

## NanoCourses 2004, Section 4

# Practical Thin Film Analysis

## *What is It?*

Introduction  
by  
Lynn Rathbun

Presented by the  
**CNF** Technical Staff  
for the education of CNF Users,  
Potential Users, and Industrial Sponsors



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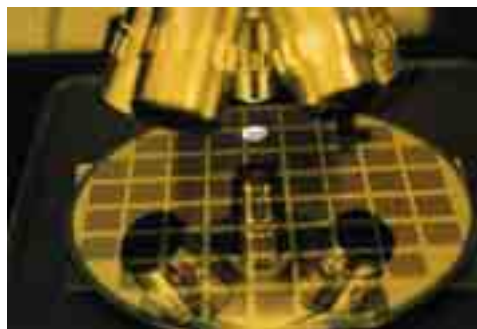


Thin Film Analysis, page 1



## Why ?

- Process Development
- In Process Monitoring
- Process Characterization / Maintenance
- Failure Analysis



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Thin Film Analysis, page 2



## What Do We Need / Want to Know

- **Electrical**
  - Resistivity
  - Contact Resistance
  - Barrier Height
- **Mechanical**
  - Stress
  - Adhesion
  - Optical
- **Chemical/Elemental**
  - Composition
  - Bonding
  - Valence
- **Index of Refraction**
  - Reflectivity
  - Adsorption
- **Physical**
  - Crystal Structure
  - Grain Size
  - Orientation
  - Texture

## Measurements and Techniques

- **Specialized Measurements**
- **Specialized Techniques**
- **Specialized Instruments**
  
- **Many Many Available**

## Planning

- **Design and Plan for Analysis and Characterization**
- **Don't analyze unless you expect to learn something**
- **Variety of Tools Available**
  - **Cornell**
  - **Outside**

## Microanalysis

- **Analysis of small volumes**
  - **High spatial resolution in at least one dimension**
- **Not necessarily the same as, or compatible with trace element analysis**

## Particle in / Particle out

- “Excite” sample
  - Generally a localized small volume
- “Look” at stuff that comes out
  
- Ions (high or low E, various kinds)
- Electrons (high or low E)
- Photons (various energies)
  
- Reflected, scattered, re-emitted, etc.



## Lots of Acronyms

- SEM
- EMPA
- AES
- SAM
- RBS
- SIMS
- ISS
- .....
- .....



## The Process

- Measure something
- Calculate what we want from the something we measure
  - Model
    - Assumptions
    - Limitations



## Cautions

- What you want to know *vs.* what you really measure
- What you measure *vs.* what you think you are measuring
- The authoritative “power” of computer generated / analyzed data
- Important that you understand SOMETHING about the analytical measurement



## **Rathbun's Laws of Analysis**

**If you ask the wrong question,  
you will get the wrong answer.**

**If you try to answer the right question  
with the wrong technique,  
you will also get the wrong answer.**



## **Optical Microscopy**

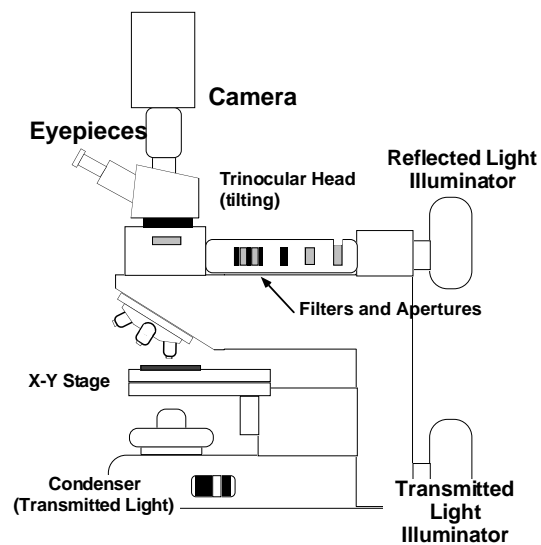


## The Semiconductor Inspection Microscope

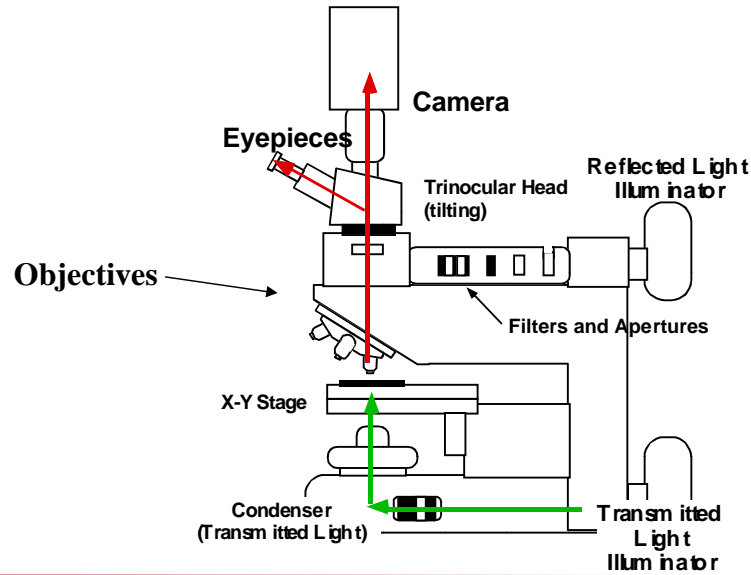
- It bears some relationship to the thing you used in high school biology...but not much
- Sophisticated optical / mechanical instrument
- Simple to use badly
- Somewhat more difficult to use correctly
- Few people in CNF ever use all its features



## Inspection Microscope-Parts



## Transmitted Light



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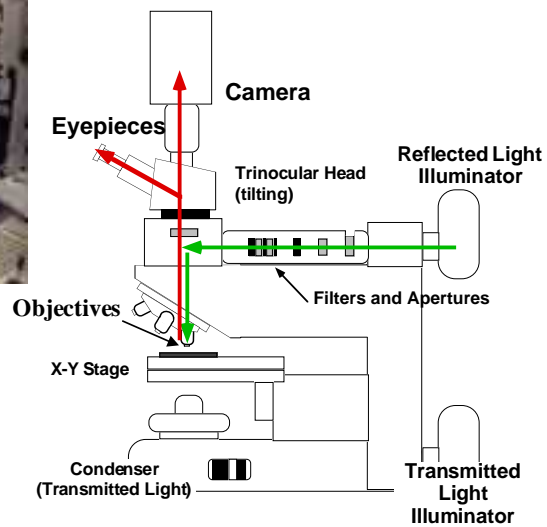
Thin Film Analysis, page 15

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## Reflected Light



Most Common



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# Magnification

- **Total Magnification Product of:**

- Objective (5x - 250 x)
- Body (1x - 2x)
- Eyepiece (10 x- 15 x)
  - Camera

- **Magnification vs. Resolution**

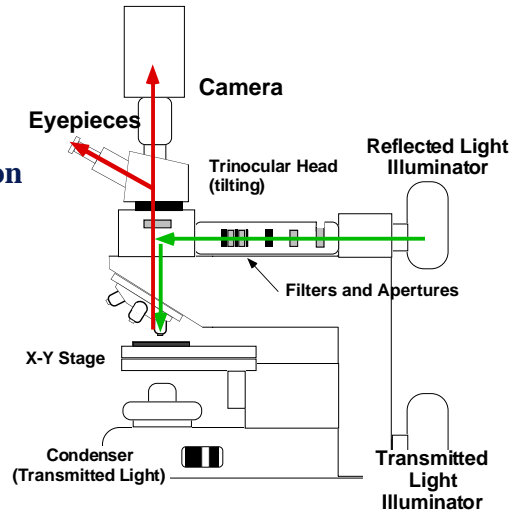
- Useful magnification
- Empty magnification

- **Good Microscope**

- 1000X

- **Great Microscope**

- 2500X



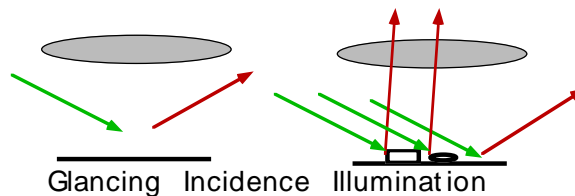
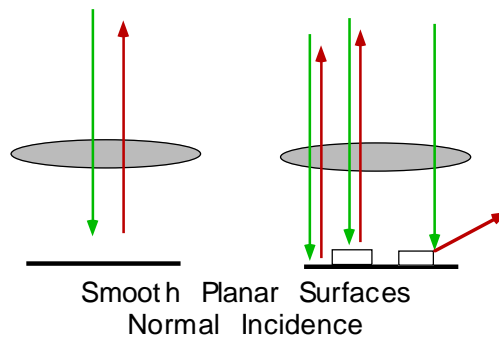
# Light Scattering at Surface

- **Plane surface**

- Angle in = Angle out

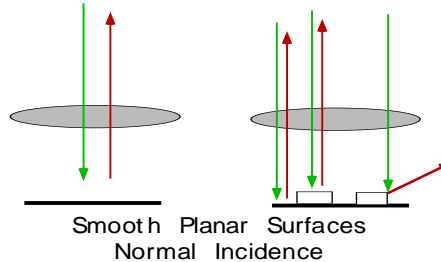
- **Edges reflect differently**

- **For glancing illumination, only edges will reflect up, perpendicular to the surface**

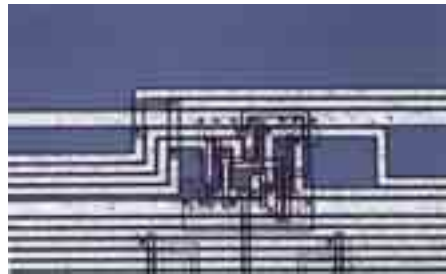


## Bright Field

- Light incident more or less perpendicular
- Flat planar surfaces reflect well
  - Appear bright
- Edges and slopes reflect to the side and appear dark
- Most common mode for general use



Bright Field, reflected light is the most common arrangement



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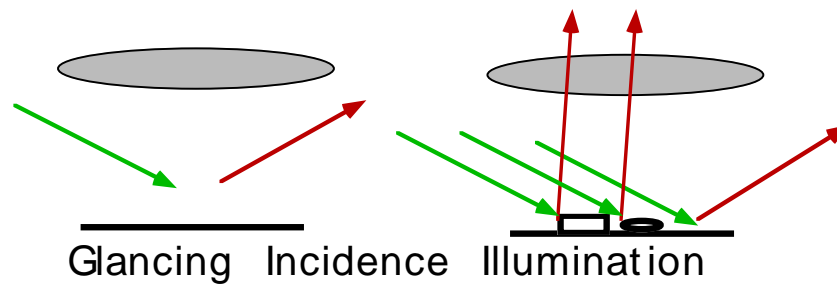


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## Dark Field

- Angular illumination
- Plane surface reflect away from lens
  - Most of sample is DARK
- Sharp edges will scatter some light into lens
  - Edges and dirt sparkles
  - Starry night



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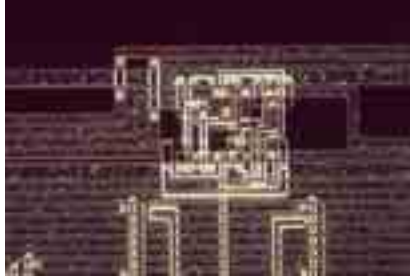


Thin Film Analysis, page 20

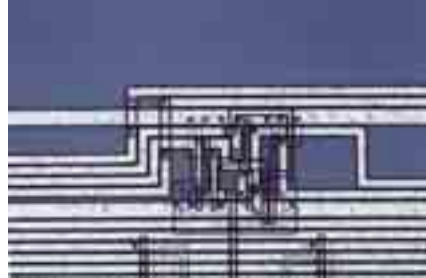
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## Dark Field vs. Bright Field

Dark Field



Bright Field

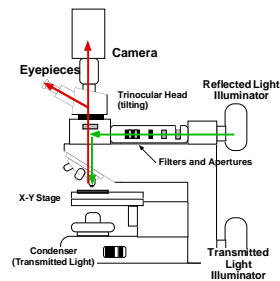


## Dark Field-Defect Detection

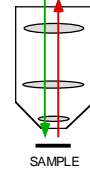


## BF/DF Objectives

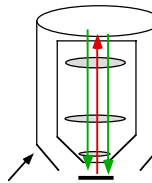
- How do we get the oblique illumination required for DF
- Combination BF/DF objective
  - Coaxial light paths
- A simple slider in the optical path directs light to the outside, oblique path



Bright Field Illumination through a BF objective

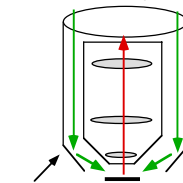


Bright Field Illumination through a BF/DF objective



Mirrored Collar

Dark Field Illumination through a BF/DF objective



Mirrored Collar

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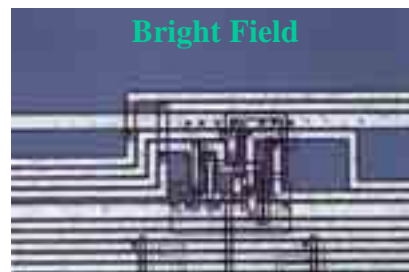


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## Other Imaging / Illumination Modes

- Polarized light
  - Optical index contrast
- Differential interference contrast
  - A false "depth" contrast achieved by interference
  - aka Nomarski
- About half of CNF scopes are set up for DIC / Polarized light



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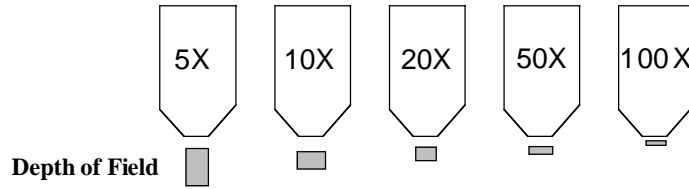
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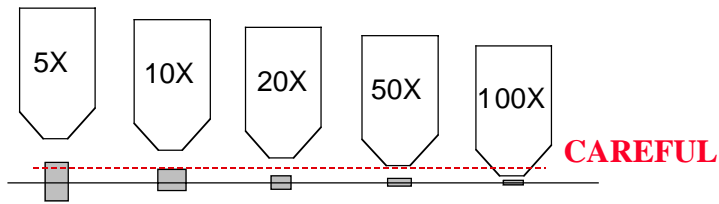
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## Depth of Field / Working Distance / Par-Focality



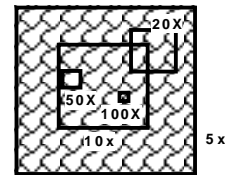
- ❖ High magnification objectives have small depth of field
- ❖ High magnification objectives have short working distance
- ❖ CNF microscopes are Par-focal



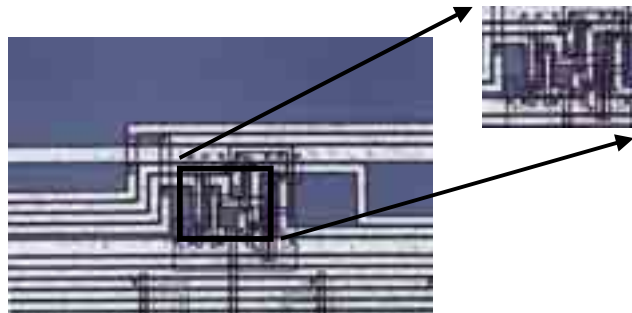
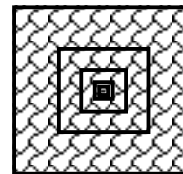
## Par-Centricity

- You would like your sample image not to “move” as you zoom in
- Precision mechanical / optical adjustment
- Most CNF microscopes are par-centric

Non-Parcentric

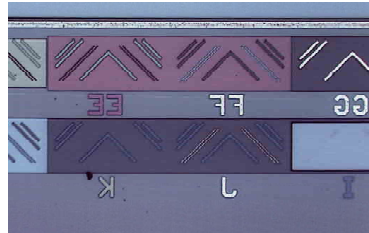


Par-centric



## Upright and Bilaterally Correct

- Left is left
- Right is right
- Top is top
- Bottom is bottom
- Text is legible
- All CNF microscopes give upright/erect images ( I think)



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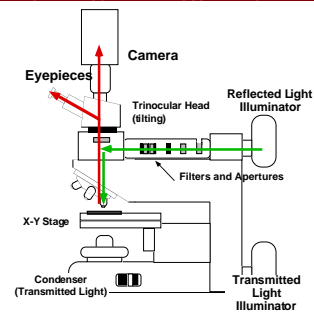


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## Hints

- Use both eyes!!!!
  - Binocular eyepieces
- Eye is part of the optical instrument
  - Wear your glasses
  - High point eyepieces
  - Proper brightness/contrast
- Illumination aperture
  - Amount of light
- Field Aperture
  - Illuminated area



Field Aperture



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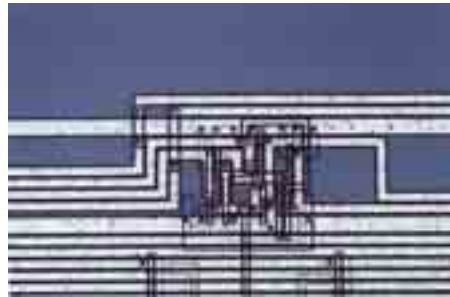
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## Confocal Microscopy

- Confocal microscopy uses optical “tricks” to create a very shallow depth of focus
  - Only features at a specific depth are imaged clearly
- Olympus BX50
  - High intensity light source required

## The Good, the Bad and the Ugly

- Micrograph Options (SEM or Optical)
  - Film
  - Thermal Prints
    - Not publication quality
  - Digital files
- Most pictures I see:
  - Too Dark
  - Too Close
  - Bad contrast



