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An Aberration-Corrected STEM for Diffraction Studies

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dedicated to the memory of Professor John Maxwell Cowley

John Cowley had far-ranging interests throughout his scientific career. But in his ASU years, two interests became preeminent: diffraction physics [1] and STEM (scanning transmission electron microscopy [2]). And in combining the two, he pushed both fields many miles forward.

John Cowley was the first to install a high-resolution polepiece in a 100 kV STEM (with C_s=1.3 mm), and the first to obtain better than 2 Å resolution STEM images. He was the first to appreciate the importance of diffraction effects in a STEM, the first to install a STEM diffraction camera, and the first to install a reconfigurable detector system [3]. He and his students and collaborators formulated the theory of high resolution imaging in the STEM [4], recorded the first Ronchigrams, and worked out the theory of their formation [5,6]. The VG STEM he procured and modified heavily was also used to record the first lattice-plane resolution EELS profiles [7].

At Nion, we are building a dedicated aberration-corrected STEM, which owes much to these concepts [8]. The aberration corrector is a 3rd generation Nion design, correcting all aberrations up to and including $C_{5,6}$ (6-fold astigmatism of fifth order) [9]. At 200 kV primary voltage, it is expected to give a probe smaller than 0.4 Å [10]. The column will also be optimized for diffraction studies, thanks to its principal features (described elsewhere in these proceedings [8]), plus the following features included specifically for diffraction studies:

> Flexible probe-forming optics: the pre-sample column is built in such a way that it will be possible to independently change the source demagnification and the probe convergence by changing excitations of lenses rather than by changing apertures, and to switch between different modes with minimized hysteresis and thermal drift. The expected range of available probe semi-angles spans from 0.5 mrad (useful for spot diffraction patterns and momentum-resolved EELS) to 50 mrad (for ultra-high resolution imaging and CBED studies).
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> Flexible post-sample optics: there are 2 post-sample coupling lenses, allowing the camera length to be varied and spiral and radial distortion in diffraction patterns to be minimized. Other lenses can be added later as needed, since the column is modular.
>
> Flexible scanning system. Double scan coils in the lower (entry side) bore of the symmetric condenser-objective lens are precisely mirrored by the descan coils in the upper bore. The resultant 4 layers of scan/descan coils can be driven very flexibly by a newly developed scan generator. Scan/descan, rock/derock, square, conical or other-mode rastering will all be supported. The resultant new capabilities are expected to include conical rock/derock scans such that the motion of the small probe on the sample is minimized even with conical semi-angles in excess of 50 mrad. This will give diffraction patterns with many advantages in terms of ease of interpretation over regular point diffraction patterns [11].

The new column is now largely constructed and is being tested, using a beam generated by a 100 kV VG gun. (The 200 kV gun is being developed in a separate laboratory and should be available shortly.) A Ronchigram of graphitized 3.4 Å fringes as it appeared within minutes of us sending the beam through the objective lens for the first time is shown in Fig. 1. The microscope was resting on the floor with no suspension system in place, and the room was rather noisy both acoustically and electrically. The fringes are very clearly visible with good contrast, showing that the stability of the new column and insulation from external disturbances has worked out very well.

Equally importantly, new software is being developed for the recording and analysis of diffraction patterns and Ronchigrams. This includes aberration analysis using crystalline samples, based on an automated analysis of fringe spacings in Bragg disk overlap regions. An example of a simulated pattern and a resultant profile of spatial frequencies of fringes as a function of position in the overlap region are shown in Fig. 2.

In summary, the new column and associated software should be well suited for diffraction analysis with small and ultra-small probes. It will also be a flexible instrument, ready for any new experiment or experimental configuration that an imaginative researcher can think of. We like to think that it will be precisely the kind of an electron microscope that John Cowley would have enjoyed using.

References

[1] J.M. Cowley, Diffraction Physics (third edition, Elsevier Science, 1995).
[2] J.M. Cowley, Ultramicroscopy **2** (1976) 3.
[3] J.M. Cowley and A.Y. Au, *in:* Scanning Electron Microscopy (IIT Research Institute, Chicago, ed. Om Johari) part I (1978) 53.
[4] J.C.H. Spence and J.M. Cowley, Optik **50** (1978) 129.
[5] J.M. Cowley, Ultramicroscopy **4** (1979) 413.
[6] J.M. Cowley, Ultramicroscopy **7** (1981) 19.
[7] J. C. H. Spence and J. Lynch, Ultramicroscopy **9** (1982) 267.
[8] O.L. Krivanek et al., these proceedings
[9] N. Dellby et al., these proceedings.
[10] O.L. Krivanek et al., Ultramicroscopy **96** (2003) 229.
[11] C.S. Own et al., submitted to Acta Cryst. A; also C.S. Own and L.D. Marks, these proceedings.

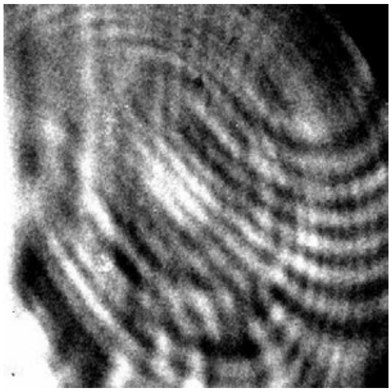

Fig. 1. Ronchigram of graphite 3.4 Å fringes recorded soon after the beam was first brought up through the new column.

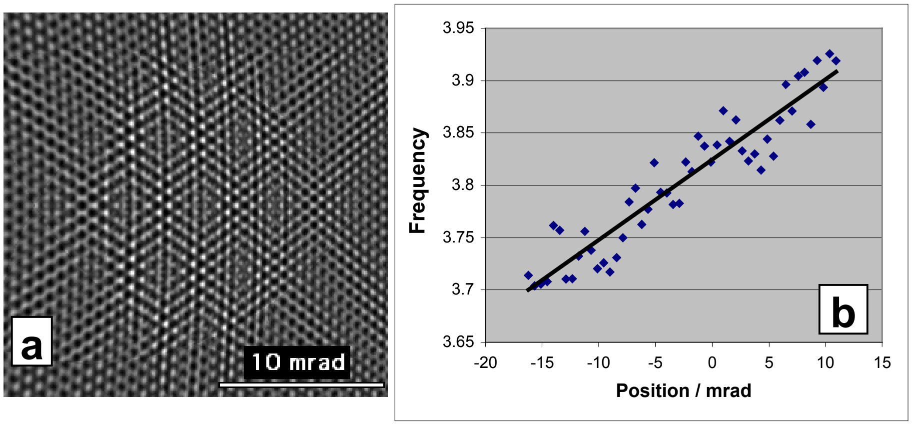

Fig. 2. a) Simulated Ronchigram from (110) silicon computed for $C_{1,0}$ (defocus) = -500 nm , $C_{2,1a}$ (coma) = 4 µm. b) Variation in the spatial frequency of the Ronchigram fringes along a horizontal line though (a), compared to the prediction for $C_{2,1a}$ = 4 µm.