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Aberration Corrected S/TEM Delivers Directly Interpretable Atomic Resolution

Guest Article

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Understanding the physical properties of a material requires detailed knowledge of its structure down to the atomic level. Transmission Electron Microscopes (TEM) and Scanning Transmission Electron Microscopes (STEM) are primary tools for investigating atomic structure. However, until recently, their resolution was limited by the spherical aberration (C_s) of the objective lens of the microscope [1]. Correcting the spherical aberration permits directly interpretable TEM image resolution better than 1 Å with better contrast and sensitivity. In STEM, aberration correction also provides higher imaging contrast, greater analytical sensitivity and unprecedented spatial resolution. However, correctors alone cannot improve the performance of the microscope if it lacks sufficient electrical and mechanical stability. Although theoretical correctors were first described in 1990, realization of their benefits had to await the design of a microscope platform capable of supporting improved optical performance they permit.

In any round magnetic lens, the parts of the object wave that carry the most interesting information passes through the outer zone of the objective lens and suffers from large additional phase shifts, introduced primarily by the third-order spherical aberration of the objective lens. As early as 1936, Scherzer proved that the spherical aberration cannot be avoided in rotationally-symmetrical electro-magnetic fields [2]. This type of aberration can be corrected by means of multipole fields, which effectively produce a negative third-order spherical aberration. In 1990, Rose described a hexapole corrector based on this principle allowing

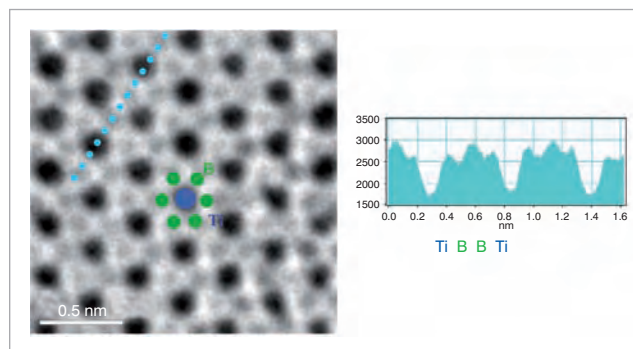


Fig. 2: C_s corrected HR-TEM image of TiB_2 along the $\langle 0001 \rangle$ zone axis. The intensity profile (right) shows the contrast of the different atomic species

compensation of the C_s -aberration of the objective lens [3,4]. By changing the multipole excitation the value of the total spherical aberration of the TEM objective imaging system can be set to any value, thus providing an additional parameter to tune the microscope to optimum imaging conditions.

In addition to this coherent aberration, incoherent disturbances also limit the information transfer of the TEM. The chromatic aberration of the objective lens in conjunction with the energy spread of the illuminating electron beam dampens the contrast transfer of the TEM at high spatial frequencies. The negative effects of chromatic aberration can be reduced by decreasing the energy-spread of the illuminating electrons [5] using a monochromator in the illumination system [6]. A smaller energy-spread also improves the analytical capabilities of the S/TEM.

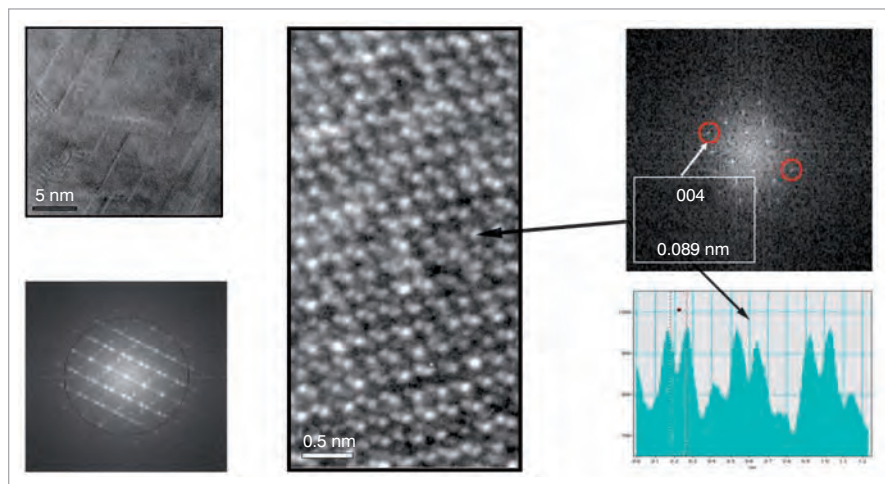


Fig. 1: C_s corrected HR-TEM image of diamond in the $\langle 110 \rangle$ zone axis

By adding a C_s corrector to the highly flexible 3-lens illumination system of TitanTM microscope, deep sub-Ångström sized probes can be formed allowing for deep sub Ångström atomic STEM imaging.

