Background, status and future of the Transmission Electron Aberration-corrected Microscope project

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The strong interaction of electrons with small volumes of matter make them an ideal probe for nanomaterials, but our ability to fully use this signal in electron microscopes remains limited by lens aberrations. To bring this unique advantage to bear on materials research requires a sample space for electron scattering experiments in a tunable electron-optical environment. This is the vision for the Transmission Electron Aberration-corrected Microscope (TEAM) project, which was initiated as a collaborative effort to re-design the electron microscope around aberration-correcting optics. The resulting improvements in spatial, spectral and temporal resolution, the increased space around the sample and the possibility of exotic electron-optical settings will enable new types of experiments. This contribution will give an overview of the TEAM project and its current status, illustrate the performance of the TEAM 0.5 instrument, with highlights from early applications of the machine, and outline future scientific opportunities for aberration-corrected microscopy.

Keywords: aberration correction; depth sectioning; monochromator; single-atom detection; light atom imaging; electron microscopy

1. Overview of the Transmission Electron Aberration-corrected Microscope project

Recent advances in aberration-correcting electron optics have led to increased resolution, sensitivity and signal-to-noise (S/N) ratio in atomic resolution microscopy. These advances have created the opportunity to directly observe the atomic-scale order, electronic structure and dynamics of individual nanoscale objects by advanced transmission electron microscopy (TEM). To take advantage of this opportunity, the Transmission Electron Aberration-corrected Microscope (TEAM) project was initiated to optimize the electron microscope around aberration-corrected electron optics and to further advance the limits of the instrument and the technique. The project brings together several leading microscopy groups supported by the US Department of Energy’s (DOE’s) Office

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of Science to jointly design and construct a new generation microscope with extraordinary capabilities (Kisielowski et al. 2008). Starting in 2000 with a series of workshops, TEAM became a project in 2004 as part of the DOE’s 20 year roadmap of Facilities for the Future of Science (see DOE website: http://www.science.doe.gov). After its completion, the instrument will be made available to the scientific user community at the National Center for Electron Microscopy (NCEM).

The vision for the TEAM project is the idea of providing a sample space for electron scattering experiments in a tunable electron-optical environment by removing some of the constraints that have limited electron microscopy until now. The resulting improvements in spatial, spectral and temporal resolution, the increased space around the sample and the possibility of setting up novel electron-optical configurations will enable new types of experiments in electron scattering. The TEAM microscope will feature unique corrector elements for spherical and chromatic aberrations, a novel atomic-force-microscopy-inspired specimen stage, a high-brightness gun and numerous other innovations that extend resolution down to the half-angstrom level. The improvement in sensitivity, brightness, S/N ratio and stability will make it possible to address major challenges such as single-atom spectroscopy and atomic resolution tomography.

The machine has been designed as a platform for a sequence of instruments, each optimized for different performance goals that were identified in a series of workshops at Argonne National Laboratory (ANL; 2000), Lawrence Berkeley National Laboratory (LBNL; 2002), Microscopy and Microanalysis (M&M) San Antonio (2003), M&M Savannah (2004) and M&M Honolulu (2005) (see TEAM website: http://ncem.lbl.gov/TEAM-project/). The most important scientific driving force that emerged from these workshops is the need for in situ experiments to observe directly the relationship between structure and properties of individual nanoscale objects. The project is guided by a Scientific Advisory Committee (see TEAM website: http://ncem.lbl.gov/TEAM-project/) and has received extensive input from the scientific community. The collaboration comprises several DOE-supported partner laboratories (ANL, Oak Ridge National Laboratory (ORNL), University of Illinois at Urbana Champaign (UIUC) and LBNL as the lead laboratory) and two commercial partners (FEI Company and CEOS GmbH). Its primary driving force has been the scientific goal to probe nanometre volumes of materials with atomic resolution.

A timeline of project milestones is given in figure 1. The TEAM instrument will be installed at the NCEM in 2009 and will be operated as a user facility to support the growing need for atomic-scale characterization in the nanoscience community. Its origin in the needs of nanoscience has imposed a set of important constraints on the project in terms of timing, goals and approach. To maximize the impact on the DOE nanoscience centres at Berkeley, Argonne, Oak Ridge, Brookhaven and Sandia National Laboratories, the project is being implemented in two stages, with an initial instrument (TEAM 0.5, without $C_c$ correction) starting operations as a user facility in October 2008 and the final instrument (TEAM I, with $C_c + C_s$ correction) becoming available to users in October 2009.

