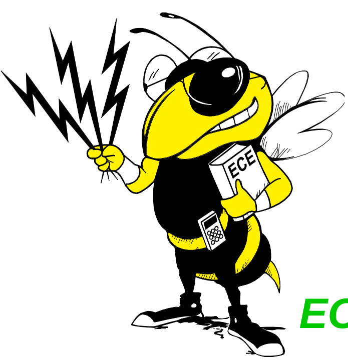
Generalized Magneto-Optical Ellipsometry

Nelson E. Lourenco

School of Electrical and Computer Engineering
85 5th Street, N.W., Georgia Institute of Technology
Atlanta, GA 30308 USA

nlourenco@mail.gatech.edu



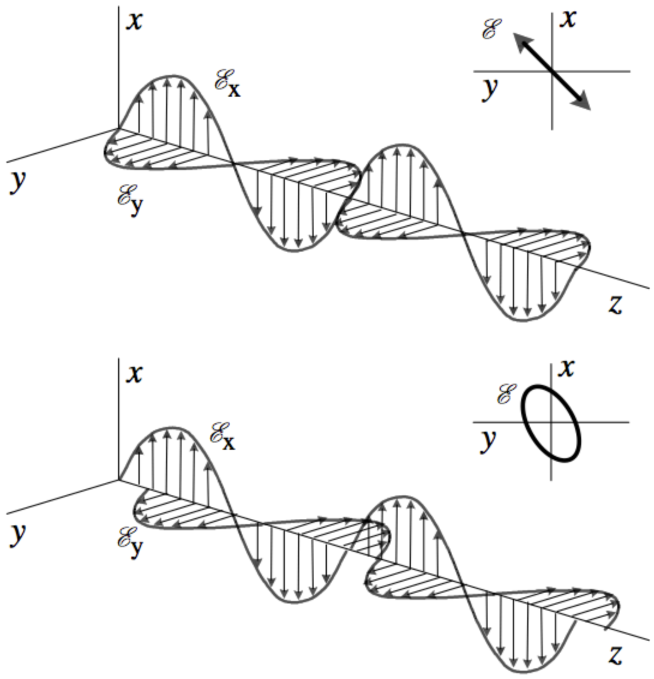
ECE 4813 - Semiconductor Materials and Device Characterization
Dr. Alan Doolittle

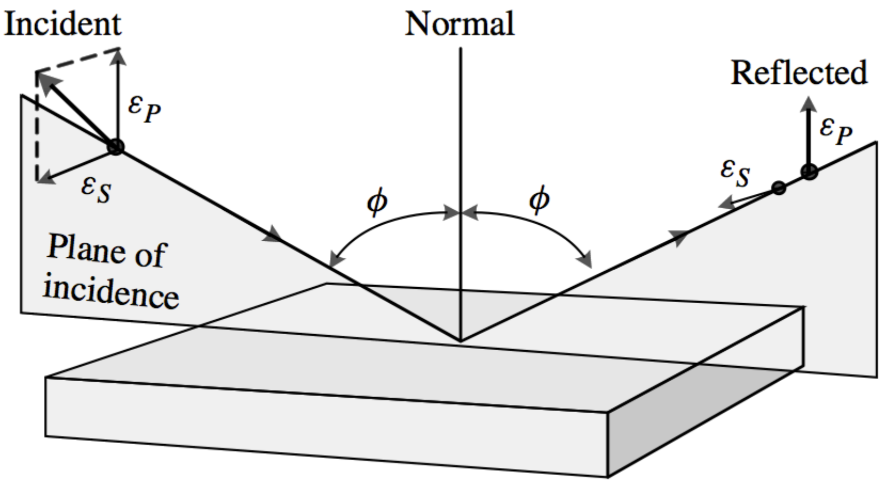
Outline

- **Fundamentals of Polarized Light**
- **Overview of Traditional Ellipsometry**
- **Magneto-Optical Characterization**
- **Generalized Magneto-Optical Ellipsometry**
- **Vector Generalized Magneto-Optical Ellipsometry (Vector Magnetometer)**

Light Polarization

- **Light can be fully polarized, partially polarized, unpolarized**
  - Fully Polarized Light
    - Linearly Polarized
    - Elliptically Polarized

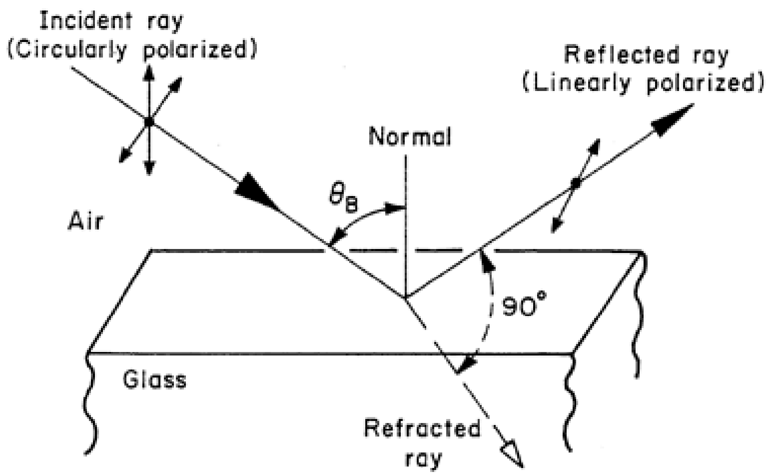


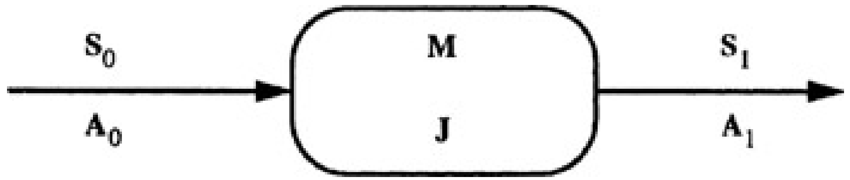


D.K. Schroder “Semiconductor Device and Material Characterization, 3rd Ed.”

Jones Calculus

- **Developed by Dr. Robert Clark Jones**
  - Developed between 1941-1956 at Harvard / Polaroid Corporation
  - Mathematical model for describing polarized coherent light
  - Randomly polarized, partially polarized, and incoherent light cannot be modeled using Jones Calculus
    - Mueller Calculus (Stokes Vectors)





G.G. Fuller "Optical Rheometry of Complex Fluids, 1st Ed."

Jones Calculus contd.

