REPORT

Third TEAM Workshop

San Antonio, Texas August 8, 2003

Organizers:

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The Third Transmission Electron Aberration-corrected Microscopy (TEAM) Workshop

EXECUTIVE SUMMARY

The Third TEAM Workshop covered scientific challenges and enabling technologies for the development of the Transmission Electron Aberration-corrected Microscope (TEAM). TEAM promises to be a new and revolutionary instrument for the characterization of condensed matter via electron scattering, and will have broad impact by facilitating unique experiments across many scientific disciplines.

This workshop highlighted a number of specific scientific challenges ranging from size-dependent behavior of nanomaterials to atomic-level imaging of oxygen in superconductors, to characterization of polymers and macromolecules. Aberration correction was recognized as a key element in meeting these challenges.

The proposed design of the TEAM instrument features a number of novel concepts and technologies, which were addressed in four breakout sessions focused on *correctors, detectors, stages* and *tomography*. Preliminary calculations and prototyping indicate that there exists no fundamental impediment to any feature of the proposed instrument. In particular, a plausible design has been achieved for a chromatic-aberration-corrected image-forming lens. Other technical challenges for TEAM include: a new electron gun design with improved monochromaticity; a completely redesigned specimen stage; novel primary electron and spectroscopic detectors; and new technique development for tomographic imaging.

It was recommended that different approaches to the TEAM scientific objectives should continue to be explored, and that the ability to synthesize, manipulate or modify the sample *in-situ* during electron beam microcharacterization should remain a high priority.

WORKSHOP PUBLICITY AND ATTENDEE PROFILE

Outreach:

The Third TEAM Workshop was publicized through e-mail solicitations, through posting on high-visibility web sites, and through advertisement at M&M2003, the annual meeting of the Microscopy Society of America. Roughly 5000 e-mails sent to target groups directed prospective participants to the official TEAM web site (ncem.lbl.gov/team3.htm). E-mail invitations for TEAM3 participation were distributed through the American Physical Society (APS) and the Materials Research Society (MRS). These e-mails targeted members of the APS Division of Materials Physics and MRS special interest groups, including Catalysis, Structural Materials, Geology, Semiconductors, Polymers and Organics, Magnetic Materials, Nanoscale Materials, and Modeling on an Atomic Scale. E-mail notifications were also sent to users of the DOE-BES Electron Beam Microcharacterization Centers (EBMCs) and to participants of the TEAM2 (LBL, 2002) and TEAM1 (ANL, 2000) workshops. TEAM3 was publicized on the web site of the Materials Research Society (MRS) and also on the M&M2003 web site, which is heavily trafficked by M&M2003 registrants and by the membership of the sponsoring societies, the Microscopy Society of America (MSA), the Microbeam Analysis Society (MAS), and the International Metallographic Society (IMS), especially in the months of June and July. TEAM3 was also publicized during M&M2003 through the distribution of flyers and through the M&M Daily Newsletter, the official publication of the meeting that notifies attendees of important daily events and last-minute changes to the program, such as late-breaking posters.

A first announcement of the workshop appeared by mid-May 2003, followed by second and third announcements by end of June and mid July, respectively (Appendix 1). The final workshop program (Appendix 2) was distributed with the third announcement. Together with the submitted abstracts it was handed out as a brochure during the workshop, which was free of registration charges.

Participation:

An estimated 150 people participated in the Third TEAM Workshop, of whom 95 preregistered and 41 registered on-site on the day of the workshop; the additional attendees present did not choose to leave contact information. The significant number of on-site registrants can be attributed to advertising at the M&M2003 meeting, including especially instrument manufacturers.

The registered participants were affiliated with universities (54), DOE laboratories (43), other federal/national laboratories (5), government agencies (2), and industry (31). Foreign countries represented (20) included Australia (3), Belgium (1), Canada (3), Germany (6), Japan (2), Mexico (1), Netherlands (2), Taiwan (1), and the United Kingdom (1). Industry participation included representatives of all major electron optical instrument manufacturers (FEI, Hitachi, JEOL, LEO, NION, CEOS, Gatan), microscope stage and accessory provider E.A. Fischione Instruments, and Hewlett-Packard, IBM, Motorola and Zyvex.
A detailed list of the registered participants is attached (Appendix 3).

WORKSHOP REPORT

The workshop was comprised of a morning plenary session outlining scientific challenges for the TEAM project, followed by four parallel afternoon breakout sessions that covered enabling technologies for the TEAM scientific objectives: electron optics, specimen stages, detectors, and tomographic methods. Abstracts were solicited in advance of the meeting for all sessions. The abstracts submitted for presentation at the workshop are attached (Appendix 4).

PLENARY SESSION: SCIENTIFIC CHALLENGES AND OBJECTIVES FOR TEAM

Summary:
The plenary morning session was attended by about 140 participants. The program featured 11 distinguished speakers from universities, national labs and scientific institutes in the US, Canada, Mexico and Germany. The session covered a broad range of scientific topics each of which will benefit directly from the development of aberration correction, from nanophase structure and stability to biology.

Chair J. Spence, ASU, Presenters:

- U. Dahmen, NCEM, LBNL
- A. Navrotsky, UC Davis
- D.C. Larbalestier, U. Wisconsin
- R.W. Scott, Texas A&M
- G.A. Botton, McMaster University, Canada
- J. Plitzko, MPI Martinsried, Germany
- G. Liu, LBNL
- V. Leppert, UC Merced
- J. Yang, U. Pittsburgh
- J. Reyes-Gasga, UNAM, Mexico

Report:
The workshop opened with a description of the TEAM objectives (U. Dahmen). To reach out to potential users of the TEAM microscope, the organizers aimed for a range of speakers with diverse backgrounds and levels of expertise, including:
-*Thermodynamics of nanomaterials* (A. Navrotsky)
-*Superconductivity* (D.C. Larbalestier)
-*Catalysis* (R.W.J. Scott)
-*Biology* (J. Plitzko)
-*Lighting with organic LEDs* (G. Liu)
-*Combination of soft - with hard nanomaterials* (V. Leppert)
-*Corrosion* (J.C. Yang)
-*Biomedical materials* (J. Reyes-Gasga)
-*Advanced electron spectroscopy* (G.A. Botton).

On several occasions invited speakers referred to goals of the TEAM project. For example, the general need was emphasized to increase the sensitivity of the current generation microscopes, which can be achieved through aberration correction and resolution enhancement. Several speakers (D.C. Larbalestier, V. Leppert, J. Reyes-Gasga) felt that the current instrument sensitivity is a most limiting factor in their investigations. Explicitly, the need for imaging of oxygen columns and a better sensitivity for spectroscopy were emphasized.
There is a general understanding that Cs correction will provide sensitivity improvements if applied to either the probe or the objective lens. Science will dictate the choice of experiments and it was pointed out from the audience (J. Silcox) that - historically - novel instrumentation paved the road for scientific breakthroughs. Several speakers adopted this view. Beyond Cs correction, the benefit of Cc correction for biological investigations was stressed (J. Plitzko).
Other talks made clear that aberration correction should not only benefit transmission electron microscopy but also should be applied to scanning electron microscopy (G. Liu) and in-situ experiments (J.C. Yang).

Breakout Session: Sample Holders and Stages - Enabling New Scientific Experiments

Summary:
From an engineering standpoint, the level of control needed to address the TEAM scientific objectives is achievable, but will require an entirely new sample stage design, image-based drift compensation, and great attention to thermal stability. The sample itself will likely be a limiting factor for achieving new experimental paradigms. The ability of the researcher to modify the sample *in-situ* during electron microcharacterization should remain a high priority.

Participation: 20-25 total. Moderator: R.D. Twesten. Presenters:
- H. Zandbergen, Delft University of Technology, The Netherlands
- N. Salmon, Lawrence Berkeley National Laboratory, Berkeley, CA
- C. Baur, Zyvex Corp., Richardson TX
- M. Wall, Lawrence Livermore National Laboratory, Livermore, CA
- E. Stach, Lawrence Berkeley National Laboratory, Berkeley, CA
- J-G Wen, Frederick Seitz Materials Research Laboratory, Urbana, IL

The major instrument manufacturers were represented at the breakout session, as were several manufacturers of microscope accessories.

Report:
The goal of the session was to explore design requirements for sample holders and stages needed to achieve the scientific goals of the TEAM project. Informal presentations by researchers with experience in sample holder design were used to define the current state-of-the-art and a starting point for discussions. While no single paradigm emerged for sample manipulation, consensus was achieved on several key points.

- Current generation side-entry stages will not meet the TEAM specifications. These stages were designed for user convenience and not ultimate performance. Two paradigms emerged for improved performance: 1) Embedding the stages directly within the objective lens; 2) Using a cantilevered system, similar to an SEM stage. Both stage designs would require a load-lock system. A dual-stage design that features a traditional side-entry stage for convenience as well as a high-performance stage, would improve functionality and flexibility, but at the price of system complexity.
- The need for 6-axis control (x,y,z, 2-tilts, plus sample rotation) was clear. Sub-nanometer closed-loop translational control and eucentric sub-milliradian tilting control will be needed to enable the reproducible positioning and orientation of the stage required for tomography. Deep sub-nanometer control is likely unnecessary. The required specifications can be achieved by careful engineering.
- The effect of thermal changes on instrument performance is likely to be a critical factor; several strategies for improving thermal stability were discussed.

Breakout Session: Emerging Detector Technologies

Summary:
A variety of detector technologies will aid in achieving the scientific goals and improve the scientific versatility of the TEAM development project. A different combination of detectors may be appropriate for different experiments, and the development of detectors optimized for different modes of signal collection is strongly recommended. Simulation can aid in the specification of various detector parameters for a given experiment.

Participation: 20-30 total. Moderator: J.S. Wall. Presenters:

- J. S. Wall, Brookhaven National Laboratory, Upton NY
- P. Mooney, Gatan Research and Development, Pleasanton CA
- N. J. Zaluzec, Argonne National Laboratory, Chicago IL
- E. A. Kenik, Oak Ridge National Laboratory, Oak Ridge TN
- M. M. G. Barfels, Gatan Research and Development, Pleasanton CA

Report:
The current state-of-the-art and opportunities for advancement of primary electron, electron energy-loss (EELS), and energy-dispersive X-ray (EDX) detectors used in electron beam microcharacterization were discussed. Primary electron detector designs typically balance spatial resolution (pixels) with temporal resolution (frame rate); spectroscopic detectors (EELS, EDX) typically balance spectral resolution with limited signal intensities.

