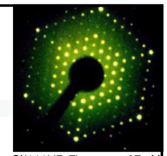
# Chapter 9




## Electron mean free path

## Microscopy principles of SEM, TEM, LEEM

9.1 Electron Mean Free Path

9.2 Scanning Electron Microscopy (SEM)

-SEM design; Secondary electron imaging; Backscattered electron Imaging

9.3 Transmission Electron Microscopy (TEM)

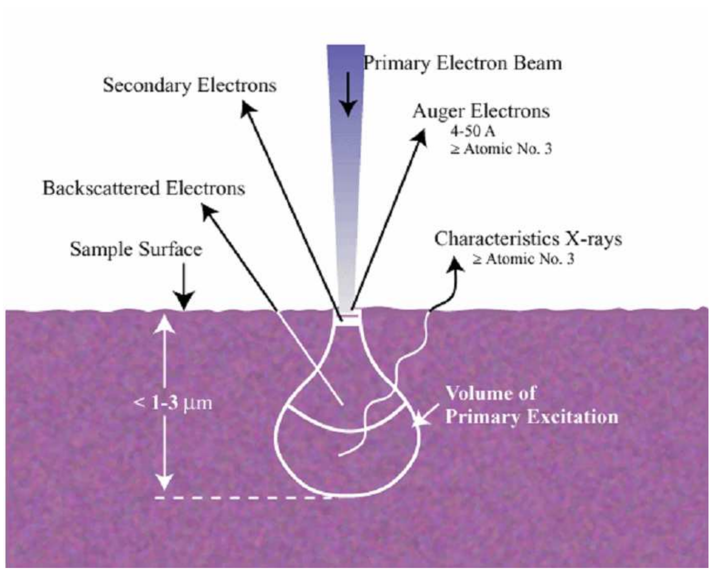
- TEM/STEM design; High Angle Annular Dark Field (HAADF)

9.4 Low Energy Electron Microscopy (LEEM)

References:

1) L. Reimer, "Scanning Electron Microscopy - Physics of Image Formation and Microanalysis", 1985.
2) R.E. Lee, "Scanning electron microscopy and X-Ray microanalysis, 1993
3) D.P. Woodruff, T.A. Delchar, "Modern Techniques of Surface Science", Chapter 2 and
4) K. Kolasinski, "Surface Science: Foundations of Catalysis and Nanoscience. 2nd ed.; 2008; pp.84-91, 107-108
5) LEEM: http://www.research.ibm.com/leem/#item2

## Electron beam interactions with the sample

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## 9.1 Electron Scattering

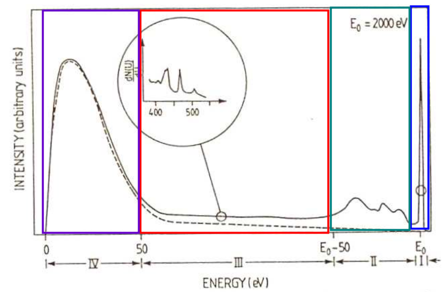
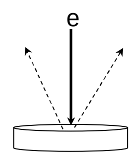


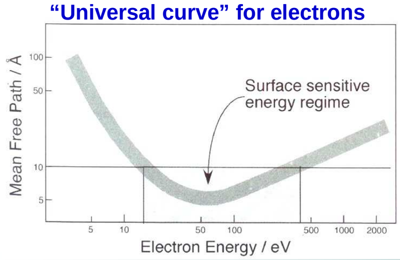

Fig. V.2. Qualitative large-scale overview of the energy distribution of electrons emitted from a surface which is irradiated by an electron beam of primary energy $E_0$.

