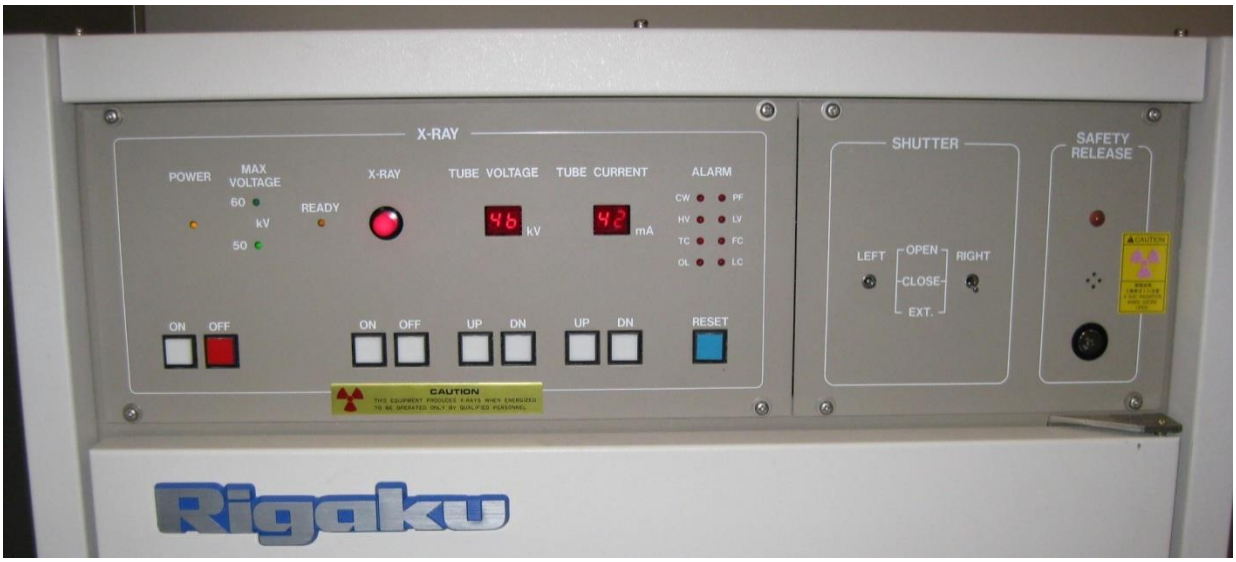


Figure 1: Top panel of the XRD1 electronics cabinet. Voltage and current are at full power (46 kV, 42 mA).

4) Open the *Rapid/XRD* software.
5) At the prompt, choose whether to continue previous measurements or not. Usually, it is preferable to select Yes. The *DataFolder* window should open.
6) In the *DataFolder* window, select the folder to which measurements will be saved.[2]
   a) Browse to your personal data folder (or a sub-folder) on D: drive.
   b) Click Insert to select the folder.
   c) Click OK.
7) Observe the goniometer positions listed under *Device Status* on the left side of the *Rapid/XRD* menu (Figure 2)
   a) If all values are realistic angles in the range -360° to 360°, proceed to the next step.

[2] The folder selection may be changed after the *Rapid/XRD* software has opened, by selecting *Project: Data Folder* from the menu.

b) If any value is 9999., on the toolbar, press Device Check to initialize the instrument.

8) Set the goniometers to convenient orientations for loading your sample as follows.
   a) On the menu bar, select *Manual: Goniometer Control*. The *Gonio control* window should open (Figure 3).
   b) Select the desired goniometer on the tab.
   c) Select Move.
   d) In the *Position* box, type or scroll to the desired angle, in accordance with the allowed range.
   e) Press OK.
   f) Repeat for any other goniometers desired.
9) On the toolbar, press CCD camera to open the *Rax Video* camera window (Figure 4).