## Summary

- **CNF has a very good stock of optical microscopes**
  - Leitz Ergoluxs
  - Older Olympus scopes
  - Olympus MX50
  - Olympus BX series
    - confocal
- **They are precision instruments**
- **They are expensive**
  - \$20,000 - \$100,000
- **Variety of imaging modes**
  - Learn how to use





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# Practical Thin Film Analysis *What is It?*

## Scanning Electron Microscopes (SEMs) by Daron Westly

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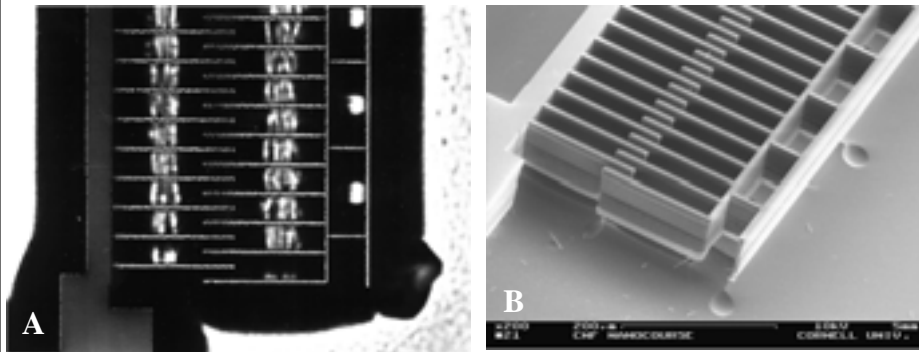


SEMs, page 1



## SEM vs. Light Optical Imaging

### Superior Depth of Focus



MEMS Comb Drive

Figure A: Optical

Figure B: SEM



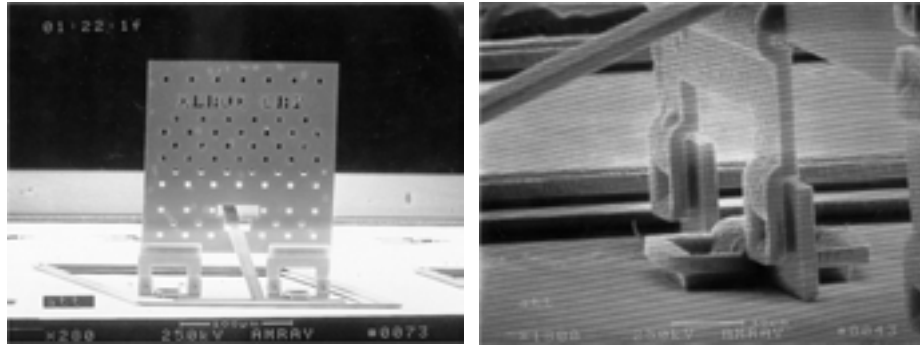
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SEMs, page 2



## SEM: Superior Depth of Field



### MEMs Mirror

by Alex Tran

*Corporate Research & Technology, Xerox Corporation*



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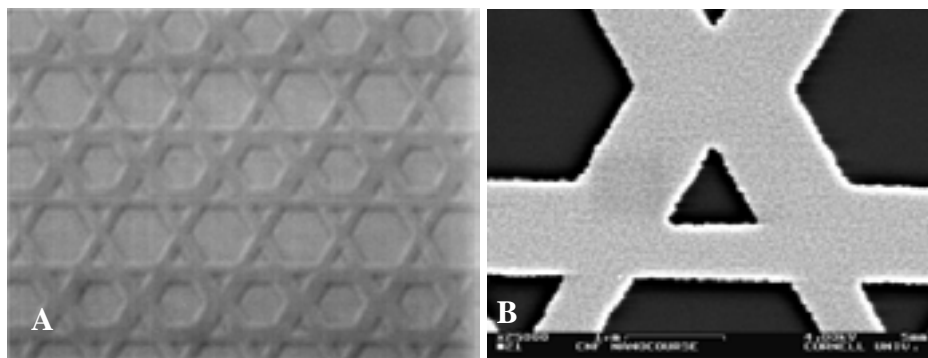


SEMs, page 3



## SEM vs. Light Optical Imaging

### Superior Resolution



### Kagome Superconductivity Structure

Figure A: Optical - 4600x

Figure B: SEM - 25,000x



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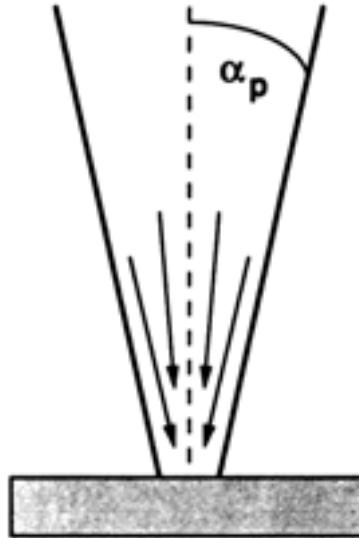


SEMs, page 4



## Depth of Field & Resolution

- Depth of field is dependent on the probe convergence angle:
  - SEM depth of field 1000X Light
- Resolving power scales with the of the illuminating radiation:
  - Light
    - $\lambda = 433\text{-}633\text{ nm}$
  - Electrons
    - $\lambda = 0.009\text{ nm}$  (20keV)
- SEM Mags to 400,000X



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## Hitachi S-4700



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## LEO 982 SEM



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## SEM Imaging

The goal of the instrument is to place a focused beam of primary electrons onto a sample, and to collect secondary electrons from the sample to form an image

### Modes of Operation:

- **High Resolution:**
  - Small Probe Diameter → Low Probe Current ( $I_p$ ) → Poor Signal / Noise (S/N) Ratio
  - Short Working Distance → Less Lens Aberrations
- **High Depth of Field:**
  - Long Working Distance → More Lens Aberrations
  - Small Aperture → Low  $I_p$  → Poor S/N
- **Microanalysis:**
  - High Probe Current → High  $I_p$  → Good S/N → Large Beam Diameter

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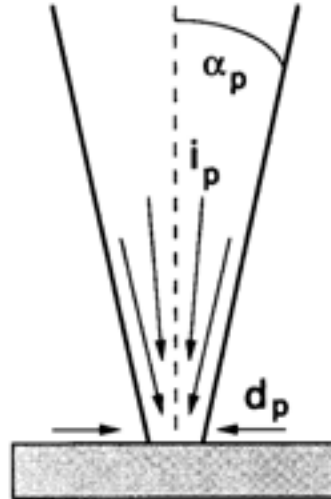


SEMs, page 8

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## Why Learn about Electron Optics?

- Three electron beam parameters determine sharpness, contrast, and depth of field of SEM images:
  - Probe diameter –  $d_p$
  - Probe Current –  $I_p$
  - Probe Convergence Angle –  $\alpha_p$
- You must balance these three depending on your goals:
  - High Resolution
  - Best Depth of Field
  - Best Image Quality and X-Ray Analysis



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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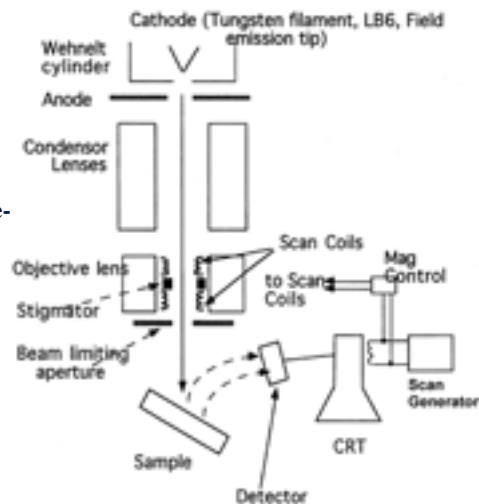


SEMs, page 9



## Electron Optics: Simplified SEM Schematic

- Gun
  - Source of electrons & accelerates electrons to 1-40keV
- Lenses
  - 1, 2, or more lenses to focus and demagnify electron beam
- Stigmator
  - Corrects lens errors
- Beam Limiting Aperture
  - Affects depth of field and  $I_p$



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SEMs, page 10



## Column: Electron Gun

### Thermionic Electron Gun

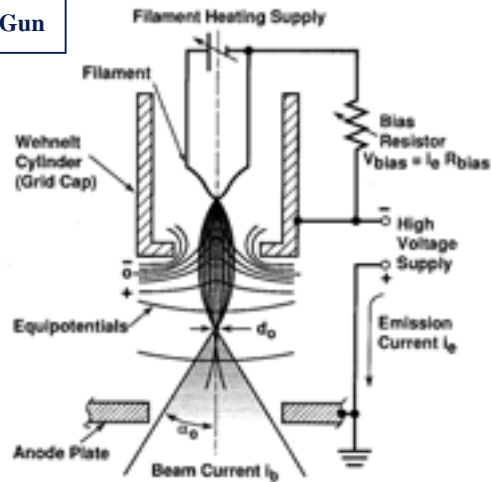


Figure 2.4. Schematic diagram of the conventional self-biased thermionic (triode) electron gun (adapted from Hall, 1966).

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



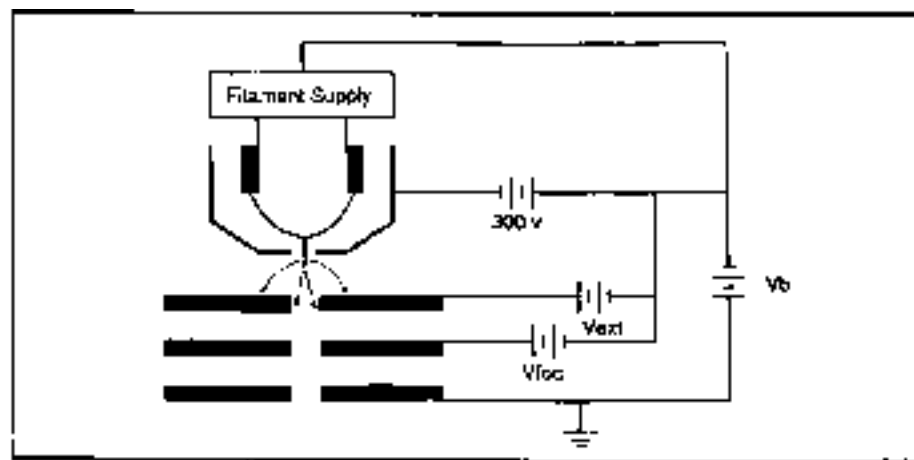
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SEMs, page 11



## Column: Electron Gun



### Thermal field emission source and electrostatic gun lens



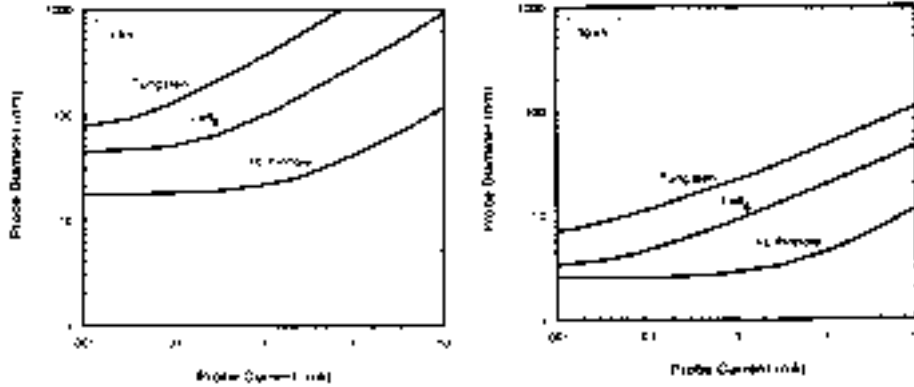
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SEMs, page 12



# Electron Sources



Probe Diameter vs. Probe Current

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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SEMs, page 13



# Column: Electron Gun

Comparison of Electron Sources at 20 kV

Source	Brightness	Lifetime	Source size	Energy spread $\Delta E$	Beam current Stability	References
Tungsten hairpin	$10^5$ A/cm <sup>2</sup> sr	40–100 h	30–100 $\mu$ m	1–3 eV	1%	a,b
LaB <sub>6</sub>	$10^6$	200–1000	5–50 $\mu$ m	1–2	1%	b,c
Field Emission						
Cold	$10^8$	>1000	<5 nm	0.3	5%	d,e
Thermal	$10^8$	>1000	<5 nm	1	5%	e
Schottky	$10^8$	>1000	15–30 nm	0.3–1.0	2%	e

<sup>a</sup> Haine and Coslett (1961).

<sup>b</sup> Troyon (1987).

<sup>c</sup> Broers (1974).

<sup>d</sup> Crewe et al. (1971).

<sup>e</sup> Tuggle et al. (1985).

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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## Column: Electron Lenses Produce a Small Spot

- **Beam diameter at crossover ( $d_0$ ) in the e-gun too large to generate a sharp image:**
  - Thermionic source size: 5 – 100 $\mu$ m
  - Field emission source size: 5 – 30nm
- **Lenses reduce diameter (de-magnify) of source of electrons and place small focused beam (1nm - 1 $\mu$ m) on specimen:**
  - Demagnification:
    - Thermionic source  $\leq$  10000
    - Field emission source 10 – 100



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SEMs, page 15



## Column: Electron Lenses

- **Condenser Lens: 1 or more Lenses**
  - Determines the  $I_p$  that impinges on the sample
  - Higher  $I_p$  – larger spot size – lower resolution – better S/N
  - Knob labeled spot size, condenser, C1, or resolution
- **Objective Lens: Final Lens**
  - Focuses the beam by controlling the movement of the probe crossover along the optic-axis (Z-axis) of the column
  - Knob labeled focus or objective
  - Pseudo working distance (WD) meter
  - The design of the lens incorporates space for the scanning coils, the stigmator, and the beam-limiting aperture

**Lens aberrations that enlarge the final probe size increase rapidly with focal length (WD). So for high-resolution work keep the WD short.**



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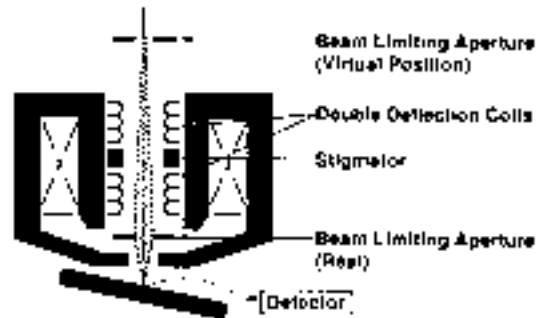
SEMs, page 16





## Column: Objective Lens

- **Stigmator:** A device that applies a weak supplemental field to make the lens appear symmetrical to the electron beam
- **Method:** Try to choose a “round” object
  - Adjust objective lens to best focus
  - Adjust x-stigmator
  - Focus
  - Adjust y-stigmator
  - etc



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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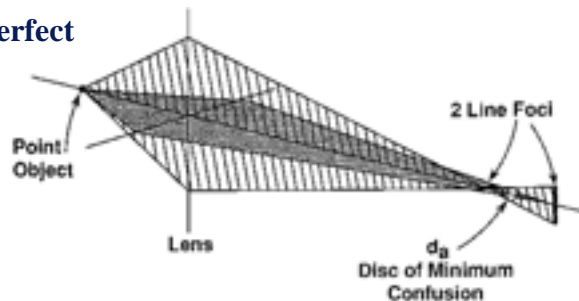


SEMs, page 17



## Column: Stigmator

- Lenses do not have perfect rotational symmetry
- **Causes:**
  - Machining errors
  - Inhomogeneities in the iron of the lens
  - Asymmetry in its windings
  - Dirty apertures
- For example, if a lens has elliptical rather than circular symmetry, electron focuses at 2 separate points at right angles to each other, thus enlarging beam diameter



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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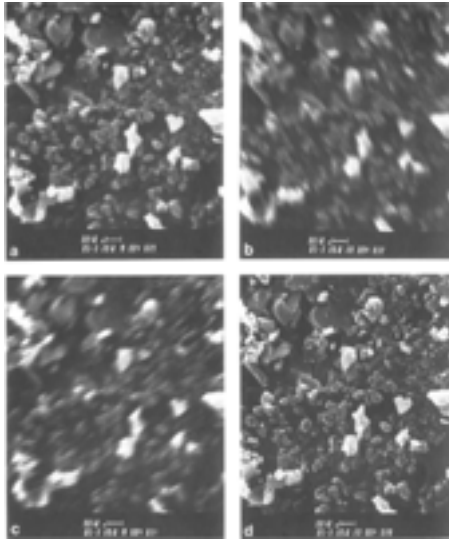


SEMs, page 18



## Astigmatism: IMPORTANT!

- Effect can be recognized by stretching of the image in two perpendicular directions, when the objective lens is underfocused and then overfocused
- At exact focus — stretching vanishes



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al., Plenum Press)

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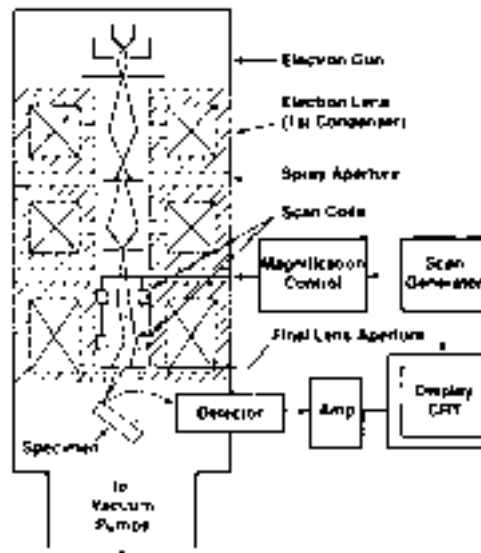


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## Scan System: Image Formation

- CRT & e-beam scanned by single scan generator
- Scan Coils
  - 2 Pairs
  - 1<sup>st</sup> bends beam off optical axis
  - 2<sup>nd</sup> bends beam back onto optical axis



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al., Plenum Press)

