User Manual for EDT-test V.1.0 Jan. 2017

HREM Research Inc.

1. Introduction:

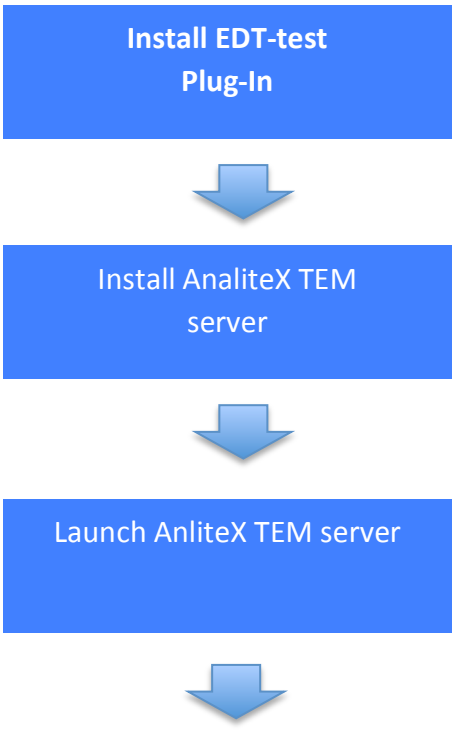
The **EDT/Collect Plug-In** for **DigitalMicrograph** (**DM**) has the routine, **EDT-test**, which is used for testing whether a user's TEM works appropriately under the control of **EDT/Collect Plug-In**. **EDT-test** works without a license key, so it is free program. The program allows users to perform following tasks:

1. To examine whether the program can control deflectors of the user's TEM.
2. To examine whether the program can control tilt action of a user's specimen holder.
3. To examine whether the program can control TEM-modes, such as to change from Diff-mode to Imaging-mode automatically in the user's TEM.

After the results of using **EDT-test**, users can decide whether the user's TEM is appropriate to install the **EDT/Collect Plug-in** program, or not.

2. Work flow:

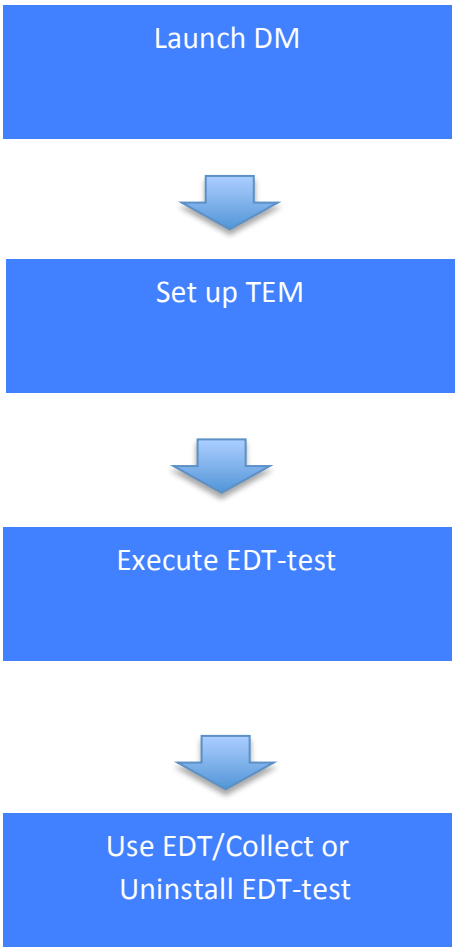
The action that users will take with this manual is summarized below. Details of these actions are described in the following sections.



Download **EDT/Collect Plug-In**. The program includes **EDT-test** and related programs. All those programs must be copied to the folder "PlugIns" of **DM**.
See the section 3.1.

EDT/Collect Plug-In includes the folder of AnaliteX TEMserver. The TEMserver folder must be put under the folder of "USERNAME", and the configuration file must be edited.
See the section 3.2.

Before the launch of the **EDT-test**, users must launch the **TEMserver**.
See the section 3.3.



Then, launch the **DM**, and chose **EDT-test** from the menu list of **EDT/Collect**.
See the section 3.4.

To execute **EDT-test** program, set up procedure of the TEM is described.
See the section 4.

