Standard Operating Procedure:
Bede D1 XRD

(You must be trained by MSE Staff or Authorized Trainer to use this machine)

Last updated by: Ying Qi January, 2014

Location: Room 2219 Dow Building

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: (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.

Typical Experiment Sequence:

1: Sign in Log sheet.
2: Turning on the machine and preparing for alignment
3: Aligning the detector and optics stage
4: Aligning the position of the sample
5: Running a rocking curve (double axis) Scan and/or Running a reciprocal space map
6: Shut down the system
7: Sign out Log sheet.

Procedures:

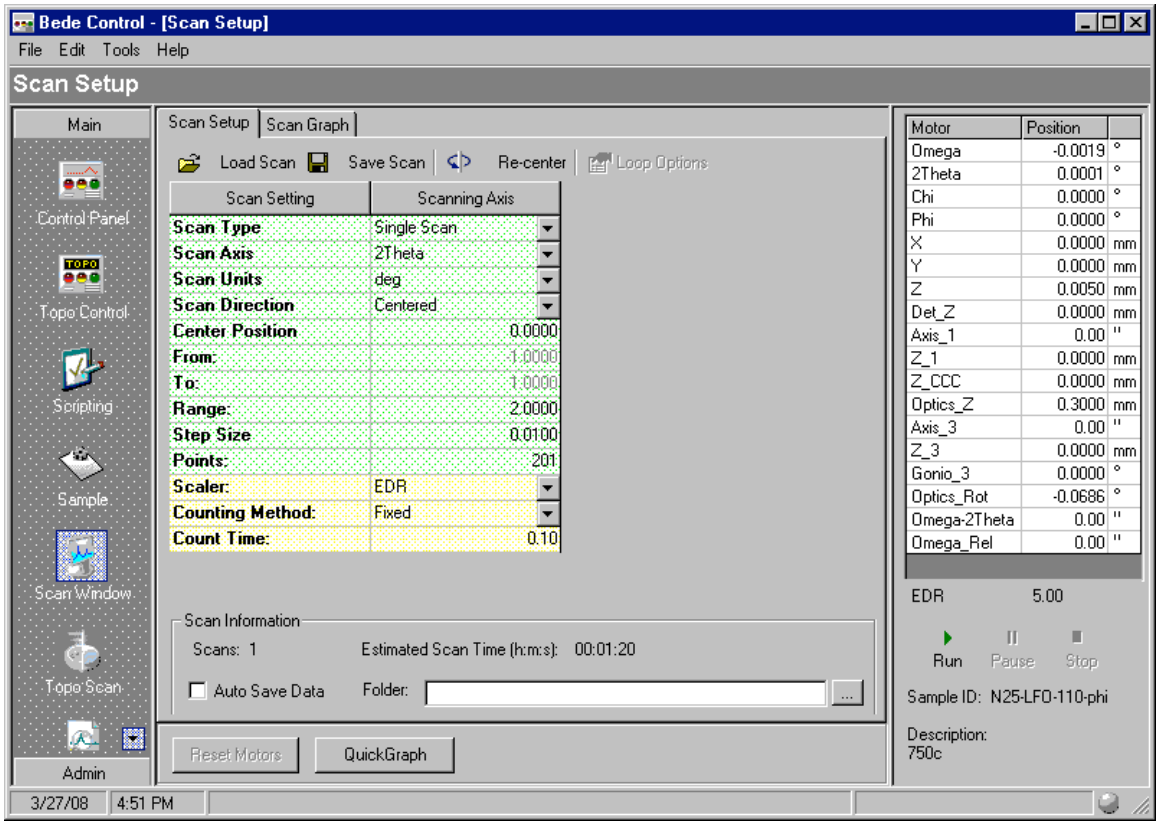
Turning on the machine and preparing for alignment:

1. Push the blue X-Ray OFF button to show current settings for current and voltage.
2. If not set as so, turn current to 5mA and voltage to -20kV while holding in the X-Ray OFF button. This is the ideal start condition.
3. Push the red X-Ray ON button. Wait until it reach and stay at -20kV, 5mA.
4. Slowly increase the voltage to -30kV and current to 20mA. Voltage first.
5. Open the enclosure and put the vertical slit into the detector stage holder on the smallest slit.
6. Make sure that the 3rd crystal is moved all the way out of the beam. These are all under computer control so do NOT move them manually!)

7. Slowly and gently close the enclosure door. Keep holding the door until it is closed completely. Don't drop it or you will damage the door and mess up alignment. Then push the "Shutter open" button to open the shutter.

