



## The GIF Quantum, a next generation post-column imaging energy filter

Alexander Gubbens\*, Melanie Barfels, Colin Trevor, Ray Twesten, Paul Mooney, Paul Thomas, Nanda Menon, Bernd Kraus, Chengye Mao, Brian McGinn

Gatan Research and Development, 5794 W. Las Positas Blvd., Pleasanton, CA 94588, USA

### ARTICLE INFO

#### Keywords:

Electron microscopy  
Electron energy-loss spectroscopy  
Energy-filtered imaging  
Post-column imaging filter  
Spectrum imaging  
ESI

### ABSTRACT

We describe a new post-column imaging energy filter for (scanning) transmission electron microscopy from 60 to 300 kV operating voltage. The completely redesigned GIF quantum has a gradient magnetic prism, dodecapole optics, a 10-times faster 40 Mpixel/sec CCD camera, a 1  $\mu$ s electrostatic shutter, and new user interface, control, and auto-alignment software. An 8 dodecapole lens system, performs full 2nd and 3rd, and partial 4th and 5th order aberration correction. The improved aberration correction has allowed the size of the entrance aperture to be nearly doubled to 9.0 mm compared to current generation post-column designs. The electrostatic shutter provides exposure control down to 1  $\mu$ s, extending the exposure time range to over 7 orders of magnitude. Spectroscopy operation has been improved with a larger 2 keV field of view at 200 kV, and a maximum acquisition rate of 1000 spectra per second. A high-speed DualEELS mode simultaneously acquires core- and low-loss spectra up to 2 keV apart. A more intuitive user interface includes new capabilities such as automated exposure control and optimized full spectrum acquisition. The auto-alignment software has been significantly enhanced to use the full flexibility of the dodecapole lens system.

© 2010 Elsevier B.V. All rights reserved.

### 1. Post-column imaging filters to date

To date, post-column imaging filters have been based on the design of the GIF200 introduced in 1992 [1]. These filters introduce energy dispersion by bending the electrons over 90° with a 100 mm radius magnetic prism. Two post prism quadrupoles magnify and focus the spectrum onto an energy-selecting slit, and 4 post-slit quadrupoles project either an energy-filtered image or an energy-loss spectrum onto a CCD camera. The prism's tilted and curved entrance and exit faces, in combination with a quadrupole and sextupole before the prism, correct the important 2nd order aberrations in the spectrum. 5 sextupoles in the post-slit optics correct the important 2nd order aberrations in the energy-filtered image. (See Table 1 for a list and a definition of various aberrations.)

Later GIF generations made changes to both the electron-optics and the CCD camera.

The GIF2000, introduced in 1997 [2], replaced the GIF200's 1 k × 1 k CCD camera, with a 2 k × 2 k CCD camera, increased the filter's magnification 2 times, and added an octupole to the post-slit optics to control a new 3rd order aberration.

Abbreviations: (S)TEM, (scanning) transmission electron microscopy; EELS, electron energy-loss spectroscopy; EFTEM, energy filtered transmission electron microscopy; ESI, electron spectroscopic imaging

\* Corresponding author.

E-mail address: [sander.gubbens@gatan.com](mailto:sander.gubbens@gatan.com) (A. Gubbens).

The GIF Tridiem, introduced in 2003 [3], added a further sextupole, 2 octupoles, and a decapole in front of the prism to correct the two 3rd order aberrations, and the dominant 4th order aberration in the spectrum. The improved aberration correction allowed the entrance aperture to be increased from 3.0 to 5.0 mm diameter while keeping the isochromaticity (the energy variation across the field of view) the same. Improvements in the stability and noise of the prism current supply allowed the Tridiem to reach an energy resolution better than 100 meV FWHM for the 1.0 mm diameter entrance aperture [4,5]. The Tridiem also incorporated the 4 port, 1 MHz UltraScan 1000 camera with an added frame transfer mode unique to the Tridiem, delivering both higher resolution and higher speed.

### 2. Need for a next generation GIF

While the GIF Tridiem series was very successful and productive, a number of considerations led us to design a dramatically improved imaging filter:

- *Field of view and transmissivity*—A post-column filter's largest field of view is limited by the magnification from the entrance aperture to the CCD camera. For a given CCD size, a larger entrance aperture will result in an increased field of view. For the Tridiem, the largest field of view depends on the TEM model but is typically around 20  $\mu$ m. The size of the entrance aperture is limited by the desired image isochromaticity in eV. A filter's

**Table 1**

Overview of the important performance parameters of the GIF quantum compared with earlier GIF generations. The aberrations are expressed in transport notation [12]. *Tijk*, *Uijkl*, *Vijklm*, and *Wijklmn*, respectively represent 2nd, 3rd, 4th, and 5th order aberrations. *i*=1 indicates an aberration in the *x* position (energy dispersive direction), *i*=3 an aberration in the *y* position. *j*, *k*, *l*, *m*, or *n*=2 represent a dependence on the angle in the *x*-direction, =4 on the angle in the *y*-direction, and =6 on the fractional momentum deviation.

