

Breaking Through the Barrier

Typical structures in nano-scale science and advanced nano-technology are becoming increasingly smaller.

Since scientists and engineers need to visualize and analyze what they develop and manufacture, there is an increasing demand for better imaging resolution and improved imaging quality.

The attainable resolution of a transmission electron microscope (TEM) is mainly determined by the properties of the objective lens like in an optical microscope. Lenses are never perfect and exhibit a variety of defects. One major lens defect, called spherical aberration and characterized by the spherical aberration coefficient, C_s , describes the effect that rays or electrons away from the optical axis are not focused into the same focal point as those propagating on the optical axis. In light optics improved manufacturing quality, sophisticated shapes of lens surfaces and combination of lenses have led to very high optical properties which result in a resolution close to the order of or even smaller than the wavelength, λ , of the visible light.

Since typical lenses in transmission electron microscopy used so far are electromagnetic round lenses, they unavoidably suffer from aberrations and have always a positive spherical aberration coefficient C_s [1]. This explains why the typical (point) resolution of a present 200 kV TEM of 0.24 nm is about two orders of magnitude worse than the wavelength of the electrons ($\lambda_{200\text{kV}} = 0.0025 \text{ nm}$). Only the coefficient C_s and the wavelength λ determine the (point) resolution. Therefore only two ways for resolution improvement exist (besides different techniques like electron holography): to shorten the wavelength by increasing the accelerating voltage or to reduce the C_s of the objective lens. Higher accelerating voltages increase the knock-on damage, i.e. the direct displacement of atoms from the crystal lattice and, also important, the costs of such microscopes. In 1990 Prof. Harald Rose showed that, in principle, it is possible to correct the C_s of the objective lens of a TEM [2]. This discovery paved the way towards sub-Ångstrom resolution for 200 kV TEMs.

The Concept of the Ultra High-Resolution TEM (UHRTEM)

Key elements of the UHRTEM are three new electron-optical components. The C_s corrector according to the concept of Rose [2] was developed by the German company CEOS (Corrected Electron Optical Systems GmbH, Heidelberg) [3,4]. It consists of two hexapole elements and two lens doublets which provide the complete compensation of the spherical aberration (of the imaging part) of the objective lens. Only this hexapole corrector is able to correct for large image sizes typical for the image mode in transmission electron microscopy.

The second component is a monochromator, which has also been devel-

oped by CEOS and is entirely integrated in the electron source which is nowadays a Field Emission [FE] system. The monochromator reduces the inherent energy spread in the electron beam from typically 0.7 eV to values smaller than 0.2 eV depending on the setting. As soon as the spherical aberration of the objective lens is fully corrected (C_s drops by three orders of magnitude to a few microns compared to a value of about 1 mm without correction) the second largest lens defect, the chromatic aberration, C_c , comes into play. For monochromated electrons the influence of the chromatic aberration is smaller. Since correction of C_c requires a rather complex arrangement of even more electron-optical elements compared to the C_s corrector, the

