GIF Continuum K3 User Manual

Models 1069, 1069HR and 1067HD

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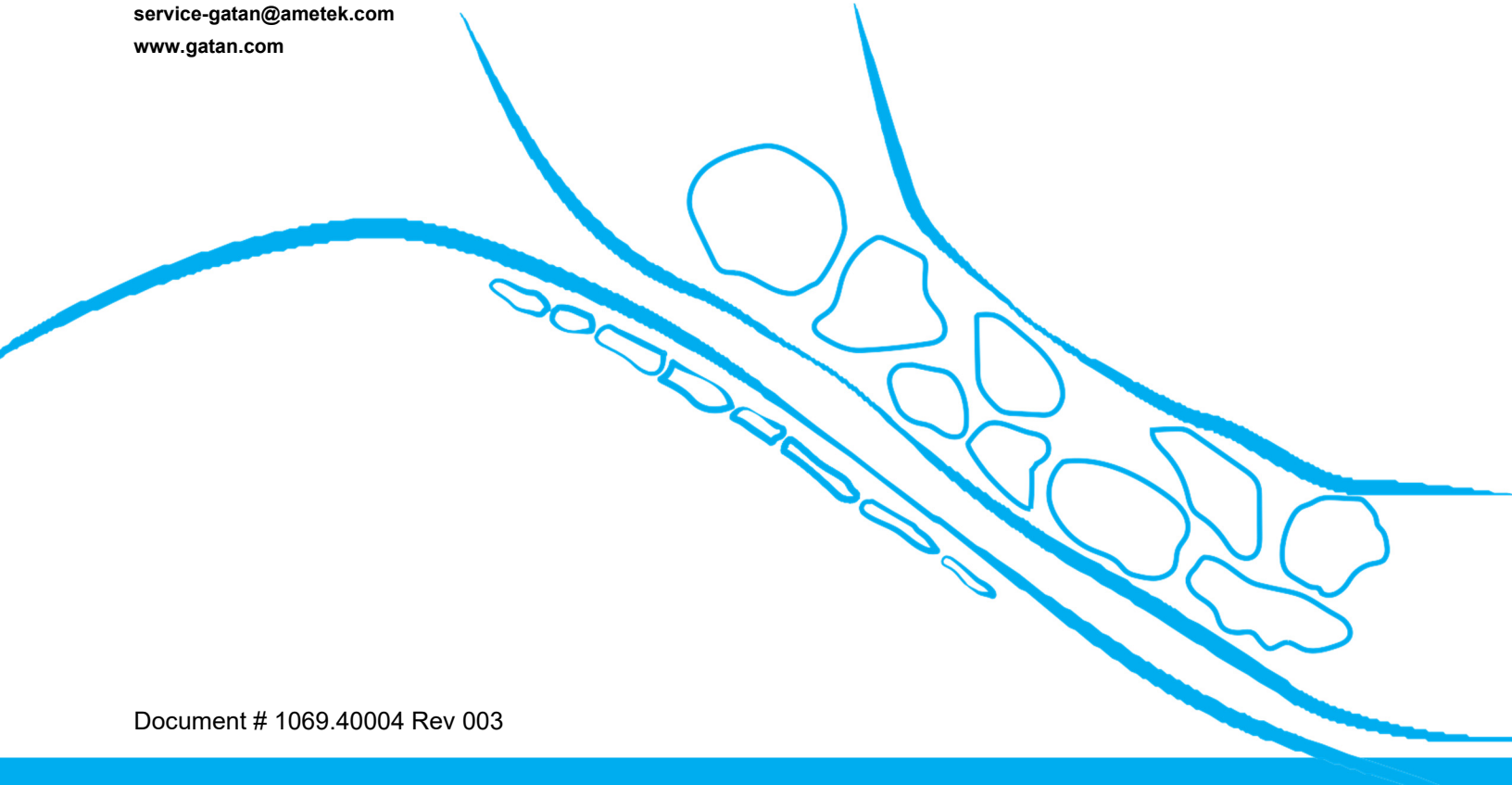

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1 Regulatory compliance and safety instructions

IMPORTANT

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.

2 Overview

The Continuum series represents Gatan's fifth and most advanced generation of post-column energy filters and spectrometers. Building on the dodecapole-based optics proven in the GIF Quantum®, the GIF Continuum™ K3® combines this advanced electron-optical design with ultra-high-speed single electron counting detection systems. This yields an imaging filter that defines the new state-of-the-art in the capture of both highly detailed electron energy loss spectroscopy (EELS) and energy-filtered transmission electron microscopy (EFTEM) data sets at 10x data throughput.

With the GIF Continuum, there is no compromise between EFTEM and EELS performance. Gatan's advanced sensor readout architecture allows the same camera sensor to be used interchangeably as a full-frame high-quality imaging device, a high-speed live viewing in-situ device, and an ultra-fast spectroscopy device. Aberration correction up to fifth-order allows the use of a 9 mm entrance aperture for EFTEM and a 5 mm entrance aperture for energy loss spectroscopy.

1.1 Hardware

The GIF Continuum K3 series imaging filters perform optimally at beam energies from 80 – 300 keV (single camera system) or 40 keV (30 keV optional) through 300 keV (dual-camera system). These can be attached to the bottom flange of nearly any modern transmission electron microscope (TEM), allowing the formation of high-quality energy-filtered images and diffraction patterns, as well as outstanding energy-loss spectral acquisition. The post-column design provides maximum flexibility in the choice of electron beam source and imaging optics. Furthermore, Gatan's quadrupole projection optics ensure the spectra are always aligned with the detector, can capture a broad range of dispersions, and optimally project to a rectangular area of the sensor detector to guarantee the best dynamic range, sensitivity, and readout speed possible.

The electron optics of the GIF Continuum fully correct focusing aberrations through fifth order. This advancement permits the use of a large 9 mm aperture at the entrance of the filter, giving the GIF Continuum a large field-of-view. The same advancements in aberration correction and new implementations of Gatan's patented tuning algorithms, allow the GIF Continuum to achieve outstanding distortion performance on the detector. Using patented algorithms, the electron optics of the system can be easily auto-tuned to ensure the system is running at peak performance. GIF performance is further optimized by the automatic saving and recalling of fine-tuned settings for each major mode and setting of the TEM imaging column ensuring peak performance in all conditions.

The GIF Continuum K3 series imaging filters include a 3.4k x 3.4k (or 5760 x 4092 for the 1067HD) K3 direct electron detector that is capable of running in electron counting mode at 3000 spectra/s. The K3 camera supports spectrum acquisition in both single and DualEELS modes. With the *in-situ* (IS) upgrade, a wealth of *in-situ* imaging modes are supported. For full details, please refer to the K3 user manual.

