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.

This SOP contains abridged instructions. It 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.

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 C.

On the Data Collection PC, you can find the document “Expanded SOP for the Bruker D8 HRXRD using XRD Commander and XRD Wizard.docx”. That SOP gives more explicit step-by-step instructions. It also gives you information about how to use XRD Wizard for collecting automatically saved scans and for collecting maps such as Reciprocal Space Maps or Wafer Maps.
| I. | Summary of the Alignment Procedure | pg 3 |
| II. | Preparing to Collect Data | pg 4 |
| III. | Align Z by Bisecting the Beam | pg 5-7 |
| IV. | Align the Symmetric Substrate Peak | pg 7-10 |
| V. | Collect Data from the Symmetric Peaks | pg 11-12 |
| VI. | Align the Asymmetric Substrate Peak | pg 13-16 |
| VII. | Collect Data from the Asymmetric Peak | pg 16-18 |

**Appendix A.** Bragg and Tilt Angles of Common Substrates | Pg 19

**Appendix B.** Using Leptos to look up the unit cell for materials | Pg 20

**Appendix C.** Using HighScore Plus to Look Up the Unit Cell and Diffraction Peak List | Pg 21-23
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
II. **PREPARING TO COLLECT DATA**

Appendix A (pg 22) gives an overview of how to use XRD Commander, which is the program that we use to manually control the diffractometer. The instructions below give some descriptions of how to perform a specific task in XRD Commander. Consult Appendix A if you need additional details.

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. When you float the mouse over the button, the name of the button appears
      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.

6. Mount the sample- see the Sample Stage SOP for instructions
• 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).
• The Receiving Slit is labeled “Antis. Slit”. In the instructions below, I will refer to it as the Receiving Slit (or Rec. Slit) when describing what it does and as the Antis. Slit when directing you to make a specific change in the XRD Commander program.
• For all scans, the scan mode should be Continuous (not 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. Drive the Antis. Slit to 0.2

2. 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

3. 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. Phi=0
      iv. Chi=0
      v. X=0
      vi. Y=0
      vii. 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. 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

- 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.

d. Repeat the Detector Scan and make sure that the peak is centered around 0° 2Theta

4. 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.

5. 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

6. 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= \textit{optimized Theta position} – 0.5
   iii. Increment= 0.005
   iv. Stop= \textit{optimized Theta position} + 0.5
   v. Scanspeed= 0.1 sec/step

7. 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

8. You might want to record the optimized value of Theta as \textit{Tilt(sample)}
   a. \textit{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
\textit{tilt(sample)}, this would indicate a miscut in the substrate
   c. Assuming that your sample is not miscut, we can use the \textit{tilt(sample)} to save time when
aligning on the Bragg peak of the substrate.

\textbf{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.
  - 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.
  - When you see multiple peaks in the rocking curve, the substrate peak will almost always
    be the most intense and sharpest
  - \textit{For very thick films, the substrate peak might not be the most intense}
  - 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 \( \frac{1}{2} \times (2\text{Theta}) + \text{tilt(sample)} \)
      i. The \( \text{tilt(sample)} \) was determined when aligning the Z position of the sample, above.
   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 use the “HRXRD Angle Calculation.xlsx” spreadsheet, found on the desktop of the data collection computer
      iii. You can use XRD Wizard, Appendix F (pg 33)

3. Optimize the substrate tilt in the diffraction plane (Omega) using a Rocking Curve
   a. **Drive** the Antis. Slit to a large value, such as 1 or 3 mm
   b. **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
   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 0.002deg
      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 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 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
    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.
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
           0.0001deg
         ▪ Using a receiving slit, the final increments might be between 0.004 and 0.0005deg
   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
    - The Omega-2Theta scan is more conventional for HRXRD
    - The 2Theta-Omega scan is easier to extract d-spacing information from
  - 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 $Tilt(Substrate) = \frac{1}{2} \times 2\Theta(optimized) - \Theta(optimized)$
         2. For each Bragg peak that you are collecting, making sure that the scan range includes them: the peak position will be $\frac{1}{2} \times 2\Theta(Bragg) + Tilt(Substrate)$
   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
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 $2\Theta(Bragg)$ position of the Bragg peak and the $\Omega(Omega)$ 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 Appendix F (pg 33), or by using the “HRXRD Angle Calculation.xlsx” spreadsheet.
   c. Based on the symmetric scan, determine the $2\Theta(offset)$ and $\Omega(offset)$
      i. $2\Theta(offset)=2\Theta(optimized)-2\Theta(optimized)$
      ii. $\Omega(offset)=\frac{1}{2} \times 2\Theta(optimized) - \Theta(optimized)$
   d. **Drive** $2\Theta = 2\Theta(Bragg) + 2\Theta(offset)$
   e. **Drive** $\Theta = \frac{1}{2} \times 2\Theta(Bragg) + \Omega(offset) \pm Tilt(Omega)$

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

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= *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 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
  - This can be either a 2Theta-Omega scan or an Omega-2Theta scan
  - The Omega-2Theta scan is more conventional for HRXRD
  - 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
  - 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 “Expanded SOP for the Bruker D8 HRXRD using XRD Commander and XRD Wizard.docx”
- 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, and relaxation

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 $Tilt(Substrate) = \frac{1}{2} \times 2Theta(optimized) - \Theta(optimized)$
         2. For each Bragg peak that you are collecting, making sure that the scan range includes them: the peak position will be $\frac{1}{2} \times 2Theta(Bragg) + Tilt(Substrate)$
      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
   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. BRAGG AND TILT ANGLES OF COMMON SUBSTRATES

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<th>Substrate</th>
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**APPENDIX B. 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\textsubscript{1-x}Ge\textsubscript{x}, Ga\textsubscript{1-x}In\textsubscript{x}As, In\textsubscript{x}Ga\textsubscript{y}Al\textsubscript{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 C. USING HIGHSCORE PLUS TO LOOK UP THE UNIT CELL AND DIFFRACTION PEAK LIST

I. Opening a PDF Reference Pattern
The computer the 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”)
**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