

Advanced Instrumentation for High resolution Electron Microscopy

Max. Haider

CEOS GmbH, Englerstr. 28, D69126 Heidelberg, Germany, haider@ceos-gmbh.de

With the emergence of the first Cs-corrected 200 kV TEM in 1997 [1], and about two years later of the first Cs-corrected STEM [2], a new class of high resolution electron microscopes is now available. These Cs-corrected TEM and STEM have several distinct advantages compared with a conventional TEM and STEM. They are, for example, much less sensitive against beam tilt and the Cs-corrected TEM offers an easier image interpretation, due to the vanishing image delocalisation and a Cs-corrected STEM has either a higher beam current-about one order of magnitude- or a reduced electron probe size leading to an improved resolution. The main advantage of such a spherical aberration corrected electron microscope, its superior resolving power, is for most of the applications very important. In materials science the resolving power defines the achievable resolution with a certain specimen if one assumes optimum specimen preparation. The main imaging mode in TEM is phase-contrast where in STEM, with its probe forming system, the high angular dark-field mode is most often used for Z-contrast. The STEM is also a routine tool for analytical purposes due to the combination of high spatial resolution with EELS or X-ray analysis.

Since the spherical aberration coefficient C_s of the objective lens of a Cs-corrected 200 kV TEM is not a fixed value anymore for a given geometry of the objective lens this aberration coefficient can be treated as a free parameter in order to optimise the contrast transfer for a certain spatial frequency band. For example, one can optimise the contrast for imaging of light atoms [3] by choosing a value of $C_s = -40 \mu\text{m}$ which allows the imaging of object details in the range of about 1.0 \AA . In a TEM with a Hexapole corrector the total spherical aberration coefficient can be varied between the original value of the objective lens and a negative C_s generated by the two Hexapole-elements.

As shown in Fig. 1. the resolution of a Cs-corrected TEM is limited by the temporal coherence of the imaging part which is the product of $\Delta E/E * C_c$. Therefore, this damping envelope can be shifted further out to high spatial frequencies either by decreasing the relative energy width $\Delta E/E$ or by reducing the chromatic aberration. For the relative energy width one could either reduce ΔE or increase the primary energy E . When increasing E up to 1 MeV the spherical aberration can still be assumed as a variable and the benefits one can achieve with a Cs corrector are the same as,

for example, with a 200 kV TEM: no contrast reversal and a strong reduction of the point spread function.

The possibility to compensate the spherical aberration C_s , which is given by the design of the objective lens, offers the opportunity to optimise the objective lens in terms of space, for example, for an improved stage with high tilt angles or various temperature ranges. The accompanied problem, when increasing the pole piece gap, an increased chromatic aberration coefficient C_c , can be reduced to a large extent when using a monochromatic electron source. This could be a Schottky emitter in combination with a monochromator in order to reduce the energy width of the primary beam down to about 0.2 eV or to compensate for the chromatic aberration C_c .

The reduction of the energy width by means of a monochromator is not only advantageous for C_s -corrected TEMs but also for imaging techniques with numerical correction of spherical aberration- such as electron holography and focus variation and, very important, for electron energy loss spectroscopy (EELS) with high energy resolution. For the latter application a small energy width even down to about 100 meV with a large probe current has to be achieved.

The concept we followed for the development of a monochromator had been proposed by Kahl and Rose [4]. It is a purely electrostatic Ω -shaped energy filter with the energy selecting slit in the symmetry plane of this filter (s. Fig.2a). The image of the electron source in the dispersive plane is a line focus, several 10 μm in height depending on the size of the gun aperture. The dispersion cancels completely after the monochromator. The load-free bipolar voltage supply provides a superior reproducibility of the beam within the monochromator. Moreover it allows a fast and hysteresis free switch to turn it on and off.

