C_S-TEM vs C_S-STEM

Duncan Alexander EPFL-CIME

Duncan Alexander: Cs-TEM vs Cs-STEM

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FEI Titan Themis @ CIME EPFL

60-300 kV

Monochromator

High brightness X-FEG

Probe Cs-corrected: 0.7 Å @ 300 kV

Image Cs-corrected: 0.7 Å @ 300 kV

Super-X EDX detector

GIF Quantum ERS energy filter

Dual-channel, Ultrafast STEM-EELS

Lorentz mode

Biprism for holography

Piezo stage

Tomographic acquisition



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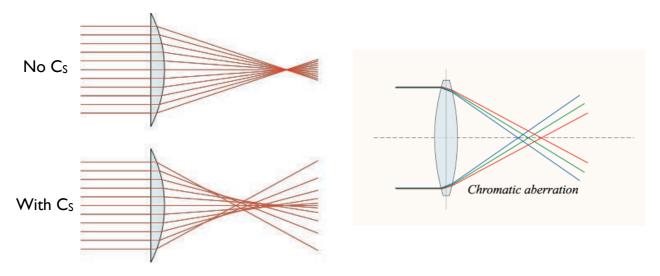
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Limitation to spatial resolution: aberrations

Electromagnetic lenses in TEM column are toroidal

Lenses inherently convergent

=> spherical aberration (C_S) and chromatic aberration (C_C)



Resolution in HR-TEM limited by aberrations, especially Cs

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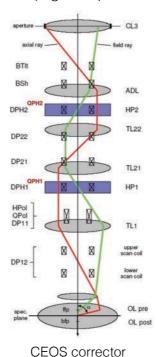
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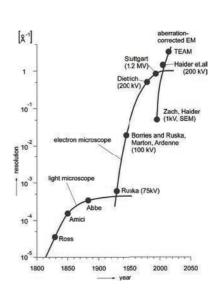
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Cs-correction (STEM and TEM)

Combination of standard radially-symmetric convergent lenses with multipole divergent lenses (e.g. tetrapoles, hextapoles) to tune C_S







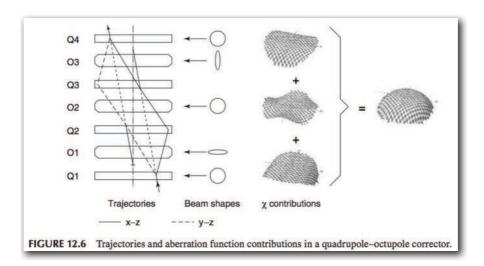
Rose J. *Electron Microscopy* **58** (2009) 77–85

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Principle of aberration correction

 Compensate C_S and other distortions with equivalent but opposite components to add together with aim of giving ideal spherical wavefront



Krivanek et al. Aberration Correction in Electron Microscopy, Handbook of Charged Particle Physics 2009, pp. 601–641

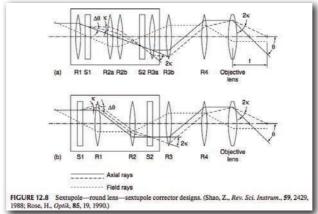
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CEOS aberration corrector

 CEOS aberration corrector used for imaging correction in CTEM also used before sample as probe-corrector for STEM; sextapole-round lens-sextapole design. This is an "indirect" corrector type; ~30 power supplies but higher power and water cooling needed.



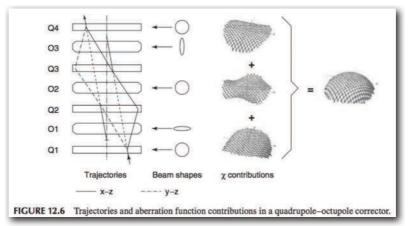
Indirect-action correctors, in which the aberration to be corrected is acted on by a combination aberration arising as a by-product of a lower-order aberration intentionally created in the corrector. The undesirable lower-order aberration coefficients are typically of the same order of magnitude as the coefficients of the aberrations to be corrected. For the small angles used in electron microscopy, this means that the effect of the lower-order aberrations on the beam are very strong. These aberrations, therefore, need to be canceled very precisely, typically by using an equivalent optical element that cancels the undesirable low-order effect while at the same time increasing the desirable higher-order effect.

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Nion aberration corrector

 First STEM aberration corrector installed on VG by Nion (Krivanek); quadrupoleoctopole design. This is a "direct-action" corrector type as now used on Nion UltraSTEM: ~70 power supplies needed but low power and which can fit onto printed circuit boards



Direct-action correctors, in which the principal aberration is corrected by a correcting element whose main effect is producing aberrations of the needed order. In these systems, undesirable by-product aberrations tend to arise that are of the same order and of similar magnitude as the aberrations being corrected. The by-product aberrations are typically canceled by creating different beam shapes in different stages of the apparatus, which permits the same type of optical element to produce different mixtures of aberration terms.

