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JEOL

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**Buildings for Advanced Technology Workshop II**
Mesa, Arizona

# Analytical Instrumentation Facility Requirements for Nanotechnology

**January 21-23, 2004**

## Outline

- Analytical Instrumentation for Nanotechnology
  - Examples
- Facility Design Requirements
- Specifications
- Environmental Effects on Instrument Performance
- Compensation After Construction
- Summary

## JEOL Core Product Groups

### Electron Optics

- Scanning Electron Microscopes (SEMs)
- Transmission Electron Microscopes (TEMs)
- Surface Analysis (Auger, EPMA)
- Scanned Probe Microscopes (SPMs)

### Electron Optical Instruments for Semiconductors

- Electron Beam Lithography
- Defect Review Tools
- Inspection Tools

### Analytical Instruments

- Mass Spectrometers
- NMRs
- ESRs



## JSM-7700F Scanning Electron Microscope

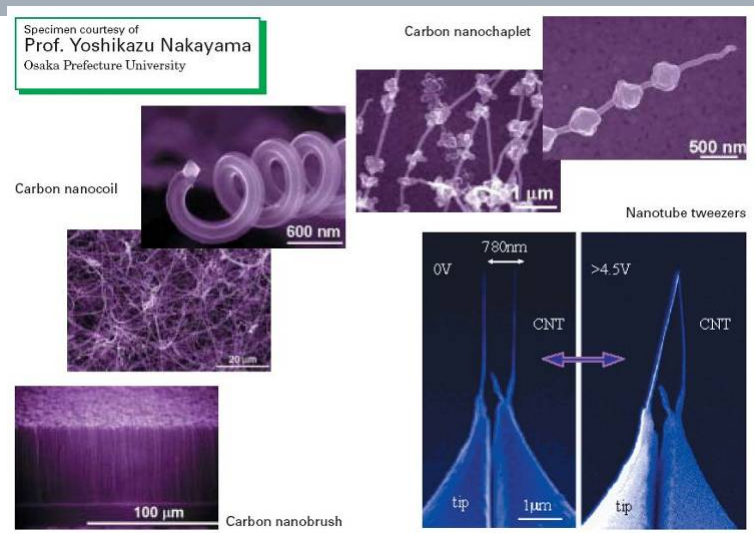
High resolution surface analysis

Resolution of to 0.6 nm

Magnification range from 25x to 2,000,000x

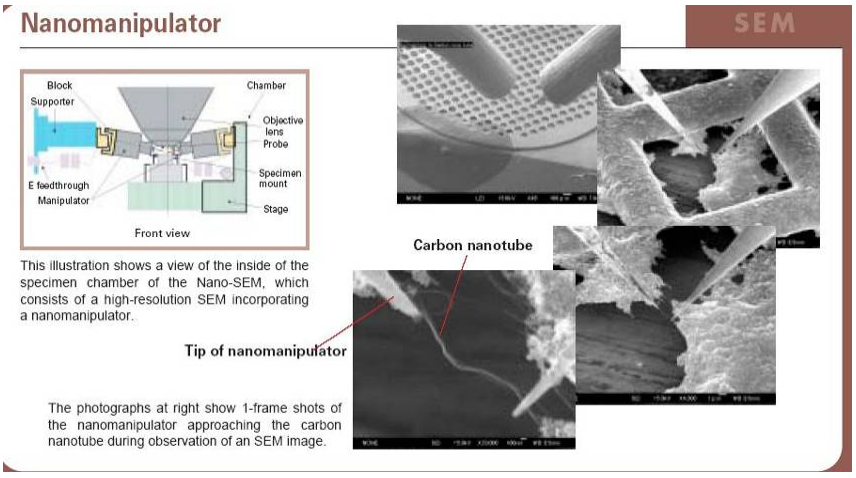
Windows 2000 based operating system

## High Resolution SEM Images of Carbon Nanomaterials





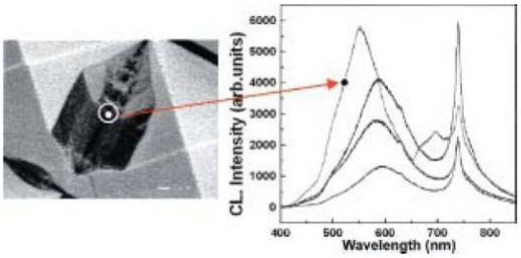

## SEM Nanomanipulator



## Combined SEM and Raman

Exterior view of SEM/Raman instrument

SEM image of multi-crystal diamond
and local Cathodoluminescence spectrum




Permits both SEM observation and Raman spectroscopy by irradiating the specimen with the electron beam of the SEM and the laser beam of a Raman spectrometer coaxial with the SEM's electron beam



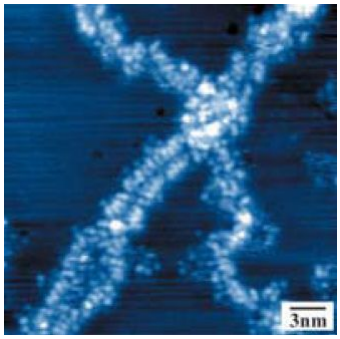
