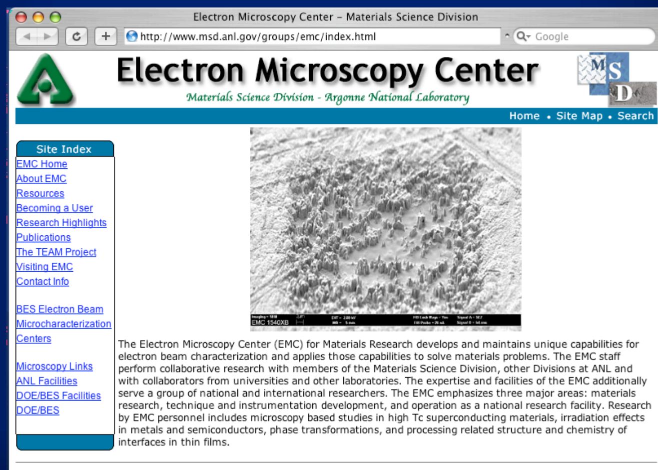
ANL Electron Microscopy Center

A DoE Basic Energy Science User Facility

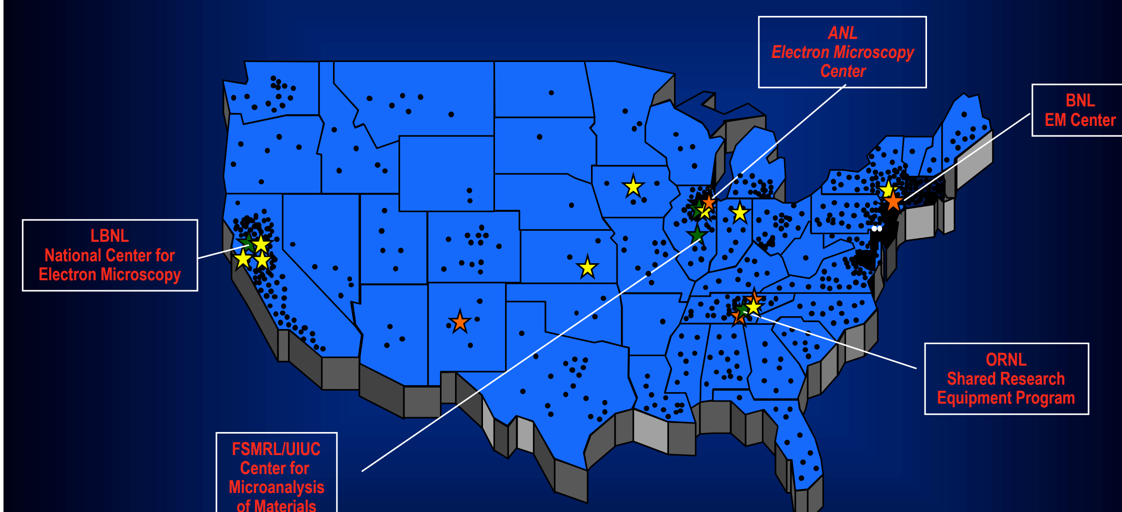


http://www.emc.anl.gov

Office of Science - BES Program
US Department of Energy

Electron Microscopy Center
Materials Science,Division, Argonne National Laboratory

US DoE BES Program 18 Scientific User Facilities - 200 Institutions - > 1400 Research Projects




EMC – Becoming a User

http://www.msd.anl.gov/groups/emc/users/index.html

Electron Microscopy Center

Materials Science Division - Argonne National Laboratory

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Becoming a User at the EMC

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The first step to becoming a user is to register for access to Argonne's facilities using this link: Argonne National Laboratory's National User Facilities On-Line Registration System.

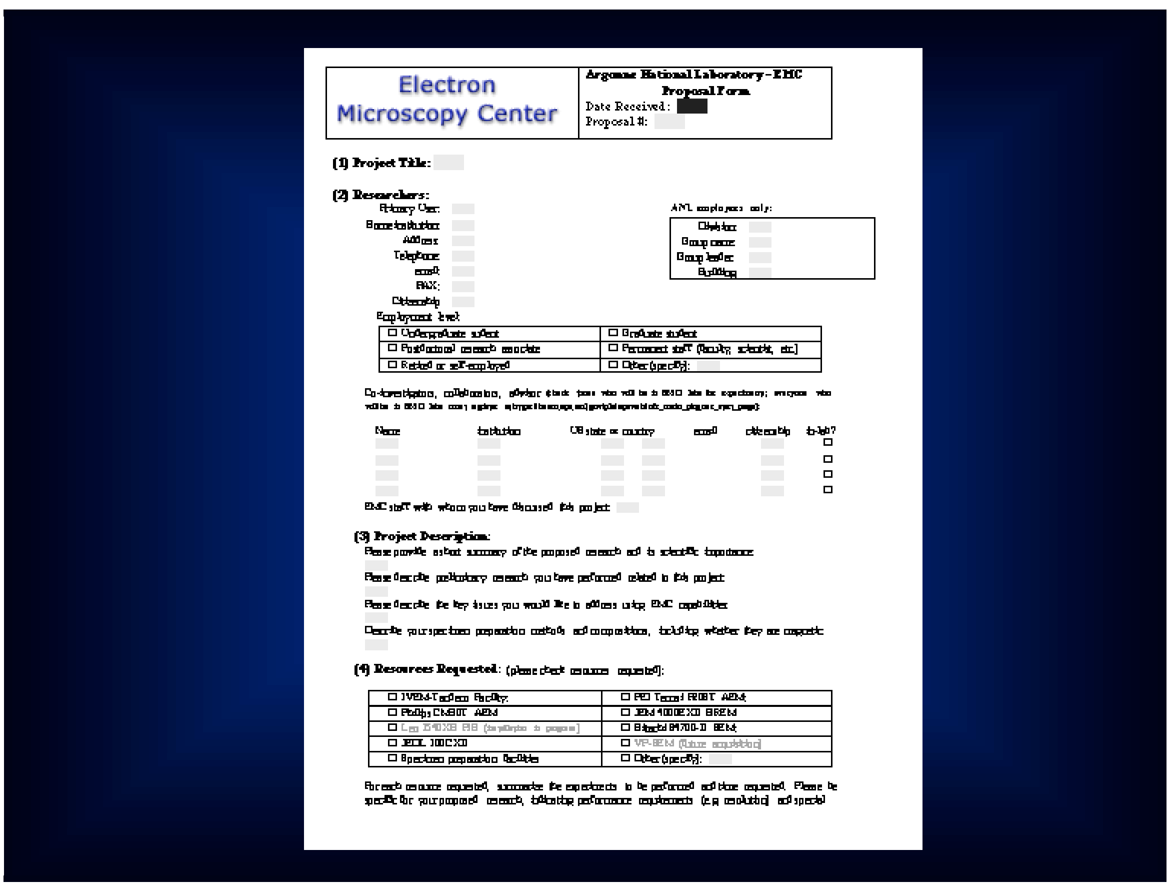
The next step is to complete an EMC Proposal form [doc - 108K]. Please fill out this form carefully, providing all of the requested information, and return it by email to emc@anl.gov. Because each research project is unique, it is strongly encouraged that a potential user discuss the proposed research with EMC staff before submitting a proposal in order to improve the review process.

When received, your proposal will be routed to the Director of the EMC for initial review and assignment to the appropriate proposal review committee. The time required for the approval process varies and depends on the nature of the proposal and the resources requested. Details on types of access is available here. Upon approval of your proposal and possible staff assignment, you will be contacted by an EMC staff member to arrange scheduling.