Additionally, higher opening angles of probe C_s -corrector tools provide extremely high probe currents in Ångström sized probes, which were only obtained before in nanometer sized probes. This performance allows for atomic resolution chemical mapping to provide not only the position of the atoms in imaging, but their chemistry as well.

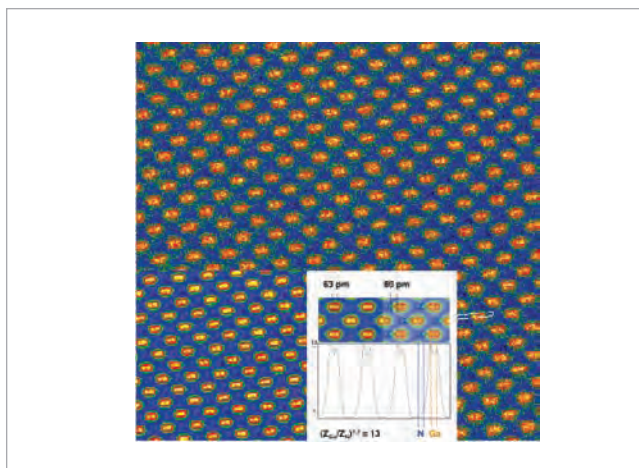


Fig. 3: HR-STEM image of GaN in [211] projection at 300kV acceleration voltage

The C_s -corrector and monochromator can only improve the information transfer of TEM if the remaining residual incoherent disturbances (mechanical, electrical and thermal instabilities) can be suppressed below the desired lateral resolution of the TEM. The FEI Titan platform was designed specifically to address the requirements of sub-angstrom resolution, aberration corrected microscopy. Disturbances due to mechanical vibrations have been reduced by introducing a design concept that separates the magnetic flux cirrus from the mechanical forces in the column. The stiffness of the column is further improved by increasing its diameter and a patented assembly technology ensures the precise mechanical alignment of the optical components.

Electrical noise also degrades performance. Noise in the electrical current supplied to the magnetic lenses and deflectors reduces the temporal coherence of the

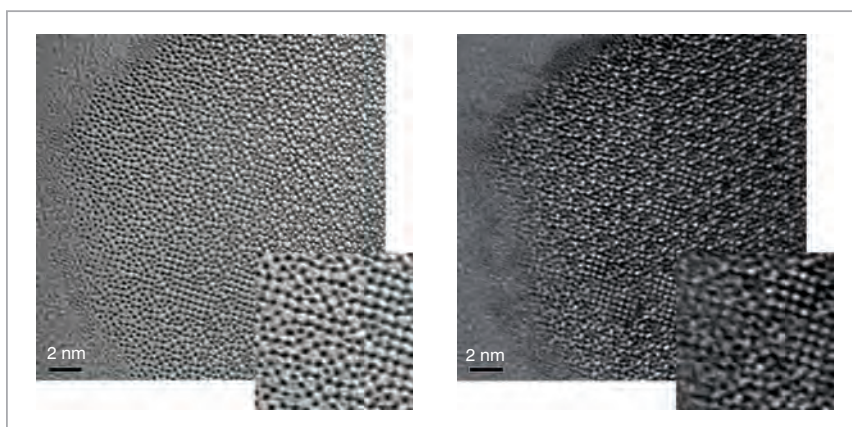


Fig. 4: Pair of C_s corrected (left) and uncorrected (right) HR-TEM images of $Nb_7W_{10}O_{47.5}$

imaging electrons and degrades the information limit of the microscope. The Titan's lens supplies attain noise levels below 0.1ppm RMS. The ultrastable high tension supply, with a noise/ripple ratio below 0.1ppm RMS, permits energy resolution down to 0.15eV.