## JSPM-5200 Scanned Probe Microscope

Can be used in a variety of environments: air, controlled atmosphere, liquids or vacuum

Specimen can be heated or cooled

Variety of STM and AFM modes

## SPM Example




Specimen courtesy of
Prof.Tomoji Kawai
Director of Intermaterial Research Center,
Osaka University.

Direct Observation of DNA Using an Ultralow Temperature STM

The double helical structure and the way the molecules face each other can clearly be seen.



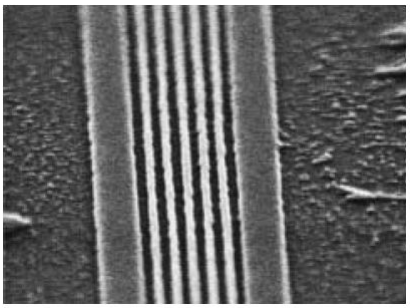
## JBX-9300FS Electron Beam Lithography System

Multiple modes

Automation

Dynamic correction system eliminates defocusing resulting from beam deflection

## InP 40nm Periodic Structure on Calixarene Resist



Specimen courtesy of
Tokyo Institute of Technology
Research Laboratory of Ultra-High Speed Electronics.



## JEOL Transmission Electron Microscopes (TEMs)

| | |
|---|---|
| 2100F: | 200kV FEG TEM/STEM |
| 2200FS: | 200kV FEG with Omega Filter |
| 2500SE: | 200kV FEG STEM/TEM |
| 2200FS ACEM: | 200kV FEG TEM/STEM with Omega and STEM Cs corrector |
| 2200F Mirai: | 200kV FEG TEM/STEM with Omega and Monochromator |
| 2200F AOTA: | 200kV FEG TEM/STEM with All of the Above |
| 3200S: | 300kV LaB6 TEM with Omega Filter |
| 3200FSC: | 300kV FEG TEM with high tilt Liquid He holder |

## JEM-2100F Field Emission TEM

New computer architecture
- Client/server compatible
- Com or dll interface
- Windows FLC/Full Capability

New vibration Isolation
- Balanced Center of Gravity
- New Goniometer Cover

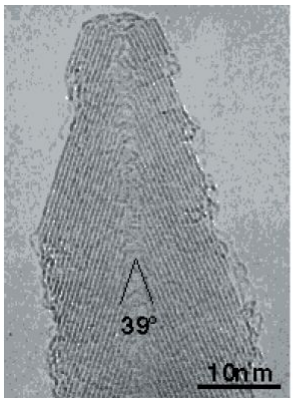
New Digital STEM
- 50MX
- 0.136nm Resolution on Factory floor during the day

