Abridged SOP for Manually Aligning a Sample and Collecting Data using XRD Commander

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This SOP describes the steps necessary to align a sample and manually collect single scans such as Rocking Curves and Coupled Scans (2Theta-Omega or Omega-2Theta).

This SOP is designed to act as a general guide that will work for most samples. You might be able to devise a more efficient procedure for your specific sample.

The main body of this SOP assumes that you know the general method for using XRD Commander, such as how to drive motors to a new position, set-up and collect a scan, optimize on a peak, zoom and redefine scan parameters by using the zoom. This SOP will instruct you to do these tasks using the keywords: Drive, Scan, Zoom, and Optimize. If you are not familiar with XRD Commander, then you should read Appendix A.

This SOP assumes that you know what (hkl) Bragg diffraction peaks you want to study and that you know how to determine the appropriate Bragg angle and tilt angle for those peaks using XRD Wizard, the “HRXRD Angle Calculation.xlsx” spreadsheet, or another method. A short list of common substrates and peaks are provided in Appendix D.

Additional SOP’s are available that will guide you through using XRD Wizard to collect and automatically save a scan and how to collect a reciprocal space map (RSM).
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I. **SUMMARY OF THE ALIGNMENT PROCEDURE**
This page provides a short reminder of the procedure used to align the sample. Following pages provide a more complete description.

I) Adjust the height of the sample by bisecting the beam
1) Use a small receiving slit (0.2mm)
2) Use a detector scan to optimize the position of the direct beam
3) Use a z-scan to optimize the position where the sample cuts the X-ray beam in half
4) Use a rocking curve to optimize the sample surface parallel to the X-ray beam
5) Repeat the z-scan and rocking curve until neither the optimal z nor the optimal Theta positions change by ± 1%.
   a) The z position is the optimal position and will not change during the rest of the measurements

II) Align on the symmetric substrate peak
1) Use a large receiving slit (1mm or larger)
2) Drive 2Theta and Theta to the theoretical values for the substrate
3) Use a rocking curve to optimize the sample tilt in the diffraction plane
4) Use a Chi scan to optimize the sample tilt in the axial plane
5) Repeat the rocking curve and Chi optimizations
6) Change the receiving slit to a small value (0.2mm or smaller)
7) Use a detector scan to optimize the 2Theta position
   a) The precision of the optimization depends on the size of the X-ray beam
8) Use a rocking curve to optimize the sample tilt
9) Use a Chi scan to optimize the sample tilt
10) Use a 2Theta-Omega scan to optimize the 2Theta position
11) Use a repetition of rocking curve and 2Theta-Omega scans to optimize Omega and 2Theta
12) Collect the data that you want

III) Align on an asymmetric peak (grazing exit or grazing incidence)
1) Use a large receiving slit (3mm)
2) Drive 2Theta and Omega to the theoretical values for the substrate
   a) Include the offset values that you determined when aligning the symmetric peak
3) Use a Phi scan to find the rotation of the sample that will let you see the asymmetric Bragg peak
4) Use a rocking curve to optimize Omega
5) Use a Phi scan to optimize rotation
6) Use a rocking curve to optimize Omega
7) Use a Chi scan to optimize Chi tilt
8) Repeat the series of optimization of Omega, Phi, and Chi until none change by +/- 1%
9) Use a small receiving slit (0.2mm or smaller)
10) Use a detector scan to optimize 2Theta
11) Use a rocking curve to optimize Omega
12) Use a 2Theta-Omega scan to optimize 2Theta
13) Repeat the series of optimization of Omega using a rocking curve, Phi, Chi, and 2Theta using a 2Theta-Omega scan until none of the optimized values change by +/-1%
14) Collect data
1. **Hardware Choices to Make Before Doing the Measurement**

1. **Decide what Incident-Beam Configuration you want to use.**

The Goebel mirror is always mounted, as is the rotary absorber. The Goebel mirror can be used by itself or it can be coupled with an incident-beam monochromator.

By default, the Ge(022)x4 asymmetric monochromator is always mounted. If you want to use a different incident-beam configuration, contact SEF staff when you make your appointment to arrange for the optic to be changed.

<table>
<thead>
<tr>
<th>Incident-Beam Configuration</th>
<th>Intensity (cps)</th>
<th>Beam Spectrum</th>
<th>Beam Divergence (arc-seconds)</th>
<th>FWHM of Si(220)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mirror only</td>
<td>170,000,000</td>
<td>Kα1+Kα2</td>
<td>108</td>
<td>0.07°</td>
</tr>
<tr>
<td>Ge(022)x4 asymmetric</td>
<td>18,000,000</td>
<td>Kα1</td>
<td>25</td>
<td>0.008°</td>
</tr>
<tr>
<td>Ge(044)x4 symmetric</td>
<td>150,000</td>
<td>Kα1</td>
<td>7</td>
<td>0.0015°</td>
</tr>
</tbody>
</table>

- The Ge(022)x4 Asymmetric Monochromator provides the best combination of intensity and resolution
  - This monochromator is recommended for HRXRD of most epitaxial films
  - This monochromator is recommended for XRR of thick films or complex multilayers
  - This optic can be used for XRR of most materials

- The Ge(044)x4 Symmetric Monochromator provides the best resolution
  - Recommended for HRXRD of lattice-matched films and multilayer superlattices
  - This optic is never used for XRR
  - It takes over an hour for this optic to be put on to the instrument, since it must be aligned.
    If you want to use this optic, please coordinate with SEF staff before you reserve the instrument.

- The Mirror, used by itself, provides very high intensity and a pseudo-parallel beam
  - Recommended for XRR of thin films, especially those <10 nm thick
  - Can be used for Bragg diffraction studies of poor quality films
  - Can be used for GIXD of polycrystalline films
  - The SEF no longer offers the use of the Bruker HRXRD with only the mirror. If you require more intensity for your research, you will get better results using the Rigaku SmartLab instead.

*Please remember that you are never allowed to remove or insert the incident-beam monochromator. If you want the monochromator changed, you must contact SEF staff to make the change for you. Requests to change the monochromator should be e-mailed to settens@mit.edu before you reserve the instrument.*
2. Decide what Detector System and Receiving-Side Optics you are going to use
There are three different receiving-side options available on the Bruker D8 HRXRD. Two of these use the Pathfinder detector system and one uses the LynxEye detector. You can change between all three beam paths from the software—no physical change to the instrument is required.

♦ **The Pathfinder detector** system is a scintillation point detector with two beam paths.
  ○ The Ge(022)x3 Analyzer crystal offers the better resolution and lowest background noise for higher quality data. However, the film must have a low defect concentration or else very little signal will reach the detector.
  ○ The variable receiving slit can be used for double-axis diffraction or for higher intensity triple-axis diffraction. This optic is used during sample alignment, for films that have a high defect concentration and therefore do not produce enough signal through the analyzer crystal, or for “quick and dirty” measurements when the highest resolution is not necessary.

♦ **The LynxEye detector** is used for high-speed reciprocal space mapping of Bragg peaks from epitaxial thin films. The detector can observe $2.7^\circ$ 2Theta simultaneously, so that it can collect a 2Theta (detector) scan without moving. This allows the LynxEye to quickly collect reciprocal space maps, since it is measuring a large portion of reciprocal space simultaneously. However, the LynxEye has limited resolution and the data tend to be noisy.

3. Checking Instrument Status and Opening Doors
1) Before opening the enclosure doors
   • Look at the interior right-hand side of the enclosure. There is a black box with several warning indicator lights.
   • The orange “X-RAY ON” lights should be lit. These indicate the generator is on and producing radiation.
   • The green “SHUTTER CLOSED” lights should be lit.
   • If the green “SHUTTER CLOSED” lights are not lit or if the red “SHUTTER OPEN” lights are lit, then do **not** open the doors.
     ○ Look at the instrument computer and determine if a measurement is in progress. If so, wait until it finishes or manually stop it by pressing the STOP button in the software.
     ○ If no measurement is in progress, then something is wrong. Do **not** attempt to operate the instrument. Contact SEF staff to report the problem.

2) To open the enclosure doors
   • On either column on the lower sides of the instrument, find the green “Open Door” button. Press this button to unlock the doors.
   • Pull the door handle out towards you. Gently slide the doors open.
   • To close the doors, gently slide the doors closed. Push the handles in towards the instrument.
III. Setting the Incident-Beam Monochromator Slits

Slits are used to control the height and width of the incident X-ray beam. Before you begin collecting data, you should check what slits are inserted into the incident-beam optics. All authorized users of the Bruker D8 HRXRD are allowed to change the incident-beam slits.