- **Polarized light represented by Jones Vector**
  - Linearly Polarized Light

    X-Direction: $\begin{pmatrix} 1 \\ 0 \end{pmatrix}$ Y-Direction: $\begin{pmatrix} 0 \\ 1 \end{pmatrix}$

  - Circular Polarized Light

    Left-Hand (LHCP): $\frac{1}{\sqrt{2}}\begin{pmatrix} 1 \\ i \end{pmatrix}$ Right-Hand (RHCP): $\frac{1}{\sqrt{2}}\begin{pmatrix} 1 \\ -i \end{pmatrix}$

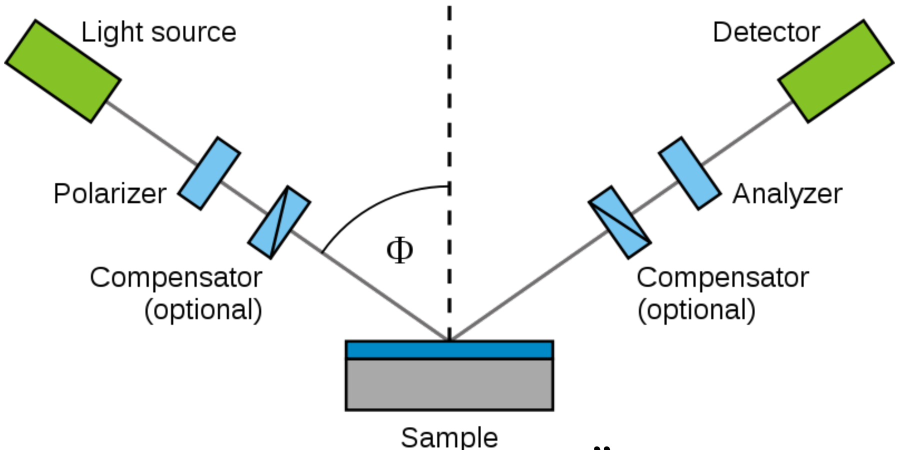
- **Linear Optical Element represented by Jones Matrix**
  - Horizontal Linear Polarizer: $\begin{pmatrix} 1 & 0 \\ 0 & 0 \end{pmatrix}$
  - Vertical Linear Polarizer: $\begin{pmatrix} 0 & 0 \\ 0 & 1 \end{pmatrix}$
  - Right Circular Polarizer: $\frac{1}{2}\begin{pmatrix} 1 & i \\ -i & 1 \end{pmatrix}$

Traditional Ellipsometry

- **Interested in optical parameters of thin films and/or semiconductor substrates**
  - Air (n_0) – Semiconductor (n_1 – jk_1) Interface
  - Air (n_0) – Thin Film (n_1) – Semiconductor (n_2 – jk_2) Interface
  - Complex Index of Refraction: $\tilde{n} = n - jk$
    - n: phase velocity in medium
    - k: absorption loss through medium

$$\rho = \frac{R_p}{R_s} = \tan(\Psi)e^{j\Delta}$$

- **Example: Null Ellipsometry (PCSA)**



R.M. Azzam "Ellipsometry and Polarized Light, 1st Ed."

Traditional Ellipsometry

- **Applications**
  - Optical Properties of Materials
  - Film Thickness
  - Film Deposition / Etching
    - Process Control
    - In-situ Monitoring

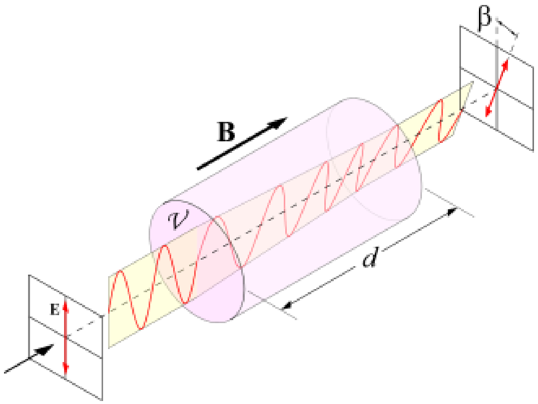
- **GT MiRC Cleanroom**
  - Woollam Ellipsometer
  - Plas-Mos Ellipsometer

Photos courtesy of GT Microelectronics Research Center

M-O Motivation

- **Traditional Ellipsometry determine optical properties, but there are also magneto-optical properties**
  - Magneto-Optical Storage Devices
    - Ultra thin-film magnetism
  - Ferromagnetic Materials
    - Rare-Earth Magnets
    - Ferrofluids

- **Faraday Effect**
  - Occurs for light propagating through magnetic fields and magnetic materials
  - Rotation of the plane of polarization

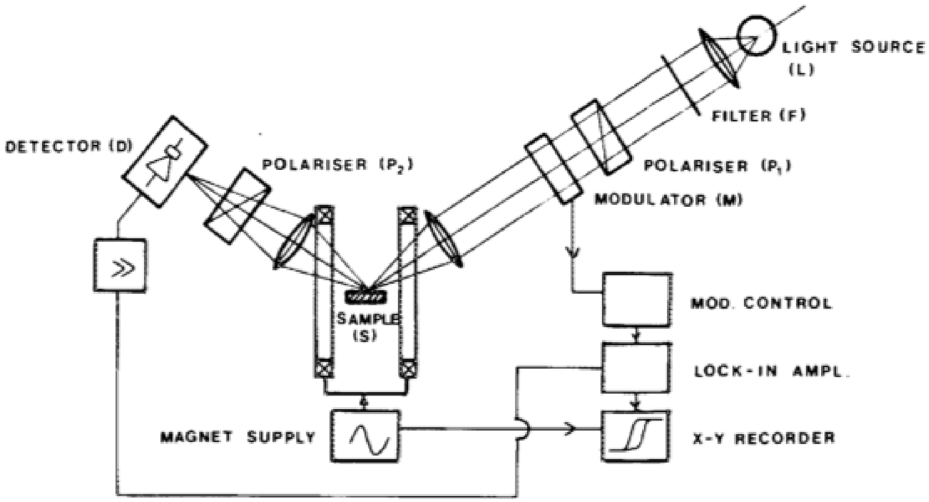


S. Mancuso `"Faraday Rotation and Models" (2000)

M-O Characterization

- **Full Magneto-Optical Characterization Process (2 Steps)**
  - Optical Characterization
    - ñ = n – jk
  - Magneto-Optical Characterization
    - $Q = Q_r - jQ_i$ (Complex Magneto-Coupling Constant)
    - Magnetization Orientation

- **Can we simplify this setup?**

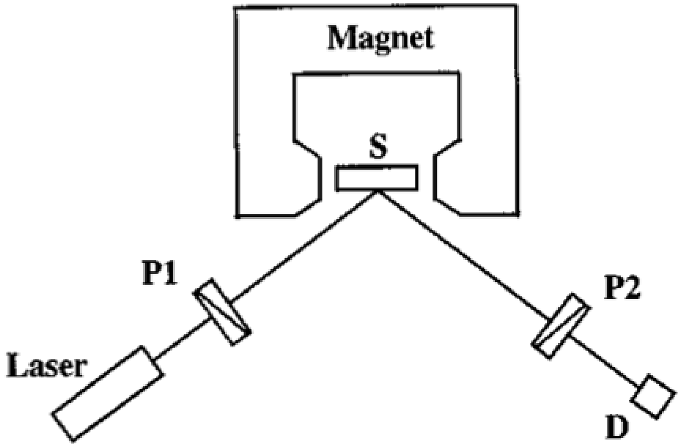


Magneto-optical Ellipsometer
P. Q. J. Nederpel & J. W. D. Martens
January 3rd, 1985

GMOE

- **Developed by Andreas Berger and Matthew Pufall at University of California – San Diego (1997)**
  - Complete magneto-optical characterization
  - Combine two-step process into one measurement

- **Measurement Setup**
  - HeNe Laser (λ=632.8 nm)
  - Rotatable Polarizers (Glan-Taylor)
  - Torroidal Ferrite Magnet
  - Photodiode Detector



A. Berger "Generalized Magneto-Optical Ellipsometry," (1997)

GMOE contd.