- CCD detectors (P. Mooney) and the means for assessing their performance are steadily improving. Current state-of-the-art is 4k x 4k pixels with 10^{-2}-1 s^{-1} frame rates at 100 keV; performance decreases at higher voltages.
- CMOS detectors (J.S. Wall) with 1.3k x 1k pixels & $5*10^{2}$ s^{-1} frame rate have a detection performance similar to CCDs and spatial resolution is rapidly increasing, driven by needs and capabilities of semiconductor industry.
- Prototype high-speed primary electron detector (J.S. Wall) with 32 x 32 pixels & 10^{4} s^{-1} frame rate can read out a convergent-beam electron diffraction (CBED) pattern with high quantum efficiency for each beam position in a raster image.
- EELS detectors (M.M.G. Barfels) are experiencing improvements in electronics stability, acceptance angle, spectral resolution and detection efficiency for monochromated beam with higher spectral resolution but lower signal intensity.
- High throughput silicon drift EDX (N.J. Zaluzec) and high spectral resolution microcalorimeter EDX (E.A. Kenik) detectors offer significant improvements in one or more performance metrics relative to current generation EDX (135 eV spectral resolution / ~$3*10^{3}$ s^{-1} count rate / 0.3 sr solid angle). Silicon drift EDX (135 eV / $2*10^{5}$ s^{-1} / 0.4 sr) ideal for X-ray mapping whereas microcalorimeter EDX (9 eV / 10^{3} s^{-1} / 10^{-4} sr) optimal for minimizing spectral overlaps and possibly detecting chemical shifts. Detector arrays or focusing optic can be used to mitigate low collection solid angle of microcalorimeter.

Breakout Session: From 2D to 3D – Tomographic Methods

Summary:
The TEAM project goal of atomic-scale tomography of individual nanostructures cannot be achieved with present methods; however, both established and newly emerging approaches to tomographic reconstruction hold promise for meeting this goal. The large electron dosage typical of tomography studies presents a significant challenge.

Participation: 20-60 total. Moderator: C. Kisielowski. Presenters:

- S.J. Pennycook, Oak Ridge National Laboratory, Oak Ridge TN
- J.C.H. Spence, Arizona State University, Tempe AZ and LBNL
- P.A. Midgley, University of Cambridge, United Kingdom
- J. Batenburg, Leiden University, The Netherlands
- F.-R. Chen, Tsing-Hua University, Taiwan
- M.P. Oxley, University of Melbourne, Australia
- W. Qin, University of Missouri, St. Louis and Motorola

Report:
The established method for electron tomography (Midgley) of acquiring a series (>100) of images over a large range ($\pm$ ~70) of specimen orientation in small (1-2) increments is well understood but time consuming and prone to radiation damage of the specimen. Tomography has been demonstrated for both STEM (BF, HAADF) & TEM (BF, EFTEM) approaches with a best resolution of 1-2 nm. The method is most promising for highly radiation-resistant materials.

- Typical rotation over a single axis creates "missing wedge" effect; interpretation of image series with two rotational axes is demanding.
- Improvement in resolution may be possible for small nanostructures (<10 nm).
- Spectroscopic tomography (e.g., EELS) is possible, but low cross sections require higher electron dosage and thus beam damage.

A number of alternative approaches to electron tomography hold promise.

- Diffractive Imaging (J.C.H. Spence) can recover projected sample shape at atomic resolution from an individual diffraction pattern. Approach cross-cuts radiation sources (electrons, x-rays, photons) but sample support presents a challenge.
- Confocal HAADF imaging (S.J. Pennycook) may provide 3D image of object from a series of images at a single orientation. Depth resolution of the technique may be limited to the spread of defocus, ~4 nm for current TEAM design. Theory needs development in order to address possible resolution limits.
- Discrete Tomography (J. Batenburg) can dramatically reduce the number of required projections for 3D reconstruction (from ~300 to ~10). Reconstruction procedures on a discrete grid has been developed and evaluated mathematically. Could significantly mitigate beam damage if atomic depth resolution achievable.

The extension of tomographic techniques to atomic resolution holds promise. Both STEM-HAADF and TEM Electron Exit Wave Reconstruction have demonstrated single atom sensitivity in exceptional cases (C. Kisielowski). Quantification of intensities from TEM Electron Exit Wave Reconstruction can be reproduced to an accuracy of about one percent independent of a particular reconstruction process (M.P. Oxley).

Breakout Session: Electron Optical Developments - Aberration Correctors, Monochromators, and Theoretical Basis

Summary:
No fundamental impediment exists for the ambitious lens designs of the TEAM instrument. Feasible designs with stabilities consistent with current technology exist for all lens elements, including a chromatic-aberration-corrected image-forming lens. Various approaches to TEAM scientific objectives, including STEM and TEM approaches and different gun-monochromator designs, should continue to be explored.

Participation: 30-40 total. Moderator: I.M. Anderson. Presenters:

- H. Rose, (retired) University of Technology, Darmstadt, Germany
- M. Haider, Corrected Electron Optical Systems GmbH, Heidelberg, Germany
- N. D. Browning, University of California, Davis CA and LBNL
- D. A. Muller, Cornell University, Ithaca NY
- M. A. O'Keefe, Lawrence Berkeley National Laboratory, Berkeley CA
- J. A. Eades, Lehigh University, Bethlehem PA
- P. Schlossmacher, LEO Electron Microscopy GmbH, Oberkochen, Germany
- Z. Yu, Cornell University, Ithaca NY

Report:
The prognosis for the proposed electron optical design of the TEAM instrument is good. Preliminary implementations of both STEM and TEM approaches to aberration-corrected electron beam microcharacterization have been demonstrated, and proponents of the two approaches are confident of the feasibility and scientific impact of next-generation lens designs. A healthy and spirited rivalry between these two groups should hasten the pace of electron optical developments. Design criteria must focus on achieving adequate signal for atomic-scale analysis with minimum specimen irradiation, rather than ultimate achievable performance, with attention to the electron optical system as a whole, thus maximizing the scientific impact of aberration correction.

- Designers of electron optical lens systems are confident of a sound theoretic basis for the various electron optical elements proposed for the TEAM instrument.
- Monochromators of several distinct designs have been developed and third-order spherical aberration (C_3) correction for both STEM probe- and TEM image-forming lenses have been successfully demonstrated over the past few years.
- A design by Harald Rose entitled the "superaplanator" has been proposed for the most technically unproven of the TEAM electron optical lens concepts, an image-forming (TEM) lens that is corrected for chromatic aberration (C_c). An imaging resolution of 0.05 nm should be achievable with a limited number of lens elements and lens stabilities of 0.2 ppm, consistent with current technologies.
- Monochromation of the incident electron beam provides advantages for spatial resolution of both TEM imaging and STEM spectroscopies, in addition to the spectral resolution of electron energy-loss spectrometry.
- Monochromators in combination with both Schottky- and cold-field-emission electron guns are expected to yield comparable probe currents in a 0.1 eV FWHM

incident electron beam; design criterion is energy spread with adequate current in a given sized probe.

- Various approaches and designs for monochromation and aberration-correction should continue to be explored.
- Increase in available current, and thus signal, is as significant a benefit of aberration correction as is the improvement in spatial resolution.
- Especially as the focal lengths of the objective lens increase, more attention must be paid to the design of other electron optical lenses (e.g. projector lens).

Discussions were wide-ranging and included practical aspects of data acquisition that help to set design criteria for electron optical developments.

- Strategies to mitigate beam damage of the specimen and make every electron count are essential: low-dose-type techniques where specimen is irradiated only when data is being collected (e.g., beam blanking); monochromation so that electrons impinging on specimen yield maximum image contrast (both STEM and TEM modes) and spectroscopic signal from volume of interest; cryogenic cooling of specimen; and the mitigation of amorphous films in specimen preparation.
- An electron dose of ~100 pC should be sufficient for STEM-EELS single atom detection.

STATUS OF THE TEAM PROJECT

For the past several years, DOE's five electron beam microcharacterization efforts, located at ANL, BNL, LBNL, ORNL and FS-MRL, have been preparing to lead a project to develop the next generation Transmission Electron Aberration-corrected Microscope (TEAM). The TEAM project will exploit recent advances in electron optics to develop a next-generation electron microscope with tunable aberration-corrected optics. Collectively, these centers have the scientific expertise and the supporting infrastructure to carry out such a project and to ensure that the resulting instrumentation benefits the entire scientific community. The goal of the TEAM project is to redesign the electron microscope around aberration corrected optics, to develop a common platform for a powerful new nanocharacterization instrument and to make this instrument widely available to the materials and nanoscience community. The resulting improvement in the spatial resolution, contrast, sensitivity, and flexibility of design of electron optical instruments will provide the unprecedented opportunity to observe directly the atomic-scale order, electronic structure, and dynamics of *individual* nanoscale structures.

TEAM is guided by a scientific advisory committee comprised of scientific leaders in the field of electron microscopy and materials science:

CB Carter - University of Minnesota
A Eades - Lehigh University
J Silcox - Cornell University
J Spence - Arizona State University
R Tromp - IBM Yorktown

The TEAM instrument will be installed at LBNL, where it will be maintained as a user instrument for research and collaboration. The long-range plan envisions utilizing the same platform to develop a variety of instruments that will be specialized for different purposes such as wide-gap in-situ experimentation, ultimate spectroscopy, field-free high resolution magnetic imaging, ultrafast high resolution imaging, diffraction and spectroscopy, and other extremes of temporal, spectral, spatial or environmental conditions. Located at different labs, these instruments will be accessible to users and collaborators from many locations as a "distributed center". Involvement of the scientific community in the TEAM project is encouraged through a series of open workshops, suggestions to the Advisory Committee as well as through collaborations and individual contacts with the partner labs:

LBNL: *U. Dahmen (Project Director), N. Browning, C. Kisielowski, E. Stach,* National Center for Electron Microscopy (NCEM)
ANL: *D. Miller, B. Kabius, N. Zaluzec,* Electron Microscopy Center (EMC)
BNL: *Y. Zhu, J. Wall,* Center for Advanced Electron Microscopy (CAEM)
FS-MRL: *I. Petrov, I. Robertson, R. Twesten, J.M. Zuo,* Center for Microanalysis of Materials (CMM)
ORNL: *I. Anderson, J. Bentley, S. Pennycook,* Shared Research Equipment Program (SHARE)

A first TEAM workshop was held in July 2000 at Argonne National Laboratory followed by a second TEAM workshop in July 2002 at Lawrence Berkeley National Laboratory.