The program calibrates beam defectors. User can examine whether the deflectors of the TEM, compensation of beam shift, tilt of user's specimen stage holders, and TEM-mode change can be controlled or not using **EDT-test** program.
See the section 5.

Users can decide whether they will use **EDT/Collect** Plug-In program, or uninstall the program from the user's TEM. Also Users can consult to HREM research Inc. See the section 6.

3. Software Setup

3.1 Install for EDT/Collect Plug-In:

EDT-test is included in **EDT/Collect Plug-In** program. **EDT-test** works without a license USB key-driver. The installation of **EDT-test** is the same as that of **EDT/Collect** except for the license key-driver.

EDT/Collect plug-in program 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-test**:

- **DigitalMicrograph (**GATAN™)
- **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** (This program is included in the folder in **EDT/Collect plug-In**)

Install of the EDT/Collect Plug-in

The **plug-in** program can be installed by drag-and-drop copy to the folder "PlugIns". The PlugIns folder should exist under a normal installation of the **DM**.

Installing IPU Plug-in

EDT-test uses some functions based on the Intel's 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**.

3.2 Install AnaliteX TEMServer:

EDT-test controls a user's TEM mainly through **Gatan EMControl plug-in**. However, some functions that are not implemented in the **EMControl** will be used through **AnaliteX TEM server**. Therefore, **AnaliteX TEMserver** must be installed and launched beforehand.

AnaliteX TEMserver program and installation manual are in a folder in the package of **EDT/Collect Plug-in**. Please read carefully details of the installation manual. Here, only the outline of Install and launch of the **AnaliteX TEMserver** is described.

AnaliteX TEMserver requires **Microsoft .NET Framework 4 or 4.5**. If user's TEM does not have this program please install beforehand from the internet site. **AnaliteX TEMserver** and related files must be placed in a folder under "USERNAME" as described in the manual of the **TEM server**.

3.3 Launch AnaliteX TEMserver:

At the first launch of **AnaliteX TEMserver,** the double clicking of AnaliteX.TEM.exe, an empty and new file is created. Then, please close **AnaliteX TEMserver**. The config file "AnaliteX.TEM.Server. exe.cofig " can be obtained by copy from a file in the folder of AnaliteX TEMserver. The config file must be edited depending on the type of user's TEM by consulting the installation manual of AnaliteX TEMserver. Put the config file in the newly created file.

Then, launch the **AnaliteX TEMserver** again. Please check that the **TEMserver** works properly consulting the installation manual.

3.4 Launch DigitalMicrograph:

When the **DM** is launched, **DM** gives warning "no license key for **EDT/Collect** ". However, user can ignore this warning; since only the subroutine **EDT-test** works without license key.

The command "**EDT-test**" will appear in the **EDT/Collect** menu bar after successful installation as shown in Fig. 1.

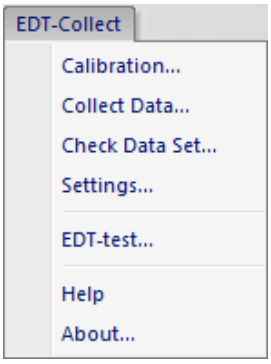


Fig.1 Pull-down menu of EDT/Collect

4. Set up TEM:

In the **EDT-test** program, the program tilts the incident electron beam at the specimen position and compensates the shift of diffraction patterns caused by the tilt of the beam by using PL-shift. In order to do this experiment users must set up their TEM to fixed the electron beam at a specimen position even after the incident beam is tilted.

1. Set the objective lens current at the standard current position. This current is for the zero deforcus position, otherwise for the eucentric specimen position.
2. In the Image-mode the incident beam must be fixed at a same position even when the bema is tilted. To fix the beam position, adjust the beam tilt compensator or adjust the beam position using the beam tilt wobbler.
3. Insert a small SA (selected area) aperture and spread the beam in the Image-mode.
4. 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 in the case of JEOL TEM; use small spot size, for example 5; use small condenser aperture; insert small SA aperture in Imaging-mode).

5. Bring the spot to the center of CCD camera.
6. Set the exposure time in the DM control panel, considering the dynamical range of the CCD camera. If the exposure time is not enough please increase later.
7. When the CCD camera becomes ready for the following tests, remove the CCD camera from the electron beam path for a while to avoid unnecessary exposure.