- **Electric Field Vector at Detector**

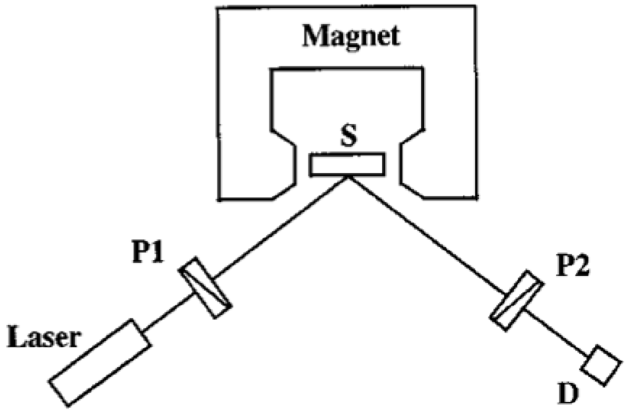
$$\mathbf{E}_D = \mathbf{P}_2 * \mathbf{R} * \mathbf{P}_1 * \mathbf{E}_L$$

- **Glan-Taylor Polarizers defined by Jones matrix**

$$\mathbf{P} = \begin{bmatrix} \cos^2(\theta) & \sin(\theta)\cos(\theta) \\ \sin(\theta)\cos(\theta) & \sin^2(\theta) \end{bmatrix}$$

- **Reflection (Jones Matrix) of sample**

$$\mathbf{R} = \begin{bmatrix} r_s & \alpha \\ -\alpha & r_p + \beta \end{bmatrix} = r_p \begin{bmatrix} \tilde{r}_s & \tilde{\alpha} \\ -\tilde{\alpha} & 1 + \tilde{\beta} \end{bmatrix} = r_p \tilde{\mathbf{R}}$$



- **Light Intensity I at detector D**

$$\mathbf{I} = \mathbf{E}_D \cdot \mathbf{E}_D^*$$

**** Linear approximation: α and β switch signs at magnetization reversal ****

GMOE contd.

- **Fractional intensity change at photodetector, δI/I**

$$\frac{\delta I}{I} = 4\,\frac{B_1 f_1 + B_2 f_2 + B_3 f_3 + B_4 f_4}{f_3 + B_5 f_5 + 2B_6 f_4}$$

$$f_1(\theta_1,\theta_2) = \sin^2(\theta_1)\sin(\theta_2)\cos(\theta_2) - \sin^2(\theta_2)\sin(\theta_1)\cos(\theta_1)$$

$$f_2(\theta_1,\theta_2) = \cos^2(\theta_2)\sin(\theta_1)\cos(\theta_1) - \cos^2(\theta_1)\sin(\theta_2)\cos(\theta_2)$$

$$f_3(\theta_1,\theta_2) = \sin^2(\theta_1)\sin^2(\theta_2)$$

$$f_4(\theta_1,\theta_2) = \sin(\theta_1)\cos(\theta_1)\sin(\theta_2)\cos(\theta_2)$$

$$f_5(\theta_1,\theta_2) = \cos^2(\theta_1)\cos^2(\theta_2)$$

$$B_1 = \mathrm{Re}(\tilde{\alpha}) \qquad B_2 = \mathrm{Re}(\tilde{r}_s\tilde{\alpha}^*)$$

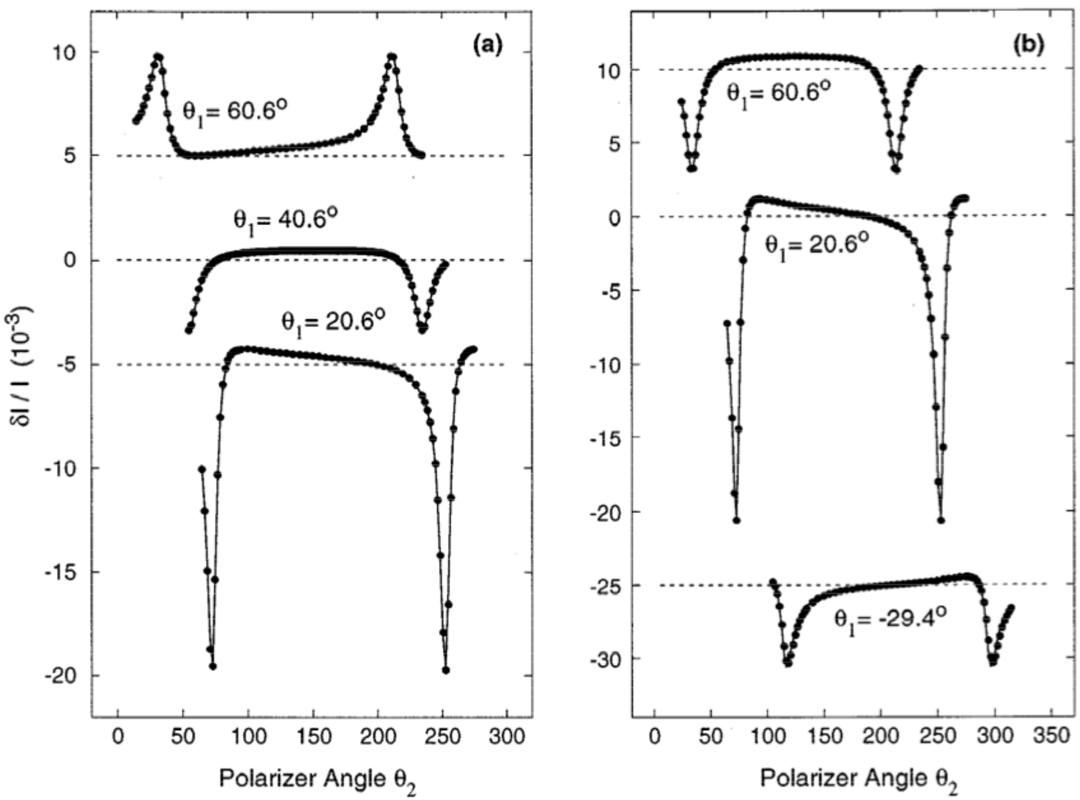
$$B_3 = \mathrm{Re}(\tilde{\beta}) \qquad B_4 = \mathrm{Re}(\tilde{r}_s\tilde{\beta}^*)$$

$$B_5 = |\tilde{r}_s|^2 \qquad B_6 = \mathrm{Re}(\tilde{r}_s)$$

A. Berger "Generalized Magneto-Optical Ellipsometry," (1997)

GMOE contd.

