Bruker D8 HRXRD

Collecting Reciprocal Space Maps using the LynxEye Position Sensitive Detector

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The SOP describes the steps necessary to align a sample and collect a reciprocal space map (RSM) using the LynxEye Position Sensitive Detector.

Because the LynxEye is a position sensitive detector (PSD) it can collect a range of 2theta data simultaneously. This allows for fast reciprocal space mapping. However, data collected with the LynxEye are noisier and have lower resolution than data collected with the Pathfinder point detector system. This makes the LynxEye most appropriate for analysis of epitaxial thin films with high defect density, such as oxide thin films.

This SOP assumes that you are familiar with:
- Background theory of the analysis of epitaxial thin films, such as covered in the lecture "Introduction to HRXRD of Epitaxial Thin Films"
- Basic operation of the Bruker D8 HRXRD instrument, as covered in the SOP "Basics of Configuring the Bruker D8 HRXRD and using XRD Commander"
- Basic collection of HRXRD data as covered in the SOP "Abridged SOP for Manually Aligning and a Sample and Collecting Data using XRD Commander"
- Running batch jobs as covered in the SOP "Using XRD Wizard to Collect Data"

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.

I. CONFIGURING THE INSTRUMENT

1. Configure the diffractometer as you normally would. The monochromator and slits will be set up exactly the same as done for normal HRXRD data collection, as described in the SOP "Configuring the Bruker D8 HRXRD and using XRD Commander"

2. Start the programs XRD Commander and XRD Wizard

3. Select the program XRD Commander

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] **Set** button
   e. Wait until the actual values (in blue) change to the desired value
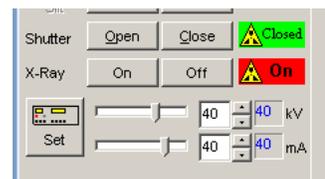
5. Mount the sample

6. Set the detector path to use the LynxEye Detector
   a. Select the Secondary Optic using the drop-down menu
      i. Select **Default Optic**
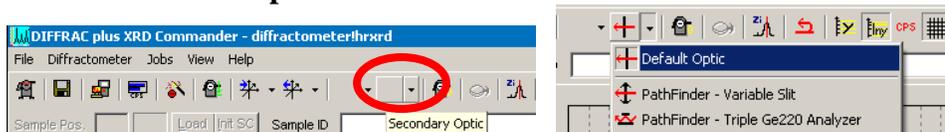
      ii. The drop down menu for the secondary optic is the second blank drop-down box in the Toolbar for XRD Commander
      iii. When you float the mouse over the button, the name of the button appears
      iv. After you select the Secondary Optic from the drop-down menu, the button will be filled with the icon for that optic.

7. Configure the Detector Settings
   a. Select the <u>Details</u> tab
   b. In the upper right-hand corner of XRD Commander, make sure that **PSD** is selected, not **Detector 1**.
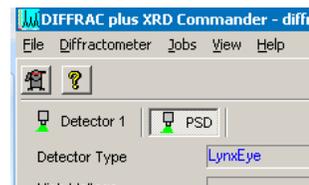
   c. The LynxEye detector has several settings that are controlled from this page and that will be changed in the course of alignment and data collection. These are described on the next page.

The LynxEye PSD can operate in two modes, **0D** and **1D**

- **0D Mod**e
  - o The detector operates as a point detector, collecting only one data point at a time
  - o The value **Opening (0D Mode)** is used to change the "virtual receiving slit". This controls what range of 2theta is observed simultaneously by the detector.
  - o This mode is used for sample alignment
- **1D Mode**
  - o The detector operates as a PSD, collecting a large range of 2theta simultaneously
  - o This mode is used for collecting the reciprocal space map (RSM)

- **To change the Mode of the Detector**
  - o Click on the Scanning Mode button **0D** or **1D** (circled in red to the right)
  - o If the Scanning Mode is 0D, then enter a value for **Opening (0D Mode)**
  - o Click on the Set Detector button (circled in blue to the right)

- During Alignment
  - o Unlike regular data collection with the Pathfinder point detector, you will not use the motor Antis. Slit to change the detector receiving slit.
  - o Instead, you will change the **Opening (0D Mode)** of the LynxEye PSD. This opening acts as a virtual receiving slit.
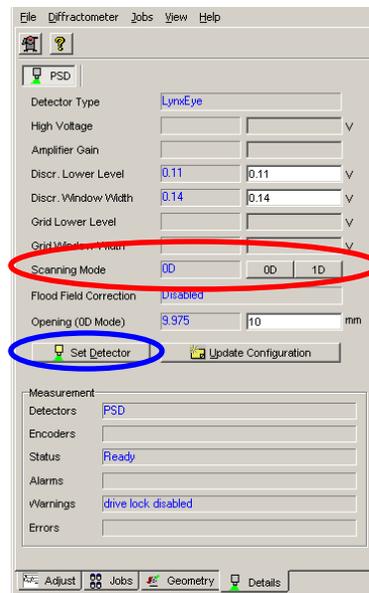
II. ALIGN Z BY BISECTING THE BEAM
You will determine the optimal Z value by bisecting the X-ray beam, as is typically done for any HRXRD measurement.

