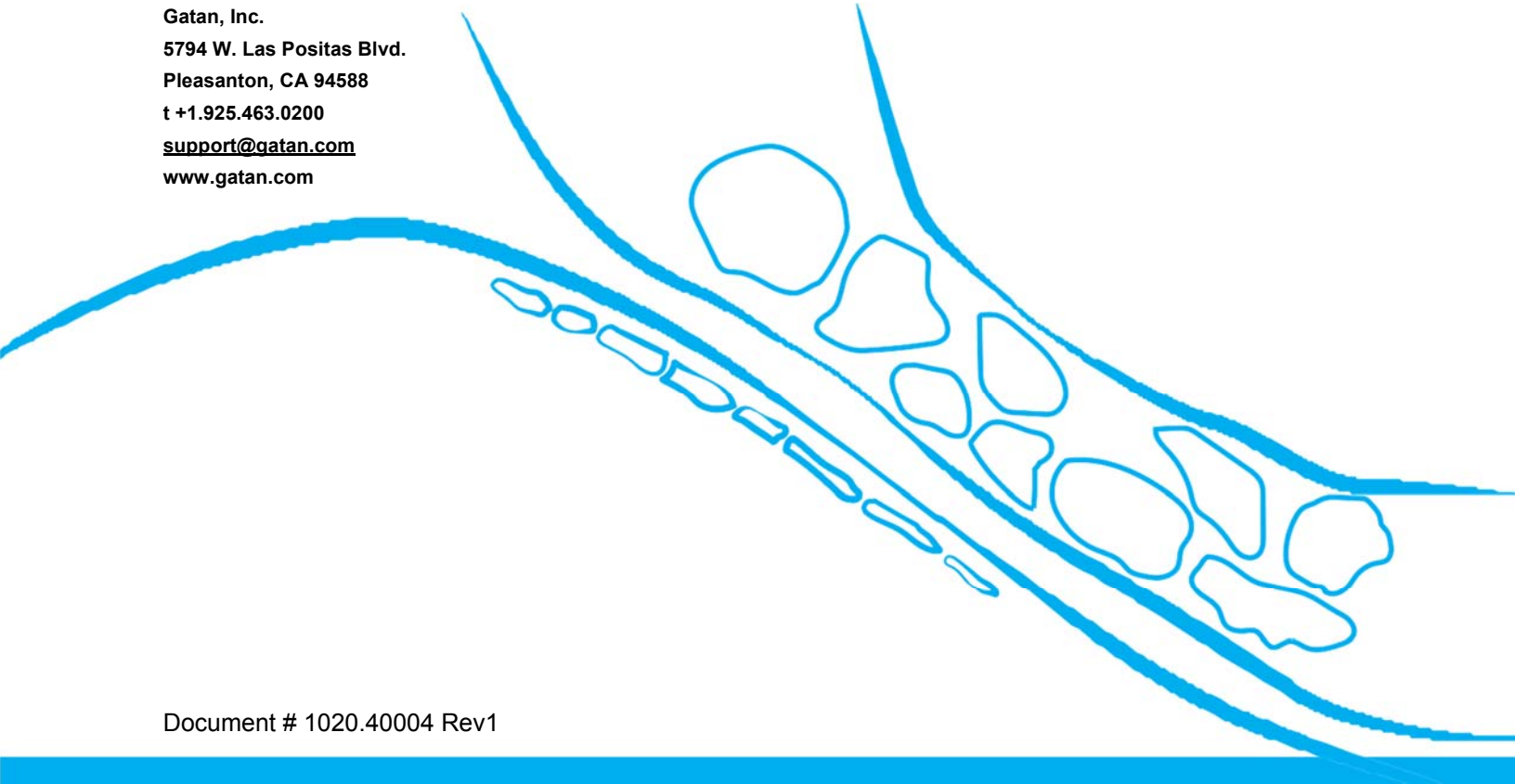
# STEMx System

## Model 1020 User Manual



**Gatan, Inc.**
**5794 W. Las Positas Blvd.**
**Pleasanton, CA 94588**
**t +1.925.463.0200**
**support@gatan.com**
**www.gatan.com**



Document # 1020.40004 Rev1

**Publisher**
Gatan Inc.

**Technical editors**
Gatan Imaging Team

**Document version**
Rev 1

www.gatan.com

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# 1 Introduction

Before installing and operating this product, and to avoid the risk of injury and potential hazards, read and review the Regulatory Pamphlet and follow all safety instructions.