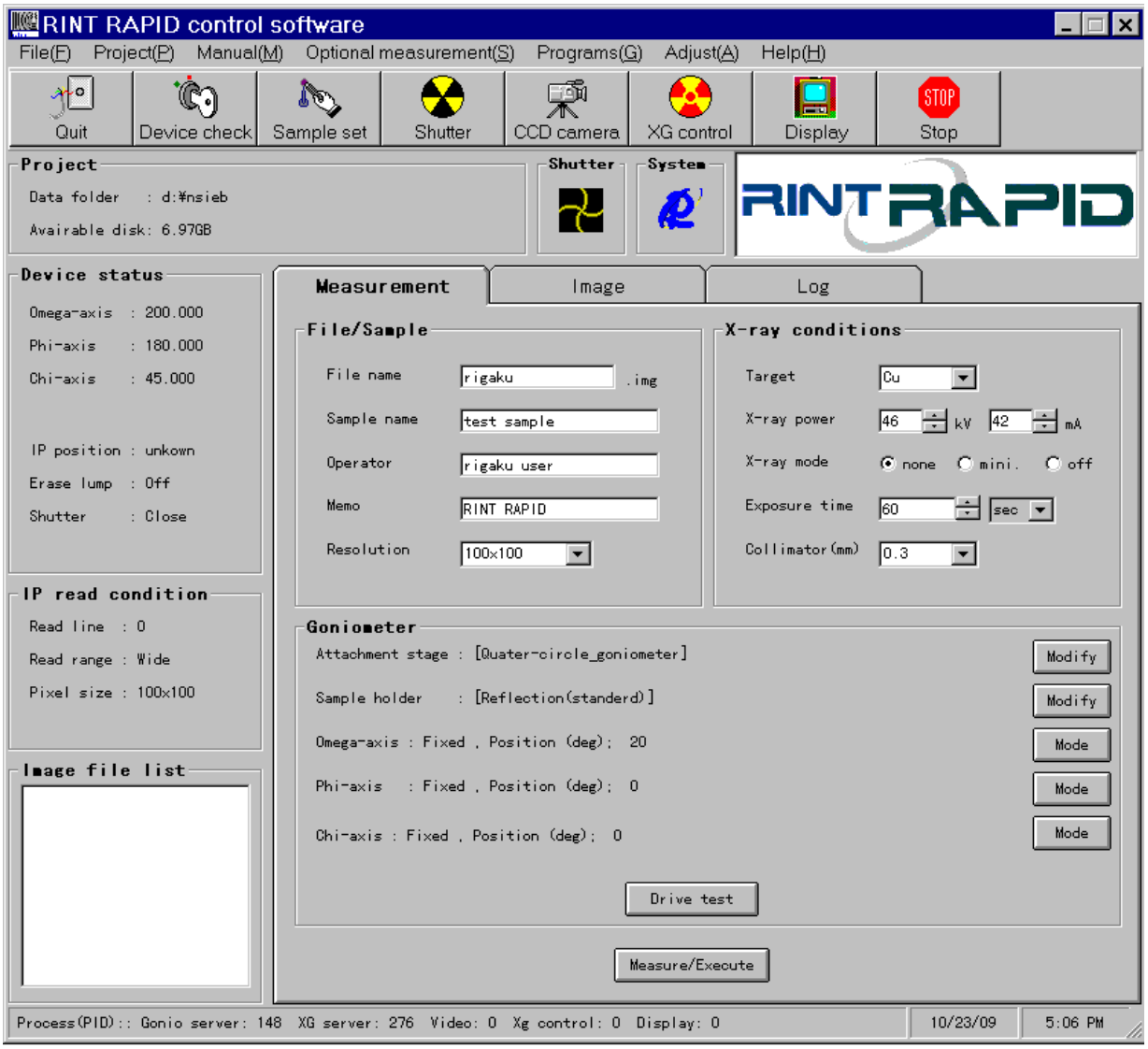


Figure 2: *Rapid/XRD* main window.

Figure 3: Goniometer control window.

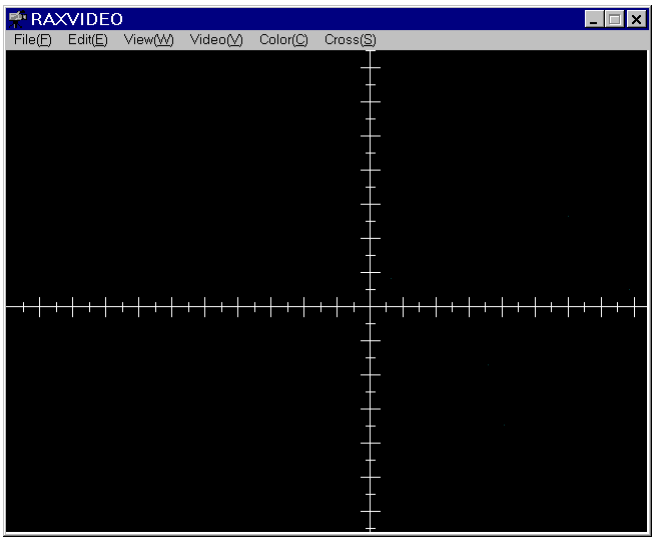


Figure 4: Camera window.