The third TEAM workshop was held at the annual meeting of the Microscopy Society of America in San Antonio/Texas, in August 2003, and is summarized in the present report. The project was presented to the Office of Basic Energy Sciences in October 2002, and in February 2003 to a subcommittee of the DOE Basic Energy Sciences Advisory Committee charged with considering facilities needs over the next twenty years. The subcommittee strongly endorsed the project and urged the development team to carefully explore collaborations with the private sector to help make a broader impact of the investment on future instrumentation. The full report is available at http://www.sc.doe.gov/bes/BESAC/20year_facilities_report.pdf.

The research and development effort leading to a TEAM instrument is currently under external peer review and aims at evaluating design options and developing instrument components in a series of specific tasks. Each task in this collaborative effort will be led by one of the partner labs. For example, the (Cs + Cc) corrector development will be headed by ANL, with contributions on Cs correction from ORNL and LBNL. Development of fast electron detectors will be led by BNL, specimen module and stage development by FS-MRL and LBNL. Evaluation of monochromators on different instruments at LBNL, ORNL, BNL and LLNL will be coordinated by LBNL. Each of these tasks will utilize expertise within the private sector wherever possible.

Installation of the TEAM instrument at NCEM/LBNL is planned for completion in FY08. More details on the project can be found at http://ncem.lbl.gov.

APPENDICES

The appendices detail public communications or official documentation of participation in the Third Transmission Electron Aberration-corrected Microscope (TEAM) Workshop.

1. Announcement

2. Final Program

3. List of Participants

4. Submitted Abstracts

APPENDIX 1:
ANNOUNCEMENT

3rd TEAM Workshop
August 8, 2003
San Antonio Texas

For the past several years, five DOE-supported electron beam microscopy efforts, located at Argonne National Laboratory, Brookhaven National Laboratory, Lawrence Berkeley National Laboratory, Oak Ridge National Laboratory, and Frederick Seitz Materials Research Laboratory have pursued the development of a next generation Transmission Electron Aberration-corrected Microscope (TEAM). You are invited to participate in a one-day workshop on the TEAM project. This workshop will be held on August 8, 2003 in San Antonio Texas, directly after the 2003 Microscopy Society of America (MSA) meeting. It is being organized in conjunction with the recently established MSA Focused Interest Group on Materials Research in an Aberration-Free Environment.

This year's one-day TEAM Workshop will provide a forum for input from the scientific community on nanotechnology applications and in-depth discussions regarding technical specifications for this planned instrumentation. Morning sessions will cover contributions that target projects involving nanotechnology applications for aberration-free electron beam microcharacterization. Contributions are invited from the scientific community in the disciplines of *Physics, Chemistry, Biology, Life Sciences, Materials Science, Earth and Environmental Sciences*. Afternoon sessions will address the technical specifications and characteristics of this new instrumentation. Discussion topics will include *aberration correctors and monochromators, sample stages, tomography approaches, and detectors.*

Information on program details, abstract requirements and registration are posted on this website: http://ncem.lbl.gov/team3.htm

Looking forward to meeting you in San Antonio,
The organizers - Christian Kisielowski, Ian Anderson, Ray Twesten, Yimei Zhu

Abstracts:
250 word abstracts are invited and should be submitted electronically to CFKisielowski@lbl.gov by *Tuesday, July 15*. When preparing abstracts, please indicate a topic/title.
Registration:
If you plan to attend, please email Jane Cavlina JLCavlina@lbl.gov
Please provide your name, institution, mailing address, phone and fax numbers.
For travel and lodging information see the MSA web page at:
HTTP://WWW.MICROSCOPY.COM/MSAMEETINGS/MMMEETING.HTML

APPENDIX 2:

FINAL PROGRAM, THIRD TEAM WORKSHOP
Friday, August 8, 2003
San Antonio Convention Center

PLENARY SESSION: SCIENTIFIC CHALLENGES AND OBJECTIVES FOR TEAM

The Mission Room (103A) – J. C. H. Spence (chair)

8:30 – 9:00 am "Introduction and Background of TEAM"
U. Dahmen, NCEM, Lawrence Berkeley National Laboratory

9:00 – 9:30 am "Looking at Nanomaterials- What We Would Love to See"
Alexandra Navrotsky, University of California - Davis

9:30 – 10:00 am "Superconducting Materials: How New Electron Microcoscopy Capabilities Could Help"
D. C. Larbalestier, X. Song, P. J. Lee, and A. Gurevich
Applied Superconductivity Center, University of Wisconsin-Madison

10:00 – 10:30 am "Bimetallic Dendrimer-Encapsulated Nanoparticle Catalysts"
R. W. J. Scott, O. M. Wilson, and R. M. Crooks
Department of Chemistry, Texas A&M University

10:30 – 11:00 am ** Coffee Break **

11:00 – 11:20 am "Applications of a Monochromated TEM in Materials Science"
G.A. Botton*, S. Lazar**, M.Y. Wu** F.D. Tichelaar**, and H. Zandbergen**
*Brockhouse Institute of Materials Research, McMaster University, Hamilton, Canada.
**National Centre of High-Resolution Electron Microscopy, Delft University of Technology, Delft, Holland

11:20 – 11:40 am "Mining Cellular Functions"
J. Plitzko and W. Baumeister, Max-Planck-Institut für Biochemie, Martinsried, Germany

11:40 – 12:00 am "Application of Electron Microscopy to the Understanding and Characterization of Nano-scale Properties within Solid State Lighting Devices"
G. Liu, N. Fromer, J. Kerr, and S. Johnson
Environmental Energy Technology, Lawrence Berkeley National Laboratory

12:00 – 12:20 pm "Quantum Dot - Organic Composites: Structure-Property Relationships at the Nanoscale"
V. Leppert, University of California - Merced

12:20 – 12:40 pm "In situ Nano-oxidation: Corrosion, Passivation and Processing"

J. C. Yang, Materials Science and Engineering Dept., University of Pittsburgh

12:40 – 1:00 pm "Electron Microscopy Characterization of Human Tooth Enamel Nanocrystals"
J. Reyes-Gasga, Instituto de Física, UNAM, México

1:00 – 2:00 pm **Lunch Break**

BREAKOUT SESSIONS (101A&B, 102A&B)

Sample Holders & Stages: Enabling New Scientific Experiments
Room 101A – Ray Twesten, University of Illinois - Urbana-Champaign

Featured Speaker:
2:00- 2:30 pm, "Sample Holders and Sample Preparation"
H. Zandbergen, Delft University of Technology, The Netherlands

Selected Contributions:
2:30 – 3:30 pm,
"Four Probe Stage and Holder for Transmission Electron Microscopes"
Christof Baur, Zyvex Corp., Richardson TX
"Sample Stages for In-situ Microscopy"
M. Wall, Lawrence Livermore National Laboratory, Livermore, CA
"Sample Stages for Electron Microscopy: An Engineering Perspective"
N. Salmon, Lawrence Berkeley National Laboratory, Berkeley, CA
"TEAM – Preliminary Stage 'Specifications' "
E. Stach, Lawrence Berkeley National Laboratory, Berkeley, CA

Roundtable Discussion: "Defining and Achieving TEAM Goals"
3:30 – 5:00 pm, E. Stach, Moderator

Detectors: Emerging Detector Technologies
Room 101B – Yimei Zhu, Brookhaven National Laboratory

Featured Speakers:
2:00 – 2:30 pm "Detector Development for Position-Sensitive Diffraction in STEM"
J. S. Wall, Brookhaven National Laboratory
2:30 – 2:50 pm "Parallel Detectors for EM"
P. Mooney, Gatan R&D
2:50 – 3:10 pm "High count rate Silicon drift x-ray detector"
N. J. Zaluzec, Argonne National Laboratory
3:10 – 3:30 pm "Microcalorimeter X-ray Detectors: Issues and Opportunities for TEAM"
E. A. Kenik, Oak Ridge National Laboratory

3:30 – 3:50 pm "Recent developments of a post-column high-energy resolution EEL spectrometer / imaging filter"
M. M. G. Barfels, Gatan R&D

Roundtable Discussion
3:50 – 5:00 pm J. S. Wall, discussion leader

From 2D to 3D: Tomographic Methods
Room 102A – C. Kisielowski, Lawrence Berkeley National Laboratory

Featured Speakers:
2:00 – 2:30 pm "Prospects for Tomography through Depth Sectioning with the STEM"
S. J. Pennycook, Oak Ridge National Laboratory
2:30 – 3:00 pm "New High-Resolution Tomographic Techniques in Materials Science and Biology"
J. C. H. Spence, Arizona State University and Lawrence Berkeley National Laboratory
3:00– 3:30 pm: "Electron Tomography of Nanoparticles and Nanocrystals"
P. A. Midgley, University of Cambridge, United Kingdom
3:30 – 3:50 pm "Mathematical Aspects of Discrete Tomography"
J. Batenburg and R. Tijdeman, Leiden University, The Netherlands

Roundtable Discussion
3:50 – 5:00 pm Selected Contributors: Fu-Rong Chen, Tsing-Hua University, Taiwan; W. Qin, University of Missouri - St. Louis and Motorola; M. P. Oxley, University of Melbourne, Australia; J. S. Wall, Brookhaven National Laboratory