The length (L) of the X-ray beam on the sample is: \( L = \frac{h}{\sin(\omega)} \), where \( h \) is the height of X-ray beam determined by the slit and \( \omega \) is the incident angle.

Slits are stored in the wooden box labeled “Slits for Bruker D8 HRXRD”

When you insert a slit, make sure it is straight vertically and that it slides all of the way in (about 1cm of the slit will be sticking out of the slot). The blades of the slit will be facing the X-ray tube (the numbers on the slit should be oriented upside down with respect to the sample position).

- **The Gobel mirror slit** should always be 0.8mm.
- **The Rotary Absorber slit** is used to restrict the beam width
  - If left empty, the beam will be about 12mm wide
  - The 6mm slit will limit the beam width to ~6mm
  - The 1mm pinhole will limit the beam width to ~1mm
- **The first monochromator slit** is used to restrict the beam height, which determines the length of the irradiated area on your sample. A larger slit will give you a larger irradiated area and therefore more intensity from your film, but it might compromise resolution if your film is not homogeneous or if the substrate is curved.
  - The 0.6 and 0.2mm slits are the most commonly used for HRXRD.
  - The 0.2, 0.1, and 0.05mm slits are used for XRR
  - The choices for this slit are 0.8, 0.6, 0.2, 0.1, or 0.05mm.
- **The second monochromator slit** should contain a 1mm height limiting slit.
- **A Soller slit to limit axial divergence** is useful if you are going to be tilting Chi in order to observe an offcut substrate or asymmetric reflections or if you are studying diffuse scatter. The Soller slit will reduce the effects of defocusing on peak shapes when Chi is tilted. CMSE SEF staff (ie Scott) must insert the Soller slit if you want one.
- **A collimator is available to limit the X-ray beam width to 1mm.** Talk to SEF staff if you would like to learn how to use this attachment.
IV. PREPARING TO COLLECT DATA

The instructions below give some descriptions of how to perform align and collect a triple-axis scan in XRD Commander. If you are not familiar with XRD Commander, read Appendix A (pg 22) which gives an overview of how to use XRD Commander.

1. Start the programs XRD Commander and XRD Wizard
2. Select the program XRD Commander
3. Select the Adjust page
   a. There are four tabs along the bottom of the XRD Commander window, labeled Adjust, Jobs, Geometry, and Details

4. Set the X-Ray Generator power to 40 kV and 40 mA.
   • Give the generator at least 30 minutes at full power to warm up before beginning your measurements!!
   a. The generator controls are located on the left-hand side of the XRD Commander window
   b. The black numbers are the desired value, the blue numbers are the current value
   c. Change the black numbers for kV and mA to the desired setting, 40kV and 40mA
   d. Click on the Set button
   e. Wait until the actual values (in blue) change to the desired value

5. Set the detector
   a. Select the Details tab
   b. In the upper right-hand corner of XRD Commander, make sure that Detector 1 is selected, not PSD.
   c. Select the Adjust tab
   d. Select the Secondary Optic using the drop-down menu
   i. Select Pathfinder-Variable Slit
   ii. The drop down menu for the secondary optic is the second blank drop-down box in the Toolbar for XRD Commander
   iii. Float the mouse over the button to see its name (Secondary Optic)
   iv. After you select the Secondary Optic from the drop-down menu, the button will be filled with the icon for that optic.
   v. If you want to use the Triple Ge220 Analyzer crystal to collect your data, you will switch to that optic later in the data collection process.
• To **Drive** a motor, type the target value in the Request value column. Once the number is typed, click the **Move Drives** button (circled in red).
• Be sure that you do NOT accidentally click on the **Initialize** button instead of the **Move Drives** button (they look very similar).
  o The **Move Drives** button has a green arrow in the icon.
  o The **Initialize** button has a yellow circle in the icon.

**V. MOUNTING THE SAMPLE**
The sample is mounted using a vacuum chuck. The thicker (ie heavier) your sample is, the more area your sample must cover in order to be held firmly in place. The vacuum is controlled through a series of holes placed along circles of different diameters. Your sample must cover all of the holes of a specific diameter in order for the vacuum to hold it. A general rule of thumb is:

• If your sample is <0.2mm thick, it must be 5 x 3mm so that it covers the innermost 2 holes
• If your sample is between 0.2 and 1mm thick, it must be 11 x 11mm so that it is large enough to cover the innermost 6 holes
• If your sample is >1mm thick, it must be 22 x 24mm so that it covers the innermost 12 holes.
• If your sample size does not meet these requirements, then you will need to mount it on a glass cover slip using an adhesive

1. **Drive** Chi to 90
   a. If Theta and 2Theta are at high angles, it will be difficult to reach the sample stage. You may want to **Drive** both of them to 0 as well.
2. Place your sample on the center of the vacuum chuck, covering the necessary holes.
3. Open and close the appropriate valves
   a. The holes open by the white valves on the side of the chuck. Each valve controls the holes along a specific diameter of circle; the diameter of the circle is labeled by the valve.
   b. The innermost holes are controlled by the valves on the left. Each valve to the right controls the next largest diameter of holes.
   c. The valve should point to the left to open the holes; point to the right to close the holes
   d. If a hole is opened but not covered by the sample, then the vacuum will not hold
4. Turn on the vacuum pump by turning on the power strip to the left of the goniometer.
5. When done, gently close the doors to the enclosure
**Important Information for Running Scans**

- In XRD Commander, the Receiving Slit is mislabeled as “Antis. Slit”. In the instructions below, it will be named “Antis. Slit” when the instructions tell you to set it to a specific value. However, when the instructions provide additional information about its function, it will be properly called the “Receiving Slit” (or Rec. Slit).
- When the instructions call for a scan, they will tell you to **Start** the scan (you press the Start button to initiate the scan). After that line, they will specify the scan parameters. You must:
  - Always set the **Scantype** before changing any other parameters
  - Always use the **Continuous** scan mode and not the Step scan mode
    - The button to the right of the Scantype drop-down menu should read “Continuous” and not “Step”. Click on the button to toggle between settings.
  - The units for the Scanspeed are controlled by the button to the right of the Scanspeed text box. Click on the button to toggle between **Sec/Step** and Deg/Min. All instructions are written for **Sec/Step**.

**VI. ALIGN Z BY BISECTING THE BEAM**

- **Why**- we need to optimize Z so that the X-ray beam is properly focused on your sample. We do this by determining the value of Z where the sample cuts the X-ray beam in half.

1. **Set** the Absorber
   a. Using the **Absorber** drop-down menu, select a value
   b. Click the **Set** button
      - If using the Ge(022)x4 monochromator, **Set** the Absorber to 78.2
      - If using the Ge(044)x4 monochromator, **Set** the Absorber to 1

2. Determine the position of the direct X-ray Beam by using a Detector Scan
   a. **Drive** the instrument to the following positions:
      i. Theta=0
      ii. 2Theta=0
      iii. Antis. Slit= 0.2
      iv. Phi=0
      v. Chi=0
      vi. X=0
      vii. Y=0
      viii. Z=-1.5
   b. **Start** a Detector Scan
      i. Scantype= Detector Scan
      ii. Start= -0.2
      iii. Increment= 0.002
      iv. Stop= 0.2
      v. Scanspeed= 0.2 sec/step
c. Make sure that the instrument intensity is within the specification.
   i. When using the Ge(022)x4 asymmetric monochromator, the peak intensity should be:

<table>
<thead>
<tr>
<th>Height Limiting Slit in First Monochromator Slit Position</th>
<th>Width Limiting Slit in Rotary Absorber Slit Position</th>
<th>Minimum Acceptable Intensity in cps</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6 mm</td>
<td>None</td>
<td>&gt; 190,000 cps</td>
</tr>
<tr>
<td>0.2 mm</td>
<td>None</td>
<td>&gt; 107,000 cps</td>
</tr>
<tr>
<td>0.2 mm</td>
<td>6 mm</td>
<td>&gt; 56,000 cps</td>
</tr>
</tbody>
</table>

ii. If the intensity is less than listed above, or if the peak is highly asymmetric, then notify SEF staff so that they can check the instrument alignment.

d. Redefine the peak maximum as 0° 2Theta
   i. click on the Zi button in the toolbar to open the Zi Determination window
   ii. Set “Enter theoretical position” to 0
   iii. Click Save and Send new Zi
   iv. If a dialog box prompts you for a password, leave the password blank and click OK
   v. Repeat the Detector Scan and make sure that the peak is centered around 0° 2Theta
      • If the X-ray beam has an odd shape, such that the Zi peak search does not properly identify the peak centroid, then see Appendix B (pg 28) for the refined procedure.