7.2 Mounting Samples on Sample Holders

Mount your sample on the appropriate sample holder on the sample preparation table.

1) Powders
   a) Place a thin layer of grease on a substrate, e.g., a piece of a microscope slide about 10 mm x 10 mm x 1mm.
   b) Press the powder sample into the grease.

c) Shake off any loose powder.

d) Bond the substrate to a flat sample holder, e.g., with double-sided masking tape (Figure 5).

e) It is most convenient, if the substrate does not extend much beyond the substrate holder.

Figure 5: Film sample on a substrate mounted on a sample holder.

2) Films or pastes

a) Thin films or thick pastes should be on a substrate, e.g., a piece of a microscope slide or silicon wafer about 10 mm x 10 mm.

b) Bond the substrate to a flat sample holder, e.g., with double-sided masking tape.

c) It is most convenient, if the substrate does not extend much beyond the substrate holder.

3) Inviscid liquids

a) Place liquid samples in a sealed capillary.

b) Place the capillary in the capillary holder and hold it in place with modelling clay. The capillary should be vertical.

4) Crystals

a) Crystals may be bonded to the end of a capillary, e.g., with modelling clay.

b) Place the capillary in the capillary holder and hold it in place with modelling clay. The capillary should be vertical.

7.3 Mounting Collimators and Sample Holders

1) Ensure that the x-ray shutter is closed by any or all of the following methods.
   a) The yellow lamp in the tower on top of the XRD cabinet must be off.
   b) The red indicator on top of the shutter motor must be off.
   c) The shutter icon in the *Rapid/XRD* software must indicate that the shutter is closed.
2) Press Door Open button  on the electronics cabinet. An audible alarm should be activated.
3) Gently open the door to the XRD cabinet. The audible alarm will continue to beep until the door is closed again.
4) Install the desired collimator (0.5 mm is typical) with the secondary beam stop attached. Note that reducing pinhole diameter by a factor of two will require a four-fold increase in measurement time to achieve the same peak intensities. For very low angle measurements, e.g., < 5°, the secondary beam stop may be removed but you must ensure that the primary beam block is correctly positioned to block the x-ray beam before ramping up the x-ray voltage and current from the standby values (20 kV, 2 mA). Failure to do so may damage the image plate.

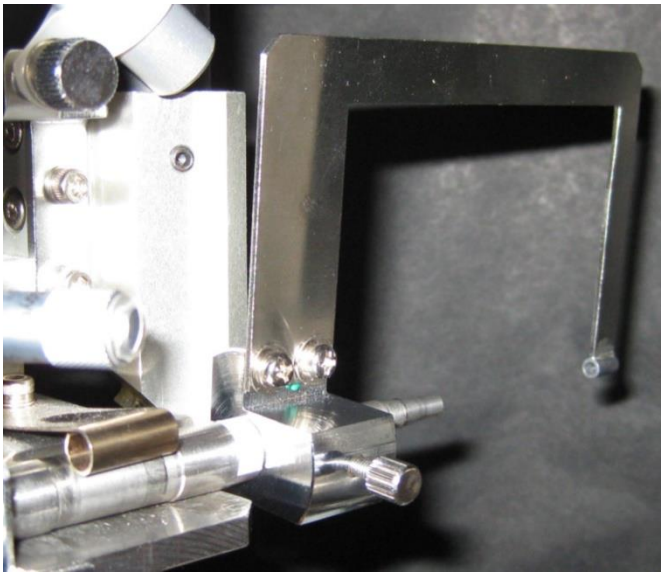
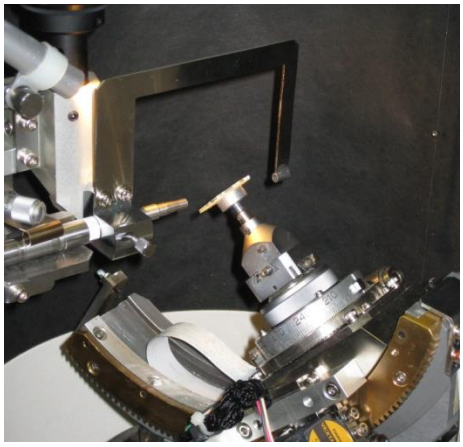


Figure 6: Collimator with secondary beam stop attached.