## 1.1 STEMx system

STEMx™ system offers 4D scanning transmission electron microscope (STEM) diffraction data collection through hardware synchronization of DigiScan™ beam control system with Gatan's *in-situ* cameras (K2® IS and OneView® IS).


***Figure 1: STEMx system box.***

### 1.1.1 Advantages

- High speed data acquisition for detail rich mapping using direct hardware communication

- Artifacts associated with specimen damage and system drift are minimized due to the shorter duration of each experiment.
- All current camera functions are supported: STEMx system is an additional option for K2 IS and OneView IS cameras, so all current available features on these cameras are also available after STEMx system is added
- Simultaneously acquire multiple signals produced in the electron microscope as part of a spectrum image
- Easy to use interface: 4D datasets are collected, stored, and processed in a single application (Gatan Microscopy Suite® (GMS) software)

## 1.2 Gatan Microscopy Suite interface

Launching GMS 3 brings up the following screen, shown in Figure 2.

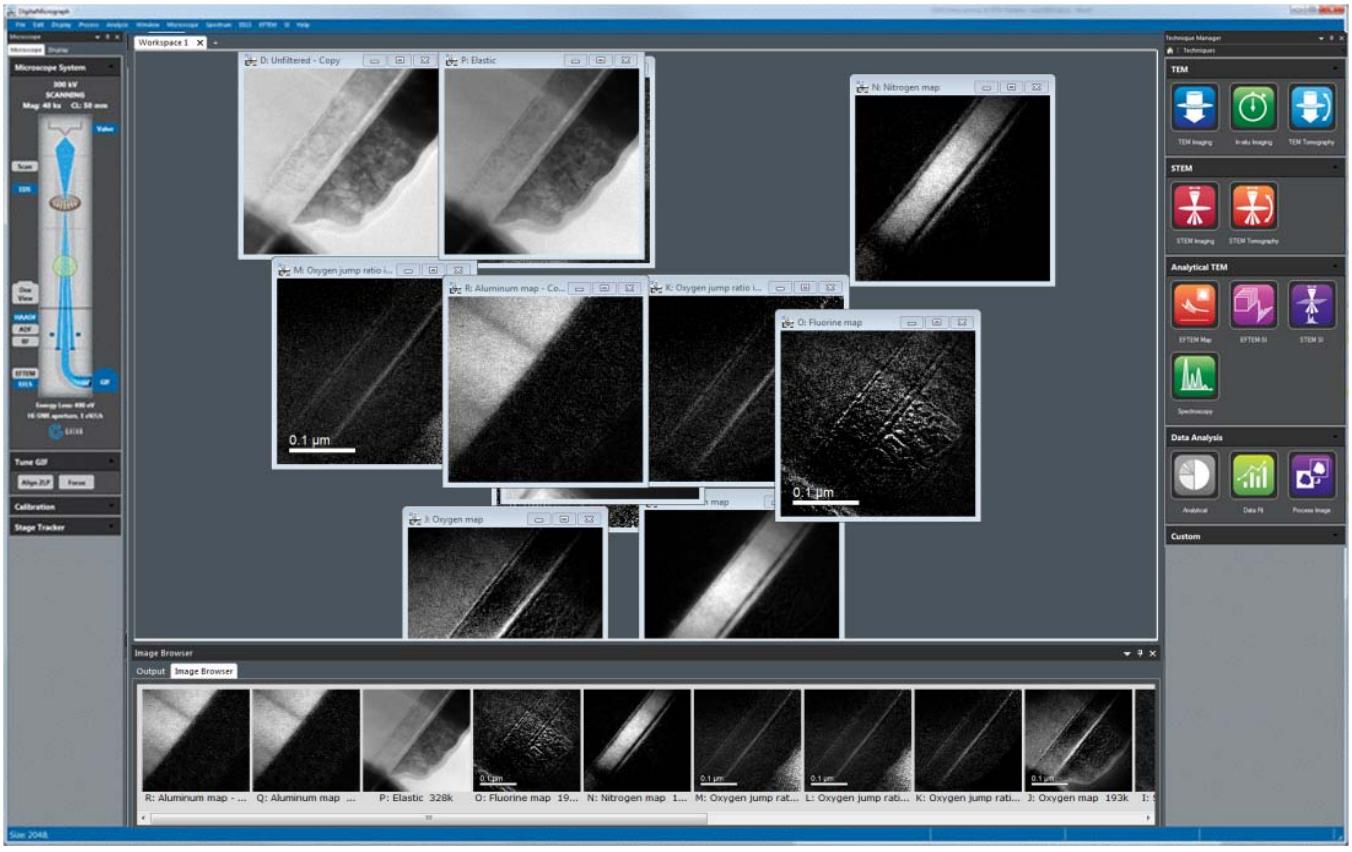


***Figure 2: The GMS 3 interface, with Microscope System panel on the left, the Workspace area in center, the Technique Manager on the right, and the Image Browser and Output areas on the bottom.***

On the left: Microscope System panel, a schematic diagram of the microscope that graphically indicates:

- Whether the camera is inserted or retracted into the beam: Inserted shown
- The current choice of either imaging or diffraction mode: Imaging mode shown

- The presence of a sample: Grid shown