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Krivanek et al. Aberration Correction in Electron Microscopy, Handbook of Charged Particle Physics 2009, pp. 601–641

Current correctors

- CEOS: C_S-corrected, "C₅ optimised": CETCOR, CESCOR, D-COR
- CEOS: C_S-C_C corrected (NCEM TEAM 1.0, Julich Titan Pico)
- CEOS: B-COR aplanatic optimised for far off-axis rays
- JEOL: unique C_S-C_C corrector (CCC project)
- JEOL: Dodecapole Cs corrector ("Grand ARM")
- Nion: C₅-C₅ corrected

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Understanding resolution in EM

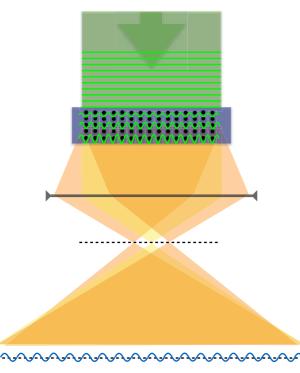
- For C_S-TEM need to understand concepts of:
 - Contrast transfer function (CTF)
 - How to use CS to optimise CTF
 - Difference between point resolution and information limit
 - Properties of the camera (MTF), sample drift, "Stobbs factor"...
- For C_S-STEM need to understand concepts of:
 - Probe size, shape, brightness, depth of field (DOF)
 - Optical transfer function (OTF); STEM first to achieve 0.5 Å res
 - Scan (in)stabilities, detectors

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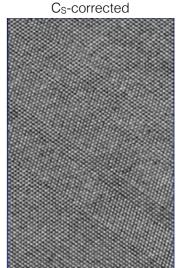
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Cs-corrected HR-TEM "interferometry"



Example: Σ3 grain boundaries in Al

Uncorrected



Images: Oikawa, JEOL

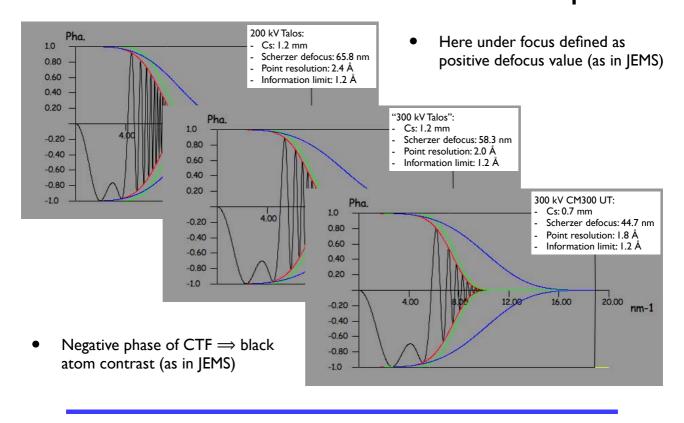
Image of "projected potential"

Reduced delocalisation in *phase contrast* image

Duncan Alexander: Cs-TEM vs Cs-STEM

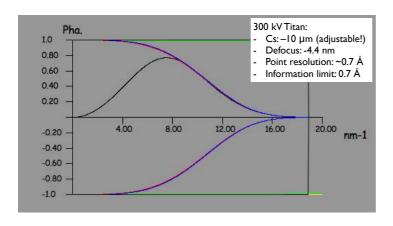
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CTF curves: uncorrected microscopes



Cs-TEM: effect on CTF

Duncan Alexander: Cs-TEM vs Cs-STEM



 Higher information limit from shifting spatial and temporal envelopes

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- Done by improved stability of instrument + monochromatic beam
- Here show negative Cs ("white atom" contrast)
- Adjust Cs, defocus to give one wide CTF pass band to information limit

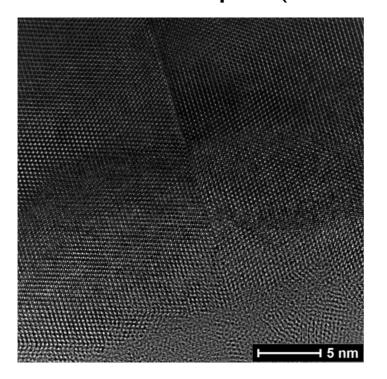
Duncan Alexander: Cs-TEM vs Cs-STEM

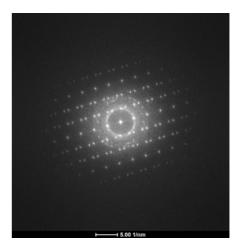
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Cs-TEM example: $(Al_xGa_{1-x})As$ nanowire