GIF Continuum K3 imaging filters may include an advanced version of the 2k x 2k Continuum camera that is capable of running in HQ, HS, and HS+ modes that yield >2000, >4000, and >8000 spectra/s, respectively. Gatan's novel ping pong spectrum mode is also available for high-duty cycle acquisition. This camera also supports a large number of *in-situ* imaging modes in the presence of an IS upgrade.

With the GIF Continuum series imaging filters, there is no need to compromise image or spectroscopy speed for image quality: One system does it all. EELS and EFTEM are option on the 1067HD.

1.1.1 Major components

The major components of the GIF Continuum are below.

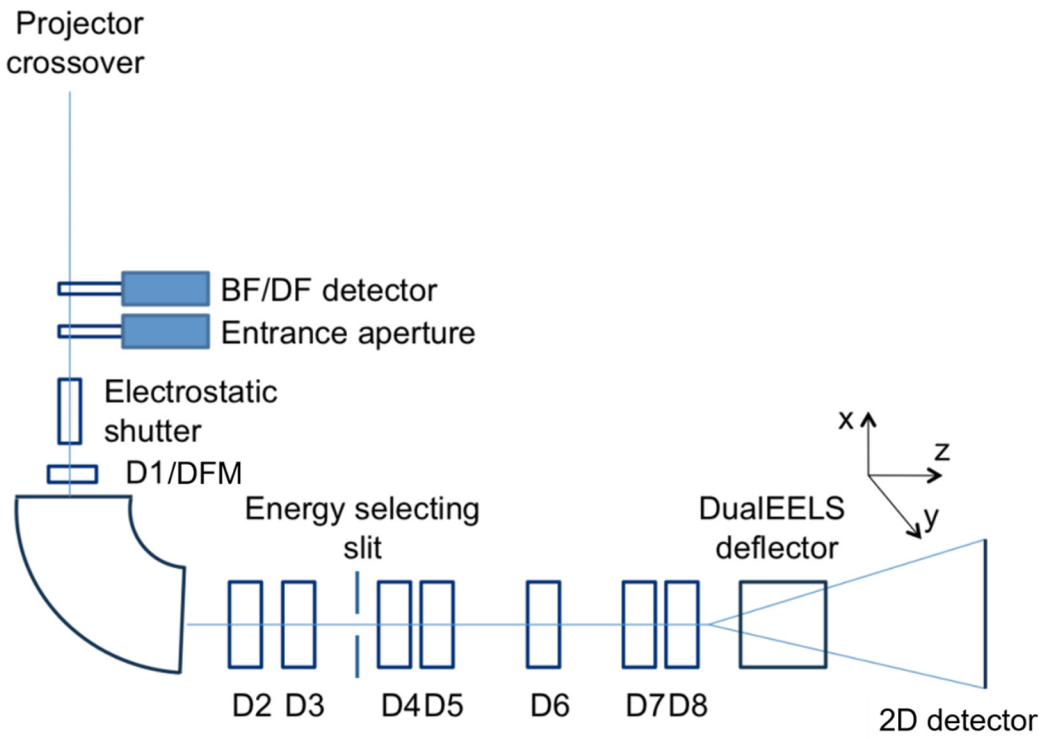


Figure 1: ***Schematic of the GIF Continuum showing the location of the key components.***

Entrance aperture

At the entrance to the system, there is a pneumatically controlled, multi-position aperture rod that contains a variety of apertures.

All Continuum models have two spectroscopy aperture positions denoted as Hi-Res (small) and Hi-SNR (large). The Hi-Res, 2.5 (1.0 on 1069HR) mm and Hi-SNR, 5.0 mm round aperture positions are for defining the collection angle (and to limit spectrum aberrations) during the EELS spectrum acquisition.

The large square aperture position is exclusively for energy-filtered imaging and diffraction work. The square aperture is a 9 mm diagonal. Its size ultimately determines both the magnification of the GIF projection system and the largest specimen area (or diffraction angle) that the GIF can view.

The final entrance aperture position contains an alignment mask. The holes of this test-pattern mask are precisely placed on a grid. The system uses images of the mask formed at the final detector plane of the GIF to align and tune the GIF imaging optics at installation. During routine usage, mask images are collected to check and, if required, fine-tune the GIF imaging properties.

Electrostatic shutter

All Continuum models are available with an internal electrostatic shutter that supports exposure times as low as 0.1 µs. This is standard for the 1069 series and comes with either the EELS or EFTEM options for a 1067HD. Since this shutter is electrostatic as opposed to magnetic, it allows extremely fast and hysteresis free beam blanking.

The shutter location is between the GIF entrance aperture and the magnetic prism. The electrostatic shutter consists of two deflector plates that are biased at opposite polarity. In the unshuttered condition, the plates are at ground potential. The plate voltages automatically scale with the GIF operating voltage and are highest at 300 kV operation.

Magnetic prism

A key element of the GIF Continuum is its 90° gradient sector magnet, which bends the primary beam through a 7.5 cm bending radius and thereby disperses it in energy. The prism has inclined pole faces that give it additional first-order focusing properties.

Dodecapoles (D1 – D8)

The GIF Continuum uses 8 dodecapole lenses rather than a series of individual multipoles. Each of the 12 poles of the dodecapoles is individually excited to generate any combination of the deflector, quadrupole, sextupole, octupole, decapole, and dodecapole lens. This enables control of the optical properties up to the 6^{th} order.

The dodecapole locations and first-order beam trajectories are chosen, such that sufficient range is available on each dodecapole to accommodate the sum of the multipole moments necessary to control the first-order properties (focus, magnification, energy dispersion), as well as correct the various aberrations.

Energy selecting slit

For energy filtering, the system includes an energy selecting silt. The slit comprises two independently controlled blades. The lower blade intercepts high energy electrons (low energy loss) while the upper edge intercepts lower energy electrons (high energy loss). The opening is computer controlled and calibrated in energy units for each operating TEM voltage. In the **retracted** state, the upper edge opens fully, but the lower edge opens to only -50 eV energy loss to prevent stray scattering. In the **inserted** state, the slit opens symmetrically around the optic axis of the GIF by the requested number of eV.

In spectroscopy mode on GIF systems, the slit opens just larger than the spectrum field of view and acts as a beam trap reducing stray electron scatter in the system.

Drift tube spectrum offset

The Continuum uses an electrically isolated drift tube to offset the energy-loss spectrum. The electrons accelerate on entering the drift tube by the voltage applied, yielding a precise energy offset. The electronics support a 2 kV range, and fast 10 µs switching at up to 5 kHz for high-speed DualEELS acquisition. New for Continuum is a full-length drift tube starting at the first dodecapole lens and ending after the last lens. This drift tube virtually eliminates lensing and deflecting effects at the ends of the drift tube and keeps the energy fixed in the spectrometer regardless of the offset.

