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EDT/Collect
for DigitalMicrograph

Data Collection for
Electron Diffraction Tomography

EDT/Collect Manual 1.0

HREM Research Inc.

Introduction

The EDT/Collect software has been developed by HREM Research Inc. in collaboration with the AnaliteX company (www.analitek.com).

The EDT/Collect DigitalMicrograph plugin allows the user to Collect beam-tilt Electron Diffraction Tomography 3D data sets.

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website: www.analitek.com

The proper reference to the data collection method as of January 2013 is

M. Gemmi and P. Oleynikov. *Scanning reciprocal space for solving unknown structures: energy filtered diffraction tomography and rotation diffraction tomography methods*. Zeitschrift für Kristallographie - Crystalline Materials: Vol. 228, No. 1, pp. 51-58 (2013).

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DigitalMicrograph is a trademark of Gatan Inc.

What does EDT/Collect program do?

Figure 1 illustrates how does **EDT/Collect DigitalMicrograph plugin** work in an experiment.

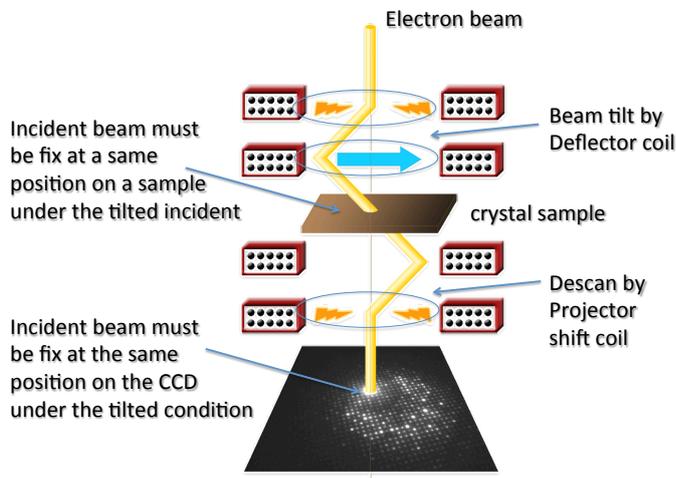


Fig. 1 Ray path diagram of EDT/Collect experiment

EDT/Collect program deflects the incident electron beam by changing deflector lens current, and the program takes a series of diffraction patterns with various diffraction conditions.

After the taking one tilt series of diffraction patterns, the program tilts the sample holder slightly. Then again the program starts to take a new tilt series of electron diffraction patterns.

By continuing this operation EDT/Collect program allows user to collect beam-tilt electron 3D diffraction tomography data, in a range say from -70 degrees to + 70 degrees, if a wide angle tilt specimen holder is used.

-In this operation the beam tilt direction by beam deflectors must be perpendicular to the sample holder tilt axis.

-Incident electron beam must illumine at the same and small position on a crystal sample during this experiment.

-Transmitted 000 electron beam must be fixed at a same position on a CCD camera.

Using EDT/Collect program these complicated experiments can be performed easily.

Technical References

- [1] P. Oleynikov. Automated Quantitative 3D Electron Diffraction Rotation Tomography. In: *Uniting Electron Crystallography and Powder Diffraction*, NATO Series B: Physics and Biophysics. 2012, p327.
- [2] Mayence A, Navarro J R G, MaY, Terasaki O, BergStrom L, Oleynikov P: Phase Identification and Structure Solution by Three-Dimensional Electron Diffraction Tomography: Gd-Phosphate Nanorods, *Inorg. Chem.*, 2014, **53**, 5067.
- [3] Sun Q, Ma Y, Wang N, Li X, Li D, Xu J, Deng F, Yoon K B, Oleynikov P, Terasaki O, Yu J: High performance nano sheet-like silicoaluminophosphate molecular sieves: synthesis, 3D EDT structural analysis and MTO catalytic studies, *J. Mater. Chem. A*, 2014, **2**, 17828.

Installing the program

EDT/Collect is available as a plugin to DigitalMicrograph of the Gatan microscopy Suite (GMS). As of 2016 the following GMS versions are supported:

- 32 bit: GMS 1.x and GMS 2.x ;
- 64 bit: GMS 2.x and GMS 3.x;

The EDT/Collect software contains only proprietary libraries such as, for example, Intel® MKL.

Software requirements

The following is a list of the software requirements necessary to run the EDT/Collect plug-in:

- DigitalMicrograph (GATAN™)
- USB Key Driver
- IPU Plug-in (Free-ware downloadable from www.hremresearch.com)
- Gatan EMControl Plug-in (a plug-in supported by Gatan to communicate with a microscope this must be installed beforehand)
- AnaliteX TEM Server (please consult the manual for the TEM server)

Software Installation

Installation of **EDT/Collect** is almost same to that of **EDT-test** program except for the license key. Please consult the manual of **EDT-test**. To examine that **EDT/Collect** program can work correctly on user's TEM, it is recommended to install the **EDT-test** at first, and please check that the program **EDT-test** works correctly. **EDT-test** is a free program. After the check by **EDT-test** program users can decide that they are going to use **EDT/Collect** or not. Once **EDT-test** program is on user's TEM, only the licence key is necessary to use **EDT/Collect**. Procedure below is the case that **EDT-test** is not in user's TEM.

Installing USB Key Driver

The user key driver should be installed by following the instructions given by the key driver installer. The key driver installer comes with EDT/Collect, or you can find it on our web site.

Installing EDT/Collect Plug-in

The plug-in can be installed by drag-and-drop copy to the folder "Plugins" (The Plugins folder should exist under a normal installation of the DigitalMicrograph.)

When the DigitalMicrograph is launched after placing the plug-ins into the Plugins folder, EDT/Collect menu commands will appear under "EDT/Collect" menu.

Installing IPU Plug-in

EDT/Collect uses some functions based on the Intel' MKL (Math Kernel Library) provided by the IPU plug-in. All the files relating the IPU plug-in can be installed by drag-and-drop copy. Please consult the ReadMe file that comes with the IPU plug-in.

Installing AnaliteX TEM Server

Please consult the installation manual for the AnaliteX TEM server. If you need any help to install/use the AnaliteX TEM Server, please contact to HREM research Inc.

Accessing the plugin from the DM menu

Each module is accessible through the main **DigitalMicrograph** menu, and EDT/Collect has a unique name that represents the content of the corresponding module as shown in Fig. 2.

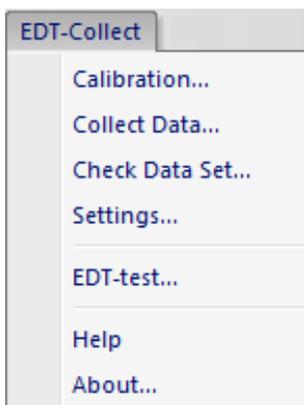


Fig.2 Pull-down menu of EDT/Collect in DigitalMicrograph.

The available commands through the main menu are:

Calibration... – the TEM beam tilt coils calibration;

Collect Data... – the main data collection module;

Check Data Set... – viewing the collected data after a series of experiment;

Settings... – changing the program settings (for service operators, not recommended to use);

EDT-test...– test program to check whether the EDT/Collect works correctly for user’s TEM or not.
This program part works without the USB license key;

Help – getting help on the plugin;

About... – displays dialog with the plugin information.