- The status of the viewing screen: Down is shown

In the center is the Workspace where images are shown.

At the bottom of the screen are two tabbed panels, one for text output and another showing recently acquired image icons

On the right is the Technique Manager, with five groups of techniques:

1 TEM: TEM Imaging; *In-situ* Imaging, and TEM Tomography

2 STEM: STEM Imaging and STEM Tomography

3 Analytical TEM: Energy-filtered TEM (EFTEM) Map; EFTEM spectrum imaging (SI); STEM SI; and Spectroscopy.

4 Data Analysis: Analytical; Data Fit; 4D STEM Analysis and Process Image

5 Custom

# 2 Operation of the STEMx system

The process of diffraction image acquisition refers to the capture of a four-dimensional data set that contains both real-space and also diffraction space information about the specimen. It is directly analogous to STEM spectrum imaging, with the key difference that a 2D diffraction pattern is acquired at each spatial position instead of a 1D spectrum. The resulting data set is referred to as a diffraction image.

In STEM diffraction imaging, the microscope is configured in such a way as to produce a probe at a particular point (x,y) on the specimen with a diffraction pattern projected onto the camera. The diffraction pattern is recorded, and stored in this pixel location, before stepping the probe to the next pixel location and repeating. Hence, the diffraction image is filled by acquiring diffraction patterns in parallel, while slowly building up spatial information pixel-by-pixel. Hence acquiring a diffraction image can be viewed as a progressive filling of the 4D data stack as illustrated below.