DualEELS deflector

The DualEELS mode of operation simultaneously acquires a core-loss spectrum and corresponding low-loss spectrum with independent exposure times. The DualEELS hardware includes an electrostatic deflector in front of the camera that alternately deflects the spectrum perpendicular to the dispersive direction to opposite halves of the camera. The voltage on the drift tube is changed between the individual exposures so that one spectrum contains the core-loss region of interest and the other the low-loss spectrum. Careful orchestration of the timing of the drift tube, electrostatic DualEELS deflector, and electrostatic shutter allows DualEELS to be performed up to 3000 or 8000 spectrum pairs per second for the K3 and fiber-coupled detectors respectively.

New for the Continuum, the DualEELS deflector is also used in the **ping-pong** readout mode of the detector. In this mode, the detector's rolling readout is synchronized with the deflector to give near 100% duty cycle in all readout modes.

2D electron detector

The Continuum models include a state-of-the-art electron counting transmission CMOS detector. The detector has 5 µm pixels arranged over a 3456 x 3456 or 5760 x 4092 active area. It can also include an optional advanced CMOS detector that is fiber optically coupled to a scintillator. The fiber-coupled detector has 18 µm pixels and 2048 x 2048 square active area. A hybrid pixel direct detector "STELA" is also available allowing counting at low primary voltages.

The counting detector uses a proprietary high-speed sensor, data pipeline, and counting algorithm to record single electron counting EELS spectra at a speed of 3000 spectra per second. The detector operates over beam energies of 80 – 300 kV. Please refer to the K3 and K3 IS user manuals for additional specifications.

The optional secondary GIF Continuum camera uses a proprietary high-speed scintillator capable of recording >8000 spectra per second without scintillator afterglow cross talk. For low-kV operation, a high brightness option is available, but it has a significantly slower response. This version is limited to only ~2000 spectra per second due to afterglow with the advantage of better performance below 80 kV. Please refer to the GIF Continuum user manual for additional specifications.

A second optional camera is also available using hybrid pixel technology. The STELA detector is a direct detection device optimized for lower primary voltages. It allows the advantages of counting all the way to 30 keV although with only 1024 x 512 pixels.

Dynamic focus module (1069HR model only)

The dynamic focus module (DFM) is an electrostatic dodecapole co-located with the first magnetic dodecapole. As an electrostatic lens, its value can be changed rapidly and without hysteresis. This lens is automatically calibrated as a function of the energy loss to dynamically refocus the spectrum as the energy offset changes. In DualEELS mode, the DFM is synchronized with the energy shift at up to 5 kHz for high speed, DualEELS acquisition with focus correction.

BF/DF detector

STEM imaging is a very efficient process allowing nearly all the electrons passing through the sample to be acquired simultaneously through multiple acquisition signals. The synchronous collection of EELS and annular dark-field (ADF) data, for instance, offers an accurate spatial correlation between spectral and image features.

An optimized ADF detector efficiently collects the electrons just outside the 5.0 mm spectroscopy aperture. The ADF detector has a scintillator with an effective inner diameter of 5.3 mm and an outer diameter of 12 mm. The light from the scintillator is coupled by a mirror to a PMT based amplifier chain. The pneumatically operated scintillator drive rod also includes a 5.7 mm outer diameter bright-field detector. In EFTEM imaging, the detector rod is fully withdrawn.

The final position houses a diffraction beam stop (standard) or a 1 mm slit (Q-Slit option) for momentum resolved EELS.

1.1.2 Electronics

Supporting electronics hardware for the Continuum consists of the main optics controller (OC), formerly known as the GIB, the OC power supply, high-voltage electronics for the electrostatic shutter, DualEELS and high-speed 2 kV spectrum offset, controller for the slit, controller for the DFM (1069HR only), and the controller for the camera(s).

The OC houses the main GIF control electronics, which drive the GIF prism current, all its dodecapole lens currents, and the entrance aperture. It also contains the OC timing master that generates the gate signals to synchronize deflectors and probe advance with the detector readout. It interfaces with the host computer via an ethernet link.

The illuminated super-G logo is **blue** when the power is on. The amber data LED flashes when data is sent between the computer and the GIB.

The OC power supply contains a toroidal transformer to reduce stray fields and supplies all the DC power required by the OC. The illuminated super-G logo on the front panel **pulses** when the system is in **standby mode**. Touch and hold the **super-G logo** for **3 s** to power on the system. The super-G logo **glows steadily blue** when the power is **on**. The over temp (**OT**) LED is lit when the power supply is overheating and has automatically shut down power. The **Status** LED is lit a **steady green** when the system operating normally; off or flashing indicates a fault.

The high-voltage electronics for the electrostatic shutter, fast drift tube, DFM, and DualEELS deflector are mounted on the filter itself to minimize connection capacitance and maximize speed. They are set up and controlled over an ethernet interface and receive high-speed gate (on/off) signals from the OC timing master. The power for these units is generated in the power tray of the Continuum rack. These controllers have four status lights: **POWER** – DC power applied, **READY** – system ready to use, **GATE SIGNAL** – external gate applied to energizes output voltage, and **OVER TEMP** – overheating fault

1.2 Software

The Gatan Microscopy Suite® (GMS) versions 3.4.2 onwards support the GIF Continuum K3 system. The software supporting the Continuum for both operation and data acquisition has been extensively revised. Automation plays a vital part in the new software interface, both during filter alignment and the optimization of parameters (e.g., camera exposure) for data acquisition.

3 Theory of operations

3.1 Electron optics

In normal operation, the Continuum uses the entrance aperture to select a part of the image or diffraction pattern that is projected by the microscope into the viewing chamber. The electrons enter the magnetic prism and are bent over approximately 90°. The exact bending angle of an electron depends on its energy; the greater the energy loss it experiences, the larger the angle the electron is bent. Besides bending the electron beam and creating energy dispersion, the prism also has a focusing action, and at some distance behind the prism, an energy-dispersed, a focused image forms of the TEM's projector lens crossover. This is the energy-loss spectrum.

