

XRD and Surface Analytics Platform

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Standard operating procedures for:

D8 Discover Plus – TXS, BCH 2118

Sample alignment for reflection measurements (HRXRD, GID, GIWAXS, micro-diffraction)

2Theta-omega scans + Rocking Curves

This SOP describes how to align a sample before a measurement session on surface diffraction (GID, IP-GID, GIWAXS), micro-diffraction or High Resolution Diffraction HRXRD (2T-om scan, Rocking Curves, Reciprocal Space Maps, Pole figures..), as well as how to perform basic scans 2T-om / RC.

GENERAL POINTS TO WATCH:

Remember that the most likely cause of serious problems are collisions between different moving parts of the goniometer and arms itself (e.g. primary optics and sample stages) and bad handling of the detector. Keep this in mind, protect the detector when you handle it and check with your eyes for potential collisions **before** moving motors.

- **DETECTOR HANDLING**
- **COLLISIONS ON GONIOMETER**

Logins:

Local PC: password

Diffrac: no pwd

Generator:

The instrument is ready to go when you see a yellow light (top, with a radiation symbol) and green (bottom) light on the left hand side of the enclosure. If the generator light/button is red and the generator off, press the red button, it should turn white (with an "I"). Press it again and let I condition.

Operating power 45 kV / 120 mA --- if too many arcs, run lower, even at 40 kV / 40 mA

Standby power 20 kV / 6mA

Data and resources:

See wiki on <https://www.epfl.ch/schools/sb/research/isic/platforms/x-ray-diffraction-and-surface-analytics/>

Instrument configuration: modify before sample alignment

Chose according to sample and resolution required.

As a general starting point for sGID-applications and HRXRD-applications you can use the Göbel Mirror, an 0.2 mm slit, an Axial Soller 2.5 ° on the primary side (add the antiscatter nozzle on position 4 for GID); the axial Soller 2.5 ° on the secondary side.

As a general starting point for GIWAXS, remember you need a point beam since you will use the 2D detector mode, you can use the Göbel Mirror, an 0.5 mm pinhole, position 3 empty slit box, and an 0.3 or 0.2 mm double collimator on position 4; empty beam path on secondary side.

Microdiffraction will require collimation optics as well, and can then be performed with either a 1D or a 2D detector.

Sample (and instrument) alignment: a minimum of 4 alignment scans

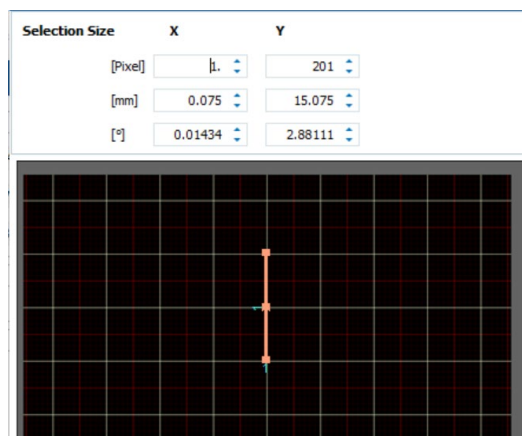
- Remember to delete offsets (Commander – clear all offsets) prior to alignment.
- Though not compulsory we typically use, in Diffrac, the application type “Powder Diffraction” for GID, GIWAXS and microdiffraction, the application type “HRXRD” for IP-GID and everything else in reflection mode and requiring sample alignment. Sample alignment is here described for the HRXRD application, but is analogous in the Powder Diffraction application (motors are named differently in some cases).