- **Example Data (from Berger & Pufall)**



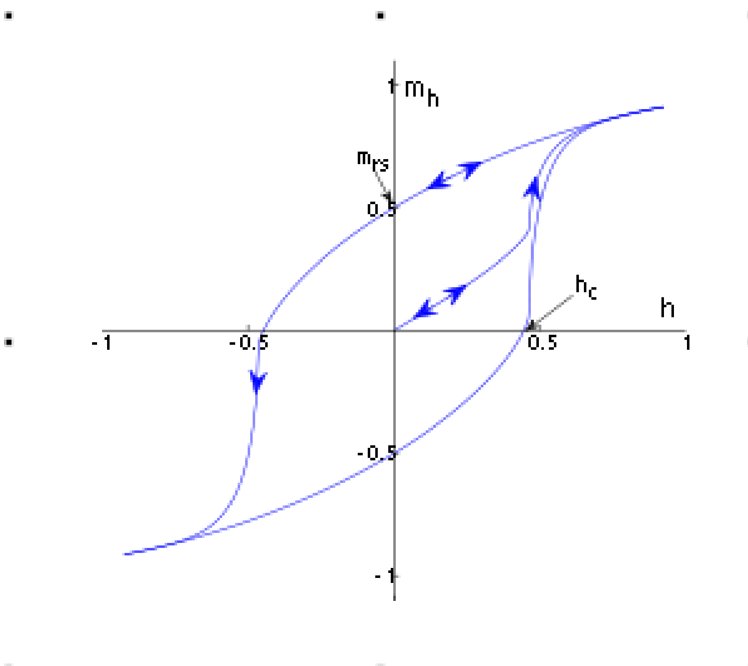
	Data set (a)	Data set (b)
B_1	$(-5.034\pm0.148)\times10^{-5}$	$(-2.727\pm0.196)\times10^{-5}$
B_2	$(1.850\pm0.023)\times10^{-4}$	$(1.232\pm0.028)\times10^{-4}$
B_3	$(1.623\pm0.169)\times10^{-5}$	$(1.980\pm0.044)\times10^{-4}$
B_4	$(-3.842\pm0.210)\times10^{-5}$	$(-4.694\pm0.074)\times10^{-4}$
B_5	1.4003 ± 0.0035	1.4279 ± 0.0048
B_6	-1.1405 ± 0.0011	-1.1511 ± 0.0015

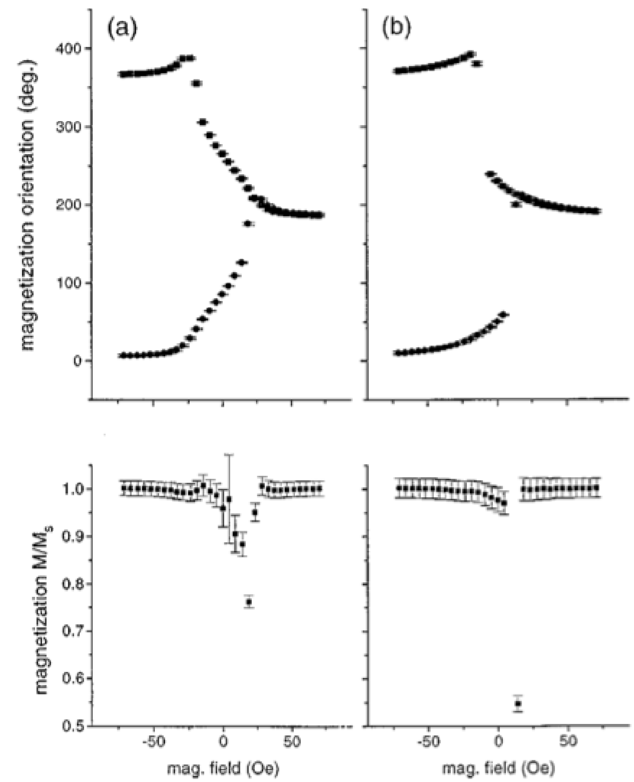
	Data set (a)	Data set (b)	Difference
n	2.403(±2.4%)	2.439(±3.3%)	1.5%
k	3.693(±1.4%)	3.561(±1.9%)	3.6%
Q_r	7.346×10^{-3}(±3.5%)	6.781×10^{-3}(±4.4%)	8.0%
Q_i	7.781×10^{-3}(±5.5%)	7.926×10^{-3}(±7.3%)	1.8%
ϕ	3.54°(±0.34°)	47.20°(±1.47°)	43.66°

A. Berger "Generalized Magneto-Optical Ellipsometry," (1997)

Vector GMOE

- **Generalized Magneto-Optical Ellipsometry can be used as a vector magnetometer**
  - Andreas Berger and Mathew Pufall
  - Measurement of H vs. M dependence





A. Berger "Quantitative Vector Magnetometry using Generalized Magneto-Optical Ellipsometry," (1997)

References

[1] D.K. Schroder, “Optical Characterization,” in *Semiconductor Material and Device Characterization*, 3rd ed. 2006

[2] G.G. Fuller, *Optical Rheometry of Complex Fluids*, 1st ed. 1995

[3] R.M.A. Azzam, *Ellipsometry and Polarized Light*, 1st ed. 1988

[4] A. Berger, “Generalized Magneto-Optical Ellipsometry,” *Appl. Phys. Lett.*, vol. *71*, no. *7*, pp. *965-967*, August, 1997.

[5] A. Berger, “Quantitaive Vector Magnetometry using Generalized Magneto-Optical Ellipsometry,” *J. Appl. Phys.*, vol. *85*, no. *8*, pp. *4583-4585*, April, 1999.

[1] D.K. Schroder, “Optical Characterization,” in *Semiconductor Material and Device Characterization* , 3rd ed. 2006

Scanning Probe Microscopy (SPM)

Brendan Gunning

ECE 4813

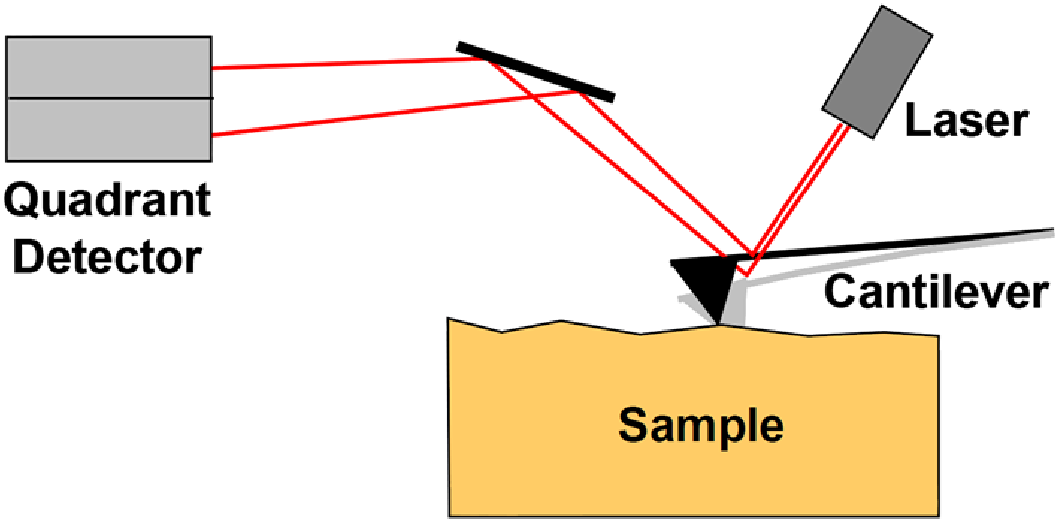
SPM Flavors

- AFM (Atomic Force Microscopy)
- C-AFM (Conductive Atomic Force Microscopy)
- BEEM (Ballistic Electron Emission Microscopy)
- EFM (Electrostatic Force Microscopy)
- KPFM (Kelvin Probe Force Microscopy)
- NSOM (Near-field Scanning Optical Microscopy)
- SCM (Scanning Capacitance Microscopy)
- STM (Scanning Tunneling Microscopy)
- And more…

Conductive Atomic Force Microscopy (C-AFM)