Plugin settings

After the installation of the EDT/Collect plugin in the **DigitalMicrograph** users should set up some parameters so that the plugin knows what CCD cameras can be used, the diffraction pattern scale bars (pixel size) for accessible camera lengths, available specimen holders and etc.

In order to access the plugin settings please choose the **Settings...** menu item from the *EDT/Collect* DM main menu. The dialog box shown in Fig. 3 will appear.

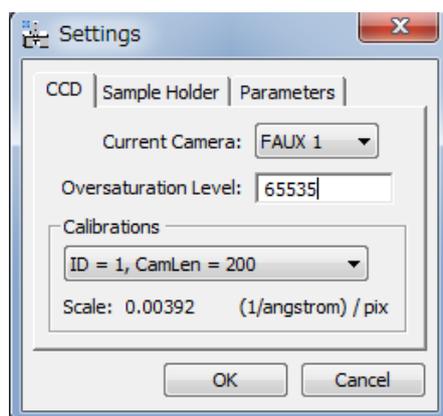


Fig. 3 Setting dialog box. The state that CCD tab is chosen.

CCD settings

The plugin will automatically identify all cameras available to **DigitalMicrograph** that can be accessed through the **Current camera** pull down list.

For each camera the user can set the **Oversaturation Level** (will be used by the EDT/Process software package to count oversaturated pixels).

Items in the pull-down menu and scale in the group box of Calibration are blank unless the TEM coils calibration is performed.

Sample holder Settings

This tab will help you to maintain the list of sample holders that can be used on your TEM. The users can add/remove (using **Add** and **Remove** buttons respectively) sample holders from the list:

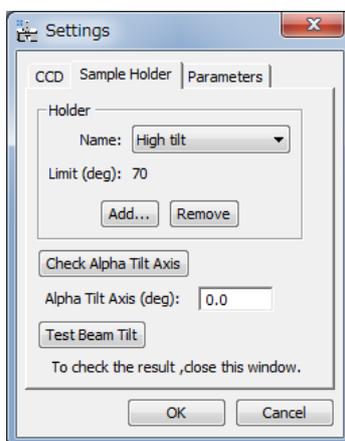


Fig. 4 Setting dialog box. The state that Sample Holder tab is chosen.

Currently selected sample holder will be removed from the list by pressing the **Remove** button. When pressing the **Add** button the following dialog will appear:

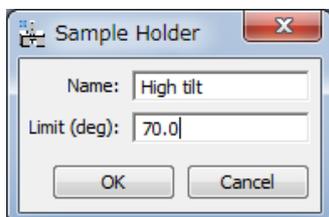


Fig. 5 Sample Holder dialog box to registrant a new holder

Here users can specify:

- name of the holder (**Name** edit box);
- tilting limit of the holder in degrees (**Limit** of tilt angle edit box);

Users must input the angle alpha in the "**Alpha Tilt Axis**" box in the Sample Holder Setting tab in Fig. 4. The alpha is the angle between y-direction, namely vertical direction, and the tilt axis of sample holder in user's camera screen. If users have already known the angle alpha user can input, otherwise the angle alpha must be estimated using "**Check Alpha Tilt Axis**". Detailed description on using "**Check Alpha Tilt Axis**" button will be described later.

"**Test Beam Tilt**" button is used to test whether the direction of beam tilt is set to be perpendicular to the sample holder tilt axis, or not. This button must be used after input of the angle alpha and

after the “**Tem coils calibration**” process. Detailed description on how to use “**Test Beam Tilt**” button will be described after the “**Tem coils calibration.**”

Setting parameters

The **Parameters** tab is used for switch of “**Invert Beam Tilt direction**”.

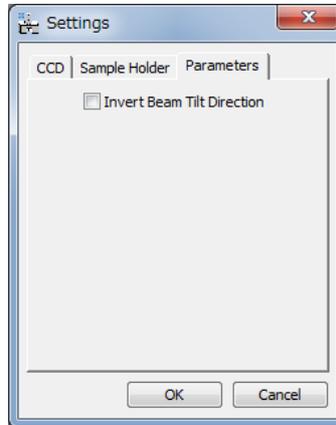


Fig. 6 Setting dialog box. Parameters tab is chosen.

If the direction of beam tilt is the same to that of the sample holder tilt in a user’s TEM, user need to put a check mark in the box of Invert beam tilt direction. User can see whether they are same or oposit by checking the collected experimental data. The procedure to see will be described in the section of “Check Data Set”.

TEM coils calibration

Since on some electron microscopes (for example JEOL) the changes of electron current for coils are done using internal units, the calibration of these coils is needed in order to:

- Convert the internal units into physical units (e.g. nm or degrees);
- Compensate the beam shift induces by one lens using the other.

The calibration of each coil is done in 2 dimensions: along X- and Y- axes of the corresponding coil. The calibration must be performed in the standard DIFF-mode or Nano Beam DIFF-mode. In case of the standard DIFF-mode insert the SA (Selected Area) aperture and focus the beam into a small spot. In case the Nano Beam DIFF-mode select the size and the brightness of the beam (same as will be used later for the data collection) and focus the beam into a small spot.

Please select the **Calibration** from the EDT/Collect pull-down menu in Fig. 2. The following dialog will be shown:

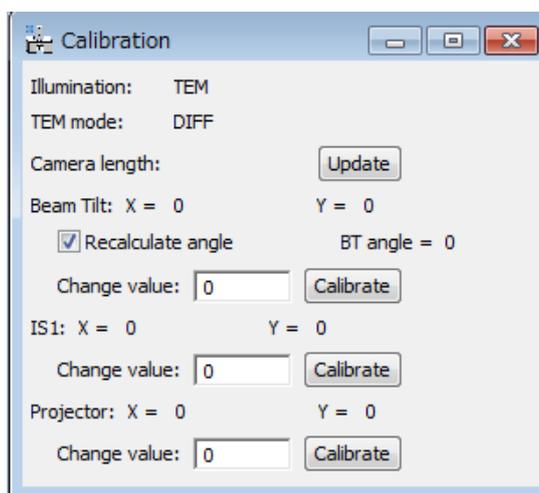


Fig. 7 Calibration dialog box.

For the calibration:

1. Set the objective lens current at the standard current position. This current is for the zero defocus position, otherwise for the eucentric specimen position.
2. In the image-mode the incident beam must be fixed at the central position even when the incident beam is tilted. To fix the beam position, adjust the beam tilt compensator or adjust the beam position using the beam tilt wobbler.
3. Select an empty area on your grid.
4. Insert a small SA aperture.
5. Spread the beam.
6. Switch into Diff-mode. Select an appropriate camera length; e.g. 300mm; and focus the incident electron beam into a small spot.

CAUTION! Be careful for the direct exposure of the CCD camera to the electron beam! Use minimum intensity beam (spread the beam using the Brightness knob of the JEOL TEM; use small spot size, for example 5; use small condenser aperture; insert small SA aperture in Image mode). Remove the CCD camera from the electron beam path when not in use to avoid unnecessary exposure.

7. Bring the incident spot to the centre of CCD camera.
8. Check the **Recalculate angle** on the Calibration dialog.
9. Set some value, or 0 in **Change value** box for the **Beam tilt**. An initial value for JEOL TEM can be 1000 units for the start.
Note: If 0 is set in the box, the **automatic** calibration will start. If a non-zero value is set, the **semi-automatic** calibration will start.
10. Press **Calibrate** button and wait while the calibration is finished.
11. Repeat the steps 9-10 for the calibration of the projector shift coils.