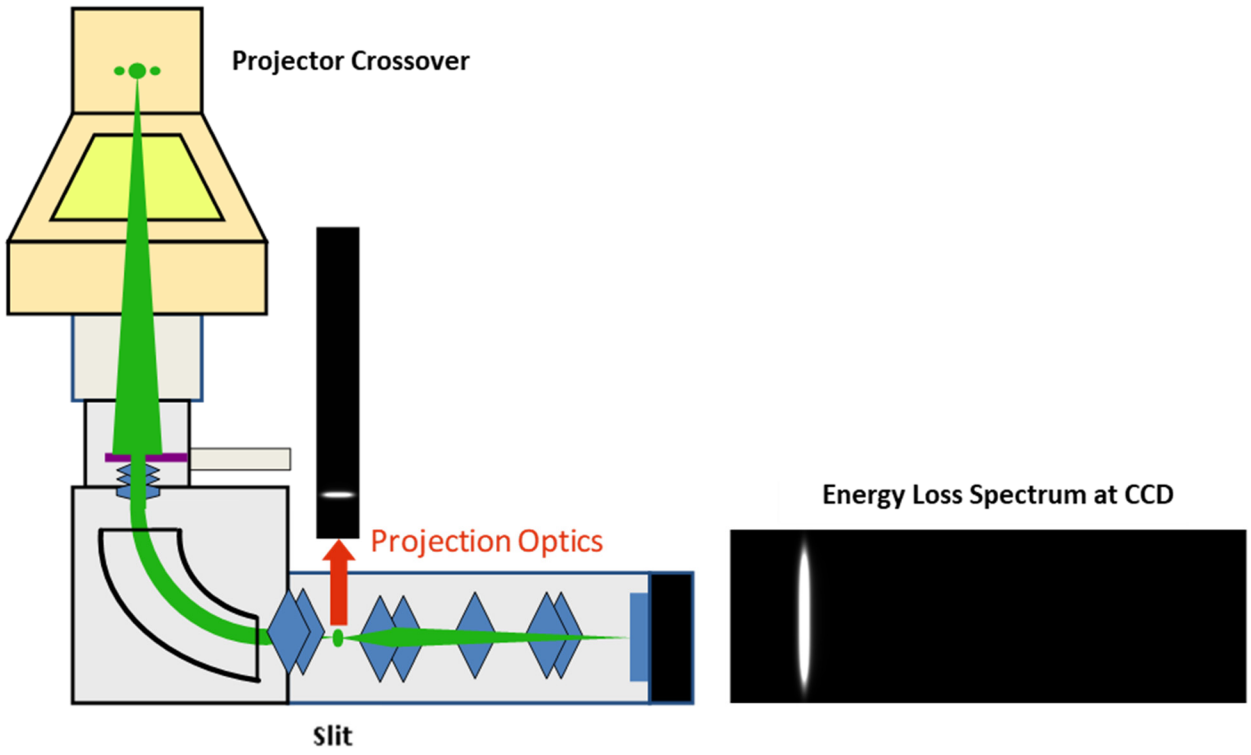


Figure 2: ***Ray path diagram showing the conjugate planes for the spectrum mode at the projector lens crossover, the energy selecting slit and the detector. The projector optics after the slit can focus either the spectrum at the slit onto the detector.***

The prism is of the gradient type and uses inclined pole faces to focus simultaneously in the x and y-direction. The entrance and exit faces are straight, and the GIF Continuum relies fully on the optics external to the prism to correct the various important spectrum aberrations.

The dodecapole locations and first-order trajectories are chosen such that enough range is available on each dodecapole to accommodate the sum of the multipole moments necessary to control the first-order properties (focus, magnification, energy dispersion), as well as correct the various aberrations.

One dodecapole in front and two dodecapoles located after the magnetic prism project the magnified focused spectrum onto the slit, correct all-important 2^{nd}, and 3^{rd} order aberrations and minimize the important 4^{th} and 5^{th} order spectrum aberrations. This aberration correction allows an entrance aperture of 9.0 mm diagonal while maintaining isochromaticity below 2.0 eV at 200 kV. The two dodecapoles, between the magnetic prism and the energy selecting slit, magnify the energy dispersion while maintaining the focus of the spectrum at the energy-selecting slit.

The energy-selecting slit is in the plane of the focused energy-loss spectrum. It can be narrowed to select a band of energies in the spectrum for energy-filtered imaging or opened for unfiltered imaging or spectroscopy. The slit design is such that the position of either edge can be independently controlled and is typically set symmetrically around the optical axis of the system.

The remaining five dodecapoles are located after the energy-selecting slit and project either an aberration-corrected energy-filtered image or an aberration-corrected energy-loss spectrum on the camera.

3.1.1 Operation in imaging mode

In Imaging mode, the electrons that were selected by the energy-selecting slit project as an energy-selected, achromatic, aberration-free version of the TEM image onto the camera. Achromatic means that no energy dispersion remains in the image (e.g., chromatic aberration); or said another way, images formed from different energies coincide. The dominant remaining chromatic blurring in the image is due to the chromatic aberration of the TEM's objective lens. The magnitude of the blurring is determined by the magnitude of the objective lens' chromatic aberration coefficient (often referred to as C_C), the angles subtended by the objective aperture, and the size of the selected energy-loss interval. The smaller the energy interval and the angles, the less blurring occurs.

3.1.2 Operation in spectroscopy mode

In Spectroscopy mode, the post-slit dodecapole assembly projects the focused energy-loss spectrum at the energy-selecting slit onto the cameras. A range of spectral magnifications or dispersions has been pre-programmed, allowing the user to optimize both the energy range projected on the detector and the magnification (resolution) of the spectrum.

3.2 Filtered imaging operating principle

EFTEM is a family of imaging techniques that utilize properties of the energy loss spectrum to increase contrast, reduce the effects of chromatic aberration, and create unique contrast effects in the image. Key applications include:

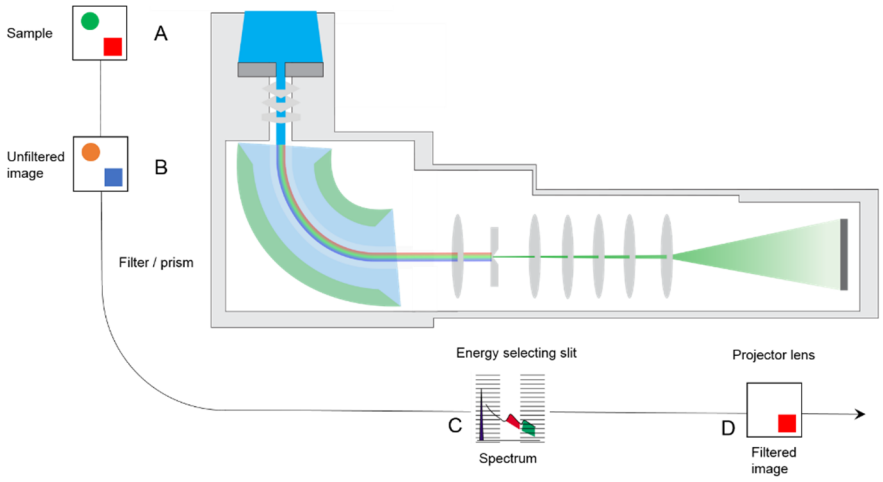
- Contrast enhancement – Improves contrast and resolution in images and diffraction patterns by allowing only a narrow band of electron energies to form the image
    - Zero-loss filtering – Boosts contrast and resolution through the removal of inelastically scattered electrons
    - Most probable loss imaging – Improves signal-to-noise ratio (SNR) in thick samples and tomography applications

- Contrast tuning – Highlights a particular material phase or property in the image
- Pre-carbon imaging – Boosts contrast of non-carbon structures in unstained biological and polymer samples

- Mapping – Creates elemental/chemical maps at nanometer resolution by forming images with inelastically scattered electrons
  - 2-window jump-ratio imaging
  - 3-window elemental mapping
  - Chemical state mapping

The principle behind EFTEM is to illuminate a very thin specimen with a beam of high energy electrons. Some of these electrons interact with the specimen and result in elastic or inelastic scattering. Inelastic scattering results in both a loss of energy and a change in momentum, which in the case of inner-shell ionization, the energy loss is characteristic of the element the electron interacted.