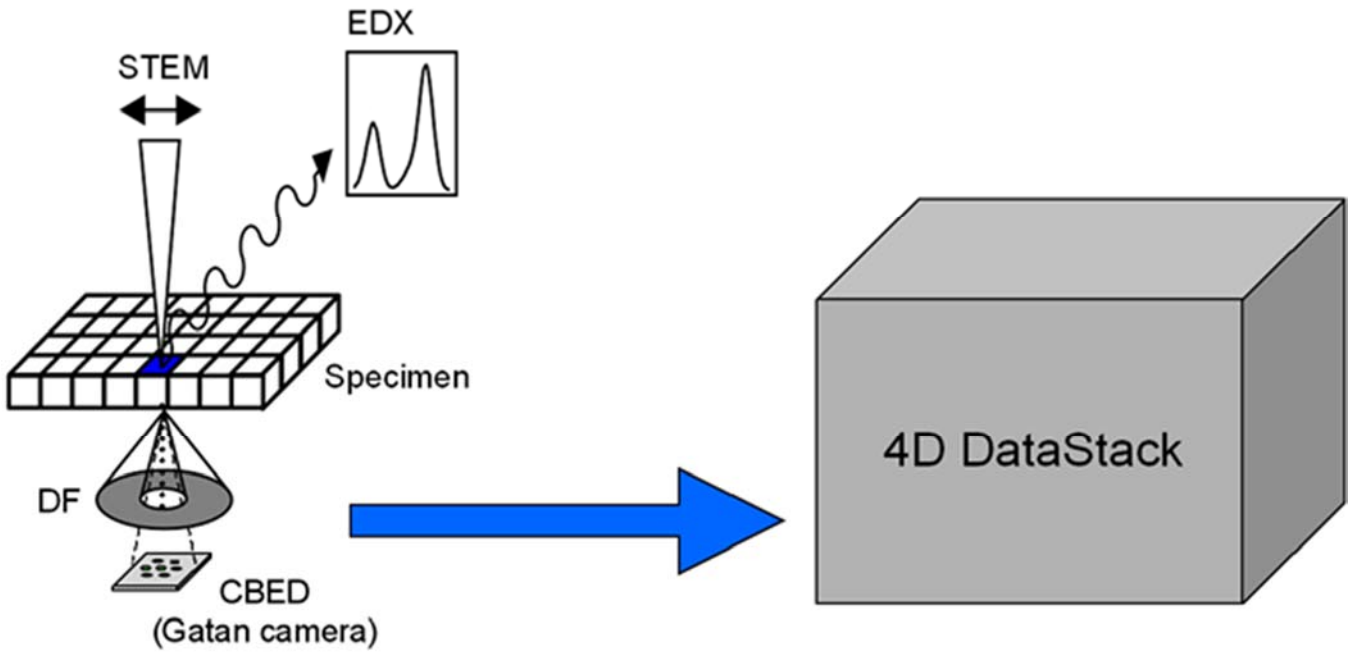


***Figure 3: STEM diffraction imaging.***

The user is strongly advised to review the general STEM SI / STEM Diffraction-Imaging help documents (available with GMS 3) for generic usage and concepts, and to review the DigiScan and camera user manuals for operation of the hardware. Here we only discuss specific procedures to STEMx operation.

STEM diffraction imaging using the STEMx system allows hardware synchronization of the scanning probe with the camera frame rate. This means that after the diffraction pattern is acquired by the camera at a given

electron probe position, a signal is passed to the DigiScan via the STEMx box, to drive the probe to the next pixel. This allows acquisition of 4D STEM datasets in up to 300 frames per second (fps) and 1600 fps for OneView IS and K2 IS cameras, respectively.

## 2.1 Acquisition

The first step in configuring diffraction image acquisition is to ensure that you have set up the microscope and, with a probe positioned using the DigiScan spot mode, can acquire a diffraction pattern on the camera that you wish to use (K2 IS or OneView IS). To configure the camera for diffraction image acquisition, it is recommended to insert the appropriate detector and use the camera UI to set-up the microscope and camera conditions to acquire the desired pattern.

Once you are satisfied with the detector set-up, diffraction image acquisition is performed using the STEM SI palette. For information regarding the general principles of data acquisition using this palette, first refer to the STEM Spectrum-Image Acquisition and STEM Diffraction-Imaging help sections.

The current memory requirements for the SI dataset are shown in the STEM-SI palette by hovering the cursor over the palette. You will be warned if the memory requirements for the acquisition exceed the amount available when starting an acquisition.

To enable the Diffraction-Image signal, select the CBED signal button on the STEM SI palette.

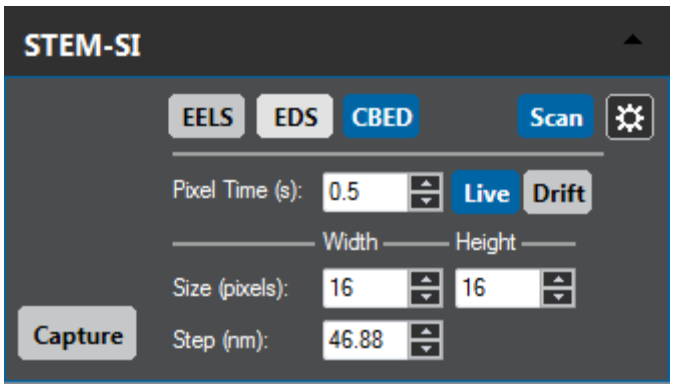


***Figure 4: STEM-SI palette.***

*Note: Only one signal can be selected that intercepts the primary beam. So, for example, EELS and CBED cannot be acquired simultaneously. Selecting one signal may automatically deselect incompatible signals.*

*Note: Selecting the CBED signal automatically expands the Camera panel which can be used to set-up and verify the camera signal.*

### 2.1.1 Diffraction SI setup

The diffraction signal acquisition parameters are accessed via the SI setup dialog opened by hitting the set-up button (gear icon) on the STEM SI palette.