Electron diffraction and microscopy:

Elastic backscattered $e^-$, ~ few % at 100eV




**"Universal curve" for electrons**




Short inelastic mean free path for electrons means that elastic scattering of electrons is **very surface sensitive**

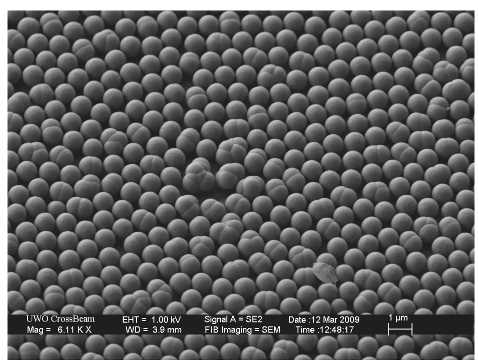


## 9.2 Scanning Electron Microscopy (SEM)

**Scanning electron microscopy (SEM)**

- topology, morphology, chemical information (BSE and EDX)

- 0.5-1000keV electron energy
- field of view 0.1 - 100 µm
- 5 nm resolution in plane
- Magnification 10x – 300,000x
- Typical operating pressure <1atms
- Non-destructive nature: though sometimes electron beam irradiation can cause sample damage

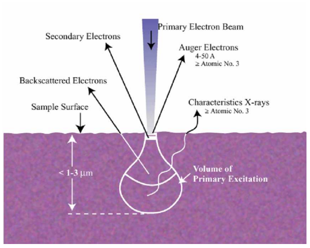



by Eric Barbagiovanni

- **Advantages**: surface, common technique
- **Disadvantages**: vacuum compatibility; coating non-conductive specimens, typical cost: US$50,000 to 300,000

## Electron beam solid interaction

**Secondary electrons (SEs)**: are produced by the interactions between energetic e's and weakly bonded valence e's of the sample




**Backscattered electrons (BSEs)**: are primary e's leaving the specimen after a few large angle elastic scattering events

**Auger electron:** incident $e^-$ kicks out an inner shell $e^-$, a vacant $e^-$ state is formed; this inner shell vacant state is then filled by another $e^-$ from a higher shell, and simultaneously the energy is transferred to another $e^-$ that leaves the sample

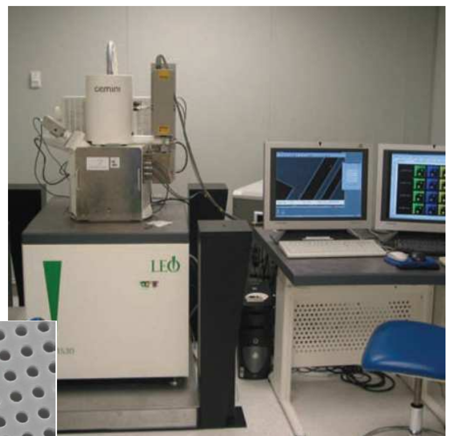
**Characteristic X-rays**: emitted when a hole is created in the inner shell of an atom in the specimen due to inelastic $e^-$ scattering, as it can recombine with an outer shell $e^-$ (EDX)

**Cathodoluminescene (CL)**: light emission arising from the recombination of e-h pairs induced by excitation of e's in the valence band during inelastic scattering in a semiconducting sample

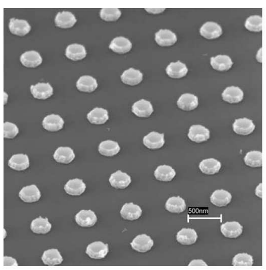


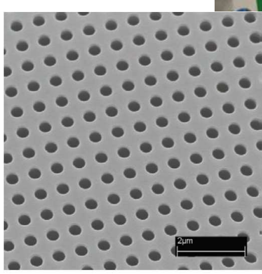
## SEM/e-beam lithography in the Nanofab

The e-beam lithography system (right) is a LEO 1530 field emission scanning electron microscope (FE-SEM) fitted with a laser interferometer controlled stage (middle right).



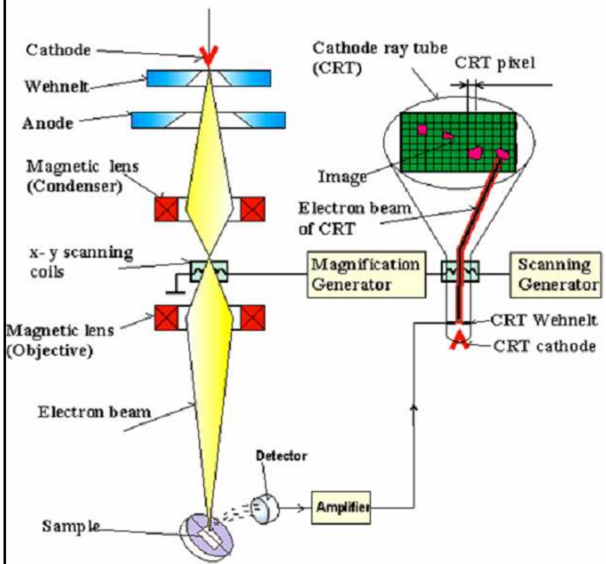

The micrograph (bottom right) shows a square array of 300nm holes on 700nm pitch written in PMMA on Si. Also shown is an array of Cr dots on Si patterned by e-beam lithography and liftoff (below).




