338
doi:10.1017/S1431927610055777
Microsc. Microanal. 16 (Suppl 2), 2010

Fast Mechanical Shutter in Hitachi HF 3300, a 60 kV to 300 kV TEM

Marek Malac*, Robert A. McLeod*, Peng Li*, Wilbur C. Bigelow•, Jane Howe°, Larry Allard°, Yoshifumi Taniguchi▲, M. Sergio Moreno■.

* National Institute of Nanotechnology, 11421 Saskatchewan Drive, Edmonton, Canada.
• Materials Engineering, University of Michigan, Ann Arbor, MI 48109-2136.
° Oak Ridge National Laboratory, Oak Ridge, Tennessee.
▲Hitachi High Technologies Corporation, Hitachinaka-shi, Ibaraki-ken, 312-8504, Japan.
■Centro Atomico Bariloche, 8400 – San Carlos de Bariloche, Argentina.

The detection of electrons in a TEM is typically performed by means of a slow scan CCD camera (SSCCD), film, or image plates. In many advanced applications the ability to reliably and quickly shutter the beam incident onto a detector determines whether usable data can be obtained or not. Various approaches were explored over the years to shutter the beam either above or below the sample. Here we report on the performance of a mechanical shutter in an HF 3300 at NINT. This type shutter was originally designed by one of the authors (WB) for the Hitachi HF 2000 cold field emission microscope in 1997. The simple and reliable design is a drop-in replacement for the factory installed shutter, either above or below sample, or for both.

As shown in Fig 1, the shutter. consists of a titanium barrel with four slits mounted in the beam path with the long axis perpendicular to the electron beam such that it intercepts the beam at a suitable position along the electron beam path. The barrel is rotated 45 degrees to change between "open" and "closed" positions. The rotation is achieved by a stepper motor mounted outside the microscope and triggered by the usual signal for the SSCCD camera (Gatan Ultrascan 1000 in case of the HF 3300 at NINT). Fig. 2 illustrates the shutter performance. The x-axis shows the nominal acquisition time on the SSCCD while the y-axis shows the number of counts summed across the entire camera. The shutter has linear response down to about 13 ms acquisition time (the readout time of the SSCCD is not affected). For shorter exposures the time required for the shutter blades to cut off the beam becomes sufficient to allow a constant residual exposure. It is likely that the acquisition time can be further decreased by tuning the program of the stepper motor controller; however, this seems unnecessary because about 20 ms acquisition time is entirely adequate for all experiments we attempted so far.

Fig. 3 a) shows is a diffraction pattern (DP) acquired from a thick (as witnessed by the Kikuchi-lines) Si <110> sample using the shortest reasonable exposure time for the factory-installed shutter. The streaking of the diffraction spots and saturation of the main beam spot prevent reliable interpretation of this pattern. Fig 3b) shows a DP acquired from gold nanorods with less than 20 nm thickness on amorphous Si substrate about 5 nm thick using the new shutter and about one fifth the exposure time. Here the 000 beam is not saturated and the diffraction spots are sharply defined, allowing this DP to be reliably interpreted

Fig 4a) shows an angular-resolved EELS spectrum acquired from a carbon support film via the Gatan Tridiem spectrometer using the spectrometer's internal shutter with a 100 ms acquisition time. The streak artifact (marked by an arrow) indicates that sweeping of the beam away from the SSCCD occurred for a significant fraction of the exposure. The streak artifact is in the energy-loss region of interest for valence loss EELS such as band-gap determination. Indeed, when a vertically summed spectrum is acquired (as done when using Digital Micrograph Turbo View mode) the shutter streak can be easily misinterpreted as the tail of zero-loss, or as transitions in the low loss region, rendering the spectrum useless. Fig 4b shows an angular-resolved EELS spectrum acquired with the new shutter from a TiOx nanobelts [1]. Clearly the streaking is not present. We have used a slightly longer acquisition time in b) to run the SSCCD near saturation. A logarithmic color scale was used in b) to ensure that no shutter artifact was present in this spectrum, whereas in a) a linear color scale is sufficient to show the artifact generated by the Gatan shutter. It should be noted that shutters that can operate with acquisition time in the order of nanoseconds can be built using electrostatic deflectors [2]. However, they require careful design of the control electronics and require voltages on the order of 3 kV. On the other hand, the mechanical shutter described here is extremely easy to build and install and provides sufficiently short acquisition times for the highly demanding applications of recording diffraction patterns and spatial- or angular-resolved EELS spectra from very thin samples.