3. Determine the Z position where the sample cuts the X-ray beam intensity in half
   a. Drive 2Theta to 0
   b. Start a Z scan
      i. Scantype= Z
      ii. Start= -1.0
      iii. Increment= 0.01
      iv. Stop= 1.0
      v. Scanspeed= 0.1 sec/step
   c. Optimize at the point on the chart where the X-ray intensity is ½ the maximum intensity
      i. If the intensity of the X-ray beam in the Z-scan does not go all the way to zero, then see Appendix C (pg 29) for details on how to deal this.

4. Make sure that the sample surface is parallel to the X-ray beam
   a. Start a Rocking Curve Scan
      i. Scantype= Rocking Curve
      ii. Start= -1
      iii. Increment= 0.01
      iv. Stop= 1
      v. Scanspeed= 0.1 sec/step
   b. Optimize on the center of the maximum
5. Iteratively improve the alignment of Z and Theta
   a. Repeat the Z and Rocking Curve scans until the optimal position for both does not change by more than ±1% between successive scans
   b. The Z Scans that you use should have parameters:
      i. Scantype= Z
      ii. Start= optimized Z position – 0.3
      iii. Increment= 0.005
      iv. Stop= optimized Z position + 0.3
      v. Scanspeed= 0.1 sec/step
   c. The Rocking Curve scans that you use should have parameters:
      i. Scantype= Rocking Curve
      ii. Start= optimized Theta position – 0.5
      iii. Increment= 0.005
      iv. Stop= optimized Theta position + 0.5
      v. Scanspeed= 0.1 sec/step

6. When you have determined the optimal aligned Z value
   a. Drive Z to the optimized value
   b. Uncheck the box next to Z so that Z will not be changed again
   c. This optimal Z value will not change for any of the scans of your sample

7. You might want to record the optimized value of Theta as Tilt(sample)
   a. Tilt(sample) indicates the Theta value when the physical surface of the sample is parallel to the X-ray beam
   b. If the tilt of the substrate peak (determined later) is significantly different than tilt(sample), this would indicate a miscut in the substrate

VII. ALIGN THE SYMMETRIC SUBSTRATE PEAK
- We want to align on the symmetric Bragg peak of the substrate.
- Measurements of your film are made relative to the substrate peak.
- In these alignment procedures, the most important thing is to align the sample so that it produces the most intense and sharpest rocking curve from the substrate peak.
  o If you are not sure what the optimal value is for a position, such as the Chi tilt, then collect rocking curves at different values for that position. The optimal position is the one that gives you the most intense and sharpest rocking curve.
- Remember, a rocking curve collected with a large Receiving Slit will allow you to see the contributions from your substrate and any film peak that has a d-spacing value close to that of your substrate.
  o When you see multiple peaks in the rocking curve, the substrate peak will almost always be the most intense and sharpest
  o For very thick films, the substrate peak might not be the most intense
  o We begin alignment using a large Receiving Slit because the optimal 2Theta may be shifted from the theoretical Bragg peak position by effects such as substrate strain and the dynamical scattering refractive index effect.
1. **Set** the Absorber to 1

2. **Drive** the goniometer to 2Theta and Theta values for the substrate’s symmetric Bragg peak.
   a. The Theta value should be equal to \( \frac{1}{2} \) (2Theta)
      i. If \( \text{tilt(sample)} \) was large, you might add that to the calculated Theta value.
   b. If you do not know the 2Theta values for your substrate, then:
      i. You can find 2Theta values for common substrates in Appendix D (pg 31)
      ii. You can look up peak position from a reference database using a program such as HighScore Plus, as described in Appendix F.
      iii. For cubic substrates, you can use the “HRXRD Angle Calculation.xlsx” spreadsheet, found on the desktop of the data collection computer
      iv. You can use XRD Wizard, as described in the “Using XRD Wizard” SOP

3. Optimize the substrate tilt in the diffraction plane (Omega) using a Rocking Curve scan
   a. **Drive** the Antis. Slit to a large value, between 1 to 3 mm
   b. **Start** a coarse rocking curve
      i. Scantype= Rocking Curve
      ii. Start= \( \text{current Theta position} - 1 \)
      iii. Increment= 0.01
      iv. Stop= \( \text{current Theta position} + 1 \)
      v. Scanspeed= 0.1 sec/step
   c. Collect a more precise rocking curve
      i. **Zoom** around the peak and click the **Use Zoom** button
      ii. Change the increment to 0.005 or 0.002 deg
      iii. **Start** the rocking curve
   d. **Optimize** on the Rocking Curve Peak
      i. Optimize on the center of mass of the parabola that defines the top half of the peak
      ii. This will not necessarily be the maximum of intensity

4. Optimize the substrate tilt in the axial direction (Chi)
   a. **Start** a Chi scan
      i. Scantype= Chi
      ii. Start= -2
      iii. Increment= 0.02
      iv. Stop= 2
      v. Scanspeed= 0.1 sec/step
   b. **Optimize** on the centroid of the peak
      i. If the peak is too broad to clearly resolve the maximum, then repeat the Chi scan using a range from -4 to 4 deg with a 0.05deg increment
      ii. **If the peak has multiple maxima or an unusual shape, then:**
         1. Determine the Chi values that correspond to each maxima and minima
         2. Collect rocking curves with Chi set to each of those values
         3. The optimal Chi position is the one that produces the most intense rocking curve
5. Optimize the Rocking Curve again
   a. **Set** the scan type to Rocking Curve
   b. **Start** the Rocking Curve using the previous scan parameters
   c. **Optimize** on the Rocking Curve

6. Repeat steps 4 and 5 (optimize Chi and optimize rocking curve) until both are optimized
   a. The optimum rocking curve and Chi positions should not change by more than ±5% between successive scans
   b. Chi should be optimized to produce the most intense rocking curve

7. Use a Detector Scan to optimize 2Theta for the Bragg peak
   a. **Drive** the Antis. Slit to 0.2mm or smaller
   b. **Start** a coarse detector scan
      i. Scantype= Detector Scan
      ii. Start= current 2Theta position – 0.5
      iii. Increment= 0.005
      iv. Stop= current 2Theta position + 0.5
      v. Scanspeed= 0.1 sec/step
   c. Collect a more precise detector scan
      i. **Zoom** around the peak and click **Use Zoom** to redefine the start and stop positions
      ii. Change the increment to 0.002 deg
      iii. **Start** the Detector Scan
      iv. **Optimize** on the centroid of the detector scan peak

8. Optimize Theta using a rocking curve
   a. **Set** the scan type to Rocking Curve
   b. **Start** a Rocking Curve using the previous scan parameters
   c. **Optimize** on the Rocking Curve
   d. The rocking curve may be much sharper once 2Theta is aligned and the receiving slit is made smaller. If this is the case, then:
      i. **Zoom** around the peak and click **Use Zoom** to redefine the start and stop positions
      ii. Change the increment to 0.002 or 0.001 deg
      iii. **Start** the Rocking Curve scan
      iv. **Optimize** on the centroid of the rocking curve

9. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
   a. **Start** a 2Theta-Omega scan
      i. Scantype= 2Theta-Omega
      ii. Start= current 2Theta position – 0.2
      iii. Increment= 0.002
      iv. Stop= current 2Theta position + 0.2
      v. Scanspeed= 0.1 sec/step
   b. **Optimize** on the centroid of the 2Theta-Omega scan
10. Optimize the Rocking Curve and Chi with the detector at the new optimal 2Theta position. For each optimization below, use the previous scan parameters for the initial scan. If the peak is significantly sharper than before, Use Zoom to redefine the start and stop positions and change the increment to a smaller value.
   a. Use a Rocking Curve scan to Optimize Theta
   b. Use a Chi scan to Optimize Chi
   c. Use another Rocking Curve to Optimize Theta again.
      i. If the optimal Theta position did not change by more than ±1%, you are done
      ii. If the optimal Theta position did change by more than ±1%, repeat steps c and d