	GIF200	GIF2000	GIF Tridiem ERS	GIF quantum ERS
Year of introduction	1992	1997	2003	2009
Operating range (kV)	80–400	80–400	80–400	60–300
Bend radius (mm)	100	100	100	75
Dispersion at slit ( $\mu\text{m}/\text{eV}$ )	5.0	5.0	5.0	2.5
<b>EFTEM</b>				
Entrance aperture ( $\emptyset$ mm)	3.0	3.0	5.0	9.0
EFTEM field of view (approximate, $\mu\text{m}$ )	12	12	20	36
Aberration correction in the image	T122 T144 T126 T324 T346	T122 T144 T126 T324 T346 U1446	T122 T144 T126 T324 T346 U1446	T122 T144 T126 T324 T346 U1244 U1446 U3444
Isochromaticity (max, eV)	< 2.0	< 2.0	< 2.0	< 2.0
Distortion max/rms (%)	1.5/1.0	1.5/1.0	1.5/0.75	0.75/0.5
<b>EELS</b>				
Aperture size at best resolution (mm)	0.6	0.6	1.0	2.5
Filter energy resolution (meV)	< 400	< 400	< 40	< 40
EELS field of view (200 kV, eV)	1024	1024	1024	2048
Aberrations corrected in the spectrum at the energy-selecting slit	T122 T144 T126	T122 T144 T126	T122 T144 T126 U1222 U1244 V12244	T122 T144 T126 U1222 U1244 V12244 W124444
Electrostatic shutter	X	X	X	✓
Minimum exposure time (ms)	50	50	50	0.001
<b>CCD CAMERA</b>				
Camera size (pixels)	1 k × 1 k	2 k × 2 k	2 k × 2 k	2 k × 2 k
Pixel size ( $\mu\text{m}$ )	24	24	14	14
Number of outputs	1	1	4	4
Read-out speed (Mpixel/s)	0.4	0.4	4.0	40.0
Frame rate 1 k × 1 k (fps)	0.4	0.1	2.5	15
Spectral speed (Hz)	10	5	30	1000

transmissivity at a given isochromaticity is effectively a measure of the collection efficiency and scales with the square of the entrance aperture. Field of view, isochromaticity, energy resolution, and transmissivity are therefore tightly linked. For the same isochromaticity, more complete aberration correction in the energy-loss spectrum will allow a larger entrance aperture, a larger maximum field of view, and a higher transmissivity.

- **Spectral speed**—Post-column filters so far have been limited to a maximum of 30 spectra per second. High brightness guns in combination with modern aberration corrected probe-forming optics have allowed significantly more current to be focused into a small probe [6,7] and atomic resolution core-loss spectrum imaging at a 1 ms exposure time per pixel is in principle possible if detectors were able to operate at the same speed.
- **Spectral field of view**—Many important edge energies often span more than 1 keV of energy loss (e.g., the O–K and Si–K edges are separated by > 1.3 keV). However, post-column filters have till now had a limited field of view of 1 keV at 200 kV operating voltage. This limitation follows from the large dispersion at the energy-selecting slit, which at 5  $\mu\text{m}/\text{eV}$  at 200 kV limits the size (in eV) of the spectrum that can cleanly be passed through the optics to the CCD detector.
- **Sub 100 kV TEM**—Increase in interest in the high-resolution study of nano-materials and light elements is driving microscopy to operate voltages below 100 kV in order to minimize radiation damage, in particular knock-on [8]. This trend has

been helped by the availability of advanced aberration corrected optics allowing atomic resolution to be obtained at operating voltages as low as 60 kV.

- **Dynamic range**—For quantitative EELS, it is necessary to capture the core-loss region of interest together with the zero loss peak and plasmon losses. The intensity range of an energy-loss spectrum exceeds that of a 16-bit CCD camera and cannot be captured in a single acquisition. Optimum and efficient acquisitions of EELS spectra, particularly in the low-loss regime, require a faster shutter (e.g. electrostatic) and a more flexible way of reading out the CCD camera [9–11].
- **User friendliness**—EELS and EFTEM have never been easy techniques and optimal results required a skillful, experienced operator with considerable knowledge of the instrumentation involved. With the right improvements in instrumental performance and more powerful software, it should be possible to automate optimized data acquisition allowing the user to concentrate on science rather than instrumentation.

With these considerations we set out to design the GIF Quantum with the following goals.

1. Open up the entrance aperture as much as possible without compromise to any of the imaging and spectroscopy specifications (isochromaticity, energy resolution, image distortion, etc.).

Download English Version:

<https://daneshyari.com/en/article/1678050>

Download Persian Version:

<https://daneshyari.com/article/1678050>

[Daneshyari.com](https://daneshyari.com)