Electron Optics: Aberration Correctors, Monochromators, and Theoretical Basis
Room 102B – Ian Anderson, Oak Ridge National Laboratory; Bernd Kabius, Argonne National Laboratory

Featured Speakers:
2:00– 2:30 pm "Outline of an Ultracorrector Compensating for all Primary Chromatic and Geometrical Aberrations of Charged-Particle Lenses"
H. H. Rose, University of Technology, Darmstadt, Germany (retired)
2:30 – 3:00 pm "Alignment and Technical Feasibility of C_c Correctors"
M. Haider, CEOS GmbH, Germany
3:00 – 3:25 pm "High Spatial and Energy Resolution EELS"
N. D. Browning, University of California - Davis and Lawrence Berkeley National Lab
3:25 – 3:40 pm "Beyond University Facilities: Opportunities in Spectroscopy"
D. A. Muller, Cornell University

Roundtable Discussion
3:40 – 5:00 pm Selected Contributors: B. Kabius, Argonne National Laboratory, M. J. van der Zande, Philips Research Laboratories, The Netherlands; M. A. O'Keefe,

Lawrence Berkeley National Laboratory; G. Benner, LEO EM Group, Germany; Z. Yu, Cornell University; L. F. Allard, Oak Ridge National Laboratory

APPENDIX 3:
LIST OF PARTICIPANTS

Name	Affiliation	Organization
Al Jassim, M.	Other Fed./Nat. Lab	NREL
Allard, L	DOE Lab	ORNL
Allan, L.	Univ/For	Univ Melbourne
Anderson, I.	DOE lab	ORNL
Arslan, I.	Univ	UC Davis
Barfels, M	Ind	Gatan
Basile, D.	Ind	Hewlett Packard
Baur, C.	Ind	Zyvex
Baumeister, W.	Other Fed. Lab/ For	Max Planck-Martinsried
Batenburg, J.	Univ/For	Leiden Univ
Beleggia, M.	DOE Lab	BNL
Benner, G.	Ind	LEO
Bentley, J.	DOE Lab	ORNL
Bleloch, A.	Other Fed. Lab/ For	Super STEM Lab
Bliss, R.	DOE Lab	LLNL
Blom, D.	DOE Lab	ORNL
Botton, G.	Univ/For	Mc Masters U
Browning, N.	Univ/DOE Lab	UCDavis, LBNL
Bruley, J.	Ind	IBM
Buchanan, R.	Ind	Gatan
Carim, A.	Gov	DOE
Carter, B.	Univ	U. Minnesota
Cavlina, J.	DOE Lab	LBNL
Chen, F. R.	Univ/For	Univ Taiwan
Connelly, T.	Ind	Gatan
Dahmen, U.	DOE Lab	LBNL
Dalaver, A.	Univ	Univ Virginia
Dickerson, P.	DOE Lab	LANL
Dickerson, R.	DOE Lab	LANL
Duscher, G.	Univ/DOE Lab	N. Carolina State U., ORNL
Eades, A.	Univ	Lehigh Univ
Egerton, R.	Univ/For	Univ. Edmonton, Canada
Erni, R.	Univ	UC Davis
Findlay, S.	Univ/For	Univ Melbourne
Fischione, P.	Ind	Fischione Instruments
Fortmann, C.	Univ	Stony Brook, SUNY
Gottschall, R.	Gov	DOE
Haider, M.	Ind	CEOS
Herring, R.	Univ/For	Univ Victoria, Canada
Hunt, J.	Ind	Gatan
Hwang, R.	DOE Lab	BNL
Iddir, H.	Univ	UC Davis
Idrobo, J.	Univ	UC Davis

Inada, H.	Ind	Hitachi
Ishizuka, K.	Ind	HREM Research
Ito, Y.	Univ	N. llinois State U.
Jing, Y.	Univ	UC Davis
Johnson, S.	DOE Lab	LBNL
Kabius, B.	DOE Lab	ANL
Kakibayashi, H.	Ind	Hitachi
Kawasaki, M.	Ind	JEOL
Kenik, E.	DOE Lab	ORNL
Kisielowski, C.	DOE Lab	LBNL
Kilaas, R.	DOE Lab	LBNL
Klie, R.	DOE Lab	BNL
Krivanek, O.	Ind	NION
Kuebel, C.	Ind	FEI
Kundmann, M.	Ind	Gatan
Larbalestier, D.	Univ	U. Wisconsin
Lau, J.	DOE Lab	BNL
Lazarov, V.	Univ	Univ Wisconsin
Leppert, V.	Univ	UCDavis
Lichte, H.	Univ/For	Dresden U.
Liu, G.	DOE Lab	LBNL
Lupini, A.	DOE Lab	ORNL
Marshall, M.	Univ	U. Illinois
Matesa, J.	Ind	Fischione Instruments
McKernan, S.	Univ	Univ Minnesota
Medlin, D.	DOE Lab	LLNL
Midgely, P.	Univ/For	Cambridge
Miller, D.	DOE Lab	ANL
Mooney, P.	Ind	Gatan
Muller, D.	Univ	Cornell
Navrotsky, A.	Univ	UCDavis
Nuzzo, R.	Univ	U. Illinois
O'Keefe, M.	DOE Lab	LBNL
Oleshko, V.	Univ	Univ Virginia
Own, C.	Univ	Northwestern Univ
Oxley, M	Univ/For	Univ Melbourne
Pan, M.	Ind	Gatan
Pennycook, S.	DOE Lab	ORNL
Petrov, I.	DOE Lab	U. Illinois
Plitzko, J.	Other Fed. Lab/ For	Max Planck-Martinsried
Qin, W.	Univ	Univ Missouri
Rabenberg, L.	Univ	Univ Texas
Rau, W.-D.	Ind For	LEO
Ray, D.	DOE Lab	PNNL
Reyes-Gasga	Univ/For	Inst. Fisica, UNAM Mexico
Rice, P.	Ind	IBM

Ringnalda,J.	Ind	FEI
Robin, D.	DOE Lab	LBNL
Rose, H.	Univ/For	Univ Darmstadt
Ross, F.	Ind	IBM
Salmon, N.	DOE Lab	LBNL
Schlossmacher, P.	Ind	LEO
Schindler, B.	Ind	LEO
Schindler, U.	Univ/For	Univ Münster
Schmid, A.	DOE Lab	LBNL
Scholfield, M.	DOE Lab	BNL
Scholes, G.	Ind	FEI
Scott, J.	Other Fed./Nat. Lab	NIST
Scott, R.	Univ	Texas A&M
Seo, D.	Univ	Texas A&M
Silcox, J.	Univ	Cornell
Son, S-K.	DOE Lab	LBNL
Song, X.	Univ	Univ Wisconsin
Spence, J.	DOE Lab/Univ	LBNL/Arizona State Univ
Stach, E.	DOE Lab	LBNL
Tanaka, N.	Univ/For	Nagoya Univ
Tao, X.	Univ	Lehigh Univ
Thomas, M.	Univ	Cornell
Treacy, B.	Ind	Spansion
Tromp, R.	Ind	IBM
Twesten, R.	DOE Lab	Univ Illinois, C-U
Van der Zand, K	Ind	Philips
Van Tendeloo, G.	Univ/For	Univ Antwerp
Voelkl, E.	Ind	NLine
Volkov, V.	DOE Lab	BNL
Von Harrach, H.	Ind	FEI
Voyles, P.	Univ	Univ Wisconsin
Wall, J.	DOE Lab	BNL
Wall, M.	DOE Lab	LLNL
Wang, C.	DOE Lab	PNNL
Weber, W.	DOE Lab	PNNL
Windl, W.	Univ	Ohio State Univ
Wu, L.	DOE Lab	BNL
Xu, X.	DOE Lab	LBNL
Yamasaki, J.	Univ/For	Nagoya Univ
Yang, J.	Univ	Univ Pittsburgh
Yu, Z.	Univ	Cornell
Zaluzek, N.	DOE Lab	ANL
Zandbergen, H.	Univ	Princeton Univ
Zheng, J.	Univ	Northwestern Univ
Zhou, J.	Univ	Univ Texas
Zhu, Y.	DOE Lab	BNL

APPENDIX 4:
SUBMITTED ABSTRACTS

Plenary Session

INTRODUCTION AND BACKGROUND TO THE TEAM PROJECT

U. Dahmen,
National Center for Electron Microscopy, LBNL, Berkeley

As an introduction to the workshop, this talk will present an overview of the Transmission Electron Aberration-corrected Microscope (TEAM) project. The TEAM project is driven by scientific needs and made possible by recent advances in electron optics. As a joint operation of five DOE-supported electron microscopy efforts (LBNL, ANL, BNL, FS-MRL, and ORNL) it involves a diverse group of collaborators and potential users. A brief review will outline the principal ideas that spawned the project, introduce its goals and its participants, summarize the involvement of the scientific community, and forecast its role in the development of microscopy instrumentation and research.

The major focus of this presentation will be the scientific opportunities that arise from the unprecedented performance of aberration corrected microscopes, from increased brightness to higher spatial, temporal and spectral resolution. The close relationship between the burgeoning field of nanoscience and the goals of the TEAM project will be highlighted with examples from a broad spectrum of research fields. Scientific examples will include needs and opportunities for atomic resolution tomography, single-atom spectroscopy, and in-situ growth, transformation or manipulation of materials during real-time observation at atomic resolution. Finally, this talk will describe the structure and the goals of the present workshop.

LOOKING AT NANOMATERIALS - WHAT WE WOULD LOVE TO SEE

A. Navrotsky,
University of California - Davis

This overview is presented from a vantage point far from TEM, namely that of a solid state thermodynamicist. Using my own work for examples, I discuss several recurring issues that TEAM may help resolve. (1) What is meant by "amorphous" at the nanoscale and what is the impact of the (gradual) increase in ordering with increasing particle size

on properties? (2) For nanocomposites and multiphase nano-aggregates, can one get information about morphology, three dimensional tomographic images, and knowledge of compositional zoning, especially at interfaces? (3) Can one get detailed structural information on "fragile" materials, including heavily hydrated phases and other structures prone to electron beam damage? This information will help relate structure, energetics, and kinetics of transformation, particularly relevant to geochemical and environmental processes.