11. Set the Receiving-Side Optic to the Value that you will use to Collect Data
   a. Your final scan can be collected using a Receiving Slit or an Analyzer Crystal
      i. If the measurement will be collected using the same size or a larger receiving slit than
         the current value, then leave the receiving slit at its current value.
      ii. If the measurement will be collected using a smaller receiving slit than the current
         one, then Drive the receiving slit size to the final desired value.
      iii. If using the analyzer crystal, then change the secondary optic to the “Pathfinder-
           Triple Ge220 Analyzer” using the drop-down menu

12. Final Optimization Sequence
   • As a starting point for each optimization below, use the previous scan parameters for the
      initial scan.
      o The peak might be significantly sharper than before, especially if you inserted the
         analyzer crystal.
         ▪ If the peak is significantly sharper than before, Use Zoom to redefine the start and
           stop positions and change the increment to a smaller value.
         ▪ Using an analyzer crystal, the final increments might be between 0.0005 and
           0.0001 deg
         ▪ Using a receiving slit, the final increments might be between 0.004 and 0.0005 deg
   a. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
   b. Optimize the Theta position using a Rocking Curve
   c. Optimize the Chi tilt using a Chi scan
      i. If you inserted the analyzer crystal, you might find that the chi plot has changed
         significantly
   d. Optimize the Theta position using a Rocking Curve
   e. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
   f. Optimize the Theta position using a Rocking Curve
      i. If the optimal Theta position did not change by more than ±1%, you are done
      ii. If the optimal Theta position did change by more than ±1%, repeat steps c through h
VIII. Collect Data from the Symmetric Peaks

- You should collect a coupled scan
  - This can be either a 2Theta-Omega scan or an Omega-2Theta scan
    - It is easier to extract d-spacing information from a 2Theta-Omega scan
    - The Omega-2Theta scan is more often referenced in HRXRD textbooks.
  - The coupled scan will allow you to determine the d-spacing of the Bragg peaks of your film, which can be used to calculate composition or relaxation of the film
  - If you do not see film peaks in the coupled scan, then your film is misoriented with respect to the substrate
  - In a simple film, the coupled scan will let you estimate film thickness
- You may want to collect a rocking curve for your substrate and film peaks
  - The Rocking Curve in a Triple-Axis Diffractometer measures the tilt distribution of the Bragg peak for a single d-spacing
  - The Rocking Curve can let you evaluate misorientation, dislocation content, mosaic spread, curvature
  - If you collect a rocking curve with a large receiving slit, this is effectively a Double-Axis Diffractometer rocking curve as long as your film is lattice matched to your substrate
    - The range of d-spacing observed in the Rocking Curve is limited compared to a conventional Double-Axis Diffractometer
- If you want to collect a Double-Axis Diffractometer Rocking Curve from a non-lattice matched film, try collecting an Omega-2Theta scan using a very large Receiving Slit

1. Collect your Coupled Scan
   a. Scantype= 2Theta-Omega or Omega-2Theta
   b. Start and Stop should be selected to encompass your film and substrate peaks, with some additional range if interference fringes are also present
      i. If using a 2Theta-Omega scan, then calculate the Start and Stop from the Bragg angles for the diffraction peaks
      ii. If using a Omega-2Theta scan, be sure to include any tilt offset determined during the alignment procedure in your start and stop angles
         1. You can calculate the $Tilt(Substrate)=\frac{1}{2}*2Theta(optimized)−Theta(optimized)$
         2. For each Bragg peak that you are collecting, making sure that the scan range includes them: the peak position will be $\frac{1}{2}*2Theta(Bragg)+Tilt(Substrate)$
   iii. You might want to use a fast, coarse coupled scan to make sure you have good start and stop values and to evaluate what scanspeed you should use.
   c. The increment should be between 0.005° and 0.0001°, depending on the optics:
      i. Ge(022)x4 and Rec Slit: 0.005 to 0.001° increment
      ii. Ge(044)x4 and Rec Slit: 0.004 to 0.0005° increment
      iii. Ge(022)x4 and Analyzer: 0.002 to 0.0005° increment
      iv. Ge(044)x4 and Analyzer: 0.0005 to 0.0001° increment
   d. Scanspeed should be at least 0.2 sec/step. More complex or defective films will require a slower scan
      i. Remember, if you are not sure what the scanspeed should be, you can use the Repeat option to repeatedly collect the scan, adding the results until you have a good enough signal for analysis.
2. Save your Scan
   - **Data are not saved automatically when you collect them with XRD Commander**
     a. Before saving the scan, you can type a description of the sample and/or experiment in the **Sample ID** line in the toolbar. This will be saved as the sample ID in the data header.
     b. Save the scan by going to *File > Save* or clicking on the **Save** icon in the toolbar.
     c. Save your data in the “Diffrac Plus Ext. RAW file (*.raw)” format.

3. Collect your Rocking Curves
   a. For each diffraction peak observed in your coupled scan that you want to collect a rocking curve for, you should:
      i. Double-click on the peak centroid (as if you were optimizing on that peak)
      ii. Record the 2Theta and Theta values that result when you double-click on the peak
      iii. These are the optimized 2Theta and Theta values that you want to use at the beginning of your rocking curve
   b. If there is a diffraction peak that you expected to see in the coupled scan, but did not, it might be misoriented with respect to the substrate. You can try collecting a scan from it by driving 2Theta to the expected value for that peak and collecting a rocking curve using a large receiving slit opening and a broad range for the rocking curve.
   c. **Drive** 2Theta and Theta to the optimized positions for the first diffraction peak that you are going to collect a Rocking Curve for.
   d. Set Scantype= Rocking Curve
   e. The Start and Stop values for the Rocking Curve should be centered around the optimized Theta position
      i. You might want to run a fast, coarse, long range Rocking Curve to determine the best scan range and scanspeed for the data collection
   f. The increment should be between 0.005° and 0.0001° depending on what optics you are using and the rocking curve width of your film
      i. Ge(022)x4 and Rec Slit: 0.005 to 0.001° increment
      ii. Ge(044)x4 and Rec Slit: 0.004 to 0.0005° increment
      iii. Ge(022)x4 and Analyzer: 0.002 to 0.0005° increment
      iv. Ge(044)x4 and Analyzer: 0.0005 to 0.0001° increment
   g. Scanspeed should be at least 0.2 sec/step. Broader rocking curves usually require a longer scan since the peak will diffract weakly
      i. Remember, if you are not sure what the scanspeed should be, you can use the Repeat option to repeatedly collect the scan, adding the results until you have a good enough signal for analysis.
   h. Save your Scan
      i. Data are not saved automatically when you collect them with XRD Commander
      ii. Before saving the scan, type a description of the sample and/or experiment in the **Sample ID** line in the toolbar. This will be saved as the sample ID in the data header.
      iii. Save the scan by going to *File > Save* or clicking on the **Save** icon in the toolbar.
      iv. Save your data in the “Diffrac Plus Ext. RAW file (*.raw)” format.
      i. Repeat for each rocking curve you want to collect.
IX. ALIGN THE ASYMMETRIC SUBSTRATE PEAK

1. **Drive** 2Theta and Theta to the values for the asymmetric Bragg peak of the substrate.
   a. Decide if you want to collect a grazing incidence (-) or grazing exit (+) asymmetric scan
      i. The grazing incidence scan is more sensitive to surface layers and will tend to produce more interference fringes; but the grazing incidence scan will tend to produce broader peaks providing less precise peak position information
      ii. The grazing exit scan will tend to produce sharper peaks providing more precise peak positions for composition and relaxation calculations; but the grazing exit scan will give less information about the surface layers and will be dominated by the substrate and thicker layers in the sample
   b. Determine the 2Theta(Bragg) position of the Bragg peak and the Tilt(Omega) required to observe the asymmetric peak
      i. You can find both of these values for common substrates in Appendix D (pg 31), by using XRD Wizard as described in the Using XRD Wizard SOP, or by using the “HRXRD Angle Calculation.xlsx” spreadsheet.
   c. Based on the symmetric scan, determine the 2Theta(offset) and Omega(offset)
      i. 2Theta(offset) = 2Theta(theoretical) - 2Theta(optimized)
      ii. Omega(offset) = ½ *2Theta(optimized) - Theta(optimized)
   d. **Drive** 2Theta = 2Theta(Bragg) + 2Theta(offset)
   e. **Drive** Theta = ½ *2Theta(Bragg) + Omega(offset) ± Tilt(Omega)