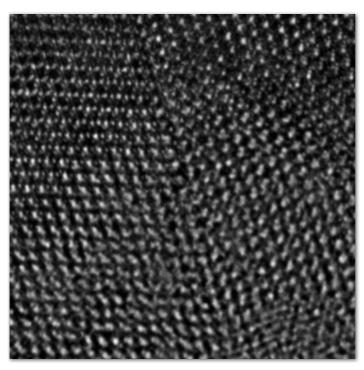
Sample courtesy of Yannick Fontana, Anna Fontcuberta-i-Morral, LMSC

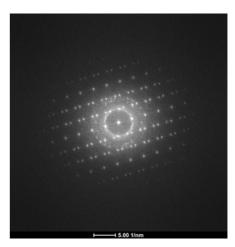
Duncan Alexander: Cs-TEM vs Cs-STEM

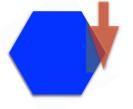
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Cs-TEM example: $(Al_xGa_{1-x})As$ nanowire





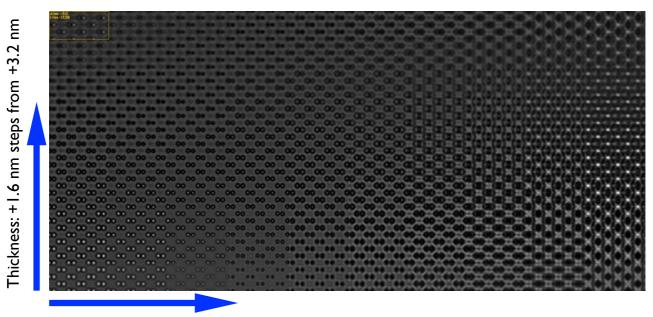


Sample courtesy of Yannick Fontana, Anna Fontcuberta-i-Morral, LMSC

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Cs-TEM [I I 0] GaAs simulation



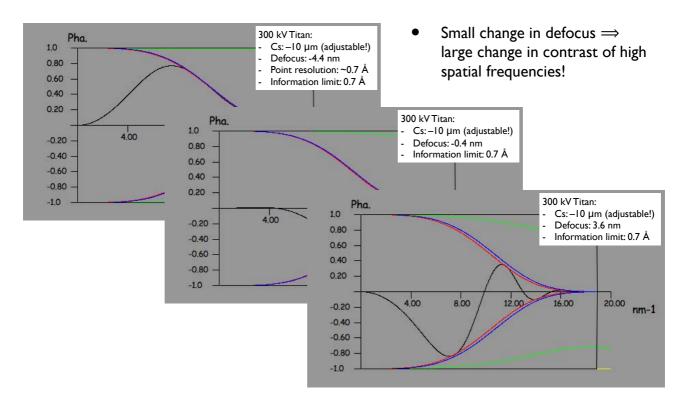
Defocus: - 2 nm steps starting from +5 nm

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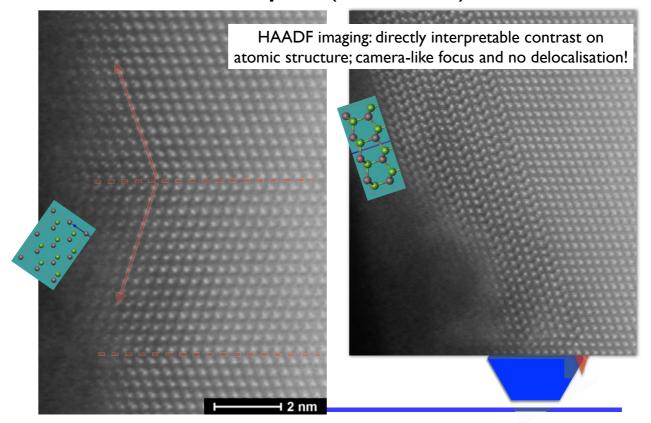
Cs-TEM: defocus effect on CTF



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Cs-STEM example: $(Al_xGa_{1-x})As$ nanowire



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Benefits of aberration correction

Analytics – STEM-EELS

Atomic resolution core-loss STEM-EELS mapping (Nion UltraSTEM)

Atomic-Scale Chemical Imaging of Composition and Bonding by Aberration-Corrected Microscopy

D. A. Muller, et al.

Science 319, 1073 (2008);

DOI: 10.1126/science.1148820

Fig. 1. Spectroscopic imaging of a La_{0.7}Sr_{0.3}MnO₃/ SrTiO3 multilayer, showing the different chemical sublattices in a 64 x 64 pixel spectrum image extracted from 650 eV-wide electron energy-loss spectra recorded at each pixel. (A) La M edge; (B) Ti L edge; (C) Mn L edge; (D) red-green-blue falsecolor image obtained by combining the rescaled Mn, La, and Ti images. Each of the primary color maps is rescaled to include all data points within two standard deviations of the image mean. Note the lines of purple at the interface in (D), which indicate Mn-Ti intermixing on the B-site sublattice. The white circles indicate the position of the La columns, showing that the Mn lattice is offset. Live acquisition time for the 64×64 spectrum image was ~30 s; field of view, 3.1 nm.