1. Set the Detector Opening to 0.1mm
   a. Select the Details tab
   b. Set the Scanning Mode to **0D**
   c. Set the **Opening (0D Mode)** to 0.1
   d. Click on the **Set Detector** button

2. **Set** the Absorber
   a. Select the Adjust tab
   b. Using the **Absorber** drop-down menu, select a value
   c. 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 8.5

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. Redefined 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 dialogue box prompts you for a password, leave it blank and click **OK**
   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

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

If the detector scans, Z scans, or rocking curves give you peaks that are unusual or difficult to analyze, then see the appendices in the SOP "Basics of Configuring the Bruker D8 HRXRD and using XRD Commander" for guidance and the best way to proceed with the alignment.

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= ***optimized Theta position – 0.5***
        iii. Increment= 0.005
        iv.   Stop= ***optimized Theta position + 0.5***
        v.   Scanspeed= 0.1 sec/step

III.   BEGIN WRITING THE XRD WIZARD JOB AND DETERMINE THE CORRECT POSITION FOR THE BRAGG DIFFRACTION PEAK

These instructions assume that you are aligning on an asymmetric peak, since asymmetric peaks generally provide the most useful information in a reciprocal space map. These instructions walk you through the beginning part of writing a batch job with XRD Wizard, up to the point where need to manually align on the Bragg peak.

1. Activate the XRD Wizard program.

2. When you started the XRD Wizard program, it should automatically generate an HRXRD job.
    a. If the job name in the top most margin of XRD Wizard does not say "[HRXRD#]", where # is an actual number, then you should create an HRXRD job
        i.   Select *File > New*
   ii.   In the dialogue window, select HRXRD and click **OK**

*XRD Wizard program walks you through several pages of that collect information for writing the data collection batch job. When you have input the information for a page, click **OK** to save that setting and proceed to the next page. The flow chart on the left-hand side of XRD Wizard will show you which step you are on and allow you to navigate.*

3. In the first page, *Scan Documentation*, you can enter information about your Experiment.
    a. The most useful thing is to fill out the **Sample** information. This will be the Sample ID in the header of the saved file.
    b. Click **OK** when you are finished entering information.

4. The second page, *Diffractometer Settings*, is already filled out. Click **OK**

5. The third page, *Measurement Geometry*, is used to collect information about the substrate.
   a. You must complete the information for the Substrate. You may also include information about one layer in your film—this is optional, though it can be useful.

   b. For the Substrate
      i. Enter the **Name**

      ii. The **Surface (mno)** designates the (hkl) of the planes parallel (or closest to parallel) the surface of your sample.
         1. For any axes that are equivalent, it is convention to put the largest value in the last place for the equivalent axes.
            a. *For example, in a cubic material a=b=c. The largest value would go to the l positon, so you would enter (001) instead of (100).*
            b. *For a hexagonal substrate, a=b≠c. Therefore, the l position would be a fixed value; for h and k, the largest value would go to the k value, so you would enter (011) instead of (101).*

      iii. The **Azimuth (pqr)** designates the lateral direction (within the plane of the sample surface) that you will use as a reference.
         1. This value should be normal to the Surface (mno).
            a. *The azimuth (pqr) is normal to the surface (mno) if $p*m + q*n + r*o = 0$.*
            b. *Both [100] and [110] would be valid Azimuth(pqr) for a Si wafer with surface(mno) of (001)*
            c. *The azimuth that you enter should be close to the projection of the diffraction peak that you want to measure. For example, if you intend to collect the (224) asymmetric peak from a (001) oriented crystal, then enter an azimuth of (110) and not (100)*

      iv. Click on **Cryst. System…** to specify the unit cell of the substrate.
         1. First, select the **Crystal System** for the substrate.
         2. Then, enter the **Lattice Parameters** (in nm, not Å)
            a. If you do not know the lattice parameter for you substrate, you can look it up in HighScore Plus, Leptos, or another reference.
         3. Click **OK**.

c. If you like, repeat the previous steps (i-iv above) to enter information for your layer.
   i. *If your sample contains multiple layers, it is usually the most useful to enter the values for the layer with the lattice parameter the most different from the substrate. For example, if your sample is a Si substrate, coated with several layers if $Ge_{1-x}Si_x$ and then capped with a layer of Ge, the Ge layer will have the lattice parameter most different from the substrate. You should enter the information for the Ge layer.*
d. Click **OK** when you have filled out all of the information for the substrate and layer.