Conductive AFM

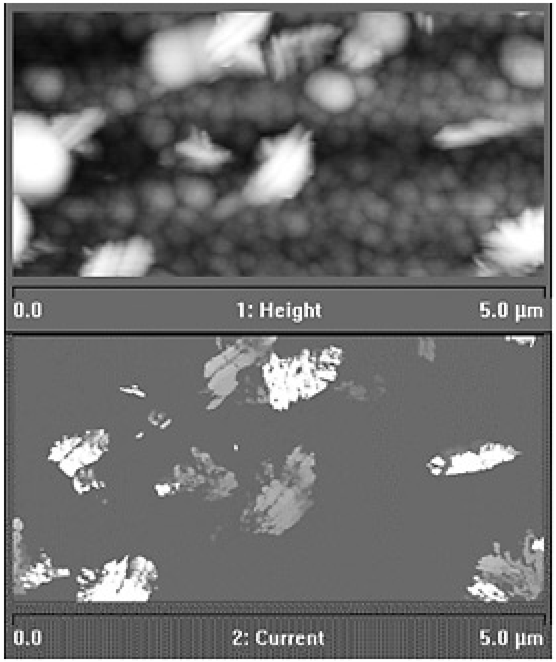
- Like AFM… but it's conductive (duh)
- Cantilever/tip is coated in conductive film (Pt, Pt-Ir, etc)
- Apply bias to tip, ground sample → contact…
- Current flows



- And… you can still get topography!

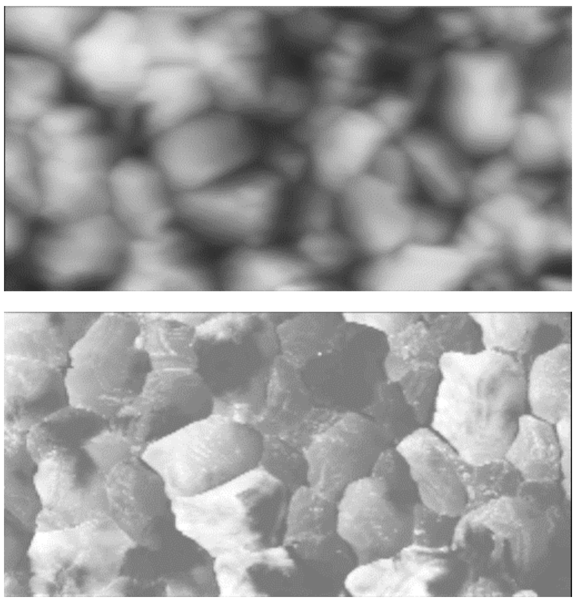
Current Mapping

- Scan across surface
- Areas with different conductivity will have different currents
- Map current like you do with topography

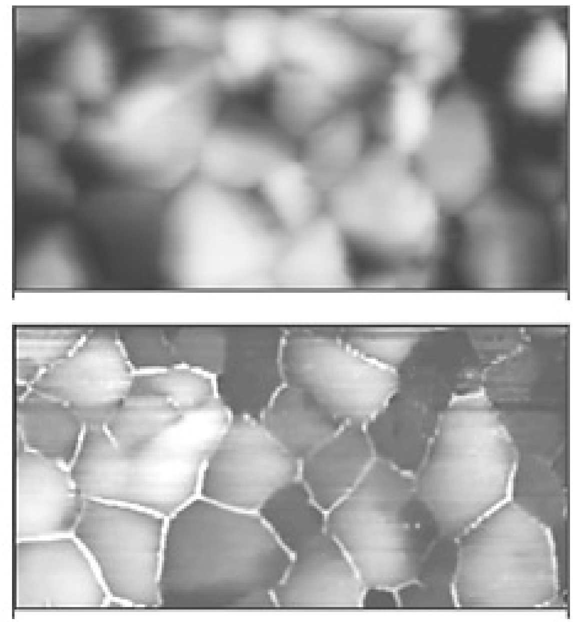


http://www.nrel.gov/pv/measurements/conductive_atomic.html

Processing Characterization



as-grown CdTe/CdS Solar Cell

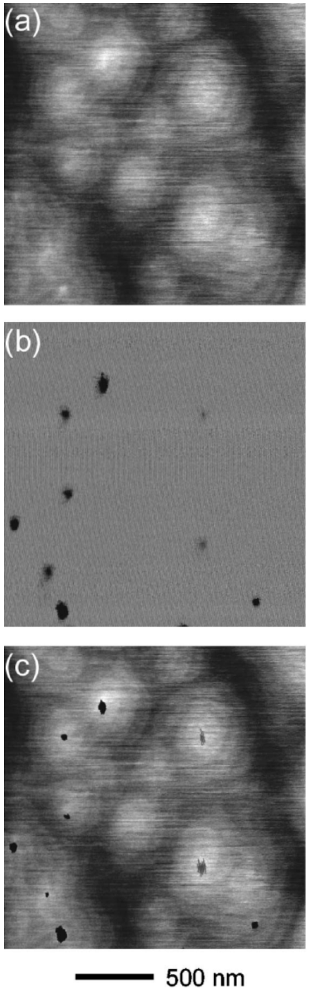


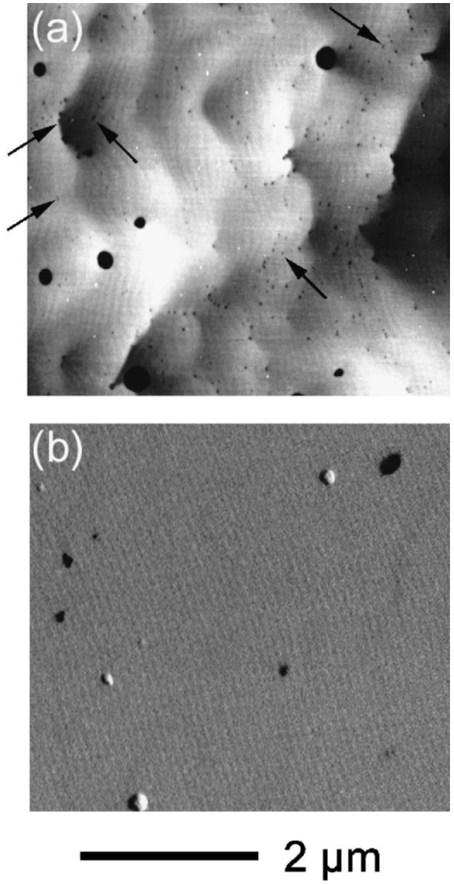
CdTe/CdS Solar Cell
after bromine-methanol etch

Moutinho et al., "*Conductive Atomic Force Microscopy Applied to CdTe/CdS Solar Cells*", *2004.*

More Current Mapping

- Mapping the current can shed light on things like:
  - Defects
  - Composition
  - Contamination