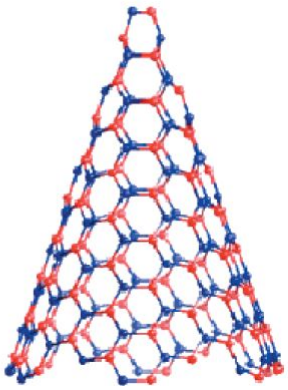
Piezo Stage built-in

Remote Control
- all functions



## Discovery of a Nanocone of Boron Nitride



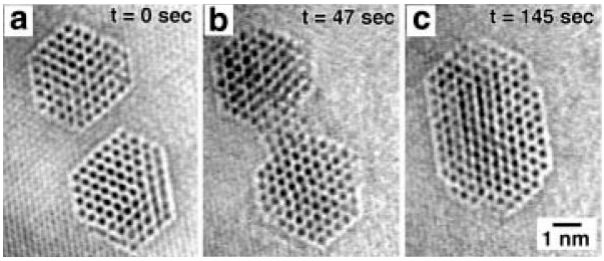



Unlike carbon nanocones, which have a tip angle of 60°, BN cones have tip angle of 39°.

Possibly could be used as a probe for high resolution AFM.

Specimen courtesy of
Dr. Yoshio Bando
Head of Integrated Strategy Office,
Integrated Strategy Office,
National Institute for Materials Science.

## Formation of Xe Nanoparticles

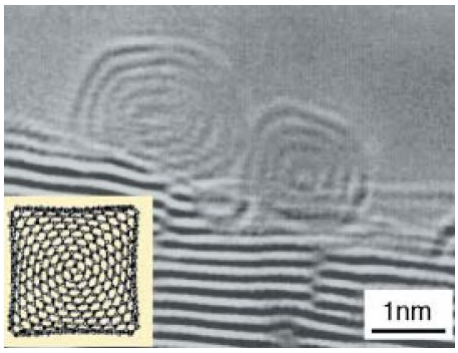



In-situ formation and combination of Xe nanoparticles in aluminum by injection of Xe ions into the Al.

Specimen courtesy of
Dr. Kazuo Furuya
Supervising Researcher Nanomaterials Laboratory,
National Institute for Materials Science.



## Discovery of Fullerene of Boron Nitride

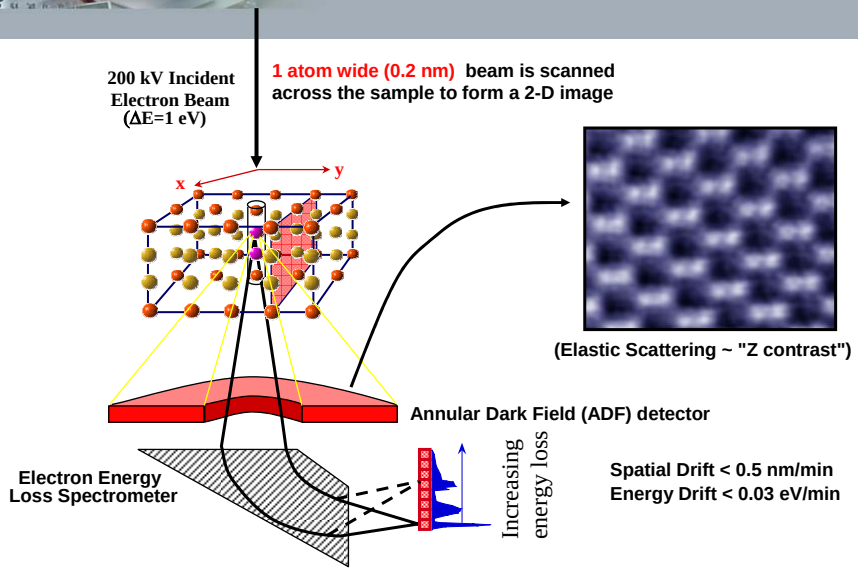



These BN fullerenes were discovered by using a new method in which a high speed electron beam is irradiated at the specimen while being imaged in the TEM.