Additional information on user requirements and user status can be found here.

If you have questions, please contact:

Rebecca Videtic
Electron Microscopy Center
Materials Science Division, Bldg. 212
Argonne National Laboratory
Argonne, Illinois 60439
Tel: 630-252-4987
Fax: 630-252-6389
Email: videtic@anl.gov


Electron Microscopy Center

Argonne National Laboratory – EMC Proposal Form

Date Received:
Proposal #:

(1) Project Title:

(2) Researchers:

Primary User:
Home Institution:
Address:
Telephone:
email:
FAX:
Citizenship:
Employment level:

ANL employees only:
Division:
Group name:
Group leader:
Building:

☐ Undergraduate student	☐ Graduate student
☐ Postdoctoral research associate	☐ Permanent staff (faculty, scientist, etc.)
☐ Retired or self-employed	☐ Other (specify):

Co-investigators, collaborators, advisor (include those who will be in EMC labs for experiments; everyone who visits the EMC labs must register)

Name	Institution	US state or country	email	citizenship	in-lab?
					☐
					☐
					☐
					☐

EMC staff with whom you have discussed this project:

(3) Project Description:

Please provide a short summary of the proposed research and its scientific importance.

Please describe preliminary research you have performed related to this project.

Please describe the key issues you would like to address using EMC capabilities.

Describe your specimen preparation methods and compositions, including whether they are magnetic.

(4) Resources Requested: (please check resources requested):

☐ IVEM-Tandem Facility	☐ FEI Tecnai F20ST AEM
☐ Philips CM30T AEM	☐ JEOL 4000EXII EHREM
☐	☐ Hitachi S4700-II SEM
☐ JEOL 100CXII	☐ VP-SEM
☐ Specimen preparation facilities	☐ Other (specify):

For each resource requested, summarize the experiments to be performed and those requested. Please be specific for your proposed research, indicating performance requirements (e.g. resolution) and special

EMC Proposal Access Guidelines

One part of the EMC mission is to provide unique instrumentation and expertise to the scientific community. Important to achieving this goal is the effective management of EMC instrumentation and staff for maximum scientific impact. Broad and fair access to these resources is achieved through a proposal review process and scheduling and access policies.

The type of access granted is based on the nature of the proposed work, the qualifications of the user, and the instrumentation requested. Research projects may be allocated a specific amount of instrument time or may be granted continuous access.

Allocated Access:

> Under *allocated access*, a user is allocated a specific amount of instrument time to complete the proposed project. Allocated access is utilized primarily to manage instrument time for those instruments that have very high demand and for projects that require exceptional staff assistance.
>
> All new users and new projects will be designated for allocated access during the qualification and feasibility period.
>
> Allocated access is utilized for research on the IVEM-Tandem, FEI Tecnai F20ST, LEO 1540XB, and all assisted user research.

Continuous Access:

> Continuous access allows a user to use specified EMC instrumentation without limits on the amount of instrument time. Continuous access is intended for those users for whom Argonne is their primary research home or who otherwise demonstrate a consistent need for access to EMC instruments. Continuous access proposals terminate at the end of each fiscal year and must be renewed annually.
>
> A user must attain at least *User* status for each instrument utilized under continuous access. Typically, the Hitachi S-4700-II, Philips CM30T, JEM-4000EXII, and JEM-100CXII are utilized under continuous access for *Users* and *Operators*.
>
> Research projects may operate under both access policies. This may be the case when a research project requires the use of several instruments, for example.

Rapid Access:

> Recognizing the occasional urgent need, a proposal may be granted rapid access at the discretion of the EMC Director. Rapid access is intended to facilitate discovery and dissemination of significant, new scientific results and may be granted if an exceptional case is presented. Rapid access is not intended to accommodate poor planning or other experimental difficulties.

EMC – Resources

http://www.msd.anl.gov/groups/emc/resources/index.html

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Facilities and Resources

The instrumentation in the Center is organized according to specialized functions. Major areas in the Center include the IVEM-Tandem, the Analytical Electron Microscopy facility, the Advanced Analytical Electron Microscope, Specimen Preparation, and the Computing Center. In addition, a variety of other resources are available through collaboration with scientists in MSD. These resources are summarized below.

IVEM-Tandem

This unique facility consists of an intermediate voltage TEM (Hitachi H-9000 NAR) interfaced to two ion accelerators. This combination allows in situ observation of ion beam modification and effects of irradiation. In addition, a number of holders allow for a variety of in situ experiments to be performed, including low temperature studies in a liquid He stage. The accelerators also are used independently for ion beam irradiation/implantation and analysis including research for other programs. More information on the IVEM-Tandem is available at http://www.msd.anl.gov/groups/ht/.

AEM

The Analytical Electron Microscopy facility consists of electron microscopes that are optimized for high resolution and/or analytical work. This part of the Center primarily serves a regional and local user community.

Current instrumentation in the AEM includes:

- FEI Tecnai F20ST – analytical TEM and STEM
- JEM-4000EXII – high-resolution TEM
- Philips CM30T – analytical TEM
- JEM-100cx-II - TEM
- Hitachi S-4700-II – high resolution SEM
- LEO 1540XB – dual beam FIB

Additional information on the AEM facility is available at here [pdf - 121K].

AAEM and Materials Microcharacterization Collaboratory (MMC)

The Advanced Analytical Electron Microscope is a STEM/TEM with various modes, Telepresence, CTEM, CBED, EELS, windowless EDXS, CCD TV-rate imaging, SIMS, gas reaction cell, energy filtering, etc. This instrument serves primarily as a test-bed for the development of new techniques and methods. More information on the IVEM-Tandem is available at http://www.amc.anl.gov.

Specimen Preparation

Specimen preparation is an important part of electron microscopy. The EMC maintains an array of specimen preparation capabilities that are available to users. While users are expected to carry out their own specimen preparation, expertise and guidance may be provided by EMC or other MSD staff. The staff in the EMC have developed a number of new methods and approaches to sample preparation.

Image Analysis Laboratory

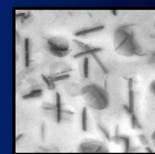
Open "http://www.msd.anl.gov/groups/ht/" in a new window behind the current window.

Microscopy & Microanalysis

Experimental methodologies which employs (electron-optical) instrumentation to spatially characterize matter on scales which range from tenths of a millimeter to tenths of a nanometer. The principle modalities employed are:

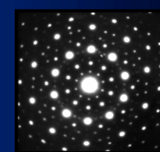
Imaging

- Transmission Electron Microscopy
- Scanning Transmission Electron Microscopy
- Scanning Electron Microscopy
- Focussed Ion Beam



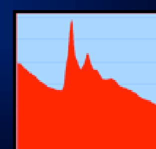
Diffraction

- Convergent Beam Electron Diffraction
- Selected Area Electron Diffraction
- Reflection High Energy Electron Diffraction



Spectroscopy

- X-ray Energy Dispersive
- Electron Energy Loss
- Auger Electron

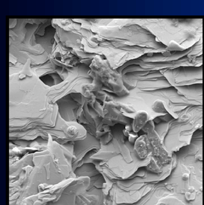


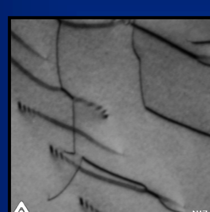
MicroCharacterization via Electron Microscopy

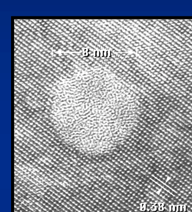
SEM
Scanning Electron Microscopy

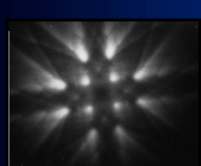
TEM - STEM - HREM
Transmission - Scanning Transmission - High Resolution Electron Microscopy