More recently: atomic resolution EDX, EFTEM – but are they as interpretable?

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Measurement precision - Cs-TEM

Is science prepared for atomicresolution electron microscopy?

Knut W. Urbar

The efforts of microscopists have given aberration-corrected transmission electron microscopy the power to reveal atomic structures with unprecedented precision. It is now up to materials scientists to use this power for extracting physical properties from microscopic atomic arrangements.

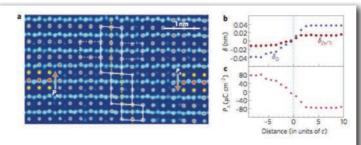


Figure 2 | Polarization domain wall in ferroelectric PZT. a, Atomic structure. Arrows give the direction of the spontaneous polarization, which can be directly inferred from the local atom displacements. The shifts of the oxygen atoms (blue circles) out of the Ti/Zr-atom rows (red circles) can be seen directly, as can the change in separation between Ti/Zr and Pb (yellow circles). b, Atomic-resolution measurements of the shifts of oxygen (δ_0), and titanium/zirconium ($\delta_{1/2,0}$) atoms as a function of distance from the wall centre (units of c-lattice parameter) in a longitudinal-inversion domain wall; c. The value of the local polarization P_c that can be calculated from these data. Adapted from ref. 12. © 2008 NPG.

An investigation using Gaussian regression analyses' revealed that such position measurements can be carried out at a precision of better than ±5 pm (at a 95% confidence level). Such a precision is far superior to that of any other microscopic technique, including the scanning transmission electron microscope or even the scanning tunnelling microscope. The standard objection to such extremely high

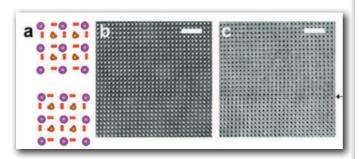
precision is that it cannot be allowed by a microscope with a 70-pm Rayleigh or point resolution 15-20. However, resolution and precision are two separate physical terms. Although resolution is defined by the minimum separation of two optically broadened intensity peaks at which these can just be separated in the image, the distance between two well-isolated peaks, fitted for example by two-dimensional Gaussians, can be measured at a precision more than an order of magnitude higher.

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Measurement precision – C_S-STEM

Mapping Octahedral Tilts and Polarization Across a Domain Wall in BiFeO₃ from Z-Contrast Scanning Transmission Electron Microscopy Image Atomic Column Shape Analysis

Albina'v. Borisevich, "** Oleg S. Ovchinnikov," Hye Jung Chang, Mark P. Oxley, ".5 Pu Yu, ** Jan Seidel, Eugine A. Eliseev, Anna N. Morozovska," Ramamoorthy Ramesh, ** Stephen J. Pennycook, ** and Sergel V. Kallinin**



VOL. 4 • NO. 10 • 6071-6079 • 2010 ACT AND

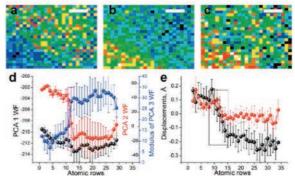


Figure 6. Comparison of the spatial variation of the column shapes and the ferroelectric polarization across the domain wall: (a) absolute value of the third PCA weight factor map (see Figure 4h), (b and c) Polarization maps for out-of-plane (b) and in-plane (c) polarization based on the Bi and Fe column displacements measured from the Z-contrast image. (d) Profiles of different PCA weight factor maps across the domain wall: first (black) (see Figure 4f), second (red) (see Figure 4g), and the absolute value of the third component (blue) (see Figure 6a); note the atomically sharp transition in the second and third PCA weight factor map profiles. (e) Profiles of out-of-plane (black) and in-plane (red) polarization maps (see b and c) across the domain wall showing a diffuse transition. Scale bars are 2 nm.

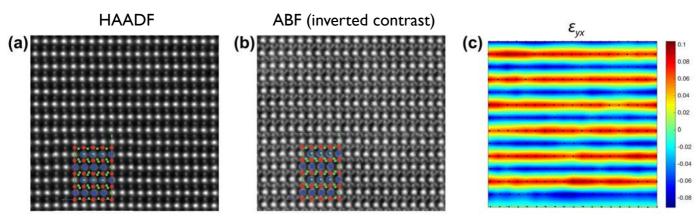
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Measurement precision - Cs-STEM