References

[1] Moreno et al. *Microscopy & Microanalysis, Vol 15, Suppl.2, p.1340, 2009.*
[2] AJ. Craven et al, Ultramicroscopy 92 (2002) p. 165.

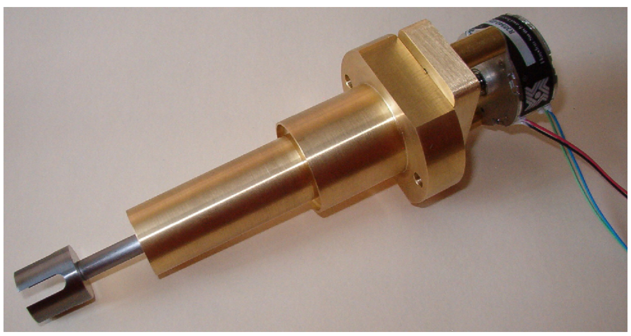

Fig. 1 Photo of shutter hardware. A titanium barrel with four slits driven by a stepper motor is used to block the beam.

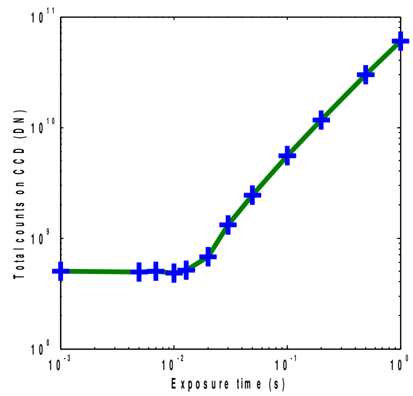

Fig. 2 Shutter response. The *y* axis is number of counts summed over the entire CCD while the *x* axis is the nominal camera acquisition time. The response is linear down to about 13 ms.

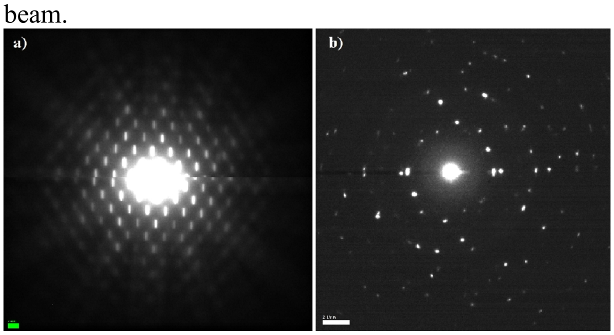

Fig 3 Diffraction pattern acquired with a) original pneumatic shutter (120 ms) on a Si <011> , and b) new mechanical shutter (18 ms) acquired on Au nanorods on SiO_2 membrane. The presence of streaking in a) makes quantitative interpretation difficult but is absent in b). The direct beam is saturated in a) but not in b) although b) is significantly thinner sample.

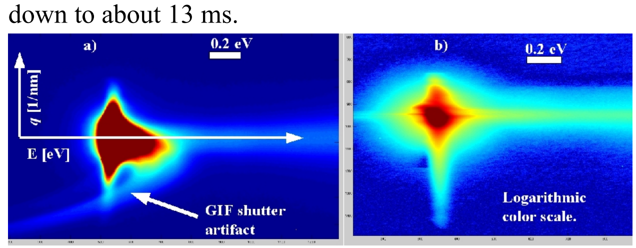

Fig. 4 a) (linear color scale) streaking, marked with an arrow, observed when using internal shutter of Gatan Image Filter in angle-resolved EELS mode with 100 ms acquisition time. b) (log scale) clean low loss region of angle-resolved EELS when using new mechanical shutter. Acquisition time was 200 ms. The energy dispersion in both cases was 20 meV/ch.

https://doi.org/10.1017/S1431927610055777 Published online by Cambridge University Press