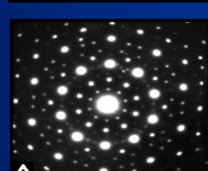
AEM
Analytical Electron Microscopy

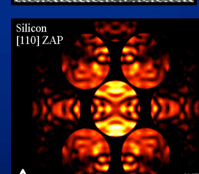


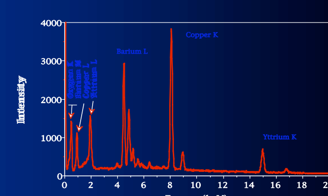


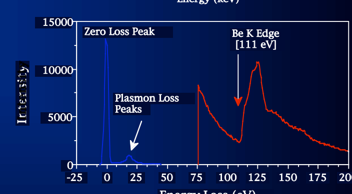




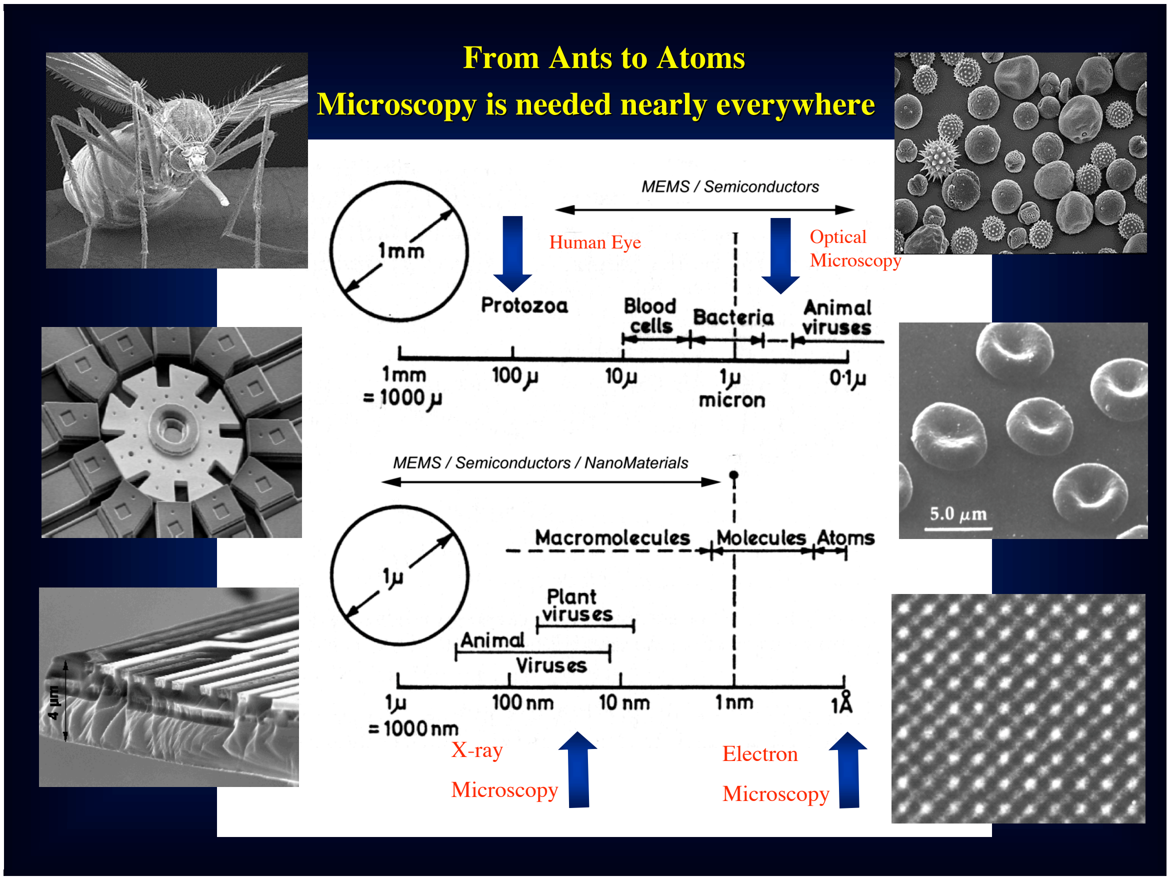


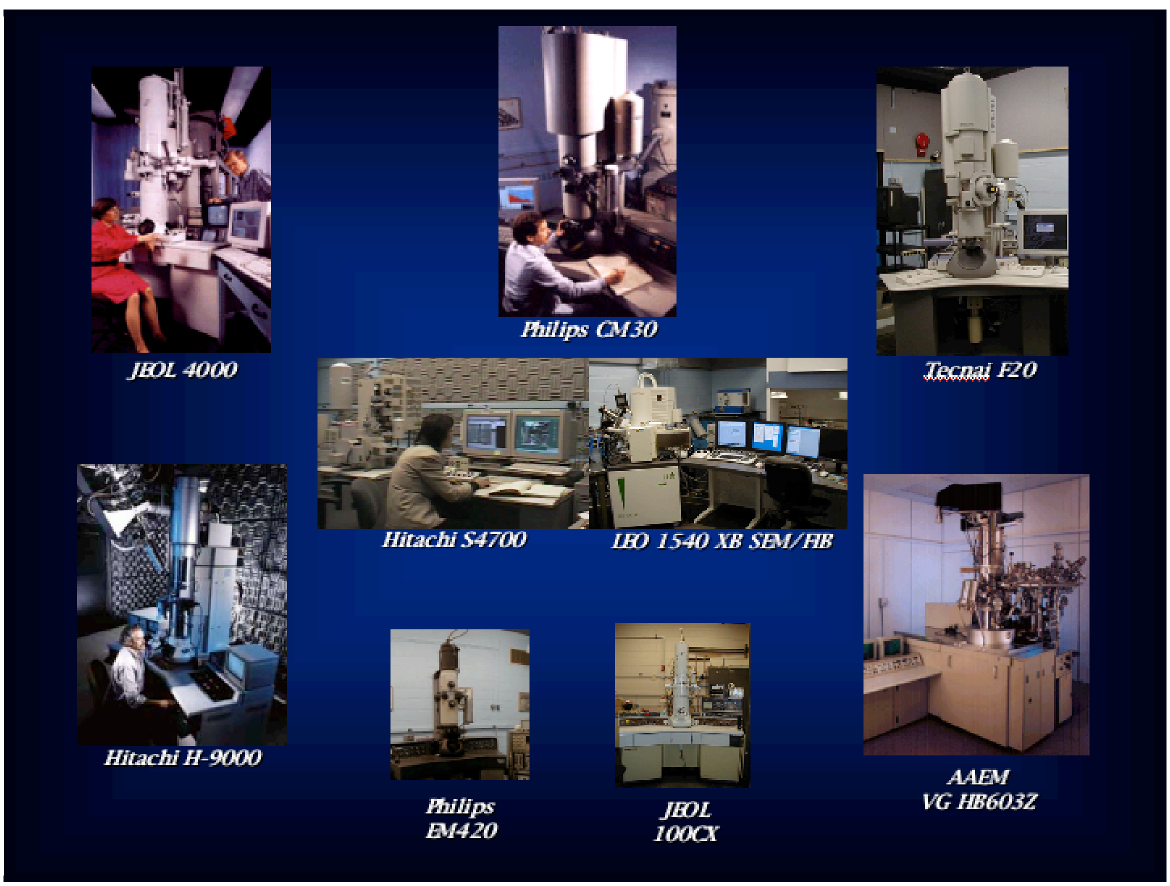




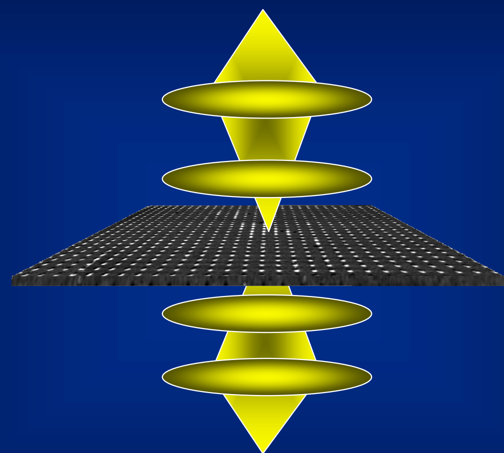


Morphology, Crystallography, Elemental , Chemical , Electronic Structure


From Ants to Atoms
Microscopy is needed nearly everywhere
MEMS / Semiconductors
1mm
Human Eye
Optical
Microscopy
Protozoa
Blood
cells
Bacteria
Animal
viruses
1mm
= 1000 μ
100 μ
10 μ
1 μ
micron
0·1 μ
5.0 μm
MEMS / Semiconductors / NanoMaterials
1μ
Macromolecules
Molecules
Atoms
Plant
viruses
Animal
Viruses
1μ
= 1000 nm
100 nm
10 nm
1 nm
1Å
X-ray
Microscopy
Electron
Microscopy
4 μm


JEOL 4000
Philips CM30
Tecnai F20
Hitachi S4700
LEO 1540 XB SEM/FIB
Hitachi H-9000
Philips
EM420
JEOL
100CX
AAEM
VG HB603Z

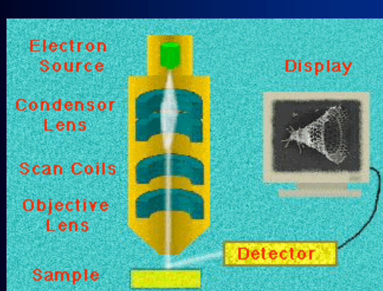
Scanning Microscopy
Deals Mainly with Near Surface Region

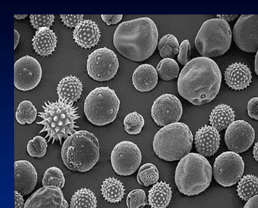


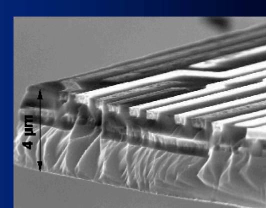
Transmission Microscopy
Deals Mainly with Internal Structure