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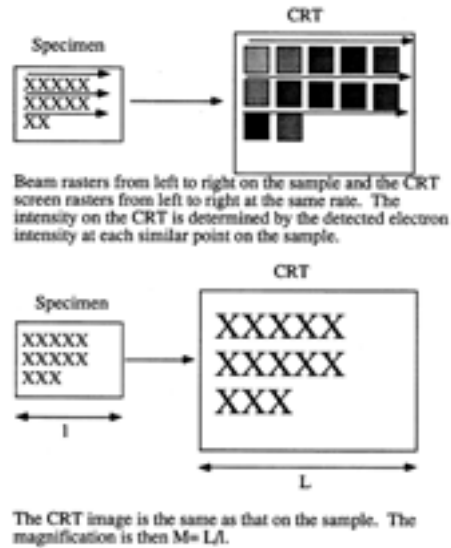
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## Scan System: Image Formation

The number of electrons detected from the specimen determine the intensity changes on the CRT

Magnification is the ratio of the size of viewing CRT to the area scanned on specimen



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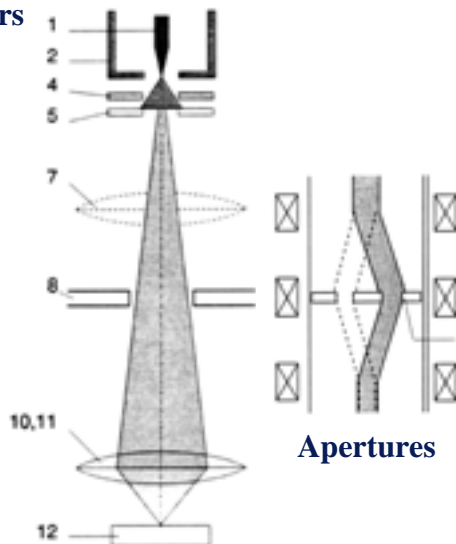
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## Thermal Field Emitter SEM Column: LEO 982

Simpler Column with no crossovers

- Schottky Field Emitter
- Suppressor
- Extraction Voltage
- Aperture
- Condenser lens
- Aperture
- 10,11) Objective Lens
- 12) Specimen



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## Column: Final (Beam Limiting) Aperture

Affects all beam parameters:

- **Large Aperture:**
  - Large  $I_p$  - Good S/N and microanalysis
  - Large  $\alpha_p$  - Poor depth of focus
  - Large  $d_p$  - Poor resolution
- **Small Aperture:**
  - Small  $I_p$  - Poor S/N and microanalysis
  - Small  $\alpha_p$  - Good depth of focus
  - Small  $d_p$  - High resolution



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SEMs, page 23



## IMPORTANT! Aperture Must Be Centered

- For optimum resolution the beam limiting aperture must be centered on the electron optical axis
- If your image is shifting while focusing the aperture is not centered
- While “wobbling” center the aperture – when properly adjusted the image will show no side to side movement when properly centered
  - It will only go in and out of focus
- Check whenever the accelerating voltage or aperture is changed, or when you just cannot get good resolution



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SEMs, page 24



## Depth of Focus at 10 mm WD

- Depth of focus is a measure of the z (vertical) distance over which two objects will still be in focus
- The depth of focus will depend on the working distance and the aperture
- Long working distances, and small objective apertures yield the best depth of field



Depth of Focus at 10 mm working distance

MAG	100x	50x	20x
aperture	80 $\mu$ m	160 $\mu$ m	320 $\mu$ m
1000x	40 $\mu$ m	80 $\mu$ m	160 $\mu$ m
10,000x	4 $\mu$ m	8 $\mu$ m	16 $\mu$ m
100,000x	0.4 $\mu$ m	0.8 $\mu$ m	1.6 $\mu$ m

\* 10 mm WD is a light microscope (10 mm WD)

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)

## Depth of Field: Effect of Aperture Size

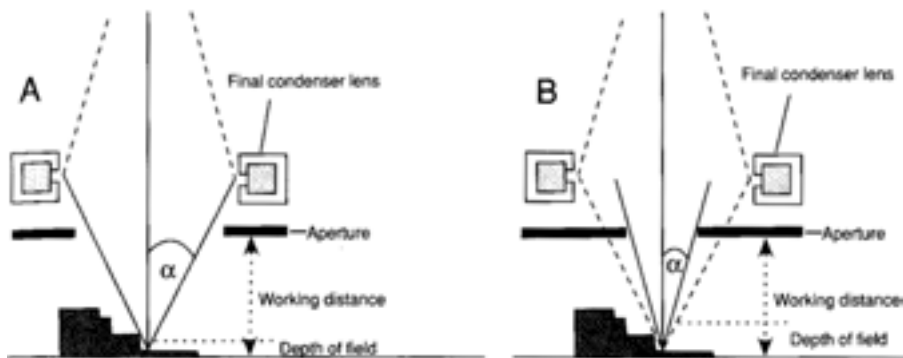


Figure 7-6. Depth of field (the depth that is in focus in the specimen) is increased by using smaller apertures as shown on right.

(Redrawn from Postek et al., 1980)

## Depth of Field: Effect of Working Distance

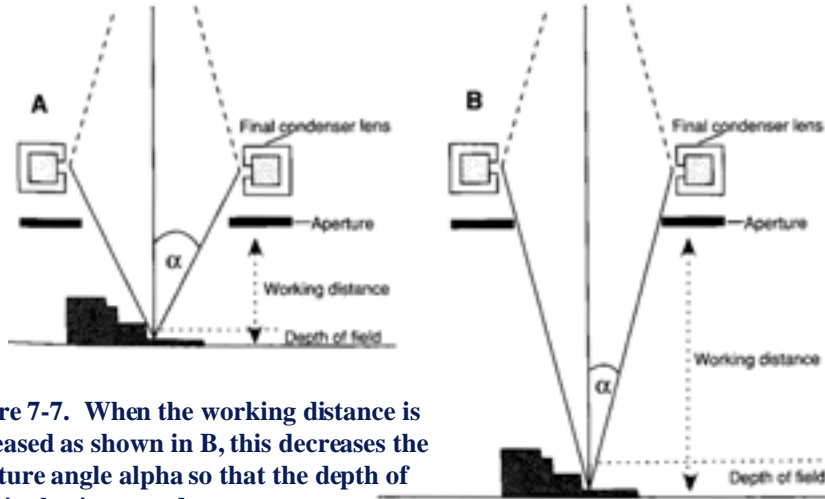


Figure 7-7. When the working distance is increased as shown in B, this decreases the aperture angle alpha so that the depth of field is also increased.

(Redrawn from Postek et al., 1980)



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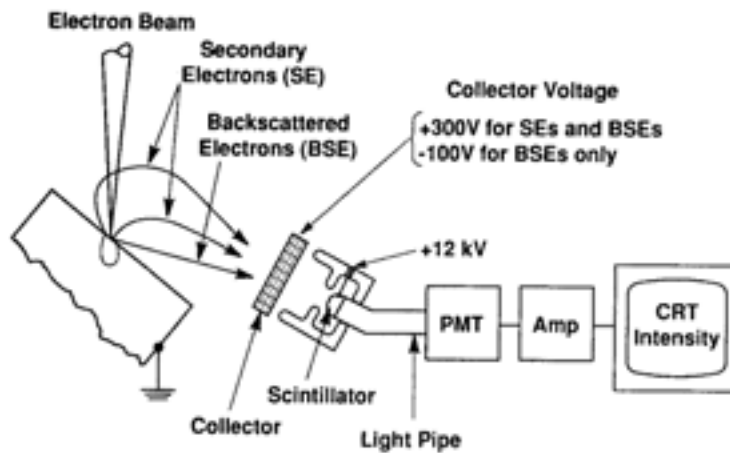


SEMs, page 27



## Electron Detectors: Collect Signals

- Secondary (SE) and backscattered electron (BSE) detectors for imaging



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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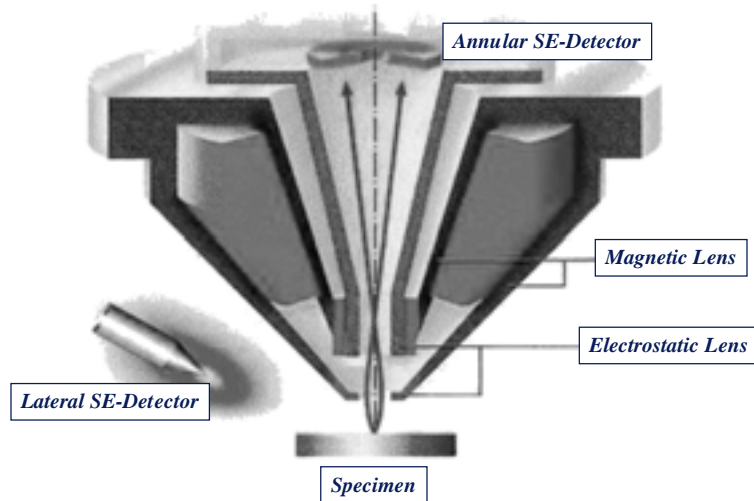


SEMs, page 28



## Dual Secondary Detectors: LEO 982 SEM

### Principle of Secondary Electron Signal Detection



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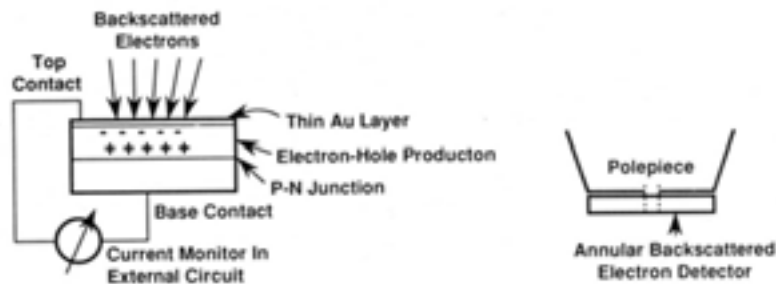


SEMs, page 29

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## Solid-State BSE Detector

- Single annular detector or smaller discrete detectors placed on pole piece of objective lens
- Size permits close proximity to specimen – provides large solid angle for high geometric efficiency
- Sensitive to high energy BSEs only, not SEs



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)

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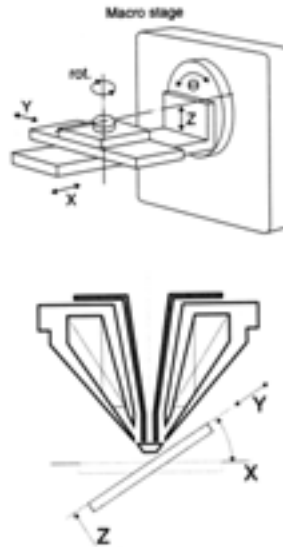


SEMs, page 30

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## Specimen Stage

- **5 Axis of Movement:**
  - X, Y, Z, Tilt, Rotate
- **Manipulation of specimen under beam:**
  - To locate areas of interest, e.g., to tilt to look at edges
  - To improve collection geometry relative to detectors
  - To change Z-height to effect depth of field
- Some are motorized



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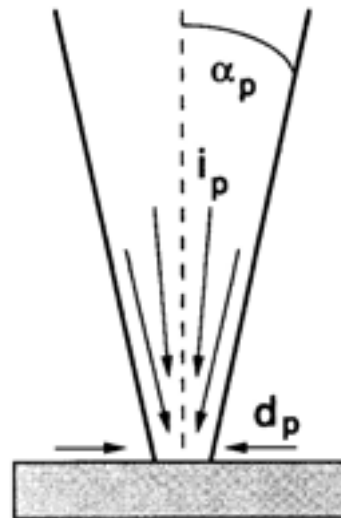


SEMs, page 31

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## Electron-Specimen Interactions

- We have great control of the beam electrons before they reach the specimen, however, once they enter the specimen, the scattering processes control subsequent behavior
- Are signals generated confined to the area of the beam impact on the specimen's surface?
  - If yes – This would result in images of the highest possible resolution
  - Answer is NO! Electron scattering degrades spatial resolution



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SEMs, page 32

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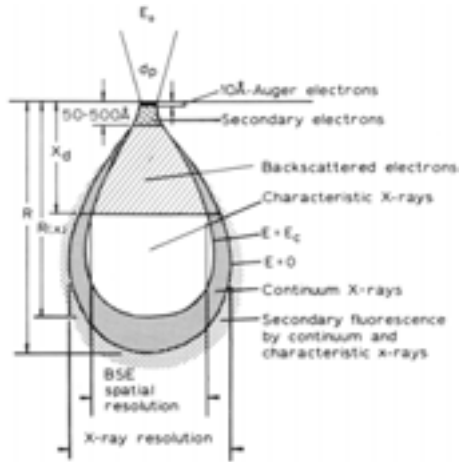


# Electron-Specimen Interactions

Rich variety of interactions that can reveal information on a specimen's:

- Composition
- Topography
- Crystallography
- Electrical potential
- Local magnetic field
- Etc.

- $E_0$ =Voltage of e-beam
- $E_c$ =Critical excitation energy
- $R$ =Range



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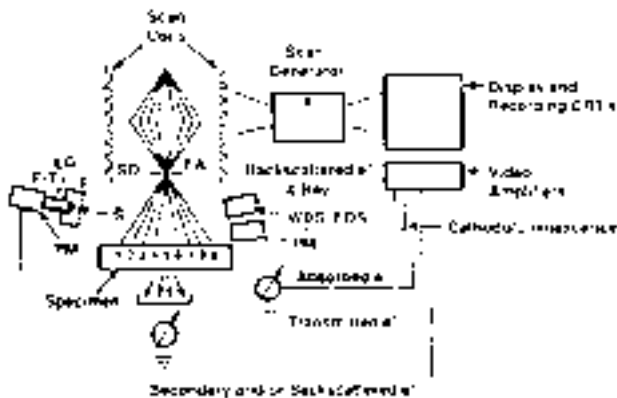
SEMs, page 33



# Signal Detection

- With the proper detector or with specialized instruments you can take advantage of the multitude of signals:

- TEM
- EELS
- SAM
- AES
- EPMA
- WDS & EDS
- Etc...



(From Scanning Electron Microscopy and X-Ray Microanalysis, Joseph I. Goldstein et al. Plenum Press)



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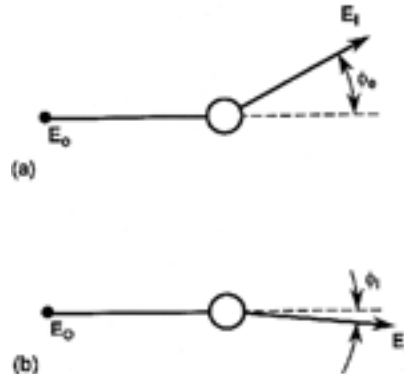


SEMs, page 34



## Electron-Specimen Interactions

- (a) Elastic Scattering
  - BSEs
  - Electron scattered by interaction with atomic nucleus
  - Direction of beam electron changed, but velocity essentially the same
    - $\Phi_e = 0 - 180^\circ$
  
- (b) Inelastic Scattering
  - SEs, x-rays, etc
  - Energy transferred to tightly bound inner shell electrons or loosely bound outer-shell electrons
  - Kinetic energy of beam electron decreases
    - $\Phi_i \leq 0.1^\circ$



(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



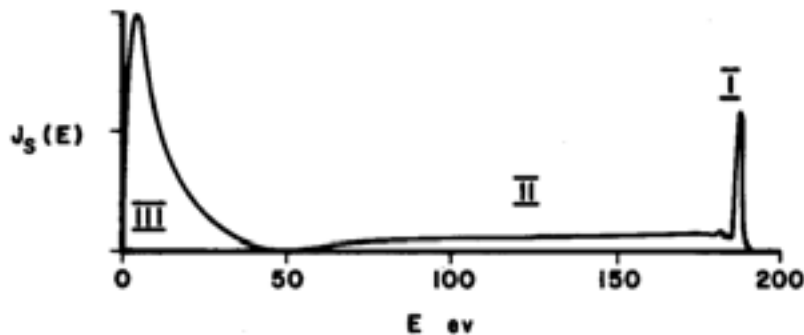
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SEMs, page 35



## Energy Distribution of Emitted Electrons



Energy distribution of secondary electrons.  $J_s(E)$  is the intensity of secondary electrons. Note that the beam energy  $E_0$  is 180 eV. (From Hachenberg and Brauer.<sup>(10)</sup>)

I = Backscattered Electrons  
 III = Secondary Electrons

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)



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SEMs, page 36



## Comparison of Coefficients

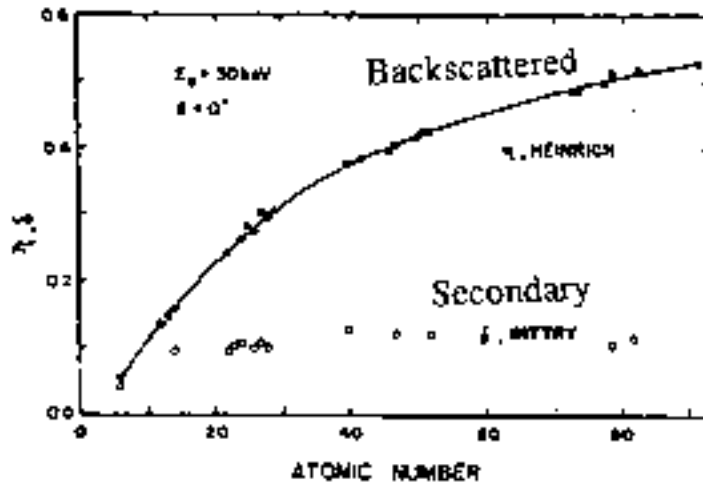
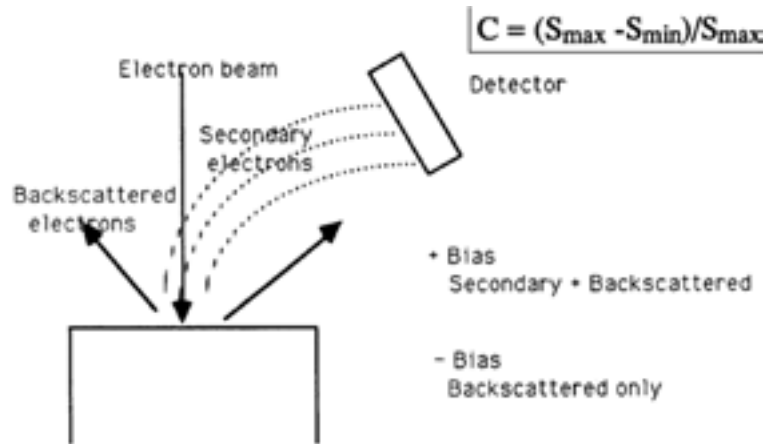


Figure 3.29. Comparison of backscattered electron coefficients and secondary-electron coefficients as a function of atomic number (Witry, 1966; Henning, 1966).