http://www.uwo.ca/fab/

## Schematic diagram of SEM




**Filament (cathode)**: free e's by thermionic emission of W, $LaB_6$

**Wehnelt Cylinder:** focuses the e-beam and stabilizes beam current

**Anode Plate**: maintains the HV difference between the anode and the cathode, and accelerates the free electrons down the column

**Condenser Lens**: reduces the diameter of the electron beam to produce a smaller spot size

**Scan Coils:** electromagnetically raster the e-beam on the surface

**Final Objective Lens:** focuses the e-beam on the surface; the smallest spot is about 5 nm (~ 1nm with a FI source)

**Detectors**: within the scope chamber, but not part of the column are the detectors

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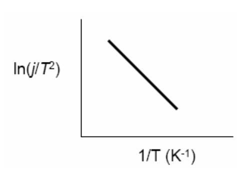
## Electron Sources: 1. Thermionic Emission

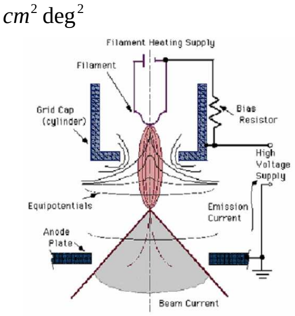
**Thermionic emission** occurs when sufficient heat is supplied to the emitter so that e's can overcome the work function, the energy barrier of the filament, $E_w$, and escape from it

- **Richardson's Equation**: (derivation – aside)
  Current density, *j*: $j = A_o(1-\bar{r})T^2 \exp(-\frac{e\phi}{kT})$

  *r* = reflection coefficient; $A_o = \frac{4\pi mek^2}{h^3} = 120.4 \frac{Amp}{cm^2 \deg^2}$

- **Richardson plot**:
  ln(j/T²) *vs* 1/T ⇒
  ⇒ straight line



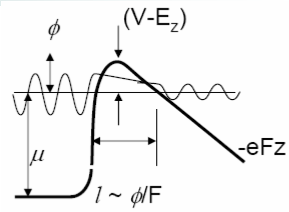




Chapter 9

# Field Electron Emission

- Electron tunneling through low, thin barrier
  - Field emission, when $F > 3 \times 10^7$ V/cm ~ 0.3 V/Å
- General relation for electron emission in high field:

$$j = e\int_0^{\infty} P(E_z, F)\, v(E_z)\, dE_z$$




- P is given by WKB approximation

$$P = const \times \exp\left( -\frac{2^{2/3} m^{1/2}}{\hbar} \int_0^{l} (V - E_z)^{1/2}\, dz \right)$$

- If approximate barrier by triangle: $\int \sim \frac{1}{2}\phi^{1/2}\frac{\phi}{F} \sim \frac{1}{2}\frac{\phi^{3/2}}{F}$

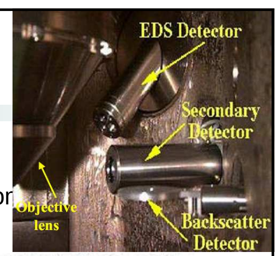
$$P = const \times \exp\left( -\frac{2^{2/3} m^{1/2}}{\hbar} \frac{\phi^{3/2}}{F} \right)$$

- Fowler – Nordheim eqn, including potential barrier:

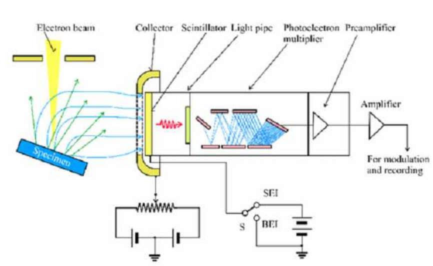
$$j = 1.54 \times 10^{-6} \frac{F^2}{\phi} t^2(y) \exp\left\{ -6.83 \times 10^7 \frac{\phi^{3/2} f(y)}{F} \right\}; \text{ where } \quad y = \frac{e^{3/2} F^{1/2}}{\phi}$$