Scanning Electron Microscopy

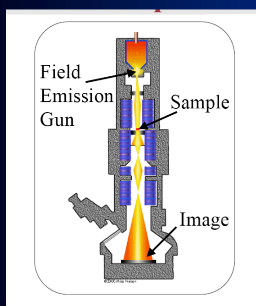
Hitachi S4700

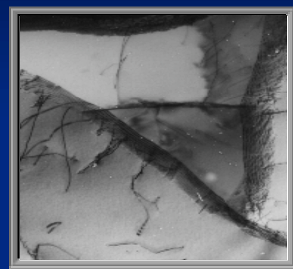


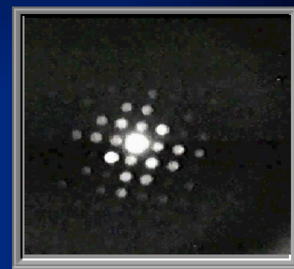


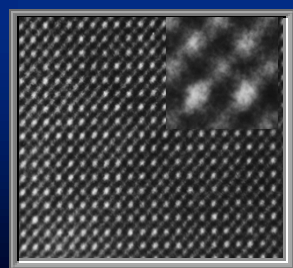


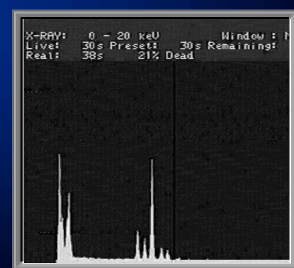
Transmission Electron Microscopy



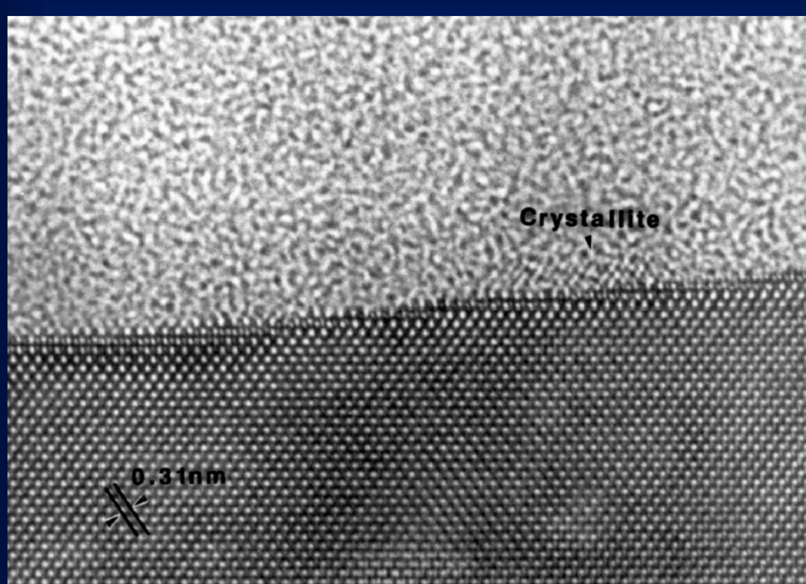




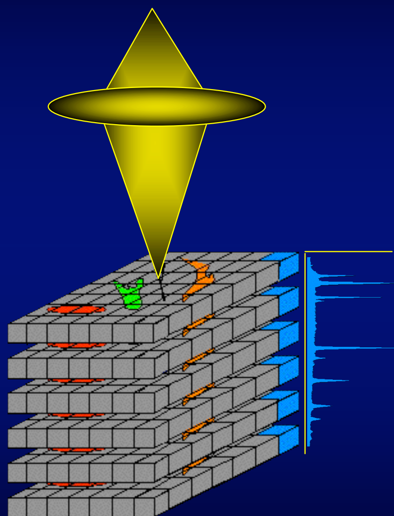


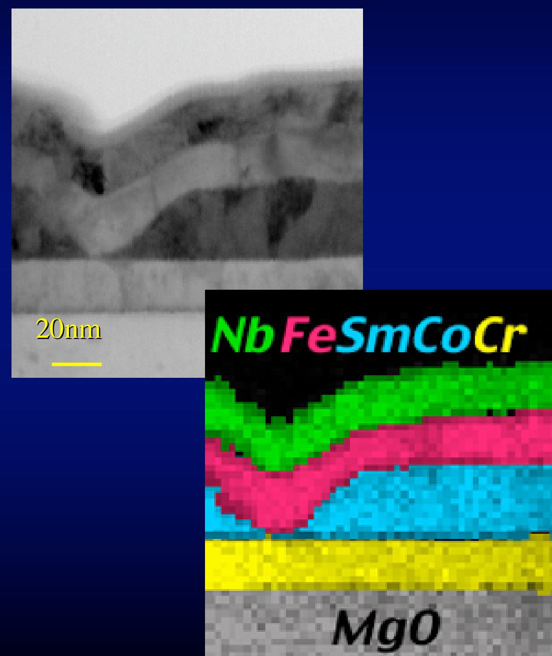


TEM-High Spatial Resolution Imaging

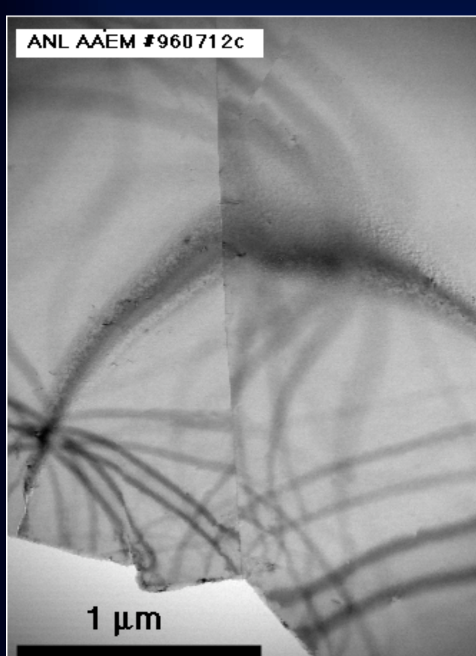


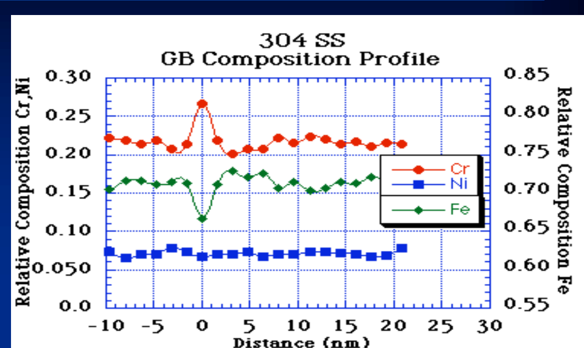
Spectral Imaging

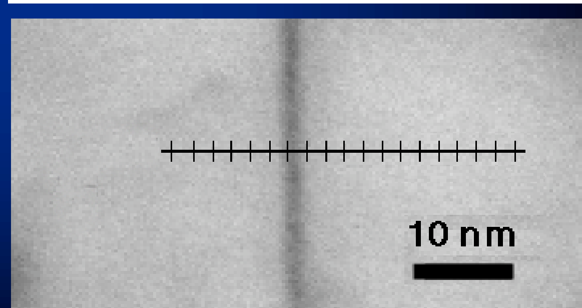




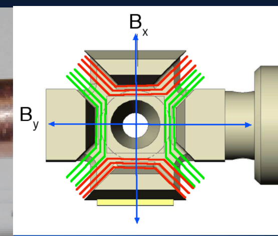
AEM - High Spatial Resolution Measurements of GB Segregation in 304 /316 SS

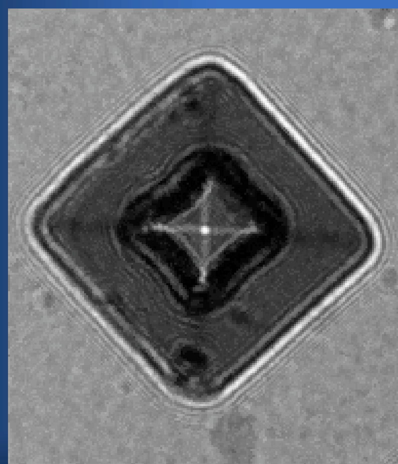


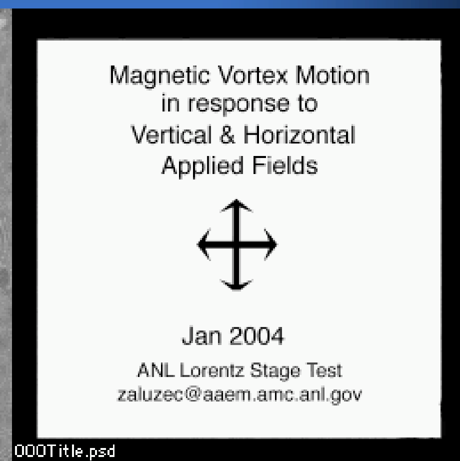