Alignment consists of a minimum of 4 alignment scans, which will reference the 2Theta scale, the Omega scale (called Theta in application type PXR), orient the sample parallel to the primary beam and bring the sample to the correct height where it shadows exactly half the primary beam. **The correct measurement height is approx. 40 mm from the base of the Eulerian cradle, adapt with spacer (1,2,3 mm) if needed!**

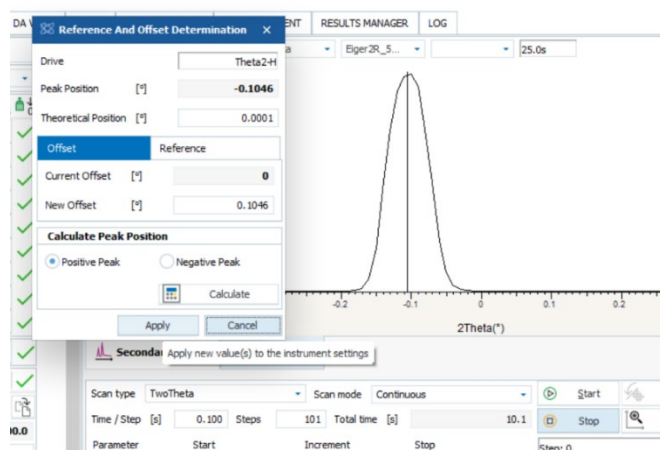
Workflow of alignment:

- 2Theta scan (references 2Theta to 0°)
- Initial height scan (brings sample to correct height to shadow half the primary beam)
- Rocking Curve scan (references Omega/Theta to 0° resulting in the sample being parallel to beam)
- Final height scan (determines final height for measurement)
- Optional: when using the Kalpha1 2-bounce monochromator a psi scan should be performed to bring the surface normal of the film into vertical position. This is not necessary when using only the mirror. Psi, RC may need to be iterated.

- All alignment scans are performed with a 0D detector, choosing a line of 1 pixel vertical to scanning direction, and about 200 perpendicular to that (on right show for detector in 2T-optimized orientation, can be different for GIWAXS). This ensures good resolution of the alignment scans. You can optionally use a Cu-absorber placed in front of the detector. This is not necessary but can be useful when you think the detector may be saturating, resulting e.g. in a broad-ish peak in the 2Theta scan.



- 2Theta scan:** make sure all offsets from the previous user have been “cleared”. Ensure the beam will not be obstructed by the stage or the sample during the 2Theta scan. Bring the X-ray tube (omega drive in HRXRD or theta drive in PXRD mode) to 0 degrees. Bring the z-stage to -1 mm, x and y either at 0 / 0 or at the positions of the current sample or measuring spot. Chose 0D detector (ROI of 1 x 200 px). Chose 2Theta scan from -0.5 to 0.5 ° 2T, step size 0.01 °, time per step 0.1s. Perform scan (see below). You should see a peak, probably not exactly at 0°. If the peak is flat or a doublet mount the Cu absorber and repeat. To determine the offset on the 2Theta scale go to “Commander – Reference and Offset determination” (see below). A window will open and you will see a line appear in the centre of the peak. Click “apply” once (!) then click “cancel” to close the window. If you want to verify that your offset is stored click on the icon “additional settings” next to the 2 Theta drive in Diffrac Commander. If you are unsure of what you did you can always “clear” the offsets again.



- Initial z-scan (height):** ensure that both the X-ray source AND the Two Theta drive are at 0°. If you have not mounted your sample yet do this now. Chose z-scan from -1 to 1.8 mm (or adjust to other values respecting these limits) at 0.01 step size and 0.1s per step. In the z-scan, you should see a plateau at high intensity which then shows a step down to zero intensity. The plateau is the primary beam, the step is due to sample absorption. The new height is found at half the initial intensity. You can zoom in into the step (left click and drag box) and double click on the curve at the correct position (i.e. half of primary beam intensity, read values of x-y axis at bottom left of Commander). By double-clicking on the curve the new z value is automatically defined as new set point for the next scan. If the z-scan is very noisy and/or low in intensity you have probably forgotten to bring the 2Theta drive to 0°. If the z-scan starts with zero Intensity (or does not show a step at all) your sample is shadowing the beam (or too low), you have to adapt the sample height manually by removing (adding) spacers.
- Rocking Curve:** at the previously determined height, chose now the rocking curve scan, and scan from -1 to 1 ° in omega, at 0.03 – 0.05 stepsize (or larger) and 0.1 s per step. You should see a clear maximum in the RC, which will be narrower for large substrates than small ones. As long as you see a clear maximum all is ok. Go to “Commander – Reference and Offset determination”. A window will open and you will see a line appear in the centre of the peak (as for the 2Theta offset in step 1). Apply the offset once (!) and cancel to close the window. You can verify your offset was stored by clicking on the “additional settings” next to the omega drive in Diffrac Commander (theta in PXRD application). Now your sample is parallel to the beam, and you probably need to correct the height again.
- Final z-scan (height):** as “initial z-scan”.