## Contrast

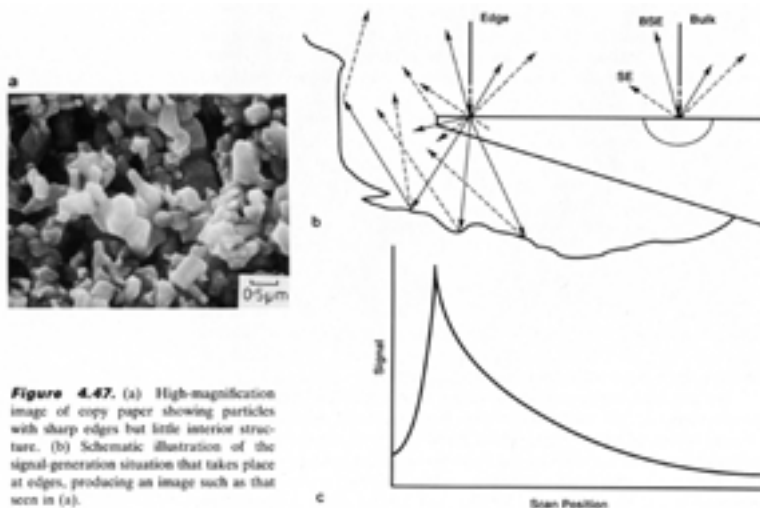
- **BSE Yield:**
  - Atomic number dependent
    - Higher atomic number  $\rightarrow$  Higher BSE yield  $\rightarrow$  Brighter image
  - Contrast in BEI is a combination of:
    - Surface topography
    - Composition - Z contrast
- **SE Yield:**
  - Less dependent on Z
  - More dependent on accelerating voltage
  - Contrast in SEI dependent on:
    - Sample orientation (emission contrast)
    - Detector position (collector contrast)
    - Surface topography – sharp edges emit more SE's

## Contrast



Note: While backscattered electron yield is larger than the secondary electron yield, secondary electrons are collected from a larger area and will contribute from 2-5 x backscattered electron signal. So SEI will give more contrast for the same beam current.

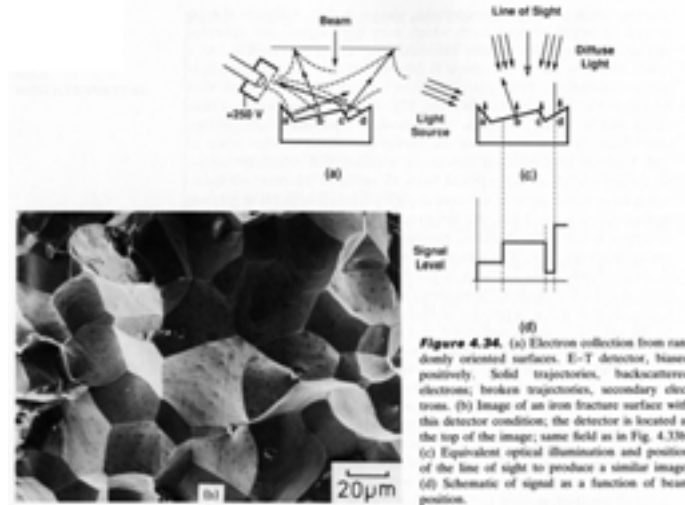
## Contrast: Sharp Edges



**Figure 4.47.** (a) High-magnification image of copy paper showing particles with sharp edges but little interior structure. (b) Schematic illustration of the signal-generation situation that takes place at edges, producing an image such as that seen in (a).

(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)

## SEI Mode

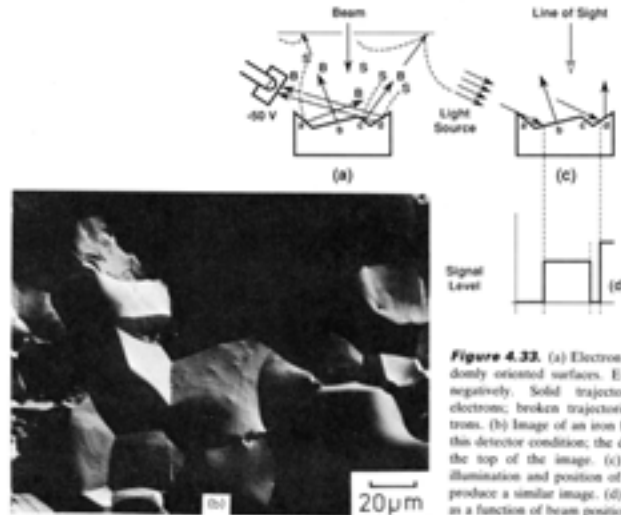


(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)

## Light Optical Analogies: SEI Mode

- With the detector positively biased, secondary electrons are attracted to it
- These electrons are detected as well as the backscattered electrons
- However there are usually many more secondary electrons than backscattered electrons
- Also some of the surfaces that are not directly facing the detector will contribute electrons to the signal
- The light optical analogy is a single light source from the side combined with diffuse lighting from the top

## BEI Mode



**Figure 4.33.** (a) Electron collection from randomly oriented surfaces. E-T detector, biased negatively. Solid trajectories, backscattered electrons; broken trajectories, secondary electrons. (b) Image of an iron fracture surface with this detector condition; the detector is located at the top of the image. (c) Equivalent optical illumination and position of the line of sight to produce a similar image. (d) Schematic of signal as a function of beam position.

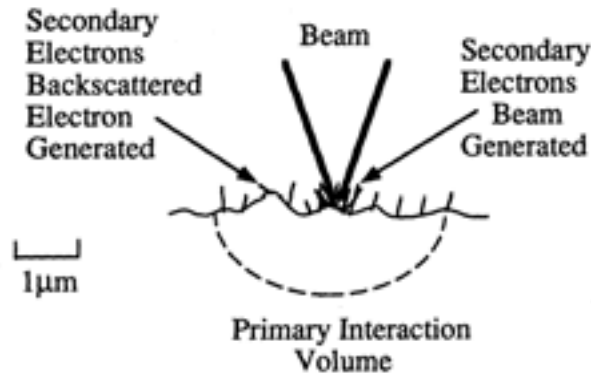
(From *Scanning Electron Microscopy and X-Ray Microanalysis*, Joseph I. Goldstein et al. Plenum Press)

## Light Optical Analogies: BEI Mode

- When the detector has a negative bias, only the backscattered electrons are detected
- The path of these electrons is generally line of sight
- The light optical analogy is a single light source from the side with the eye looking down along the beam path
- This mode gives strong topological contrast

## Sample Volume Effects: Image Degradation

- SEI is most susceptible to this effect:
  - The backscattered electrons originating deep in the sample create secondary electrons when they emerge from the surface
  - The distance that the back-scattered electron emerges from the sample can be far from the beam spot (several beam spot diameters)



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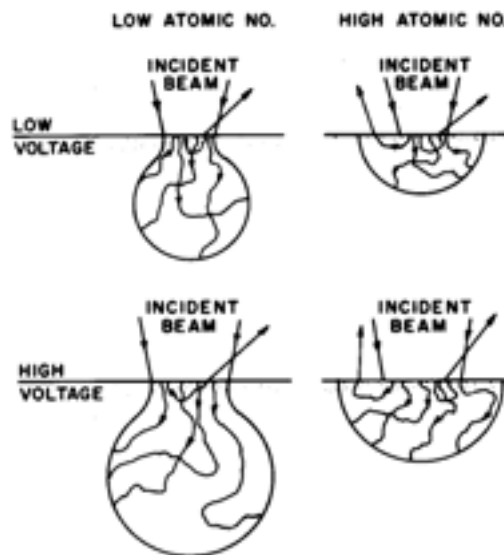


SEMs, page 45

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## Interaction Volume

- Interaction volume dependence on accelerating voltage and atomic number:
  - Section through specimen surface illustrating the variation of electron scattering with voltage and atomic number  
(from Duncumb and Shields)



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SEMs, page 46

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## Sample Preparation

- **Advantage of SEM - little preparation needed!**
- **Mount on appropriate stub making electrical contact**
  - Use conductive adhesive or clips to secure sample
- **Various mounts:**
  - Whole wafer mounts
  - Small piece mounts
- **Cleave to look at cross-section**
- **Non-conducting samples will charge causing sample drift, image distortion, and dark or bright image**
  - Sputter coat with metal, e.g., Au-Pd
  - Evaporate C for x-ray analysis
  - Use low voltage
- **Mark specimen with a “Sharpie” if area of interest difficult to locate**



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SEMs, page 47



## Cheat Sheet

### To increase depth of field:

Long working distances  
Small Aperture

### Surface Sensitivity:

Lower accelerating voltage

### High Resolution:

Higher accelerating voltage  
Reduce spot size  
smaller aperture  
or condenser lens  
Short working distance  
Slower scan rate

### Charging or insulating sample:

Low accelerating voltage -  $\geq 1.5$  kV  
Sputter coat with Au-Pd

### Better S/N:

Larger Aperture  
Slower scan rate

### In-lens Detector - LEO 982:

Sample normal to beam  
 $\leq 4$  mm WD from pole piece

### Focus and stigmatism:

Adjust focus and stigmatism at higher mags than working mag  
Adjust to best focus  
Adjust 1 or both stigmator controls  
Iterate between focus and stigmatism (roundish structures helpful in adjusting stigmatism)  
Center apertures after changes in accelerating voltage or aperture



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SEMs, page 48





## Equipment Available in CNF

- LEO 982 SEM's (2)
  - Thermal Field Emission
  - 4.0 nm @ 1kV
  - 2.0 nm @ 30kV
  - For Low Resolution Work

### Hitachi S-4700

- Cold Field Emission
- 2.1 nm @ 1kV
- 1.5 nm @ 15kV
- For High Resolution Work



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SEMs, page 49



## Beware!!!

Ultimate Resolution is Often  
Sample Limited  
**NOT**  
Instrument Limited

- Ultimate resolution on an SEM tested with an ideal sample:
  - Au islands on Carbon
  - High Z material on low Z material
  - Few SEs created by BSEs
  - Strong contrast
- Your sample is not likely to be as ideal



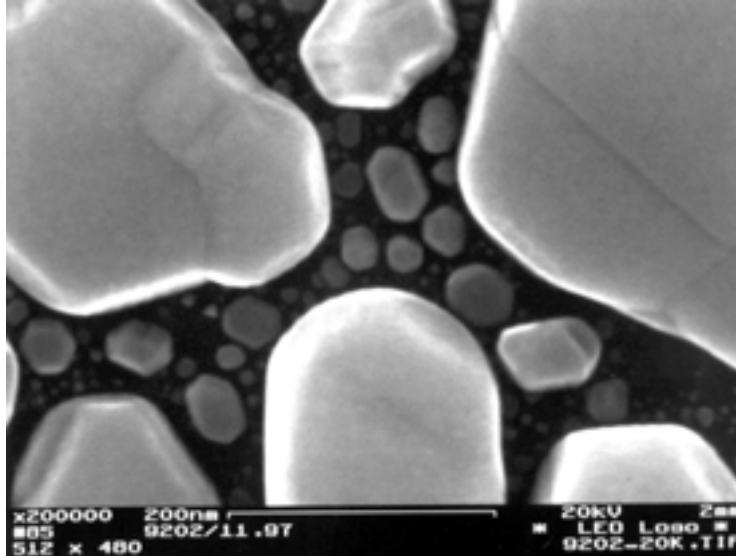
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SEMs, page 50



## Gold on Carbon Resolution Standard



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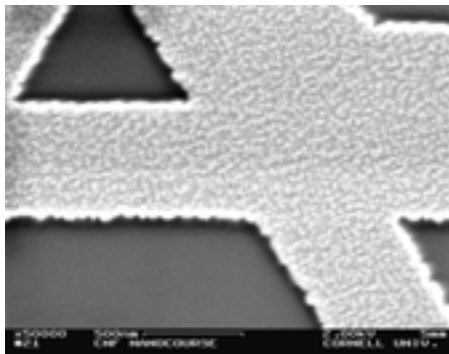
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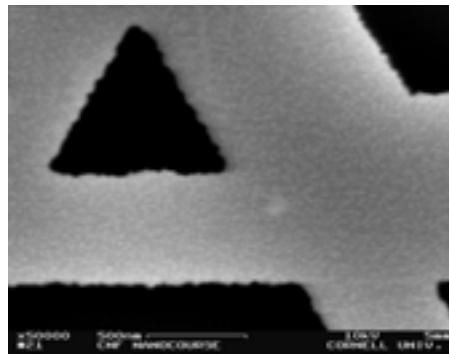
SEMs, page 51

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## Low vs. High Accelerating Voltage



2 KeV



10 KeV

Kagome Superconductivity Structure

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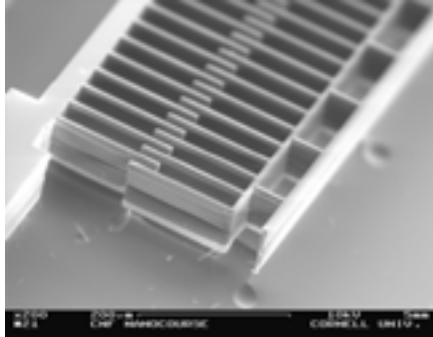
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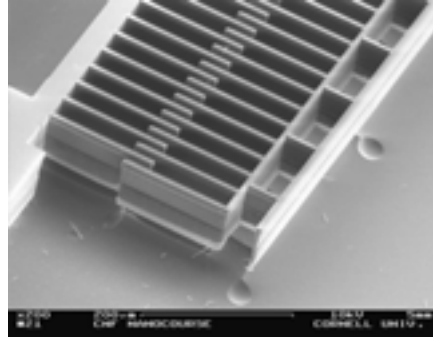
SEMs, page 52

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## Depth of Field: Effect of Aperture Size



WD - 5mm, Aperture size 120  $\mu\text{m}$



WD - 5mm, Aperture size 20  $\mu\text{m}$

MEMS Comb Drive



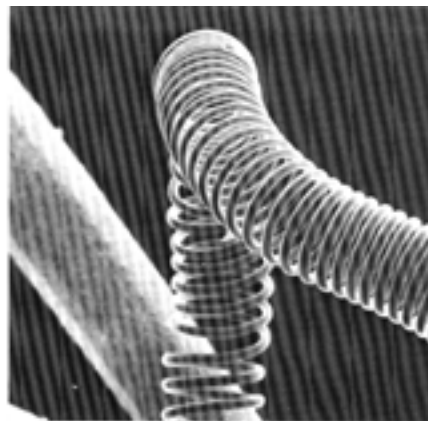
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SEMs, page 53

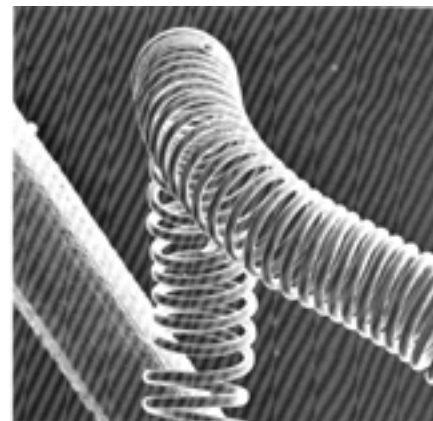


## Depth of Field: Effect of Working Distance



(c) W. D. 13 mm

Aperture size 100 $\mu\text{m}$ .



(d) W. D. 37 mm

Aperture size 100 $\mu\text{m}$ .