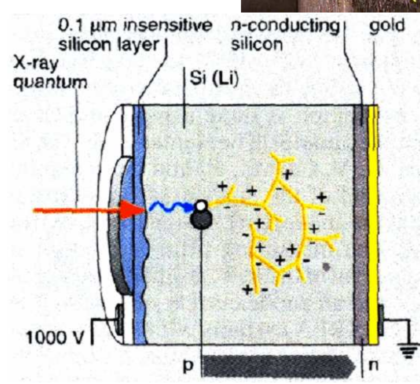
# SEM Detectors




Everhart-Thornley (E-T) detector

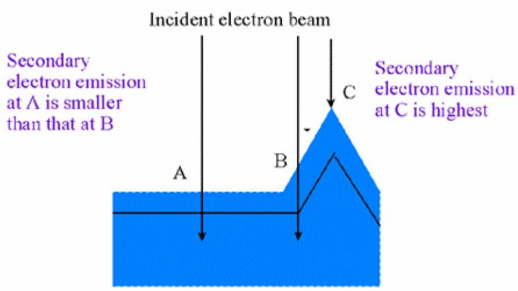


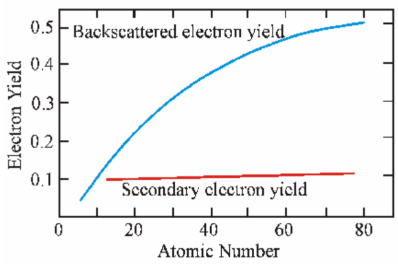

EDX spectror



## Contrast of secondary electron micrograph

Contributions from (a) sample topography and (b) compositional contrast




**Q:** Why do the backscattered electron micrographs, rather than secondary electron micrographs reveal the compositional contrast?

## 9.3 Transmission Electron Microscope (TEM)

Multipurpose machine!

**Elastic scattering:**

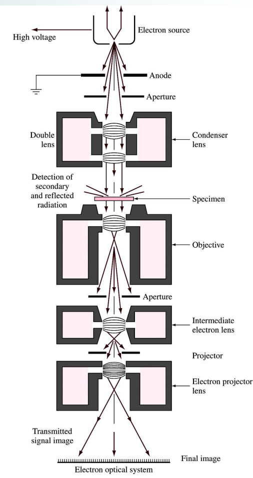
• atomic structure (lattice parameters, orientation) (~1pm)

• microstructure and defects (~1nm-1µm)

**Inelastic scattering**

• Chemistry EDX (~ 1nm) and EELS

- 100-3000keV electron energy
- resolution in plane 1nm (TEM) 0.6Å (HRTEM, current record)
- **Advantages**: atomic resolution and depth resolution
- **Disadvantages**: difficult sample preparation, need UHV

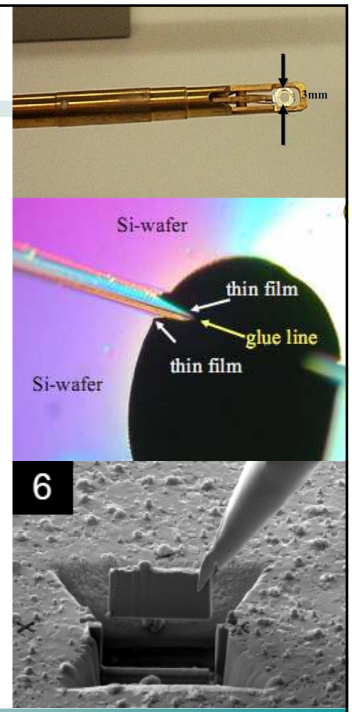



http://www.thebiotron.ca/modules-imaging.php

http://www.brockhouse.mcmaster.ca/facilities/tem.html

http://videolectures.net/kolokviji_gloter_tem/
http://www.ccmr.cornell.edu/igert/modular/docs/1_electron_microscopy.pdf
http://www.rodenburg.org/RODENBURG.pdf