To be able to address materials issues across disciplines, the machine is designed to operate between 80 and 300 kV. Aberration correction for the illumination system and the image will allow 0.5 Å resolution in both scanning transmission electron microscopy (STEM) and TEM operating modes. On the
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**2. Instrument parameters and performance**

TEAM 0.5 is a specially designed microscope based on a commercially available FEI Titan 80-300 (scanning) transmission electron microscope platform. The instrument is isolated within a sound-proof enclosure placed inside a separate environmentally isolated laboratory space and operated from a remote workstation. Standard instrument alignments are performed with the aid of a high-sensitivity camera that transfers the information on the fluorescent screen in real time to the graphical user interface. The electron source of TEAM 0.5 consists of a novel high-brightness Schottky-type field emission gun. With an energy
Table 1. Typical residual axial aberration coefficients of the illumination aberration corrector and the imaging aberration corrector of TEAM 0.5 for operation at 300 kV. First-order aberrations, $C_1$ and $A_1$, are manually optimized. Adapted from Kisielowski et al. (2008).

<table>
<thead>
<tr>
<th>aberration coefficient</th>
<th>illumination (nm)</th>
<th>imaging (nm)</th>
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<tbody>
<tr>
<td>three-fold astigmatism $A_2$</td>
<td>24</td>
<td>43</td>
</tr>
<tr>
<td>second-order coma $B_2$</td>
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<td>38</td>
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<tr>
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<td>$-341$</td>
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<td>97</td>
<td>$1.0 \times 10^3$</td>
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<td>90</td>
<td>921</td>
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<tr>
<td>five-fold astigmatism $A_4$</td>
<td>$10.2 \times 10^3$</td>
<td>$13.4 \times 10^3$</td>
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<tr>
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<td>fifth-order spherical aberration $C_5$</td>
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<tr>
<td>fifth-order star aberration $S_5$</td>
<td>$6.6 \times 10^3$</td>
<td>$430 \times 10^3$</td>
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spread of 0.8 eV (full width at half maximum; FWHM), the brightness of the gun exceeds $4.5 \times 10^9\,\text{A\,cm}^{-2}\text{sr}^{-1}$ at 300 kV. A Wien-filter-type monochromator (Tiemeijer 1999) is capable of reducing the energy spread to 0.13 and 0.08 eV at 300 and 80 kV, respectively. The instrument uses the standard three-condenser lens system of the Titan 80-300 column to provide variable probe convergence angles in STEM mode and adjustable parallel illumination in TEM mode.

An advanced probe corrector derived from the hexapole-type corrector from CEOS corrects for objective lens illumination aberrations (Haider et al. 2000; Müller et al. 2006). The imaging aberration corrector is a hexapole-type corrector from CEOS (Haider et al. 1998). The high-resolution Ultratwin objective lens has an inherent third-order spherical aberration coefficient $C_3 (= C_s)$ of 0.66 mm (300 kV) and a (relativistic) coefficient of chromatic aberration $C_c$ of 1.64 mm. The system includes a retractable Gatan US1000 slow-scan CCD camera and a high-resolution GIF Tridiem 866. In its current configuration, TEAM 0.5 is operated either at 300 or 80 kV. All optical elements are activated, including both aberration correctors, independent of the operation mode. This minimizes thermal drift because, at a given accelerating voltage, switching between different operation modes does not change the thermal load on the system.

Both correctors on TEAM 0.5 correct for coherent lens aberrations, in particular the spherical aberration of the objective lens, and partially for higher order aberrations and resulting parasitic aberrations. The correctors impose a slight increase in the chromatic aberration of the system, changing the inherent $C_c$ of the objective lens from 1.64 to approximately 2.1 mm with the correctors activated, for both TEM and STEM operating modes.