   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: In this set up we need not to use a specimen.

5. Excecute EDT-test:

5-1. Start up of EDT-test

Clicking "**EDT-test**" command in Fig. 1 will open the following graphical user interface (GUI) shown in Fig. 2. Users can chose following items; Calibrate deflectors, Acquire a data set, Test stage tilt, Reset deflectors, and Change TEM mode.

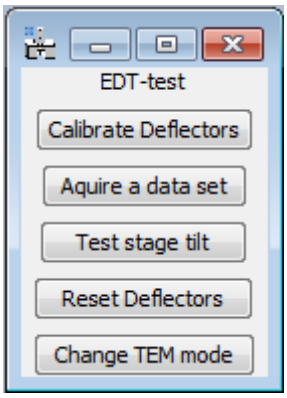


Fig.2 EDT-test GUI with five buttons.

Note: If the communication with TEM server is failed, the message shown in below will appear. In such case, click "OK" button and close **DM**. Please check carefully terms in this message box. If users find any mistakes, please correct and reset them! Then, launch the **TEMserver**, and then **DM**!

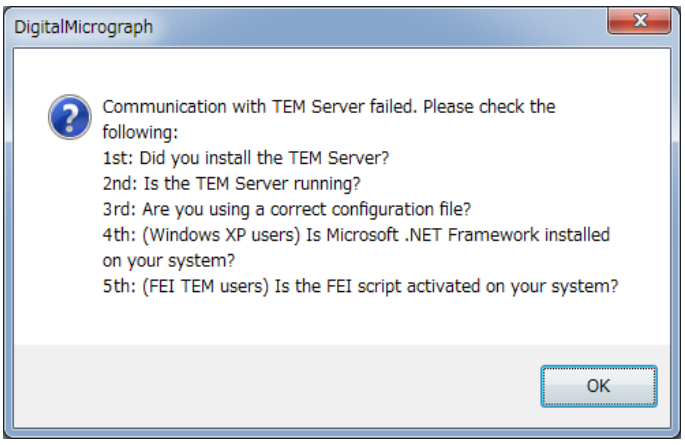


Fig. 3 message in case of failure of communication.

5-2. Deflector Calibration

By pressing the "**Calibrate deflectors**" button in Fig. 2 a new dialog box will appear. Following the message in the dialog box users can perform the calibrations on TEM deflector coils automatically. At the end of calibration the calibration chart shown in Fig. 4 will appear. If the graph in the chart is not linear, the calibration may be unsuccessful. In such a case please re-check the set up conditions in section 4.

CAUTION! Be careful for the direct exposure of the CCD camera to the electron beam! Use minimum intensity beam.

Note: If the beam goes out from the SA-aperture during the calibration, the calibration may fail. Please adjust so that the beam is fixed at the SA-aperture position and spread the beam as mentioned in section 4.