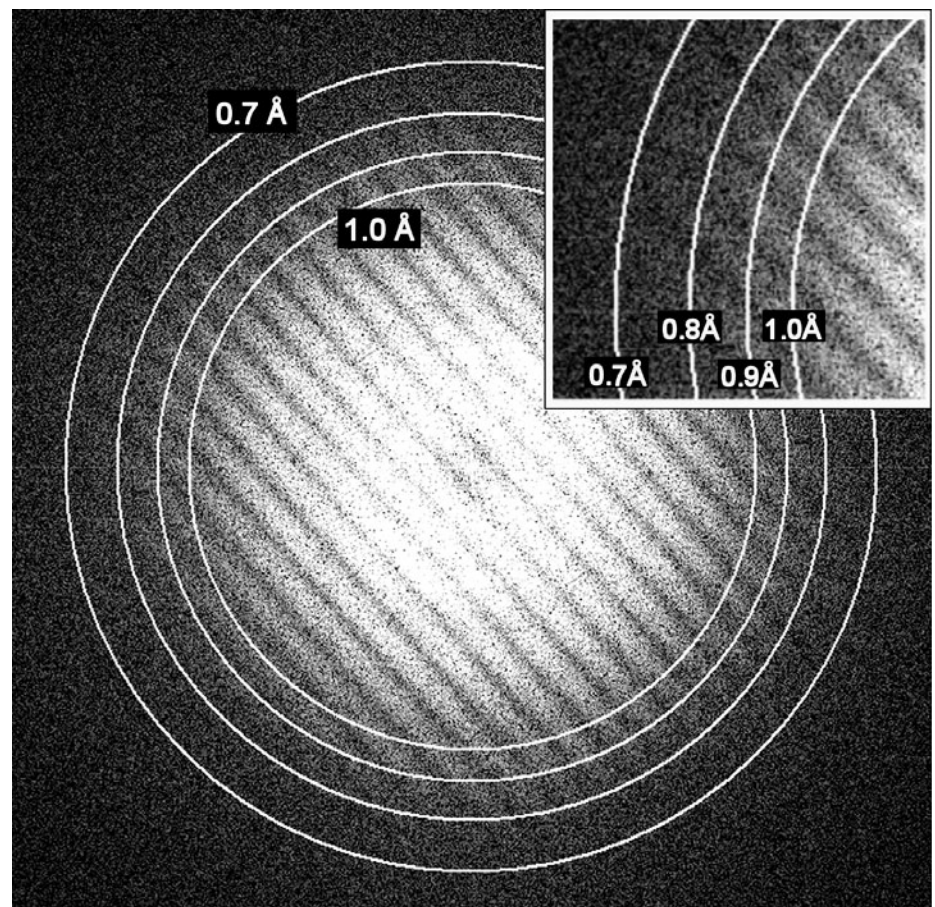


Fig. 1: Young's fringes pattern revealing useable image resolution of 0.8 Angstrom. Inset shows image resolution of even down to 0.7 Angstrom for certain image directions, nearly equal the theoretically achievable resolution limit.

reduction in energy spread is the method of choice. The combination of the monochromated FE source with a C_s corrector for the objective lens enables breaking the barrier of sub-Ångstrom imaging resolution.

Last but not least the UHRTEM comprises an In-column energy filter of the corrected Omega design [5]. An In-column Corrected Omega filter disperses the electrons according to their energy (like a prism disperses the different colors of the visible light) and thus providing an electron energy loss spectrum (EELS) of the specimen in the dispersive plane, revealing detailed information on the chemical elements and their bonding. Furthermore, the In-column Corrected Omega filter is also an imaging unit since it generates an achromatic image of the specimen (to which electrons of all energies contribute). By selecting a certain energy range by inserting a slit in the dispersive plane, the image only contains information of specific electron energy losses within the specimen (EFTEM = energy filtered TEM). This can be used e.g. to remove inelastically scattered

electrons (for contrast enhancement) or to select element specific energy losses for elemental distribution images (electron spectroscopic imaging). Therefore, the In-column Corrected Omega energy filter adds analytical capabilities to the UHRTEM without influencing its outstanding imaging resolution.

A new Record Milestone in Sub-Ångstrom TEM Imaging

During the qualification phase of the UHRTEM, a new record milestone was achieved [6]. In order to demonstrate the achieved imaging resolution, a so-called Young's fringes pattern (Fig. 1) has been generated from two micrographs recorded at 800,000 times magnification and image acquisition times of one second. The two images were taken from slightly different locations of an amorphous tantalum thin film, subsequently added and Fourier transformed to generate the fringe pattern shown in Fig. 1. The fringe pattern helps to distinguish signal from noise. The energy spread of the field emission source was reduced by

the monochromator to 0.2 eV and a residual spherical aberration of the objective lens (C_s value) of approximately $-3\ \mu\text{m}$ was obtained. Four ring insets, calibrated by gold lattice reflections, indicate the 1.0, 0.9, 0.8 and 0.7 Ångstrom resolution limits (from inside to outside in Fig. 1). For all image directions, the fringe contrast clearly extends to the 0.8 Ångstrom ring and even to the 0.7 Ångstrom ring for certain image directions which is close to the theoretical limit for a 200 kV FE-TEM.