The monochromator is incorporated in a UHV chamber above the accelerator and is driven at extractor potential of the Schottky-emitter. With a gun lens and its movable aperture the beam can be focused into the energy dispersive plane. The selectable gun-lens aperture allows the selection of the emission angle of the electron source, hence, the beam current. The energy selecting slit system consists of ten selectable slits with a width of from 0.5 μm up to 5 μm in 0.5 μm steps. The dispersion of the monochromator depends linearly on the extractor potential, which defines the energy of the electrons within the monochromator. At 4 kV extractor voltage the dispersion is 12.0 $\mu\text{m}/\text{eV}$ and, with the given width of the slit at this extractor setting the theoretical limit of the smallest energy width one might achieve is about 40 meV FWHM.

The monochromator has been constructed and incorporated into the accelerator UHV chamber. In a first step, the electron probe has been analyzed without acceleration in a low voltage SEM. For the measurement of the energy width a faraday cage has been designed with an

electrically isolated grid in front of it in order to decelerate the electrons. The energy resolution of this spectrometer has been measured to be 40 meV. The first results obtained with this monochromator are shown in Fig. 3. The measured energy width of the filtered beam is $\Delta E = 90$ meV FWHM, still having a beam current of about 1 nA. The aperture at the tip-potential is 5 mrad in this case. When using a smaller aperture (3 mrad) the filtered beam current was reduced to 0.36 nA and the energy width was measured to be $\Delta E = 81$ meV FWHM. This demonstrates a very good agreement with the specifications in the German SESAME project. Which assumed 200 meV for the filtered beam before entering the accelerator. This development has been carried out in collaboration with Zeiss/Germany for their new generation of advanced TEMs.

The second possibility to improve the point resolution is to compensate the chromatic aberration by means of a Cc/Cs-corrector. This would combine several advantages such as offering the opportunity to increase the pole piece gap - allowing, for example, various types of stages, in-situ observation and Lorentz microscopy- or to use larger energy windows when working with an in-column energy filter. For this Cs/Cc-corrector an electric-magnetic quadrupole system is the only feasible choice. Unlike a hexapole Cs-corrector a quadrupole corrector strongly alters the path of the paraxial rays.

The "minimum" quadrupole Cc-corrector requires two magnetic and two combined electric-magnetic quadrupoles arranged anti-symmetrically with respect to the mid-plane of the system in order to avoid a two-fold chromatic astigmatism \overline{Cc} . The correction of the spherical aberration can be achieved by additional octupole elements which act symmetrically on the path of rays. The path of the fundamental rays for each section is determined by the sum of the focusing and defocusing strength of the magnetic and electric quadrupoles. Hence, for each element a negative chromatic aberration is introduced in one section. The dependence of the refraction power on the beam energy is different for electric and magnetic quadrupoles, since only magnetic interaction depends on the velocity of the electron. For small energy deviations the change of the refraction power of a magnetic quadrupole is only half as large as in the case of an electric one.

Unfortunately, the minimum quadrupole corrector described above introduces strong off-axial aberrations. Therefore, it can be applied only in a low-voltage SEM-[5]. For the TEM more advanced designs with reduced off-axial aberrations have been proposed by Rose-[6] and we are investigating a new class of correctors based on a double-symmetric arrangement of electric-magnetic quadrupoles. For these highly symmetric systems the geometrical aberrations of third order can be controlled completely.