• Analysis of LaVO₃ film grown under epitaxial strain on DyScO₃ substrate



- Simultaneously acquired HAADF and ABF scan image series
- HAADF series corrected for linear and non-linear scan distortions to produce aligned and averaged image; alignments applied to ABF series also
- HAADF shows anti-ferroelectric charge ordering of La cations;
 ABF also shows projected rotations of O anion octahedra

Meley et al. APL Materials 6 (2018) 046102

Duncan Alexander: Cs-TEM vs Cs-STEM

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The move to lower kV

- Before C_s -correction highest resolution by minimising λ (MeV instruments with $\lambda < I_{pm}$)
- Light materials (graphene, nanotubes, ...) suffer knock-on damage. Some thresholds:
 - Bulk graphene: 86 keV
 - Graphene edge atom: 36 keV
- Therefore need low kV 80 kV max but 60 kV better which have long wavelengths
- Aberration correction now mandatory for atomic resolution
- Notable projects: Suenaga CCC project (30 kV aim), Ute Kaiser's Salve project (20 kV aim), both with combined C_S - C_C correctors; new UltraSTEM (20 100 kV range)

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Imaging organic molecules – Cs-(S)TEM

Cs-TEM (80 keV beam): imaging of molecule as weak phase object

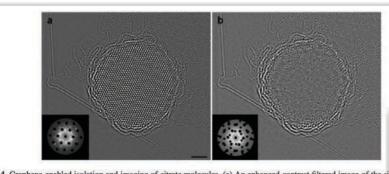


Figure 4. Graphene-enabled isolation and imaging of citrate molecules. (a) An enhanced-contrast filtered image of the cinanoparticle. (Inset) The graphene reflections were subtracted in a digital diffractogram of the entire image. Scale bar reg An image of the citrate molecules. (Inset) The graphene and gold reflections were masked in the digital diffractogram to citrate.

Lee et al. Nano Letters 9 (2009) 3365-3369

Cs-STEM (200 keV beam): imaging of molecules by HAADF; need very clean (un-contaminating) sample

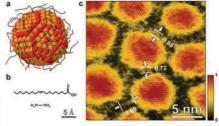


Figure 1. Geometric arrangements of the oleic acid lagands. (a) Schematic representation of the as-synthesized PbSe nanocrystal with oleic acid ligands. (b) Chemical structures of oleic acid and hydrazine molecules. (c) Atomic-resolution HAADF-STEM image of PbSe nanocrystals suspended on a ~ 5 nm thick a:Si TEM grids. Atomic planes along different crystallographic directions are visible for majority of the nanocrystals. The oleic acid ligands are attached to nanocrystals and extend to less than 1 nm radially from the surface of the nanocrystals. Indicated dimensions are in nanometers.

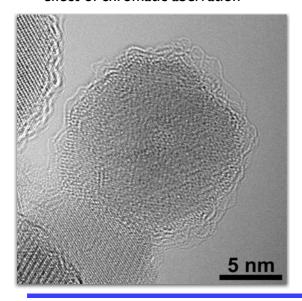
Gunawan et al. Chem. Mater 26 (2014) 3328-3333

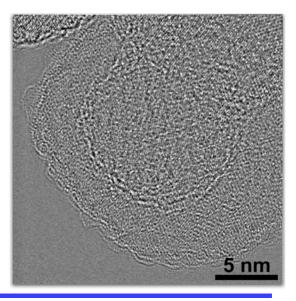
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Imaging organic molecules – Cs-TEM

- Iron oxide nanoparticles functionalised with folic acid
- Aberration-corrected phase contrast imaging at 80 keV using Titan Themis at CIME
- Incident electron beam monochromated to improve temporal coherence/reduce effect of chromatic aberration





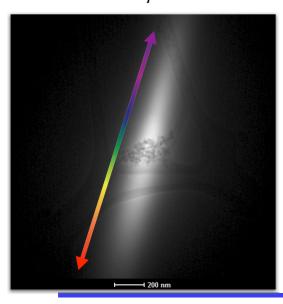
Duncan Alexander: Cs-TEM vs Cs-STEM

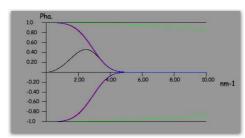
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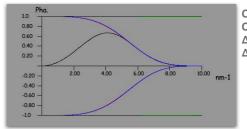
"Rainbow" mode illumination

- Use Wein filter to monochromate incident electron beam => inhomogeneous illumination with energy dispersion on one axis
- Significant reduction of energy spread; increase of temporal envelop of CTF
- See: Tiemeijer et al. Ultramicroscopy **I I 4** (2012) 72–81





