

Standard Operating Procedure: Rigaku Rotating Anode XRD



Last Modified by : Ying Qi (yqi@umich.edu)

March 27, 2014

Location :

Gerstacker Building B115

Hazards : [The following materials and equipment associated with this procedure present exposure or physical health hazards. Safety precautions are prudent and mandatory.]

This unit produces a high intensity X-ray beam. Take all precautions to avoid exposure to the x-rays

Engineering Controls :

[Prior to performing this procedure, the following safety equipment must be accessible and ready for use: (e.g. chemical fume hood, biological safety cabinet, laminar flow hood, chemical spill kits)]

None.

Protective Equipment :

[Prior to performing this procedure, the following personal protective equipment must be obtained and ready for use: (e.g. acid resistant gloves, safety eyewear, lab coat, chemical splash apron)]

You must wear **radiation dosimeters** when operating this equipment. The dosimeters will be checked every 3 months to determine whether you have received any x-ray exposure.

Waste Disposal :

[This procedure will result in the follow regulated waste which must be disposed of in compliance with environmental regulations]

None.

Accidental Spill :

[In the event that a hazardous material spills during this procedure, be prepared to execute the following emergency procedure]

Not Applicable.

Instructions:

[Please follow these detailed instructions when using this equipment]

1. **Vacuum system** : The rotating anode system is under high vacuum of 10^{-4} to 10^{-5} Pa during operation. On the front panel is a small area with 3 lights; If the first light is on, the system is in the 10^{-3} Pa range; if two lights are on, it is in the 10^{-4} Pa range, and if all 3 lights are on, the system is in the 10^{-5} Pa range. Ideally, you should only operate the machine when all three lights are lit. The system can be run in the 10^{-4} range.



2. **Water flow**: Water is continually flowing through the turbo-molecular pump, which pumps down the system. The water flow should never be turned off by a user. Water must also flow through the rotating anode portion of the system while the beam is energized. Water is vital because the electron bombardment of the Cu anode produces a great deal of heat which must be carried away by the cooling water. There is an interlock which will prevent you from turning on the beam when the water isn't flowing through the anode.
3. **Safety interlocks** : The system is designed to protect users from accidentally being exposed to the beam. The system is enclosed in tinted plastic panels. Under normal conditions, the beam will not turn on when the panels are open. In addition, several safety lights are in circuit with the x-ray beam and turn on when the beam is energized. If any of these lights burn out, the circuit will open and the beam will not turn on. When the x-ray shutter is open, a red light will turn on right next to the shutter. In addition, the shutter light on the front panel will turn on. As stated above, under normal conditions the beam will not stay energized when the panels are open. Thus, if you open a panel, the beam will turn off (or "crash"). However, users do need to go into the enclosure sometimes in order to change samples. There is a bypass mechanism that will allow this.



Turn the **FS release** key, and a beeping will start. This beeping indicates that you can now open the enclosure panels and change the sample. When you are done changing samples, close the panel(s) and the beeping will stop. The shutter will not open if any of the front or right side panels are open. This protects the user from accidentally opening the door and working inside the enclosure with the beam on. But for added safety, always make sure the shutter is closed before you open the enclosure. There is lever

attached to the shutter circuit inside the enclosure, which also must be pushed down for the shutter to open. This lever is located on the top of the metal enclosure surrounding the sample on the left side of the rotating anode. The heavy top must be placed onto the metal enclosure to push down this lever. Otherwise, as just stated, the shutter will not open.

Typical Experiment Sequence:

1. Sign in Log sheet.
2. Starting up the system and turning X-Ray on
3. Preparing your sample and putting it in
4. Running a scan
5. Shutting down system
6. Sign out Log sheet.

Procedures:

1: Starting up the system and turning X-Ray on

01. On the **VACUUM CONTROLLER** panel, check the vacuum system to make sure that all three lights are on. If only two lights are on, it mean the vacuum is not perfect but ok to use with care. If it dropped to one light please inform Lab Staff that something is wrong with the vacuum system.



a.

02. On the **XG CONTROLLER** panel All 6 red lights on the right side must be off for you to energize the beam. **If you see water dripping or squirting from the rotating anode assembly, turn the water off at once and notify a member of the MSE staff immediately.** This means that the water seal has failed and must be replaced. Prolonged running with leaking water seal may cause expensive repairing for Anode assembly.



a.