To illustrate the EFTEM process, consider a thin sample (A) in the TEM that produces an unfiltered TEM image (B), which has a lot of image detail but is hard to interpret. The prism transforms then the image into a spectrum (C). Next, an energy-selecting slit filters the spectrum. The selected part is transformed back into an energy-filtered image (D). Then a suitable detector collects the final energy-filtered image.



If only the main energy (zero energy loss) electrons form the image, a zero-loss filtered image is obtained, the filtering prevents the out of focus, inelastically scattered electrons, from contributing to the image plus enhances image contrast and resolution. In addition to zero-loss filtering, you can adjust the system to select electrons that have lost a specific amount of energy to obtain additional contrast effects and compositionally sensitive images.

Jump-ratio imaging is a technique that requires two energy-filtered images, one just before the ionization edge (pre-edge) and one just after the edge (post-edge). The resultant images are divided pixel-by-pixel to yield a qualitative map that is bright when the element is present and dark where it is not.

The 3-window technique requires two images before the ionization edge and one after. It uses the pre-edge images to compute the approximate background contained in the post-edge window. Once the background is determined and removed pixel-by-pixel, the resultant map shows a signal that is proportional to the element concentration in the sample.

Alternatively, it can obtain a series of images over a broad range of energies. Results then contain a continuous range of energies. This allows quantitative analysis and improves the accuracy of mapping, where more than one element is involved. This EFTEM data stack is known as a spectrum image.

3.3 Spectroscopy operation modes

When the Spectroscopy mode is selected, an image of the projector lens crossover projects onto the camera. When properly adjusted, the image is a sharp line for electrons of one energy; electrons of another energy produce another sharp line, displaced in the dispersive direction.

The projector crossover is present under all normal TEM or STEM operating conditions (if the projector lens were not excited strongly enough to form a small crossover, the image on the final viewing screen would only be a few millimeters in diameter). The crossover moves up and down a little depending on the exact operating mode, but small adjustments of the pre-prism quadrupole **FOCUS X** can easily compensate for this movement. Sometimes **FOCUS Y** must also be adjusted. Thus, the spectrometer operation and microscope operation are almost completely independent.

The acquired spectra correspond to whatever feature was in the center of the microscope viewing screen because this part is selected by the entrance aperture of the spectrometer when you lift the screen. It can be a precipitate image in TEM, a Bragg beam from a diffraction pattern, etc. Three basic operation modes are possible: **TEM Imaging**, **TEM Diffraction**, and **STEM**.

3.3.1 TEM imaging

With the TEM in the Imaging mode, an image is formed on the viewing screen, while the projector crossover contains a small diffraction pattern. Because the spectrometer projects an energy-dispersed diffraction pattern onto the detector, this is also known as the *diffraction-coupled* mode. The camera length L of the pattern in the projector crossover is given by

$$L = h/M$$

and the diameter dp of the projector crossover therefore by

$$dp = (2\,ß\,h)/M$$

where

- h is the distance from the projector crossover to the viewing screen
- M is the image magnification
- ß is the half acceptance angle as defined by the objective aperture

In practical terms, with h = 50 cm and at M = 10,000x, L = 50 µm. A diffraction pattern encompassing angles to 50 mrad is, therefore, only 5 µm in diameter at the projector back-focal plane, and it becomes even smaller at larger microscope magnifications. This means that good energy resolution can be attained in the TEM Imaging mode while accepting practically all the electrons scattered from a specimen area. If a smaller range of scattering angles is required, this can be selected using the objective aperture.

Note: Operating in TEM Imaging mode for spectroscopy should not be used except for the initial examination, as it may give the false analysis results described below.

The TEM Imaging mode, at first sight, appears to allow the selection of a small specimen area for microanalysis by the spectrometer while illuminating a larger area of the sample. This might seem a useful way to get a spectrum from a small particle or interface. However, the image seen on the TEM screen is from the region of the EELS spectrum that has the most electrons, which for a thin sample is the zero loss. The TEM objective focuses the electrons that have lost energy at a different location due to its chromatic aberration (Cc). This means the observed image at the energy loss in the spectrum comes from a different area of the specimen.

In TEM Imaging mode, the spectrum's intensity can be easily modified by changing the illumination focusing and/or the size of the condenser aperture. The selected specimen area is directly visible on the viewing screen. It can change by translating the specimen, shifting the image electronically, adjusting the microscope magnification, and modifying the spectrometer entrance aperture. The collection efficiency can be very high in this mode. These characteristics make the TEM Imaging mode ideal for the initial examination of any specimen.

Quantitative analysis, however, is made nearly impossible in the TEM Imaging mode by the fact that electrons of different energies are spread over areas of different size in the image. If the illumination focuses on a small area or the specimen is not homogeneous, the intensity ratio between low-energy edges and high energy edges can be changed by as much as a factor of 10 in the imaging mode by merely changing the focus of the objective lens. As a consequence, attempting to quantify the specimen composition without taking account of this effect is likely to be highly spurious. It is, therefore, almost always better to collect spectra for quantitative analysis in the diffraction mode.

An exception occurs when the illumination is highly defocused (over tens of microns), the specimen is homogeneous over tens of microns, and the size of the illuminated area on the viewing screen is much larger than the spectrometer entrance aperture. In this case, the electrons that miss the spectrometer entrance aperture due to chromatic aberration are replaced by a similar number of electrons that enter the aperture in error also due to chromatic aberration. Operating under these conditions is sometimes useful on contamination-prone specimens, or when one needs to spread the illumination over a large area to minimize radiation damage while maintaining some spatial resolution or a high collection angle.

3.3.2 TEM diffraction

With the TEM in diffraction mode, a diffraction pattern is formed on the viewing screen, while the projector crossover contains an image of the illuminated area of the specimen. Because the GIF projects this image onto the detector, this mode is sometimes called, again somewhat confusingly, "image-coupled" mode. The magnification of the image in the projector crossover is related to camera length by

$$M = h/L$$

And the diameter D_p in eV at the slit of the projector crossover is therefore determined by

$$D_p = (h \cdot d_S)/L \cdot K_S$$

where

- d_S is the diameter of the illuminated specimen area
- h is the distance from the projector crossover to the GIF entrance aperture
- L is the camera length at the GIF entrance aperture
- K_S is the spectrometer energy dispersion and magnification factor or 0.3 eV/µm at 200 kV

For example, a 3 µm diameter-illuminated specimen area gives rise to a 3 µm projector crossover size for L = 70 cm and h = 70 cm. This permits better than 1 eV energy resolution at 200 kV. The TEM Diffraction mode, therefore, makes possible acquiring spectra from large areas. Although you should carefully monitor the size of the illuminated area when using small camera lengths if the energy resolution is not to worsen appreciably.