Note that with STEMx licenses installed, DigitalMicrograph® (DM) selects hardware synchronization by default. If you are using STEMx with a OneView IS camera, and you wish to apply software synchronization for your data acquisition, check Force software synchronization (during this session).

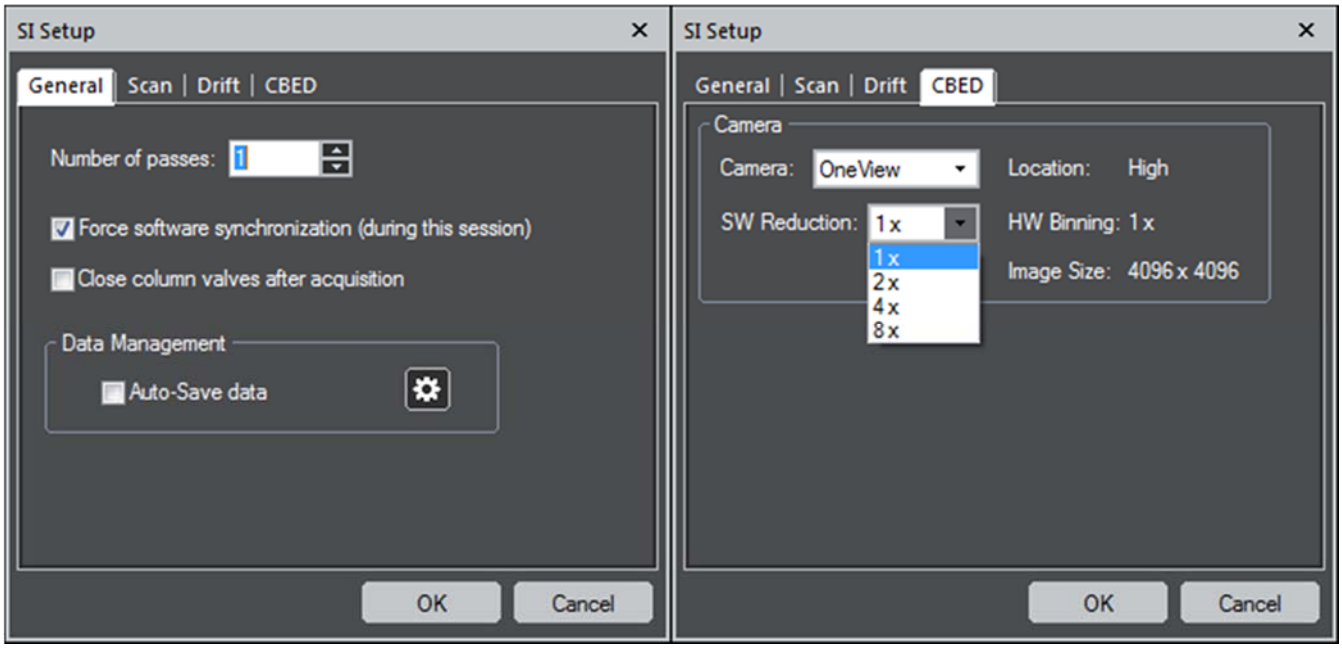


***Figure 5: SI Setup window.***

Use the CBED page to select the camera you would like to use for diffraction imaging and additional SW Reduction to be applied to the diffraction images (software reduction is not applicable to K2 IS).

*Note: In addition to one of the* in-situ *cameras (OneView IS or K2 IS), STEMx can be used to collect hardware synchronized diffraction images using a Gatan imaging filter (GIF). Both the GIF and the* in-situ *camera can be connected to STEMx at the same time, and the signal is automatically switched between the two according to the user selection in DM.*


*Figure 6: Clock-in connections to the back of STEMx system box.*

### Diffraction-Image acquire

Once the set-up parameters for the STEM diffraction image component have been configured, the actual acquisition is set-up and instigated using the normal STEM spectrum imaging procedure. Please refer to the STEM Spectrum-Imaging help section for a detailed description of the procedure.