5) Switch the stage lamp on.
6) Align the notch on the sample holder with the knob on the stage and screw the holder onto the stage.
7) Set the goniometers to convenient angles for positioning your sample as follows.
   a) On the menu bar, select *Manual: Goniometer Control*. The *Gonio control* window should open (Figure 7).

Figure 7: Goniometer control window.

   b) For the flat sample holder, chi = 45° is usually a good choice. For capillaries, chi = 0° is usually best.
   c) Set phi = 270° for convenient access to the z-axis control knob.



1) Adjust the z-axis position knob on the sample holder line of focus in the camera window passes through the cross-hairs. [3] For samples which are relatively featureless, e.g., with smooth, defect-free surfaces, it may help to temporarily pull the stage lamp out of the lamp holder and move it by hand to get the best illumination.
2) Adjust x-axis and y-axis position knobs as desired. If you intend to rotate or oscillate your sample, ensure that the desired measurement location on your sample is at the eucentric position. This may be checked, e.g., by changing phi by 180°. For samples on capillaries, it is important that the capillary be vertical.
3) Turn the stage lamp off.
4) Close the door to the XRD cabinet gently and smoothly.
5) If the x-ray generator switches off, go to the electronics cabinet (See Figure 1).
   a) Ensure that the READY lamp is illuminated. If not, contact the tool owner.
   b) In the X-RAY section, press the ON button to switch the x-rays on. The READY lamp should switch off, the X-RAY lamp should illuminate, the x-ray voltage should slowly ramp up to 20 kV, and, finally, the x-ray current should slowly ramp up to 2 mA.
   c) Next time, close the door more gently and smoothly.
6) Set desired measurement angles. For the flat sample holder, typical values are:
   a) Omega = 200°. Omega should be >180°.
   b) Chi = 45°.
   c) Phi = 180°.

7.4 Data Acquisition

Set the measurement parameters in the *Measurement* panel of the *Rapid/XRD* window as follows.

[3] Do not adjust the position of the cross-hairs in the camera window. They have been positioned at the eucentric point of the goniometers.

File/Sample Panel

1) Enter the file name, sample name, and other relevant information.
2) *File name*: type the desired file name. Use letters, numbers, hyphens, and underscores only. Remember the OS is Window NT.
3) *Sample name:* Any characters are acceptable. This just goes into the file header.
4) *Operator:* Your name.
5) *Memo:* Any comment. This just goes into the file header.
6) *Resolution:* 100x100.

X-ray conditions Panel

1) *Target:* Cu.
2) Set *X-ray power* to 46 kV and 42 mA.
3) Set *X-ray mode* to None.
4) Set *Exposure time* to the desired value.[4]
5) *Collimator:* Select the correct collimator size, or the closest to it if the exact value is unavailable. This just goes into the file header, it does not affect the measurement.

Goniometer Panel

1) *Attachment stage:* [Quarter-circle_goniometer].
2) *Sample holder:* [Reflectometer (standard)].
3) *Omega-axis:* Fixed or oscillating. Change by pressing the Mode button.
4) *Phi-axis:* Fixed, oscillating, or rotating. Change by pressing the Mode button.
5) *Chi-axis:* Fixed.[5] Change by pressing the Mode button.

[4] Note that total measurement time will be 2 min longer because 1 min is required to erase the image plate prior to measurements and 1 min to read the image plate after measurements.

[5] The chi axis appears to have the option for oscillation but it does not work. If chi is set to oscillate, the measurement will crash.

Execute your measurement as follows.

1) If the goniometer positions in the *Goniometer* panel differ from the current positions shown in the *Device status* panel, press Drive Test to move to the initial measurement position. "Measurement in progress" should appear at the upper right and the goniometers should move.
2) When the "Measurement in progress" message disappears, press Stop.
3) Press Measure/Execute. The system will erase the image plate, expose the image plate, and then read the image plate. During the read phase, the image may be observed in the *Image* panel.