SUPERCONDUCTING MATERIALS: HOW NEW ELECTRON MICROCOSCOPY CAPABILITIES COULD HELP

D. C. Larbalestier, Xueyan Song, Peter J Lee, and Alex Gurevich
Applied Superconductivity Center, University of Wisconsin-Madison, Madison WI 53706, USA

When exposed to a magnetic field, most useful superconductors are penetrated by high densities of real supercurrent vortices whose cores contain quantized magnetic field in units of 2×10^{-15} Wb. High bulk, not just surface supercurrents exist when these vortices are pinned so that a vortex density gradient can occur. To maximize this superconducting critical current density J_c a high density of strong pinning interactions are needed. The best way to do this is by subdividing the superconductor into 10-25% of nanosize, normal phase, well dispersed in a continuous superconducting matrix. This permits strong elementary pinning interactions with the normal vortex cores. The vortex core diameters – 2 superconducting coherence lengths, _ - of high field superconductors are very small, typically 1-10 nm. In the widely used low temperature superconductors, Nb-Ti and Nb_3Sn, optimizing vortex pinning is all the needs to be done, because any polycrystalline superconductor is always continuously connected, even across grain boundaries. But to understanding flux pinning at the next level of detail requires very specific information about the local electronic state of the pin on scales of 0.1_, that is the atomic scale. For the high temperature, cuprate superconductors grain boundaries are multiply connected barriers to current flow except under specially textured, low-angle conditions. At HTS grain boundaries, carrier density is strongly depressed for reasons that are still poorly understood. At Nb_3Sn grain boundaries, superconductivity is depressed but just enough to make the GBs good pins but not good obstacles. Local knowledge of the electronic state on a scale of ~0.1_ would be very valuable to understand this transition would be very valuable. In as much as MgB_2 appears to be a well connected low-T_c rather than a poorly connected high-T_c material, study of its grain boundaries is also very interesting. We will summarize these important issues and seek to define questions that new generations of analytical microscopes could answer.

BIMETALLIC DENDRIMER-ENCAPSULATED NANOPARTICLE CATALYSTS

R. W. J. Scott, O. M. Wilson, R. M. Crooks
Department of Chemistry, Texas A&M University, College Station, Texas

The synthesis, characterization, and catalytic activity of bimetallic dendrimer-encapsulated nanoparticles (DENs) will be discussed.[1,2] These materials are prepared by co-complexation of different ratios of metal salts with interior tertiary amines of poly(amidoamine) (PAMAM) dendrimers, followed by chemical reduction, or alternatively by deposition of a second metal onto metallic DEN seeds. These syntheses yield stable, near-monodisperse, water-soluble bimetallic DENs. The size of the nanoparticles can be varied between 1 to 3 nm through control of the metal:dendrimer ratio. In addition, dendrimer templates offer the possibility of multi-step sequential design of DEN catalysts that allows for the synthesis of a large range of bimetallic and multimetallic architectures with tunable catalytic properties. While evidence that individual nanoparticles are bimetallic has been obtained using single-particle x-ray energy dispersive spectroscopy (EDS), we wish to further examine the atomic structure of these bimetallic DENs: *i.e.* whether individual particles have random alloy vs. core-shell structures, in order to correlate this information to their catalytic properties. Other interesting avenues of research include probing the catalyst-dendrimer interface and the nanoparticle geometry and location within the dendrimer interior.

(1) Crooks, R. M.; Zhao, M.; Sun, L.; Chechik, V.; Yeung, L. K. Acc. Chem. Res. 2001, 34, 181-190.
(2) (2) Scott, R. W. J.; Datye, A. K.; Crooks, R. M. J. Am. Chem. Soc. 2003, 125, 3708-3709.

APPLICATIONS OF A MONOCHROMATED TEM IN MATERIALS SCIENCE

G.A. Botton*, S. Lazar, M.Y. Wu** F.D. Tichelaar**, H. Zandbergen****
***Brockhouse Institute of Materials Research, McMaster University, Hamilton, Canada.**
****National Centre of High-Resolution Electron Microscopy, Delft University of Technology, Delft, Holland.**

The recently developed monochromators in the transmission electron microscope open new prospects for applications of electron energy loss spectroscopy in physics and materials science. The enhanced energy resolution (for example see the TiL23 edge, Figure 1) makes it possible to clearly resolve fine structure changes due to small structural environment modifications and bonding in the absorption edges. More important, however, is the impact monochromators have in the analysis of the low energy losses (Figure 2, [1]). The improvements in energy resolution make it possible to measure

energy gaps using a small probe. This dramatically enhances the prospects of quantitative analysis of local electronic properties.
After discussing the general instrument used for the experiments we will discuss examples of application of high-resolution EELS in a broad range of materials. These include the analysis of metal-insulator transitions in oxides with strongly correlated electrons, perovskite structures with various substitutional changes of the cations and the analysis of the low losses in GaN semiconductors and low-dimensional structures. Correlation of the low energy measurements with photoluminescence spectroscopy and defect states in the gap will be presented.

Reference:
[1] S. Lazar, G.A. Botton, F.D. Tichelaar, M.Y.Wu and H. Zandbergen, Ultramicroscopy, In press, SALSA 2002 proceedings.

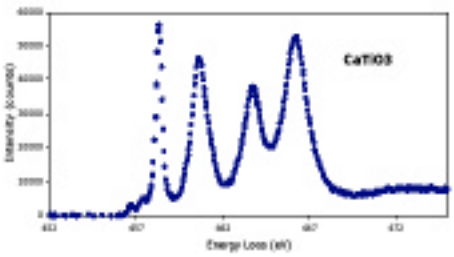

Figure 1. Ti L_{23} edge in $CaTiO_3$ obtained with EELS and monochromator showing the crystal field splitting. This spectrum shows that synchrotron-quality data can be obtained from small areas (here, few nm in size).

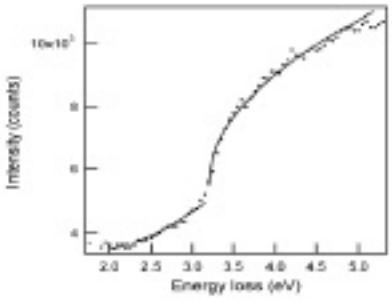

Figure 2. Low-loss energy spectra in GaN showing a perfect fit to the parabolic band above 3.2eV due direct transitions across the energy gap and an additional indirect-type band due to defects states in the gap.

MINING CELLULAR FUNCTIONS

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D-82152 Martinsried, Germany

The long prevailing view of a cell as a membrane-bound reaction compartment filled with freely diffusing and colliding macromolecules can no longer be maintained. There is growing awareness that fundamental cellular functions are carried out by ensembles of macromolecules, protein complexes or 'molecular machines'. And, as in a factory, the operation of these machines must be coordinated to give rise to a stochastically variable supramolecular architecture. On this level of structure, the cell is, by and large, an uncharted territory. None of the existing imaging techniques enables the study of pleiomorphic structures, such as organelles or whole cells with a resolution of a few nanometers, as is required for identifying macromolecules *in situ* and for describing their

interaction networks. Therefore, there is a strong incentive to develop methods, ideally non-invasive, to study the supramolecular architecture in a cellular context.
However, cryo-electron tomograpy (ET) is an imaging technique, which allows the three–dimensional (3D) visualisation of cells within 4 to 6 nm resolution. ET is by no means a new imaging technique, but with the advent of computer-controlled electron microscopes and the automation of elaborate image acquisition procedures, it became possible to obtain molecular-resolution tomograms of structures as large and complex as whole prokaryotic cells or thin eukaryotic cells embedded in amorphous ice. Tomograms of cells at molecular resolution are essentially 3D images of the cell's entire proteome, but with the current resolution, one can address only larger complexes in a cellular context. To widen the scope of cellular electron tomography it will be necessary to improve the resolution. Theoretical considerations and ongoing instrumental improvements, such as liquid helium cooling, improved detectors and dual-axis tilting, make a resolution near 2 nm a realistic goal. Taken together, these developments should help to push the limits of cryo-ET and brighten the prospects to explore the uncharted territory of the molecular architecture of the cytoplasm.

APPLICATION OF ELECTRON MICROSCOPY TO THE UNDERSTANDING AND CHARACTERIZATION OF NANO-SCALE PROPERTIES WITHIN SOLID STATE LIGHTING DEVICES

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Solid State Lighting is an emerging technology that is projected to significantly reduce the electric lighting loads within US buildings. Progress in this technology is dependent upon improvements in the performance of light emitting diodes (LEDs) and organic light emitting diodes (OLEDs). In both LEDs and OLEDs, improving phosphor efficiency is essential to achieve high-efficiency lighting systems, and recent advances in the fabrication of quantum dot and nanocrystal based phosphors show promise in this direction. However, both the spectral characteristics and the conversion efficiency of these nanophosphors are highly dependent on size, shape, and external environment. Combining optical spectroscopy with high spatial resolution electron microscopy allows the direct comparison of the efficiency and color output of these nanophosphors with their surface and internal structure, at the single particle level. The potential for a full 3D map of the nanoparticles would give unparalleled understanding of the size and shape dependence of emission from these particles.
Even more can be gained in the study of OLED devices. Studying the polymer-electrode interfaces with high-resolution electron microscopy can lead to understandings that will improve the charge-transfer between layers, and to significant increases in device efficiency and lifetime. Improvements in device performance have been shown by inclusion of semiconductor nanoparticles in the polymer matrix, but understanding the nanoparticle-polymer interface is critical for continued enhancements. Mapping the

charge distribution in 3D of an operational device in real time would provide key information about device performance, helping to identify non-radiative traps, defects and other phenomena that can dramatically lower efficiency or lifetime of the devices.