03. Make sure that all the door panels are closed.
04. Push the **T-REV** button on the XG CONTROLLER panel of the Rigaku. This will start the anode rotating at a high rate of speed.
05. Check to make sure the **READY** light is lit. If it is not, check that the panels are closed again and that the voltage and current knobs are turned all the way CCW (to zero.)
06. If the **READY** light is lit, push the ON button right below the **READY** light. This will turn the beam on. The voltage and current meters are located just to the right. You will see the voltage come up to 20 KV and the current up to 10 mA. These are the minimum possible settings on the rotating anode unit.

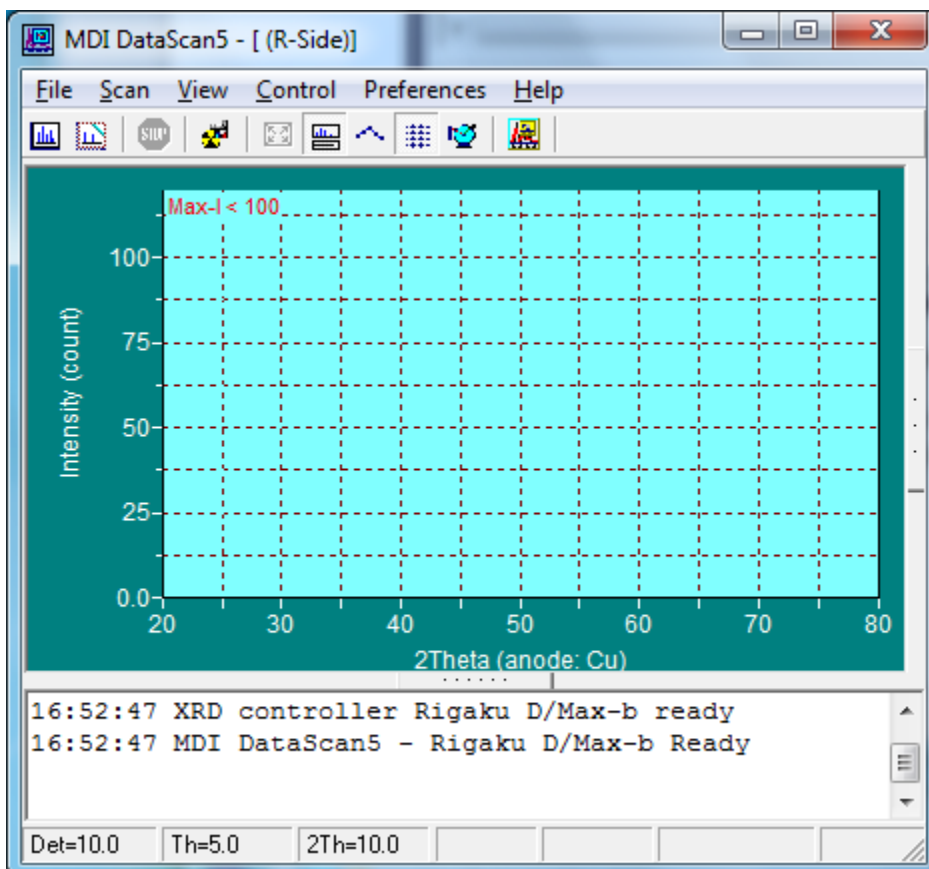
07. Turn the voltage up to 40 KV in 10 KV increments. This means you should turn the outer knob. The small, inner knob changes the KV in smaller increments and we do not use it. While you are turning up the voltage, keep an eye on the vacuum lights. Sometimes heating up the anode causes the vacuum to degrade three lights to two lights on, and if so you should wait a little while for the vacuum to recover to three lights on. Do not whip the voltage up quickly in any case -- give it a few seconds between every turn of the knob.
08. Now turn the amps up to 100 mA in 10 mA increments. Follow the same procedure (keep an eye on the vacuum lights) for the amps as for the volts.

2. Preparing your sample and putting it in

01. Most bulk samples are easy to prepare. Simply cut them to a suitable size (minimum of 0.5 cm X 0.5 cm) and thickness (max of ~ 3 mm). Samples are generally attached to an Al or glass plate, and then fit vertically into the clips.
02. Powder samples are generally packed securely into a special quartz holder (see member of MSE staff for a holder.) If the powder is likely to fall out, it should be secured with a binder or covered with tape. Make sure the binder and the tape are not crystalline to avoid getting any interfering peaks in your pattern.
03. 3. Put your sample into the clips