Where C60 fullerenes have a soccer ball like structure, these BN fullerenes are shaped like a die.

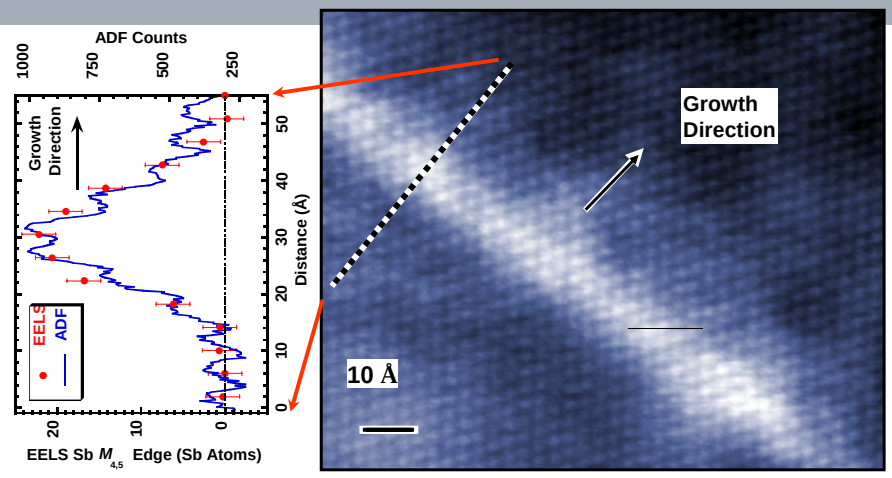
Specimen courtesy of
Dr. Yoshio Bando
Head of Integrated Strategy Office,
Integrated Strategy Office,
National Institute for Materials Science.

## Scanning Transmission Electron Microscopy (STEM)






## Demonstration of Single-Atom Sensitivity:
### Imaging Sb Atoms in a δ-doped layer




Detection Limit is ±2 atoms by EELS, ±0.5 atoms by ADF

David Muller, Paul Citrin, Hans Gossman, R. Vanfleet, *Phys. Rev. Lett.* 1999

## Facility Design Requirements

General

- Room Size
- Lighting
- Heating
- Ventilation
- Privacy
- Safety

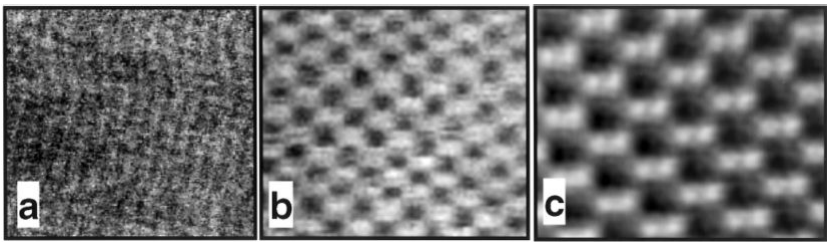
Special requirements for analytical instrumentation

- Mechanical vibration
- Magnetic fields
- Electrical disturbances
- Environmental changes
  - Temperature
  - Pressure



## Cs Correctors, Monochromators, Omega Filters


## Performance Improvement




After installation | After 6 months | After 1 year

Improvements from a) to b) are due to improvements to the microscope. Those from b) to c) are due to improvements in the room environment.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)



## Environmental Effects Upon Performance

- Vibrations
  - Acoustic noise
  - Mechanical
- Electrical disturbances
- Magnetic fields
  - Electromagnetic interference (EMI)
- Air flow
- Thermal fluctuations
- Pressure fluctuations