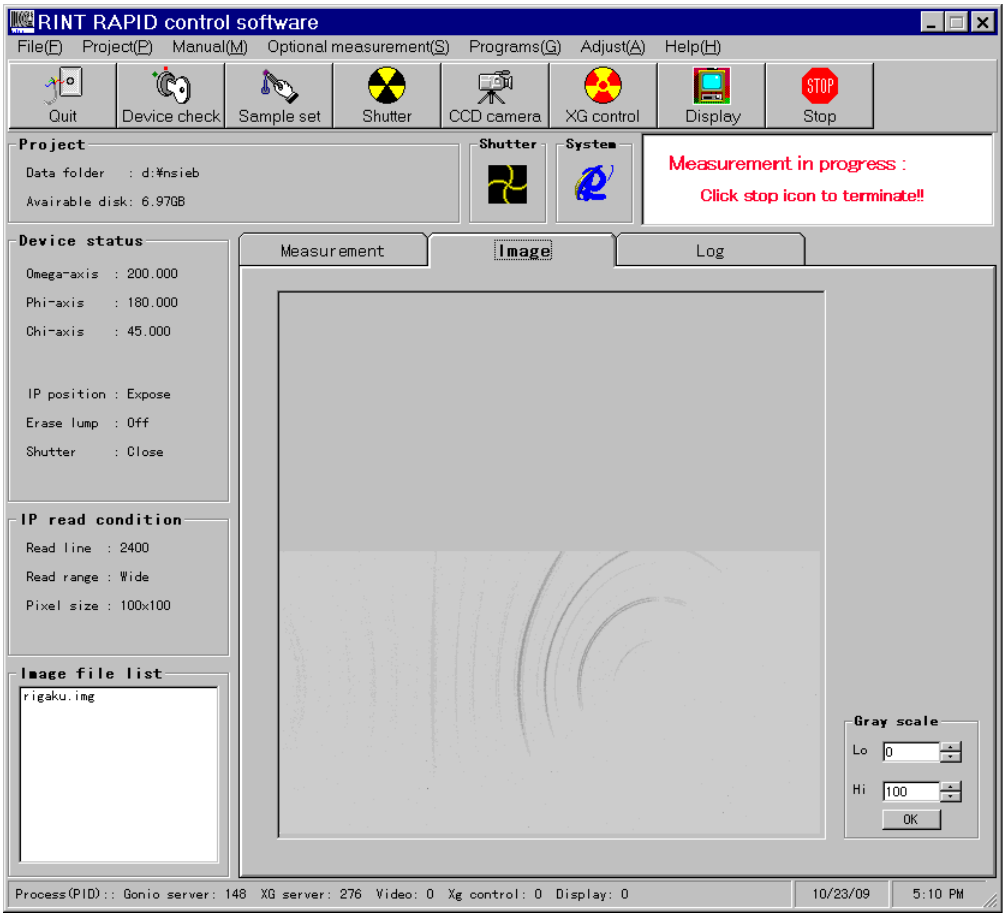
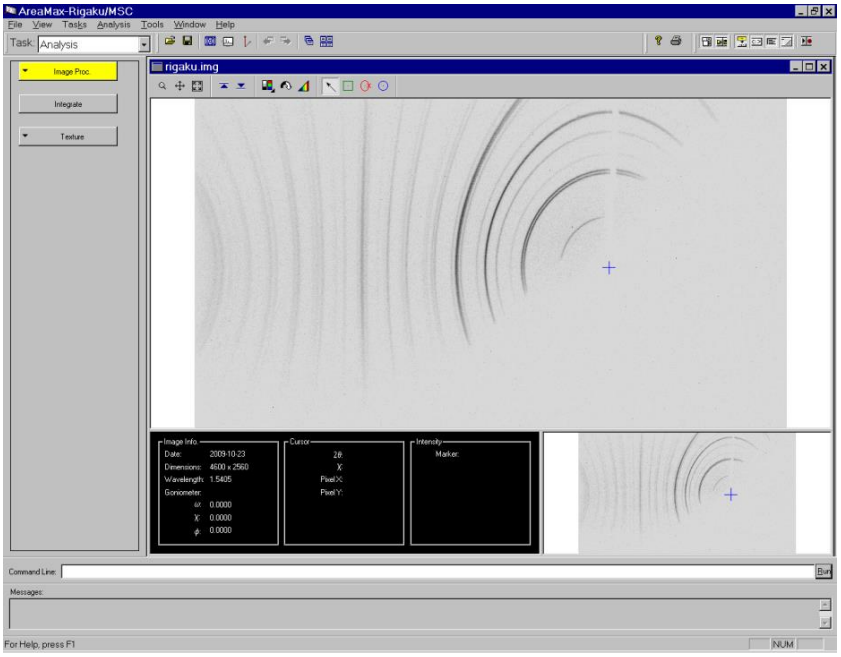


Figure 8: *Image* panel of the *Rapid/XRD* window.

