# **SmartAxis**

## User Manual

## **1.Introduction**

The SmartAxis software is specifically developed to facilitate real-time and precise alignment of zone axes in crystal materials, catering to the micro- to nanoscale range. It seamlessly integrates with the hardware, providing online control and optimization capabilities for efficient and accurate zone axis alignment.

Zone axes alignment in the context of transmission electron microscopy (TEM) refers to the precise orientation of a crystalline sample so that the electron beam is directed along a specific crystallographic direction or zone axis. This alignment is critical for several advanced TEM techniques, including Selected Area Electron Diffraction (SAED) and High-Resolution TEM (HRTEM), where image and diffraction pattern quality are highly dependent on the sample being correctly oriented with respect to the incident electron beam.

Accurately aligned zone axes are crucial for:

1. **Symmetrical Electron Diffraction Patterns**: Proper zone axes alignment results in symmetrical diffraction patterns, which are essential for the structural analysis and characterization of the material.

2. **High-Resolution Imaging**: For HRTEM imaging, the zone axis alignment ensures that atomic planes are parallel to the beam, allowing for the visualization of atomic columns and defects within the crystal lattice.

3. **Phase Identification and Crystallography**: By aligning different zone axes, researchers can identify various phases within multiphase materials and study their crystallography.

4. **Defect Analysis**: Proper alignment allows for the study of defects such as dislocations, stacking faults, and twins, which can have significant effects on material properties.

Software like SmartAxis facilitates this alignment by automating the control of TEM's beam tilt and holder positioning, ensuring that the electron beam is accurately aligned with the desired crystallographic axis, hence improving the quality of data obtained from the TEM analysis.

### **2.**Calibration

To match the software with an existing microscope equipped with a double-tilt holder, two microscope parameters must be determined initially: the alpha and beta tilt axis with respect to the beam tilt. This calibration step is required only once for each microscope. To calibrate the alpha and beta tilt, a sample with a known preferred orientation of a specific zone axis is needed. We recommend using a single crystalline Silicon sample prepared using a focused iron beam (Fig. 1a).

Here are the steps to calibrate the tilt axes:

a. Firstly, align the sample perfectly into the zone axis using the double-tilt sample holder (Fig. 1a).

b. Increase the alpha tilt of the double-tilt holder by 3°. The crystal will be tilted off from the zone axis and positioned close enough to a pole to identify a Laue circle (Fig. 1b).

c. Adjust the beam tilt to realign the selected-area electron diffraction (SAED) pattern back to the zone axis. This can be done using the multi-function knobs under the "Dark Field" mode.

d. Click on "Tool" -> "Calibrate (A)" to store the calibration of the alpha tilt and complete the calibration of the double-tilt holder in the alpha direction.

e. Realign the zone axis by using the double-tilt holder. Similarly, complete the calibration of the beta tilt in the double-tilt holder to ensure accurate alignment of the zone axis.



Fig. 1. An experiment for calculation of tilting angles between a double-tilt holder and beam tilt with a Si crystal. (a) SAED pattern along [100] zone axis; (b) SAED pattern after tilting the crystal with the alpha tilt of double-tilt holder for 3° from the [100] zone axis. (c) SAED pattern after tilting the crystal with the belt tilt of double-tilt holder for 3° from the [100] zone axis.

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## 3. User Guide for SmartAxis Software

Fig. 2. Introduction to the SmartAxis Software Interface

The software primarily features three functions: Stage Tilt, Axis Tilt, and TEMstate. Each function is described below.

#### **1. Stage Tilt Function**

The Stage Tilt feature is primarily used for zone axis alignment. Utilizing the Beam Tilt functionality (available in Thermo Fisher Scientific's TEM instruments in TEM mode and can be activated through Dark Field), it tilts the incident electron beam. By adjusting the diffraction pattern to be symmetrical and then clicking on 'Stage Tilt Value', the calculated tilt angles for the dual-axis stage, alpha and beta, will automatically populate in the 'Stage\_A' and 'Stage\_B' fields. With the 'Axis Tilt' checkbox unchecked, clicking 'Apply' completes the crystal zone axis calibration.

### 2. Axis Tilt Function

After ticking the 'Axis Tilt' checkbox, click on 'Refresh Stage' to fetch the current alpha and beta angles of the dual-tilt stage, which will be filled into 'Start A' and 'Start B', representing the starting tilt angles. 'Axis Angle' indicates the angle at which the crystal needs to be rotated in reciprocal space. This function is used for tilting along a specified zone axis, in a specified direction using 'Beam Tilt'. When the 'Axis Tilt' function is enabled, pressing 'Apply' will tilt the stage in the specified direction of 'Beam Tilt' to the angle stored in 'Axis Angle'. The maximum tilt angle for the dual-tilt

stage is 35°; attempting to exceed this limit will result in a failed application.

### **3. TEMstate Function**

TEMstate allows users to capture the electron microscope's parameter settings at various states—from 'Get one' to 'Get Six'—storing temporary states such as Magnification, Defocus, Intensity, Mode, etc. Then, using 'Set one' through 'Set Six', these temporarily stored states can be applied for rapid state switching. Additionally, users have the option to specify which parameters to store and apply with checkboxes. Note that parameter capture and application are only available in the TEM mode of the microscope.

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