Note that Diffraction-Images acquired with a K2 IS camera will be directly stored into the capture PCs in binary format. After data acquisition is complete, a smaller sub-sampled dataset of what has been collected will be displayed in DM. This shows the user that data acquisition is complete and allows them to examine the data to decide how to proceed with the experiment. If the user decides to keep the collected dataset, they can use the Data Manager palette to extract this dataset into a 4D cube and store it in DM format for further processing.

### Align STEM Diffraction-Image

Once a STEM Diffraction-Image is acquired, it is often desirable to align the direct beam diffraction spot which can move around with pixel position within the diffraction-image. This can be performed using the Align SI By Peak sub-menu item in the SI menu. Please refer to the STEM Diffraction-Imaging help section for a detailed description of the procedure.

### Diffraction-Image calibration

To ensure that STEM Diffraction-Images are acquired with the correct calibration, ensure that both the DigiScan and the camera are correctly calibrated for the appropriate STEM acquisition modes. Please refer to the sections on using calibrations in DM and camera calibration for details.

Once the acquisition devices are calibrated, all diffraction image data will be acquired with the appropriate spatial and diffraction calibrated units written to the dataset. By default, the calibrated center of the diffraction space will be set to the center of the diffraction pattern (i.e., pixel position [127, 127] for a diffraction pattern of size {256, 256}). Generally, it is advisable to set up your STEM diffraction image acquisition such that the diffraction pattern center corresponds closely with the camera image center. However, it is often desirable to refine this. Please refer to the STEM Diffraction-Imaging help section for a detailed description of the procedure.

## 2.2 Analysis

As mentioned previously, diffraction images acquired with the OneView IS camera are in a 4D cube format; on the other hand datasets acquired with the K2 IS camera are originally stored onto the capture PCs as binaries and would have to be extracted into 4D cubes before any further processing can be done.

### 2.2.1 Using K2 IS data manager with diffraction images

Datasets collected with combination of STEMx and K2 IS are saved on the capture PC in binary format. Similar to the K2 IS *in-situ* datasets, these need to be extracted into a DM format before further processing. This can be done using the K2 IS Data Manager. The user is referred to the K2 IS user manual for instructions on how to use the Data Manager; here we only list its specific applications to STEMx datasets:

1 Select the desired dataset from the list:

   a. Dataset Info will be displayed on the right.

   b. Similar to a K2 IS dataset the player can be used to view and play through the diffraction images.

2 **Optional**: Right click on the image and use the rectangular ROI tool to draw an ROI on the diffraction pattern. As the dataset is extracted into a Diffraction-Image, all diffraction patterns will be cropped to this selected region.

3 Click on Extract

   a. Browse to the folder where you want to save the Diffraction-image after it is extracted.

   b. Select the Sampling interval (only real space) and binning (only diffraction pattern).

   c. Click ok. This will add this task to the task list.

4 Repeat 1 to 3 and add as many tasks as you want to the task list.

5 Once ready to process all the listed tasks, click on Process. Data manager will start from the top of the task list; it extracts the STEMx dataset using the parameters selected as described above. Once it is done with one task, it saves the extracted diffraction image (4D cube) to the folder you had specified, closes the dataset and starts the next task.

### 2.2.2 Diffraction image analysis

For details of diffraction image Visualization, Summing and Mapping the user is referred to the STEM Diffraction-Imaging help.

### 2.2.3 2D strain mapping

Microscope setup for data acquisition with the purpose of 2D strain mapping is critical:

- Set the convergence angle such that diffraction discs do not overlap

  Smaller convergence angles translate to larger probe sizes and decreased spatial resolution, so that is a consideration. On the other hand, as in any other image processing algorithm signal to noise ratio is very important. Larger discs spread the signal over more pixels, so there will be more pixels for disc fitting (good), but fewer counts per pixel (bad). Optimizing this depends on the thickness of the sample.