2. Use a Phi scan to find the rotation of the sample that will let you see the Bragg peak
   a. **Drive** the Antis. Slit to 3mm
   b. **Start** a coarse Phi scan
      i. Scantype= Phi
      ii. Start= -15
      iii. Increment= 0.5
      iv. Stop= 105
      v. Scanspeed= 0.1 sec/step
      vi. You can often tweak the scan range if you know approximately where the sample should be rotated
         1. The flat on a Si(001) wafer usually indicates the [110], so you can find the (224) or (115) by making the flat square with the X-ray tube. The (044) would be rotated 45° from this.
   c. If you do not see a peak in the Phi scan
      i. Try collecting again with a larger range
      ii. Try going to the other asymmetric peak (grazing incidence vs grazing exit) and recollect the Phi scan
      iii. Manually change Phi in 5° increments and collect rocking curves, looking for the Phi that allows you to see a peak in the rocking curve
         1. This works best in grazing incidence mode
         2. After you find a signal in the rocking curve, **Optimize** Theta and then collect a Phi scan around that position
d. Collect a more precise Phi scan
   i. **Zoom** around the peak and click the **Use Zoom** button
   ii. Change the Increment to 0.05
   iii. **Start** the Phi scan
   iv. **Optimize** on the center of the peak maximum

3. Optimize the substrate tilt in the diffraction plane (Omega) using a Rocking Curve
   a. **Start** a coarse rocking curve
      i. Scantype= Rocking Curve
      ii. Start= **current Theta position – 1**
      iii. Increment= 0.01
      iv. Stop= **current Theta position +1**
      v. Scanspeed= 0.1 sec/step
   b. Collect a more precise rocking curve
      i. **Zoom** around the peak and click the **Use Zoom** button
      ii. Change the increment to 0.005 or 0.002deg
      iii. **Start** the rocking curve
   c. **Optimize** on the Rocking Curve Peak
      i. You want to optimize on the center of mass of the parabola that defines the top half of the rocking
      ii. This will not necessarily be the maximum of intensity

4. Optimize the substrate tilt in the axial direction (Chi)
   a. **Start** a Chi scan
      i. Scantype= Chi
      ii. Start= -2
      iii. Increment= 0.02
      iv. Stop= 2
      v. Scanspeed= 0.1 sec/step
   b. **Optimize** on the centroid of the peak
      i. If there are two peaks, then optimize in between those peaks
      ii. If the peak is too broad to clearly resolve the maximum, then repeat the Chi scan using a range from -4 to 4 deg with a 0.05deg increment
      **iii. If the peak has multiple maxima or an unusual shape, then:**
         1. Determine the Chi values that correspond to each maxima and minima
         2. Collect rocking curves with Chi set to each of those values
         3. The optimal Chi position is the one that produces the most intense rocking curve

5. Optimize Theta using a Rocking Curve again
   a. **Set** the scan type to Rocking Curve
   b. **Start** the Rocking Curve using the previous scan parameters
   c. **Optimize** on the Rocking Curve
6. Optimize Phi again
   a. **Set** the scan type to Phi
   b. **Start** the Phi scan using the previous scan parameters
   c. **Optimize** on the centroid of the peak
   d. If necessary, **Use Zoom** to redefine the start and stop positions for a more precise scan

7. Optimize Theta using a Rocking Curve again
   a. **Set** the scan type to Rocking Curve
   b. **Start** the Rocking Curve using the previous scan parameters
   c. **Optimize** on the Rocking Curve

8. Repeat steps 4, 5, 6 and 7 (optimize Chi, Theta, and Phi) until all are optimized
   a. The optimum rocking curve and Chi positions should not change by more than ±5% between successive scans
   b. Chi should be optimized to produce the most intense rocking curve

9. Use a Detector Scan to optimize 2Theta for the Bragg peak
   a. **Drive** the Receiving Slit to 0.2deg or smaller
   b. **Start** a coarse detector scan
      i. Scantype= Detector Scan
      ii. Start= \( \text{current 2Theta position} - 0.5 \)
      iii. Increment= 0.005
      iv. Stop= \( \text{current 2Theta position} + 0.5 \)
      v. Scanspeed= 0.1 sec/step
   c. Collect a more precise detector scan
      i. **Zoom** around the Bragg peak and click **Use Zoom** to redefine the start and stop positions
      ii. Change the increment to 0.002 deg
      iii. **Start** the Detector Scan
      iv. **Optimize** on the centroid of the detector scan peak
         1. This peak will be very broad in grazing incidence geometry and sharper in grazing exit geometry

10. Optimize Theta using a rocking curve
    a. **Set** the scan type to Rocking Curve
    b. **Start** a Rocking Curving using the previous scan parameters
    c. **Optimize** on the Rocking Curve
    d. The rocking curve may be much sharper once 2Theta is aligned and the receiving slit is made smaller. If this is the case, then:
       i. **Zoom** around the rocking curve peak and click **Use Zoom** to redefine the start and stop positions
       ii. Change the increment to 0.002 or 0.001 deg
       iii. **Start** the Rocking Curve scan
       iv. **Optimize** on the centroid of the rocking curve
11. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
   a. **Start** a 2Theta-Omega scan
      i. Scantype= 2Theta-Omega
      ii. Start= current 2Theta position – 0.2
      iii. Increment= 0.002
      iv. Stop= current 2Theta position + 0.2
      v. Scanspeed= 0.1 sec/step
   b. **Optimize** on the centroid of the 2Theta-Omega scan

12. Optimize Theta, Chi, and Phi with the detector at the new optimal 2Theta position
   a. For each optimization below, use the previous scan parameters for the initial scan. If the peak is significantly sharper than before, **Use Zoom** to redefine the start and stop positions and change the increment to a smaller value.
   b. Use a Rocking Curve scan to Optimize Theta
   c. Use a Chi scan to Optimize Chi
   d. Use another Rocking Curve to Optimize Theta again.
   e. Use a Phi scan to Optimize Phi
   f. Use another Rocking Curve to Optimize Theta again.
      i. If the optimal Theta position did not change by more than ±1%, you are done
      ii. If the optimal Theta position did change by more than ±1%, repeat steps c and d

13. Final Optimization Sequence
   a. Set the Receiving Side Optic
      i. If the measurement will be collected using the analyzer, insert it now
      ii. If the measurement will be collected using a smaller receiving slit than the current one, then **Drive** the receiving slit size to the final desired value.
      iii. If the measurement will be collected using the same size or a larger receiving slit than the current value, then leave the receiving slit at its current value.
   b. For each optimization below, use the previous scan parameters for the initial scan. If the peak is significantly sharper than before, **Use Zoom** to redefine the start and stop positions and change the increment to a smaller value.
   c. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
   d. Optimize the Theta position using a Rocking Curve
   e. Optimize the Chi tilt using a Chi scan
   f. Optimize the Theta position using a Rocking Curve
   g. Optimize Phi using a Phi Scan
   h. Optimize the Theta position using a Rocking Curve
      i. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
      j. Optimize the Theta position using a Rocking Curve
         i. If the optimal Theta position did not change by more than ±1%, you are done
         ii. If the optimal Theta position did change by more than ±1%, repeat steps c through h
X. COLLECT DATA FROM THE ASYMMETRIC SCANS

- You should collect a coupled scan
  o This can be either a 2Theta-Omega scan or an Omega-2Theta scan
  o The Omega-2Theta scan is more conventional for HRXRD
  o The coupled scan will allow you to determine the d-spacing of the Bragg peaks of your film, which can be used to calculate composition and/or relaxation of the film
  o If you do not see film peaks in the coupled scan, then your film is strained or misoriented with respect to the substrate
    ▪ If your film is highly strained, you might need to collect a Reciprocal Space Map to measure the film peaks. This is explained in the Using XRD Wizard SOP
- You may want to collect a rocking curve for your substrate and film peaks
  o The Rocking Curve in a Triple-Axis Diffractometer measures the tilt distribution of the Bragg peak for a single d-spacing
  o The Rocking Curve can let you evaluate misorientation, dislocation content, mosaic spread, curvature, and relaxation
  o If you collect a rocking curve with a large receiving slit, this is effectively a Double-Axis Diffractometer rocking curve as long as your film is lattice matched to your substrate
    ▪ The range of d-spacing observed in the Rocking Curve is limited compared to a conventional Double-Axis Diffractometer
- If you want to collect a Double-Axis Diffractometer Rocking Curve from a non-lattice matched film, try collecting an Omega-2Theta scan using a very large Receiving Slit