Cs: -10 μm Cc: 1.5 mm ΔV: 800 meV Δf: -9.9 mm



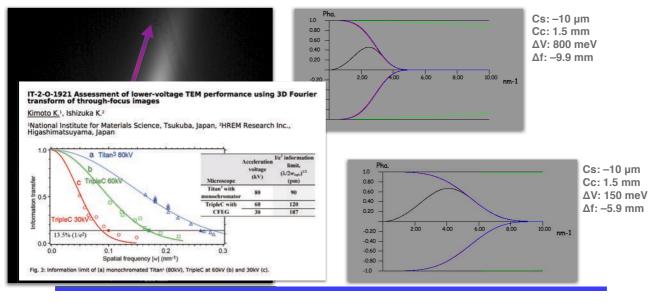
Cs: -10 μm Cc: 1.5 mm ΔV: 150 meV Δf: -5.9 mm

Duncan Alexander: Cs-TEM vs Cs-STEM

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"Rainbow" mode illumination

- Use Wein filter to monochromate incident electron beam => inhomogeneous illumination with energy dispersion on one axis
- Significant reduction of energy spread; increase of temporal envelop of CTF
- See:Tiemeijer et al. Ultramicroscopy **II4** (2012) 72–81

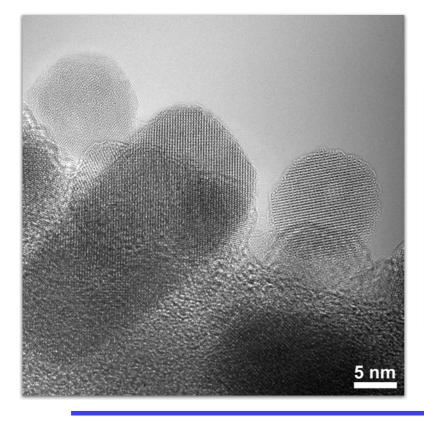


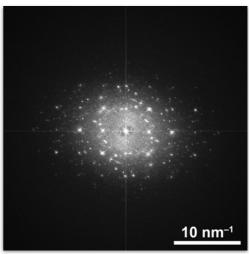
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"Rainbow" mode illumination





Iron oxide nanoparticles

Duncan Alexander: Cs-TEM vs Cs-STEM

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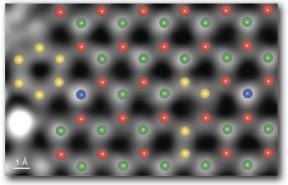
Doped graphene, BN monolayer – C_S-STEM

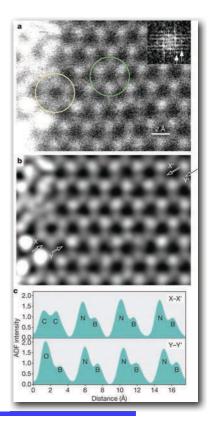
Atom-by-atom structural and chemical analysis by annular dark-field electron microscopy

Ondrej L. Krivanek¹, Matthew F. Chisholm², Valeria Nicolosi³, Timothy J. Pennycook^{2,4}, George J. Corbin¹, Niklas Dellby¹, Matthew F. Murfitt¹, Christopher S. Own¹, Zoltan S. Szilagyi¹, Mark P. Oxley^{2,4}, Sokrates T. Pantelides^{2,4} & Stephen J. Pennycook^{2,4}

- Analysis of monolayer materials: low kV essential to prevent knock-on damage; here 60 kV used (knock-on threshold for bulk graphene ~86 kV) with Nion UltraSTEM
- Medium-angle ADF (MAADF) gives intensity I $\propto Z^{1.7}$ but with increased signal intensity compared to true HAADF image. (This intensity is needed for imaging single atom by single atom; $\beta = 58-200$ mrad.) Direct atom assignment by intensity.

Krivanek et al Nature 464 (2010) 571





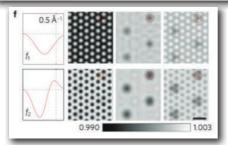
Duncan Alexander: Cs-TEM vs Cs-STEM

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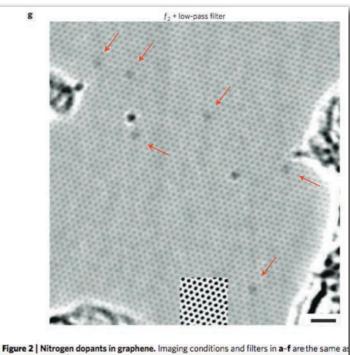
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Doped graphene, BN monolayer – C_S-TEM

Experimental analysis of charge redistribution due to chemical bonding by high-resolution transmission electron microscopy



We have shown that it is possible to obtain insights into the charge distribution in nanoscale samples and non-periodic defects from HRTEM measurements. For our examples of the nitrogen substitutions in graphene and hBN layers, we can assign experimentally observed contrast features to details in the simulated electron distribution. We can detect a single light substitution atom in graphene, which is possible only because of the electronic effect. In the case of hBN, the charge redistribution leads to a loss of the elemental contrast difference. Instead, the ionic character of the material is experimentally confirmed for the single layer. One key ingredient here is the extraordinary stability of the samples under the low-voltage electron beam, which allows us to obtain extremely high signal-to-noise ratios from long exposures. The precisely defined, ultrathin sample geometry enables a straightforward analysis. The DFT-based TEM image calculation is irreplaceable for the interpretation of experimental results in these materials, and can provide insights beyond the structural configurations.