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

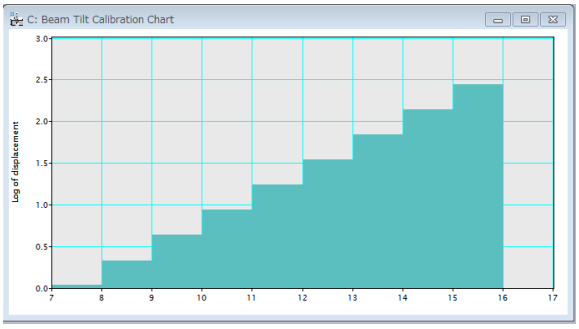


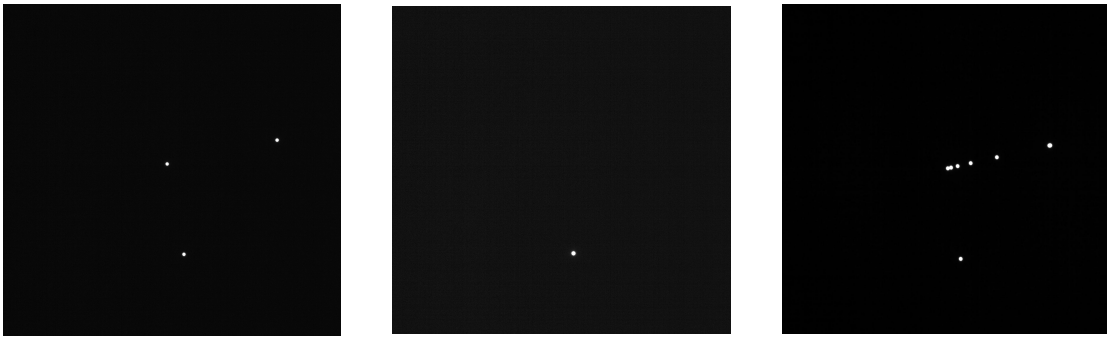
Fig. 4 Calibration chart indicating the relation between an internal parameter to control deflector coil current and tilted electron beam position in CCD images. The chart is on log vs. log.

In this calibration process the program find the relations between internal parameters that control electron beam deflector current and actual beam tilt angle. The beam tilt angle is automatically estimated from the beam position in a CCD image. The calibrations are performed for beam tilt coils x and y, then also for PL-shift x and y to compensate the beam shift which is cased by the incident beam tilt.

The automatic process that is carried out during the calibration is as follows. The program

1. reads internal parameters for deflector coil current for x- and y-axes (let's call them as X_0 and Y_0);
2. records the incident beam position (original pattern);
3. changes the internal parameter for x-axis of the coil (so it becomes X_0+*CV, CV* is changed value for internal parameter);
4. records the incident beam position (X-modified pattern);
5. changes the x-axis back to the original X_0 value and changes y-axis value of the coil by the *CV* (so it becomes Y_0+*CV*);
6. records the incident beam position (Y-modified pattern);
7. restores the coil for y-axis back to Y_0;
8. calculates the shifts of the spot using normalized cross-correlation;
9. if the shifts is small, the CV value is automatically doubled and go to the step 3;
10. saves the calibration matrix (the direction of the coil x- and y-axes and the measured deviations along x- and y-axes).

The images appear after the calibration should look similar to Fig. 5:



Current **Shifted** **Cross-correlation**

Fig. 5 Images appear after the calibration process.

The **Current** pattern in Fig. 5 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 on the **Current** pattern can be interpreted as shown on the Fig. 6:

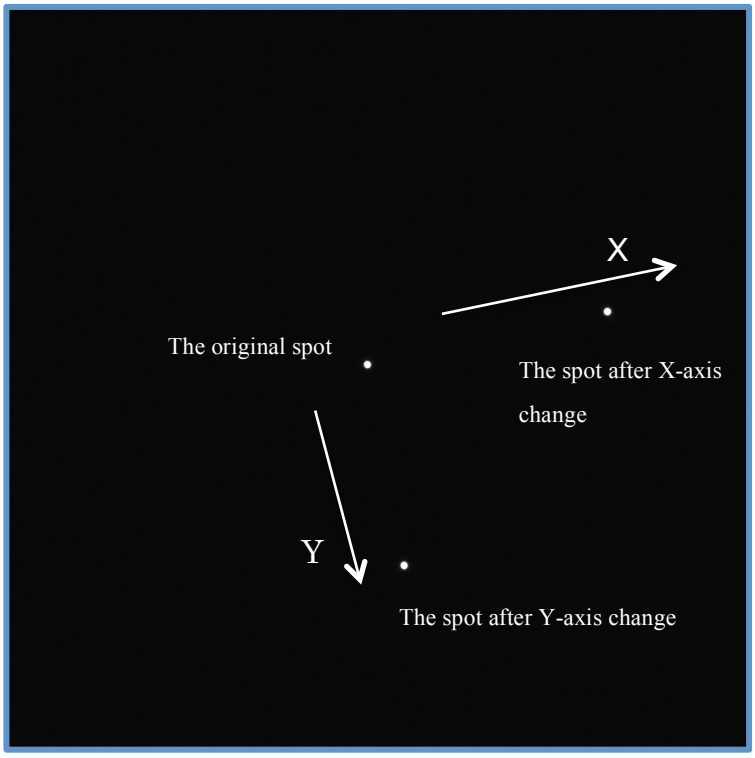


Fig. 6 Explanation on the **Current** pattern in Fig. 5.

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 **Shifted** pattern in Fig. 5 shows the last pattern with the deviation of the y-axis of the coil.