- Depending on the camera sensor size any crystal unit cell, the camera length varies; set the camera length such that the first 2 – 3 orders of diffraction spots can be imaged on the camera

- Ideally, the sample should be oriented very near the zone axis, so that strong transfer to diffraction discs in multiple directions is possible

  If it is not possible to tilt on-zone, try to find a near zone tilt that strongly illuminates discs in 2 directions, even if those are not the closest discs to the center spot.

- It is also an important point that the condenser aperture must be well focused in the diffraction plane.

- Sample thickness should be enough such that there is a reasonable amount of diffraction scattering. On the other hand, if the sample is too thick, there is a lot of fine structure in the diffracted discs, which can lead to difficulty in edge finding. If there are enough black lines/streaks/texture in the diffraction disc that a significant fraction of the disc circumference is black instead of bright, strain precision will suffer.

In order to calculate a 2D strain map from a diffraction image:

1 Open the diffraction image in DM. Note the size of the dataset and the available physical memory before opening the dataset.

2 Select 4D STEM Analysis, under the Data Analysis section in the Techniques page.

3 Right click on the dataset and select the “U” tool to specify the reference unstrained area in the sample.

*Note: It is not always possible to have a perfect unstrained region in the sample. In the case of epitaxial grown layers, often the interest is to measure how the strain varies with distance from interface, so one reference lattice can be used (substrate) for all layers. In this case the effective strain with respect to the reference is reported, which might mean that the epitaxial material can be unstrained with respect to its own bulk lattice, but reports a strain with respect to the substrate. If a definitive reference lattice (such as the substrate) is not available, one can use the average lattice over the field of view as the reference. In this case, perturbations are preserved, but not necessarily the absolute lattice parameter. In cases where material within the field of view are not epitaxial, or have different crystal structures/directions (like a grain boundary), use a separate reference lattice for each region.*

4 Click on Reference to calculate the reference diffraction pattern from the selected area.

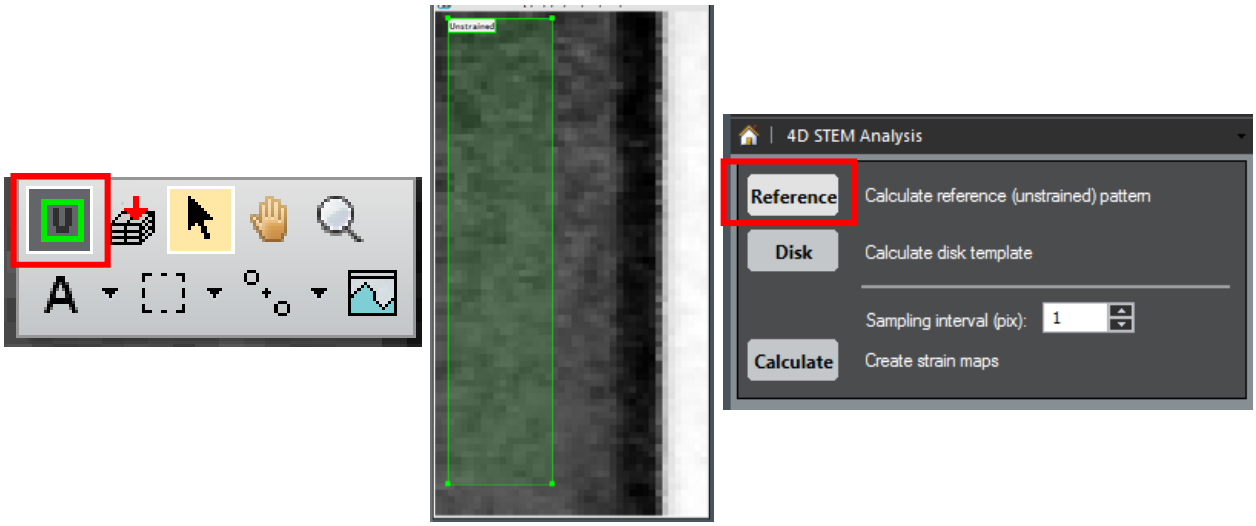


***Figure 7: Select the U tool to specify the unstrained reference area and click on Reference to calculate the unstrained pattern.***

5 Specify the central beam and the U and V vectors by adjusting the size and position of the red circles on the reference diffraction pattern.

6 Adjust the size and position of the green circle (spots) on the reference diffraction pattern. This specifies how many orders of diffraction spots will be used for the strain calculations. Larger selection will elongate the calculation time.

