

How to use the CrystalPack Module

The Thermo Scientific™ CrystalPack™ Module is a software package designed to assist you when working with electron crystallography. It allows for zone axis tilt, K-space construction, tilting for two-beam conditions, and other functionalities.

It is particularly useful for:

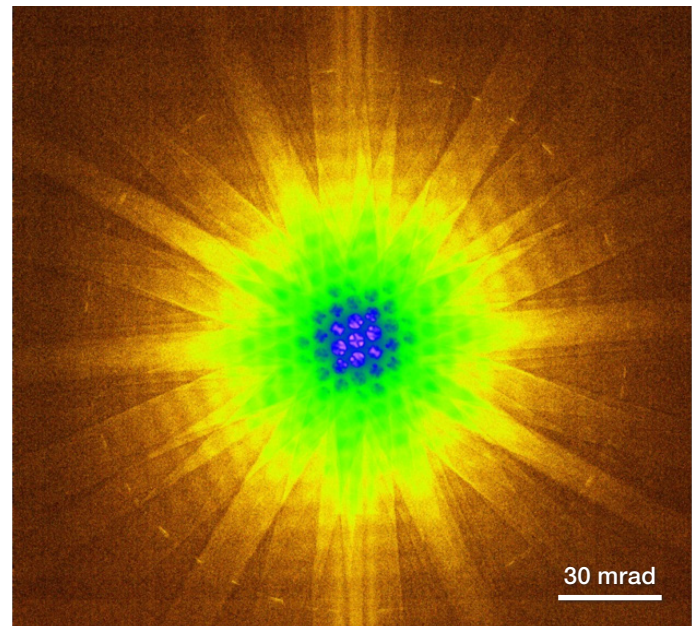
- Fast zone axis search and tilt
- Zone axis identification
- Low-symmetry/high-index zone axis identification and tilt
- Zone axis implicit search of beam-sensitive materials

General workflow

- Holder compucentricity calibration
- K-space construction when necessary
- Usage of Smart Tilt

Prerequisites

- CrystalPack Module includes: Compucentricity, K-space control, and Smart Tilt when installed
- Diffraction and imaging (i.e., camera length and scanning/transmission electron microscopy [S/TEM] magnifications), SmartCam remote operation are all well calibrated
- Mechanically stable holder
- TEM specimen possessing a distinct feature, prepared, and properly mounted in the chosen holder



Detailed workflow

Holder compucentricity calibration

1. This compucentricity calibration file represents a computed position correction to correct tilt-induced holder off-eucentric and lateral movements.
2. Ensure that the CrystalPack Module is loaded into a single workspace including the **three** parts: **Compucentricity**, **K-space Control**, and **Smart Tilt** (Figure 1). It is advised that these control panels be grouped into the same tab for convenience.

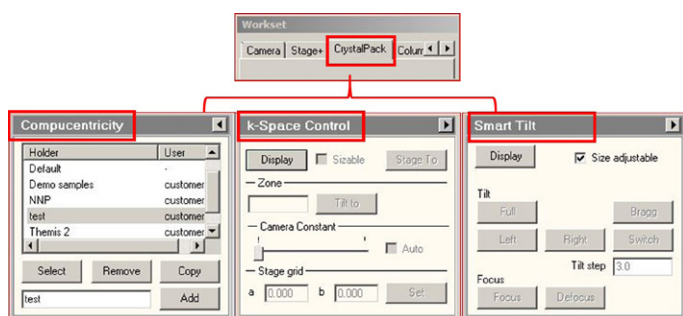


Figure 1. CrystalPack Module.

3. Load a pre-aligned TEM or STEM field emission gun (FEG) register.
 - 3.1 Alternatively, you can align the microscope in TEM or STEM mode using **Direct alignment**.
4. **Compucentricity calibration and test:**
 - 4.1 Regarding how the compucentric β tilt works, refer to Figure 2.
 - 4.2 Mount the sample in the chosen double-tilt holder.
 - 4.3 Load the holder in the microscope and wait for the drift to settle; this will take approximately half an hour.