The illumination aberration corrector is an improved version of the hexapole corrector (Haider et al. 2000), suitably upgraded to minimize the impact of six-fold astigmatism $A_5$ (Müller et al. 2006). It is configured to fully correct coherent axial aberrations up to fifth-order spherical aberration $C_5$. Table 1 gives a list of typical residual aberration coefficients that are measured after fine-tuning of the corrector at 300 kV.
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The imaging aberration corrector fully corrects for coherent axial aberrations up to third-order and partially compensates for fourth- and fifth-order aberrations. Whereas off-axial aberrations are negligible for the illumination aberration corrector, they must be considered in the setup of the imaging corrector. The ‘aberration-free’ area in the imaging mode (15.9 mrad) is smaller than in the illumination mode (25.2 mrad). This is due to the fact that it is possible to compensate fourth-order aberrations, six-fold astigmatism $A_5$ and fifth-order spherical aberration $C_5$ with the improved illumination aberration corrector, but these coefficients, including third-order aberrations, are larger on the imaging side.

In STEM mode, the illumination aberration corrector is used to reduce the effect of coherent aberrations on the electron probe and to increase the probe convergence angle such that both lateral and depth resolution are improved (van Benthem et al. 2006). The electron probe that provided the highest lateral resolution was obtained by using a semi-convergence angle of 30 mrad, with a probe current of approximately 50 pA. For this setting, even without employing the monochromator, the chromatic aberration due to an inherent energy spread of approximately 0.8 eV (FWHM) does not severely affect the probe size.

The performance of TEAM 0.5 in STEM and TEM is illustrated in figure 2. A STEM image of GaN in [211] orientation (figure 2a) shows the 0.63 Å distance between Ga dumbbells clearly resolved. The corresponding diffractogram in figure 2b shows Fourier components beyond the 50 pm marker indicated by the circle. The Fourier diffractogram in figure 2c from high-resolution TEM images of an Au test sample supported on an amorphous carbon film shows Young’s fringes extending beyond the 50 pm mark indicated by the circle. It was found that, without the use of the monochromator, Young’s fringes only reached to about 70 pm. For a discussion of the relationship between information transfer and resolution, see den Dekker & van den Bos (1997), Van Aert et al. (2006) and Peng et al. (2008), and for a discussion of the use of Young’s fringes to measure information transfer, see Barthel & Thust (2008).

Figure 2. Performance of TEAM 0.5 in STEM and TEM at 300 kV. (a) Aberration-corrected high-resolution STEM image of GaN in [211] orientation, showing the 0.63 Å distance between Ga dumbbells clearly resolved (see inset model). Scale bar, 2 nm. (b) The corresponding diffractogram shows Fourier components beyond the 50 pm marker indicated by the circle. (c) The Fourier diffractogram from high-resolution TEM images shows Young’s fringes extending beyond the 50 pm mark indicated by the circle.
3. Initial applications

This section describes a selection of initial applications of the TEAM 0.5 instrument to a range of scientific issues. The examples serve to demonstrate the performance of the microscope, as well as its ability to address problems in materials science. Several of these examples are from publications that have appeared recently or are presently under review or in preparation.

(a) Detection of buried defects

An important limitation for electron microscopy is imposed by the projection geometry. It has been pointed out that the focal depth of an aberration-corrected microscope in the STEM mode can be reduced by increasing the convergence angle of the probe (Borisevich et al. 2006) or by using a confocal geometry (Frigo et al. 2002; Nellist et al. 2006). This makes it possible to retrieve detailed structural information from defects contained within the crystal volume (Lupini et al. in press). Figure 3 shows high-angle annular dark field (HAADF) STEM images of an Au [111] crystal with the electron beam focused on the crystal surface (figure 3a) and defocused by 7 nm (figure 3b). The image in figure 3a shows a typical $\Sigma 3\{112\}$ grain boundary separating two grains related by a $60^\circ$ rotation about the axis of projection. When the probe is focused 7 nm into the crystal, the lower right segment of the boundary remains unchanged, indicating that this part of the boundary spans the whole thickness of the foil. At the same time, the upper left segment almost disappears, indicating that it is not present in the lower part of the sample, while a new boundary segment appears at the lower left. This buried segment is another $\Sigma 3\{112\}$ boundary that branches off the main segment, as shown in the three-dimensional schematic in figure 3c. Not shown in this schematic is the $\{111\}$ segment parallel to the surface that connects the upper and lower branch. This geometry, termed 'double positioning' (Dickson & Pashley 1962) is a result of the low stacking fault and twin boundary energy of Au and the large anisotropy of $\Sigma 3$ grain boundaries. A more detailed description of the bicrystal geometry can be found in Westmacott et al. (1999). From calculated probe profiles, we estimate a
possible depth precision significantly better than 6 nm. This kind of sensitivity to three-dimensional defects can be of great utility in studying buried interfaces, precipitates and even dopant atoms.