6. In the next page, ***Sample Alignment***, you specify the Bragg peak you will collect data from.
   a. This page will calculate the appropriate Theta and 2Theta angles for the aligning the peak
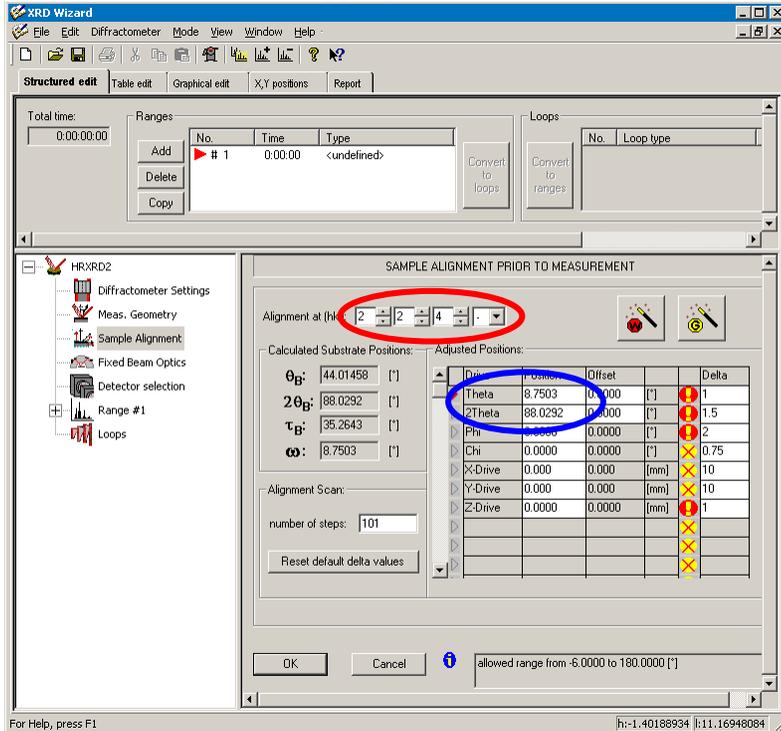
   b. Specify, using **Alignment at (hkl)** (circled in red), the Bragg diffraction peak that you want to study.
      i. In this example, hkl is set to (004).

   c. The fourth box in the **Alignment at (hkl) line** lets you specify s, +, or -.
      i. **S** indicates a symmetric scan,
      ii. **+** specifies a grazing exit asymmetric scan,
      iii. **–** specifies a grazing incident asymmetric scan
      iv. To decide if you should us a grazing incidence (-) or grazing exit (+) asymmetric scan
         1. *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*
         2. *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*

d. The entries Theta and 2Theta (circled in blue) will be calculated to indicate the starting positions for aligning the sample on that Bragg peak
  i. If there is a significant tilt of your sample, determined during Z alignment or alignment of a symmetric peak, then you can compensate for that tilt
    1. In the column Offset for Theta, enter *–Tilt(Sample)*
    2. Be sure that the offset has the opposite sign from the *Tilt(Sample)*

e. The field Calculated Substrate Positions shows you several values
  i. θ_B is equal to ½ $2\theta_B$ and is the value that omega would be for a symmetric scan
  ii. $2\theta_B$ is the Bragg angle for the diffraction peak
  iii. τ_B is how much the sample must be tilted to observe an asymmetric Bragg peak
  iv. ω is the omega value necessary to observe a peak, and is ω= $\theta_B \pm \tau_B$
  v. The Theta value calculated for the drive position is Theta= ω - Offset

IV. ALIGN ON THE BRAGG DIFFRACTION PEAK

These instructions assume that you are aligning on an asymmetric peak, since asymmetric peaks generally provide the most useful information in a reciprocal space map. If you are aligning on a symmetric peak, then you can just skip the steps with instructions to optimize phi

1. **Drive** 2Theta and Theta to the values for the asymmetric Bragg peak of the substrate.
   a. These values were calculated in XRD Wizard, as described above, or you may know them from another source.

2. Use a Phi scan to find the rotation of the sample that will let you see the Bragg peak
   a. Set the Detector Opening to 5mm
     i. Select the Details tab
     ii. The Scanning Mode should already be **0D**
     iii. Set the **Opening (0D Mode)** to 5
     iv. Click on the **Set Detector** button
       1. *An opening of 5mm will cover ~1deg 2theta simultaneously.*
       2. *You can make this bigger, up to 14mm (2.8deg 2theta) if you are less confident about the true Bragg peak position*
       3. *You can make this smaller if you know the Bragg position and you are worried about interfering signal from film layers or other sources*

   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. Tweak the scan range if you know approximately where the sample should be rotated
       1. *For example, the flat on a Si(001) wafer usually indicates the [110], so you can find the (224) or (115) peaks by making the flat square with the X-ray tube. The (044), which projects along the [010], 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

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 with each maxima or minima
         2. Collect rocking curves with chi set to each of those values
         3. The Chi position that produces the most intense rocking curve is the correct chi position