## Sample preparation



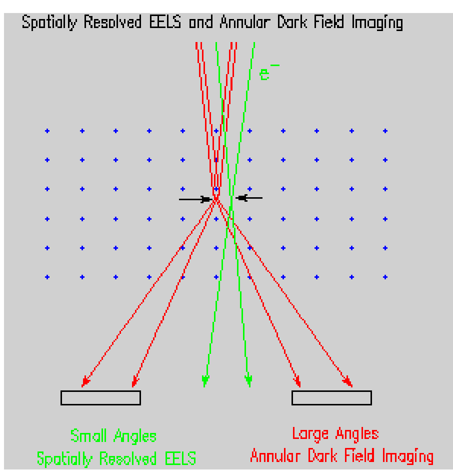

**Cross-section preparation (1-1.5 days)**

- gluing face-to-face
- cutting a slice
- mechanical polishing down to a thickness of 30µm
- ion milling until perforation

**FIB (see Appendix I):** a bit faster…



## STEM- High Angle Annular Dark Field (HAADF)




Detect only scattering at high angle, primary sensitive to the atomic number and thickness

"vacuum": black

High Z elements: bright

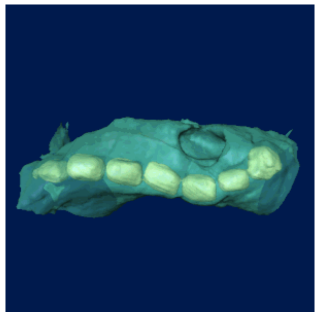
Low Z: grey…

http://www.research.ibm.com/atomic/batson/adfstem.htm

## HAADF STEM Tomography

HAADF images show little or no diffraction effects, and their intensity is ~ $Z^2$.

This imaging technique proves ideal for tomographic reconstruction as it generates strong contrast that has a fully monotonic relationship with thickness.

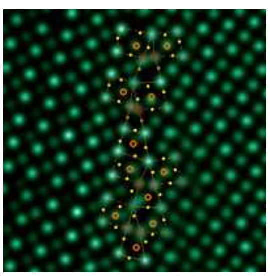


Magnetite crystals in bacteria strain MV-1, in this preperation the cell is preserved around the crystals.

The tilt series was acquired from +76 degrees to - 76 degrees; each crystal is ~60nm long.

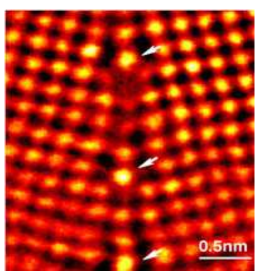
http://www-hrem.msm.cam.ac.uk/research/CETP/STEM_Tomo.html

## Applications of HRTEM



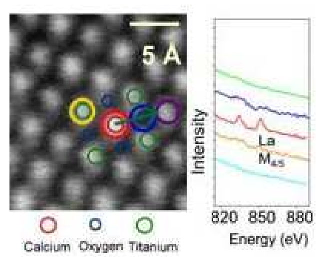
Direct Determination of Grain Boundary Atomic Structure In $SrTiO_3$

McGibbon MM et al., *Science* **266,** 102 (1994)




Impurity-Induced Structural Transformation of a Grain Boundary

Y. Yan et al, *Phys. Rev. Lett.* 81, 3675 (1998)




Single Atom Spectroscopy

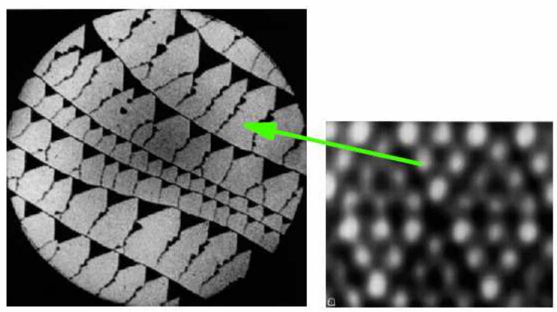
M. Varela et al., *Physical Review Letters* **92**, 095502 (2004)

**http://stem.ornl.gov/highlights.html**

## 9.4 Low Energy Electron Microscope (LEEM)

**LEEM history**

- 1962 Invention by Ernst Bauer
- 1985 Operational LEEM instrument (Telieps and Bauer)
- 1991 IBM LEEM-I (Tromp and Reuter)
- 1998 IBM LEEM-II
- 2006 SPECS FE-LEEM P90