(b) **Li-rich precipitates in Al–Li–Sc–Zr alloys**

Al–Li alloys exhibit low density and high strength-to-weight ratio. These characteristics are of great interest for aerospace and cryogenic applications. The alloy’s mechanical properties are based on a fine dispersion of coherent Al₃Li metastable precipitates formed by congruent ordering and spinodal decomposition. Other elements, such as Sc and Zr are added to Al–Li alloys to further improve their properties. These alloys contain a fine distribution of core/shell precipitates consisting of an Al₃(ScLi) core surrounded by a shell of pure Al₃Li. Hardness values up to 1330 MPa have recently been demonstrated in similar alloys (Krug et al. 2008). Because Li atom positions are closely linked to the electronic structure of precipitates and therefore the alloy’s physical properties, it is important to characterize the Li partitioning. However, direct imaging of atomic columns of light elements, particularly when surrounded by heavier elements, has been limited by the sensitivity and resolution of conventional transmission electron microscopes, and extensive data analysis has been required to detect the weak signal from light elements such as Li (Shao-Horn et al. 2003).

By applying the TEAM 0.5 instrument in TEM and STEM modes to this alloy, we were able to clearly image Li columns in Al₃Li and detect the distribution of Li and Sc in these precipitates. Figure 4 illustrates the microstructure of the alloy using energy filtered jump ratio maps of Li and Sc, indicating that Sc is confined to the core, while Li is primarily located in the shell. In figure 4c, an aberration-corrected HAADF STEM micrograph of the core/shell interface confirms that the core region also maintains L1₂ order, with the bright Sc atom columns defining a square pattern (see schematic of L1₂ structure in figure 5) that is strictly aligned with the Li sublattice in the shell (missing columns in figure 4c).

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From a focal series of TEM images recorded on the instrument, we reconstructed the exit-plane wave function of one of the precipitates. The phase of the exit wave in figure 5a reveals the Al$_3$Li long range order, where Al columns can be clearly distinguished from Li columns (cf. schematic in figure 5b). These observations demonstrate the increased sensitivity of aberration-corrected microscopy and the complementary nature of TEM and STEM imaging modes.

(c) Graphene layers

Since it recently became possible to isolate single layer graphene sheets, a large research effort is now directed at the characterization of their lattice imperfections, which control electronic, thermal and mechanical properties of graphene (e.g. Son et al. 2006; Heersche et al. 2007). In contrast to defects in carbon nanotubes, the structure and dynamics of defects in a planar graphene layer remain largely unexplored.

At 200 and 300 kV, rapid radiation damage makes the study of graphene sheets by electron microscopy quite challenging. But, by using the TEAM 0.5 instrument at 80 kV in the monochromated aberration-corrected mode, it has been possible to achieve the necessary resolution below the critical threshold for displacement damage (Meyer et al. 2008a,b). This offers the capability to resolve every carbon atom in a suspended single-layer graphene sheet. Experimentally, it was found that, even for single-shot data acquisition, each atom in the field of view is detected with a signal well above the noise. This is illustrated in figure 6a, which shows a large-scale view of a graphene sheet partially covered by organic residue. Contrast from a single adatom is clearly apparent above the background in the region marked by an arrow. S/N ratios can be further boosted by reconstructing the electron exit wave function from focal series of images (figure 6c) to obtain S/N ratios of 8–10 for the detection of a single carbon atom. In principle, such values even allow the unambiguous detection of single hydrogen atoms if they can be kept in place during the imaging process.

It was possible to directly image theoretically predicted configurations such as the Stone–Wales defect shown in figure 6b, and explore their real-time dynamics. These defects are well known to be involved in the coalescence of fullerenes and...
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Figure 6. Graphene images recorded at 80 kV. Point defect visible in large-scale view (see arrow in (a); scale bar, 2 nm) and (b) a detailed analysis of a Stone–Wales defect made of pentagon–heptagon rings. Adapted from Meyer et al. (2008a). (c) Reconstructed electron exit wave function.

nanotubes (Kim et al. 2003; Yoon et al. 2004), and their mobility is relevant for the plastic response of carbon nanotubes under strain. However, in this work, it was observed that, for graphene sheets, the separation of pentagon–heptagon pairs is clearly not the favoured pathway. In contrast to highly curved graphene structures, all multiple pentagon–heptagon defects were observed to relax to the original unperturbed lattice. These results indicate significant differences in defect dynamics of flat and closed-shell graphenes such as nanotubes or fullerenes.