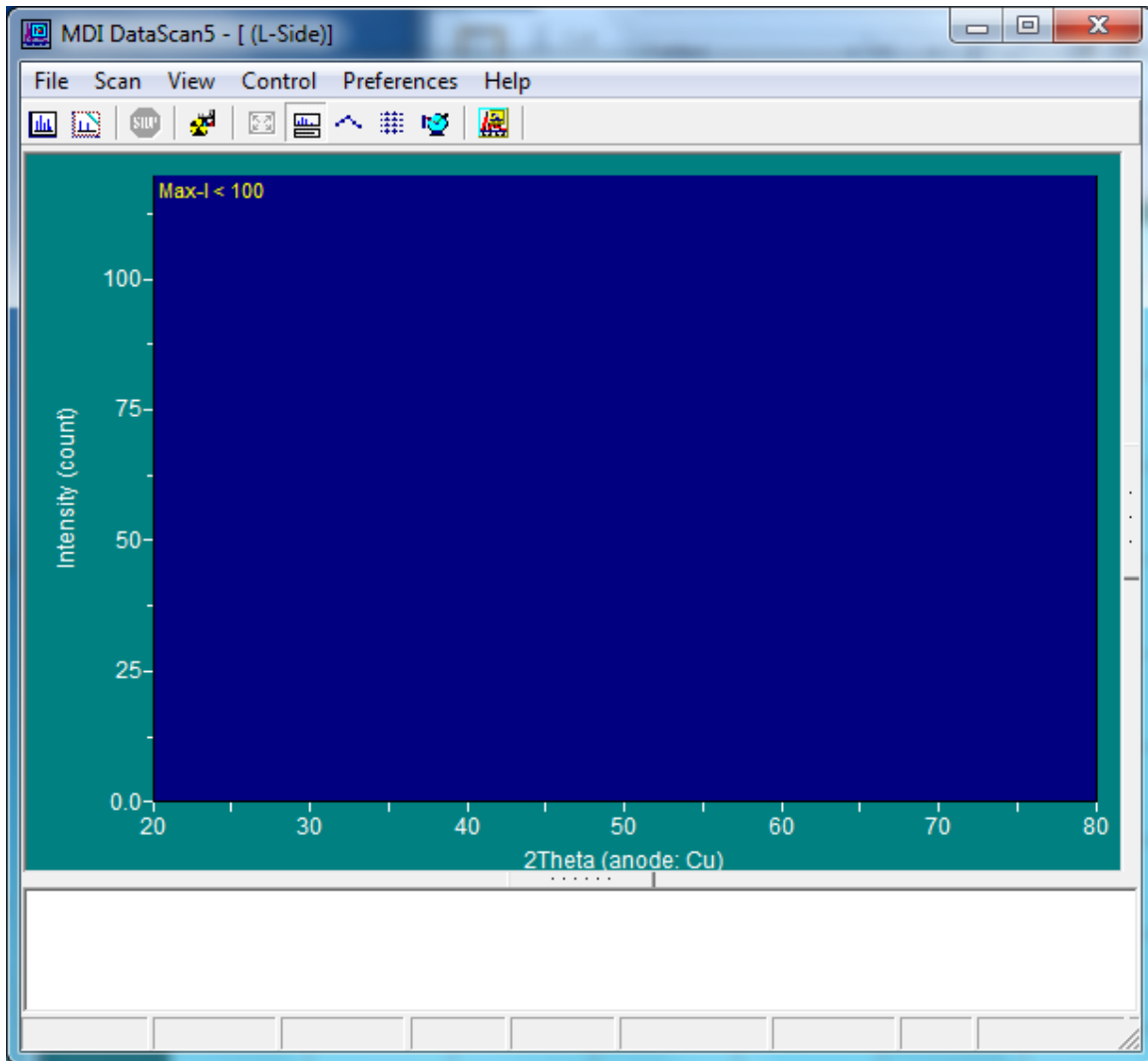
3. Running a scan

01. Launch program "MDI DataScan5" if it is not opened yet.

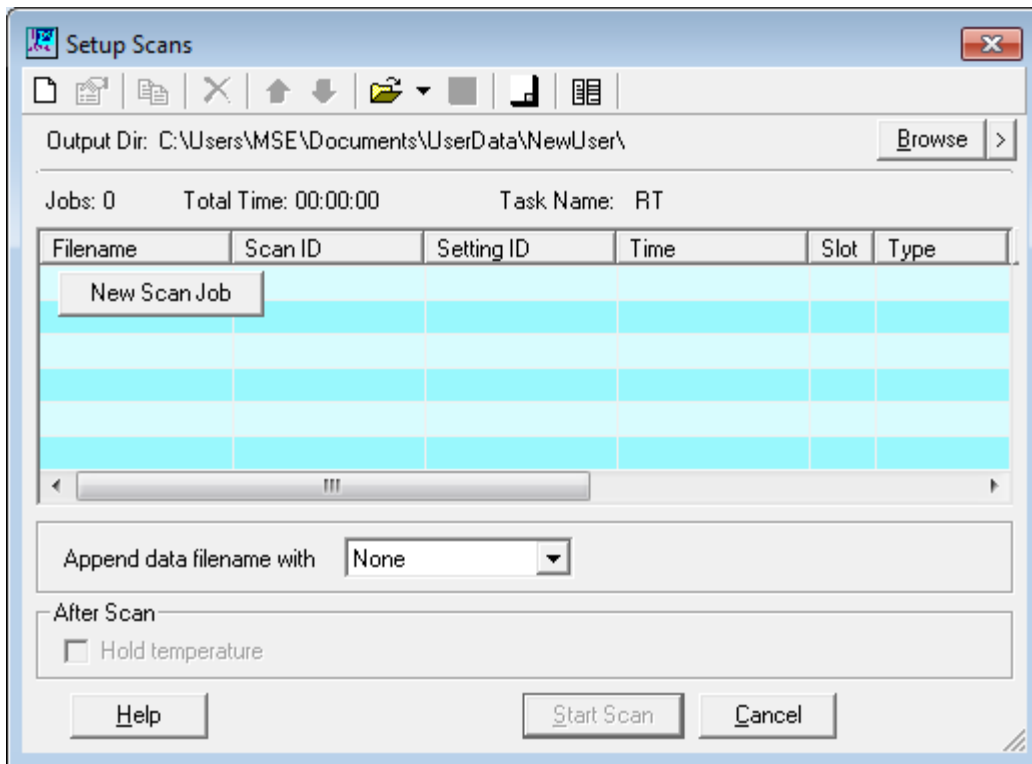


(it might start doing Datum on Right side. wait until it says "Rigaku D/Max-b ready" on the lower message window)