The **Cross-correlation** pattern in Fig. 5 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***.

5-3. Acquire data set

Users can test the beam deflector and the compensation of beam shift using "**Acquire a data set"** in Fig. 2. By clicking this button the program makes two data stacks which consist of seven pictures obtained by changing the incident beam direction at -1.2°, -0.8°, -0.4° 0.0°, +0.4°, +0.8° +1.2°.

One of the data set is taken by only the beam tilt without compensation by descan. You can see these pictures using the **slice tool** of **DigitalMicorgraph (DM).** (If you are not familir with **DM slice tool** please consult the manual of **DM**.) Seven spots must be located with the same distance and the same direction. Users can check the tilt direction and the tilt angle by measuring the distance between the spots. Fig. 7a shows the summing of the seven spots using the **slice tool**, and the image shows that the beam tilt is performed correctly.

> **Note**: Accurate beam tilt angle between two spots can be measured by comparison with a specimen that shows diffraction spots with known Bragg angles.

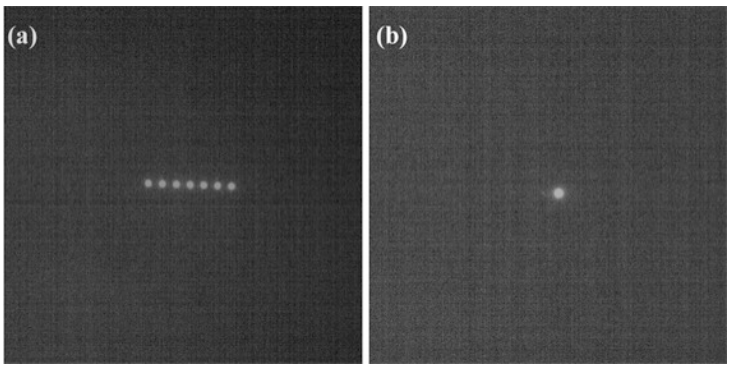


Fig. 7 (a) and (b) An image by summing up of images in a data set without compensation and with compensation by PL-shift, namely descan.

Another data set was taken by the beam tilt with compensation of the beam shift so called descan. You will see that the incident beam spot is fixed at the center in these 7 pictures. Fig. 7b shows the summing up images with compensation by PL-shift. Fig.7b shows that the descan is performed correctly

If the results are not successful, the calibration in section 5-2 may not be performed correctly.

5.4 Test of stage tilt

By clicking "**Test stage tilt**" in Fig. 2 users can check whether tilt of a specimen holder can be controlled properly by this program. Please select an appropriate single-tilt ***specimen holder***.

By clicking this button the specimen holder tilts from the present stage angle successively by +0.25°, +0.5°, +1.0°, +2.0° (total 3.75°). The current angle, intended angle and actual angle are shown in the results window. Users can check that the move of specimen stage by their eye.

5.5 Reset deflectors

By choosing "**Reset"** button in Fig. 2, the deflectors are reset to the state before the start of this test program.

5.6 Test of TEM-mode Change

Users can check whether the EDT-test program can change the TEM-mode automatically for the TEM. By clicking "**Change TEM-mode**" button in Fig. 2 a dialog box appears. Following the dialog message, users must switch into Diff-mode by manual at first. Then please follow the message in the dialog box to check whether the EDT-test program can change the TEM-mode automatically from Diff-mode to Imaging-mode (SAMAG-mode in JEOL microscope), and then from Imaging-mode (SAMAG) to Diff-mode.

> **NOTE:** Even when the automatic mode change is failed, **EDT/Collect** program can be used for experiments by manual change of the TEM mode. Please send the log-file to support at HREM research Inc. in case of failure. We hope we can update the **EDT/Collect** for the user's microscope.

6. Use EDT/Collect or uninstall EDT-test:

If users decide to use the whole part of **EDT/Collect program**, the license key is required. If any trouble, please consult HREM research Inc.!

If users want to uninstall all programs related to EDT-test, delete the **EDT/Collect** folder that users put in the "**PlugIns**" folder of **DM**, and delete also **AnaliteX TEMserver** folder that users put under the "USERNAME", also delete the folder that was created for the config file for **AnaliteX TEMserver**.