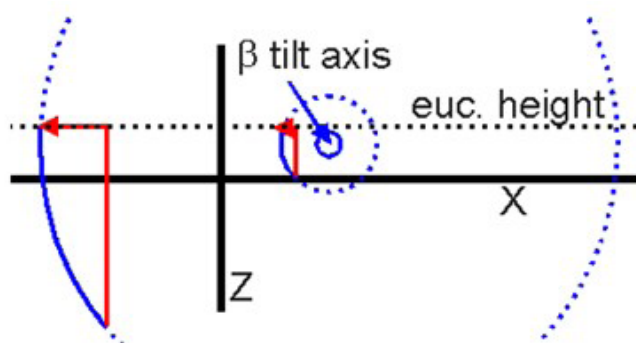


Figure 2. Schematic showing compucentric β tilt axis. The thick black lines show the locations of the X and Z stage axes, while the dotted black line indicates the eucentric height. The central blue circle is the β tilt axis, which is displaced from its "ideal" X 0, Z 0 location. The dotted blue circles indicate the path traveled by a point when tilted around β . Tilting around β for the point lying on the smaller dotted blue circle requires a small correction to X and Z (red arrow lines) to bring the area back to the same position in space (but a different stage position). For the point lying on the larger dotted blue circle, the correction (red arrow lines) is much larger because it is further away from the tilt axis itself. The corrections are applied automatically when compucentric tilting is used.

- 4.4 Find a distinct feature around center (X, Y ~ 0) and ensure the area at eucentric height. Note that eucentric height means a tilt axis, which can be achieved by minimizing the feature movement while a tilt wobbling.
- 4.5 Go to the CrystalPack tab and select the **Compucentricity** panel (Figure 1) and create a specific compucentricity file name for the selected holder and press **Add**.
- 4.6 Press **Select** to choose the holder to get calibrated. Flip out the additional controls by pressing the arrow button (located in the top right corner).
- 4.7 Go to the **Calibrate** page, press **Start**, and follow the automatic routine for calibration (Figure 3). This routine performs a tilt series in 5° steps from $0 \rightarrow -25 \rightarrow 0 \rightarrow 25$ degrees and calculates an interpolation for stage movement corrections caused by the beta tilting.
 - 4.7.1 It is critical to ensure centering the feature and correcting eucentric height as accurately as possible for each tilt, which is given in the instructions during the calibration routine.
- 4.8 Find a recognizable feature around X shift of approximately $-400 \mu\text{m}$ and ensure the eucentric height, run another round of the compucentricity calibration procedure described in 4.7; when completed, click **Replace**.
 - 4.8.1 This procedure aims at finding more realistic values for the rotation of the β tilt axis in the direction of the α tilt axis of the stage and for the rotation of the β tilt axis in the vertical direction, i.e., a non-existent γ tilt axis of the stage.
- 4.9 Find a recognizable feature around X shift of approximately $+400 \mu\text{m}$ and ensure the eucentric height. Then, run another round of the compucentricity calibration procedure from 4.7. When completed, click **Compare/Add** to add another point for calibration.
 - 4.9.1 This procedure aims at adding another point so that the compucentric tilting can be applied for a broader area.
 - 4.9.2 Note: If there were not distinct features over the positions mentioned above, other positions can be used as well, except the positions far from the center, i.e., X shift is larger than $500 \mu\text{m}$ or around the end of the range.

4.10 By performing the steps 4.7–4.9, the β tilt axis can be found, and it can correct the β -tilt-induced movement at a region far away from the center.

4.10.1 Note: Different holders certainly require separate calibrations. To obtain best possible compucentricity calibration for practical use, it is strongly advised that you perform compucentricity calibrations even for the same holder with regard to different types of samples; for instance, a FIB sample, copper grid sample, 3-mm disc sample, and so on. When a specimen does not have a distinct feature around the positions mentioned above, the compucentricity calibration may not be as accurate, but it will still ease tilting.

