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First experimental test of a new monochromated and aberration-corrected 200 kV field-emission scanning transmission electron microscope

T. Walther[a,*], E. Quandt[a], H. Stegmann[b], A. Thesen[b], G. Benner[b]

[a]*Center of Advanced European Studies and Research (caesar), Ludwig-Erhard-Allee 2, D-53175 Bonn, Germany*
[b]*Carl Zeiss Nano Technology Systems GmbH, Carl-Zeiss-Str. 56, D-73447 Oberkochen, Germany*

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Abstract

The first 200 kV scanning transmission electron microscope (STEM) with an imaging energy filter, a monochromator and a corrector for the spherical aberration (C_s-corrector) of the illumination system has been built and tested. The STEM/TEM concept with Koehler illumination allows to switch easily between STEM mode for analytical and TEM mode for high-resolution or in situ studies. The C_s-corrector allows the use of large illumination angles for retaining a sufficiently high beam current despite the intensity loss in the monochromator. With the monochromator on and a 3 μm slit in the dispersion plane that gives 0.26 eV full-width at half-maximum (FWHM) energy resolution we have obtained so far an electron beam smaller than 0.20 nm in diameter (FWHM as measured by scanning the spot quickly over the CCD) which contains 7 pA current and, according to simulations, should be around 0.12 nm in true size. A high-angle annular dark field (ADF) image with isotropic resolution better than 0.28 nm has been recorded with the monochromator in the above configuration and the C_s-corrector on. The beam current is still somewhat low for electron energy-loss spectroscopy (EELS) but is expected to increase substantially by optimising the condenser set-up and using a somewhat larger condenser aperture.

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1. Introduction

Recent advances in transmission electron microscope instrumentation include monochromators for the electron sources, energy filters that can be used as either spectrometers or imaging filters and correctors for the spherical aberration of electromagnetic lens systems. Here, we describe tests of the first 200 kV field-emission gun (FEG) (scanning) transmission electron microscope (FEG-(S)TEM) with both a beam monochromator and an aberration corrector for the illumination system.

A monochromator narrows the energy spread of the primary electron beam, which enables better energy resolution in electron energy-loss spectroscopy (EELS) [1–5]. It also improves the temporal source coherence for imaging by minimising the defocus spread due to electrons of different primary energy [6] and thereby extends the information limit [7] attainable in high-resolution transmission electron microscopy (HREM). For transmission electron microscopes several different monochromator (MC) designs have so far been built, incorporated into existing microscope columns and tested. Examples include simple Wien filters [8], retarding Wien filters [9], double [10] and multi-pole Wien filters [11] and electrostatic Omega-type filters [4], all of which have been shown to decrease the width of the energy distribution of the electron emitter. However, the improvement of the energy resolution by incorporating such a MC between electron source and condenser system has typically compromised the ability of

*Corresponding author. Tel.: +49 228 9656 198; fax: +49 228 9656 116.
E-mail address: walther@caesar.de (T. Walther).

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the illumination system to focus the electrons into a small beam as necessary for nano-probe TEM or scanning transmission electron microscopy (STEM). Fig. 1a depicts the energy resolution attained in present TEM/STEM systems with different MC designs [1,12,13] as a function of the spatial resolution, which shows the typical trade-off between spectral and spatial resolution.

Imaging energy filters used in TEM or STEM may be divided into two classes, namely in-column filters such as the magnetic prism with electrostatic mirror filter [14], the electromagnetic Omega-filter [15–17], its aberration-improved version in the forms of the so-called Corrected Omega or 90° filter [18] and the Mandoline filter [19] and, on the other hand, post-column filters mounted below the microscope column such as the Gatan imaging filter [20], and improved versions thereof such as the GIF2000 [21] and the Tridiem filter. The spectroscopy and imaging performance of filters fully corrected for second-order and partially for third-order aberrations (such as 90° and GIF2000) is rather comparable. The energy resolution of a standard electron microscope without MC is usually limited by the energy distribution of the primary electrons from the heated filament or the thermally assisted Schottky-type FEG. With a MC, on the other hand, the energy stability of the whole instrument, in particular of the high-voltage tank and the current supplies for both MC and spectrometer, have now started to limit the spectral resolution attainable [2].