2Theta – Omega scan:

The 2T-om scan is used to measure d-spacings (lattice parameters) and identify phases. This scan is done with a 1D detector, and the configuration described above. Your sample is now aligned, remove the Cu absorber in case you used it, and ensure the 2.5 ° axial Soller is placed in front of the detector (large or small Soller).

- Chose 1D detector and define ROI. This will depend on your sample (resolution, crystallinity, peak overlap). You can start at 100 x 200 px if you are unsure (a smaller window in scan direction will increase resolution).
- Choose start and stop 2Theta values, increment and time per step. Rule of thumb for increment: at least 9-10 points above the FWHM of the peak.
- Start scan and save when finished.

Rocking curve:

The RC is used to measure mosaicity (tilt distribution of a certain hkl plane) and performed with the 0D detector mode. It is measured for one single peak. Usually one measures a 2T-omega scan prior to the RC in order to obtain current experimental 2Theta values for peaks of interest. The RC is then performed at the exact 2T and omega values of the peak. This provides the diffraction condition for (hkl). During the RC the two drives are moved simultaneously, while maintaining 2T and om constant, which is analogous to performing a “rocking” movement of the sample.

- In your 2T-omega scan, if you double click on a peak maximum (ex the one you would like to perform a RC on) the values 2Theta and om will automatically be transferred as new set points of the drives. You can also type the 2Theta value manually into the setpoint box.
- Chose the ROI according to the sample’s mosaicity (smaller region = better resolution but longer measurement time). Optimize the ROI (ex 20 x 200) then use for samples you want to compare.
- Chose the scan type RC and verify that your drives are set to the correct set points (green circle below), and that the 2Theta value in the scan (red circle below) is also correct. If not, type correct values (for example taking them from your 2T-om scan you probably did prior to this). In the example below the system is set up to perform a RC around 2T = 31.56° and om = 2T/2, in a range of om = 10...20°, a step size of 0.03° and a time per step of 0.2s.
- Define the measurement range in omega (!!!), NOT 2Theta. (ex below from 10-20°). Start the RC scan.
- If your curve is flat, there is no alignment of crystallites (i.e. your sample is powder-like). The narrower the RC the lower the film’s mosaicity. If you have a broad peak and cannot capture the shoulders then increase the range of the RC. If you have a very sharp peak make sure the FWHM criterion is respected (10 point above FWHM). Note that at low angles (approx. 5-10 om) some primary beam intensity will always contribute somewhat to the scan intensity, but does not indicate a peak.

| Parameter | Unit | Actual | target |
|---------------|--------|---------|---------|
| g0-H | [°] | 0.0000 | 15.7800 |
| a2-H | [°] | -0.1045 | 31.5601 |
| ector | [°] | -0.1045 | 0.0000 |
| f | [°] | 0 | 0 |
| i | [°] | 0.0000 | 0.0000 |
| n-H | [mm] | 0.0000 | 0.0000 |
| | [mm] | 0.000 | 0.000 |
| | [mm] | 0.000 | 0.000 |
| | [mm] | 1.8050 | -1.0000 |
| able rotation | [/min] | 0.0 | 0.0 |

ray Generator

| | | | |
|------|------|--------|-----|
| age | [V] | 45 | 45 |
| ent | [mA] | 120 | 120 |
| W | [W] | 5400.0 | |
| iter | | Set | Off |