The Benefit of Artefact-free Imaging

The possibility to reduce the main lens defect, the spherical aberration, by three orders of magnitude to a value close to zero not only increases the resolution, but it also improves the image quality in general since other effects are close related to the C_s value. In the following two of them are addressed.

The first one is the so-called delocalization. What does delocalization mean? Due to a non-zero C_s value, electrons from one point of an object are not imaged into a single point but rather into a small disk smearing out the information (the information is no longer „localized“ but „delocalized“). In atomic resolved images of periodic structures delocalization, although present, is not easily visible. However, as soon as non-periodic structures are imaged or the periodicity is terminated in at least one direction (e.g. by an interface or surface), the effect of delocalization is striking. To our knowledge the first example of this kind, a CoSi_2/Si interface, was shown [3, 4]. Only the aberration corrected image of this interface revealed its atomic structure and showed that the CoSi_2/Si interface is atomically flat. Therefore, for all types of thin film research artefact-free imaging is prerequisite for a successful process control and development. Since any kind of process control has to give a clear feedback to process engineers, whether the selected parameters led to satisfactory results or not, the new class of C_s -corrected TEMs could define the future standard tool for process control especially in the nano-scale science and research independent of the class of materials. An impressive example of artefact-free imaging taken recently is shown in Fig. 2. A silicon nano-wire could be imaged with the UHRTEM without delocalization, resulting in a crisp and clear image of the interior structure and the outer shape of this special nano-structure.