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. Set the Detector Opening to 0.2mm
      i. Select the <u>Details</u> tab
      ii. The Scanning Mode should already be **0D**
      iii. Set the **Opening (0D Mode)** to 0.2
      iv. Click on the **Set Detector** button

   b. **Start** a coarse detector scan
      i. Scantype= Detector Scan
      ii. Start*= <u>**current 2Theta position – 0.5**</u>*
      iii. Increment= 0.005
      iv. Stop= *<u>**current 2Theta position + 0.5**</u>*
      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. 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
    d. Optimize the Theta position using a Rocking Curve
    e. Optimize Phi using a Phi Scan
    f. Optimize the Theta position using a Rocking Curve
    g. Optimize the 2Theta position of the Bragg peak using a 2Theta-Omega scan
    h. 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

V. FINISH WRITING THE XRD WIZARD BATCH JOB
This step assumes that you have already aligned your sample using XRD Data Collector

1) Copy the aligned positions from XRD Commander To XRD Wizard
   a) In XRD Commander, click on the **Transmit Drive Positions** button (circled in red)
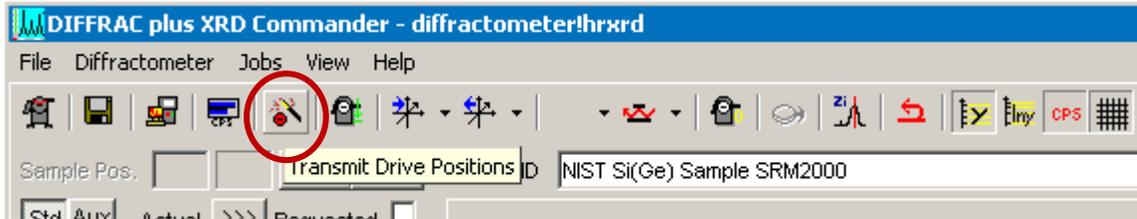
   b) In XRD Wizard, you should be on the *Sample Alignment* page
   c) Click on the Get Positions button (circled in blue)
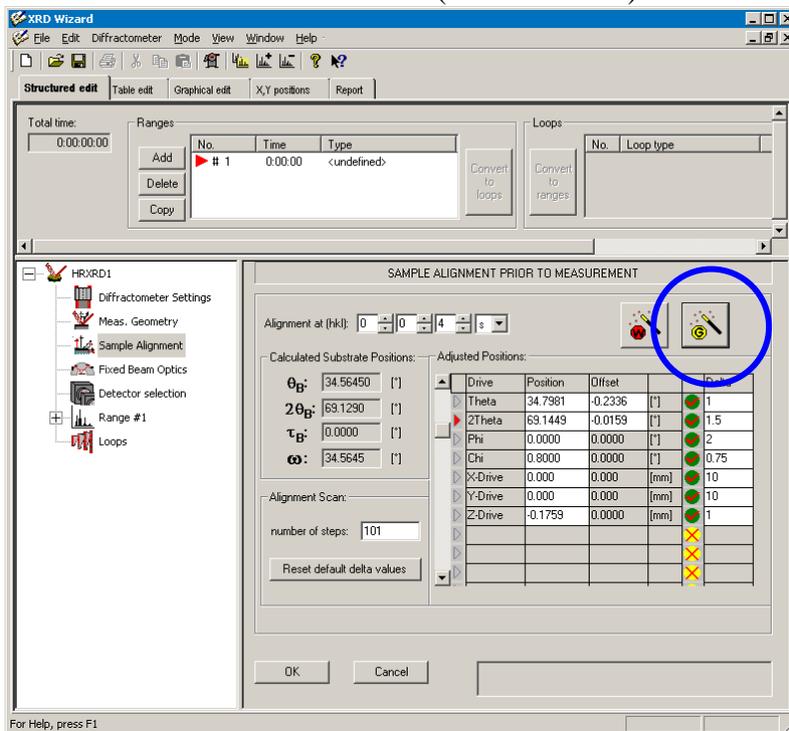
   d) The aligned values for all drives are loaded in the Position Column
   e) The offsets are automatically calculated for Theta and 2Theta
   f) Each drive receives a check mark (red check mark in green circle) indicating that this is the aligned value
   g) Click **OK**
   h) If you get a message "There is at least one range defined which is referred to the alignment reflex. Do you want to change the reflex in the range(s) too?", click **YES**

2) In the next page, *Fixed Beam Optics*, there are no parameters to set
   a) The parameters "Detector Slit" and "Antiscattering Slit" do nothing
   b) Click **OK**