02. Select File\Other side, (Do not close window for R-side, it will close MDI DataScan5 and L-side will not working)



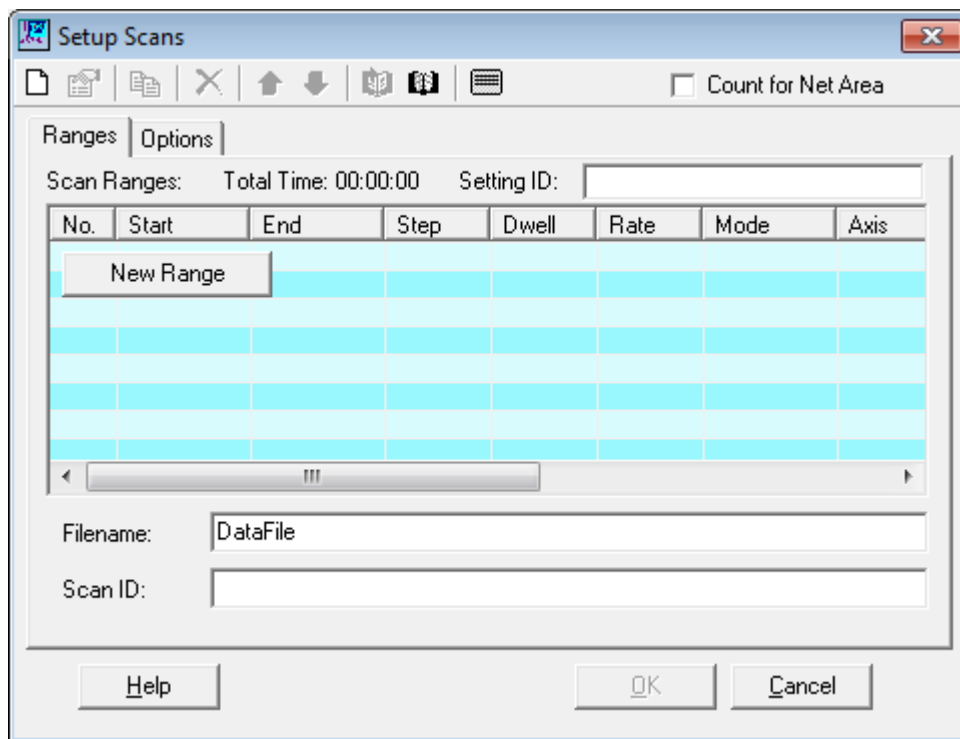
03. In the new window "MDI DataScan5-[(L-Side)]", Click on First icon on the top manu "Runtime Scan"