Aligning the detector and optics stage:

1. Launch "**Bede Control**" software. Use student/student as username/Password.
2. Run a scan over 2 theta. To do this, enter the "Scan Window" and set up a scan with the following parameters in windows:



3. Uncheck "Auto Save Data" if it is checked. Then click Green Arrow "Run" button.
4. After the scan is finished, check both the intensity and the location of the peak. If the intensity is less than 200,000 counts, the optics stage needs to be realigned (step 5~step 10). Please ask Ying Qi (yqi@umich.edu) if you need help. If the intensity is more than 200,000 counts proceed to step 10. Remember to click "Reset Motors" button before exiting the data window!
5. *Remove the detector slit,*
6. *Adjust Axis 1 with step of 1 arcsec. Change the direction if intensity drops while adjusting Axis_1 until reach maximum intensity.*
7. *If nobody used Bede on the same day before you and you could not get intensity above 200,000 cps, please ask Ying Qi (yqi@umich.edu).*

8. *Put the detector slit back in.*
9. *Re-run the scan over 2theta. Note the intensity and the location of the peak.*
10. Move 2theta to the angle where it has the highest intensity and calibrate this to zero by pushing "C" then entering zero and hitting OK.

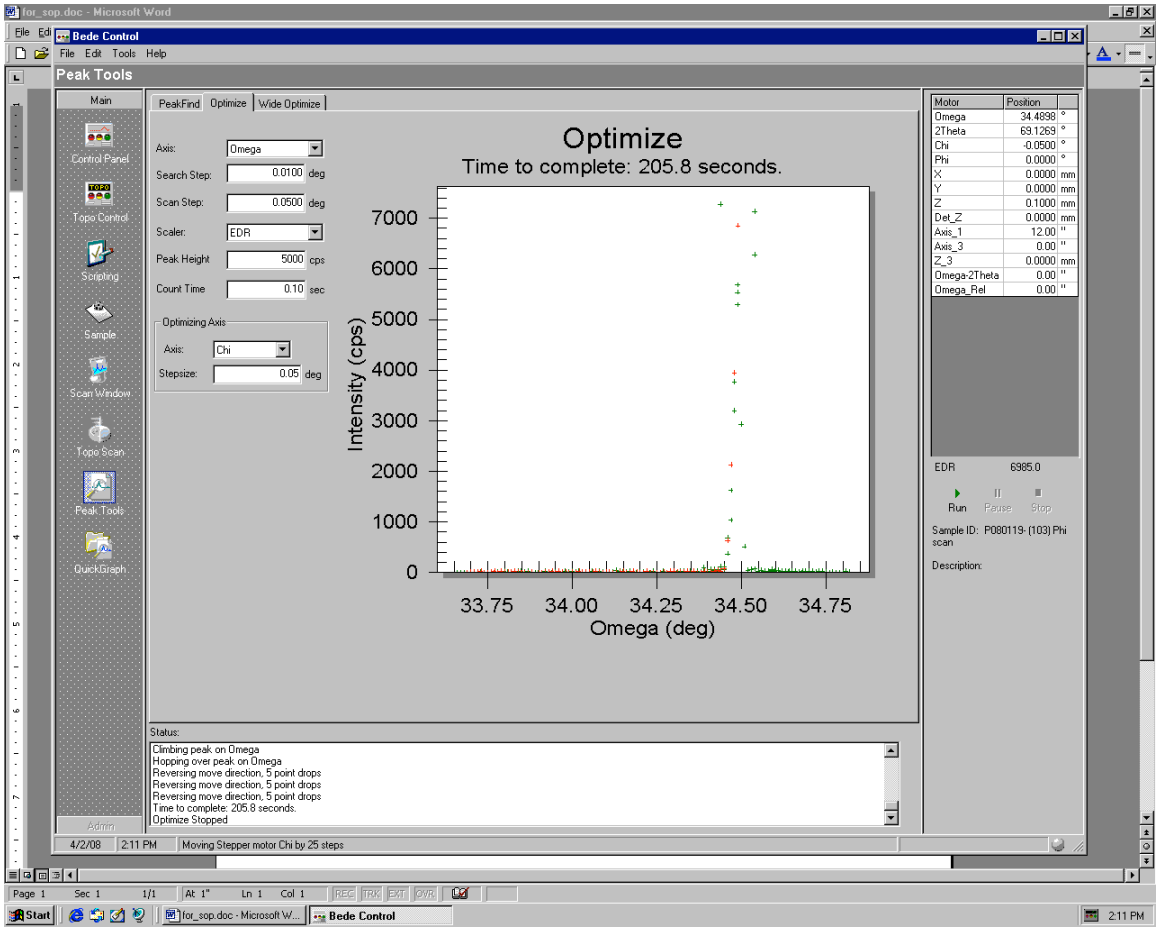
Aligning the position of the sample:

1. Open the enclosure and load your sample to the sample stage. Close the enclosure and turn on shutter.
2. Adjust Z axis so the intensity is cut in half: If you have Zero counts then your sample is completely blocking the beam and you need to move the sample towards the back of the machine (+Z). If you have the same intensity as before, your sample is not in the beam at all and you need to move it towards the front of the machine (-Z). Do this in the "Control Panel" menu by choosing the Z axis and adjusting Z position in microns until about half of the highest intensity.
3. Adjust the Omega axis until highest intensity is achieved.
4. Repeat 2,3 steps as needed.
5. Calibrate Omega axis to zero. Leave Z axis as is.

Running a rocking curve (double axis) Scan:

Note: Do not let Detector expose to direct strong X-ray beam at 40kV/40mA. Make sure shutter is closed before you increase X-Ray intensity.

1. Make sure shutter is closed when you increase the X-Ray intensity. Increase the intensity to -40 kV and 40 mA.
2. Remove the detector slit. **This is Critical because when moving to the Bragg angle the arm may run into the slit!!!**
3. Move the 2theta and the Omega axis to the Bragg angles of your sample substrate. Move 2 theta first and then Omega.
4. These values may not be precise. You will need to run a centered scan of the omega axis of approximately 3000 arc seconds. Move the omega axis to the maximum of the peak. Do not calibrate it to zero.
5. Adjust the position of Omega and Chi to maximize the intensity and narrow the FWHM: You will need to iterate between the two. Option 1: Move chi one step (usually 0.05 degree), then do Omega scan to check the maximum intensity change. Move chi another step to the direction that leads to higher intensity until highest intensity and smallest FWHM are achieved. Another option is to use "Optimize" in the "Peak Tools" menu:



Set the scanning axis as Omega and the optimizing axis as Chi with step size of 0.05º. The Omega and Chi will automatically move to the peak position after the scan.

6. You are now ready to collect your rocking curve data. Check "**Auto save data**" box and choose a folder where you want to save your data. You have two type of scans depending on your application:
   - Theta rocking curve is used when the range of the scan is small and there is no fringe detail. No detector slits are used for this scan. Scan over the omega axis and collect data.
   - Theta/2theta rocking curve is used when the scan area is large and/or fringe detail exists. Place the narrowest slit in the detector slit holder. Re-optimize the 2theta axis so that a maximum intensity is detected. Run a scan over theta/2theta.

Running a triple axis scan in preparation for reciprocal space mapping:

1. Do all the steps above: align detector and sample.
2. Put in the smallest detector slit if not already in.
3. Adjust 2theta to a maximum
4. Move the 3rd crystal, Z3, into place (-46 mm from the hardware limit). This limits the amount of information entering the detector to a few arc seconds.
5. Move Det Z by 4.3 mm to compensate for the 3rd crystal placement.

6. Adjust Axis 3 I arc seconds to achieve maximum intensity. The intensity should drop about 1/3 of intensity before 3rd crystal in. if it is not, the alignment needs to be perfected.
7. Run a theta/2theta scan to find the range over which you should run the reciprocal space map in that direction. If your scan has only the substrate peak and the film peak is missing you need to perfect the alignment of the system.
8. Run a scan over omega to determine the width of the film peak for the reciprocal space map.

Running a reciprocal space map:

1. Do all of the steps for "running a triple axis scan"
2. Set up the scan.
    a. Define the movement of the "Loop" axis (also called the offsetting axis). In this steps of 2 arc seconds and a speed arc sec/sec. Add this axis to the scan setup list.

Setup the range and step-size for the scanning axis, in this case theta/2theta axis. Make sure that the parent axis of the theta/2theta scan is Omega. This is the range determined previously from the theta/2theta scan.

    b. Set up the setting for the scaler. Make sure that the parent axis of the scaler is the theta/2theta axis.
    c. Be sure to set "Auto-save" to yes!
3. Please return Z3 to -20mm and Det_Z to 0 after you are done.

To shut down the system:

1. Close the shutter.
2. Return all of the Omega and 2theta to the "zero" position for the next user. Omega first then 2theta
3. Turn down mA to 5 and kV to -20
4. Turn off the X-rays by push the X-Ray OFF button.
5. Close the "BEDE Control" program.
6. Take out your sample and leave the sample holder inside the door.
7. Slowly and gently close the enclosure door. Keep holding the door until it is closed.
8. Sign out.