Calibration of IS1 coil is not necessary unless Nano Beam Diffraction mode is used.

Note: Once you have set the objective lens current, do not change it.

Note: Once you have chosen a camera length in calibration you should not change the length in the following test.

Note: If the beam goes out from the SA-aperture during the calibration, the calibration may fail. Please adjust the beam tilt compensator as mentioned above beforehand.

In case of **automatic calibration**, the calibration chart as shown in Fig. 8 will appear at the end. If in case of failure the chart is not linear. Please try **semi-automatic** calibration in case of failure.

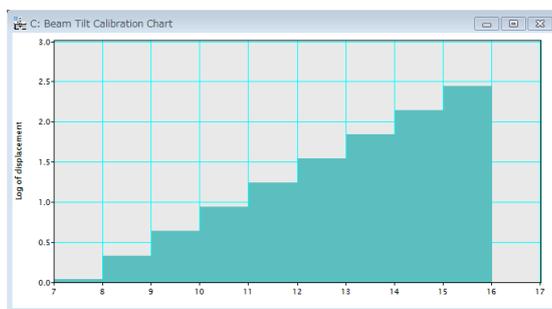


Fig. 8 A relation between a parameter for coil current, CV-value, and tilted electron beam position in log vs. log axis.

The calibration of the selected coil is done automatically according to the following steps, when you press **Calibrate** button. The calibration program

1. reads the user-supplied **Change value** from the panel (let's call it CV value);
2. reads current coil values for x- and y-axes (let's call them as X_0 and Y_0 values);
3. records the diffraction pattern (original pattern);
4. changes the x-axis value of the coil by the CV value (so it becomes X_0+CV);
5. records the diffraction pattern (X-modified pattern);

6. changes the x-axis back to the original X_0 value and changes y-axis value of the coil by the CV value (so it becomes Y_0+CV);
7. records the diffraction pattern (Y-modified pattern);
8. restores the coil y-axis value back to Y_0 ;
9. calculates the shifts of the spot using normalized cross-correlation;
10. if the shifts is small and the check box for Automatic Change value is selected, the CV value is automatically doubled and go to the step 4;
11. saves the calibration matrix (the direction of the coil x- and y-axes and the measured deviations along x- and y-axes).

The correct coil calibration images should look similar to the following:

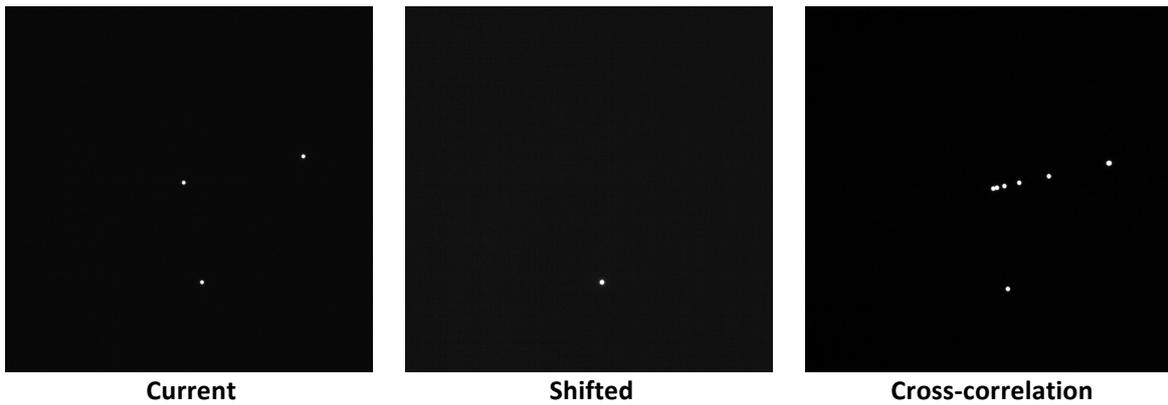


Fig. 9 Coil calibration images appear after the calibration process.

The **Current** pattern in Fig. 9 is a sum of 3 recorded diffraction patterns – the original and 2 with induced changes along x- and y-axes of the coil. The 3 spots seen in the **Current** pattern in Fig. 9 is explained in Fig. 10:

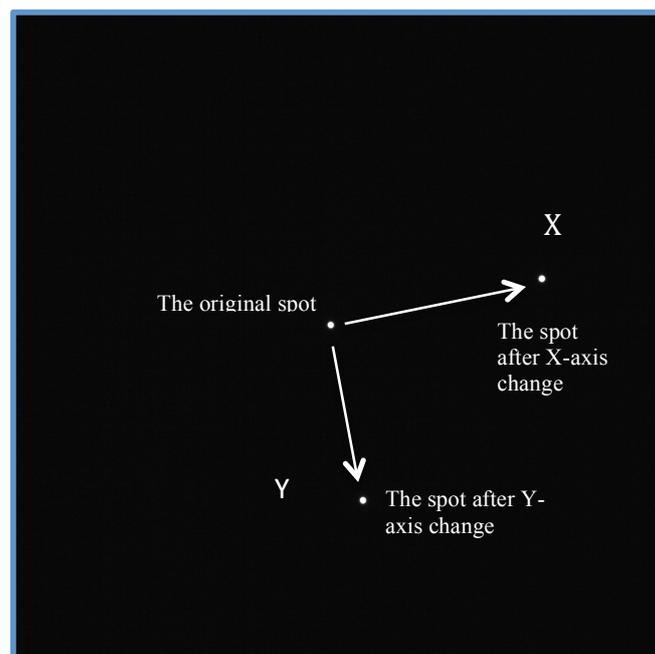


Fig. 10 Explanation on the Current pattern in Fig. 9.

NOTE: the calibration will be more precise if the 2 spots obtained from the induced changes of x- and y-axes of the coil are well separated (close to the edge of the CCD frame). These positions depend on the **Change value** for the corresponding lens. The x- and y- axes in this image are that for the beam tilt coil, but not for the coordinate in this CCD image.

The **Shifted** pattern in Fig. 9 shows the last pattern with the deviation of the y-axis of the coil.

The **Cross-correlation** pattern in Fig. 9 is the sum of 2 individual cross-correlation patterns: (1) the original pattern and the pattern where the x-axis has been changed; (2) the original pattern and the pattern where the y-axis has been changed. Since in each case the cross-correlation shows a single peak then the sum of the 2 cross-correlation functions must show **2 well-separated peaks**.

Note: If the calibration of a camera length for a selected CCD camera and for a selected nominal camera length has not been carried out beforehand for **DigitalMicrograph**, the warning message appears and TEM coil calibration stops. In such case please calibrate beforehand the camera length consulting the manual of **DigitalMicrograph**.

If the calibration of TEM coils is performed for a new camera length and for a new selected camera, as seen in the CCD setting dialog box, a new item with a new ID number appear in the pull down menu. Recalibrate for the same experimental condition will overwrite the data.