The combination of C_s -corrector and monochromator technology minimizes the most dominant aberration of C_s and C_c . This allows for high tension flexibility (300-60kV) in S/TEM applications while maintaining the atomic resolution in imaging and chemical mapping at lower acceleration voltages. The lower voltage range enables the examination of delicate light materials (Graphen, SWCNT etc.) with higher contrast and better S/N and avoids sample destruction by knock-on damage processes.

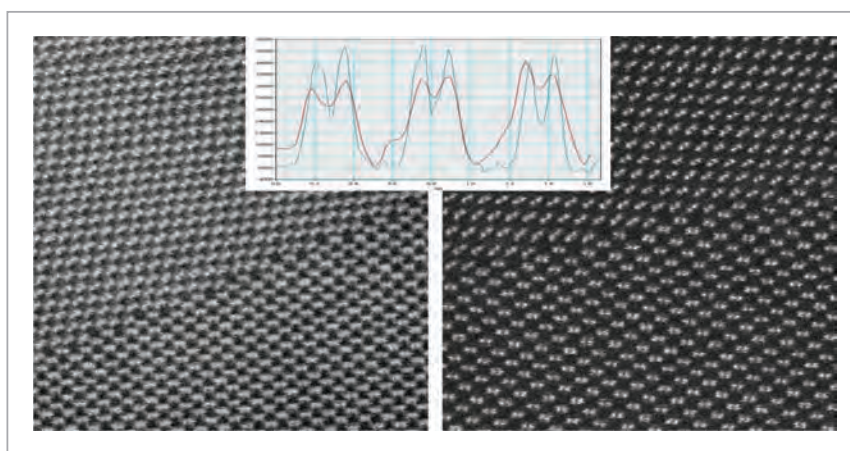


Fig. 5: Pair of C_s corrected (left) and uncorrected (right) HR-TEM images of a silicon grain boundary

Long term stability, including mechanical drift and thermally induced drift in optical alignments, is equally important. The Titan's constant power lenses permit optical changes in the optical power of the lens without changing the total electrical power dissipated by the lens, thus providing a stable thermal environment and avoiding lengthy waits for the system to achieve equilibrium after every change in operating conditions.

The high resolution and improved sensitivity that can be obtained in C_s corrected STEM are depicted through the following sample images.

Improved Resolution

The combination of corrector optics and a highly stable column has drastically improved the image resolution to the sub-Ångström level in HR-TEM and HR STEM. An example of this improved resolution is shown in Fig. 1. The dumbbell

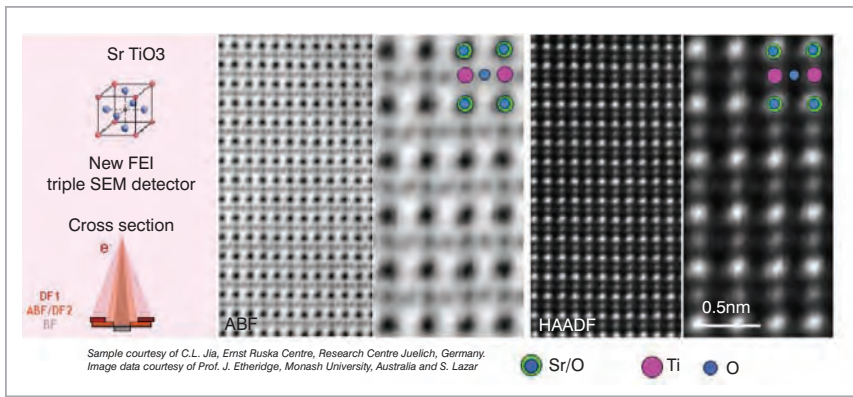


Fig. 6: Annular bright-field (ABF) and high-angle annular dark field (HAADF) STEM image of SrTiO₃ in [110] projection

structure with a distance of 89 pm (0.089nm) can be resolved in the image. This is not an artifact as this resolution cannot be obtained by double diffraction effects.