In the TEM Diffraction mode, the angular acceptance range of the spectrometer is limited by the spectrometer entrance aperture. It can be varied by changing the camera length or selecting a different aperture size. The diffraction pattern visible on the viewing screen makes it possible to monitor the specimen phase, thickness, and diffraction condition. Assuming that no selected area diffraction aperture is used, it collects the spectrum from the whole illuminated area of the specimen, and the spatial resolution is therefore determined purely by the probe-forming performance of the microscope. In this case, unlike in imaging mode, chromatic effects do not appreciably change the collection efficiency of the spectrometer in the diffraction mode, making quantitative chemical analysis of small sample areas much more reliable.

The diffraction mode is ideal for acquiring the high-quality spectra necessary when looking for low-concentration elements, or when performing EXELFS analysis. It is also beneficial when closely monitoring the diffraction condition and/or collection angles, e.g., in channeling experiments.

3.3.3 STEM

In a correctly set up STEM mode, the diffraction pattern displays on the viewing screen, and the projector crossover contains an image of the probe. Electron-optically, this mode is similar to the TEM Diffraction mode.

In STEM mode, it is easy to image a large area of the sample even though the illumination is focused into a small probe and to position the probe accurately on the feature of interest. This makes the STEM mode ideal for examining small areas of the sample (e.g., precipitates, interfaces, cell membranes).

In modern probe-corrected STEM systems, the convergence angles can be many 10s of milliradians, making the bright-field disk very large. To collect the entire direct beam plus the energy-loss signal, very short camera lengths are required. This can compromise the energy resolution of the system since the crossover size is inverse to the camera length.

3.4 Selecting an energy loss

In both imaging and spectroscopy, it is possible to shift between different energy losses by moving the spectrum at the energy-selecting slit. This can be done by adjusting the:

- Prism current
- Voltage on the drift tube
- TEM high voltage

Each method is discussed below.

3.4.1 Adjust the prism current (Shift)

This approach adjusts the current through the magnetic prism. The disadvantage of this method is that the absolute energy of the electrons observed will change and the image will go out of focus due to the

chromatic aberration of the TEM objective lens. Refocusing the objective lens is tedious and extremely difficult for the faint inner shell-loss images.

It's most useful to apply the prism current energy shift in TEM diffraction or STEM, where you can reliably examine the area of interest on the specimen using this method. Another disadvantage of using the prism current energy shift is the small hysteresis that can be observed when the current is varied over a large range. Therefore, we do not recommend using this method for precision edge onset measurements.

Shift using the magnetic prism does allow very large energy offset, however, and must be used when the offset required exceeds the drift tube voltage.

3.4.2 Adjust the voltage on the drift tube (drift tube offset)

The second method applies a voltage to the electrically isolated drift tube through the magnetic prism. In response to such an applied voltage, the electrons accelerate by a corresponding number of eV while they traverse the drift tube giving less deflection in the prism. This method has the same disadvantage as the previous one in that the absolute energy of the electrons used for imaging will change, the objective lens needs to be refocused, and it is not suitable for EFTEM.

The advantages of this method are its fast response (<10 µs), its accuracy, and the absence of hysteresis; for focused probe work, it does not affect the probe on the specimen as change focus or HT would.

For spectroscopy applications where a high accuracy edge onset measurement is required, we recommend applying energy offsets up to 2000 eV with the drift tube voltage. Energy offsets above 2000 eV can be achieved by combining prism current (energy shift) and drift tube control.

3.4.3 Adjust the TEM high voltage (HT offset)

The third way of selecting an energy loss is by changing the primary energy (high voltage) of the microscope. Let us assume that the zero-loss peak is in the center of the energy-selecting slit, and the imaging filter is selecting electrons of the primary energy E_0. If the primary energy then increases by E_1, the new image still forms with electrons of energy E_0 defined by the prism current and energy selecting slit. Thus, the electrons making it through the slit have lost precisely E_1. Since the absolute energy of the imaged electrons in the objective lens has not changed, it does not need to be refocused.

The change in the primary energy results in a change of specimen illumination focus (e.g., size of the illuminated area), but this is automatically compensated by a small adjustment of the last condenser lens.

Most TEMs are designed to raise the HT up to 3 kV for this purpose.

4 Installation

IMPORTANT
Only a trained Gatan Service Representative can install the Continuum system. See the Gatan website for a service office nearest you.

4.1 Software installation

All required software to operate the Continuum is preinstalled on the system computer. Gatan supplies backup copies of the installation software, license files, and factory checkout data with the system.

It may be necessary to install software upgrades during the life of the system. Due to the complex nature of the overall system, we recommend conferring with Gatan Service when performing any software upgrades. For details of the installation process, please see the PDF document **GMS Installation Guide** included on the GMS Installer.

The minimum version required for the GIF Continuum K3 is GMS 3.4.2. The minimum version of GMS for a 1067HD is 3.5.2.

5 Getting started

After a Gatan-authorized engineer installs the Continuum, the system is ready for use. All setup and calibration for routine work should be complete.

As a significant extension of the TEM/STEM electron-optical column, a GIF and its specialized detector system add many new imaging and analytical capabilities to the host microscope. Most of these techniques require specific system setups plus special data acquisition and analysis software.

Rather than covering the entire gamut of EELS and EFTEM techniques, please visit www.eels.info for instructions on how to set up and operate your TEM-GIF system to perform data acquisitions that form the basis for almost all EELS and EFTEM experiments:

- Zero-loss EFTEM imaging
- Core-loss EFTEM imaging
- EELS acquisition in TEM image mode
- EELS acquisition with simultaneous STEM imaging

For details on more advanced techniques (e.g., EFTEM Spectrum Imaging, STEM EELS Spectrum Imaging, or EFTEM Tomography), please refer to the user manuals provided with the technique-specific software packages. To view these manuals, open the **DigitalMicrograph software**, select the **Help** menu, then use the **Search (F1)** function. Alternatively, you can use the following shortcut to access the documentation directory on the desktop (C:\ProgramData\Gatan\Documentation).

6 Care and maintenance

6.1 General precautions

Very little is required to maintain the Continuum. The following are some general precautions users should adhere to in the care and maintenance of their Continuum system.

- Warm detectors and shut off the system before venting the detector system. Always vent to dry air or nitrogen.
- Operate only in a clean and dry environment.
- Regularly check air inlets on electronics for blockage and dust accumulation. Clean with a dust-free vacuum if necessary.
- Regularly check water flow and quality.
- Perform a regular backup of the computer system. In particular, the Gatan preferences folder: **C:\ProgramData\Gatan\Prefs**.
- Avoid changing the electromagnetic environment around the system. Time-varying fields may affect the system's resolution. A change in the DC field may require a Gatan Service visit to realign the system.
- Regularly run the detector heat cycle for the system cameras.