## Acoustic Noise - External

Planes, trains and automobiles

Machinery

Facility noise

- Other laboratory equipment
- People

Room must be acoustically shielded from outside

- Thick walls
- Insulation
- Heavy doors
- Room-in-room design



## Acoustic Noise - Internal

Air conditioning system

Cooling fans

Electronic noise

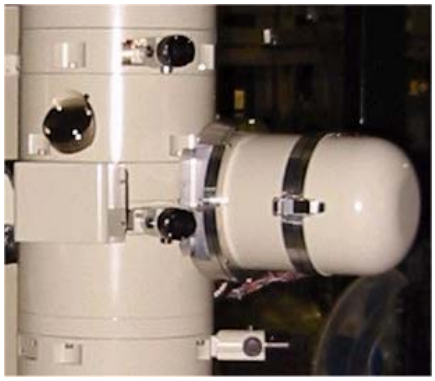
Human noise

Eliminate as many of the sources of noise as possible

In-room sound dampening system

- Foam
- Baffles

## Improved Stage Stability



New dampers and clamshell



## Mechanical Vibrations

- Large range of resonant frequencies
  - Specimen holders tend to resonate at high frequencies
  - Microscope lenses resonate at lower (<30Hz) frequencies
- Planes, trains and automobiles
- Heavy machinery
- Elevators
- HVAC
- Laboratory equipment
- Human induced
- Associated microscope equipment
  - HV supplies
  - Cooling supplies
  - Vacuum system

## Mechanical Vibrations

- During design, ensure that building frequencies are as high as possible
  - Much easier to attenuate high frequency vibrations
- Location, location, location
  - Away from loading docks, storerooms, stockyards
- Isolated pad for facility
  - Decouple laboratory from rest of building
- Solid foundation for instrument
  - Ground floor
- Isolated pad for each instrument
  - Decouple instrument room from rest of laboratory
  - Decouple instrument from auxiliary equipment
- Vibration dampening
  - Equipment away from instrument
  - Instrument itself



## Electromagnetic Interference

- Electricity supplies
  - High-voltage transmission lines
  - Local substation
    - Transformers
- Subways, electrical trains
- Three-phase supply
  - If loads uneven – current in neutral conductor
- Mains
- Equipment motors
  - Elevators, hoisting cranes, AC plant
- Other equipment
  - Arc-welders
  - Arc-discharge equipment – coaters, etc.
- Grounding
- Lighting
- Monitors

## Electromagnetic Interference

For a straight conductor, the magnetic field is given by Ampere's Law:

$B = 2I/d$

- B=magnetic field in mG
- I=current in mA
- d=distance in m

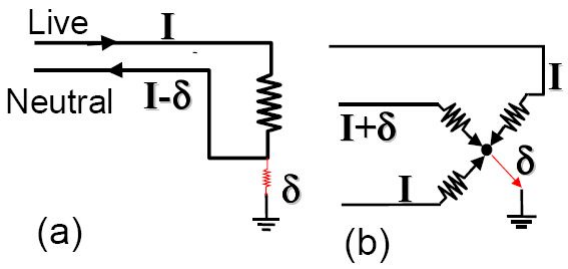
Direction of field given by right-hand rule

A 0.3 mG rms magnetic field can be detected in a 0.3 nm STEM image

At 1m distance from a wire carrying 0.5 mA, a 1mG field will be detected – enough to degrade 0.3 nm performance



## Origin of Ground Currents

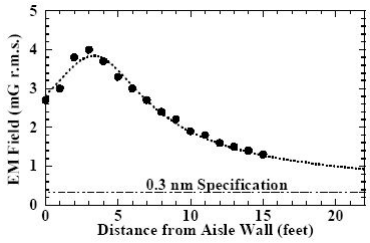



Single phase circuit Three phase circuit

Net AC field generated will be proportional to the current δ lost to ground

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)

