

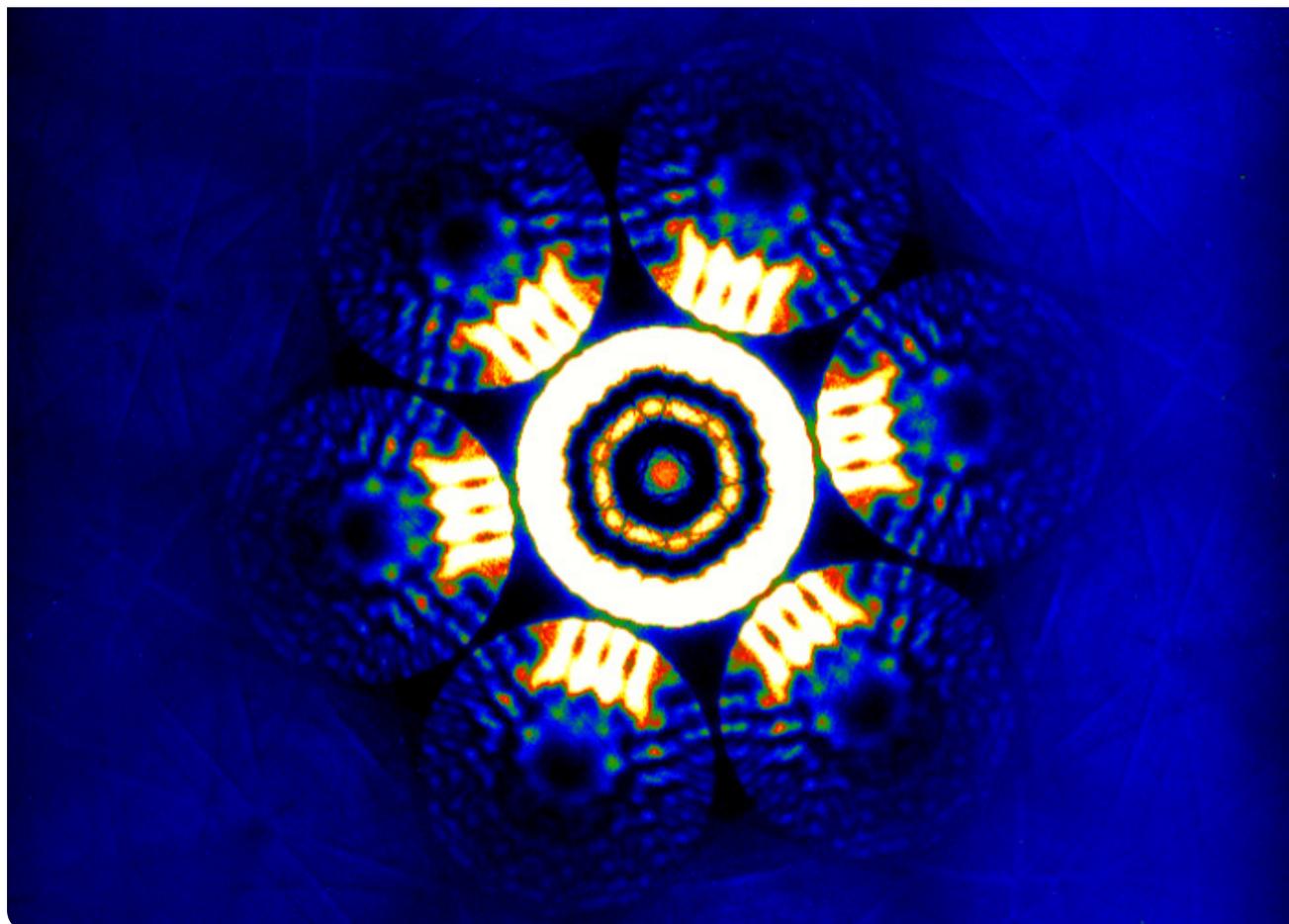
CrystalPack Module

Are you a metallurgist? Or a materials scientist working a lot with the same type of single crystalline samples? Or looking for an easy way to tilt from zone axis to zone axis automatically in less than a minute?

The Thermo Scientific™ CrystalPack™ module enables users to do compucentric β tilting, rapid toggling between diffraction conditions and can automatically tilt to the relevant zone axes.