3) In the next page, *Detector Selection*, specify the detector that you are using
   a) Use the drop-down menu to set the Detector to **"PSD: LynxEye"**
   b) In the field *PSD electronic window*
      i)   Select the radio button **enter value**
      ii) Enter in the scan range that you would like to use for your detector scan.
           (1) For example, if you want to scan 10deg 2theta, you would enter 10
      iii) If your substrate peak and film peaks are close together (within 2.5 degrees), then select the radio button **use default**.
           (1) This will only collect data over a 2.7 degree range, but the detector will not have to move during data collection so it will be much faster!
   c) Click **OK**
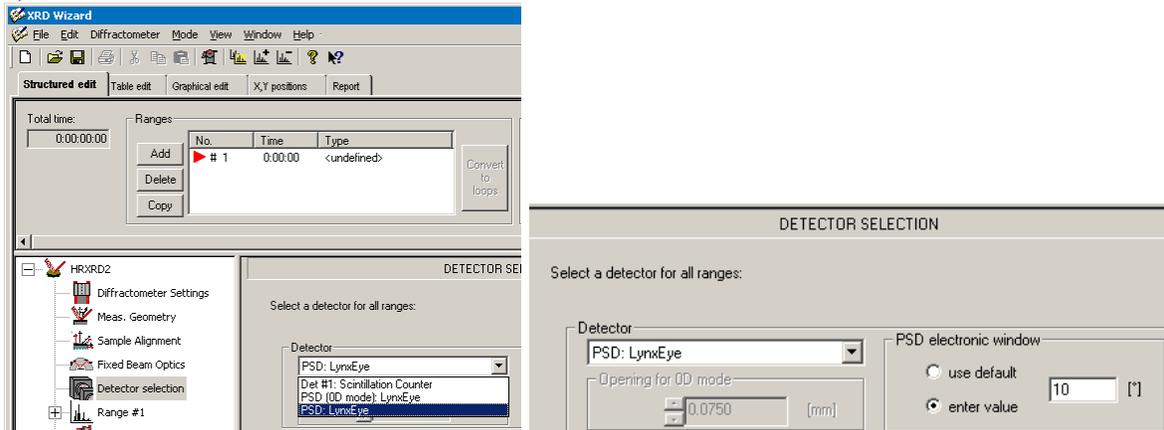
4) The next page is **HRXRD Scan Settings**
   a) In **Scan Type**, use the drop-down menu to select **PSD-Fixed Scan**
   b) The **Diffraction setup** should always be set to + (circled in red)
      i)   *Note: this designation does not correspond to grazing exit or grazing incident.*
   c) The **Diffracted beam path** should be set to LynxEye button, labeled below
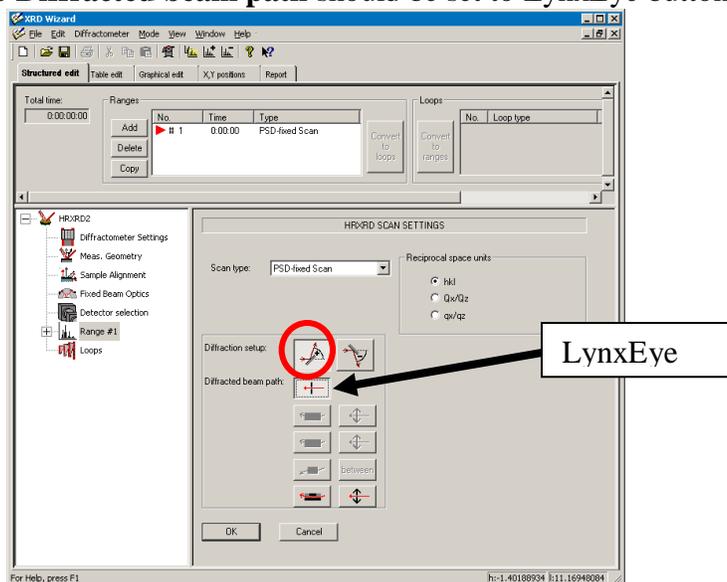
   d) Click **OK**

5) The next page contains the *Scan Parameters*
   a) The Start and Stop parameters are specified using Relative positions
      i) The Reference Position are shown in a table in the lower-right corner
      ii) The **Rel. start** is the range before the Reference Position that the scan will begin
      iii) The **Rel. stop** is the range after the Reference Position that the scan will stop
      iv) The total distance between Rel. start and Rel. stop should equal the value that you entered for the *PSD electronic window* in step 3 on the previous page**.**
      v) *For example, for the image below the **PSD electronic window** was set to 10 deg. The Reference Position for 2Theta is 88.0292deg. The Rel. start is -5deg, so the scan will start at 83.0292deg. The Rel. stop is 5deg, so the scan will stop at 93.0292deg. The total scan range is 10 deg.*
      vi) *If you use a total range of 2.7 deg, the detector will not have to move during data collection. This will result in much faster data collection.*

   b) The **Resolution** is the step size, which is a function of the LynxEye detector geometry. Enter **0.00767 deg**. *You must type this value in; do not use the up/down arrows.*

   c) The **Time** the amount of time each data point is observed.
      i) The minimum allowable value is 9.5 sec. There is no maximum value.
      ii) *If you are using a range of 2.7 deg, so that the detector does not have to move, then the minimum allowable time is 1 sec.*

   d) The **Total Scan Time** calculated by the program is wrong. To calculate the scan time, use the equation: $total\ scan\ time = \frac{scan\ range + 2.7}{Resolution} * \frac{Time}{192}$
      i) *If you are using a range of 2.7 deg, so that the detector does not have to move, then the calculated **Total Scan Time** will be correct. It will be equal to the value that you entered for **Time** in the scan rate.*