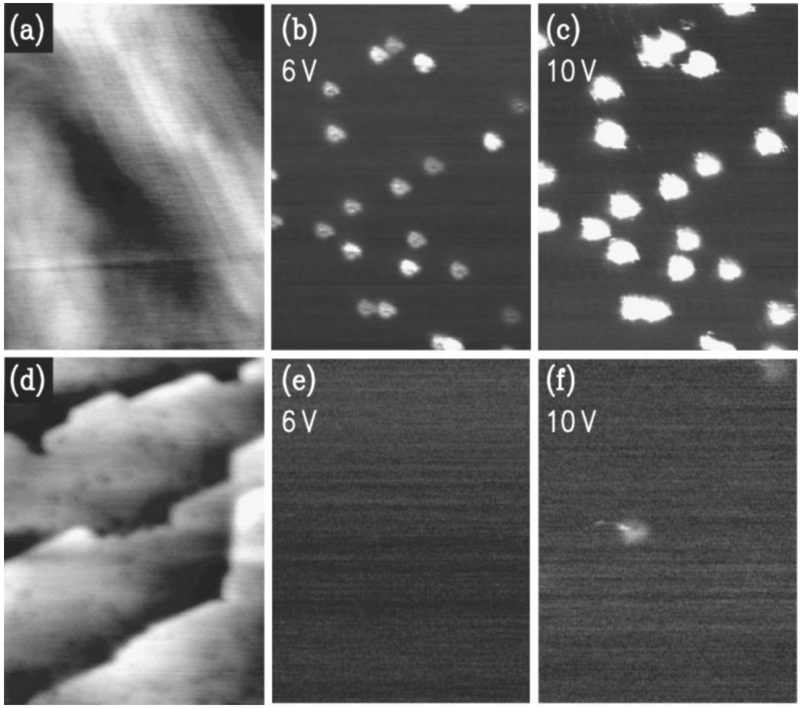




Hsu et al., *"Direct imaging of reverse-bias leakage through pure screw dislocations in GaN films grown by molecular beam epitaxy on GaN templates", 2002.*

More Current Mapping

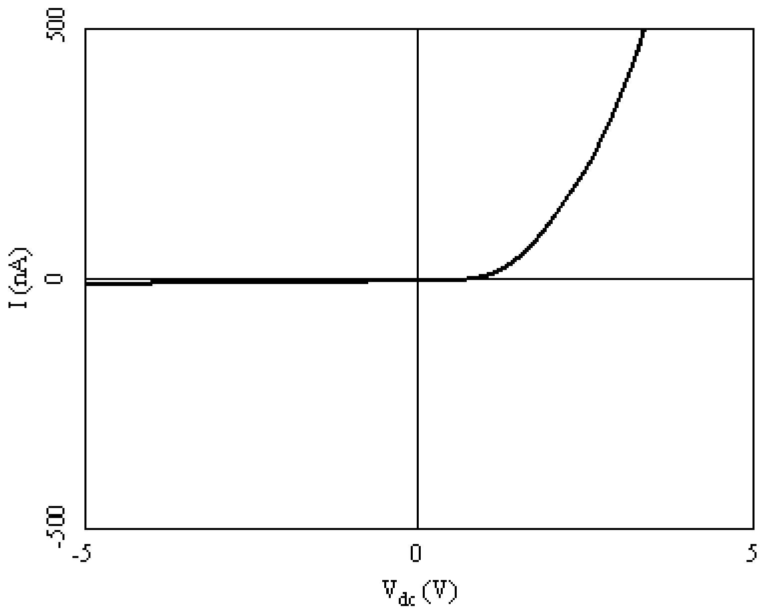
- Top samples are GaN grown with just N_2
- Bottom samples grown with H_2 and N_2
- H_2 passivated dangling bonds, reducing electrical activity



Dong et al., *"Effects of hydrogen on the morphology and electrical properties of GaN grown by plasma-assisted molecular-beam epitaxy"*, 2005.

Scanning Current-Voltage Microscopy (SIVM)

- Tip is held at one x-y location, contacting surface
- Sweep voltage → measure current flow



I-V curve of CdTe/CdS taken by conductive AFM

Moutinho et al., "*Conductive Atomic Force Microscopy Applied to CdTe/CdS Solar Cells*", *2004.*

Unintended Side-Effects of C-AFM

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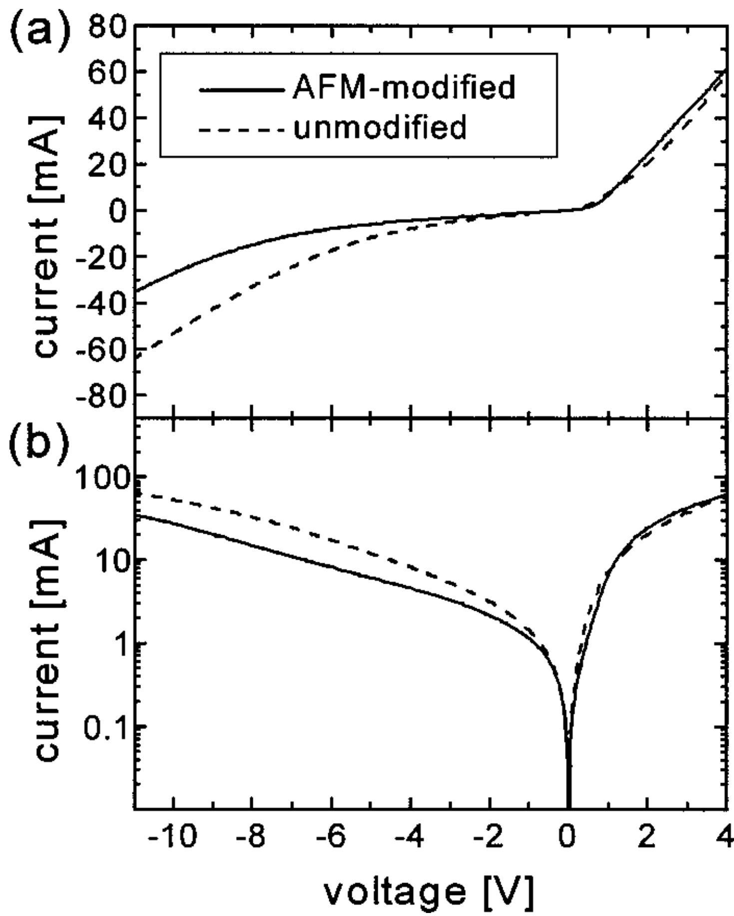
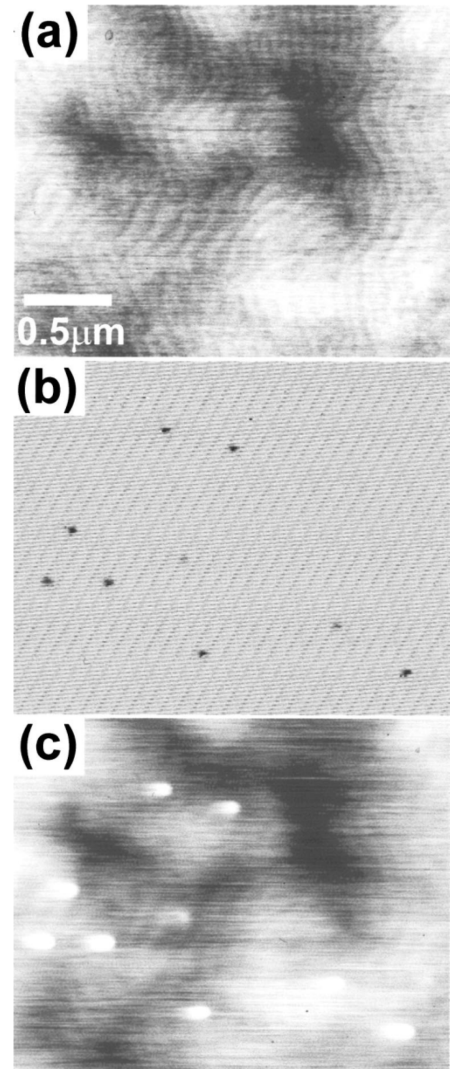
- er
- en