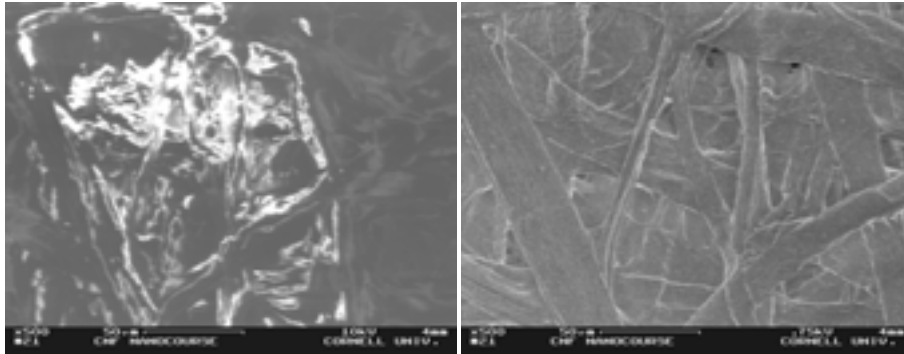
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SEMs, page 54



## Sample Charging: Paper

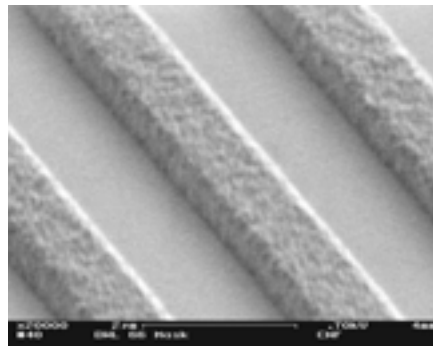
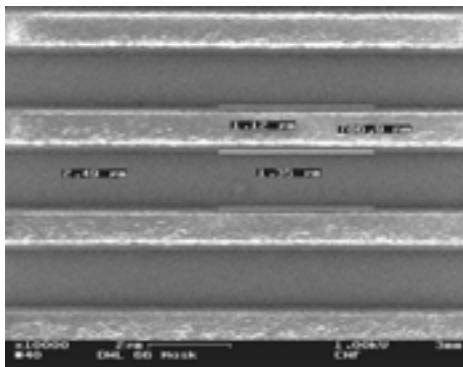


10 KeV Accelerating Voltage

750 eV Accelerating Voltage

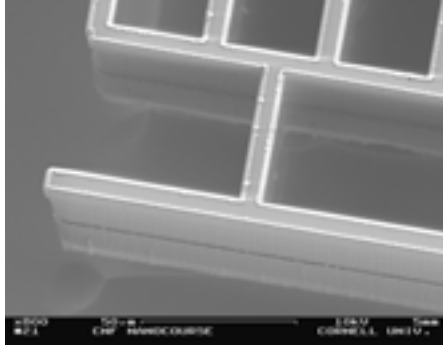
## Low Voltage Inspection of Resist

Below: At low accelerating voltages ( $\leq 1500V$ ) it is possible to view nonconducting samples uncoated. Note charging at 1kV below – 700V at right no charging.

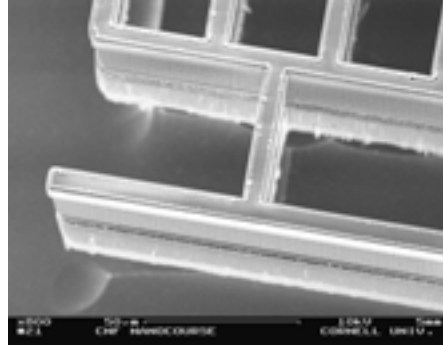


Above: Positive resist on Si wafer – tilted 30° to view sidewall profile above. Normal to beam at left for measuring.

## Lateral SE-Detector vs. Annular SE-Detector



“Normal” SE-Detector



“In-Lens” SE-Detector

### MEMs Comb Drive



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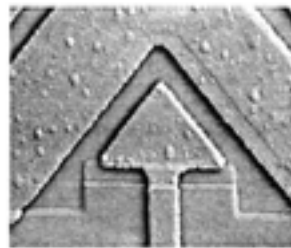
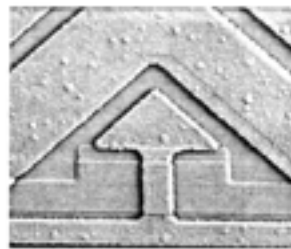


SEMs, page 57



## Tilt Compensation

Set tilt compensation to tilt angle to compensate for foreshortening in Y direction



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SEMs, page 58



## Dynamic Focus

Allows entire sample to be in focus at high tilt angles

- Dynamic focus ON (45)
- Adjust amplitude
- Scan rotation (40) must be OFF and the sample aligned in Y direction



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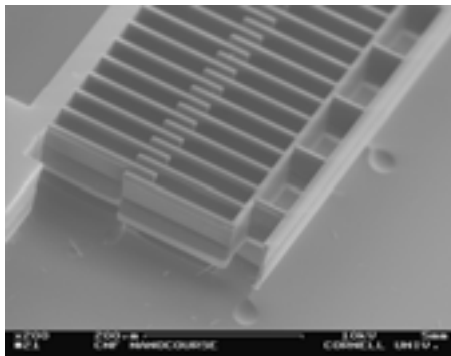
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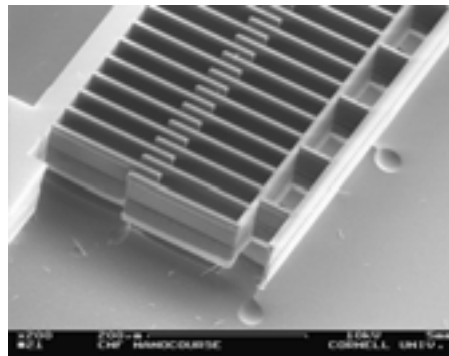
SEMs, page 59

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## Signal Processing



No Signal Processing



GAMMA 3, Contrast Doubled

MEMS Comb Drive

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SEMs, page 60

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## References

- **SEM:**

- Scanning Electron Microscopy and X-Ray Microanalysis, Goldstein, Newbury, Echlin, Joy, Romig, Lyman, Fiori, and Lifshin
- Scanning Electron Microscopy, L. Reimer, Springer Verlag, #45 in the optical sciences series

- **TEM:**

- Practical Microscopy of Materials Science, J. W. Edington
- Electron Microscopy of Thin Crystals, Hirsch, Howie, Nicholson, Pashley, and Whelan
- Introduction to Analytical Electron Microscopy, Hren, Goldstein, and Joy
- Transmission Electron Microscopy, L. Reimer, Springer Verlag, #36, optical sciences series
- Electron Microscopy, Bozzola and Russell, 1992

- **EPMA:**

- Microprobe Analysis, Anderson (ed.), Wiley-Interscience



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SEMs, page 61



## NanoCourses 2004, Section 4

# Practical Thin Film Analysis

## *What is it?*

### Electrical & Optical Measurements

by

### Phil Infante

Presented by the  
**CNF** Technical Staff  
for the education of CNF Users,  
Potential Users, and Industrial Sponsors



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Measurements, page 1



## Resistivity Measurement

- Resistivity ( $\rho$ ): A measure of the inability of a layer to support the conduction of electrical carriers (electrons or holes)

$$\rho = [neu]^{-1} \text{ (units = ohm-cm)}$$

$n$  = carrier concentration

$e$  = electronic charge

$u$  = carrier mobility

- For thin layers, it is useful to introduce the concept of resistance per unit area, or Sheet Resistance:

$$R_s = \rho/t \text{ (units = ohm/square)}$$

$t$  = thickness of layer



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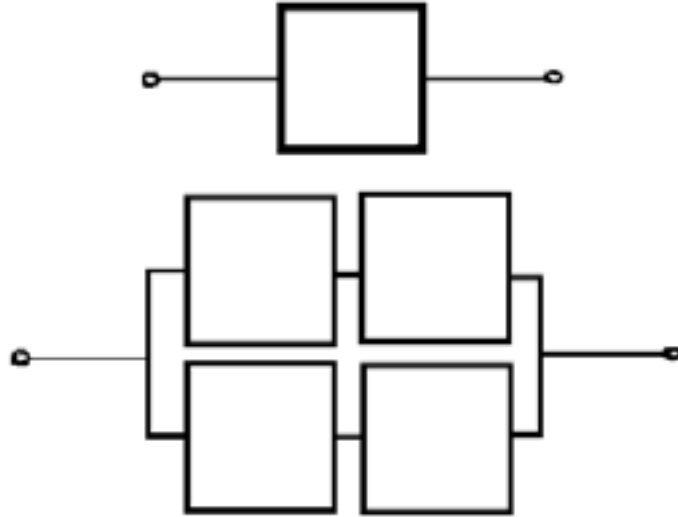


Measurements, page 2





## Sheet Resistance Example



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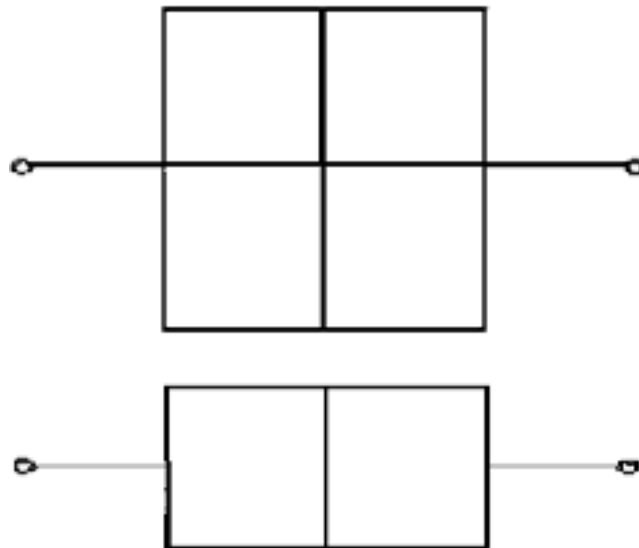
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Measurements, page 3

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## Sheet Resistance Example



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Measurements, page 4

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## Sheet Resistance

- A rectangular sample would have a resistance given by:

$$R = V/I = R_s (l/w)$$

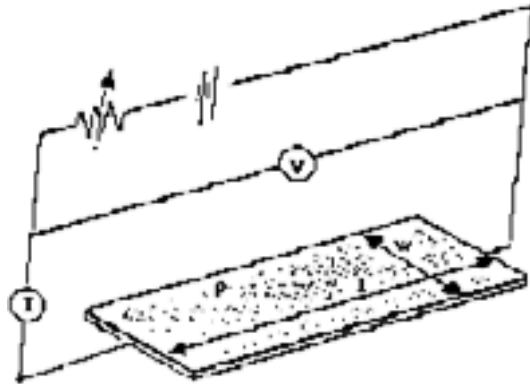
- In most films the carrier concentration, and therefore the resistance will vary with depth  $z$ :

$$\rho(z) = [n(z)e\mu(n)]^{-1}$$

(note  $\mu$  is a function of  $n$ )

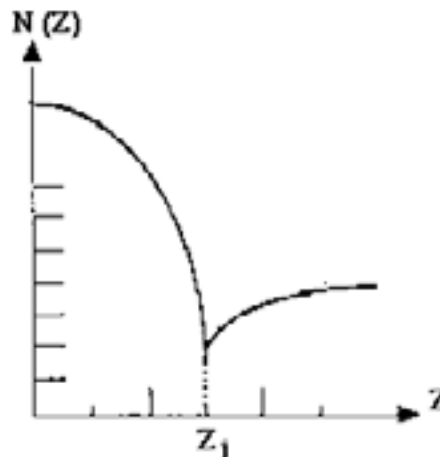
So the sheet rho is:

$$R_s = \left[ \int_0^t n(z)e\mu(n) dz \right]^{-1}$$



## Film Thickness

- For a p-n junction, the thickness  $t$  equals the junction depth  $z_j$
- Also, the top film is much less resistive than the bulk
- You can assume:
  - $t =$  the film thickness



## Van der Pauw Structures

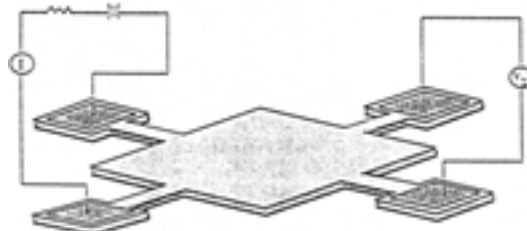
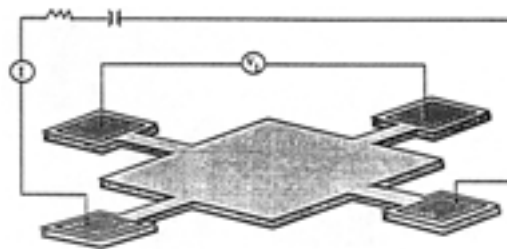
- Two methods to measure  $R_s$ :
- First Method: Van der Pauw Structure (*See next page*)

$$\begin{aligned}R_s &= (\pi/\ln 2)(R_1 + R_2)/2 \\ &= 4.532(R_1 = R_2)/2\end{aligned}$$

$$R_1 = V_1/I, R_2 = V_2/I$$

- The measurement is geometry-independent because of the paired resistances,  $R_1$  and  $R_2$
- Problems:
  - Needs to be patterned, may need metallization on pads to carry current to high rho structures

## Van der Pauw Structures



## Four Point Probe

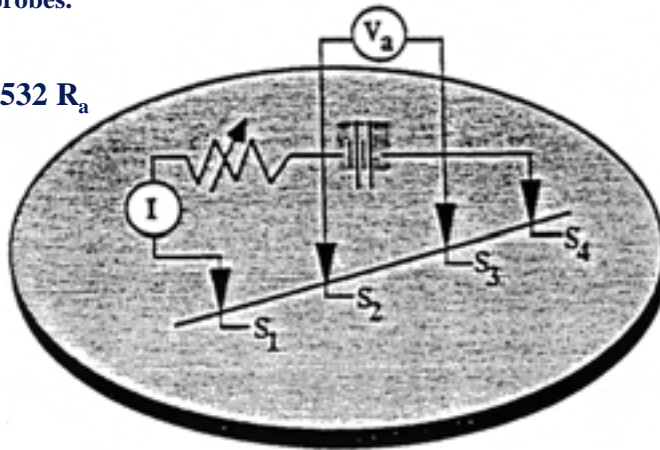
- **Second Method: Four Point Probe**

- Force a current through two probes, measure the voltage drop across the remaining probes.

$$R_s = (\pi/\ln 2) R_a = 4.532 R_a$$

$$R_a = V_a/I$$

Note:  $R_a$  is the average resistance obtained by reversing the polarity of the current supply. This eliminates voltage offsets in the measuring circuit.



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Measurements, page 9

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## Four Point Probe

- To compensate for geometric effects, do dual configuration measurement, similar to Van der Pauw structure (*See next slide*)

$$R_s = k(\zeta) R_a$$

$$\zeta = R_a/R_b$$

- For the ideal case of equal probe spacing on an infinite sheet:

$$\zeta = 1.2619$$

$$k = \pi/\ln 2 = 4.532$$

- This gives a measurement accuracy of +/- 0.5%

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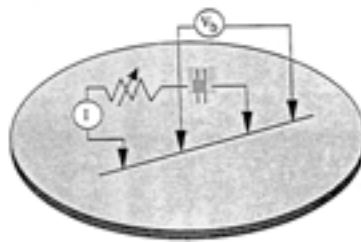
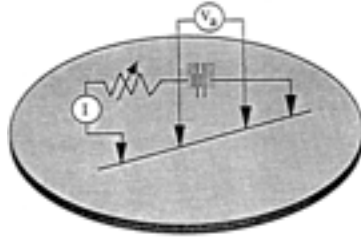
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Measurements, page 10

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## Dual Configurations



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Measurements, page 11  
CNF Na

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## Prometrix VersaProbe VP-10

- At the CNF, we have a Prometrix VersaProbe VP-10, a lovely instrument that does all this for you automatically
- Measurement range = 5 microhm/square to 5 megohm/square
- Things to remember:
  - 1) Oxide on your film produces a contact resistance. The probes can break through the 10 monolayers present on silicon. If there is much more than this on the film, the oxide should be removed.
  - 2) Whole wafers are the most convenient measurement vehicle. Fragments of wafers can be done, but it isn't as much fun.
  - 3) NO ALUMINUM FILMS MAY BE MEASURED ON THE VP-10. Aluminum lodges in the pits in the probe tips, oxidizes, adds a resistor to the measurement circuit, and screws up future measurements. Aluminum MAY be measured with the manual 4-point probe.
  - 4) Geometry effects come into play when the "infinite film" becomes smaller in area (sic). Make sure your "infinite film" is at least a 1 cm by 1 cm square.
  - 5) Grain boundaries can cause  $\zeta$  to be too high -- "ratio error." Do single configuration test instead of dual configuration.

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Measurements, page 12

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# Interferometry (Leitz MPV-SP)

- A method of measuring film thickness based on the interference analysis of the light reflected from the surface of the object to be measured
- Our Leitz SP focuses white light on the sample and analyzes the reflected signal from 400 to 800 nm
- Measurement Principal
- The Leitz manual gives an excellent explanation of the measurement principal, so here it is:



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Measurements, page 13



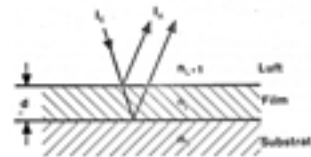
## 3.3 Measuring

### 3.3.1 Principle

The measuring principle is based on the interference analysis of the light reflected from the surface of the object to be measured.

A normalized spectral curve is necessary for the determination of film thicknesses, i.e. the intensity measured from the point in question must be divided by that from the substrate. This ensures that substrate- and instrument-specific influences are largely eliminated.

To make this clear, the following example considers the special case of light incident perpendicularly on/in, transparent, non-absorbent films on a non-absorbent substrate.



$$R = \frac{I}{I_0} = \frac{r_{01}^2 + r_{12}^2 + 2 r_{01} r_{12} \cos \delta}{1 + r_{01}^2 r_{12}^2 + 2 r_{01} r_{12} \cos \delta}$$

$$\text{where } \delta = 4d n_1 \cdot \frac{2\pi}{\lambda}$$

the reflection coefficient between air and film

$$r_{01} = \frac{1 - n_1}{1 + n_1}$$

the reflection coefficient between film and substrate

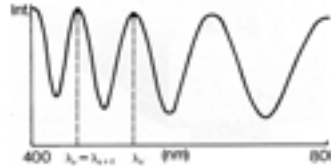
$$\frac{n_2 - n_1}{n_2 + n_1}$$

$n_1$  and  $n_2$  are the refractive indices of film and substrate,  $\lambda$  is the wavelength, and  $d$  is the film thickness

The film thickness  $d$  is determined from the peak positions (at least two peaks in the normalized spectral curve):

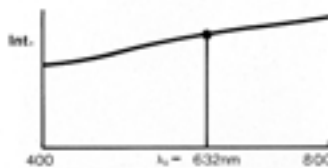
$$d = \frac{\lambda - \lambda'}{4n_1} \cdot \frac{n_2 + n_1}{n_2 - n_1}$$

where  $p$  and  $q$  are the ordinal numbers,  $\lambda_p$  and  $\lambda_q$  are the wavelengths of peaks  $p$  and  $q$ , and  $n_1$  is the refractive index of the film.