Check Alpha tilt axis and Beam Tilt test

1. Estimation of angle Alpha and set the angle

In Fig. 4 we have a box “ **Alpha tilt axis**” to input the angle Alpha. If user does not know the angle Alpha, it can be determined using “**Check Alpha Tilt Axis**”. By clicking this button the program takes one series of diffraction-patterns by changing the sample-holder-tilt angle. There may be some ways to estimate the angle alpha using this button. Two examples are described in **Appendix**. After the estimation of the angle alpha users must input the angle alpha into the box “**Alpha Tilt Axis**” in Fig.4.

2. Beam tilt test

After the input of the angle alpha, users should test whether the beam-tilt-direction is perpendicular to the sample-holder-tilt-axis, namely whether the beam-tilt-direction and the sample-holder-tilt-direction are parallel (or anti-parallel) to each other or not. “**Test Beam Tilt**” button in Fig. 4 is used to check these.

In Diff-mode without specimen in the beam path, by clicking “**Test Beam Tilt**” button, the incident beam is tilted without compensation by descan, namely by PL-shift. Users can chose the tilt range and tilt steps following the suggestion in dialog box. The results of the beam tilt test are recorded by CCD camera. The program crates one image stack. The image stack can be seen using **slice tool** of **DigitalMicrograph(DM)**. If users are not familiar with the **DM slice tool**, please consult to the manual of **DM**. Figure 11 is the result of summing up of the recorded pictures using the **DM slice tool**. It can be seen that the beams are tilted in the same distance, and along the same direction. It can be confirm that this beam-tilt-direction is perpendicular to the sample-holder-tilt-axis, namely parallel to the sample-holder-tilt-direction in this case. The procedure to obtain the sample-holder-tilt-direction is shown in Fig 16 and 17 in **Appendix**. The beam-tilt-direction is set to be parallel to the sample-holder-tilt-direction, so that it is concluded that the beam-tilt-direction is set correctly by the input of the estimated angle alpha.

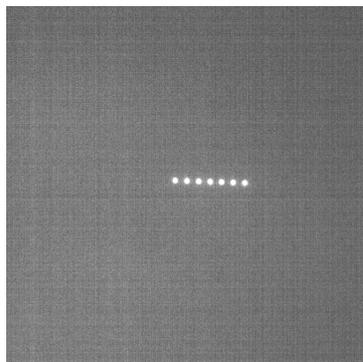


Fig. 11 Result of the beam tilt test.

Collecting 3D EDT data sets

NOTE: It is strongly recommended to run the TEM coils Calibration module before the data collection. The data collection of 3D Electron Diffraction Tomography data can be performed using the “**Collect Data...**” module from the main “EDT/Collect” menu. The Data Collection dialog box is shown in Fig. 12.

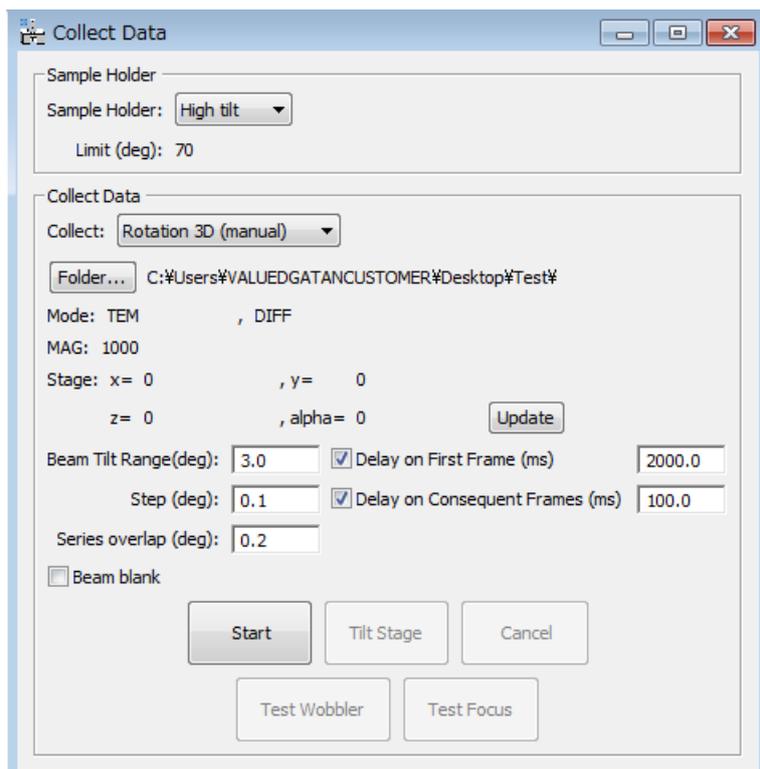


Fig. 12 Data collection dialog box that appears after choosing “**Collect Data...**” in the pull-down menu of EDT/Collect in Fig. 2.

The group box “**Sample Holder**” can be used to select the appropriate sample holder that is currently in use (see section “Sample holder settings” on how to manage a list of sample holders). It is required to choose the sample holder prior to the data collection. The actual limit of the tilt angles will be shown below the selected sample holder type (“**Limit (deg): 70**” on the figure above).

The group box “**Collect Data**” should be used for the data collection.

The settings that will be used for the data collection are:

- Collection type can be chosen from the **Collect** pull-down box (currently “**Rotation 3D (manual)**”).

- The folder where the acquired frames will be stored can be chosen using the browse button (“**Folder...**”). The selected folder will be shown next to the button, for example, (“**C:\Users\user_name\Desktop\Test**” on the figure). When the data folder is specified, the grey out **Start** button will become active.
- The **Beam tilt range** (in degrees) corresponds to the *full angle* that will be covered by the beam tilt of TEM. In case of 3° the beam tilt will vary from -1.5° to +1.5°. The **Beam tilt step** (in degrees) specifies the beam tilt step that will be used in order to cover the range of the beam tilt angles specified in **Beam tilt range**. In this example the program will change the beam tilt in the range from -1.5° to +1.5° (covering 3°) with a step of 0.1°, and automatically acquire 31 frames;
- The **Series overlap** (in degrees) defines the overlap between consequent goniometer tilts. This value is in order to compensate the mechanical imperfections of the stage. In ideal case the goniometer should tilt by the same value as the “**Beam tilt range**” (3° in this case). However, if the value of “**Series overlap**” is different from 0 then the real goniometer tilt will be “**Beam tilt range**” *minus* “**Series overlap**” ($3.0^\circ - 0.2^\circ = 2.8^\circ$ in our example). The “**Series overlap**” value can be kept as 0.2° for most data collections;
- The **Delay for the first Frame** will be applied to the first frame of the beam tilt series after the stage tilt. For the stage tilt the microscope mode is switched between DIFF and Imaging modes (or SAMAG mode in JEOL microscope), and this delay will be effective for some microscope to stabilize the lens settings.
- The **Delay on consequent frames** (milliseconds) and the **Beam blank** check box are used for some types of CCDs that are sensitive for the beam position changes. Some CCD cameras (such as Gatan ES500W) use a fluorescent screen so that there can be a trace left by the strong beam while it moves during the beam tilt or shift. It may decay slowly and can be noticed on the CCD. In order to compensate for these effects please check the **Beam blank** and put some delay (500-1000 ms should be generally enough) for the Consequent Frames.

NOTE: The collected data set must occupy an **individual folder**. Every new data collection must be pointed into a new folder otherwise all-previous data will be overwritten with the new dataset!

Data collection step-by-step

The data collection will start by pressing the **Start** button at the bottom of the panel.