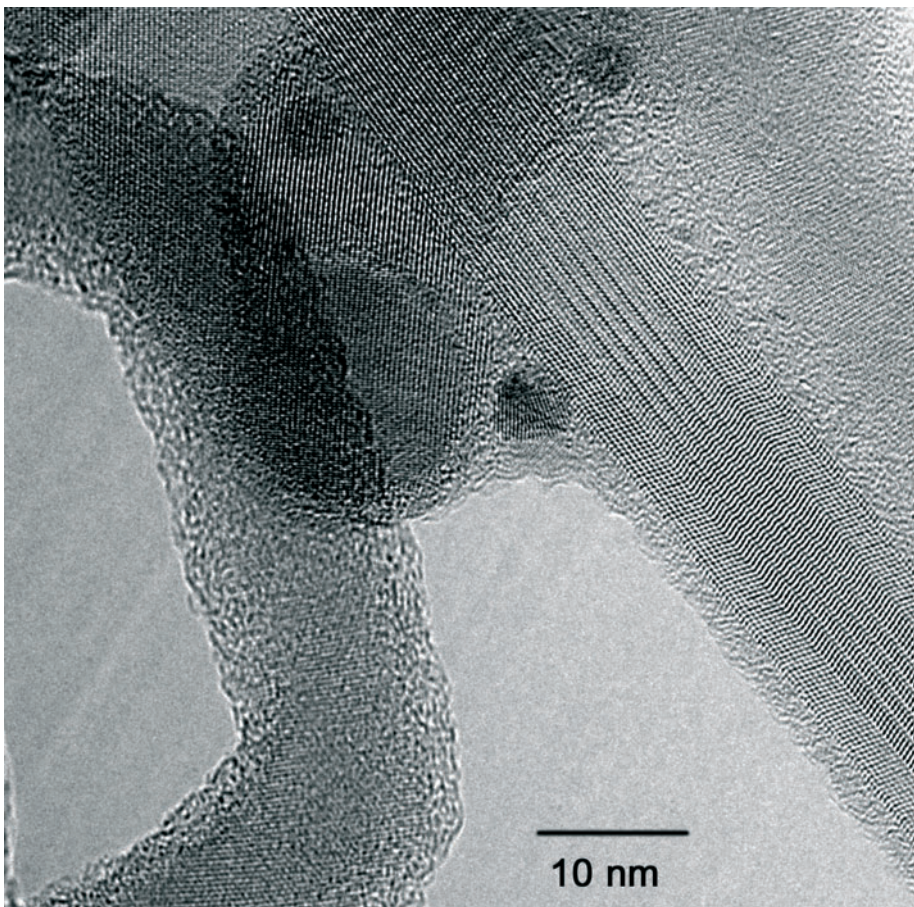


Fig. 2: Image of a single Si nano-wires on a carbon support film. The magnification was 400,000 times and a $C_s < 3\ \mu\text{m}$ was used. Specimen courtesy of Prof. Brian A. Korgel and Prof. Miguel J. Yacaman [Chemical Engineering, University of Texas, Austin]

Secondly, in a TEM with aberration correction of the objective lens a small beam tilt does not cause other image faults like astigmatism or coma. High-resolution TEM imaging of crystalline structures requires their precise orientation to the optical axis. To achieve this by tilting the sample is in many cases time consuming. In a C_s -corrected TEM this final alignment of orientation of the sample towards the electron beam direction can be reversed by tilting the axis of electron beam towards the orientation of the crystalline sample. This makes it

much easier to precisely align a specimen area e.g. close to a defect of interest just by tilting the beam and leaving the specimen stage untouched. As a consequence, specimen throughput can be increased.

Outlook

After almost no improvements in imaging resolution during the 1990's, the correction of the spherical aberration of the objective lens of a TEM has opened up a new era in transmission electron microscopy. Artefact-free imaging up to the utmost imaging resolution with the freedom to tilt the beam without deteriorating effects on the image quality will find many new applications with unprecedented insights into materials research problems at the nano-scale.

The advantages of a C_s corrector, i.e. the artefact-free imaging and a new freedom in permissible beam tilts, is available independent of the presence of a monochromator. The monochromator in addition to a C_s corrector is only necessary to push the imaging resolution to the deep sub-Ångstrom regime.

Although the entire potential of the new technology of aberration correction and monochromatisation is far from being exploited, the impact on transmission electron microscopy as a process control and research tool for nano-technology and nano-science should not be underestimated.

Acknowledgement

The authors thank all their co-workers and colleagues, especially the entire R&D team for all contributions and continuous support which made all the new developments possible.

Libra and Omega are registered trademarks of Carl Zeiss SMT Nano Technology Systems Division.

References

- [1] O Scherzer, Z. Physik 101 (1936), 593-603
- [2] H Rose, Optik 85 (1990), 19-24
- [3] M Haider et al., Ultramicroscopy 75 (1998), 53-60
- [4] M Haider et al., NATURE, Vol. 392 (23 April 1998), 768-769
- [5] S Lania, H Rose, D Krahl, Optik 75 (1986) 56
- [6] <http://www.smt.zeiss.com/nts> and follow "New resolution record"
- [7] For more information see: www.smt.zeiss.com/Libra

Peter Schlossmacher

Marko Matijevic

Alexander Thesen

Gerd Benner

Carl Zeiss SMT– Nano Technology Systems Division
Rudolf-Eber-Str. 2 · 73447 Oberkochen · Germany
Tel. +49 7364/20-0 · Fax +49 7364/20-4537
schlossmacher@smt.zeiss.com · www.zeiss.de

Short CV

Peter Schlossmacher received his Ph.D. at the Technical University (RWTH) Aachen (Germany) for a TEM study on defects in GaAs performed at the Research Center in Juelich. Thereafter he moved to the Research Center in Karlsruhe heading a TEM laboratory at the Institute for Materials Research and working mainly on TiNi-based shape memory alloys. Since 2002 he works for Carl Zeiss NTS as International Business Development Manager and in Technical Sales & Marketing for the entire TEM business.



Fig. 3: The Carl Zeiss SMT Libra 200 FE with 200 kv Schottky-FE system, 300 mm-diameter stable column and In-column Corrected Omega energy filter [7]