1. Collect your Coupled Scan
   a. Scantype= 2Theta-Omega or Omega-2Theta
   b. Start and Stop should be selected to encompass your film and substrate peaks, with some additional range if interference fringes are also present
      i. If using a 2Theta-Omega scan, then calculate the Start and Stop from the Bragg angles for the diffraction peaks
      ii. If using a Omega-2Theta scan, be sure to include any tilt offset determined during the alignment procedure in your start and stop angles
        1. You can calculate the \( Tilt(Substrate) = \frac{1}{2} \times 2\Theta(Bragg) \) – \( \Theta(optimized) \)
        2. For each Bragg peak that you are collecting, making sure that the scan range includes them: the peak position will be \( \frac{1}{2} \times 2\Theta(Bragg) + Tilt(Substrate) \)
      iii. You might want to use a fast, coarse coupled scan to make sure you have good start and stop values and to evaluate what scanspeed you should use.
   c. The increment should be between 0.005° and 0.0001°, depending on the optics:
      i. Ge(022)x4 and Rec Slit: 0.005 to 0.001° increment
      ii. Ge(044)x4 and Rec Slit: 0.004 to 0.0005° increment
      iii. Ge(022)x4 and Analyzer: 0.002 to 0.0005° increment
      iv. Ge(044)x4 and Analyzer: 0.0005 to 0.0001° increment
   d. Scanspeed should be at least 0.2 sec/step. More complex or defective films will require a slower scan
      i. Remember, if you are not sure what the scanspeed should be, you can use the Repeat option to repeatedly collect the scan, adding the results until you have a good enough signal for analysis.
2. Save your Scan
   a. Data are not saved automatically when you collect them with XRD Commander
   b. Before saving the scan, you can type a description of the sample and/or experiment in the Sample ID line in the toolbar. This will be saved as the sample ID in the data header.
   c. Save the scan by going to File > Save or clicking on the Save icon in the toolbar
   d. Save your data in the “Diffrac Plus Ext. RAW file (*.raw)” format

3. Collect your Rocking Curves
   a. For each diffraction peak observed in your coupled scan that you want to collect a rocking curve for, you should:
      i. Double-click on the peak centroid (as if you were optimizing on that peak)
      ii. Record the 2Theta and Theta values that result when you double-click on the peak
      iii. These are the optimized 2Theta and Theta values that you want to use at the beginning of your rocking curve
   b. If there is a diffraction peak that you expected to see in the coupled scan, but did not, it might be misoriented with respect to the substrate. You can try collecting a scan from it by driving 2Theta to the expected value for that peak and collecting a rocking curve using a large receiving slit opening and a broad range for the rocking curve
   c. Drive 2Theta and Theta to the optimized positions for the first diffraction peak that you are going to collect a rocking curve for.
   d. Set Scantype= Rocking Curve
   e. The Start and Stop values for the Rocking Curve should be centered around the optimized Theta position
      i. You might want to run a fast, coarse, long range Rocking Curve to determine the best scan range and scanspeed for the data collection
   f. The increment should be between 0.005° and 0.0001° depending on what optics you are using and the rocking curve width of your film
      i. Ge(022)x4 and Rec Slit: 0.005 to 0.001° increment
      ii. Ge(044)x4 and Rec Slit: 0.004 to 0.0005° increment
      iii. Ge(022)x4 and Analyzer: 0.002 to 0.0005° increment
      iv. Ge(044)x4 and Analyzer: 0.0005 to 0.0001° increment
   g. Scanspeed should be at least 0.2 sec/step. Broader rocking curves usually require a longer scan since the peak will diffract weakly
      i. Remember, if you are not sure what the scanspeed should be, you can use the Repeat option to repeatedly collect the scan, adding the results until you have a good enough signal for analysis.
   h. Save your Scan
      i. Data are not saved automatically when you collect them with XRD Commander
      ii. Before saving the scan, type a description of the sample and/or experiment in the Sample ID line in the toolbar. This will be saved as the sample ID in the data header.
      iii. Save the scan by going to File > Save or clicking on the Save icon in the toolbar
      iv. Save your data in the “Diffrac Plus Ext. RAW file (*.raw)” format
      i. Repeat for each rocking curve you want to collect
APPENDIX A. HOW TO USE XRD COMMANDER

This section describes:

A. Screen Layout
B. Changing the Generator power
C. Driving Goniometer Motors
D. Collecting a Scan
E. Using the Chart Area
F. Changing the Detector Configuration

A. Screen Layout

There are four pages in XRD Commander, each accessed using the page tabs on the bottom of the screen. The pages are Adjust, Jobs, Geometry, and Details. To see a page, click on the appropriate tab.

The most heavily used page, the Adjust page, is illustrated below. The Adjust page is used to control goniometer motors, to execute single measurements, and to show the status of job (batch) measurements.

There are three common actions that you will execute using the Adjust page: changing the generator power, driving goniometer motors, and collecting a single scan measurement.
B. Changing the Generator Power
Controls for the X-Ray Generator are on the left-hand side of XRD Commander, either in the middle or along the bottom (depending on screen resolution). A close up of this area is shown below.

- There are two lines, one for the voltage, kV and one for the current, mA.
- The blue numbers in the right column indicate the current setting.
- The black numbers in the left column indicate the desired setting.

To change the generator power:
1. Set the black numbers to the desired setting
   1. the operating power is 40 kV and 40 mA
   2. the standby power is 40 kV and 10 mA
2. Click on the Set button
3. Wait until the actual values (in blue) change to the desired value

The generator should always be on. If the X-Ray line indicates that the generator is off or in stand-by mode, then something is wrong. Contact SEF staff for help.

C. Changing the Detector Setting
In the Details page, you can switch between the Pathfinder detector system and the LynxEye detector.

In the Details page, the upper-right corner of the XRD Commander window has two buttons to let you switch between Detector 1 and PSD

- Select Detector 1 if you are using the Pathfinder detector system
- Select PSD if you are using the LynxEye
  - Changing the detector that you use requires that you also physically change the detector that is mounted on the diffractometer. Contact SEF staff if you want to make this change.

If you are using the LynxEye detector, you can change the scanning mode between 0D and 1D. The 0D mode uses the detector as a point detector. You can then define the Opening (0D mode). If the LynxEye is in its 0° rotation, this value is the virtual receiving slit size. If the LynxEye is in its 90° rotation, then this is the width of the detector.

The 1D mode uses the detector as a linear position sensitive detector, which resolves many angles of 2theta simultaneously.
To select the 1D mode, click on the button for Scanning Mode that says 1D. To select the 0D mode, click on the button for Scanning Mode that says 0D.

In 0D mode, you must designate the Opening (0D Mode). To do so, type the opening that you want in the line Opening (0D Mode), then click the Set Detector button. The actual value of the opening, shown in blue in the Opening (0D Mode) line, should change.

**D. Driving Goniometer Motors**

You drive a motor to change the position of the goniometer—for example, driving the 2Theta motor changes the position of the detector.

To Drive a motor, use XRD Commander. In the left-hand side of the XRD Commander window, pictured right, the actual positions of the motor are shown in blue in the left column. The black numbers in the right column are the Requested (ie target) positions of the motors.

To change a motor position:
1. type the desired number for that motor into the Requested value field
2. make sure that there is a checkmark in the box to the right of the Requested field
   a. make sure that motors that you do not want to do not have checkmark next to them
3. click on the Drive button in the toolbar (circled in blue)

In the picture above, all motors have been told to move—because all motors have a checkmark next to them. However, the motors Theta, 2Theta, Phi, X, Y, and Z all have a requested position that is the same as the actual position—therefore, those motors will not actually move. The only thing that will change is that the Antiscatter Slit will move from 0.008 to 3, and Chi will move from 0 to 90.

*** On our system, the motor Antis. Slit actually controls the Receiving Slit***

If, at any point, the Actual drive positions (blue numbers in the left column) do not have a proper numerical value but instead read “---“ or a number followed by dashes, like “23.---“, then the instrument must be reinitialized. Contact SEF staff for help.
E. Collecting a Scan
A scan will collect the intensity of the X-ray beam observed by the detector as a motor(s) is moved. Scans collected using XRD Commander are not automatically saved—you must manually save them after they are collected.

Scans are configured and executed using the bottom of the XRD Commander window.

Note: XRD Commander uses the terms Theta and Omega interchangeably. Both refer to the incident angle of the X-ray beam. Technically, this axis should always be called Omega; it just happens that sometimes (i.e. in most powder diffraction scans) Omega is equal to Theta (i.e. equal Omega = ½*2Theta).