The CrystalPack module consists of three parts:

1. **Compucentricity** provides the functionality for compucentric tilting of the beta tilt axis of a double-tilt specimen holder, i.e. tilting that is pseudo-eucentric through corrections applied to the other stage axes.
2. K-space / reciprocal space and its manipulations to orient crystals can be difficult to handle and **K-Space Control** is designed to make working in diffraction considerably easier. The program assists in indexing diffraction patterns so that zone axes become simple to determine. The program can then be instructed to set the tilts to that orientation automatically.
3. In order to provide a match between the orientation of the k-space display and the image on the TEM, **Smart Tilt** is used.



1. TEM Compucentricity

Compucentricity (see Fig. 1) provides the functionality for compucentric tilting of the beta tilt axis, i.e. tilting that is pseudo-eucentric through corrections applied to the other stage axes. Compucentricity thereby minimizes the sideways movement of the image when the beta tilt is changed. Compucentricity can only be applied to software-controlled motions such as those of Smart Tilt and k-Space Control, and does not apply to manual motions which are under direct user control. When applied to a calibrated double-tilt holder, the image movements resulting from (even large) changes in beta-tilt angle can be reduced to values as small as 1 to 2 micrometers whereas real displacements that can amount up to several hundreds of micrometers on the X and Z axes.

The beta tilt axis of double-tilt holders is non-eucentric (see Fig.2); beta-tilt eucentricity would require changing the height of the beta tilt axis relative to the alpha tilt. The behavior of the non-eucentric beta tilt is, however, predictable when the location of the beta tilt axis is known. By deriving the latter from a calibration procedure, corrections can be applied to the other stage axes (predominantly X and Z) to compensate for the non-eucentric motion. The accuracy of the compensation strongly depends on the accuracy of specimen-position measurement. The effective compensation accuracy is of the order of 1 to 2 micrometers, independent of the actual change in beta tilt applied. Since manual beta tilting is typically done slowly, the compucentric corrections would be worse than any typical image movement during manual tilting. Compucentric corrections are therefore only used when (larger) software-controlled changes in beta tilt are applied.

Compucentricity consists of software for the compucentricity calculations and holder calibration procedure. Its use is through the regular microscope user interface Stage control or through k-Space Control and Smart Tilt. Compucentricity consists of :

- Establishing the current stage position and the required new tilts
- Calculating what corrections should be made to the stage axes to keep the feature of interest centered on the screen
- Moving the CompuStage to the new position

Since specimen holders deviate from the 'ideal' model, a correction can (and for good results should) be made for this 'non-ideal' behaviour through a calibration procedure. Data are stored for each specimen holder separately. Prior to calibration holders must therefore be identified by name.

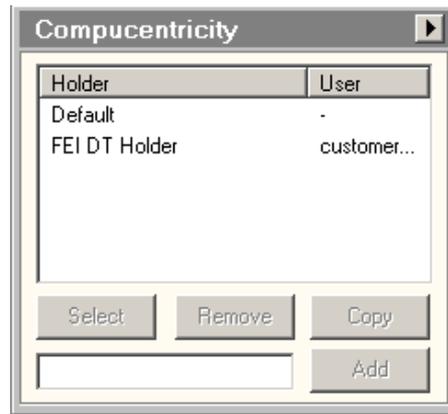


Figure 1. Compucentricity control panel

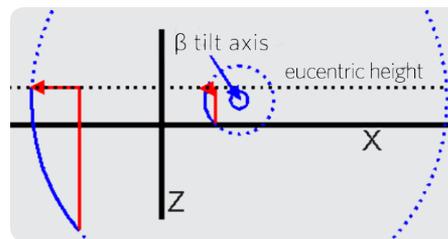


Figure 2. Schematic diagram of the effect of the β tilt. The thick black lines show the locations of the X and Z stage axes, while the dotted black line indicates the eucentric height. The central blue circle is the β tilt axis, which is displaced from its 'ideal' $X=0, Z=0$ location. The dotted blue 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.

2. K-Space Control

K-space/reciprocal space and its manipulations to orient crystals can be difficult to handle and k-Space Control is designed to make working in diffraction considerably easier. The program assists in indexing diffraction patterns so that zone axes become simple to determine. Once one or more zone axes have been defined and their orientation relative to the specimen stage determined, a stereographic projection of the structure is shown. With the grid of the goniometer tilt axes overlaid, it is easy to assess whether any desired orientation is within reach of the tilts. The program can then be instructed to set the tilts to that orientation automatically.