QUANTUM DOT - ORGANIC COMPOSITES: STRUCTURE-PROPERTY RELATIONSHIPS AT THE NANOSCALE

V. J. Leppert
University of California – Merced

Block copolymer systems offer the opportunity for templating nanoparticles of various compositions, including semiconductors, metals and oxides. Specifically, particle size, morphology, inter-particle distance, and packing arrangement may be controlled during the in-situ growth of the nanoparticles in the polymer matrix. This is achieved through the proper selection of the relative weight percents of the sequestering and matrix polymer blocks (determining packing arrangement and morphology), and by the length of the entire polymer chain (determining size and spacing). Transmission electron microscopy, and more specifically electron energy-loss spectroscopy, offers unique opportunities for studying templated nanoparticles, the polymer matrix, and the interfacial region between the two; as well as the details of phase segregation of the inorganic phase in the organic medium. Specific examples of polymer-templated structures that will be discussed include GaN quantum dots formed in a PS-P4VP matrix from the in-situ formulation and decomposition of cyclotrigallazane; and a block copolymer lithium ion battery material incorporating gold nanoparticles formed in-situ, also containing carbon nanotubes.

IN SITU NANO-OXIDATION: CORROSION, PASSIVATION AND PROCESSING

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Aberration corrected (scanning) transmission electron microscopy ((S)TEM) provides an exciting opportunity for increasing the pole-piece gap and improving spatial, spectral and temporal resolution - all perfect for developing in situ experiments. As an example, I will focus on the nano-oxidation reactions.
Understanding oxidation process is of fundamental and practical importance because corrosion, passivation, thin film growth (e.g. ferroelectrics), and some catalytic reactions, involve oxidation. Yet, a surprising paucity of knowledge concerning the transient oxidation stages, from the nucleation to coalescence, still exists. Furthermore, as engineered materials approach the nanometer regime, controlling their environmental

stability at this scale will be crucial to their performance and durability. In situ ultra high vacuum transmission electron microscopy (UHV-TEM) is ideal for exploring the complex kinetics and energetics of nano-oxidation since this technique provides real-time information at the nanoscale under controlled surface conditions. In this presentation, I will present both in situ and ex situ nano-oxidation experiments to exemplify the present limitations and the potential of aberration-corrected (S)TEM for in situ.

ELECTRON MICROSCOPY CHARACTERIZATION OF HUMAN TOOTH ENAMEL NANOCRYSTALS

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Tooth enamel is the most mineralized tissue of human body. Its composition is close to 96% hydroxyapatite. It is well known that prisms that are easily observed by SEM form enamel. The main transversal size of these prisms is around 5 _m in diameter. It is realized that many 200-nm crystals form the prisms, when they are observed with TEM. These crystals are elongated along the longitudinal section. HREM images of these enamel crystals shows that they exhibit a line of 1 to 1.5 nm thick along their centers in conjunction with the {100} lattice fringes of hydroxyapatite unit cell. This line has been named “dark line”, although its contrast is focus dependent: it appears dark in defocus, disappears when the image goes through focus, and is white in over-focus. The occurrence of this line is of particular interest because it seems to undergo preferential dissolution during early stages of caries. Several ideas on its structure have been proposed. However, the absence of any of the well known electron microscope contrasts for them debates easily some of these suggestions, and up to now many questions on the nature and role the line plays in the enamel grain structure remain unanswered. The analysis with an Aberration Corrected TEM of this subject is quite important in the next future to support any possible model of the enamel crystal structure.

Breakout Session: Sample Holders & Stages

SAMPLE HOLDERS AND SAMPLE PREPARATION

H. Zandbergen,
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In recent years we have developed a number of specimen holders for a pole gap of 2.4 mm (FEI Ultratwin lens). The development was done because commercial holders are not available or not having the right specifications.

- Double tilt cooling holder operating at about 100 K allowing a resolution of 1.4 Å
- Double tilt holder with high beta tilt ±50°
- Double tilt (beta tilt ±30°) vacuum transfer holder
- Single tilt holder allowing 360° rotation for tomography
- Double tilt (beta tilt ±20°)) rotation (rotation 70°) holder
- Holder to place specimen on lower pole piece for resolution tests

A multispecimen (4 specimens) double tilt holder (beta tilt ±50°) was designed for a Tecnai-Supertwin, to be integrated a remote experimentation environment, such that a remote operator can investigate 4 specimens without on-site assistance.

The double tilt vacuum transfer holder will be coupled to a low energy (about 100 eV) ion milling system, thus allowing the removal of surface layers (for instance oxides) and subsequent transfer into the electron microscope keeping the specimen in vacuum. The development of several other holders like a 1atm holder is in progress.

The resolution and further performance of these holders and the ion mill set-up will be presented.

Breakout Session: Detectors

DETECTOR DEVELOPMENT FOR POSITION SENSITIVE DIFFRACTION IN STEM

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Image simulation using atomic coordinates (up to 10,000,000 atoms), multi-slice calculations and wave optics for probe formation and imaging allows us to compare STEM and TEM imaging quantitatively. One interesting case is an "amorphous" thin specimen where a highly confined STEM probe illuminates only a few atoms, giving convergent beam electron diffraction (cbed) pattern on the detector, rich in detail. It appears that a set of such patterns recorded as a function of beam position, defocus and tilt should allow one to extract atomic coordinates of the specimen atoms.
In order to test this, we have constructed a fast 32x32 element detector which can be placed in the detector plane to read out integrated electron counts every 100 microseconds. This should be fast enough to record STEM images of 512x512 points in 30 sec. An active matrix pixel detector has been fabricated from high resistivity silicon in the Instrumentation Division, BNL(1, 2). This is now in the testing stage and results will be presented. Comparison with commercial CCD cameras for electron microscopes will be made. Pros and cons of various electron detectors will be discussed.
Simulation of expected cbed patterns as a function of specimen and microscope parameters, especially dose, permits testing of information retrieval methods. This is underway and results will be presented. Finally, quantitative comparison of TEM and STEM for other specimen types will be described, particularly improvements possible with aberration correctors.

1. W. Chen, G. DeGeronimo, Z. Li, P. O'Connor, V. Radeka, P. Rehak, G. C. Smith, and B. Yu (2002) "Active Pixel Sensor on High-Resistivity Silicon and Their Readout," IEEE Transactions on Nuclear Sciences 49, 1006.
2. W. Chen, G. DeGeronimo, Z. Li, P. O'Connor, V.Radeka, P. Rehak, G. C. Smith, J.S. Wall and B. Yu (2003) "High resistivity silicon active pixel sensors for recording data from STEM" Nuc. Inst. & Meth. in Phys. Res. (in press).

PARALLEL DETECTORS FOR EM

P.E. Mooney, B. Bailey and D. Joyce
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Electronic parallel detectors for image and spectrum capture in electron microscopy have evolved in size, resolution, speed, bit depth and sensitivity in their approximately 15 year history. They have replaced traditional modes of acquisition in all but a few applications and have enabled entirely new applications hinging on the availability of image feedback for automation. Further development depends on our ability to understand the way in which key factors in the detection process limit total system performance. Reciprocal space noise power analysis has brought about a clarification of the key issues involved in determining practical resolution and sensitivity of detectors and has allowed a quantification of a detector's ability to deliver specimen information from the impinging EM image. It will be shown how physical limitations on detector performance and new techniques for mitigating those measures can be evaluated using these techniques. In addition, an attempt at a quantitative history of parallel detection capability will be given with a projection of what might be possible in the future. Beyond evaluation and improvement of the detector itself, these techniques will allow a quantitative reassessment of basic microscope operation. Magnification, kV and dose choices, which have evolved over time to make best use of film, may change in the pursuit of a new global optimum. Ways in which this might happen and areas for further investigation will be presented.

RECENT DEVELOPMENTS OF A POST-COLUMN HIGH-ENERGY RESOLUTION EEL SPECTROMETER / IMAGING FILTER

M.M.G. Barfels, C. Trevor, P. Burgner, B. Edwards, H.A. Brink, M. Kundmann, P. Mooney and J.A. Hunt
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Two years ago the first sub-50 meV high-resolution post-column electron-energy-loss-spectrometer was installed on a 200 kV monochromated TEM at the University of Delft, demonstrating a system energy resolution of 100 meV [1]. This high-resolution spectrometer is based on 4th order aberration correction optics, high-stability and low-noise electronics, and advanced automated alignment software. Two imaging filters (HR-GIF) based on similar optics have also been installed at Technical University of Graz and Lawrence Berkeley Laboratory.

While aberration correcting optics significantly improve the energy resolution of the new GIF, they also make a larger filter entrance aperture practical, yielding a 16 um field of view for EFTEM and a 120 mRad collection semi-angle for energy-filtered diffraction.

High-stability and low-noise electronics are critical to the design. The low intensity of a monochromated electron source necessitates longer exposures with little drift. Noise, primarily in the prism current supply limits the ultimate energy resolution. Redesigned electronics reduce drift and noise by a factor of 20 and 5 respectively.

Much of the progress made over the past two years has been to ensure that advanced features of this instrument are as easy to use and robust as the standard GIF instruments.

Recently, we have added the latest detector technology to this new-generation GIF. The new 2K x 2K CCD detector with 4-port readout combines a narrow point-spread for

excellent EELS detection with EFTEM image frame rate > 10 Hz and spectrum readout rate > 30 Hz. The high sensitivity of the detector permits live focusing of energy-filtered images at 750 eV loss and beyond. Late results obtained with the new detector will be presented.

[1] H.A. Brink, M. Barfels, B. Edwards and P. Burgner, Proceedings of Microscopy and Microanalysis, 908-909 (2001).

Breakout Session: Tomographic Methods

PROSPECTS FOR 3D ATOMIC RESOLUTION TOMOGRAPHY THROUGH DEPTH SECTIONING WITH THE STEM

S. J. Pennycook, A. R. Lupini, A. Borisevich, M. Varela and S. Travaglini
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Aberration correction not only brings substantial improvements in resolution, contrast and signal/noise ratio, but the increased aperture angle opens up techniques that have never before been possible with electrons. Confocal imaging has revolutionized optical microscopy by facilitating 3D tomography through depth sectioning. ADF STEM is ideal for tomography since the images show no contrast reversals with focus, a key requirement. The maximum aperture angles available in STEM are increasing with each new generation of corrector, while at the same time the depth of field is reduced. For imaging a zone axis crystal, theory has shown that optimal coupling into the 1s states occurs when the size of the probe matches the size of the 1s state, with an aperture ~ 25 mrad semiangle giving a probe FWHM ~ 0.04 nm. With larger probe-forming apertures the non-1s component becomes dominant. The high angle components of the probe are traveling at a large angle to the zone axis. Therefore, even in a zone axis crystal, they are scattered *only kinematically* and come to a focus at a unique depth in the specimen.