Normalized spectral curve for a 1100nm thick  $\text{SiO}_2$  film on a Si substrate.

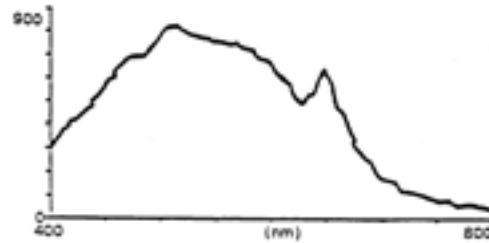
For single-layer films with a thickness of about 10 to 80 nm (ordinal number  $p=0$ ), the thickness is determined, according to the first equation given above (for  $R$ ), from the ratio of the intensity of the light reflected from the film to that of the light reflected from the substrate, at a selected wavelength (that of the He-Ne laser, i.e.  $\lambda_0 = 632.8$  nm).



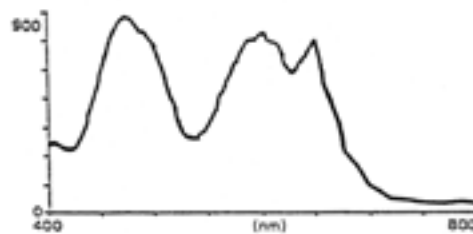
Normalized spectral curve for a 50 nm thick  $\text{SiO}_2$  film on a Si substrate.

## Measurement Sequence

- 1) Measure and record the reflectance spectrum for a **BARE SUBSTRATE** (2 nm native oxide can be ignored)



- 2) Measure and record the reflectance spectrum for the **film on the substrate**



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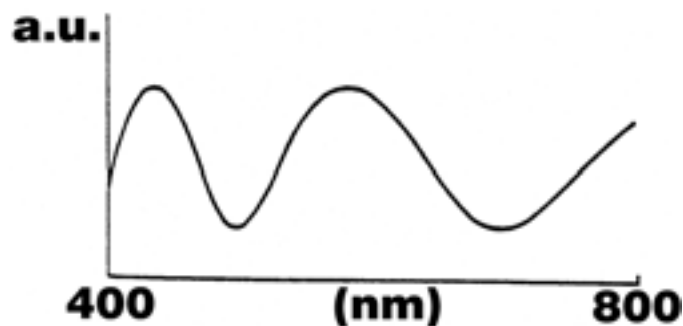


Measurements, page 15

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## Measurement Spectrum

- 3) Normalize the measurement spectrum by dividing by the calibration spectrum



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Measurements, page 16

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## Measurement Spectrum

- The film thickness is calculated from the normalized spectrum
- For films < 80 nm (thin film program)
  - Measure reflected intensity from substrate at 550 nm. measure refl. intensity from film at the same wavelength
  - Calculate thickness from intensity ratio



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Measurements, page 17



## What can (and can't) the Leitz do?

Minimum measurement area = 2  $\mu\text{m}$  by 2  $\mu\text{m}$ . Yes, this is very small. But the signal to noise ratio, and hence the accuracy, improves as the area gets bigger. So make sure your sample has a nice large area for Leitz measurement.

The Leitz can measure the thickness of the top layer of a film-on-film-on-substrate sample (e.g. polysilicon on  $\text{SiO}_2$  on Si). You must know the thickness of the intermediate layer within 10%. Also the intermediate layer should be no thicker than 200 nm. (Empirical observation by yours truly for polysi on  $\text{SiO}_2$ . You are welcome to try other combinations. If you find combinations that work with thicker intermediate films, please tell me.)

If the film and substrate have refractive indices that are very close (like polysilicon on silicon) don't expect good results. It helps to have an intermediate layer of a different refractive index.



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Measurements, page 18





## What can (and can't) the Leitz do?

Film thickness range = 10 nm to 15  $\mu\text{m}$ .  
Accuracy < + /- 2 to 5%  
for films > 100 nm depending on material,  
< 2 nm for films < 100 nm.



## First Thing to Remember:

- DON'T BELIEVE EVERYTHING YOU READ !!!
- While this instrument is quite sophisticated, the principal is such that the software is easily confused
- When trying something "unconventional" for the first time (e.g.  $\text{SiO}_2$  on PMMA on GaAs), it is a good idea to verify the measurement via some other technique



## Second Thing to Remember:

- DON'T TRY TO FOOL IT !!!
- It is easily fooled.
  - For example, if you tell it to measure  $\text{SiO}_2$  and you really have photoresist, it will give you an answer that it thinks is perfectly logical
- Make sure you have an area large enough to measure
- If you are measuring film on film make sure you know the thickness of the intermediate layer



## Example 1

Examples of samples that can and cannot be measured:

Oxidize Silicon - Thermal Oxidation

Pad Oxide - 30 nm
P-Type Silicon

Yes. Use thin film or thick film programs.

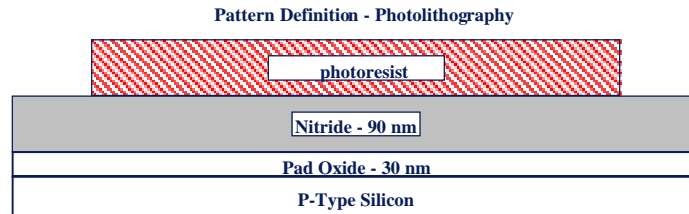
Silicon Nitride Deposition - LPCVD

Nitride - 90 nm
Pad Oxide - 30 nm
P-Type Silicon

Yes. Use nitride on oxide program.



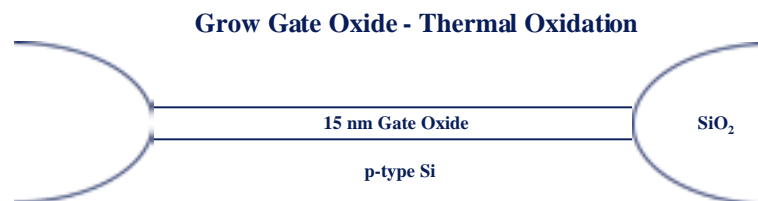
## Example 2



No. Cannot measure 3-layer structure. However, you can ignore the 30 nm pad oxide and get good results.

Use resist on nitride program.

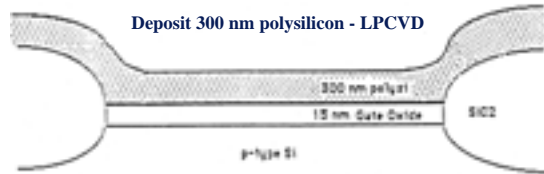
## Example 3



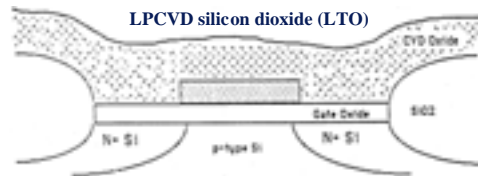
Yes, provided the gate area is at least  $2\mu\text{m} \times 2\mu\text{m}$ .

Use the thin film program.

## Example 4



**Yes. Use polysilicon on oxide program.**

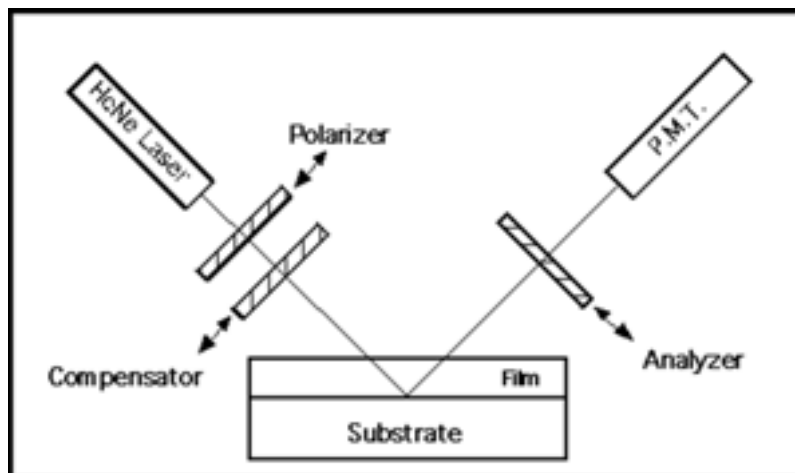


**No, in gate area (3 films).**

**Yes, in source-drain area provided the area is large enough.  
Use oxide on Si and ignore 15nm gate oxide.**

## Ellipsometry

Apparatus:



## Ellipsometry

- Principal:

- Monochromatic light (633 nm in our case) passes through two polarizing grids.
- The first is called the "polarizer," the second is the "compensator."
- This produces light that is elliptically polarized. This light reflects off the sample and experiences a change in the ellipticity of polarization.
- A third polarizing grid, called the "analyzer," is adjusted along with the polarizer to achieve a reflected signal of minimum intensity.
- This is called a "null." The positions of the polarizer and analyzer are recorded at this point.

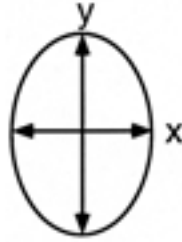


## Null Points

- Null points are measured in four "zones," corresponding to the four quadrants of the ellipse. The positions of the analyzer and polarizer for all four zones are fed into a computer which calculates  $\Delta$  and  $\Psi$ . These are angles that describe the shape of the ellipse. From this the program calculates the thickness and refractive index of the film.
- Very accurate and precise (+/- 1 monolayer for some films).
- Can measure films < 10 nm thick -- >
- USE INSTEAD OF LEITZ FOR FILMS < 20 nm.
- Can measure refractive index of bare substrates.
- Maximum film thickness 1  $\mu\text{m}$ .
- Refractive index of very thin films may be different than that of "bulk" material so you may have to assume an index (i.e. fudge it).



Before passing through film



After passing through film

$\Delta = y^1/x^1$   
 $\psi = \text{Phase shift}$



## Spot Size

- Spot size is large - 1 mm diameter. Measurement area should be 0.5 cm by 0.5 cm. The bigger the better.
- Refractive index of film should be less than 3.0 --> you can't measure polysilicon.

## NanoCourses 2004, Section 4

# Practical Thin Film Analysis

## *What is It?*

Auger Electron Spectroscopy  
by  
Lynn Rathbun, Ph.D.

Presented by the  
**CNF** Technical Staff  
for the education of CNF Users,  
Potential Users, and Industrial Sponsors



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Auger & Analysis, page 1



## Auger Electron Spectroscopy

- One of several techniques for materials analysis commonly used
  - Surface analysis
  - Thin film analysis
  - Not bulk analysis
    - Auger Electron Spectroscopy
    - Auger
    - Auger Microprobe
    - Scanning Auger Spectroscopy
    - Scanning Auger Microprobe
    - AES
    - SAM



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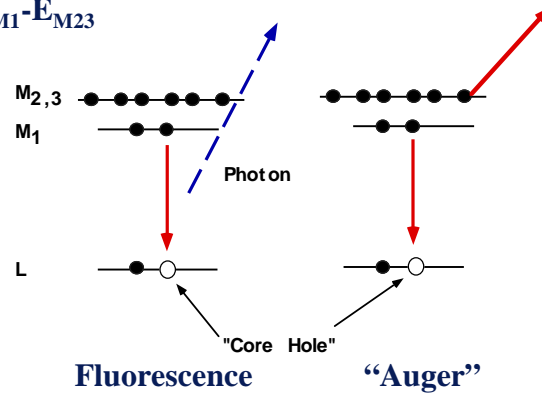


Auger & Analysis, page 2



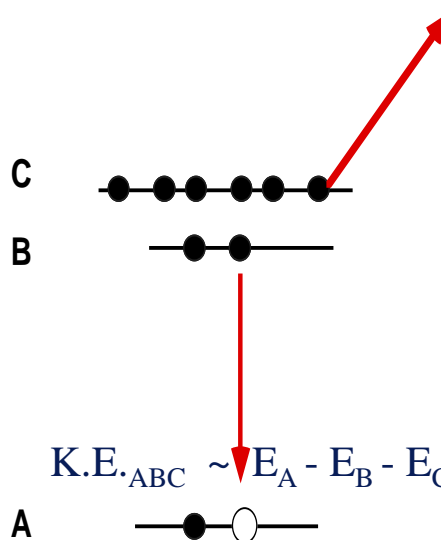
## Auger Transition

- Radiationless electron transition in an atom
- Fluorescence yield dominates only for transitions  $> 10\text{KeV}$ , i.e. certain heavy atoms transitions
- Kinetic energy  $\sim E_L - E_{M1} - E_{M23}$



## Auger Transition

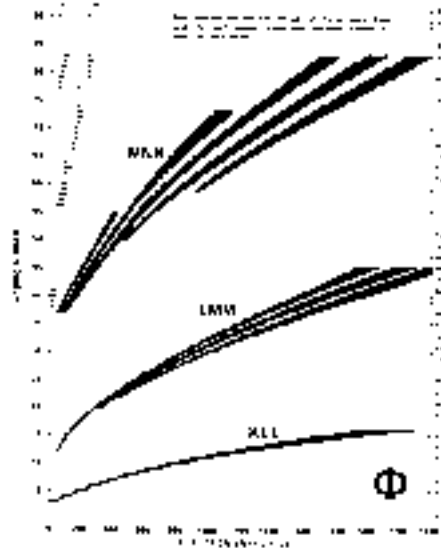
- Electron of a fixed energy, independent of excitation method
- Characteristic of atom
- Not an energy loss process
- Labeled by the 3 atomic shells involved, i.e KLL, LMM, LVV, etc
- Commonly kinetic energies up to 2500 eV



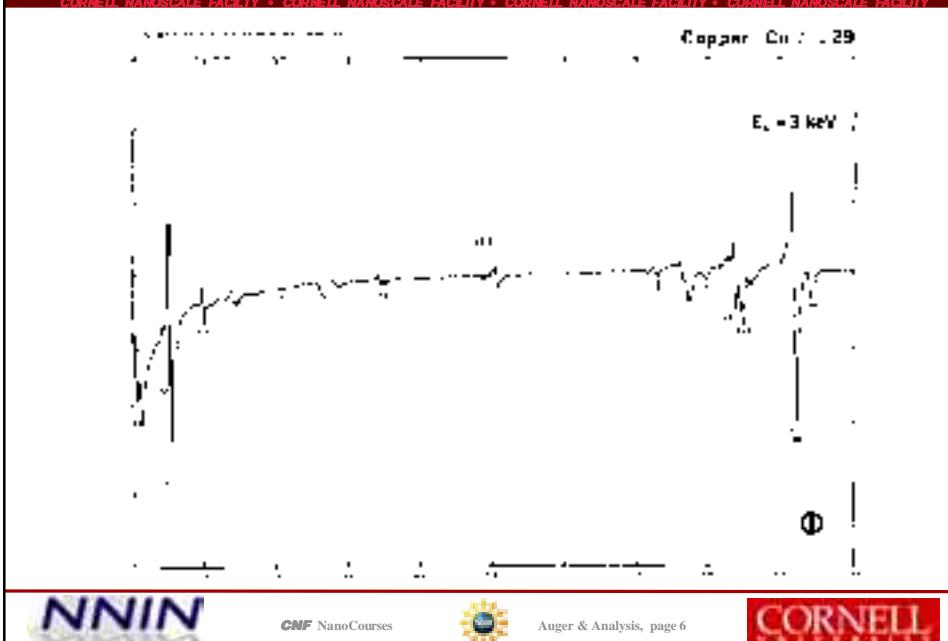


## Auger Transition Bands

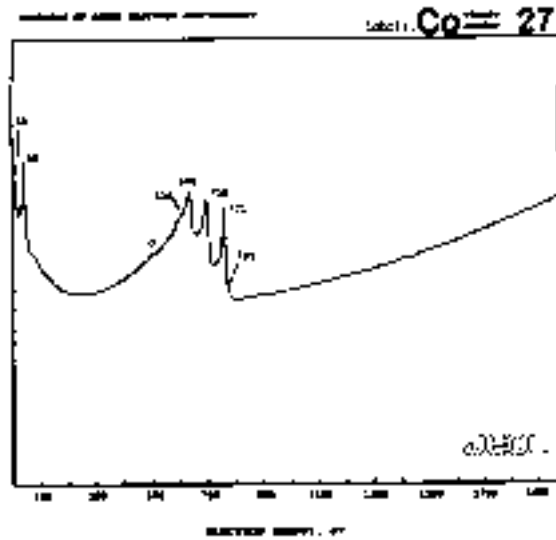
- A set of energy bands with unique transitions for each element



## Spectral Example, 1



## Spectral Example, 2



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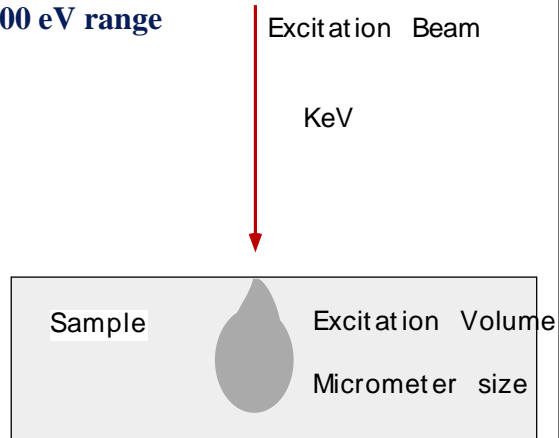


Auger & Analysis, page 7

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## Excitation

- Auger decay presumes a “core hole,” i.e. an excitation
- High energy electron beam impacting a solid surface
- Threshold varies 200-2000 eV range