Greater Sensitivity

Along with the improvement in lateral resolution, the

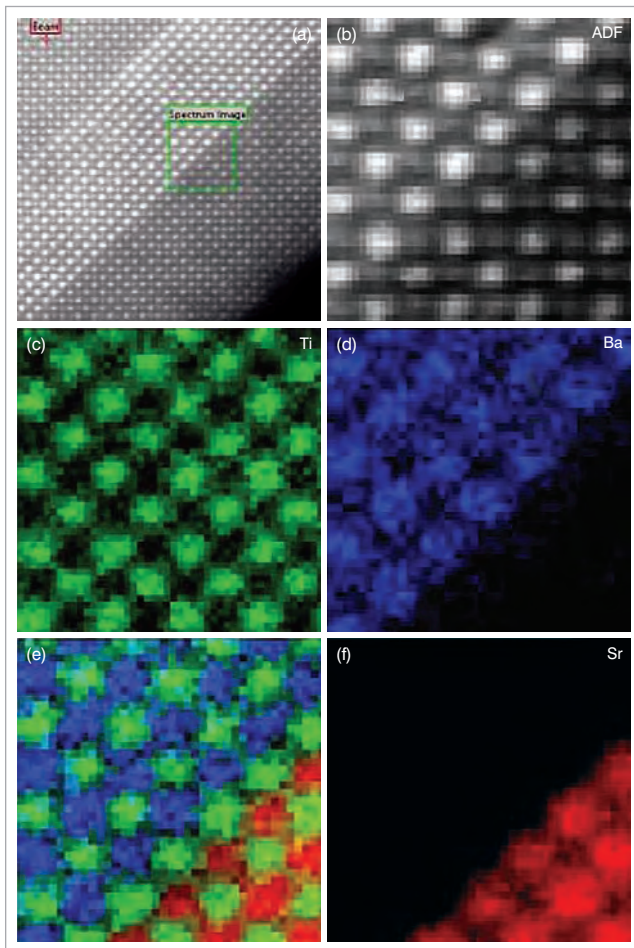


Fig. 7: Atomic resolution EELS mapping on SrTiO₃/BaTiO₃ interface. (a) spectrum image, (b) Annular Dark Field (ADF) image, (c, d and f) chemical maps showing atomic positions of the Ti, Ba and Sr respectively and (e) overlay of Ti, Ba and Sr maps

C_s-corrected Titan microscope also enhances the sensitivity in atomic imaging and spectroscopy. Figure 2 shows this effect on a TiB₂ sample along the hexagonal <0001> zone axis. The lattice of the extremely light element boron can be imaged in HR-TEM imaging, revealing the hexagonal boron rings around the heavier titanium atom in the center of the ring.

Another example of enhanced sensitivity is clearly depicted in Fig. 3, where the distance between the Ga atoms of 63 pm can be clearly resolved in the unprocessed image.

Comparison Between C_s Corrected and Uncorrected Images

The benefit of corrector optics technology can be demonstrated by comparing images taken on the same area with and without the spherical aberration correction. As observed in Fig. 4, in the corrected image each dark dot represents the position of Nb or W (inset left side) where as in an uncorrected image the atoms can have bright or dark contrast, so that the atomic positions cannot be extracted directly (inset right side)

Similarly, in Fig. 5 the improvement in the resolution is clearly noticeable in the form of higher contrast in the image. The intensity profile in Fig. 5 shows the gain in contrast especially in the depth of the minimum between the silicon dumbbell distances.

Contrast

Spherical aberration correction offers remarkable advantages for Z-contrast STEM. Figure 6 shows the annular bright-field (ABF) and high-angle annular dark field (HAADF) STEM image of SrTiO₃ in [110] projection. The oxygen columns can be visualized using angular bright field imaging (ABF) and the heavier elements of titanium and Strontium can be imaged using conventional HAADF STEM imaging. The images were taken at 200kV using a probe C_s-corrected Titan with the new FEI triple bright field dark field detectors (see schematics on the left side)

Atomic Resolution Electron Energy-Loss Spectroscopy (EELS)

EELS is capable of giving structural, chemical and electronic information about a solid down to the atomic level. Spherical aberration correction helps to increase the aperture angle that increases the beam current by several orders of magnitude which is advantageous for chemical mapping. Figure 7 shows atomic resolution EELS mapping on SrTiO₃/BaTiO₃ interface. In the

Annular Dark Field (ADF) image of SrTiO₃/BaTiO₃, the atomic positions of the Ti, Ba and Sr can be clearly imaged by the EELS map of the interface. The result was obtained at 80kV acceleration voltage to minimize beam damage effects using a probe C_s corrected Titan.

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