## Magnetic Field Generated by Power Bus

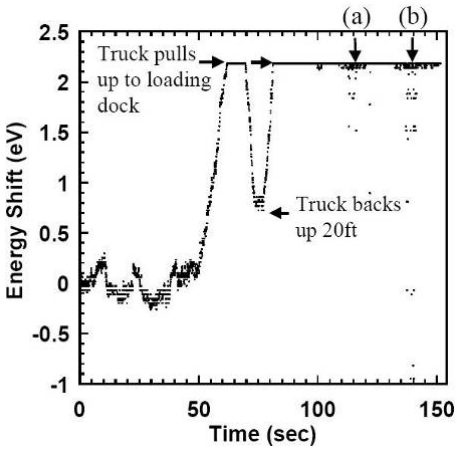



Measured magnetic field from a power bus located 3 feet below the floor. The bus was rerouted.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)



## Spectrometer Sensitivity



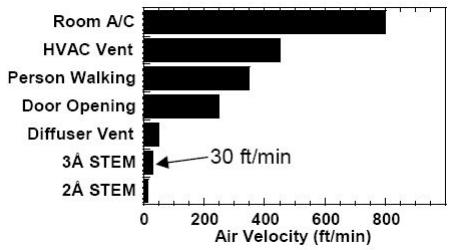

Measured deflection of zero loss peak due to truck at loading dock 20 feet from column.

1 eV deflection seen for each liter of engine capacity.

At 50 feet, spectrometer ceases to function as a truck detector.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)

## Airflow

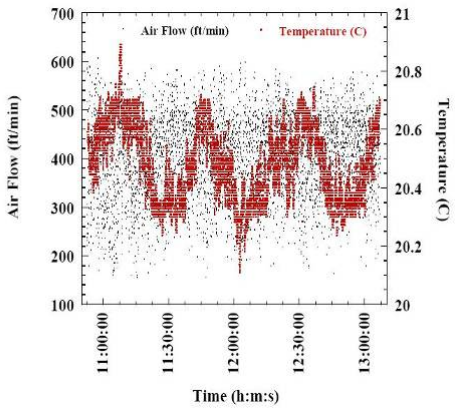



Airflow across a column must be 15 ft/min or less for <0.2 nm resolution STEM work.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)



## Airflow and Thermal Fluctuations




400 ft/min airflow corresponds to 96 air changes per hour.

Heat load in room determines air flow rate:

$q=mC_p\Delta T$

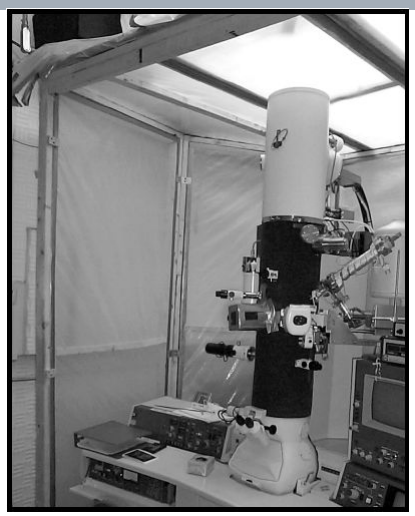
q=Heat load
m=mass of air moved
Cp= Specific heat of air
ΔT=Temperature gradient

Heat load was about 4kW giving temperature gradient of ~3 degrees.

Most of heat load from power supply and electronics.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)

## "Gazebo" Shielding



Gazebo design greatly stabilized beam fluctuations in the 1-10 sec time frame.

Further wrapping of column with bubble wrap or neoprene improves temperature stability.