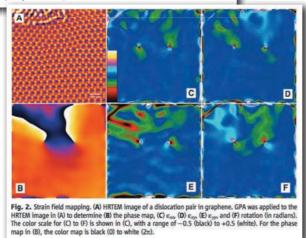


C_S-TEM of dislocations in graphene

Dislocation-Driven Deformations in Graphene

Jamie H. Warner, ¹* Elena Roxana Margine, ¹ Masaki Mukai, ² Alexander W. Robertson, ¹ Feliciano Giustino, ¹ Angus I. Kirkland ¹

The movement of dislocations in a crystal is the key mechanism for plastic deformation in all materials. Studies of dislocations have focused on three-dimensional materials, and there is little experimental evidence regarding the dynamics of dislocations and their impact at the atomic level on the lattice structure of graphene. We studied the dynamics of dislocation pairs in graphene, recorded with single-atom sensitivity. We examined stepwise dislocation movement along the zig-zag lattice direction mediated either by a single bond rotation or through the loss of two carbon atoms. The strain fields were determined, showing how dislocations deform graphene by elongation and compression of C-C bonds, shear, and lattice rotations.



SCIENCE VOL 337 13 JULY 2012

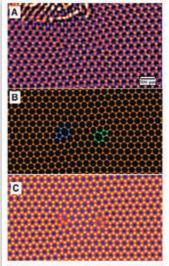


Fig. 1. Imaging edge dislocations. (A) HRTEM image showing two opposing (1,0) edge glide dislocations in graphene. (B) Structural model representing the dislocation pair (blue and green) in (A). (C) HRTEM image simulations using the atomic model in (B) as a supercell. False color is used for the images to aid visual inspection.

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Studies of monolayer MoS₂

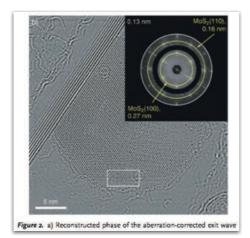
2010: Cs-TEM, 80 kV, TEAM 0.5 microscope

High-Resolution Microscopy

DOI: 10.1002/anie.200906752

Imaging ${
m MoS_2}$ Nanocatalysts with Single-Atom Sensitivity**

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copy are provided in the Supporting Information. Figure 2 shows the experimentally restored phase of a complex exit wave function of the catalyst, and reveals a nanometer-sized particle situated on a thin graphite flake. The wave reconstruction was performed to recover all obtainable information contained in a series of 15 HRTEM images of the nanoparticle acquired at different focal points [12] In such a phase image, the intensity maxima relate to atom positions and the contrast can reflect the chemical composition as represented in the projected scattering potential. Whilst the hexagonal shape of the nanoparticle is consistent with previously obtained images of this material, [14] the regular lattice in the interior part of the particle is now clearly visible. A lattice

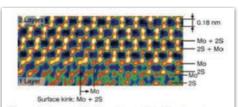
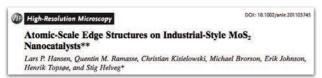


Figure 3. A close-up of the phase image obtained from the region near the bottom edge of the MoS₂ nanocrystal (partly contained in the white box of Figure 2 b). A red-green-blue color scale was applied to improve readability. The upper region is assigned to a double-layer MoS₂ slab, while the lower region is a single-layer slab. More generally, the chemical composition of individual columns can unambiguously be determined. A kink from a Mo + 2S column to a Mo column is indicated along the surface step from the double-layer to the single-layer MoS₂.

Studies of monolayer MoS₂

2011: Cs-STEM, 60 kV, SuperSTEM



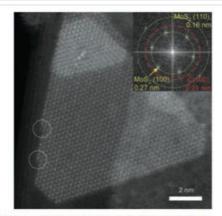
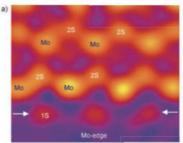


Figure 1. High-resolution STEM image of a MoS, nanocrystal supported on a graphite support. The insert is a fast Fourier transform (FFT) of the image and shows hexagonally arranged spots at the 0.27 nm and 0.16 nm lattice distances, corresponding to the MoS; (100) and (110) lattice planes, respectively. Moreover, a hexagonal set of lattice distances at 0.21 nm, corresponding to graphite (100), is also revealed. At the low-indexed edges, kink sites may be present as indicated by white circles.