7 Click on Disk to calculate the disk template.

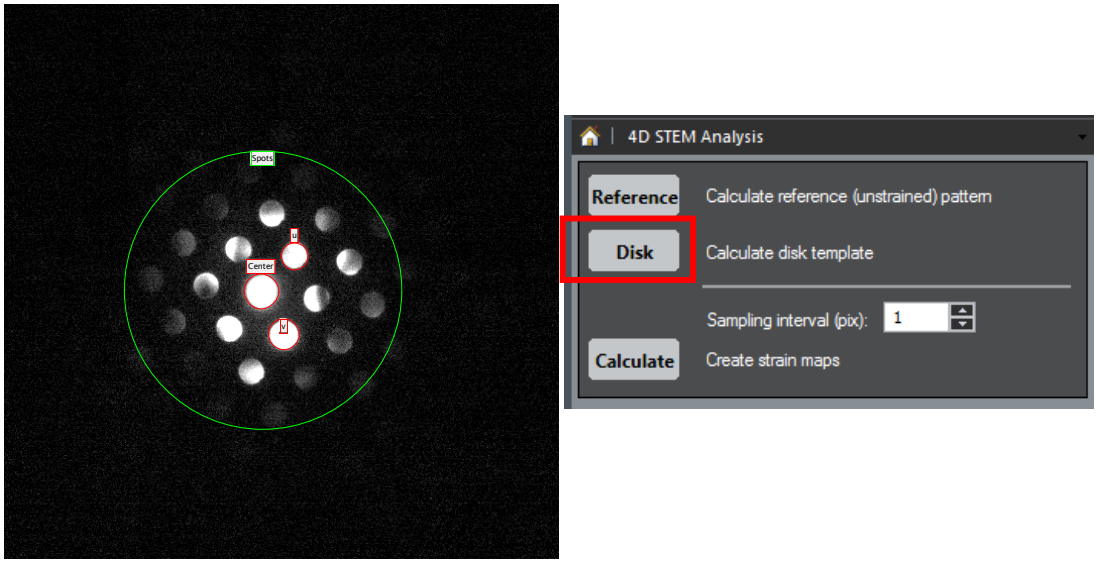


***Figure 8: Adjust the central beam, u and v vectors and calculate the disk template.***

8 Once the disk template is calculated, you are ready to start the strain calculations.

a. Set the Sampling Interval (subsampling is done with equal number of pixels in X and Y directions in the real space and reduces the calculation time).

b. Click on Calculate.

Progress bar shows the progress and the remaining time to completion. Once the calculation is done, complete 2D strain maps, shear map and rotation map will be displayed in DM.

### 2.2.4 Volume tools

In many cases it is useful to reduce the size of the diffraction image. This is possible using the Volume menu. This menu provides a set of tools which can be applied for processing and manipulation of both 3D and 4D datasets. Some of these tools are only applicable to 3D, some only applicable to 4D and some shared between the two types of datasets. User is referred to the Volume help section for more details.

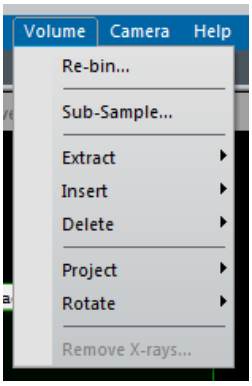


***Figure 9: Volume tools.***

## 2.3 Power on/off sequence for STEMx

### 2.3.1 Power on

To start from the power off:

1 Close DM if it is open (or power on the PC if it is off)

2 Plug in the power cable into the back of the STEMx box. Note that there is no power switch on the STEMx box. Gatan logo lights up when power is connected.

3 Launch DM software


***Figure 10: Power connection to the back of STEMx system box.***

### 2.3.2 Power off

Under normal circumstances, you can leave the STEMx plugged into power. If necessary, you can power down completely:

1 Close DM

2 Unplug the power cable from the back of the STEMx box.

## 2.4 Software

Install the GMS license using the Gatan license CD. Installation instructions are included with the CD.

*Special note: For optimal performance do not run antivirus applications during operation of camera and do not load third party applications other than those recommended or verified by Gatan.*