Initial results will be presented using the 300 kV HB603U aberration-corrected STEM, and simulations will be presented for future generation corrected instruments. Ultimately, atomic resolution should become viable *in depth* as well as laterally, making possible 3D tomography to reveal the precise location of impurity atoms at interfaces and grain boundaries. In principle, simultaneous EELS or EDX could also be used for spectroscopic 3D tomography of particular atoms or specific electronic structures.

DIFFRACTIVE IMAGING FOR TOMOGRAPHY OF ORGANICS AND INORGANICS.

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The opportunities which aberration correctors provide for atomic-resolution tomography will be reviewed. Tomographic imaging using soft X-rays (which is well established) will be compared with TEM, and a comparison made of the radiation damage in each case. An aberration-free three-dimensional image can be reconstructed from electron

diffraction patterns (and a few low-tilt HREM images), using the iterative "HiO" algorithm and a compact support condition along the beam direction to solve the phase problem. This Gerchberg-Saxton-Feinup algorithm was first used to reconstruct electron microscope images from diffraction patterns by Weierstall et al (Ultramic 90, p.171), and the first atomic-resolution images of a double-walled nanotube were recently obtained from electron diffraction patterns by this method (Zuo et al, Science, 300, p. 1419 (2003)). This appears to offer a useful improvement to existing cyromicroscopy methods for TEM of protein monolayers, especially those membrane proteins used for drug delivery, which are difficult to crystallize. The extension of this method to tomographic atomic resolution imaging of in organic nanostructures using both medium energy X-rays and electrons will be discussed.

ELECTRON TOMOGRAPHY OF NANOPARTICLES AND NANOCRYSTALS

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The push for nanotechnology and the increasing use of nanoscale materials brings with it the need for high spatial resolution imaging and analysis. The transmission electron microscope (TEM) is a remarkably powerful and versatile instrument and in many ways ideal for such characterisation. Conventional use of a TEM is to section the object of interest and examine 2D slices assuming either uniformity in the 3rd dimension or speculating on the 3D structure from the projection. However, as devices and structures become truly 3-dimensional, by growth or design, a single projection will not be adequate for a complete description. Stereo microscopy offers some insight into the 3D nature of an object but for true quantitative 3D analysis, one has to turn to tomography as a way to reconstruct the 3D object from a tilt series of 2D projections. Electron tomography has been used with great success in the biological sciences for about 30 years: the 3D structure of viruses and macromolecules have been determined with remarkable accuracy using tomography based on series of bright field images. However, in the physical sciences, for a general, probably crystalline, object, diffraction (and Fresnel) contrast prohibits the use of (coherent) BF images for electron tomographic reconstruction. Other, incoherent, signals must be used. In Cambridge, electron tomography has been developed using scanning transmission electron microscopy (STEM) high-angle annular dark-field (HAADF) imaging and energy-filtered TEM (EFTEM). Used correctly, both techniques give predominantly incoherent signals which can be exploited as a basis for electron tomography. In this talk, the effectiveness of this new method will be discussed highlighting the advantages (and disadvantages) of these signals for tomography. Using a number of animations, it will be shown how this new form of tomography is particularly advantageous for the study of heterogeneous catalysts, allowing the 3-D distribution of sub-nm particles to be viewed with relative ease, and how STEM tomography in particular can be used to study the faceting of nanocrystals and quantum dots. In the

physical sciences, the spatial resolution and field of view of this technique complements perfectly the ultra-high resolution technique of atom probe tomography and the much lower resolution X-ray micro-tomography.

DISCRETE TOMOGRAPHY

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The introduction of QUANTITEM in the 1990s gave rise to a wide range of mathematical questions. If we have the ability to measure the projections of atomic structures in crystals, are we also capable of reconstructing these structures from the measured data? Is the reconstruction unique or are there more atom configurations
that correspond to the same projections? In this talk I will first give an impression of the most important mathematical results that have been obtained so far. These results are mostly related to the question how hard DT problems are from a computational point of view, the uniqueness of the resulting reconstruction and the stability of this reconstruction when the measurements contain errors.
Recently I developed a new algorithm that is very effective on a large class of images, having certain structure properties. I will show some results obtained with this algorithm, demonstrating its capabilities. Although the original motivation for research in DT was its application in electron microscopy, research has branched into many directions and has diverged from its original context. In order to successfully apply DT in electron microscopy it is critical that the problem model is further refined. Hopefully this will lead to the adaption of existing algorithms and the development of better ones, to make accurate reconstructions of atomic structures possible.

ELECTRON TOMOGRAPHY & TEAM

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The ability to detect single atoms in a more routine manner by STEM and HRTEM is an outstanding performance improvement in electron microscopy that came along with advancements of the instrument technology over the last years. Together with recent improvements in quantifying scattering processes in thin samples it principally enables electron tomography on an atomic scale. Further, the perspective unfolds to record several hundred images at deep sub Ångstrom resolution without inflicting substantial radiation damage to many hard materials by the development of aberration corrected instruments operating at lower voltages (100 – 200 kV). Such progress would further stimulate research with electron tomography. Since aberration correction will be very

effective in instruments operating at lower voltage [1], it seems natural to promote electron tomography within the TEAM project. Approaches to electron microscopy will be discussed.

"Advantages of chromatic aberration correction for material science research" B. Kabius, D. J. Miller, this workshop.

ABERRATION CORRECTION AND POSSIBLE STRUCTURAL TOMOGRAPHY IN COMPLEX TEM

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We will present a new digit method to correct the lens aberration from a series of de-focus images under non-linear imaging condition. This method involves retrieving the image wave with Transport Intensity Equation (TIE)/ self-consistent wave propagation with Gerchberg-Saxton Algorithm and exit wave reconstruction from image wave under non-linear imaging condition. The structural information can then be quantitatively determined from the exit wave by 1) fitting the exit wave with the S-state model 2) fitting the exit wave with multislice model. The whole reconstruction procedures are being implemented into a user-friendly program.
Progress in hardware development of the micro-electrostatic phase plate to get the phase (complex signal) will be also briefly reported and discussed. The micro-lens is manufactured with micro-machining technique. The discussion will be extended to the possibility of constructing structural tomography by integrating the structural information from several different crystallographic orientations.

ON-LINE DETERMINATION OF NANOCRYSTAL LATTICE PARAMETERS

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The three-dimensional lattice parameters of a selected crystal can be inferred from lattice image information on three sets of non-parallel lattice planes. Today, with sufficiently wide tilt-capability, such data can come from phase-contrast (or less easily Z-contrast) images taken along two low-index zone axes of the crystal (cf. *Ultramicroscopy* **94**, p. 245-262). Higher spatial resolution in images will lessen the requirement for wide-angle tilting, but also increase the geometric complexity of the task due to the involvement of

lower symmetry orientations. In addition to some "fancy inverse crystallography" for the design of tilt protocols, our experience so far also suggests that issues of tilt-stage precision, on-line computer support, and off-axis fringe visibility as a function of specimen thickness will have to be considered before routine on-line determination of the lattice parameters of an arbitrary nanocrystal becomes possible in practice.

EXIT WAVE RECONSTRUCTION AT ATOMIC RESOLUTION

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An iterative method for exit wave function reconstruction based on wave function propagation in free space is presented. The method, which has the potential for application to many forms of microscopy, has been tailored to work with a through focal series of images measured in a high resolution transmission electron microscope. Practical difficulties for exit wave reconstruction which are pertinent in this experimental environment are the slight incoherence of the electron beam, sample drift and its effect upon the defocus step size that can be utilised, and the number of image measurements that need to be made. To gauge the effectiveness of the method it is applied to experimental data that has been analysed previously using a maximum likelihood formalism (the MAL method).

Breakout Session: Aberration Correctors, Monochromators, and Theoretical Basis

OUTLINE OF AN ULTRACORRECTOR COMPENSATING FOR ALL PRIMARY CHROMATIC AND GEOMETRICAL ABERRATIONS OF CHARGED-PARTICLE LENSES

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A novel ultracorrector is outlined which compensates for the primary and secondary first-order chromatic aberrations and all third-order geometrical aberrations of electron optical systems with a straight optic axis. This corrector is well suited for realizing an aberration-free high-resolution in situ transmission electron microscope. The telescopic ultracorrector consists of two identical symmetric quadrupole septuplets, which are separated by a distance such that the back principal plane of the first unit matches the front principal plane of the second unit. Its quadrupole fields are excited with opposite polarity with respect to those of the first septuplet. As a result the corrector only introduces aberrations with rotational and fourfold symmetry. Octopoles are incorporated to compensate for the third-order aberrations of the entire system consisting of round lenses and the ultracorrector. The octopoles must be placed and excited symmetrically with respect to the plane midway between the two septuplets. By choosing special locations for the octopoles, it is possible to successively eliminate the individual third-order aberrations in such a way that each subsequent correction does not affect the aberrations nullified in the preceding correction steps. The correction procedure must start with the elimination of the chromatic aberrations since this correction affects the third-order geometrical aberrations. The chromatic correction is performed by constructing the quadrupoles located at astigmatic images as crossed electric and magnetic quadrupoles. These elements act partly as quadrupole elements and partly as first-order Wien filters affecting only electrons whose energies differ from the nominal energy. The chromatic aberration is eliminated by properly adjusting the electric and the magnetic quadropole strengths.