indexed edge types. [12] The detailed structural information was obtained from a phase image representing a reconstruction of a through-focus series of HRTEM images of the sample. However, due to edge reconstructions during the acquisition of the consecutive images, a determination of the atomic-scale structure of the catalytically important outermost edges was not directly possible. These edge reconstructions are most likely caused by knock-on damage induced by the incident electron beam (with a kinetic energy of 80 keV), whereas ionization damage is expected to be negligible due to the electrical conductivity of the sample. The knock-on damage may be reduced by lowering of the primary electron energy below its threshold energy, [13] which is approximately 66 keV for MoS2 (see the Supporting Information). Only quite recently, HRSTEM under such conditions became possible. [14] Herein, we report HRSTEM images, acquired at low beam energy (60 keV), to obtain atomic-scale information about the edge structure of the industrial-style MoS2 nanocatalysts and we compare the images with edge strucel catalyst studies and DFT calcula-



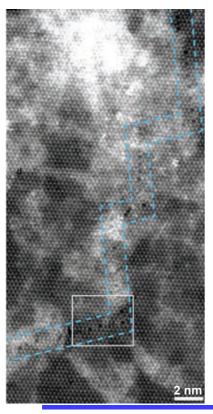
STEM image, which is probe-deconvoluted and colorized for improved visibility. The arrows point to single sulfur atoms that terminate the Mo edge as designated by an intensity analysis, b) Ball models (top and side views, respectively) of differ

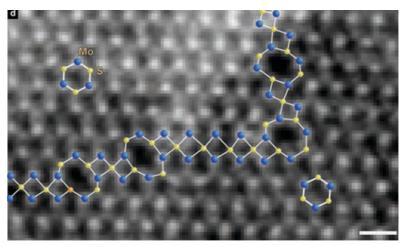
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Titan Themis Cs-STEM: CVD monolayer MoS₂

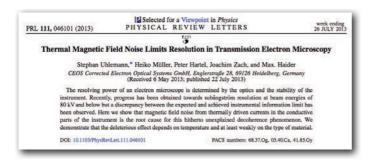




"Large-area MoS₂ grown using H₂S as the sulphur source" Dumitru Dumcenco et al. 2D Materials **2**(4) 2015

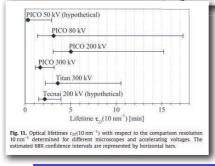
- 80 keV beam; even if below knock-on threshold can have beam-induced chemistry with residual gas molecules in column because not UHV (e.g. water etching).
- UHV or sample heating can be essential to good work!

Other limits



On the optical stability of high-resolution transmission electron microscopes

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iong as the optimum image is not available for a direct comparison, a further aspect can explain in a literal sense the low visibility of the problem: as has been shown in the previous sections, the optical lifetime decreases dramatically with increasing resolution, and as a consequence ultra-short lifetimes below 1 min can be obtained for the sub-Angström resolution regime. One has to bear in mind that mainly the extra high-resolution information below 1 Å is then adversely affected by aberrations. This extra-high resolution is however responsible only for a small part of the total

6. Conclusion

The usability of high-resolution transmission electron microscopes for controlled quantitative work depends crucially on the achievable level of optical stability, especially when aiming at Angström or even sub-Angström resolution. Our investigation shows that full aberration correction is only possible by providing also sufficient optical stability for the targeted resolution regime.

I. Barthel, A. Thust / Ultramicroscopy 134 (2013) 6-17

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C_S-TEM

- Harder to align precisely on zone axis (need to flip from diffraction to image)
- Interpret via: focal series reconstruction; negative Cs imaging; simulation
- Easy to obtain fringe image but precise Scherzer focus potentially challenging
- Contrast inversions with thickness remain; but can image very thick samples
- Damage: beam intensity spread, but total dose may be higher
- Coherent imaging: CTF determines resolution limit
- Atomic column analytics with (C_C-corrected) EFTEM less proven
- Camera properties important (MTF, "Stobbs factor")
- Picometer measurement precision
- Dynamics studies 25 fps easy, 1000 fps now possible (good for ETEM)
- Can still image samples which contaminate, e.g. organic molecules

C_S-STEM

- Easier to align precisely on zone axis (always in diffraction mode)
- Interpret via HAADF/MAADF/BF/ABF/ iDPC image
- Very limited DOF but very precise focus; camera-like focus
- Arguably thickness insensitive: sample first nms of thickness
- Damage: strong local intensity, but total dose may be lower
- Incoherent imaging: OTF determines resolution limit for HAADF
- Atomic column analytics with STEM-EELS; STEM-EDX also works
- Scan instabilities and detector noise important; need very stable scan
- Equally good precision
- Slower, but possible to follow movement of single atoms
- Need contamination-free samples only UHV possibility

Duncan Alexander: Cs-TEM vs Cs-STEM

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