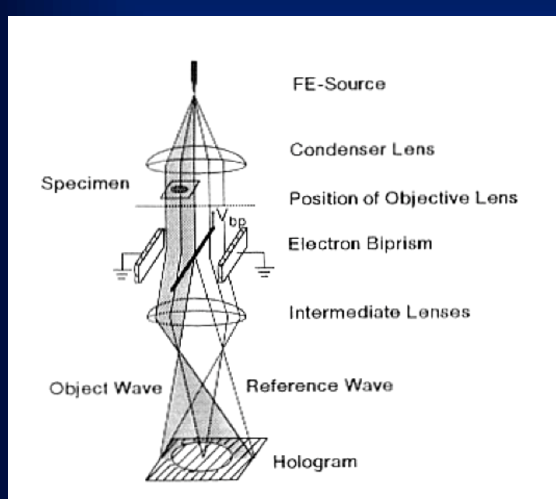
In-Situ Stage for Dynamic Magnetization Studies

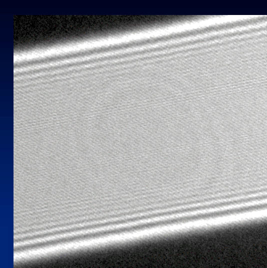


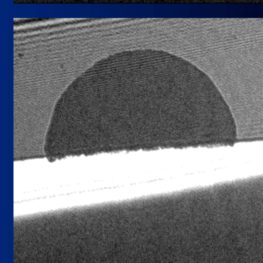


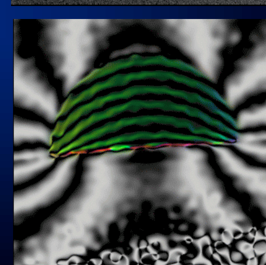


High Coherence Electron Holography









Feynman's Challenge

It would be very easy to make an analysis of any complicated chemical substance; all one would have to do would be to look at it and see where the atoms are. The only trouble is that the electron microscope is one hundred times too poor … I put this out as a challenge: Is there no way to make the electron microscope more powerful?

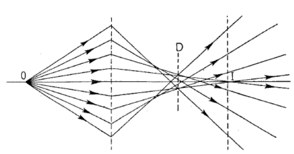
– Richard P. Feynman, 1959

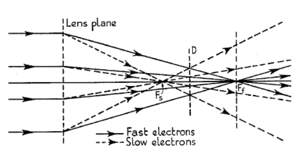
"There's Plenty of Room at the Bottom"

What are the limitations:?