   e) Click **OK** when you are done entering the scan parameters.
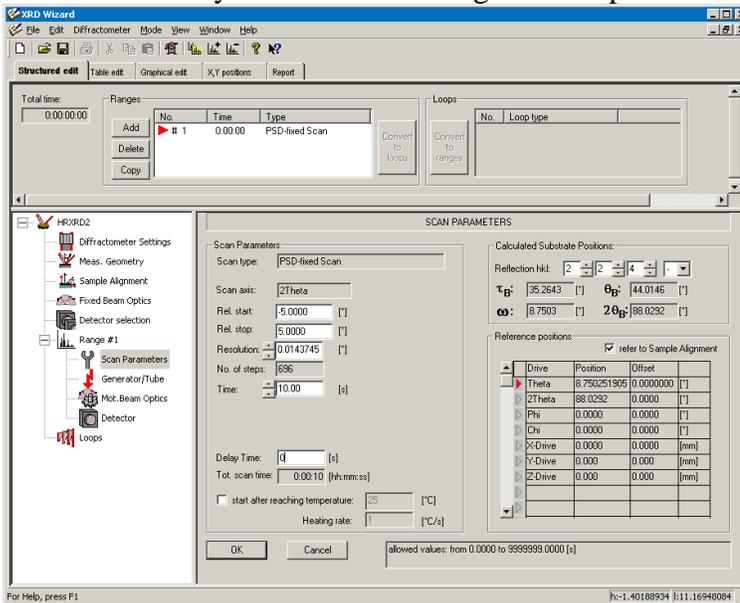
6) The next page is the *Generator Settings*
   a) Make sure Voltage is set to 40 kV and Current is set to 40 mA
   b) Click OK

7) The next page is *Motorized Beam Optics*
   a) The value Antiscattering Slit is for the Pathfinder, which you are not using. This value doesn't matter
   b) The **Rotary Absorber** should be set to **0**
   c) Click **OK**
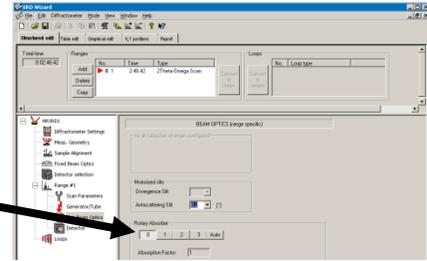
8) In the next page, ***Detector Type***, you should not change any settings. Click **OK**.

9) The next page is the ***Loops*** page
   a) This page is used to design the 2-axis map you will be collecting
   b) The detector scan that you wrote is displayed as the first column (in red)
   c) The loop is used to tell the instrument to change a parameter, and then repeat the scan

   d) Before you create the loop, you might want to look at the reciprocal space map
      i) Click **OK** on the loops page.
      ii) Click on the tab Graphical Edit (circled in red below)
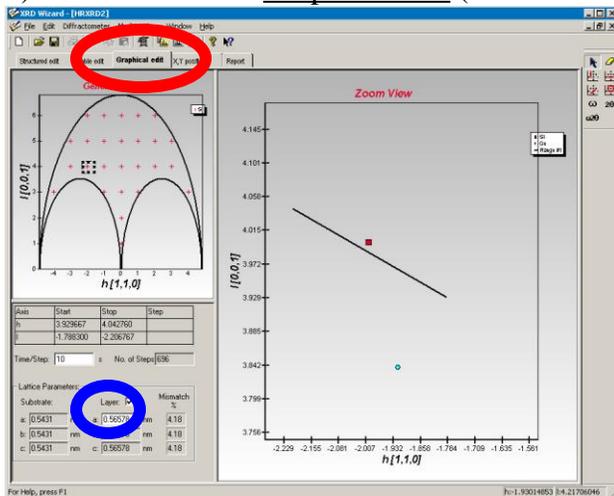

      iii) The Graphical Edit shows a picture of reciprocal space and draws the area of reciprocal space that will be covered by the detector scan that you created.
      iv) The substrate reciprocal space point is shown as red square. The film reciprocal space point, for a relaxed layer, is shown as a blue circle if you check the Layer option (circled in blue above)
         (1) Float the mouse over a reciprocal space point to see the Miller indices
         (2) The crystal system and lattice parameters are taken from the ***Measurement Geometry*** page that you filled out earlier
      v) **Zoom** in around the diffraction peak that you are collecting by left-clicking and dragging down and to the right.
         (1) The line shows the area that will be observed by the detector scan that you created
      vi) This map can give you an idea of how much space you need to cover with your RSM