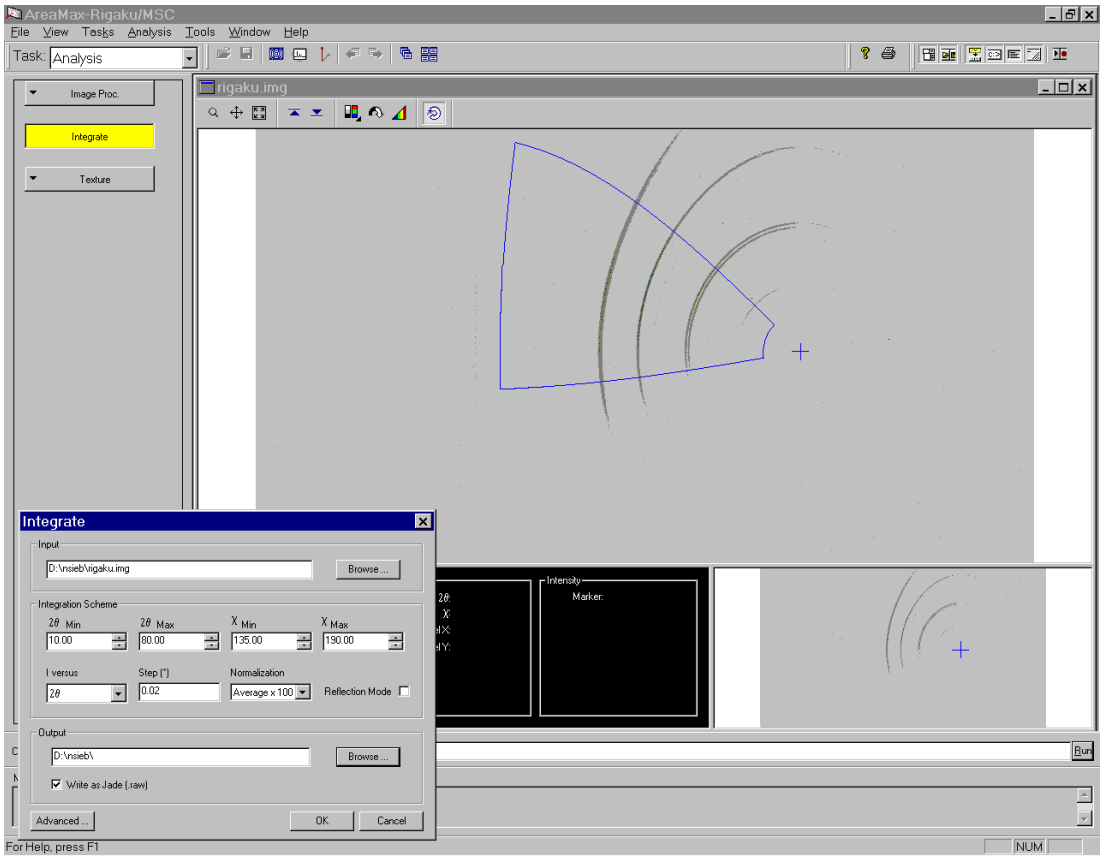
7.5 Data Reduction

Your 2D image from the image plate may be integrated over the azimuthal angle (χ) to get a 1D scan using the *Area Max* application.

1) Open the *Area Max* application.
2) At the prompt, type xray for the user name. Leave password blank.
3) Choose your data folder (or a sub-folder) as the input and output folders.
4) Open the image file created by *Rapid/XRD* by selecting *File: Open Image.*



5) Select the desired colour scheme by clicking  .
6) Open the intensity scale window by clicking  . Adjust the intensity scale until the 2D image is clear.
7) Select Integration on the left panel.



8) Select the data you wish to integrate using the radial (2θ) and azimuthal (χ) ranges.