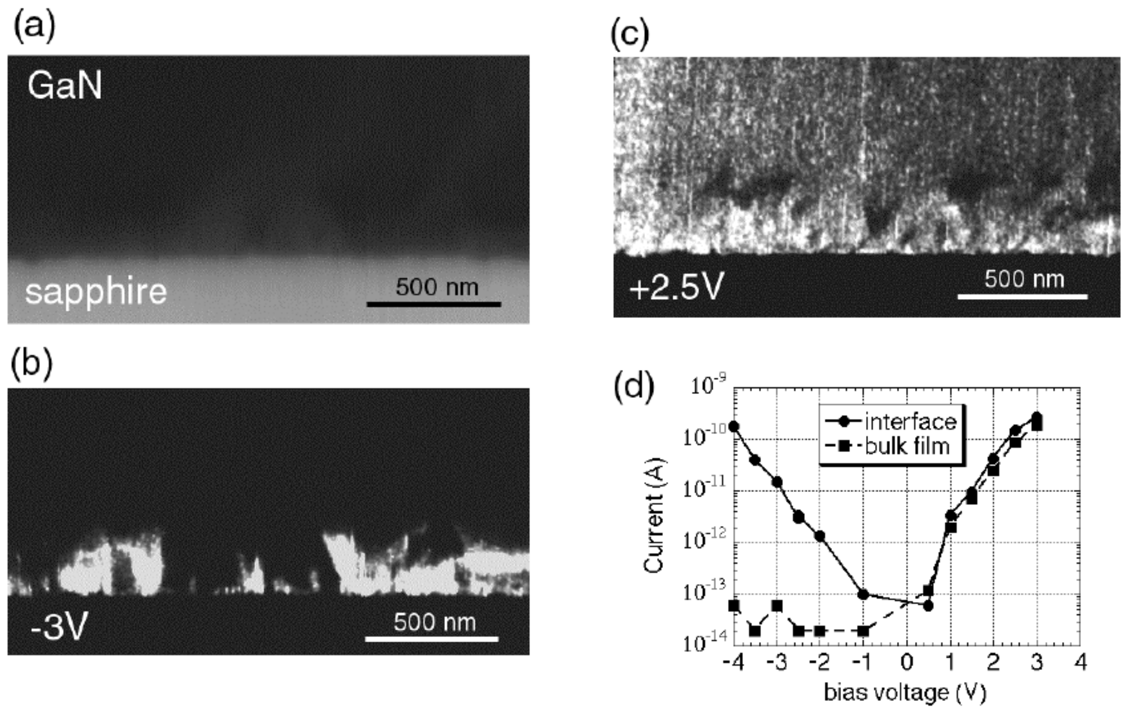




Miller et al., *"Reduction of reverse-bias leakage current in Schottky diodes on GaN grown by molecular-beam epitaxy using surface modification with an atomic force microscope"*, 2002.

Cross Sectional C-AFM

- Look at cross section to probe:
  - Multiple layers
  - Interfaces

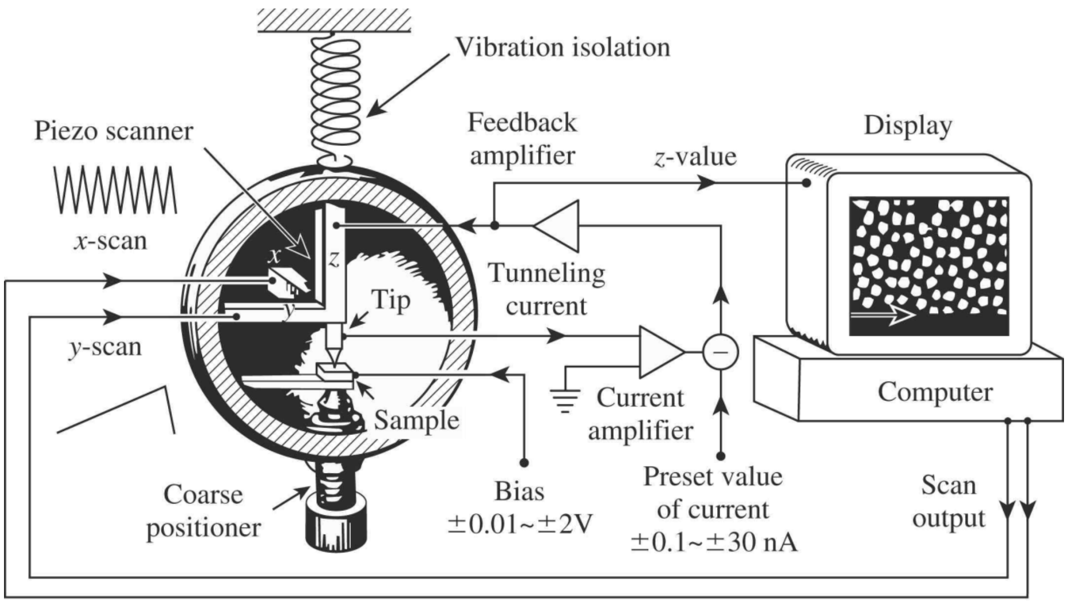


Hsu et al., *"Scanning Probe Studies of Defect Dominated Electronic Transport in GaN"*

Scanning Tunneling Microscopy (STM)

STM Apparatus and Procedure

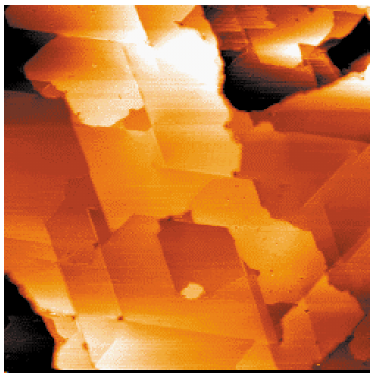
- Coarse control brings tip (W, Pt-Ir, or Au) close to sample
- Once close enough, z-piezo brings tip within tunneling range (~5Å)
- Z-piezo steps down until preset tunneling current is reached
- As the tip rasters, changes in topography will increase/decrease current
- Feedback raises/lowers tip to maintain constant current
- The distance the tip was raised/lowered forms the topography image



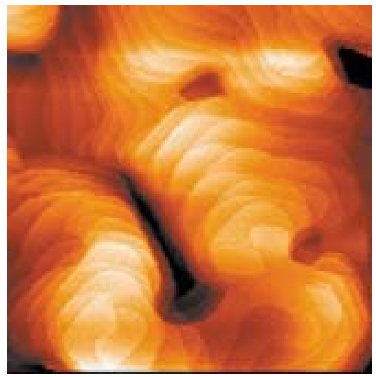
STM Images

GaN on 6H-SiC

Regular (0001)