Aberrations

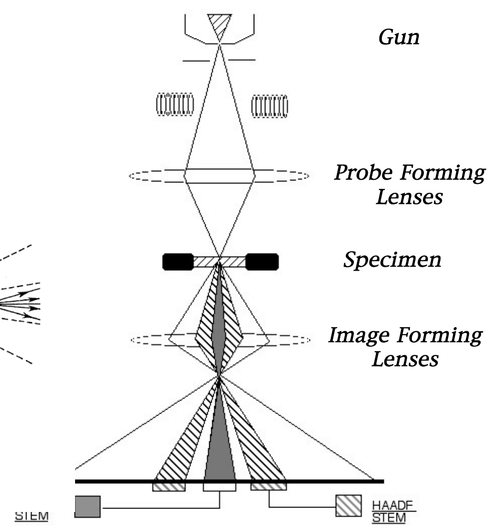
- Spherical
- Chromatic





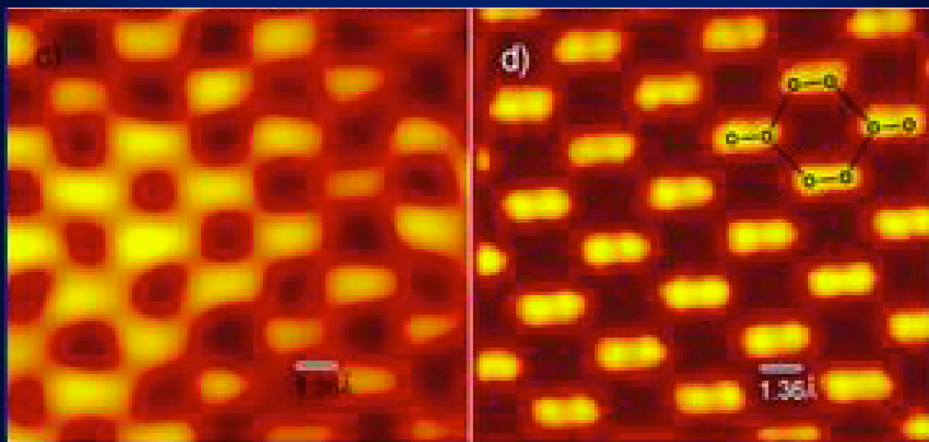
$$r_{sph} = C_s \beta^3$$

$$r_{chr} = C_c \frac{\Delta E}{E} \beta$$



What is the Limit of Current Technology In Aberration Correction

Silicon (110)



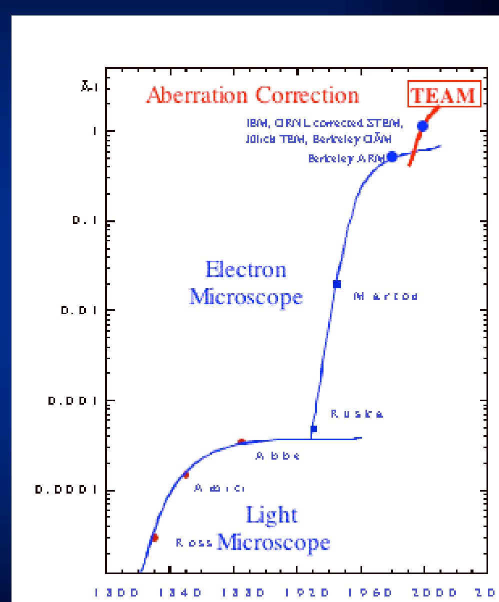
Before Correction *After Correction*

P.E. Batson IBM Yorktown, Jan 2004

Office of Science - BES Program
US Department of Energy

TEAM Project

- *A Series of Breakthroughs in electron optics has minimized the barrier that is limiting the performance of electron microscopes*
- *Simultaneous advances in electronics, detectors, MEMS and computers enables the technology to be practically implemented*
- *Nanoscience and Nanotechnology Centers need atomic level characterization*
- *DOE recognizes the importance and is investing in the development*



H. Rose, 2000

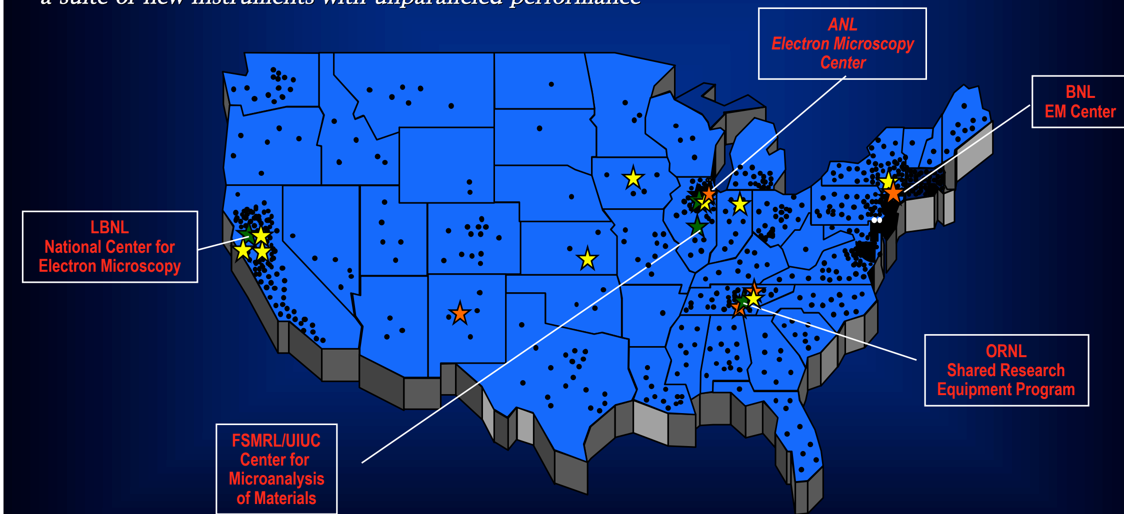
Transmission Electron Aberration-corrected Microscope (TEAM)

Office of Science - BES Program
US Department of Energy

TEAM Project

•Goals: To capitalize on recent advances in electron optics to design, build and operate the next generation of electron microscopes capable of 0.5 Å resolution in real time and with 3D with single atom sensitivity

•Resign the basic electron microscope around aberration corrected optics to serve as a platform for a suite of new instruments with unparalleled performance



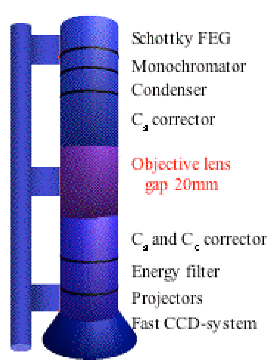
US DoE BES Program 18 Scientific User Facilities - 200 Institutions - > 1400 Research Projects

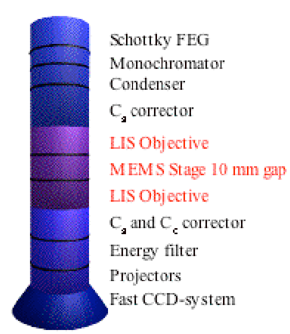
Office of Science - BES Program
US Department of Energy

TEAM Project

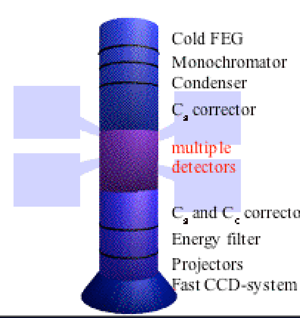
Based on TEAM platform, develop suite of aberration-corrected instruments for