e) Return to the <u>Structured Edit</u> tab

f) Create a loop
   i) In the Miscellaneous menu on the right side (circled in green), select **Omega rel.**
   ii) In the field **Add loop,** click the button **at end**
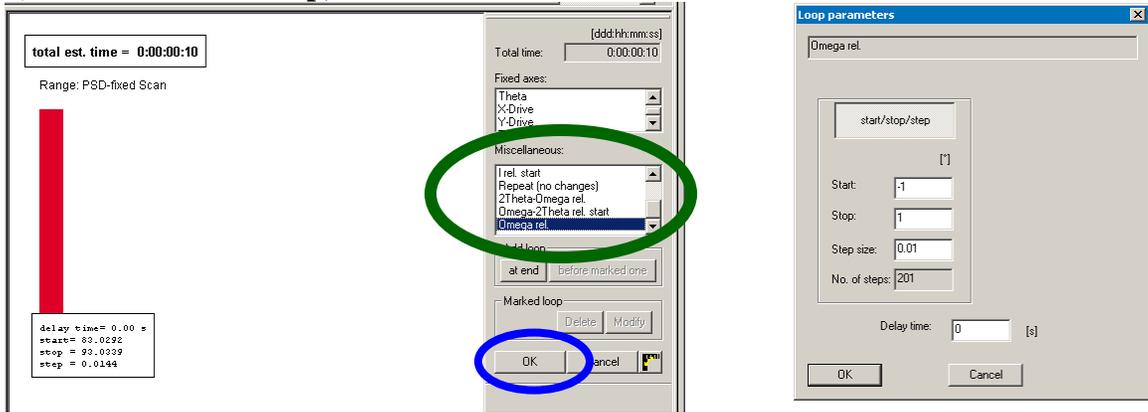
   iii) The *Loop Parameters* window opens
   iv) Set the **Start**, **Stop**, and **Step size** increments for that loop.
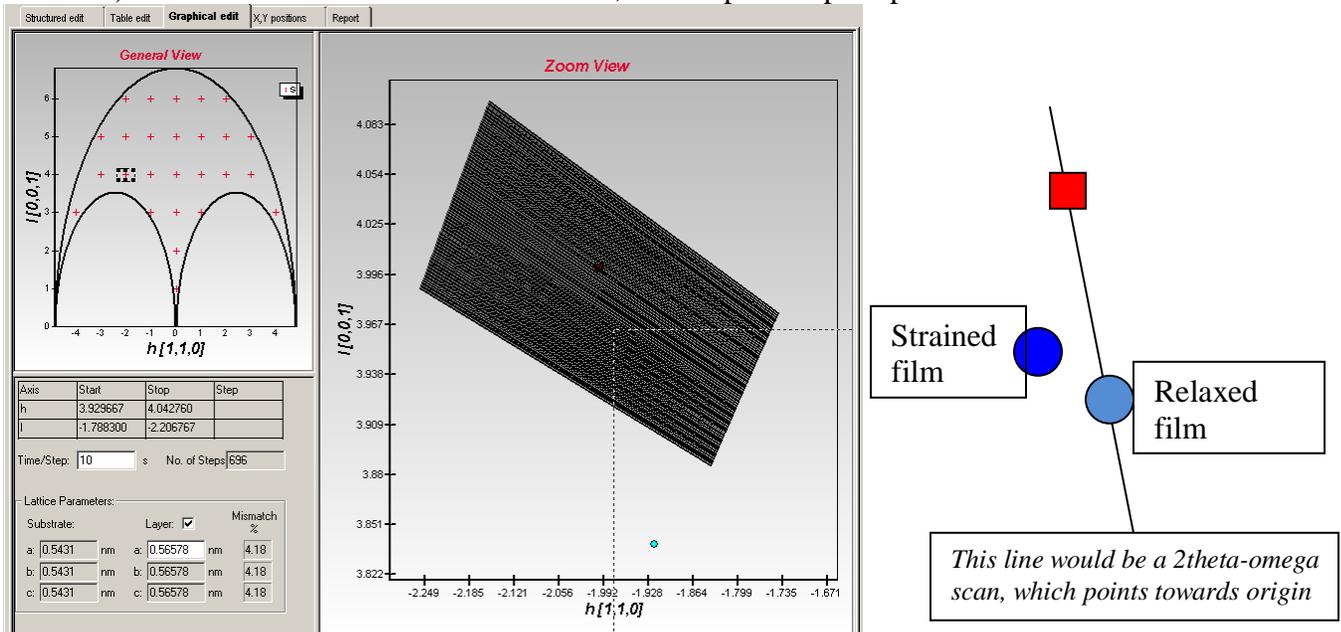   v) Click **OK**

g) Click **OK** in the *Loops* window (circled in blue above)
   i) This saves the loop and returns you to the first page of information

h) Select the <u>Graphical Edit</u> tab
   i) Make sure the program you just wrote will cover both the substrate and the film peak
   ii) Remember that if the film is strained, the reciprocal space point will be shifted

iii) In the example above, the program would not measure the film peak

i) If the program will cover the desired reciprocal space, then save it
   i) Go to *File > Save*
   ii) Save the program as a ***.dql** file