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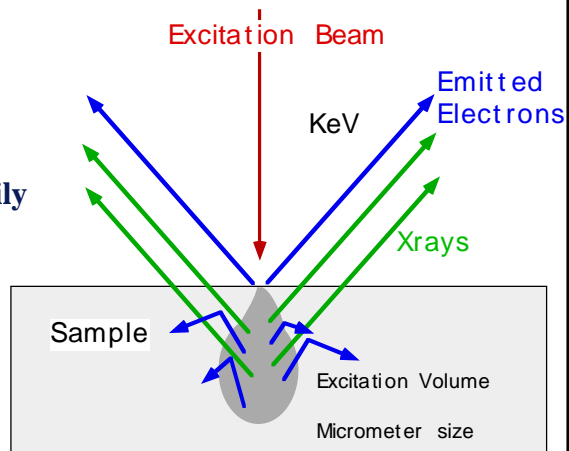


Auger & Analysis, page 8

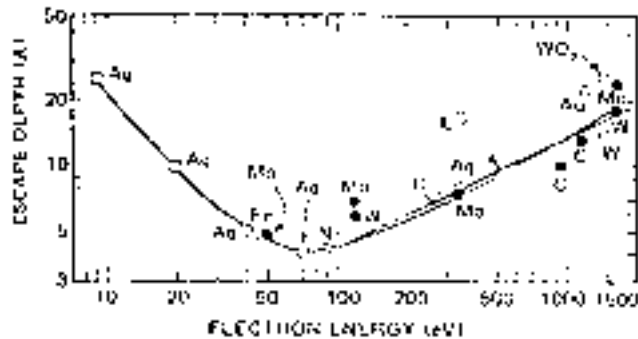
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## Excitation and Escape

- Atoms within entire bulb are being excited
- Decay by
  - X-ray fluorescence
  - Auger electrons
- X-rays easily escape
- Electrons can only easily escape from surface without energy loss

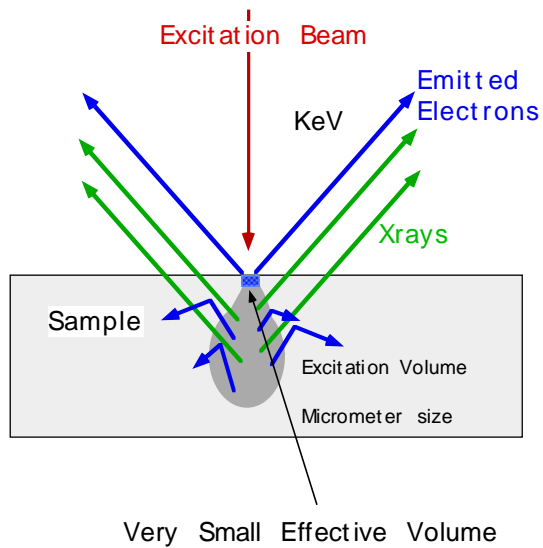


## Electron Escape Depth



- Elastic Mean free path for electron in solid is very short
- Unscattered “Auger” electrons only come from the very surface region.
- **VERY SURFACE SENSITIVE**

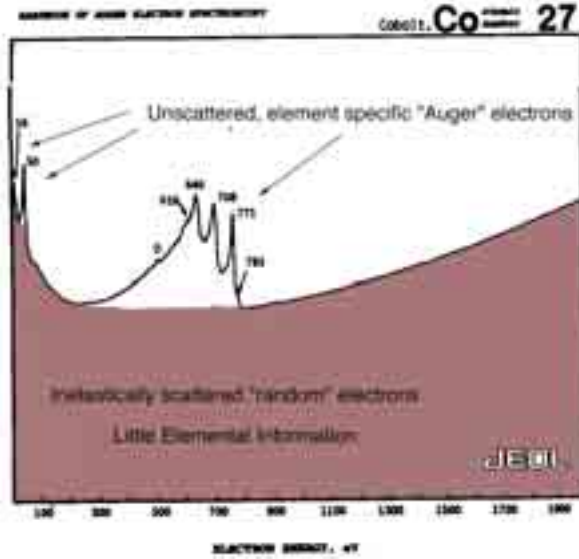
## Surface Sensitive !!!



## An Auger Spectroscopy Tool

- If we can excite and detect Auger electrons from a solid — we can identify atoms !!!
  - Surface Sensitive
    - Clean Surface
    - UHV
- An Ultra high vacuum, SEM like tool, with an energy analyzer

## N(E) vs E Spectrum



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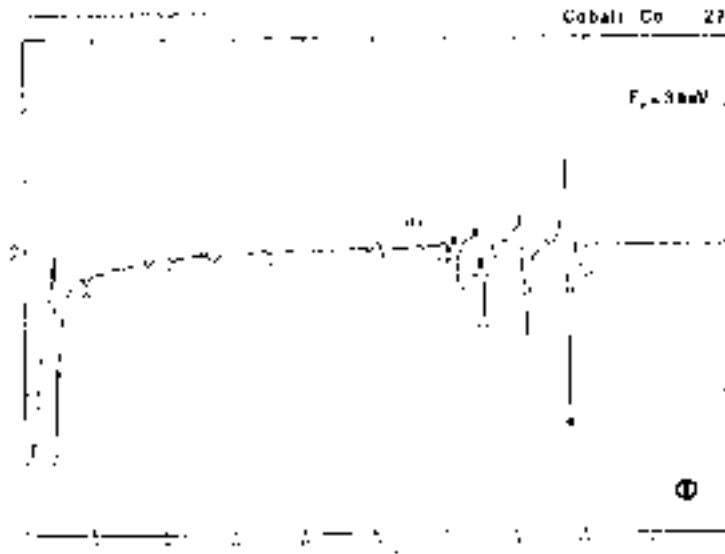
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Auger & Analysis, page 13

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## Derivative N(E) vs. E Spectrum



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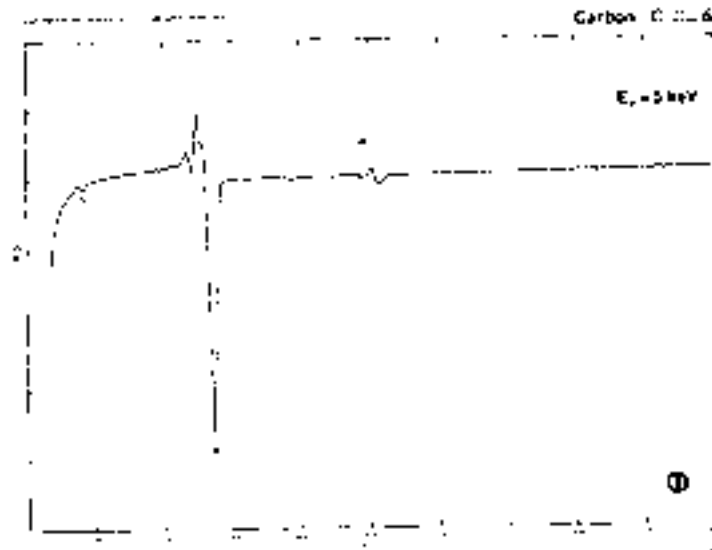
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## Carbon Auger Spectrum



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## Auger Spectroscopy

- **Elemental Analysis Technique**
  - What atoms not what molecules
  - Not chemical analysis
- **Surface Analysis Technique**
  - Not bulk

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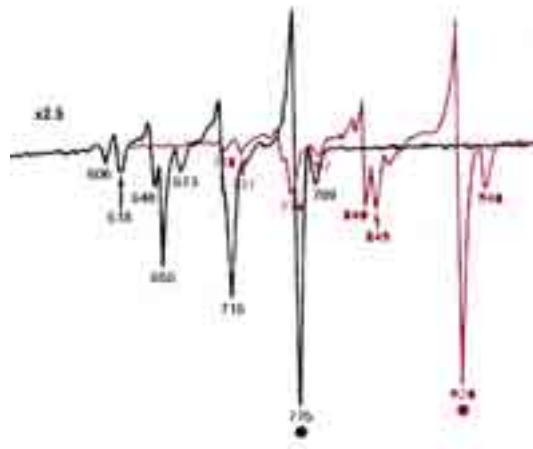
Auger & Analysis, page 16

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## Limitations

- Insulators
- Electron beam sensitive materials
- Interferences
  - Peak overlap

## Cobalt and Copper Superposition



## Capabilities

- Sensitivity to approx. 1%
- Quantity Accuracy
  - Limited
  - Empirical “sensitivity factors”
  - 1- 5%
  - Surface not Bulk !!!!
- Resolution
  - Limited by ebeam size and current
  - < 100 nm lateral
  - Bigger beam==> more current ==> more signal



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Auger & Analysis, page 19



## Accuracy limitations

- Surface vs. bulk
- Matrix effects
  - Electron scattering
- Chemical effects
  - Small changes in peaks effect accuracy even if not useful for “chemical” identification



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Auger & Analysis, page 20





## Scanning Auger

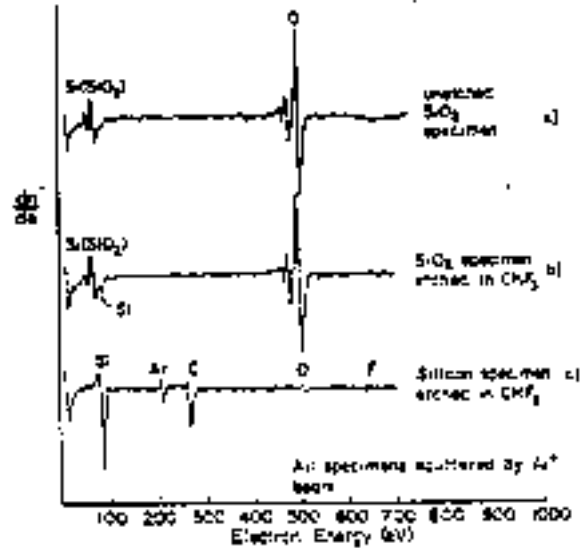
- **Combination of SEM imaging techniques with Auger depection**
  - Point analysis
    - Best S/N
  - Composition var. along a line
  - Compositional “maps”
    - Low S/N
    - Qualitative



## Use of Scanning Auger

- **Failure Analysis**
- **Process documentation / characterization**

## Residue Study: Example



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## Thin Film Analysis

- Auger is fundamentally a surface analysis technique
- What about under the surface
- Slowly erode surface and look at surface vs. time
- Destructive
  - Auger depth profiling
  - Auger sputter profiling

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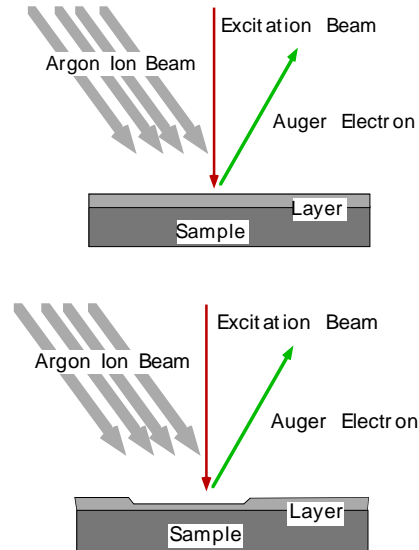


Auger & Analysis, page 24

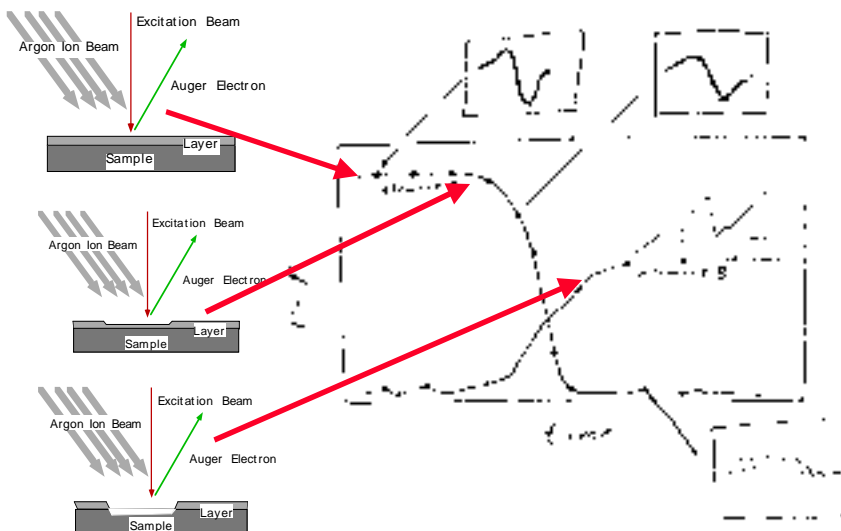
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## Sputter Depth Profiling

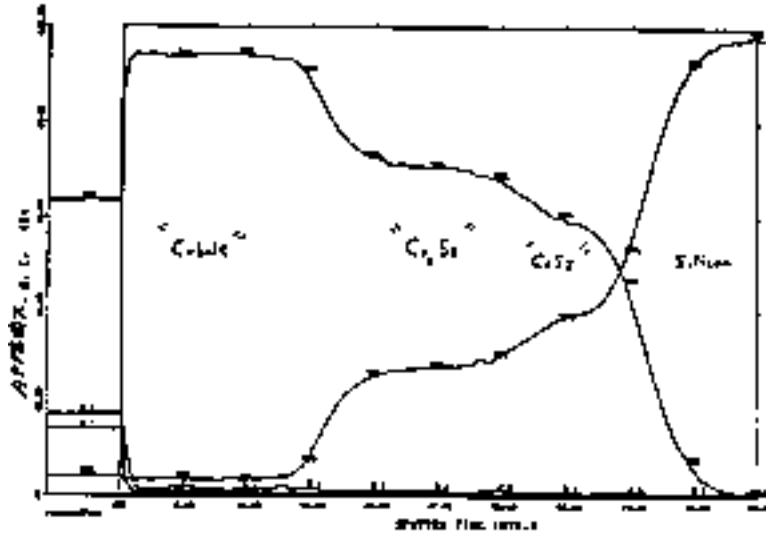
- Ar ions
  - 500-5000 eV
  - 10 A/min - 1000 A/min
  - Depths to ~ 1  $\mu\text{m}$
- 
- Erode a crater
  - Monitor signal vs time
- 
- Destructive!!



## Auger Depth Profile



## Auger Depth Profile



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## An Auger Depth Profile is NOT ----

- Composition vs. depth -----NOT NOT NOT!!!
- Signal vs. time ---- Yes
- Assumptions:
  - Composition as a function of signal
  - Depth as a function of time
  - Uniform

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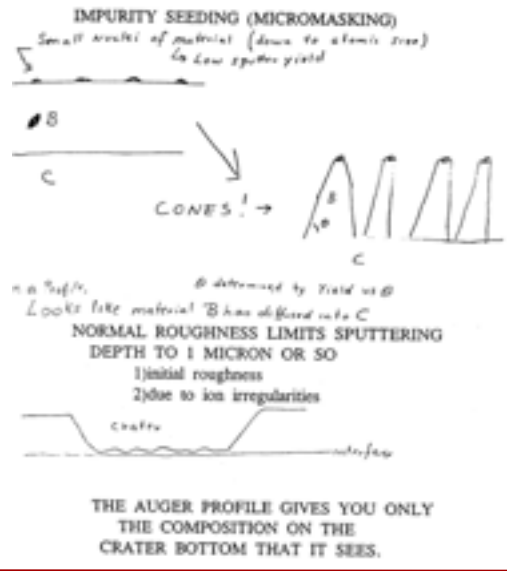
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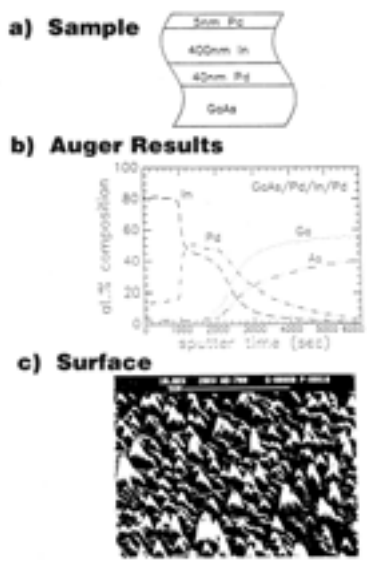
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# Micromasking and Roughness



# Depth Profile Artifacts



## Depth Resolution

- Near surface
  - 3 nm in good case
  - 10 nm common
- Worse the deeper you go
- NOT A TOOL FOR MEASURING DEPTHS

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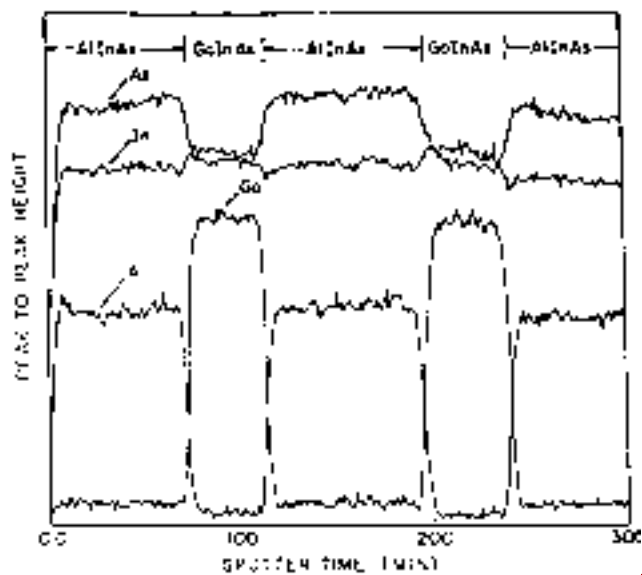
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## High Quality Depth Profile



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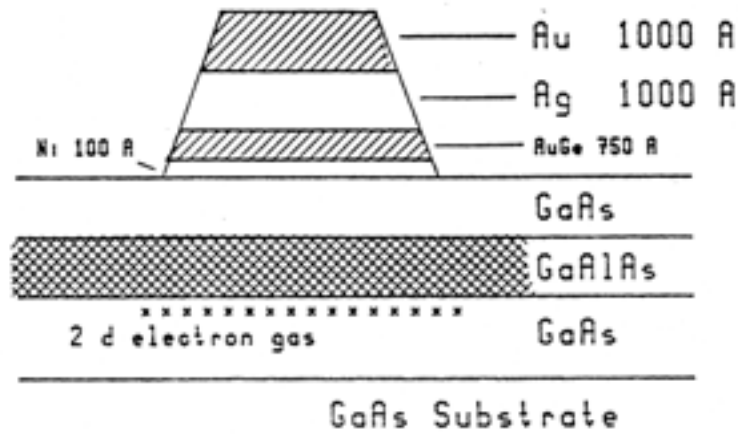
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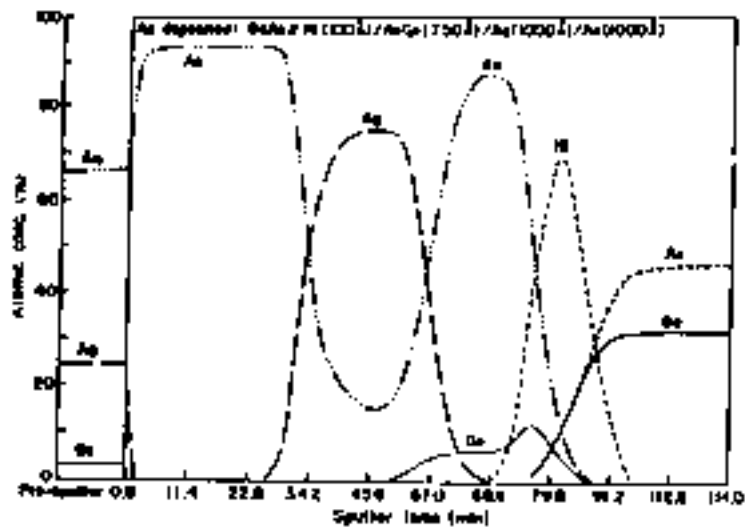
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## Ohmic Contact Example



## Ohmic Contact Profile



## Summary-Depth Profiles

- Ion Milling allows extension of surface analysis technique into thin film analysis
- Localized high resolution depth profile
- Other methods of thin films analysis

## Scanning Auger



- Powerful tool for surface characterization on a submicron scale
- Reasonable sensitivity
- Reasonable accuracy
- Reasonable speed
- High spatial resolution imaging !!