(d) Au nanobridges

The atomic structure of 90° ⟨110⟩ tilt grain boundaries in Au is important as a model system for the behaviour of high-angle grain boundaries in face-centred cubic materials. One particular inclination of this boundary is of special interest because it is characterized by a direction in which the two grains are incommensurate. The structure of this boundary has been investigated by high-resolution microscopy (Penisson et al. 1999; Radetic et al. 2002), and atomistic simulations predict vanishing static friction for gliding along this direction (Lançon et al. 2000). However, the ability to investigate the atomic structure has been limited by the S/N ratio of high-resolution images, the geometric constraints of the sample and the stability of samples under the influence of the electron beam. Initial applications of TEAM 0.5 to this grain boundary have produced outstanding images with single-atom sensitivity and a contrast that is strongly dependent on defocus.

Figure 7 shows one image from a 15-member focal series that illustrates this sensitivity. The image shows a grain boundary contained in a nanobridge of Au, connecting two grains rotated 90° relative to each other about the beam direction. Owing to the low stacking fault energy of Au, the boundary is dissociated into twin-related segments, which are readily discernable by direct inspection of the structure.

From experimental line profiles, it was determined that a single Au atom is recorded with a S/N ratio of 10 in a single lattice image and attenuates the incoming electron beam by approximately 25 per cent, while two-atom columns nearly double this attenuation. The arrow at the left-hand side of the bridge points out a single atom that remains after another atom escaped from observation following the previous frame. This conclusion is based on a comparison of
Figure 7. TEM lattice images of an Au nanobridge connecting two grains that are rotated relative to each other by 90° around a common (110) axis. The image is part of a 15 member focal series, recorded in time intervals of 1.5 s. The left arrow points to a single atom. The arrows on the right point out 13 two-atom columns, some of which disappear in the following image due to the influence of the electron beam. Adapted from Kisielowski et al. (2008).

Figure 8. Time sequence of a nanobridge connecting two grains rotated by 90° around a common (110) axis. Current density $4 \times 10^5$ A m$^{-2}$, monochromator off. In (a) and (b), the grain boundary displays an extended structure, visible as an apparent curvature of the lattice planes. An abrupt structural change between frames (b) and (c) leads to a multiply twinned arrangement that remains stable during continued irradiation in (e) and even after the connection is severed in (f).

simulated and observed contrast for single- and double-atom columns over a range of defocus conditions, as shown in the adjacent graph. The contrast difference between one and two atoms is particularly clear in the defocus range between $-4$ and $-8$ nm.

Similar simulations show that the row of atoms between arrows at the right of the image is two atoms high, with a systematic contrast variation along the row that is likely to reflect differences in $Z$-height.

The effect of electron irradiation on the structure of the sample is illustrated in the image sequence shown in figure 8. The images were recorded at 300 kV over a period of about 2 min at a beam current of $4 \times 10^5$ A m$^{-2}$, and demonstrate
the rate of sputtering caused by the electron beam. Throughout this sequence, the contrast level and high S/N ratio of images makes it possible to identify the atomic structure directly and with high precision.

The crystallography is identical to that in figure 7—the nanobridge accommodates a $90^\circ$ misorientation between the grains on either side of the image. The apparently curved lattice planes visible (figure 8a,b) are due to the well-known dissociation of the $\{111\}/\{112\}$ boundary. In figure 8c, the bridge has relaxed into a new, multiply twinned structure that remains stable even after the connection between the grains is severed (figure 8f).

Although the dynamic features observed here have not yet been analysed in detail, it is clear that the improved capabilities of the new microscope will enable direct atomic-level observation of phenomena, such as crystal growth in three dimensions and with a time resolution greatly exceeding the current rate of about 1 s.