To configure a scan:
1. select the Scantype from the drop-down menu
   - this indicates what motor(s) moves as the X-ray intensity is plotted
   - Locked Coupled: moves 2theta and theta, forcing theta=½*2theta
   - Unlocked Coupled: moves 2theta and theta; preserves any initial offset between 2theta and theta so that theta=½ 2theta + offset
   - 2Theta/Omega: the same as unlocked coupled
   - Omega/2Theta: the same as 2Theta/Omega, except that start and stop angles are defined in terms of theta instead of 2theta
   - Rocking Curve: moves only the incident-angle omega (i.e. Theta)
   - Detector Scan: moves only the detector-angle 2theta
   - Chi: moves the tilt of the sample around the x-axis
   - Phi: moves rotation of sample around the sample normal
   - X, Y, or Z: moves the sample motor x, y, or z
   - use the button to the right of the drop-down menu to change between Continuous or Step Scan modes. The actual setting will be displayed on the button. For HRXRD, this should always be Continuous mode.

2. input the Start, Stop, and Increment values
3. set the Scanspeed
   - use the button to the right of the input field to switch between units: Sec/Step or Deg/Min. The actual setting will be displayed on the button. All instructions in this SOP refer to the Scanspeed in units Sec/Step
4. press the Start button
Some important notes about scans:

- When you select a **Scantype**, the values from the last scan of that type (for start, stop, increment, and Scanspeed) will automatically be input into those boxes. This may or may not be correct for your desired scan—be sure to check those values!
- Stop a scan in progress by clicking the **Stop** button
- Change the units for Scanspeed by clicking the button that says **Sec/Step** or **Deg/Min**.
  - Using **Sec/Step**, you can be sure that all data are collected with the same statistics even if you change the increment. As you change the increment, the resulting scan time will change.
  - Using **Deg/Min**, the scan time will be the same no matter what increment you select. However, the scan intensity will change depending on the scan increment and range.
- You can select the scan to be **Continuous** or **Step Scan** mode by clicking on the button next to Scantype drop-down menu.
  - **Continuous** mode counts the X-ray photons at all positions of the detector, and integrates them into discrete data points as indicated by the Increment value. The detector is continuously moving. This is preferred for faster scans.
  - **Step Scan** mode counts the X-ray photons while the motor is stationary at discrete positions as determined by the Increment value. Photons scattered between those data points are not measured. This is preferred for slower scans.
- The **Cont** button is used to extend the range of a scan without having to redo the entire scan
  - After your scan is finished, you can change the **Stop** value and then press the **Cont** button. The scan will resume from where it finished and run until the new **Stop** value.
  - If you click the **Cont** button without changing the **Stop** value, then the previous scan will be repeated. The results of the two scans will be added together, improving the average count statistics.
- While a scan is in progress, you can click on the repeat button in the upper toolbar. While this button is active, the scan you repeat on a continuous loop. The results of each scan will be added to the previous scan, improving the average count statistics.
  - This is a good way to collect data when you are not sure what an appropriate scan rate would be; you can select a fast scan rate and let the scan repeat until you have enough intensity for your analysis.
  - To stop the scan repetition, click on the repeat button. The current scan will complete, and then the data collection will finish.

**To save the results of this measurement**

1. Type in a sample or scan description in the line “**Sample ID**” below the toolbar
2. Click on **File > Save**
3. enter the filename and location for the data to be saved
4. make sure the format is set to **Diffrac Plus Ext. RAW File**
E. Using the Chart Area

The chart area shows the current scan data. If the cursor is moved into the graphical area the shape is changed to a crosshair.

**Plotting Options**

If you right-click in the chart area you will be able to select from several different options. The right-most buttons in the toolbar will also allow you to change plotting options such as changing the y-axis between linear and log scale, show grid, and show intensity in counts or cps (counts per second).

**Zoom**

To zoom in on a region in the chart area (for example, a peak in a rocking curve):

1. position the cursor to the upper left corner of the region that you want to zoom in on
2. left-click and drag the mouse to the lower right corner of the region that you want to zoom on.

You can unzoom three ways:

- In the chart area, left-click and drag the mouse up and to the left; release the mouse button and the chart area will unzoom
- Right-click in the chart area and select “Zoom Reset”
- Click on the zoom reset button in the toolbar

**Use Zoom**

This is an important button when aligning a sample. After a scan has finished, you can Zoom on a feature in the chart area. If you click **Use Zoom**, the zoom region will be used to redefine the start and stop values for a scan.

- This is used heavily when aligning a sample—you perform a fast scan over a large range using a coarse increment; then zoom in around the peak of interest; click **Use Zoom**; change the increment to a smaller value; and then do a more precise scan of the peak

**Optimize on a Peak**

After you have collected a scan, you can double-click on any point in the data display. This will send the corresponding motor position to the ‘requested drive position’.

- For example, after collecting a Rocking Curve you can double-click on the peak of the rocking curve. The requested omega position will be updated for that optimized position.
  - When optimizing on a peak, you want to optimize on the centroid or center of mass. Imagine a parabola defining the top of the peak, and click on the centroid of the parabola.
  - To get the best result when optimizing on a peak while using the analyzer crystal:
    - Double-click on a point to the left of the peak that you want to move to
    - Click on the Drive Motor button
    - Double-click on the point of the peak that you want to optimize on
    - Click on the Drive Motor button
  - Alternatively, you can position the cursor at the optimal position for the peak, read the x value from the cursor position information area, and then type that value into the requested drive position field.
APPENDIX B. WHAT TO DO IF THE Zi OPTION DOESN’T WORK BECAUSE OF AN ODD PEAK SHAPE

Because of artifacts in the beam shape due to the Gobel mirror, the X-ray beam may not be a smoothly defined peak (see picture below). In this case, the autopeak search executed when you press the Zi button will not work, because it will define the peak at the wrong position.

You have two alternative ways to redefine the zero position of the X-ray beam.

1. Use a smaller slit in the second monochromator slit position to restrict the height of the X-ray beam.
   a. The beam aberration is usually only seen with a 0.6mm or larger slit.
   b. A 0.2mm or smaller slit almost always gives you a good peak shape.
   c. You can use the smaller slit for your data collection or change it back to a larger slit after you have redefined zero.

2. Manually calculate the centroid of the beam
   a. Using the mouse cursor, determine the X-position of the left and right edge of the peak at an intensity approximately ½ the maximum
      i. In the picture above, the X-positions at 1100 counts were determined
   b. Calculate the point halfway between those two positions. This is “center(actual)”
   c. Click on the Zi button
   d. Read the Calculated peak position in the Zi Determination dialogue window.
   e. If Calculated peak position is equal to Center(actual), then click the Save and Send new Zi button.
   f. If the Calculated peak position is not equal to Center(actual), then
      i. Calculate the value offset = Calculated Peak Position – Center(actual)
      ii. Enter the value offset in the field “Enter theoretical position”
      iii. Click the Save and Send new Zi button
APPENDIX C. OPTIMIZING Z WHEN THE X-RAY BEAM IS WIDER THAN YOUR SAMPLE

Your Z-scan should look like the picture below: the intensity starts at $I_{max}$ and linearly decreases to zero as the sample is inserted into the X-ray beam.

When your sample is wider than the X-ray beam, it will completely block the X-ray beam at some point. In this scenario, it is easy to determine where your sample bisects the beam.

1. **Determine $I_{max}$** - the maximum intensity of the X-ray beam before the sample starts blocking it.
2. **Optimize** at the point on the chart where the X-ray intensity was $\frac{1}{2}$ the maximum intensity
   - $I_{max}$ in the graph above is 150,000 cps
   - The beam is completely blocked at ~0.6mm
   - The X-ray beam intensity is 75,000 cps (ie $\frac{1}{2} I_{max}$) at 0.398mm.

If the X-ray beam is wider than your sample, you will observe one of two effects:
1. The minimum intensity never reaches 0, but rather arrives at a different minimum value
2. The intensity exhibits two plateaus of low intensity as z increases: the first plateau intensity is not zero and the second plateau intensity is zero (shown below)
   - the first plateau is produced when the sample is intercepting the X-ray beam, but the X-ray beam is wider than the sample and so some radiation gets by the sample and into the detector
   - the second plateau is produced when the sample holder intercepts the X-ray beam. Most sample holders are wide enough to completely block the X-ray beam.