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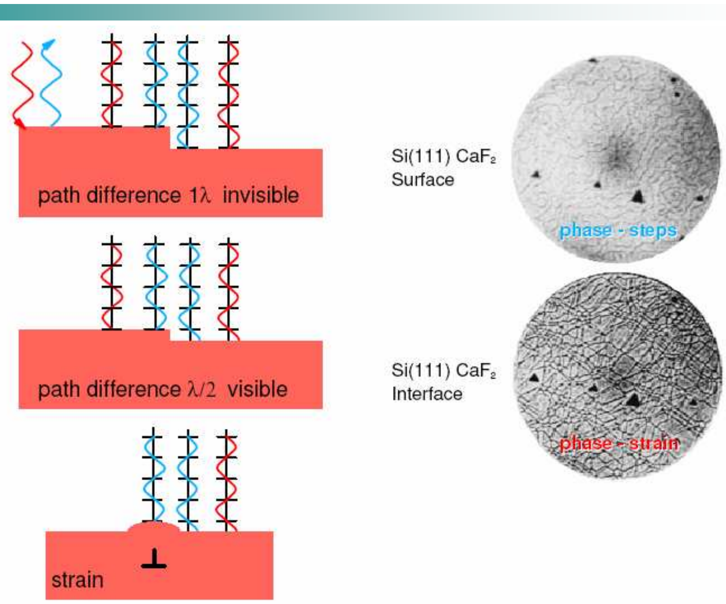
## Phase contrast

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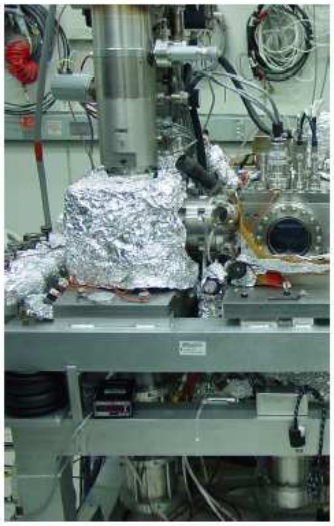
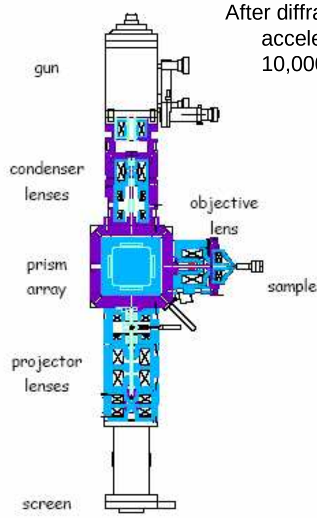
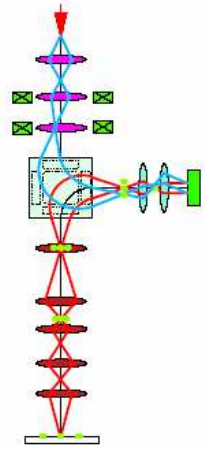
# IBM LEEM II



Surf. Reviews and Lett. 5 (1998) 1189




After diffraction, electrons are accelerated from ~ 1 eV to ~ 10,000 eV

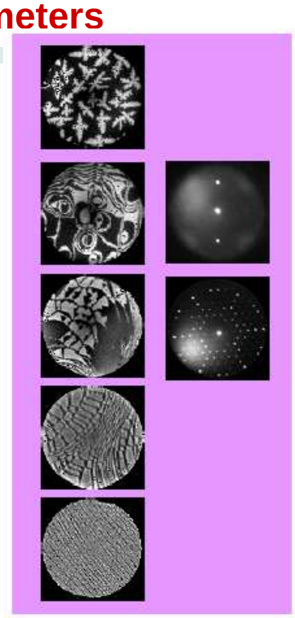




# LEEM operating parameters

- 0 - 100 eV electron energy
- field of view 1 - 100 µm
- 5 nm resolution in plane
- vertical resolution: atomic steps, 0.1 nm
- in situ growth, etching
- RT – 1200°C

⇒ extremely useful tool to study crystal growth in situ



http://www.research.ibm.com/leem/

* From R.M Tromp