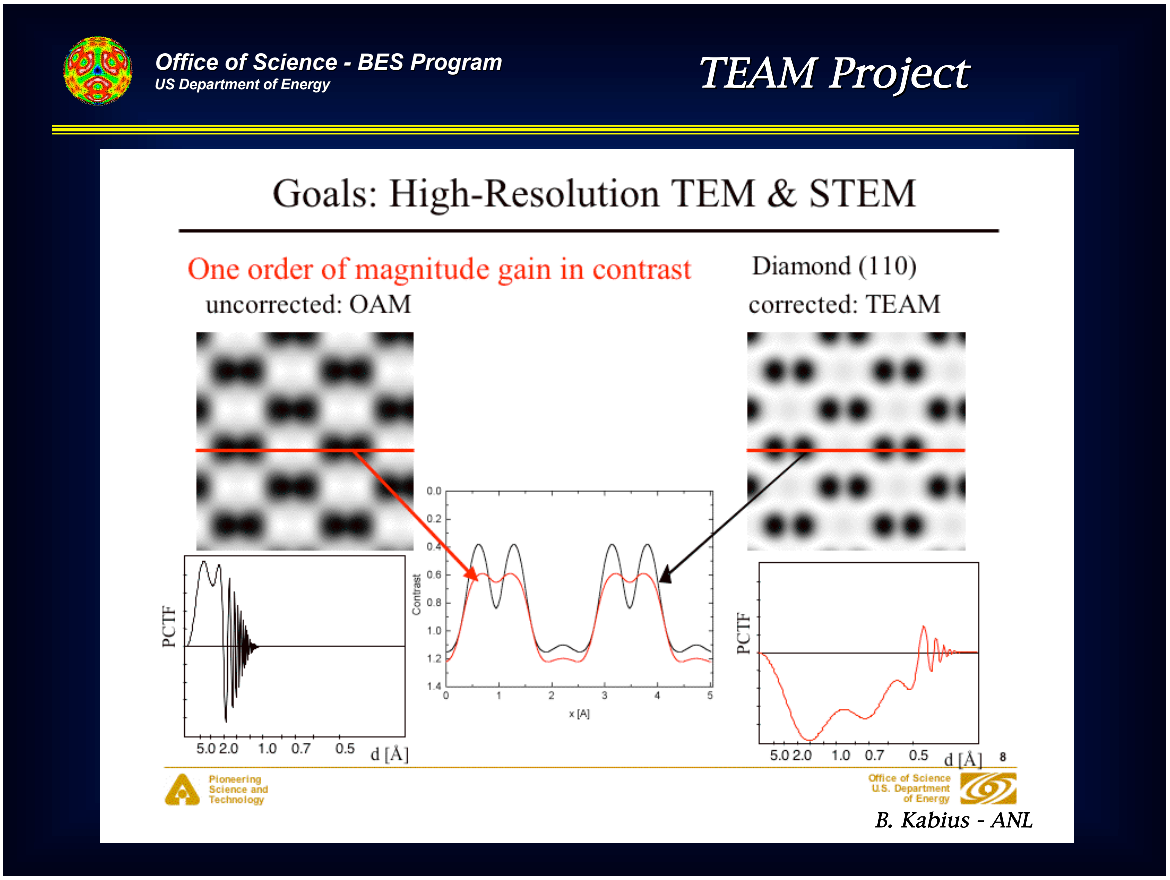
– In-situ synthesis
– UHV environment
– Ultimate microanalysis
– Fast dynamics
– Lorentz imaging, …

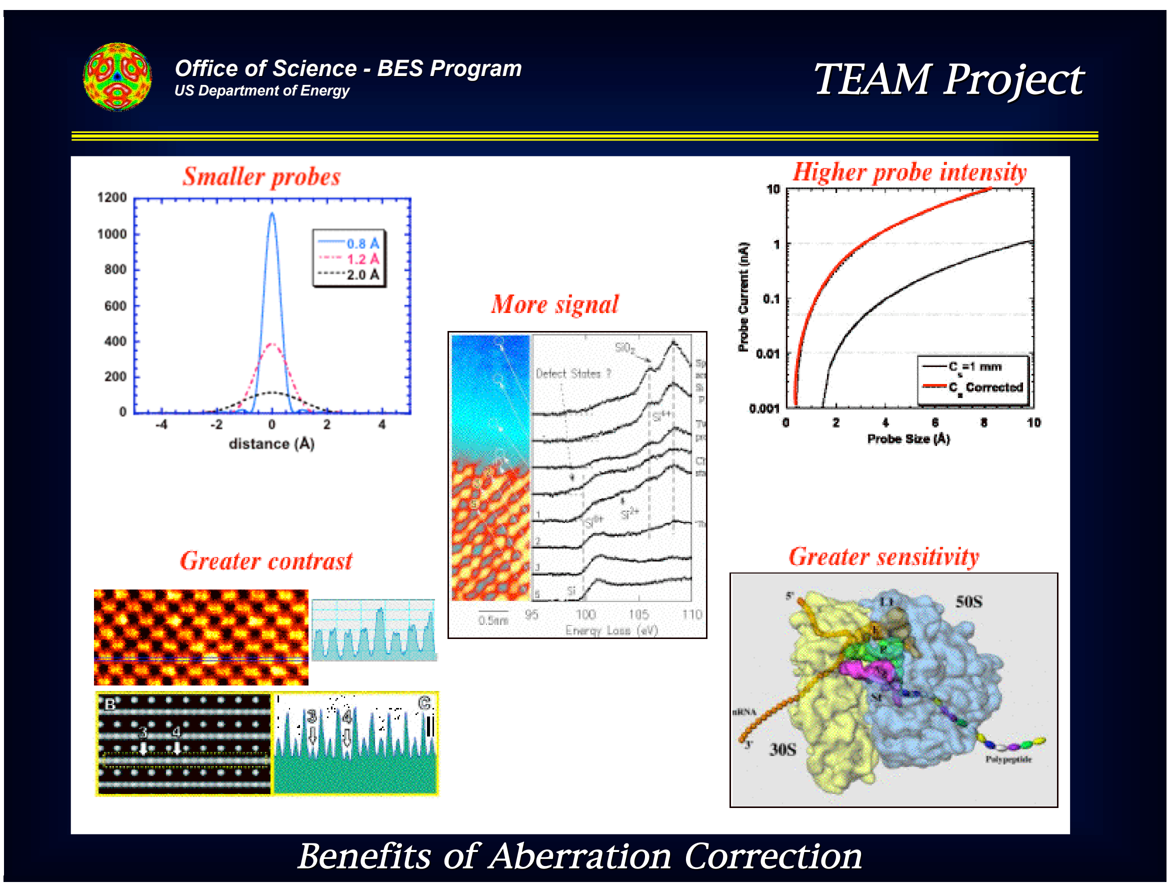




- Stimulate revitalization of electron optics in US.
- Instruments to be remotely accessible as a "distributed facility".
- Proximity to nation's synchrotron light and neutron sources and Nanoscience Centers.
- Well established user programs with missions that are aligned with BES science goals and provide national coverage - geography, scientific expertise, user community, instrumentation.




Office of Science - BES Program
US Department of Energy
TEAM Project
Goals: High-Resolution TEM & STEM
One order of magnitude gain in contrast
uncorrected: OAM
Diamond (110)
corrected: TEAM
PCTF
5.0 2.0 1.0 0.7 0.5 d [Å]
Contrast
x [A]
PCTF
5.0 2.0 1.0 0.7 0.5 d [Å]
8
Pioneering Science and Technology
Office of Science U.S. Department of Energy
B. Kabius - ANL


Office of Science - BES Program
US Department of Energy
TEAM Project
Smaller probes
0.8 Å
1.2 Å
2.0 Å
distance (Å)
More signal
Defect States ?
Energy Loss (eV)
Higher probe intensity
Probe Current (nA)
Probe Size (Å)
Cs =1 mm
Cs Corrected
Greater contrast
Greater sensitivity
50S
30S
mRNA
Polypeptide
Benefits of Aberration Correction

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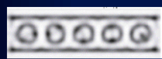
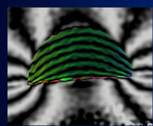
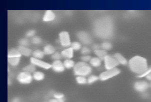
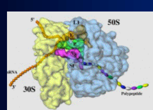
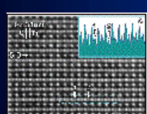
US Department of Energy

Scientific Challenges

- Nanomaterials: Syntheis , properties, assembly, electronic structure
- Semiconductors: End of the Road Map in Si Tech.
- Magnetic Materials: fundamental understanding of magnetic nanostructures
- Photonic Materials: Effects of dopants, point defects
- Catalysis: Energy, environment, transportation: controlled chemical processs
- Hard/Soft Materials: Increased sensitivity and resolution
- Oxidation/Corrosion: Real Time In-situ Experiments
- SuperConductivity: Imaging and Spectroscopy at the highest levels.