If the X-ray beam is wider than your sample, you have two options:
If the X-ray beam is wider than your sample, you have two options:

1. Reduce the width of the X-ray beam
   a. insert the 6mm width-limiting mask into the first monochromator slit position or insert the 0.6x2 slit in the second monochromator slit position to reduce the width of the X-ray beam
   b. Use a Z-scan to determine if the sample completely blocks the X-ray beam
   c. The sample may need to be centered vertically (y-axis) in order to completely block the X-ray beam.
      i. **Optimize** Z at a position in the middle of the first plateau in peak intensity (when only the sample and not the sample holder is intersecting the X-ray beam).
      ii. **Start** a Y scan
         1. Scantype= Y scan
         2. Start = -10
         3. Increment = 0.05
         4. Stop = 10
         5. Scanspeed = 0.1 sec/step
   d. **Optimize** on the minimum in the Y-scan
      i. this is the point where the sample is best centered to block as much of the X-ray beam as possible.
   e. If the sample now completely blocks the X-ray beam (the intensity at the minimum of the Y scan is zero), then use the procedure above to optimize Z
   f. If the beam is still slightly wider than the sample (the intensity at the minimum of the Y scan is not zero), then follow the procedure below in step 2

2. You can optimize Z even if the X-ray beam is wider than the sample
   a. Determine the intensity corresponding to ½ the difference between the maximum intensity and the intensity when the X-ray beam is blocked as completely as possible by the sample (ie the first plateau intensity).
   b. **Optimize** Z on that point on the graph.
   c. The fact that the X-ray beam is wider than your sample should not interfere with your data collection—the excess X-ray beam may contribute a small amount to the background noise
### APPENDIX D. BRAGG AND TILT ANGLES OF COMMON SUBSTRATES

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**APPENDIX E. USING LEPTOS TO DETERMINE THE LATTICE PARAMETERS OF YOUR SUBSTRATE**

The program Leptos contains a database of common materials for wafers and epitaxial thin films. To access this database to determine the crystal system and lattice parameter of your material:

1) Start the Leptos program
2) Go to File > Open MDB
   a) The **Materials Database** interface will open

3) From the list on the left-hand side, select your material

4) If your sample is a line compound (Si, GaAs, etc)
   a) The Crystal System will be shown in the upper header (circled in red)
   b) The lattice parameters will be shown in the Unit Cell information (circled in blue)

5) If your sample is a graded solution (Si$_{1-x}$Ge$_x$, Ga$_{1-x}$In$_x$As, In$_x$Ga$_y$Al$_{1-x-y}$As, etc) you can determine the precise lattice parameter for your expected composition by using the Concentration Sliders
   a) The concentration sliders are located in the lower-right hand corner (circled in green)
   b) Move the slider for x and/or y to change the composition of material
   c) The Unit Cell information (circled in blue) will change the lattice parameter based on the composition that you specify

6) Write the crystal system and lattice parameters found in Leptos in XRD Wizard
7) Click **Cancel** in the Leptos **Materials Database** window to close the database without saving any changes.
   a) Click **Yes** in the dialogue window that opens
8) Return to XRD Wizard or whatever else you were doing.
Appendix F. Using HighScore Plus to Look Up the Unit Cell and Diffraction Peak List

I. Opening a PDF Reference Pattern
The computer that runs the Bruker HRXRD has as copy of the Crystallographic Open Database (COD) installed as its reference database. Computers in the data analysis lab have the more complete Powder Diffraction File (PDF), which is a database published by the International Center for Diffraction Data (ICDD).

To Retrieve PDF Patterns using the PDF Reference Number
1) Go to the menu Reference Patterns > Retrieve Pattern By > Reference Code
2) In the dialog box, type in the Reference Code(s) for the PDF card(s) that you want to open
3) Click Load
4) The cards will be loaded into the Pattern List in the Lists Pane

In older literature, you may see reference to JCPDS cards. The JCPDS database was the predecessor to the PDF. You can use the original JCPDS reference number to retrieve that entry in the PDF database.

To search the PDF Database for References
If you do not know the reference code for the PDF card for the material that you are interested in, you can search the PDF database for the relevant entries.

1) Go to the menu Reference Patterns > Retrieve Pattern By > Restrictions
2) In the Restrictions dialogue window that opens, you define parameters to restrict which patterns will be retrieved from the database. If you defined no parameters at all, then all patterns in the database would be retrieved.
   a) Each tab in the Restrictions window allows you to control a different subset of search parameters. In each of the tabs, you can set the parameters for searching the PDF database for reference patterns.
   b) The options available in the tabs are described on the next page (pg 22)
   c) Each tab that is being used to constrain the search will be highlighted with a red flag
   d) The information bar at the bottom of the window tells you how many patterns will be retrieved with the current set of restrictions.
3) Once you have set up your search parameters, using as many or as few Restrictions as you like, then you click Load to perform the search and find the reference patterns.
   a) The reference patterns will be added to the Pattern List pane.
   b) If you have other reference patterns already loaded, the Combine Patterns dialogue will ask you to specify how you want to combine the new patterns with the previous. Make your choice and click OK.
   c) The Restrictions window will remain open, allowing you to retrieve more references
      i) If you are done finding reference patterns, then click Close.
The tabs that you can use to constrain your search of the reference database are:

**Chemistry**

This is the most common restriction that you will use.

- You have three fields to specify what compositions you want to find in the database:
  - **“All of:”** all of these elements together must be present in each reference pattern
  - **“At least one of:”** one or more of these elements must be present in each reference pattern
  - **“None of:”** none of these elements must be present in each reference pattern
  
  - If you click on the button **Add Rest to None Of**, then every element not listed in **“All of:”** or **“At least one of:”** fields is added to the **“None of:”** field

- The **Min** and **Max Number of Elements** controls how many unrestricted elements are allowed in the retrieved reference patterns

**Example:**

- In this example, 2146 patterns will be retrieved. These patterns would include FePO₄, LiPO₄, and LiFePO₄; but would also include any material that contains P and O, Li or Fe, and any other element—for example, Fe₂As₃O₁₂ or RbFeP₂O₇.

- In this example, only 181 patterns will be retrieved. All elements except Li, Fe, P, and O were added to the “None of:” field by clicking the Add Rest to None Of button. Patterns loaded would include FePO₄, LiPO₄, and LiFePO₄.

**Quality**

This restriction is only useful if you are accessing the PDF database. If you are using the COD database, do not use this entry. is another very useful restriction to use.

- It is highly recommended that you check the entry **“Skip marked as deleted by ICDD”**

- Patterns with the Star quality mark are the highest quality. First try searching for only Star quality cards. If that does not retrieve an entry, then try including Indexed quality cards too.

- You can also use this tab to exclude data that was not collected at standard pressure and temperature (**“Skip non-ambient temperature”** and **“Skip non-ambient pressure”**)

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**Subfiles**

This tab is used to restrict the search to certain subfiles of the PDF. Data in the PDF comes from several different sources. The data is organized into *subsets* depending on the source and *subfiles* depending on characteristics of the material. In HSP, you can search for entries from a single subset/subfile, a combination of subsets and subfiles, or from all subsets.

**Crystallography**

This restriction tab can be used to specify parameters of the crystal structure for all retrieved entries, such as the Crystal System (cubic, hexagonal, …), Density (theoretical density based on crystal structure), or lattice parameters. This restriction is not used that often.

**Strings**

This restriction is used to search for text in the PDF reference, such as a specific Mineral Name (hematite, etc) or Compound Name (polyethylene, etc). This restriction is not used that often.

**MANIPULATING THE REFERENCE CARDS THAT YOU HAVE LOADED**

Once you have searched the database and added reference cards, you can view them in the Main Graphics pane and manipulate them in the Lists Pane and Object Inspector.

In the Lists Pane, go to the Pattern List tab. You can:

- Make the pattern Visible or Hidden by checking/unchecking the Visible column
- Change the pattern color by using the drop-down menu in the Display Color column
- Open the reference card by double-clicking on an entry
  - The entry will contain information such as formula, unit cell information, the reference that the entry came from, and a list of peak positions and intensities.
  - You can use the peak list in the reference card to decide where you want to scan. A card might list several peaks at low angles (below 20deg 2theta) that are very informative. After 55.74deg 2theta, all of the diffraction peaks are weak. Therefore, a good scan range would be from 5deg to 56deg 2theta.

In the Object Inspector, you can manipulate the display of the Reference Pattern (color, scaling)

- Click on an entry in the Pattern List
- go to the Object Inspector
  - Settings in the Display section of the Object Inspector will let you change the visual display of the reference pattern in the Main Graphics
  - Other sections display information about the reference card