6.2 Electron optics

The system settings are stored on the computer and reload on startup. Regular user-level tuning should set the system in its optimal state. However, mechanical changes or an extensive re-alignment of the lower TEM column may require a Gatan Service visit to bring the system back into specification.

6.3 Detector system

The following are some general precautions users should adhere to in the care and maintenance of their camera.

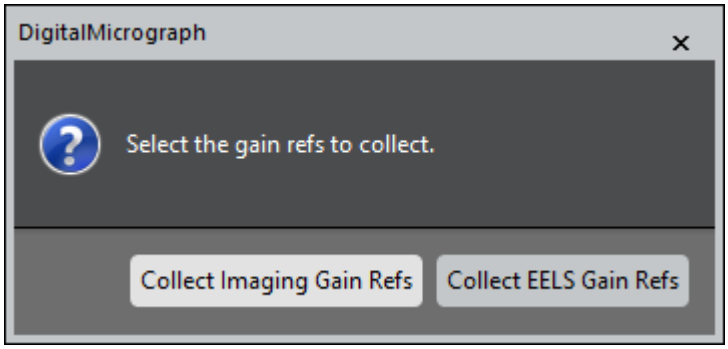


Figure 3: ***During gain reference collection, choose either imaging or spectroscopy modes from the posted dialog.***

6.3.1 Image/spectrum quality (Reference images)

The quality of the reference is key to spectral and image quality. We recommend updating the detector gain reference images regularly (weekly to daily), and always after sensor annealing (see below). Reference images for spectroscopy are recorded in a separate mode from imaging. Choose the correct reference type from the posted dialog (Figure *3*). Since this requires uniform illumination on the sensor, it is best to acquire the EELS gain reference while still in EFTEM mode and after initial tuning of the electron optics. Please see the K3 or Continuum detector manuals, plus the online help for additional instructions.

6.3.2 Camera cooler precautions

IMPORTANT
The camera must be warmed before venting the detector chamber or anytime the vacuum will be static for greater than ~ 1 h.

The camera should always be operated in the cooled state. Check the camera monitor to observe the camera temperature and health status.

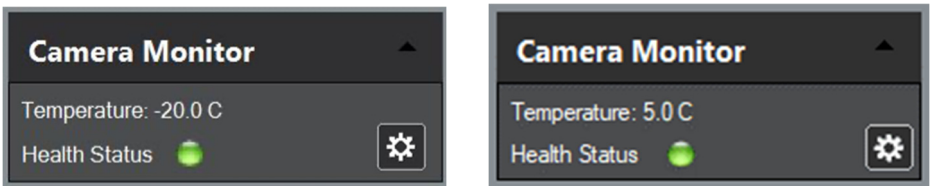


Figure 4: ***The K3 (left) and GIF (right) Camera Monitor provides simple confirmation the camera is running optimally. Use the Setup button to the lower right to access more detailed information.***

The K3 operating temperature is -20 °C. The fiber-coupled GIF camera operates at +5 °C. In both cases, the camera cooler may overheat if the water flow is disrupted or reduced. Once water flow is reestablished, the camera needs to be power cycled to reset the cooler. Shut down the **GMS** software, power off the camera for **10 s,** then **restart**, wait at least **30 s** before starting the GMS software. After the camera is stable, acquire fresh gain references for the best quality images and spectra.

Check the flow of cooling water periodically. If the flow rate of the cooling water deviates significantly from the value originally set, make sure the lines are not obstructed and adjust the pressure regulator to bring the flow back to the original level.

6.3.3 Sensor Maintenance and precautions

IMPORTANT
Minimize exposing the detector to the electron beam when the camera is not in use.

Minimize exposing the detector to high beam intensity when acquiring diffraction patterns or spectra.

K3 camera

The K3 camera has a theoretical saturation limit of 15 000 e^-/pix/s in linear mode. Avoid beam conditions that give rise to this level of brightness on the sensor. An additional layer of protection is provided by dynamic sensor protection (DSP), which automatically and continuously monitors beam intensity across the sensor. If a single pixel level exceeds the DSP threshold, the K3 camera automatically retracts. In general, most K3 experiments are performed in electron counting mode. Here, the target operating dose rate is ~40 e^-/pix/s, meaning the chance of overexposure or sensor DSP trip events in counting mode is low.

To provide enhanced utility for high-dynamic-range signals, e.g., those encountered during EELS or electron diffraction experiments, the K3 camera control has a control parameter **Live Time** in addition to the View Time. The K3 operates in rolling shutter mode and so always runs at the maximum frame rate of the chosen operating mode. Live Time enables the fast shutter to remain closed for part of a frame acquisition, which provides beam attenuation that enables the capture of bright features such as the zero-loss peak or diffraction spots without causing sensor damage.