## **Auger Microprobe is----**

- Not a tool for chemical analysis
- Not a tool for insulators
- Not a tool for trace elements
- Not a tool to measure film thicknesses
- Not a tool for bulk analysis
  
- Pick the right tool for the job



## **Electron Spectroscopy for Chemical Analysis**

## Chemical Information

- Auger
  - Elemental Information Only
  - Peaks are wide
- Chemical Information
  - Bond type
  - Valence
  - Compound identification
    - Graphite vs. diamond vs. CO vs. hydrocarbon..etc



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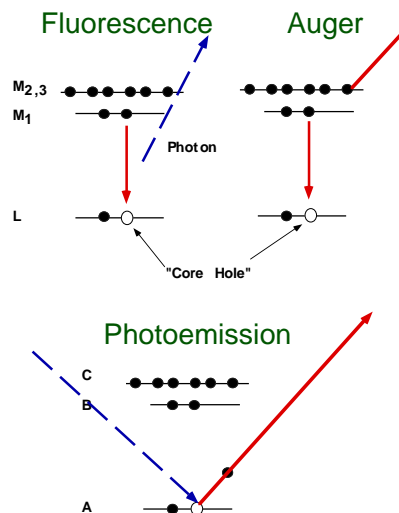


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## ESCA or XPS

- X ray photo-emission
- Electron spectroscopy for chemical analysis
- A photo-emission process
  - X ray in
  - Electron out
  - A single core electron process



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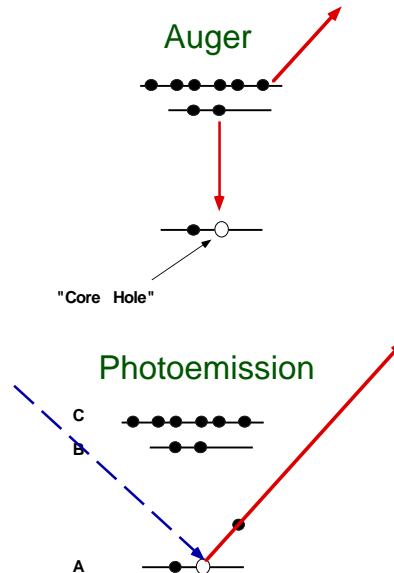


Auger & Analysis, page 40



## Electron Transitions

- Auger
  - 3 electron process
  - Wide
- Photoemission
  - 1 electron process
  - Core electron
    - Sharp narrow level
  - Bonding into valence levels changes core levels slightly by screening
  - **Identify type of bonding**



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## XPS

- $KE = h\nu - E_b$
- Typically fixed energy x ray source ~ 1 keV
- Accurate measurements of absolute peak position
  - < 0.1 eV

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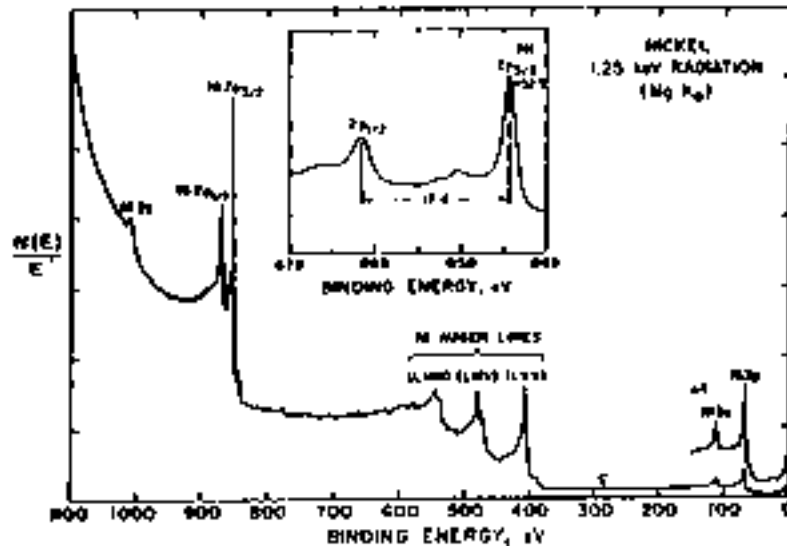
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## XPS Spectrum



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## XPS as an Analytical Tool

- **Tables of peak positions**
  - Type of bond or type of bonded species
- **X ray source**
  - Not focused well
  - Poor spatial resolution
  - 0.1 - 1 mm
- **Surface Sensitive**
  - UHV
- **Can do insulators**
  - Not significant charging effects

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Auger & Analysis, page 44

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## **XPS as an Analytical Tool**

- **Quite complementary to Auger**
- **Sacrifice spatial resolution and scanning for chemical information and insulators**
  
- **Common analysis tools in most industrial laboratories**
- **Research systems at CU but no generally accessible drop it in analytical tool**



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## **Secondary Ion Mass Spectroscopy**



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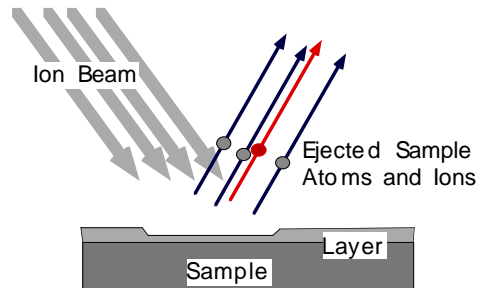


Auger & Analysis, page 46



## Secondary Ion Mass Spectroscopy (SIMS)

- **Ion Beam In**
  - Argon
  - Cesium
  - Oxygen
- **Sputtered Sample Out**
  - Inherently destructive
- **Analyze ions not atoms**
  - Major problem
  - Element variations
  - Conductivity variations



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## SIMS

- **Incredibly Sensitive for some materials**
  - Wide variation in sensitivity and detectability
    - Between elements and between samples and compounds (conductivity)
  - Sensitivity to 10<sup>15</sup>/cc for Phosphorus in Si
    - Vs. 10<sup>19</sup> or more for Auger/ESCA
- **Quantitation is difficult**

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## **SIMS Applications**

- Dopant Profiles
- Commercial services have best characterized instruments



## **Summary:**

### **Thin Film and Microanalysis**



## NanoCourses 2004, Section 4

# Practical Thin Film Analysis

## *What is It?*

### Atomic Force Microscopy (AFM) by John Treichler

Presented by the  
**CNF** Technical Staff  
for the education of CNF Users,  
Potential Users, and Industrial Sponsors

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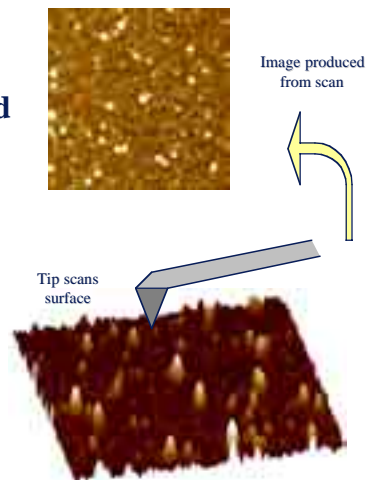


AFM, page 1

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## Overview of AFM

- Probe scans surface
- Surface interacts with probe
- Surface interaction is interpreted
  - Topography
  - Friction/Adhesion
  - Viscoelasticity
  - Electric/Magnetic fields
- Two modes used at Cornell
  - Contact
  - Tapping



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AFM, page 2

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## Advantages of AFM

- Nondestructive measurements
- Angstrom to nanometer resolution in x, y, and z
- Material properties
- Can better separate between height and material differences
- Data format offers easy analysis of many surface characteristics
  - Roughness
  - Arbitrary slice angle
  - Data may be filtered



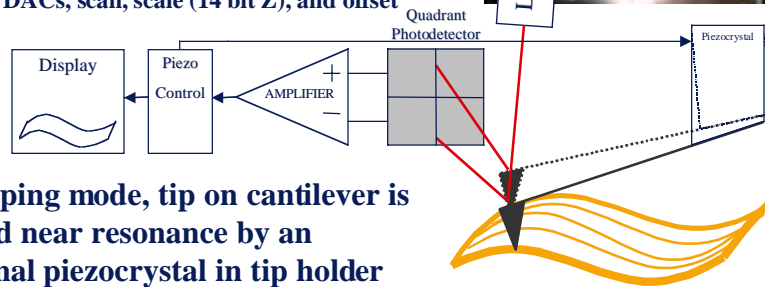
## Limitations of AFM

- Surface profile is always convoluted with tip profile
  - Measured side wall angle is limited to tip angle
  - It can be difficult to separate surface and tip profile
- Limited range in x, y and z
- Surfaces with loose particles can not be cleanly imaged
- Tip control is not closed loop on CNF AFM
  - Small slope changes can not be separated from piezocrystal nonlinearities
- High resolution scans can be time consuming



## Operation

- Tip deflection is sensed by reflected laser spot on photodetector
- Tip is moved in X, Y and Z by piezocrystals
  - Maximum range: 100um X & Y, 5um Z
  - 16 bit DACs, scan, scale (14 bit Z), and offset

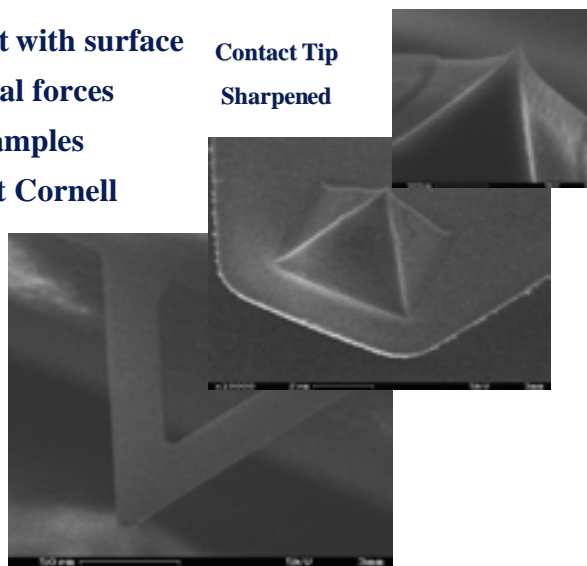


- For tapping mode, tip on cantilever is vibrated near resonance by an additional piezocrystal in tip holder

## Contact Mode

- Tip maintains contact with surface
- Can measure frictional forces
- Can damage softer samples
- Not in common use at Cornell

Contact Tip  
Sharpened



## Contact Mode

- Same sample
  - Data are: height, friction, and deflection

Height



Friction



Deflection



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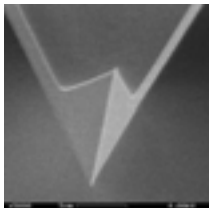


AFM, page 7

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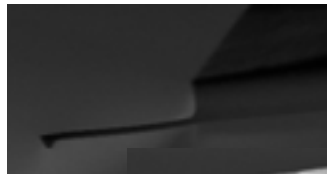
## Tapping Mode

- Tip on resonating cantilever makes intermittent contact with surface
- Amplitude of oscillation decreases as tip strikes surface
- Avoids friction and adhesion to surface fluid layer or trapped charges

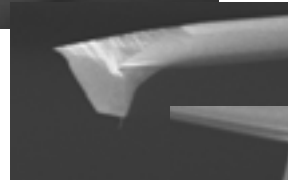


Supplied Tip

Radius is 5 to 10nm



Blade Tip  
Tapping Mode



X 30,000

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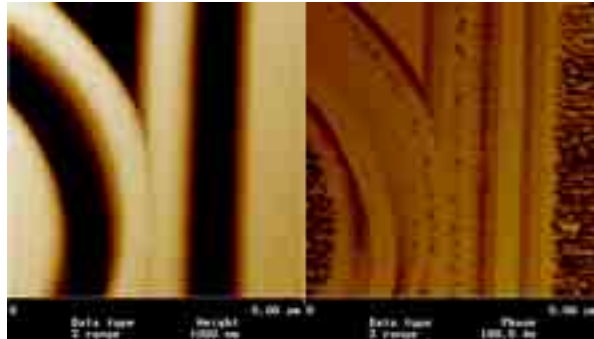


AFM, page 8

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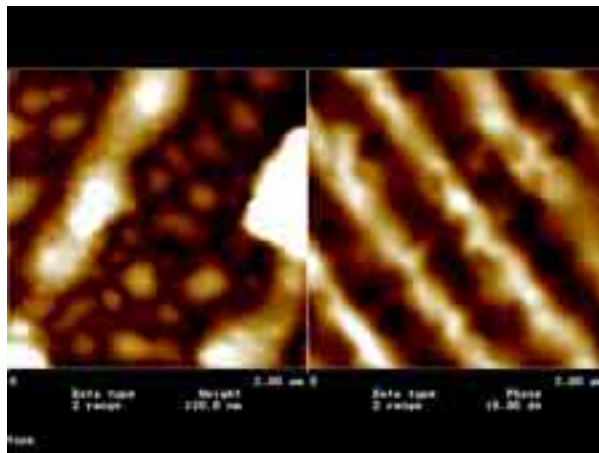
## Phase Plots

- Phase of cantilever relative to driving voltage can be plotted
- Phase plots can show variations in composition, adhesion, and viscoelasticity
- Surface can simultaneously be scanned for height and phase

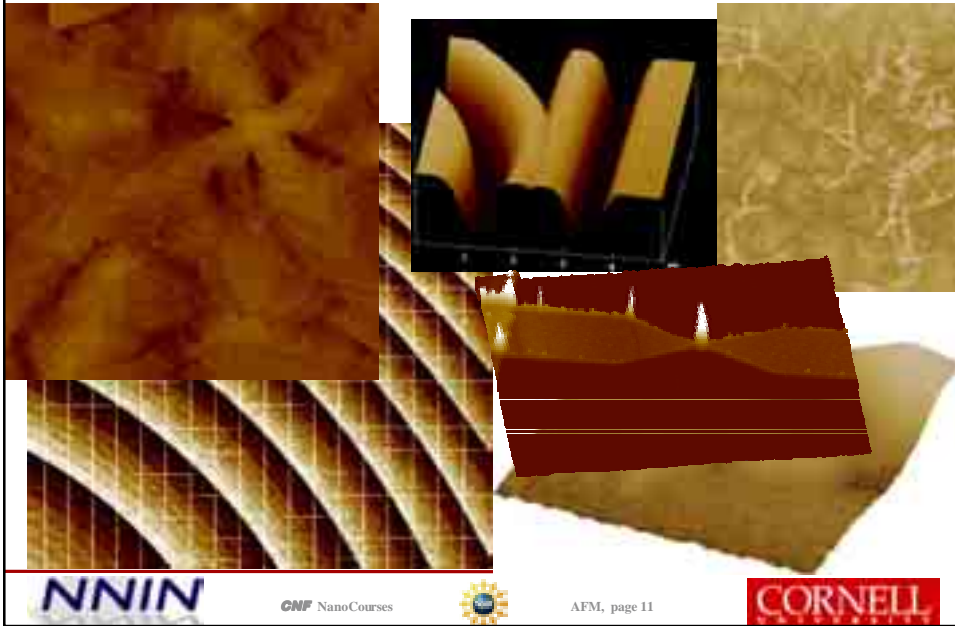


## Phase and Interleave Scanning

- Interleave scanning
  - Surface is scanned for height, tip is lifted, and surface is rescanned for phase
  - Used to image electric and magnetic fields



## Tapping Mode, Images From Users



## Imaging Problems

- Improper scan parameters
- Dirty or worn tip profile is convolved with sample's profile
- Dirty sample
- Vibration
- Sample too flexible