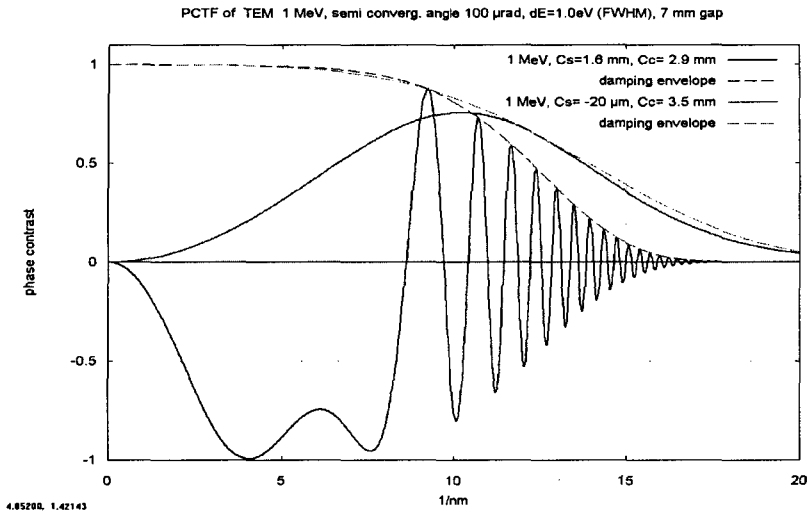


Fig. 1 Phase contrast transfer function for a 1 MeV TEM with 7 mm gap and a Schottky emitter. The PCTF of the uncorrected and the corrected TEM are shown and the gain of resolution and contrast improvement are due to the Cs-correction.

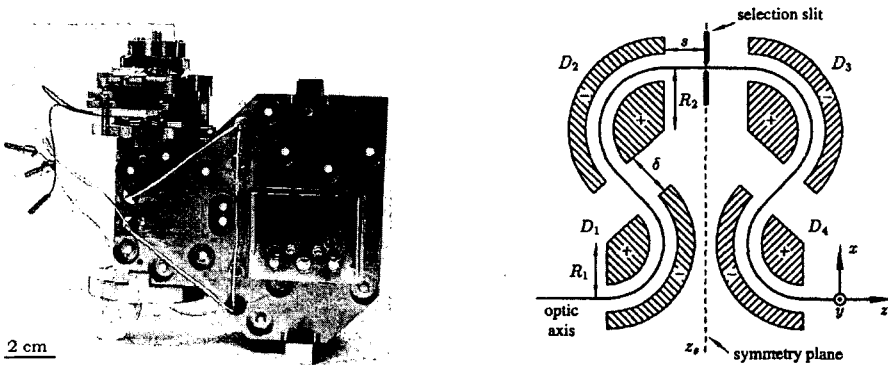


Fig. 2 a) Schematic drawing of the monochromator and b) the monochromator, turned by 90 to the right compared with the drawing, after finishing the construction.

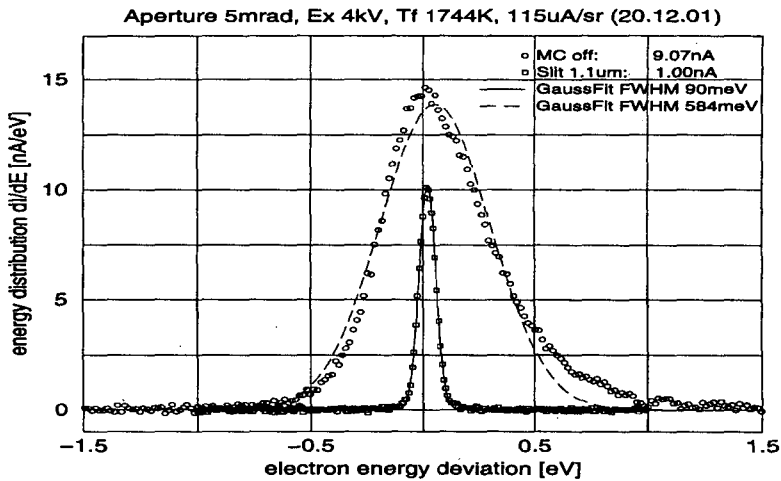


Fig. 3 Measured energy distributions of the Schottky emitter without (circles) and with filtering (squares). The energy slit was 1.0 m and the dispersion $12\mu\text{ m/eV}$.

References:

- [1] Haider M., et al, Nature 392 (1998) 768
- [2] Batson P.E., Delby N. and Krivanek O., Nature 418 (2002) 617
- [3] Jia C.L., Lentzen M. and Urban K., Science 299 (2003) 870
- [4] F. Kahl F. & H. Rose, Proc. EUREM Brno/Cz, Vol.III (2000) 1459
- [3] S. Uhlemann & M. Haider, Proc. M&M Vol. 8 Suppl.2 (2002) 584CD.
- [4] H. Rose, Ultramicroscopy, 93 (2002) 293.
- [5] J. Zach & M. Haider, Nucl. Instr. Meth. A363 (1995) 316.
- [6] H. Rose, private communication.