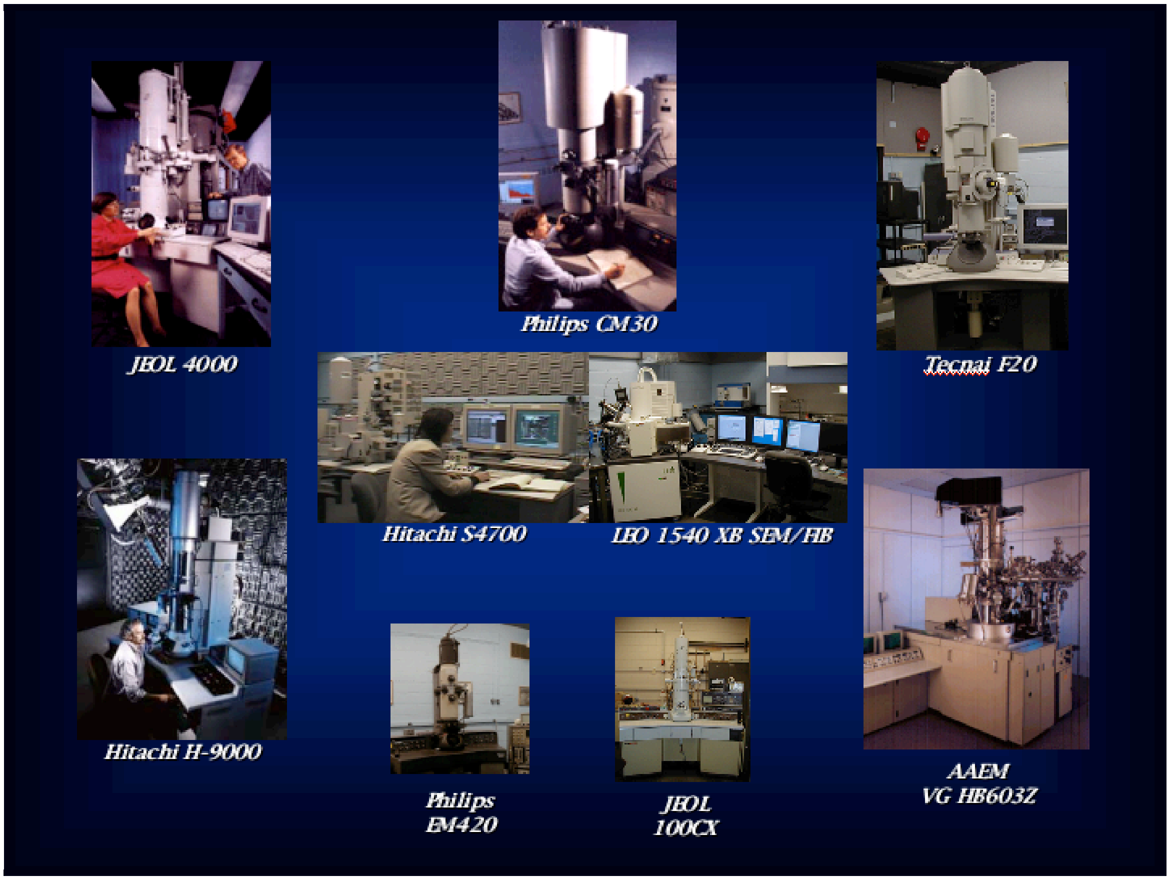
Office of Science - BES Program
US Department of Energy

TEAM Project

Extraordinary Scientific Opportunities
using direct observation of individual nanostructures

- *three dimensional atomic-scale structure, shape and defect distribution*
- *spectroscopic characterization and location of individual dopant atoms*
- *direct imaging of atomic scale structure of glasses*
- *electronic structure of individual point defects*
- *non-spherical charge density and valence electron distributions*
- *in-situ synthesis of novel nanoscale structures*
  e.g. electron beam lithography with sub-Angstrom beams
- *in-situ observation of properties and response of materials to external variables*
  temperature
  stress/strain
  environment & chemical activity
  applied electric & magnetic fields

..... All with unprecedented spatial, spectral and temporal resolution



Characteristics and Capabilities of Argonne's Electron Microscopes

Instrument	Operating Modes	Specimen Holders	Resolution
JEOL JEM-100CXII 10 – 100 kV	CTEM, Lorentz (LMI) at high-magnifications*, SEM, STEM.	*Double Tilt (±60˚ α, ±36˚ β):* double specimen, Be cup for EDXS, & cooling (93 K) with Be cup. *Single Tilt:* double specimen & bulk.	~0.7 nm point ~0.3 nm line (standard)
Philips CM30T ≤ 50 – 300 kV	CTEM, CBED, hollow-cone DF, light element EDXS, PEELS, electron dosimetry.	*Double Tilt (±60˚ α, ±30˚ β):* Be cup for EDXS, cooling (93 K) with Be cup, & heating (1250 K). *Single Tilt:* one. *Tilt/rotate (±60˚, 360˚):* one.	~0.25 nm point ~0.14 nm line probe size ≥ 9 nm
JEOL JEM-4000EXII 100 – 400 kV	HREM, CTEM, TV-rate video, digital imaging via slow scan-rate CCD camera, electron dosimetry.	*Top-Entry Double Tilt (±20˚):* three. *Top-Entry Zero Tilt:* two.	~0.165 nm point ~0.1 nm line
VG HB603Z* 50 – 300 kV (cold FEG)	STEM/TEM with various modes, Telepresence, CTEM, CBED, EELS, windowless EDXS, CCD TV-rate imaging, SIMS, gas reaction cell, energy filtering, etc.	*Double Tilt (±60˚):* Be cup for EDXS, cooling with Be cup. *Single Tilt:* heating.	Lattice: ~0.2 nm (STEM) ~0.4 nm (CTEM) Probe size < 1 nm
Hitachi H-9000NAR* 100 – 300 kV	CTEM, *in situ* ion irradiation, ion dosimetry, TV-rate high resolution video, light element EDXS.	*Double Tilt (±30˚):* heating (1250 K) & cooling (15 K – RT). *Single Tilt:* straining, electrical bias, & ion/electron dosimetry.	~0.25 nm pt-pt ~0.14 nm lattice
Hitachi S-4700-II SEM 0.5 – 30 kV (cold FEG)	SEI & BEI, light element EDXS with quantitative mapping.	5-axis motorized stage. Maximum sample size: 27 mm (H) x 150 mm (dia.).	SEI resolution: 1.5 nm at 15 kV 2.5 nm at 1.0 kV
FEI Tecnai F20ST 80 – 200 kV (Schottky FEG)	CTEM, STEM (BF/ADF, HAADF), CBED, light element EDXS, PEELS, Energy-filtered imaging (EFI), Lorentz magnetic imaging (LMI), electron holographic imaging (EHI), TV-rate video, etc.	*Double Tilt (±40˚ α, ±30˚ β):* Be cup for EDXS. *Single Tilt (±40˚):* one. Compustage. Other Philips holders.	~0.24 nm point ~0.1 nm line Cs obj. ≈ 1.2 nm Cc obj. ≈ 1.2 nm probe ≈ 0.2–1 nm