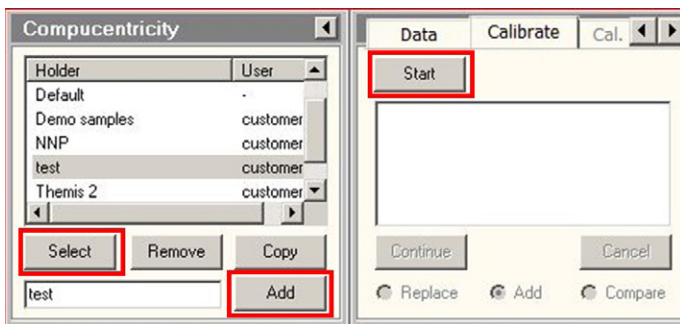


Figure 3. Compucentricity functionality and calibration.

4.11 **Compucentricity test:** Go to the **Option** page in the flap-out of **Compucentricity** (Figure 3), select the box **Use compucentricity tilting** and INPUT a tilt angle of 20 or -20 degrees. Press **set B** and, after the stabilization, measure the distance that the feature moves (Figure 4). This value should be less than 2 μm .

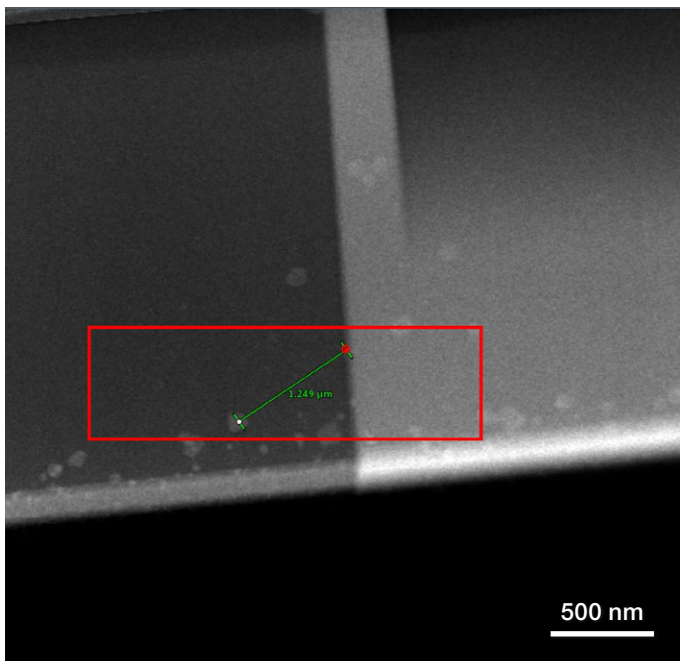


Figure 4. An example showing the center feature movement with 20-degree β tilt after compucentricity calibration.

K-space construction

Crucial prerequisite: The material's crystallography is known prior to the experiment.

1. Find the crystallographic information (symmetry information including the lattice constant, crystal systems, and centering, or simply its space group information) from literature for the investigated material BEFORE it is put into the microscope.
2. Select the compucentricity calibration for the current holder with the sample type combination.
3. Accurately find two known zone-axes with index zones as low as possible for subsequent identification and save these two stage positions in the **Stage** panel. The zone axes can be found either manually or by using **Smart Tilt**. Regarding how to use Smart Tilt, refer to the next section: **Usage of Smart Tilt**. These zone axes need to be identifiable.
4. Click **Display** in the K-space control window in the CrystalPack panel (Figure 5). A floating window of the K-space of a simple cubic lattice appears with a simulated diffraction pattern, including Kikuchi line. When closing the **K-space control** floating window, DO NOT use the **cross** of the window, but instead click **Display** in the control panel again.
5. Go to the **Crystal** flap-out and click **New** and then input the crystallography information into the system (Figure 5). The floating window of K-space will simultaneously be updated with the new crystal. For visualization of K-space, the **Display** page offers several options for displaying Kikuchi patterns, diffractions, spots, etc. The display option is up to your preference.
6. Ensure the **Compucentricity** option (Figure 5) is selected during the entire procedure.
7. The crystal file can be also saved by pressing **Save** or **Save as**. Then, it can be loaded from saved crystal files by pressing **Open**.