HIGH SPATIAL AND ENERGY RESOLUTION EELS

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In conventional microscopes, the effects of lens aberrations are balanced by the use of apertures that severely limit the signal levels that can be obtained. Such limitations mean that spectra are usually very noisy, if they are acquired approaching atomic spatial resolution (short acquisition times), or have a spatial resolution that is limited to a few nanometers. This degradation of spatial resolution is increased if monochromators are used to improve the energy resolution of the spectra from the ~1eV in conventional microscopes to a more useful ~0.2eV (further reducing the signal levels). In addition to the improvements in imaging techniques, aberration correctors in both TEM and STEM modes can therefore dramatically improve the ability to perform electron energy loss spectroscopy (EELS) with high energy and spatial resolution, by simply increasing the signal levels. The ability to routinely obtain atomic resolution spectra with ~0.2eV energy resolution will lead to detailed characterization of the electronic properties of interfaces, defects and individual nanostructures. Furthermore, the improved accuracy of the spectroscopic methods will permit a direct correlation of the experimental results with computational analyses. Here, preliminary results from monochromated Schottky field emission microscopes and Cs corrected cold field emission microscopes will be presented. These results will be used as a basis for possible spectroscopic developments to be incorporated into the first stage of the TEAM project.

BEYOND UNIVERSITY FACILITIES: OPPORTUNITIES IN SPECTROSCOPY

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By combining monochromators with aberration-corrected optics, atomic-scale electron spectroscopy limited only by the lifetime of the excitation itself becomes a very real possibility. With such unprecedented spatial and spectral resolution, the microscopic underpinnings of structural and electronic phase transitions could be examined directly. As one such example, low temperature energy loss experiments should shed light on hotly debated problems such as charge-disproportionation, localization and conduction mechanisms in strongly correlated systems such as oxides or organic thin films. Very little is known about the microstructure and electrically active defects in polymer electronics, but if radiation damage can be ameliorated at low temperatures, the electronic structure and core edges are well suited to EELS analysis. The challenges involved in coupling a monochromator, Cs corrector and stable, low drift, low temperature stage, and the expertise required to keep such a system operating make the endeavor well suited to a national facility. This would also avoid the “jack of all trades” syndrome that so often afflicts university microscopes which must be all things to all users. By drawing on a national user base, optimization for specific tasks becomes more practical. That the time required for EELS data analysis will likely greatly exceed the data collection time makes

student travel to such a facility more practical and tolerable. This is in fact reminiscent of those x-ray synchrotron or astronomy facilities that enjoy sustained external university collaborations.

ADVANTAGES OF CHROMATIC ABERRATION CORRECTION FOR MATERIAL SCIENCE RESEARCH.

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During the last 10 years several concepts for aberration correction for electron microscopes (SEM, TEM, STEM) have been realized in order to achieve a higher spatial resolution. These correctors, which correct for spherical aberration, have already proven to be valuable tools for material science research. Lens systems for correction of chromatic aberration for TEM have been proposed but they are not presently feasible due to the current stability requirements of 10^{-8} for the multi pole elements. Recently, new designs for chromatic aberration correction were suggested that require a stability of 10^{-7} which is attainable with present technology. Contrast transfer calculations show that a point resolution of 0.5 Å at 200 kV and 0.6 Å at 100 kV would be possible with such a corrector system. This allows access to 3D HRTEM at defects in crystalline material and atomic resolution of amorphous or glassy material. In addition to improving resolution, C_c correction increases the sensitivity of HRTEM images. Such a correction system also enables high resolution TEM at lower voltage, an important aspect to minimize radiation damage.
Furthermore, using lower voltages without compromising the resolution significantly is very helpful for energy filtered imaging, which to date has been limited primarily by chromatic resolution. A gain in resolution for elemental maps of up to an order of magnitude can be achieved by C_c correction. In situ measurements of mechanical properties and in situ experiments in general benefit from thick samples where chromatic aberration is a limiting factor. Therefore, C_c correction provides important advances for these topics as well. C_c correction also provides significant benefits for STEM mode because it enables beam currents which are about 10 times larger than that which can be realized using only C_s correction.

DEVELOPMENT OF A C_S PROBE CORRECTOR FOR FEI TEM/STEM SYSTEMS

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The resolution of present day Scanning Transmission Electron Microscopes is limited by the spherical aberration of the objective lens. Elimination of this aberration by introduction of a multipole aberration corrector, results in an improved resolution or an increased current for a given probe size. Advances in manufacturing technology and computing power have made the operation of such an electron-optical device feasible.

At Philips Research an aberration corrector has been developed that corrects for the spherical aberration of the objective lens. Ultimately, this results in a sub-0.1 nm probe size, or an analytical probe with 100 times more current compared to an uncorrected probe.
The corrector has been built into a modified Tecnai F20 Super Twin. Accurate alignment of the condenser, objective lens and corrector modules has been achieved by mechanical design. Magnetic cross-talk between the modules has been eliminated.
For the development of the quadrupole-octupole type corrector special emphasis was put on the design and construction of the multipoles, to prevent saturation and to minimize cross-talk. The attained mechanical precision of only a few micrometers relaxes the requirements on the power supplies. Software has been developed which integrates the microscope and corrector control as well as computer assisted alignment routines.
Experiments have shown that by means of correction of the spherical aberration, the optimum opening angle has been increased more than two-fold. The results of these experiments will be discussed.
With the C_s probe corrector switched on, TEM images have been acquired at various magnifications, indicating that TEM functionality in the Tecnai remains possible with the C_s corrector switched on.

TOWARDS HALF-ÅNGSTROM RESOLUTION: FROM OÅM TO TEAM

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Sub-Ångstrom resolution is important for nanotechnology. Metal atoms can be routinely imaged in TEM specimens at resolutions from 2Å to 1.5Å. Better resolutions (~1Å) are required to "see" lighter atoms such as carbon [1], nitrogen [2] and lithium [3]. Once CS is corrected, microscope information limit controls resolution. The one-Ångstrom microscope (OÅM) project at LBNL has demonstrated the capability of 0.78Å resolution at 300keV [4]. The Transmission Electron Achromatic Microscope (TEAM) is proposed [5] to reach resolutions of 0.5Å using hardware correction of CS [6], a monochromator (to reduce electron-beam energy spread and improve its information limit beyond that of the OÅM), and chromatic aberration correction to allow a range of electron energies to be focussed together.
Methods employed in design and implementation of the successful OÅM project [1] can be used to determine appropriate parameters for the TEAM [7]. Calculations show that a

CC corrector is not required for TEAM to reach 0.5Å at 300keV or 200keV, provided that energy spreads can be reduced to 0.4eV and 0.2eV respectively. These values allow substantial beam current. At lower voltages, TEAM would require stricter limits on energy spread to reach the targeted 0.5Å resolution. No improvement in HT stability is required to improve the information limit *per se* since the monochromator determines the energy spread in the beam. However, improved HT will improve the beam current statistics (number of electrons passing through the monochromator) by placing more of the electrons closer to the center of the energy-spread distribution [8].'

[1] M.A. O'Keefe et al., *Ultramicroscopy* **89** (2001) 4: 215-241.
[2] C. Kisielowski et al., *Ultramicroscopy* **89** (2001) 4: 243-263.
[3] Yang Shao-Horn et al., *Nature Materials* (in press).
[4] M.A. O'Keefe, E.C Nelson, Y.C Wang & A. Thust, *Philosophical Mag. B* **81** (2001) 11: 1861-1878.
[5] B. Kabius, C.W. Allen & D.J. Miller, *Microscopy & Microanalysis* **8** (2002) 2: 418-419.
[6] M. Haider, G. Braunshausen & E. Schwan, *Optik,* **99** (1995) 167-179.
[7] M.A. O'Keefe, NTEAM meeting (May 20, 2002) and NTEAM Workshop (July 18-19, 2002), LBNL.
[8] Work supported by the Director, Office of Science -- through the Office of Basic Energy Sciences,
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STATUS AND FUTURE DEVELOPMENTS OF ABERRATION-CORRECTED AND IN-COLUMN ENERGY FILTERED 200KV FEG-TEMS

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We present LEO's concept and components for state-of-the-art and future high-end TEM instruments dedicated to utmost point resolution for high-resolution imaging and best energy resolution for analytical applications. The concept of a "hanging column" was realized applying a specially developed support frame in which the electron-optical column with increased diameter is fixed close to its center of gravity like a pendulum providing highest mechanical stability. The 200kV FEG Schottky Field Emitter source is designed to house a dispersion-free monochromator of the electrostatic Omega-type. The SATEM instrument (Sub-Ångstrom-Transmission-Electron-Microscope) [3] aiming at a point resolution below 0.9 Å is equipped with such a monochromator and a Cs-corrector for the imaging system [1,2]. The Cs corrector allows tuning of the Cs value down to zero or even to negative values improving the point resolution down to the fundamental limit given by incoherent damping. The monochromator narrows the FWHM of the energy spread which shifts the envelope function of temporal coherence below 1Å. A newly developed in-column energy filter of corrected Omega type is integrated into the SATEM

and the new 200 kV FE series TEM instrument. Multi-pole correction elements and a higher dispersion increase isochromatic field of view, maximum acceptance angle for CBED and transmissivity. Energy resolution of this corrected Omega filter is only limited by the energy spread of the source even in the case when the FE source is equipped with a monochromator. First results of the SATEM will be reported demonstrating the progress of system integration.

References
[1] M. Haider et al., Nature **392** (1998), 768
[2] H. Rose, Optik **34** (1990), 19
[3] G. Benner, A. Orchowski, M. Haider, P. Hartel, M&M 2003 conference (2003)

ARTIFACTS IN ABERRATION-CORRECTED ADF-STEM IMAGING*

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As the STEM probe advances into sub-Å region in size thanks to the introduction of aberration corrector, the peak intensity in the ADF images increases for a zone-axis crystal and the lowest signal (background) drops. The introduction of an experimental black level may clip the lowest signal without being noticed and introduce unintended high-frequency artifactual details into the high-resolution lattice images. We present the multislice simulation results of such possible situations. Three simulated STEM probes of sizes 0.8 Å, 1.2 Å and 2.0 Å are scanned on the surface of a $<\bar{1}10>$ oriented Si/Ge crystal. The simulation results suggest that high-frequency artifact peaks will appear in the power spectra when an artificial black level clips the lowest signal. Therefore, care must be taken when interpreting the resolution limit of the microscope from images taken with nonzero black level setting, especially in case of sub-Å microscope. The simulation result is compared with an experimental image and they agree with each other. The analysis suggests that aberration corrected STEM provides sensitive low level detail.

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