4. Future directions

The capabilities of the new level of aberration-corrected instruments described here significantly exceed those of previous generations of microscopes. This opens up new opportunities to explore materials at the atomic level. Some of these opportunities can be realized now without the need for further developments. All that is required is good ideas and good samples. The TEAM 0.5 microscope is available free of charge to any qualified user, after peer review of a scientific user proposal (for details, see http://ncem.lbl.gov/).

Other opportunities will require new developments of instrumentation or technique. Among these, one of the most important is the original driving force for the TEAM project itself, namely the development of a tunable electron-optical observatory for materials research. By utilizing the additional space available in the objective lens for dynamic experiments, it will be possible to advance to a new level in the fundamental structure/property correlation that lies at the heart of materials science. At this level, it will be possible to correlate the properties of a single nanostructure to its local atomic structure. The aberration-corrected beam will offer a high-resolution probe for imaging and spectroscopy. Any experiments that provide a direct correlation between the physical behaviour of a single nanoscale object and its atomic or electronic structure, composition or bonding will contribute greatly to our understanding of the nanoworld.

Some important developments that are needed to make this a reality are faster, more sensitive electron detectors, a multitude of in situ stages that allow other probes and stimuli to interact with the sample and that can introduce gaseous (or liquid) environments or impose fields or gradients.

The notion of a tunable electron-optical environment includes the ability to tune the electron energy to the material or problem at hand. The TEAM instrument is based on the community’s consensus that the range of 80–300 kV covers the important region determined by the balance between usable sample thickness, tolerable radiation damage and achievable resolution. However, this balance is shifting. The success of aberration correction, especially when chromatic aberration can be minimized, makes lower-voltage microscopy more accessible. At the same time, many future samples may no longer be foils thinned

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down from bulk materials, but small nanoparticles or molecules supported on single-atom thick sheets such as graphene or similar compounds. For such samples, aberration-corrected imaging at much lower voltages may become advantageous. In view of the higher brightness electron sources, and the greatly improved S/N of this new generation of microscope, it will be important to re-examine the sensitivity to radiation damage, especially as light elements are becoming accessible.

The need for a tunable electron-optical environment is particularly urgent at the interface between hard and soft materials. The two branches of microscopy have evolved along different lines so that simultaneous imaging of soft and hard materials is currently not possible. By combining a phase plate with an aberration corrector, a cryostage and a highly sensitive direct electron detector, it will become possible to image such interfaces as that between a polymer and a semiconductor or the interfaces that control the process of biomineralization. This is an anticipated growth area where new developments in electron microscopy instrumentation and techniques will have a great impact.

Dynamic observations of atomic processes such as quasi-melting of Au particles (Iijima & Ichihashi 1986; Smith et al. 1986) or the motion of interfaces or defects in compound semiconductors (Sinclair et al. 1988) were among the most effective early demonstrations of the power of electron microscopy. The S/N in TEAM 0.5 enables a quantitative measurement of such atomic processes. With a sufficiently fast detector, it will be possible to record observations, such as those shown in figure 8 above, with a high enough rate and quality to allow direct and quantitative comparison with atomistic simulations on the same scale. To go beyond the range of milliseconds necessitates major developments in dynamic electron microscopy, such as those that are currently underway at Livermore and at CalTech.

There is a great need for techniques and software to support three-dimensional tomography. The current state of the art is defined by the substantial body of knowledge developed in biology. For materials science, additional features need further development, including accurate stage control for $k$-space navigation, the ability to combine information from different datasets such as depth sectioning in STEM imaging, tilt series or external constraints. To minimize exposure, it will be important to develop more efficient techniques of eucentric sample tilt with high accuracy. Appropriate image analysis techniques are needed to assure a linear relationship between image intensity and projected thickness. This is particularly important for phase contrast imaging and tomography using other signals such as magnetic or electric fields or selected features in the energy loss spectrum. As part of the TEAM project, a software framework is being developed, which will serve as a platform for tomography.

5. Summary

The scientific opportunities described here represent only a small sample of the great range of possibilities opened up by the current developments in aberration-corrected optics. This is a good time to capitalize on the success of this effort by pursuing other developments that are now within reach. The approach taken under the TEAM project of a parallel collaborative development is new for
the microscopy community. It is to be hoped that the successful progression of the project will set a precedent that forms a solid base for future projects and opens the possibility of larger scale developments in electron microscopy.

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