The display of k-Space Control can be tuned to suit the needs at a particular moment. See figure 3. All elements of the display (the stereogram grid, grid of the CompuStage axes, zone-axis positions and indices, Kikuchi lines, spot diffraction patterns and structure projections) can be turned on or off. Maximum indices for zone axes and Kikuchi lines can be chosen to make the display less crowded at low camera constants or reveal more detail at high camera constant.

The orientation of the stereographic projection can be changed instantaneously in various ways, for example by clicking with the cursor on any zone axis within reach of the CompuStage tilts. Thus, different zone axes with their spot or Kikuchi line patterns and their structure projection can be selected for viewing. Tilting can be achieved simply by selecting an orientation and then pressing Move. k-Space Control will then instruct the CompuStage to tilt to the new orientation. Additionally since Compucentricity is present, tilting can be selected to be compucentric (correcting for non-eucentric behavior of the beta tilt axis). In order to provide a match between the orientation of the k-space display and the image on the TEM, Smart Tilt is used.

In short, k-Space Control is software that uses an input set of crystal-lattice parameters and lattice type to

- Create a stereographic projection of the crystal's zone axes
- Simulate the electron diffraction patterns and Kikuchi lines that would be seen in the TEM when the crystal is tilted to various orientations.

The Kikuchi- and diffraction-pattern simulations can aid the identification of diffraction patterns from the crystal's zone axes in the TEM.

In conjunction with the automatic control of the CompuStage and the ability for on-line diffraction pattern measurement of the microscope, k-Space Control becomes a powerful tool for crystallographic research with the TEM. The orientation of the crystal can be determined with respect to the tilt axes of the CompuStage and the crystal can then be tilted by the software to any zone axis within the range of the specimen tilts.

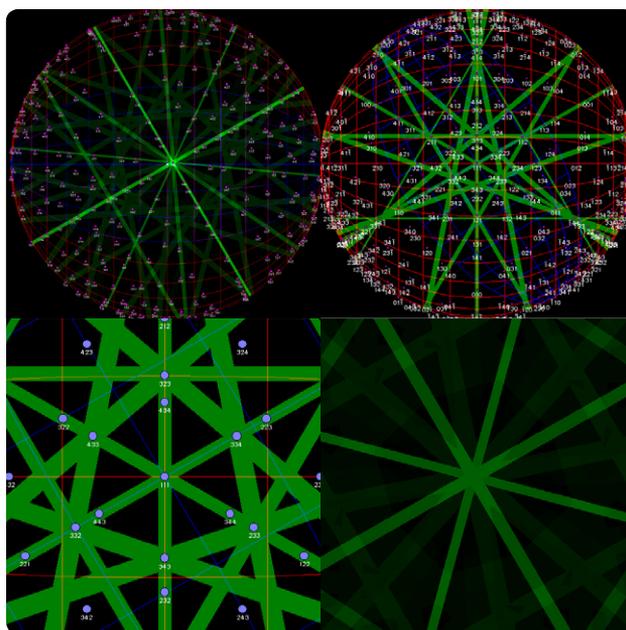


Figure 3. Various examples of the stereographic projection. A User can setup what is displayed; zone axes, indices, Kikuchi lines (green) and stage grid (blue) and more.

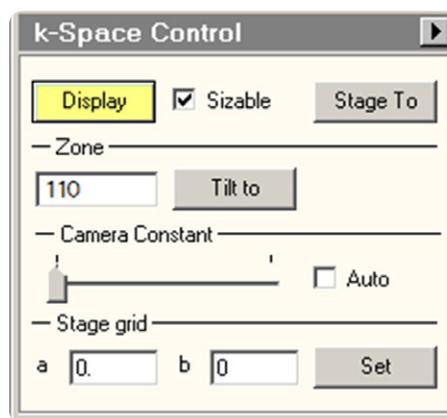


Figure 4. K-Space Control control panel

3. Smart Tilt

Diffraction experiments can be difficult, especially in combination with the non-eucentric β tilt. Users often find it difficult to predict by how much the stage must be tilted to bring a certain feature of the diffraction pattern (zone axis or systematic row) into alignment with the optical axis.