Due to the unavoidable spherical aberration (or opening error) of a round electromagnetic lens, commonly expressed in terms of its spherical aberration constant, C_s, paraxial electrons cannot be focussed into a point [22]. The resulting Airy disc in the back focal plane decreases the resolution of the system. C_s-correctors have long been discussed theoretically [23,24], but only recently have electromagnetic multi-pole lenses become manufacturable to the precision and with the computer alignment necessary for operation. Again, two design principles can be distinguished. The hexapole design is based on a combination of two hexapoles with some additional transfer lenses [25], which have been implemented in TEMs where they have been shown to improve the resolution [26] and to decrease the delocalisation of images from sharp interfaces [27]. An octupole/quadrupole design, on the other hand [28,29], was favoured for implementation in dedicated STEM where the spatial resolution was improved even more drastically, down to about 0.08 nm at 120 kV [30] and 0.07 nm at 300 kV [31], respectively, which is very close to the ultimate resolution limit predicted earlier for 200 kV [32]. It should, however, be noted that inelastic electron scattering by bound electrons is less localised than elastic scattering by atomic nuclei so that the spatial resolution obtainable in energy filtered TEM (EFTEM) or EELS imaging modes is expected to be reduced significantly [5].

In the following, we describe a new type of 200 kV scanning transmission electron microscope (STEM) with both a MC and a corrector for the spherical aberration of the illumination system, which has been designed and built by Carl Zeiss NTS GmbH. The MC is of the electrostatic Omega type [4] and the C_s-corrector is based on the hexapole design [25,33]; both components are developed and manufactured by CEOS GmbH. The microscope column features a Schottky-type FEG cathode and an in-column corrected Omega-type imaging filter fully corrected for second-order aberrations (90° filter). The STEM/TEM concept with Koehler illumination allows to switch easily, i.e. without hysteresis due to thermal drift settling after large current changes as would be required in case of only two condenser lenses, between STEM mode for analytical work and TEM mode for high-resolution and in situ studies [34]. With the C_s-corrector between the MC and the specimen (more precisely, between the condenser lenses and the objective lens) it is possible to form a very small electron probe with a current sufficient for high-resolution annular dark-field imaging, similar to a dedicated STEM instrument. The C_s-corrector also allows the use of large

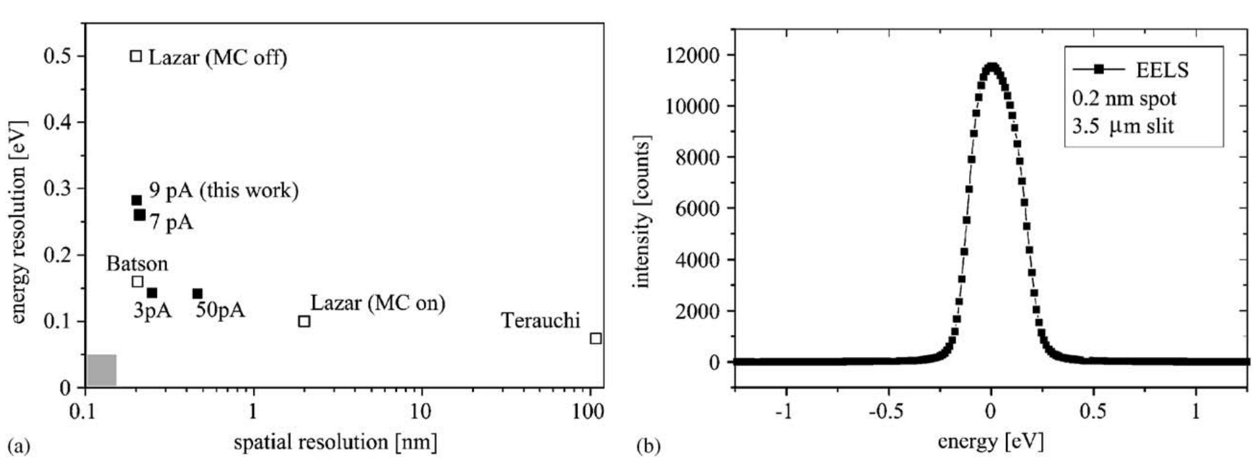

Fig. 1. (a) Comparison of the trade-off between energy vs. spatial resolution for different designs of monochromators (MCs). An ideal instrument would be at the bottom left corner of this graph (grey rectangle). Labels near the open squares refer to the first authors of the corresponding references [1,12,13]. Solid squares are measurements from this study, and the labels state the probe current obtained with a 3 or 3.5 µm slit aperture in the MC. (b) EELS of the zero loss peak with the smallest STEM probe of $\leqslant$0.2 nm and 3.5 µm MC slit aperture.