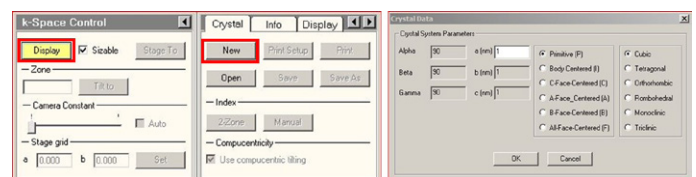


Figure 5. Diagrams showing K-space control functionality.

8. To establish the K-space orientation for a crystal, there are two approaches where one is **2Zone** and the other is **Manual**. We strongly recommended you use the **2Zone** method as directed below:
 - 8.1 Click **2Zone** under the **Index** menu. The **Two Zone Indexing** floating window will appear (Figure 6).
 - 8.2 Go to the saved stage position for one zone axis and, if necessary, adjust the stage (tilt) to be accurately at the zone axis.
 - 8.3 Go to diffraction and ensure that the zone axis, as well as the diffraction pattern, are centered.
 - 8.4 Press **Calculator** for the first zone axis. The **Zone Axis Calculator** will appear (Figure 6).
 - 8.5 Press **Start** and follow the steps to index a crystal. For each indexing, it is recommended to use at least THREE diffracted beams in addition to the transmitted beam (Figure 6).
 - 8.6 Press **Calculate** to index the diffraction pattern and select the proper zone axis index.
 - 8.6.1 Note: If this indexing does not match as expected, please check the following:
 - Is the crystallographic information correct?
 - Or press **Setup** (Figure 6) to set larger tolerances of matching constraints for indexing.
 - 8.7 Repeat 8.2–8.6 for the second zone axis found earlier. After completion, press **Index** to establish the K-space orientation. It must be realized that these two zone axes should be crystallographically consistent with the tilts, meaning that the indexes chosen should match the measured angle between two stage positions.

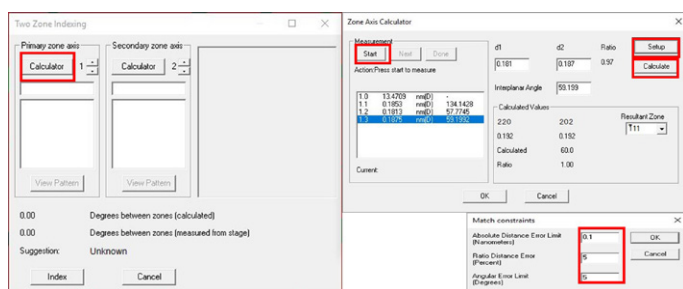


Figure 6. 2Zone windows to construct K-space.

- 8.8 Alternatively, the **Manual** approach (Figure 7) can be used for indexing K-space, provided that one zone axis can be found and the exact orientation for north direction is known for the diffraction pattern.

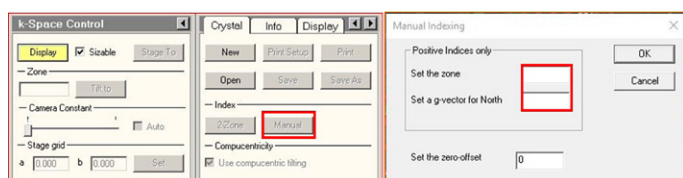


Figure 7. Manual approach to construction of K-space.

8.9 K-space control test:

- 8.9.1 Choose one zone axis that is allowed by the tilt range and input the zone axis chosen into **Tilt to** and press **Tilt to** (Figure 8).

8.9.1.1 Note: Be aware that the double-tilt holder has a certain tilt range. In particular, the tilt range may be reduced at an extreme stage position. Thus, do not tilt to any zone axis outside of the tilt range. The tilt range must be within the stereographic projection range indicated by the blue lines under the reasonable stage position; otherwise, caution needs to be paid. If, by accident, an extreme tilt is requested and performed, immediately press **ESC** on the keyboard to stop the tilt.