The data collection contains the following steps:

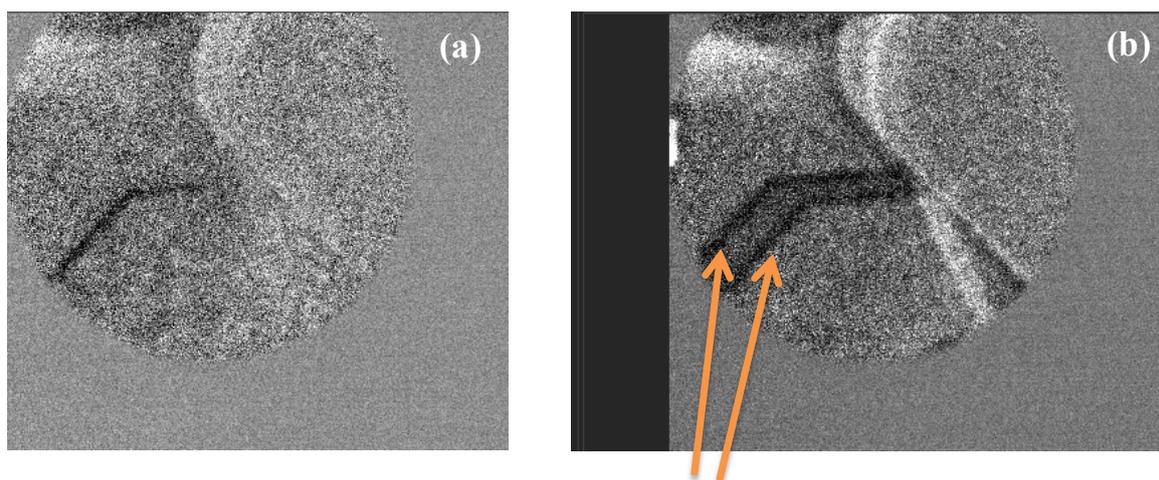
1. Check the sample position and height (using **Test Wobbler** buttons).
2. Collect the beam tilt data (using the **Collect Data** button).
3. Tilt the stage (using the **Tilt stage** button).
4. Manually track back and correct X-Y position, and correct the Z position using CCD camera (using **Test Wobbler**)
5. Pressing **Stop viewing** button and switch into the DIFF mode.
6. Go back to 2 and repeat the same procedure.
7. Finish the data collection by pressing the **Cancel** or **Finish** button.

1. Checking the sample height

There are two ways to check the crystal height in imaging mode (SAMAG mode): (i) the dynamic wobble of the beam by tilting and (ii) subtract two frames recorder with opposite beam tilts.

The procedure to use dynamic wobble is as follows. By Press the **Test Wobbler** button, the dynamic wobble test starts, this button is similar to the (JEOL) X-WOBBLE. In this case the data collection program induces 2 opposite beam tilts ($\pm 0.5^\circ$ in our case) and switches the CCD into the view mode for the observations. The correct sample height will be the one when the crystal relative movement will be minimum.

Figure 13 shows the subtraction procedure to correct the crystal height. The frame subtraction produces the following images in the case of Fig. 13a the correct sample height – notice the average gray level of the inner part of the SA aperture *with minimum* number of distinguishable features and in Fig. 13b the sample height is wrong – there are 2 shadows of the crystal corner.



Two shadows of the crystal due to the sample movement caused by the beam tilt

Fig. 13 Images with SA apertures for the correction of the sample height using subtraction procedure. **a**: correct sample height case. **b**: wrong height case.

By pressing the **Stop Viewer** button the view mode will stop, and the **Collect Data** button becomes active. The **Stop Viewer** button becomes **Tilt Stage**, but the **Tilt Stage** button becomes active only after the data collection process.

2. Collecting data

By pressing the **Collect Data** button, data will be collected automatically with the beam tilt series. All the frames will be saved to disk in the DM3/DM4 format. The typical status of the data collection is shown in Fig. 14 below:

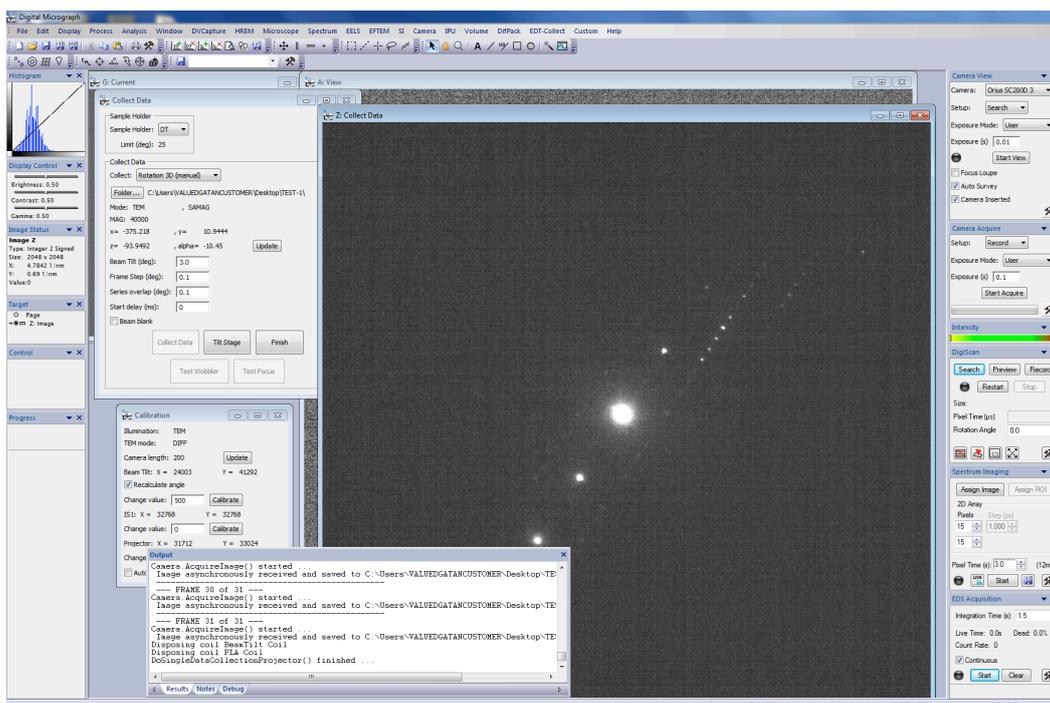


Fig. 14 Typical status of display during data collection.

NOTE: During the data collection of the beam tilt series all buttons (except **Cancel**) will be disabled. By pressing the **Cancel** button, you can stop the current data acquisition. After the one sequence of data collection, the **Cancel** button becomes **Finish** button. If users want to finish the data collection, press the **Finish** button. If users want to continue the data collection at an increased stage-angle of the specimen, please press the **Tilt Stage** button to increase the tilt angle of the specimen stage.

3. Tilting the stage

By pressing the **Tilt Stage** button the specimen stage will be tilted from the current stage-angle. As it was mentioned previously, the stage will be tilted by the angle that is calculated as the **Beam tilt angle** *minus* the **Series overlap**. In the case that is presented in this manual the stage tilt will be 2.8° ($3.0^\circ - 0.2^\circ$).