!! The time calculated for the loop is wrong!! It is based on the incorrect total scan time.
- *The time for the measurement is labeled as "Total Time", circled in black below.*
- *To calculate the correct total time for the loop, use the following equation:*
   $total\ loop\ time = (total\ scan\ time) * (number\ of\ loops) + (number\ of\ loops) * 5$
- *If you are using a scan range of 2.7 deg, and therefore not moving the detector during the scan, then the calculated total loop time will be correct.*
- *Use seconds as the units for all of the calculations.*

j) If the program would not cover the desired reciprocal space, then edit it

   i) If you need to change the detector scan, then:
!!!!!You must delete the loop before you edit the Scan Parameters!!!!!
      (1) Go to the *Loops* page by clicking on the entry **Loops** in the left hand hierarchal tree (circled in blue)
      (2) Click on the green boxes that identify the **Theta Rel** loop (circled in green)
      (3) Click on the **Delete** button (circled in red)
      (4) Click **OK**
      (5) Then go to the *Scan Parameters* page by selecting the entry in the left-hand hierarchal tree
      (6) Edit the Rel Start and Rel Stop for the detector scan
         (a) Click **OK**
      (7) Return the *Loops* page
   (8) Recreated the **Theta Rel** loop
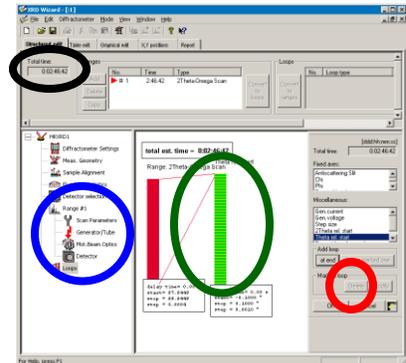
   ii) If you need to change the **Theta Rel** loop, then:
      (1) Double-click on the green boxes that identify the **Theta Rel** loop, circled in green
      (2) Edit the **Start** and **Stop** in the *Loops Parameters* window
      (3) Click **OK**
      (4) Click **OK** in the *Loops* page to save the change

   iii) Check the new program in the Graphical Edit tab

   iv) Continue editing the program and then save it once it will sufficiently cover the desired reciprocal space
      (1) Go to *File > Save*
      (2) Save the program as a ***.dql** file

VI. USE XRD COMMANDER TO RUN THE MEASUREMENT JOB YOU CREATED IN XRD WIZARD

1) In XRD Commander, select the menu *Jobs > Create Jobs*
2) In the ***Create Jobs*** table, enter values for Parameter File, Raw File, Script, and PSD-Subsampling
    a. **Parameter File**—select the *.dql file that you created in XRD Wizard
    b. **Raw File**—navigate to the folder where you want to save your data. Enter the filename and click the **Open** button
    c. **Script**—select the script **"measure_psd.vbs"**
        i. *If you used a scan range of 2.7 deg, so that the detector does not move during the measurement, then select **measurev4.vbs** for the script*
    d. **PSD-Subsampling**- enter **2**
        i. *If you used a scan range of 2.7 deg, so that the detector does not move during the measurement, then enter **1** for **PSD Subsampling***
3) You can configure XRD Commander to run multiple jobs. It will execute each measurement job in the order listed
4) Click the **Start** button to start the measurement.
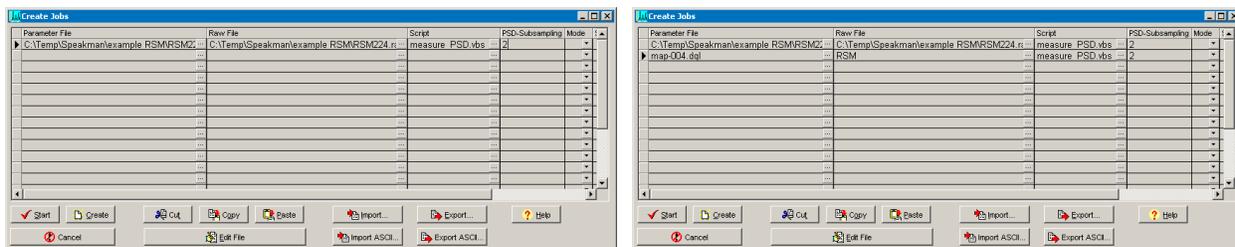
Tip for running multiple scans, including a symmetric and asymmetric peak
1) *You can write a program using the Pathfinder for the symmetric scan and the LynxEye for the asymmetric RSM.*
2) *Start with a quick alignment on the symmetric peak to determine if there is much sample tilt*
3) *Then align on the asymmetric peak and write XRD Wizard job as described above*
4) *Return to the symmetric peak and align on it using the same phi determined for the asymmetric scan*
5) *Write the XRD Wizard job for the symmetric peak as described in the SOP "Using XRD Wizard to Collect Data".*
6) *When you create the batch job in XRD Commander, be sure that you use the correct Script and PSD-Subsampling*
    a. *When data are collected using the LynxEye in scanning mode (range larger than 2.7deg), then **Script** is "measure_psd.vbs" and **PSD-Subsampling** is 2*
    b. *When data are collected using the Pathfinder or using the LynxEye in stationary mode (range is 2.7deg), then **Script** is "measurev4.vbs" and **PSD-Subsampling** is 1*