David Muller and John Grazul, *Journal of Electron Microscopy* **50(3)** : 219-226 (2001)



## Pressure Fluctuations

Sample under vacuum (~1x10-6Pa) while sample holder is at ambient (~105 Pa)

Changes in the room pressure causes a deflection of 0.1 nm/Pa

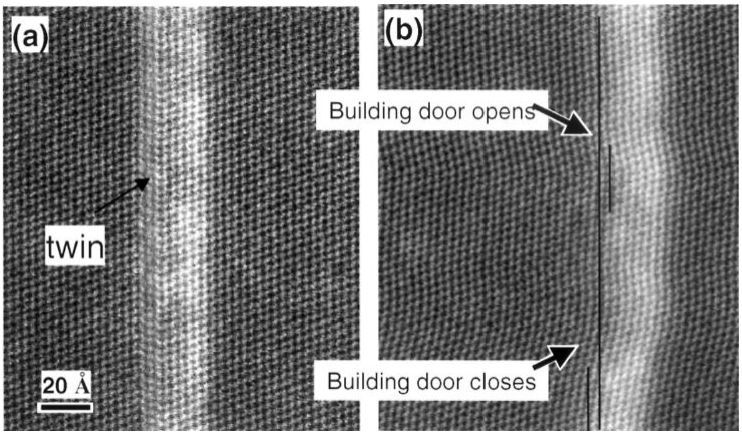
In typical building, A/C system overpressures by 10Pa

- Opening and closing of doors changes this

Weather changes cause slow drift

- Typical low pressure cell is 1600 Pa lower than air in front
- This causes 160 nm of drift as the front passes

## Image Shift with Door Opening

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## Facility Design – Network

Newest generation of TEMs are all PC driven

Many use client-server architecture

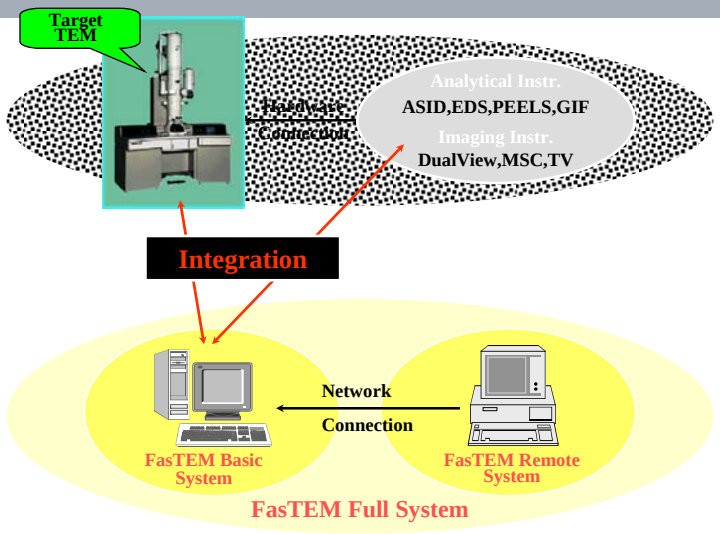
Remote microscopy is becoming more important

- Instrument sharing
- Instrument isolation
- Remove operator from microscope environment

Network requirements for remote microscopy

- Fast
  - LAN:10BaseT, 100BaseT
  - WAN: 1Mbps or more (T1 T2 T3...)
- Secure
- Internet2 is ideal

## Remote Microscopy - FasTEM






## Compensations After Construction

### Field cancellation systems

- Passive
- Active
- Can only correct one point on microscope column
  - Must decide between imaging and spectroscopy

### Vibration dampening systems

- Passive
- Active
- Both most effective at low frequencies (10Hz and below)
  - Most common vibrations are from AC motors (30Hz, 60Hz and 120Hz)

### Shielding

- Mu-metal

### Acoustic dampening

## Summary

### Facility location

- Close enough to loading dock, but not too close
- Away from airports, train lines, highways
- Away from power lines, power stations

### Room location

- Ground floor or basement
- Away from loading dock, storeroom
- Away from elevators
- Away from interfering laboratory equipment
- Decoupled from rest of facility



## Summary

### Room design

- Separate instrument from auxiliary equipment
- Vibration dampening if necessary
- Sound dampening
- No hidden or buried cables
- Minimize airflow over instrument
- Stable temperature
- Stable pressure
- Lighting

## The (Nearly) Ideal TEM Facility