Smart Tilt gives assistance to the user in tilting with a double-tilt specimen holder. The assistance takes various forms:

A display representing the viewing screen of the microscope with the tilt axes and tilt angles overlaid. The display gives an indication of which axis and direction to use to achieve a particular tilting direction. It is also possible to double-click on the screen display and the stage will automatically tilt to the orientation indicated by the cursor position (see Fig. 6). The display is kept synchronized with the camera length of the microscope;

The possibility to double-click with the left-hand button of the mouse anywhere on the 'viewing screen' displayed and the feature corresponding to that location will be brought to the center of the diffraction pattern by tilting (see fig. 7).

Accurate measurement of tilts necessary to bring certain features in the diffraction pattern along the optical axis or tilt them half-way ('Bragg' tilting to go from a diffracted beam in Bragg condition to the systematic row). After Bragg tilting it is possible to tilt left or right around the systematic row afterwards in user-defined tilt steps.

For both CBED and SAED diffraction, the program provides a method for rapid toggling between a focused and a defocused (shadow image) diffraction pattern.

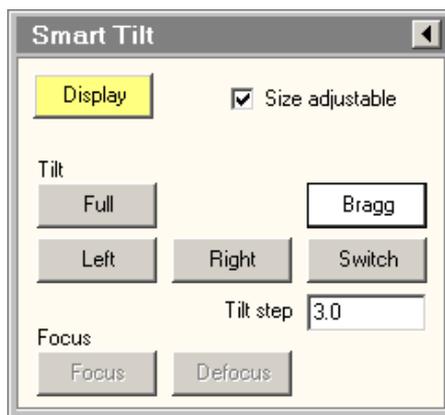


Figure 5. Smart Tilt control panel

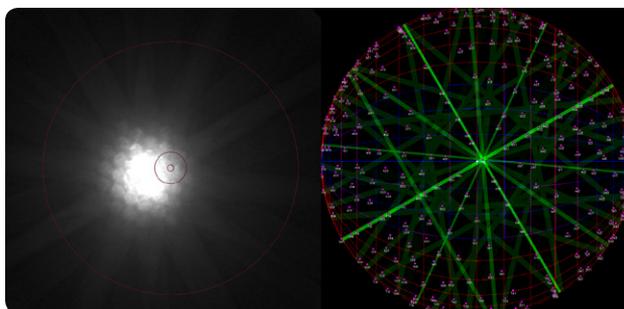


Figure 6. SmartCam and stereographic projection aligned. In the stereographic projection the zone axes (pink) with indices, Kikuchi lines (green) and stage grid (blue) are shown.

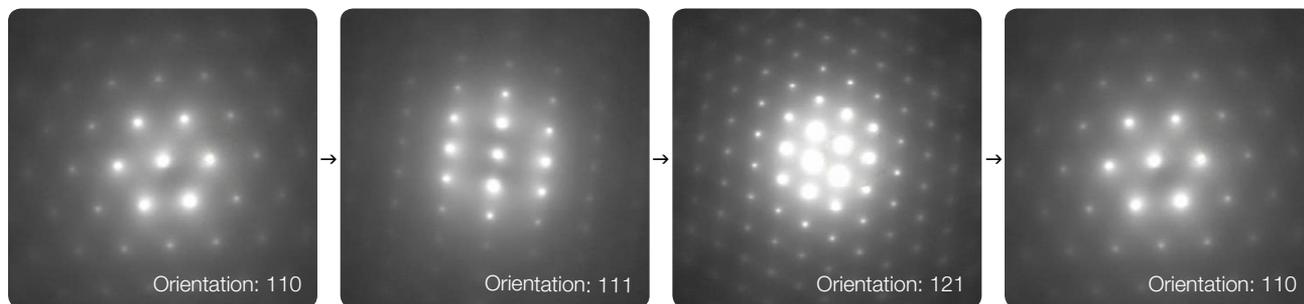


Figure 7. SmartCam with different Si zone axes. The user can simply enter the zone axis and with a single mouse-click the sample is tilted automatically to the desired zone-axis in a very reproducible way, in less than a minute. This example shows tilting from zone axis to zone axis on Si [110]: from 110 to 111 to 121 & back to 110.

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