8.9.1.2 Note: Numbers on the keyboard indicate a normal number, while the numbers on the small keypad indicate the negative number with “bar.”

- 8.9.2 Press **Stage to** to go to the selected zone axis and finetune the holder tilts and sample centering for the most accurate zone axis centering.

- 8.9.3 Acquire a diffraction pattern for indexing and verifying if K-space control produces the correct results.

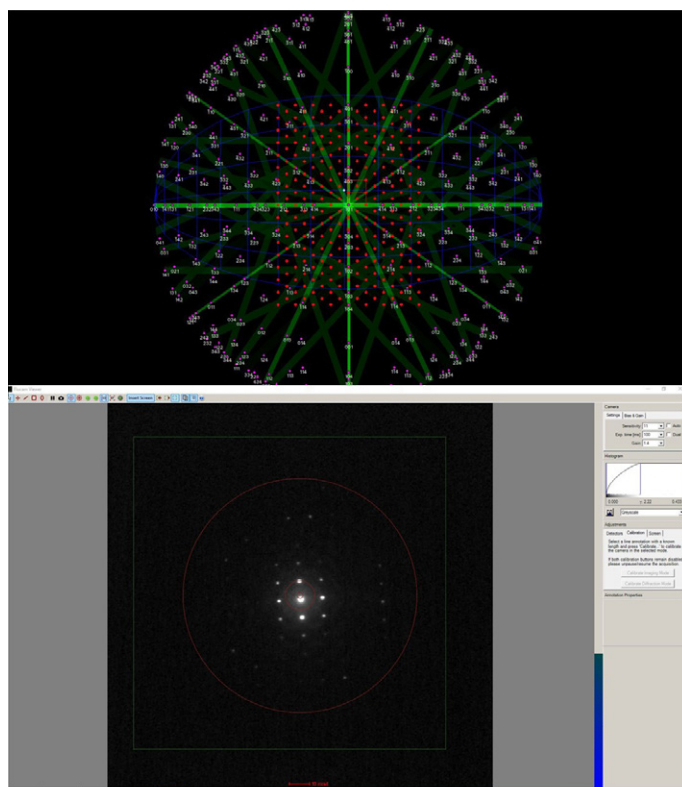


Figure 8: One application example showing the reconstruction of silicon K-space (upper figure) and, accordingly, a low-symmetry zone axis $[3\bar{2}3]$ found and tilted (lower figure) automatically using K-space control.

Using Smart Tilt

1. Select the correct compucentricity holder calibration, and ensure that **Use Compucentric Tilting** is selected (Figure 7).
2. Choose a suitable diffraction approach (for example: SAED, CBED, Kikuchi pattern, etc.), which may depend on the material. For example, for beam-sensitive materials, it is advised to use SAED with a small condenser aperture.
3. Click **Display** under Smart Tilt in the CrystalPack panel. The **Smart Tilt** floating window will appear (Figure 9). When closing the **Smart Tilt** floating window, DO NOT use the **cross**; instead click **Display** again.
4. Choose the area of interest and go to diffraction.

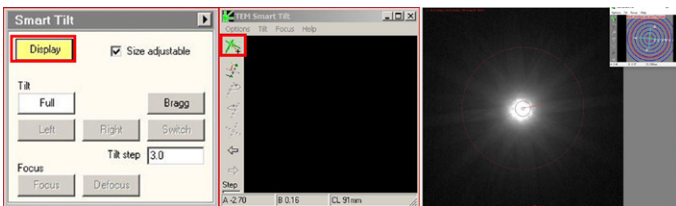


Figure 9: Smart Tilt functionality for zone-axis tilt. Right figure shows $\langle 110 \rangle$ Si zone axis found using Smart Tilt.

5. Press **Zone axis tilt** in the **Smart Tilt** window, following the instructions to do the zone axis tilt: Center the transmitted beam → Center diffracted beam → Find the area of interest back. Iterate this procedure until it reaches the required accuracy.