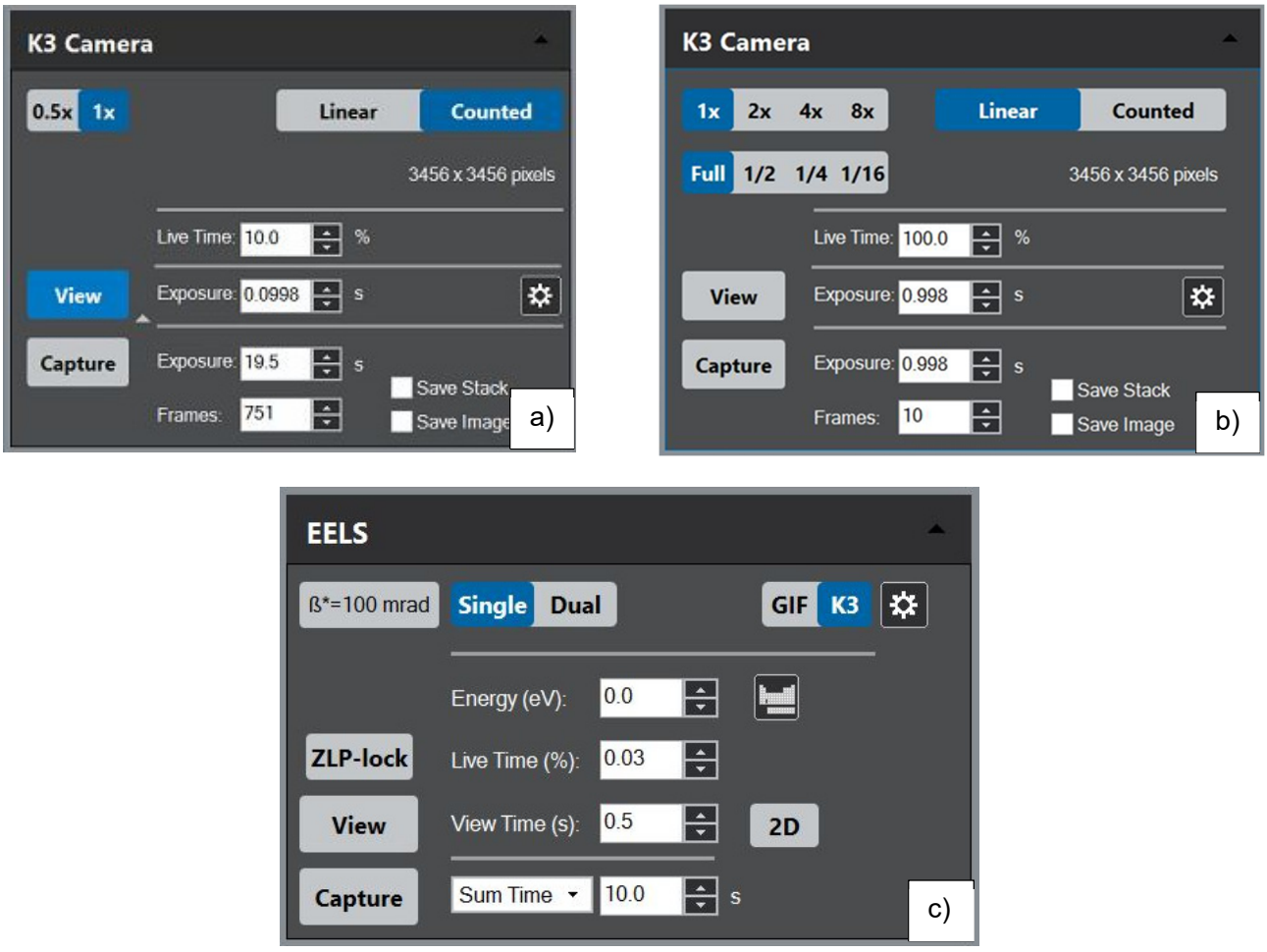


Figure 5: ***K3 detector control palette for standard K3 on a 1069 (a), the K3 IS on a 1069 (b) and K3 for EELS on a 1069 or a 1067HD with the EELS option. 1067HD's will show a size of 5760 x 4092 rather than the smaller size shown for the 1069 here but otherwise the palettes are the same. The Live Time determines the fraction of time the shutter is open during each hardware frame. The View or Capture Exposure Time determine the number of hardware frames summed to give a single image or spectrum.***

Live Time is available in both imaging and EELS modes (Figure 5). A Live Time of 100% means that the electrostatic shutter is fully open, and there is no beam attenuation. The minimum Live Time means the electrostatic shutter pulse-width is giving the maximum beam attenuation.

As a general rule, start at the minimum Live Time and incrementally increase until you achieve the target intensity. Then increase the View Time to sum frames until you reach the target signal-to-noise ratio.

Increasing or decreasing the View Time does not affect image or spectrum saturation. Saturation is only affected by changing Live Time.

Regularly anneal the K3 sensor to increase the sensor lifetime (Figure 6). This is particularly important for high brightness applications such as electron energy loss spectroscopy or *in situ*. To perform a heat cycle, set the camera temperature >25 °C to open the heat cycle dialog. We recommend an anneal cycle of 12 h at 50 °C at least once per week.

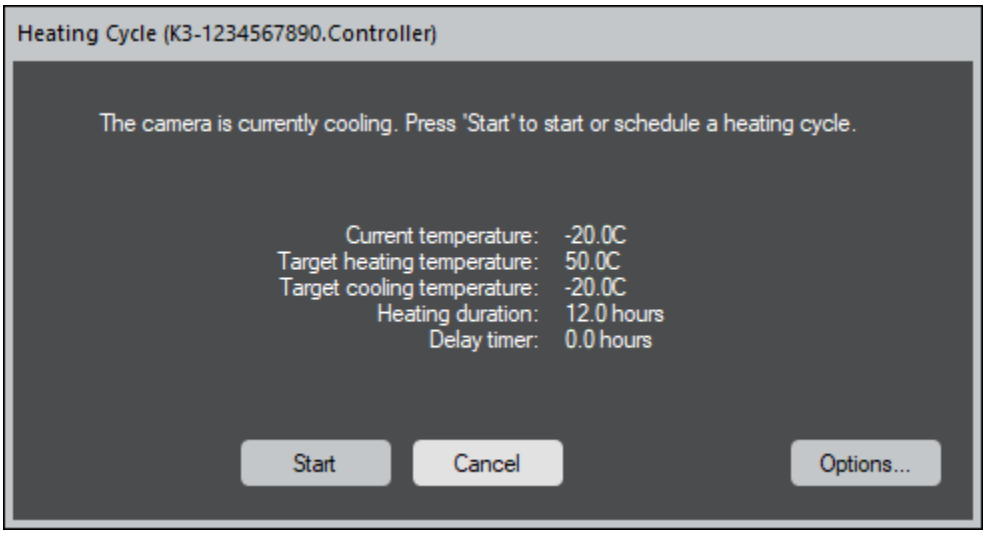


Figure 6: ***K3 camera heating cycle. Use the heat cycle anneal out trapped charge on the sensor and evaporate volatile contaminants. Acquire fresh gain and dark references after running a heat cycle.***

Continuum camera

It is important to protect the scintillator camera from being unnecessarily exposed to an intense unscattered beam. As a general rule, if you avoid conditions that would saturate any part of the sensor area in less than 0.1 s, the sensor lifetime should exceed the lifetime of the rest of the system. On the other hand, each time any sensor pixels are intensely saturated, the sensor efficiency may be permanently impaired for those pixels, resulting in a variation of the sensor efficiency across the image field.

If you think you have damaged the scintillator, take an unprocessed image of uniform illumination. If the spot turns out dark, you may have damaged the scintillator! If it is bright, spread out the beam and set the illumination, so you have near saturation for a 0.1 s exposure. Continue to expose the scintillator for 3 min and take another picture. The bright area should be gone, and the scintillator should operate normally.

Warm the scintillator periodically. Some microscopes have a significant vapor pressure of diffusion pump oil in the viewing chamber. Since the camera is colder than its surroundings, it can act as a cold trap that accumulates an oil film. While this thin film does not affect imaging, if it becomes thicker or coalesces into droplets, an increasingly stronger gain correction may become necessary.

Other problems caused by the oil film are charging and the sticking of dirt, which could otherwise be blown off easily. If left for too long, the hydrocarbons chains in the oil may be cracked by the electron beam, making them nonvolatile and un-dissolvable. Then it is necessary to send the camera back to have the scintillator serviced by Gatan. Fortunately, these problems can be avoided by adhering to the practice described below.

If you have a clean vacuum in the camera chamber area, warm the scintillator once or twice a month to 35 °C. But if you have back-streaming from your diffusion pump, you need to do it once a week. The camera should only be warmed for 15 h or overnight, but not longer. If the camera is used sufficiently without warming, the electron beam can harden the trapped oil making the scintillator challenging to clean.