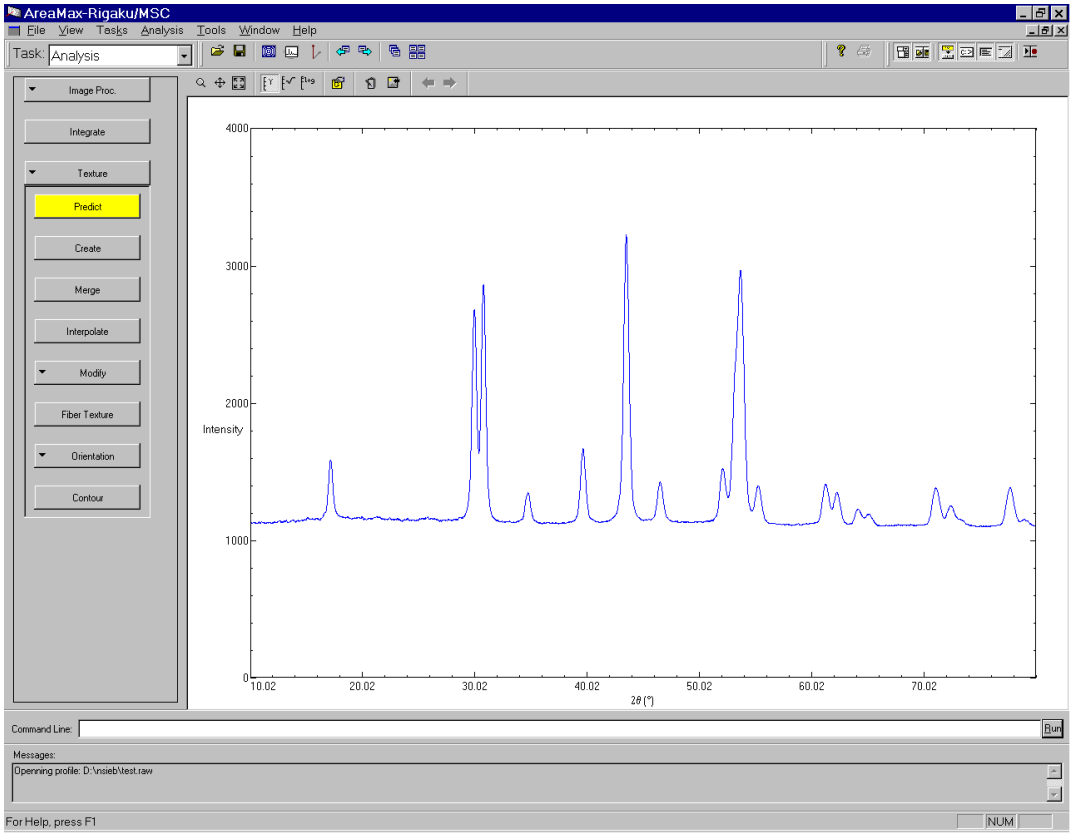
9) Select the step size, usually 0.02° or 0.01°.[6]

10) Select the file output as .raw or .rpf. *Excel* can open .rpf files. *Jade* can open .raw files.

11) Press OK.

12) A 1D scan will be displayed and the output file created.

[6] If the step size is small, e.g., 0.01°, and the 2θ range is large, e.g., 140°, *Area Max* may crash after integrating your 2D image. You should get the desired output file but you will need to re-open *Area Max* if you wish to continue working with it.



7.6 Wrapping Up

1) Close *Area Max.* **At the prompt to save the configuration, select No.**
2) Close *Rapid/XRD*.
3) Slowly turn the X-ray current down to 2 mA. Step down at approximately one step per second.
4) Slowly turn the X-ray voltage down to 20 kV. Step down at approximately one step per second.
5) Press Door Open button  on the electronics cabinet. An audible alarm should be activated.
6) Gently open the door to the XRD cabinet. The audible alarm will continue to beep until the door is closed again.
7) Remove the sample holder from the goniometer.
8) Clean up any powder which may have fallen off your substrate.
9) Close the door to the XRD cabinet gently and smoothly.

10) If the x-ray generator switches off, go to the electronics cabinet (See Figure 1).
    a) Ensure that the READY lamp is illuminated. If not, contact the tool owner.
    b) In the X-RAY section, press the ON button to switch the x-rays on. The READY lamp should switch off, the X-RAY lamp should illuminate, the x-ray voltage should slowly ramp up to 20 kV, and, finally, the x-ray current should slowly ramp up to 2 mA.
    c) Next time, close the door more gently and smoothly.
11) Remove your sample from the sample holder.
12) Return the sample holder to its container.
13) Clean up the sample preparation bench. There is a glass disposal container for any unwanted glass.
14) Complete your entry in the log book, noting any issues encountered.
15) Complete your ticket on the Nanofab user web site.