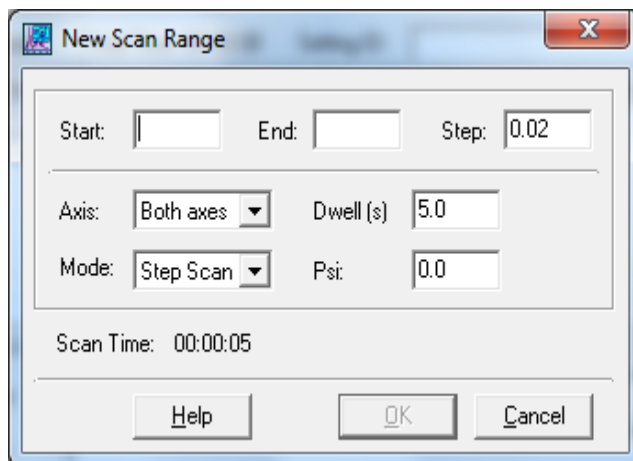
04. In "Setup Scans" Window, Click Browse to choose folder where you would like to save you data. Please save all data in C/Users/MSE/My Documents/UserData/(your unique name)/...

You can specify new scan job or open saved job.

Click New Scan Job:



Click New Range:



Input Mode(Step Scan or Continuous) first. then input desired 2theta Start,End,Step, Dwell(StepScan) or Rate d/m(Continuous). Do not change Axis (Both axes) and Psi (0.0). Then click OK.

Setup Scans

Count for Net Area ☐

Ranges Options

Scan Ranges: Total Time: 00:03:22 Setting ID:

No.	Start	End	Step	Dwell	Mode	Axis	Psi
1	26.0	27.0	0.01	2.0	Step Scan	Both axes	0.0

Filename:

Scan ID:

Help OK Cancel

Input Filename, Scan ID as wished. Then OK

Setup Scans

Output Dir: C:\Users\MSE\Documents\UserData\NewUser\ Browse >

Jobs: 1 Total Time: 00:03:22 Task Name: RT

Filename	Scan ID	Time	Type	Delay	Hold	Temp
☒ First Scan	First Sample	00:03:22	Routine ...	None	NA	NA

Append data filename with

After Scan

☐ Hold temperature

Help Start Scan Cancel

Make sure the checked mark is on the condition that you specified and uncheck the scan condition from previous users.

05. Click Start Scan, then conform the next window and click OK.

Data will be saved as "filename".MDI. You can check the pattern through File\Pattern Viewer\New window. And also you can process it with MDI Jade 2010.

Special considerations for single crystal samples

Single crystal samples, if aligned properly, will yield extremely high diffraction intensities. If these intensities are powerful enough, they can "fry" the detector, which is not set up to handle intensities much higher than 300,000 counts. If you are looking at a single crystal, please lower the voltage and current down to values of about 30 kV and 10 mA; if the intensities from your peaks turn out to be too low, you can then raise the voltage and current. But don't start at 40 kV and 100 mA and fry the detector; we will be VERY upset with you!

Special considerations for low 2theta scans

Most people work with 2 theta ranges between 2 and 100 degrees. On occasion, however, you may need to study the very low 2 theta range. If you will be working at 2 theta below 2 degrees, see a member of the MSE staff for special advice on how to proceed. The danger, of course, is the same as that for the single crystal samples -- the detector may catch a piece of the direct beam and get fried. There are special slits you will need to use to cut down the direct beam.

4. Shutting down

When you are finished using the rotating anode, if you are NOT the last user of day for rotating anode XRD, you can skip steps 01 ,02 and go to step 03.

01. Shut down the machine. First, turn down the current to 10 mA. Then turn down the voltage to 20 KV. Do both of these fairly quickly but not too quickly - 1 step per second is fine. Wait one minute after you turn the voltage and current down to their minimum values to let the anode cool a bit before turning it off.

02. Next, push the Off button. This will turn off the X-rays.

03. Remove your sample and clean up the lab as necessary.

04. Sign out user log sheet.

5. Fix Common Rigaku Problems

01. Vacuum Problem?

If you do not see any water leaking from top of X-Ray tube area, you can try this to recover the vacuum:

Case 1, if vacuum lights off with red light on, push the red OFF button, and then push the ON button. Then if vacuum lights come on, you are ready to turn the X-Ray on. If vacuum lights still off, go to case 2.

Case 2, turn the operate knob to stop, make sure the "Ready" light on the left panel is on, turn the Operate knob to Operate position again. Then you have to wait the vacuum pump down to 3 lights again. It might take 0.5 ~ 2 hour depending on how long the pump has been off.

02. No X-Ray detected?

Close the DataScan5 program.

Cycle the power for the D-max controller that is between the XRD and computer. Swing open the door, turn the Power switch on the right most off, wait a few seconds and turn it back on. Close the door.

Reopen the DataScan5, wait a couple of minutes for the XRD to finish Datum, then you are ready to choose other side and use it