The **Tilt Stage** button becomes the **Cancel tilt** button during this tilting stage action. If users want to stop the stage tilting action, press the **Cancel Tilt** button.

When the **Tilt Stage** button is pressed, the data collection program will switch the CCD into the view mode for the crystal tracking. When the stage tilt action finishes, the **Cancel Tilt** button becomes **Stop Viewer**.

4. Correction of X, Y, Z position of specimen

After the specimen stage tilt, the specimen may move. Track back the specimen manually is a crucial step during the data collection. The user must move the crystal back so that it will occupy the appropriate position. The crystal tracking can be easily achieved with the X-Y stage control of the microscope. Then, the specimen height must be checked and corrected, by pressing the **Test wobbler** button. After the correction of Z, please press the **Stop wobbler** button.

5. Stop viewing mode

If X, Y, Z positions of the specimen are appropriate, please press the **Stop Viewer** button. Then, the view mode will stop, and the **Collect Data** button becomes active. Your TEM goes into DIFF mode automatically.

6. Repeating the Collect Data

Then, by clicking **Collect Data** button, we go back to the process “**2. Collecting data**”.

7. Finish/Cancel the data collection

The data collection starts from the stage tilt angle that users set initially. Repeating the data collection procedure will increase the stage tilt angle. Press the **Finish** or **Cancel** button at any time to finish or stop the data collection.

NOTE, in this dialog box various buttons will change as follows

- Start/Collect Data
- Tilt Stage/Cancel Tilt/Stop Viewer
- Cancel/Finish
- Test Wobbler/Stop Wobbler.

Check Data Set

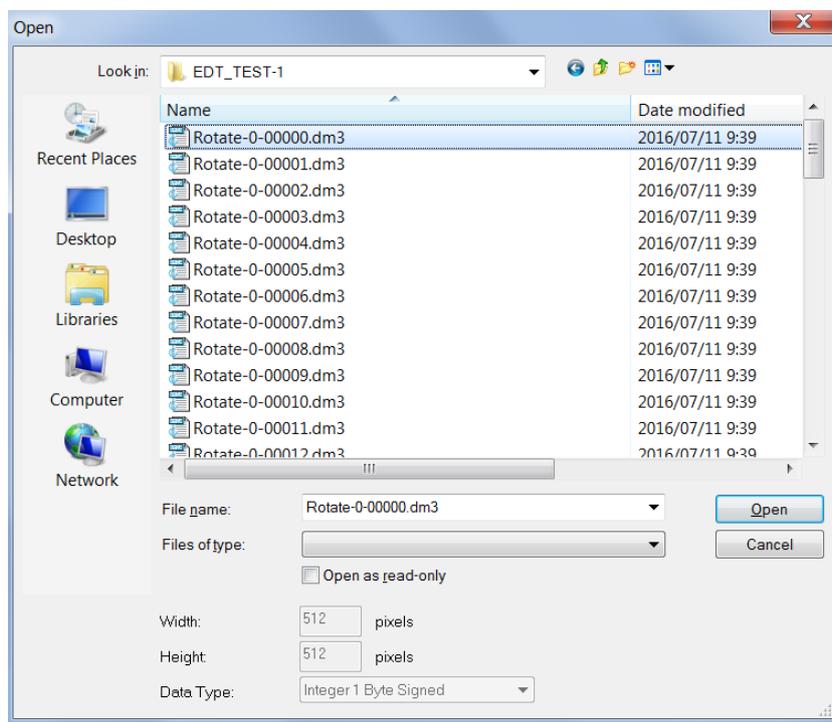


Fig. 15 Dialog box that appears by selecting “Check Data Set...” in the pull-down menu of EDT/Collect.

By selecting “Check Data Set” from the pull down menu, files selection menu window as seen in Fig. 15 will appear. The files were saved with names “Rotate-n-m.dm3/dm4”, n is an index for the specimen stage tilt, m is an index for beam tilt in one series.

If users select a single data in one series of the beam tilt, the program makes a data stack from all collected data in one series of beam tilt. The created data stack can be viewed by using **Slice tool** in **DM**.

If users select multiple data, the program makes a data stack from the selected data, and users can view through the data stack using Slice tool similarly. The selected data is sorted. This may be convenient to see the continuity of different series of collected data.

If the beam-tilt-direction is anti-parallel to the specimen-holder-tilt-direction, data at the end part of a series of beam tilt will continue to the beginning part of the next beam tilt series. If the beam-tilt-direction is parallel to the specimen-holder-tilt-direction, data at the beginning part of beam tilt series is similar to that at the end part of the next beam tilt series. In this case please check the box of invert beam tilt direction in parameter setting dialog seen in Fig. 6.

Data processing

All the recorded frames and the data collection log file will be stored in the selected folder for the current data collection. Do not change/erase/modify the files in the folder! The whole folder however can be moved to any other location.

The collected data set can be further processed with the data processing program **EDT/Process** from **AnaliteX**.

Bug reporting

There are log files created by the program during every run. These logs are stored in a special folder that depends on the Windows version.

Appendix

Estimation of angle Alpha using crossing points of Kikuchi-lines

The easy procedure to estimate the angle alpha is to use Kikuchi-lines. Set a relatively thick specimen to see kikuchi-lines. Please switch into Diff-mode, the camera length must be the same to that used in calibration. By clicking “**Check alpha Tilt Axis**” button in the Sample Holder dialog box seen in Fig. 4, the program takes one series of diffraction-patterns by changing the specimen stage tilt angle. Users can set the tilt range and tilt steps by following the suggestion in the dialog box which will appear after clicking “**Check alpha Tilt Axis**” button.

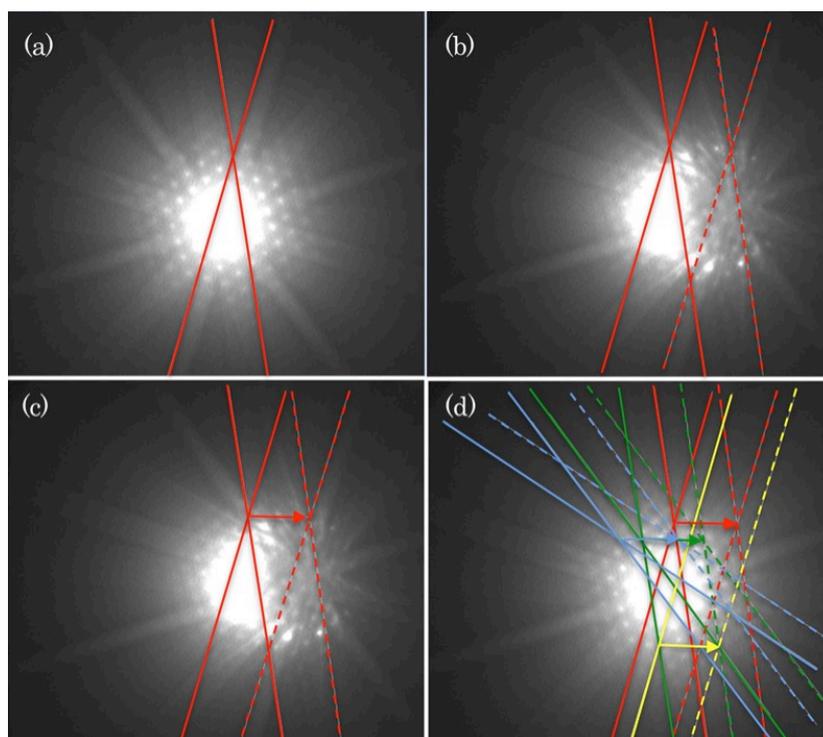


Fig. 16 a, b, c, d, diffraction-pattern from a crystal specimen at relatively thick position. **a** and **d** show the Kikuchi-pattern before the sample holder tilt, **b** and **c** show the pattern after the holder tilt.

You can save the set of diffraction patterns data with your convenient name and look through using **DM slice tool**.

Choose two Kikuchi-lines with a crossing in a diffraction pattern taken before the sample holder tilt. Red lines in Fig. 16a indicate the two Kikuchi-lines. Fig. 16b shows the result after the sample holder tilt by a few degrees. The movement of the Kikuchi-lines can be seen. The moved Kikuchi-lines are indicated by red broken lines. The moving direction of the crossing point is shown by a red arrow in Fig. 16c. The direction can be measured using **DM line tool**.

You can find many crossing points of Kikuchi-lines move towards the same direction, so that you may measure the direction from many crossing points as shown in Fig. 16d. Different color lines indicate different Kikuchi-lines with different crossings. The moving direction can be obtained as an averaged result. The moving direction can be the sample-holder-tilt-direction.

Sample holder tilt axis is determined as a perpendicular to the sample-holder-tilt-direction. The alpha is defined from the y-axis direction to the holder tilt axis in the counter clockwise rotation in the CCD camera images, thus the alpha is obtained as $+178.1 \pm 1.0$ degrees in the Fig. 16 case. Tolerance within ± 2 degrees for the measured alpha is accurate enough.

Estimation of angle Alpha using Laue circles

If users do not have a specimen with clear Kikuchi-lines, users can measure the angle alpha by observing Laue-circles. If users tilt a specimen slightly from an exact zone axis direction, users can see an excitation of Bragg reflections with circular form as seen below Fig 17a. This circle, Laue circle, appears at intersection between Ewald sphere and the zeroth order Laue zone plane. Fig. 17a shows a state that sample holder is tilted slightly towards (-) direction from an exact zone axis. Laue circle "A" can be seen. Fig. 17b is taken under a condition slightly tilted towards (+) direction. Laue circle "B" can be seen.

Fig 17c indicates Laue circles "A" and "B" on the same picture. If users determine centres of circles, users can determine the moving direction of the centres. A red arrow in Fig. 17c indicates the moving direction. This moving direction is sample holder-tilt direction, and it is perpendicular to the sample holder tilt axis. A specimen with a large unit-cell, so that user can see a clear Laue circle because of many Bragg reflections, is preferable for this estimation.

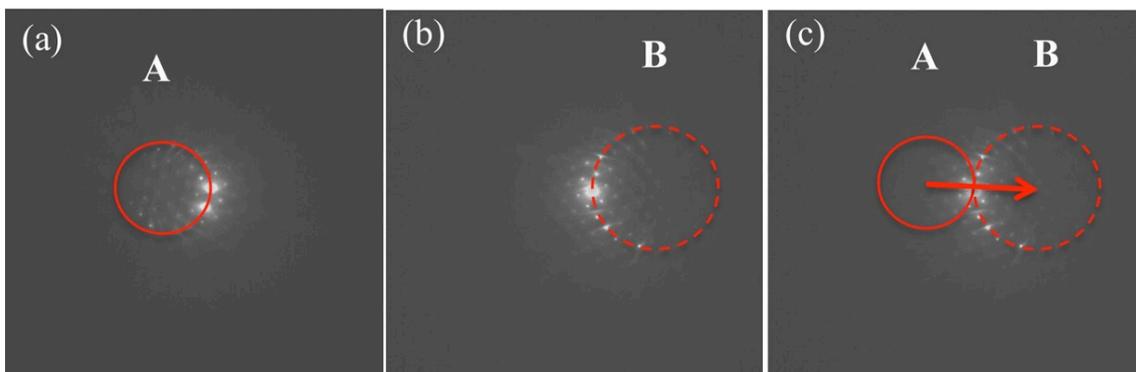


Fig. 17 a: Sample holder is slightly tilted towards the (-) direction from an exact zone axis. Laue circle "A" can be seen. **b:** Sample holder is tilted towards the (+) direction slightly. Laue circle "B" is seen. **c:** indicates Laue circles "A" and "B" on a same picture.

Please input the estimated angle alpha at the "Alpha Tilt Angle" in the Sample Holder dialog box in Fig. 4.

Relations between sample-holder-tilt-axis and sample-holder-tilt-direction in a CCD camera image

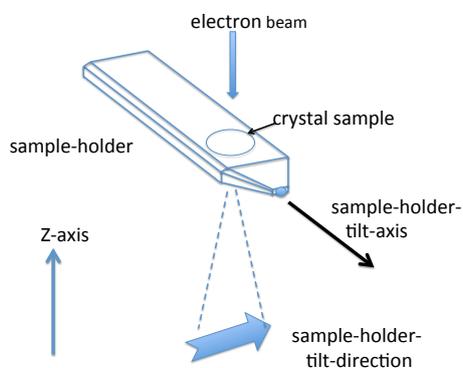
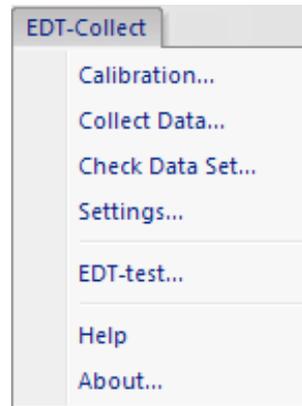


Fig. 18 Relation between sample-holder-tilt-axis and sample-holder-tilt-direction

Figure 18 illustrates a tip of sample holder and it shows the relation between sample-holder-tilt-axis and sample-holder-tilt-direction. Sample-holder-tilt-direction is the direction that Kikuchi-patterns and Laue-circles move along by the tilt of sample holder. Sample-holder-tilt-axis is the axis that the holder moves respecting to. Sample-holder-tilt axis and sample-holder-tilt-direction are perpendicular to each others. In an actual CCD image these two axes are seen to be rotated in some angles respecting to z-axis, though these two axes are still perpendicular to each others.

Quick Reference Guide

The EDT-Collect Main Menu



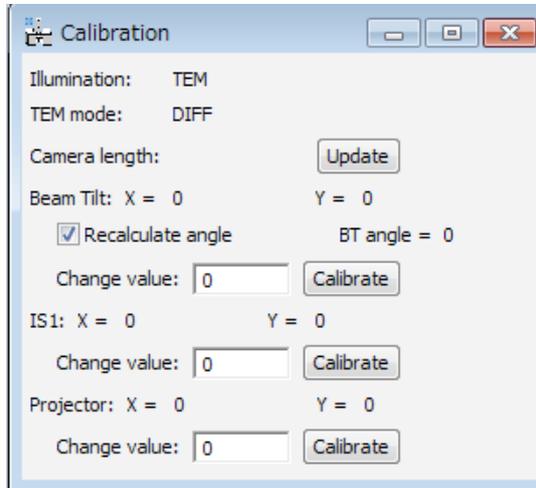
The commands in the EDT-Collect menu are described below.

Command	Description
Calibration... (see Dialog)	Opens the dialog for calibration.
Collect Data... (see Dialog)	Opens the dialog for data collection.
Settings... (see sub menus)	Opens the dialog for parameter setup.
TED-TEST	Test program to check whether the EDT/Collect works for user's TEM
Help	Opens Help document
About...	Opens About dialog

Calibration Dialog

The components of the dialog are described below.

Dialog

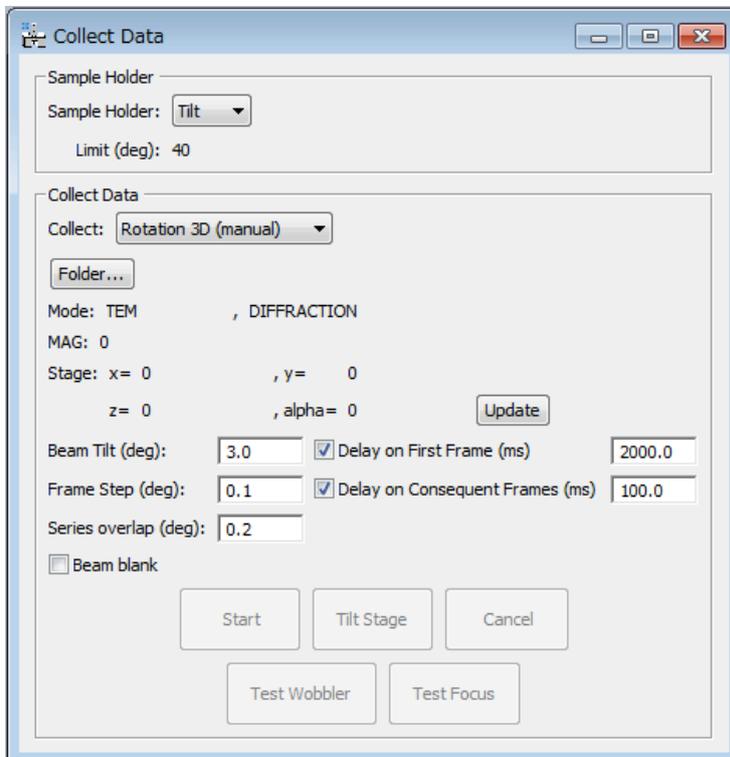


Component	Description
Illumination	Displays the current illumination mode
TEM Mode	Displays the current imaging/diffraction mode
Camera length	Displays the current camera length (or magnification)
Update	Updates above three parameters
Beam Tilt	Displays the current beam in DAC
Recalculate angle	When checked, the beam tilt angle for the “Change value” is displayed at “BT angle” as a mean value for x and y-tilts. The CCD should be calibrated beforehand.
Change value	Suggested DAC value to shift the beam (zero (0) means an automatic search for this value)
Calibrate	Executes a calibration
IS1	Displays the current image shift 1 in DAC
Change value	Suggested DAC value to shift the beam (zero (0) means an automatic search for this value)
Calibrate	Executes a calibration
Projector	Displays the current projector alignment (PLA) angles
Change value	Suggested DAC value to shift the beam (zero (0) means an automatic search for this value)
Calibrate	Executes a calibration

Collect Data Dialog

The components of the dialog are described below.

Dialog



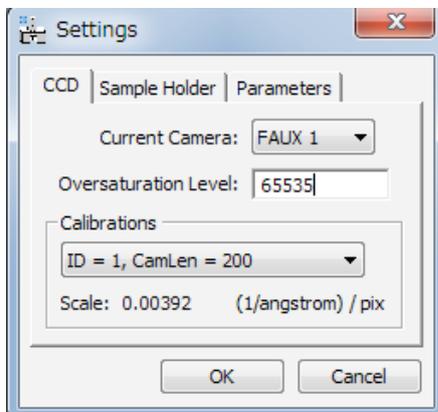
Component	Description
Sample Holder	
Sample Holder	Sample holder selector
Limit	Displays the maximum tilt angle
Collect Data	
Collect	Data collection mode selector
Folder	Browse for a folder to save the data
Mode	Displays the current microscope modes
Mag	Displays the current mag/camera length
Stage	Displays the current stage position
Update	Updates the microscope and stage parameters
Beam Tilt (deg)	Maximum rotation angle using the beam tilt
Frame Step (deg)	Rotation step for the beam tilt series
Series overlap (deg)	Overlap rotation angle between two series of the beam tilt frames.
Delay on First Frame (ms)	If checked, the specified delay is applied for the first frame of the beam tilt series.
Delay on Consequent Frames (ms)	If checked, the specified delay is applied for the consequent frames of the beam tilt series.
Beam blank	When checked, the beam will be blanked between exposure frames.

Data Collection Buttons	
Start / Collect Data	Starts the whole data collection or the frame series.
Tilt Stage	Starts manual stage rotation. The View window will open.
Cancel / Finish / Stop Viewer	Cancel or Finishes data collection, or stops the Viewer.
Test Wobbler / Stop Wobbler	Starts or Stops wobbling the beam wobbler for sample height adjustment.
Test Focus	Not-implemented.

Settings Dialog

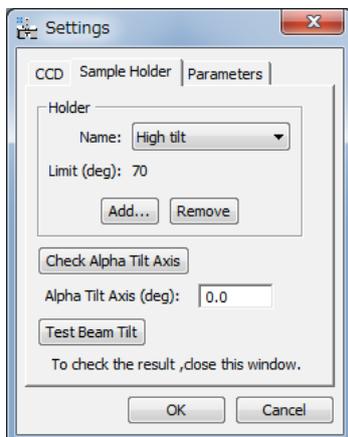
The components of the dialog are described below.

CCD Tab



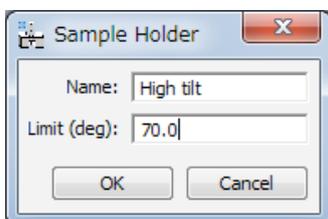
Component	Description
Current Camera	Camera selection
Oversaturation Level	CCD saturation level
Calibrations	
Calibration ID etc.	Calibration selector
Scale	CCD pixel calibration for this mode

Sample Holder Tab



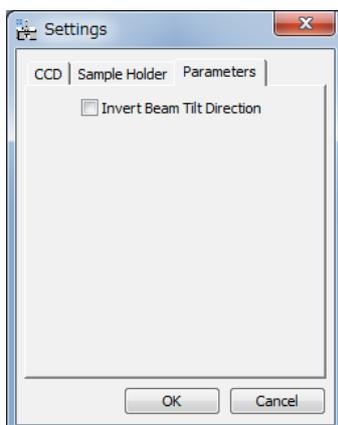
Component	Description
Sample Holder	Sample holder selection
Limit	Maximum tilt angle
Add...	Opens a dialog to add the stage information
Remove	Removes the stage entry
Check Alpha Tilt Axis	For experimental measurement of the angle Alpha
Alpha Tilt Axis (deg)	Angle between an axis of tilt stage for specimen and Y-direction in CCD
Test Beam Tilt	Use to check beam tilt direction

Sample Holder Add Dialog



Component	Description
Name	Stage name
Limit (deg)	Maximum tilt angle

Parameters Tab



Component	Description
Invert Beam Tilt Direction	If checked, the beam tilt direction of the frame series is inverted during the data processing. This parameter has no effect on data acquisition.