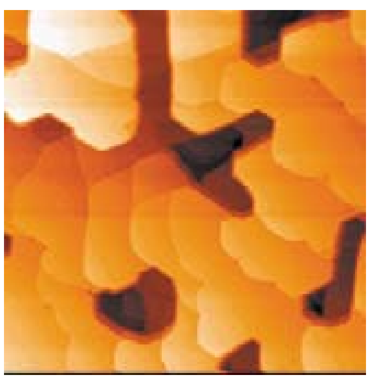


200 nm

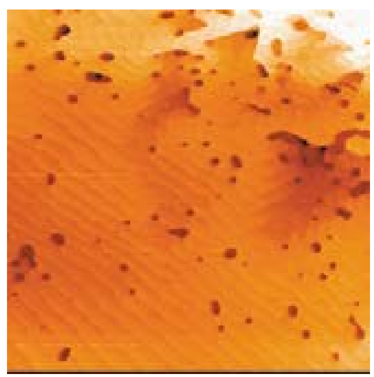


1 μm

0.4º off-cut



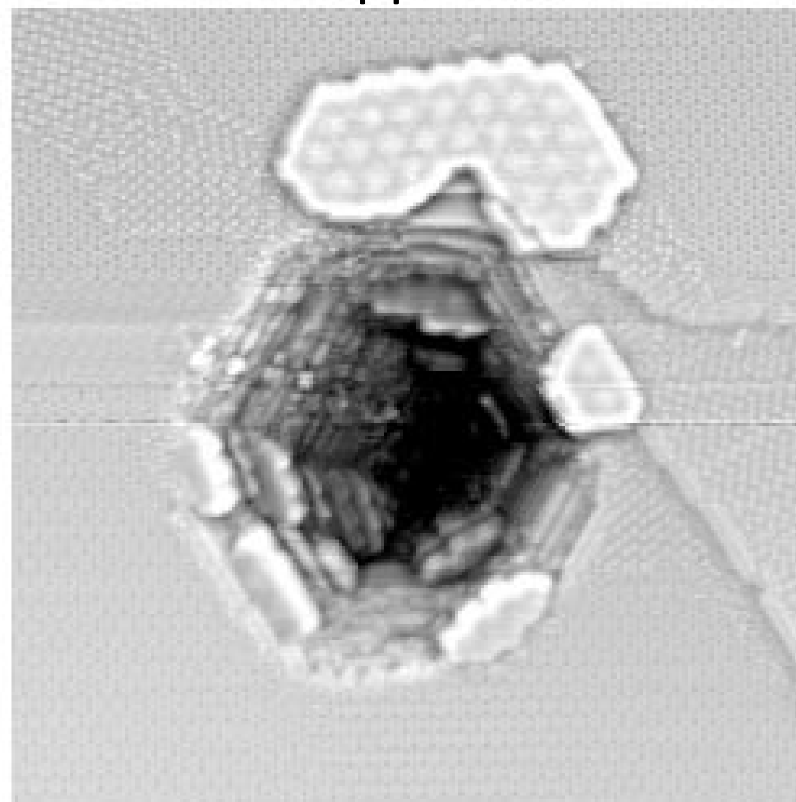
500 nm



2 μm

Cui et al., *“Suppression of Spiral Growth in Molecular Beam Epitaxy of GaN on Vicinal 6H-SiC (0001)”, 2001.*

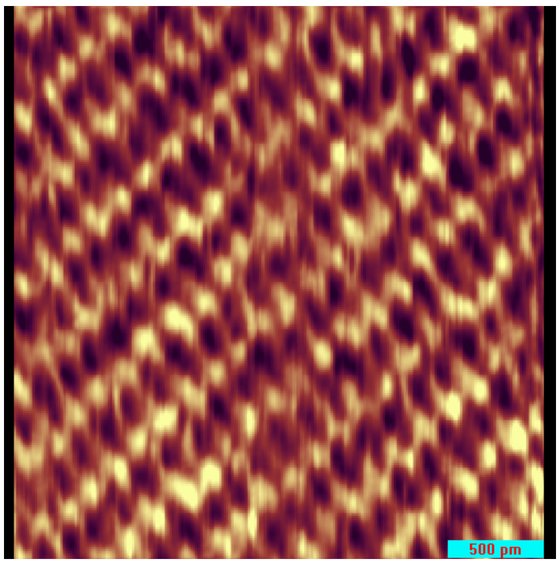
Pit in N-polar GaN on Sapphire



Feenstra et al., “Reconstruction of GaN and InGaN Surfaces”, 2000.

Our STM Images

HOPG – Highly Ordered Pyrolytic Graphite (3nm scan size)



Dirty, non-annealed Gold (726nm scan size)

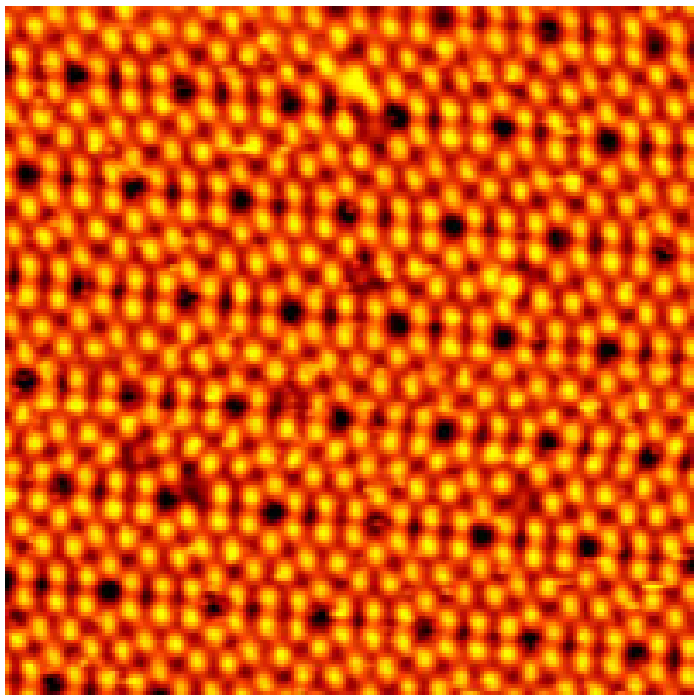
Me! 2010

Empty vs. Filled States

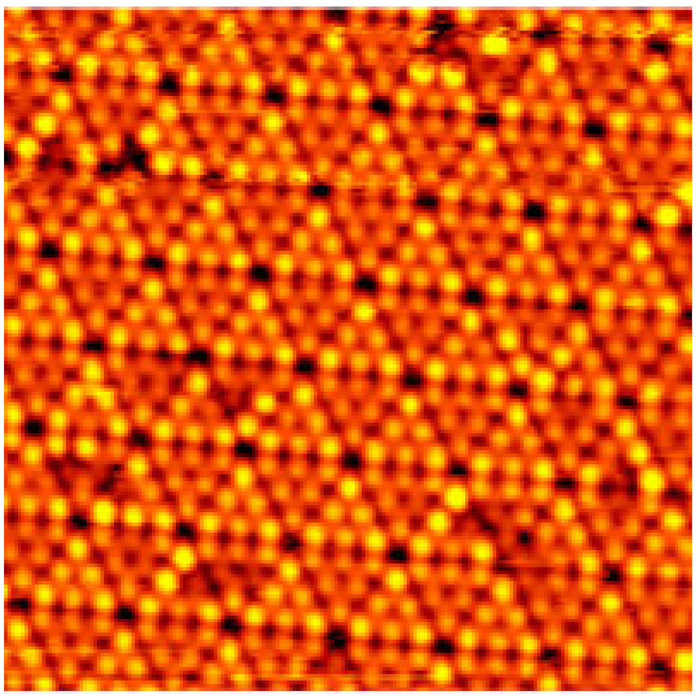
7x7 surface reconstruction of Si (111) – 1.5nA tunneling current

15nm

15nm



+2V bias = Empty states



-2V bias = Filled states

http://physics.usask.ca/~mitchell/facilities.html

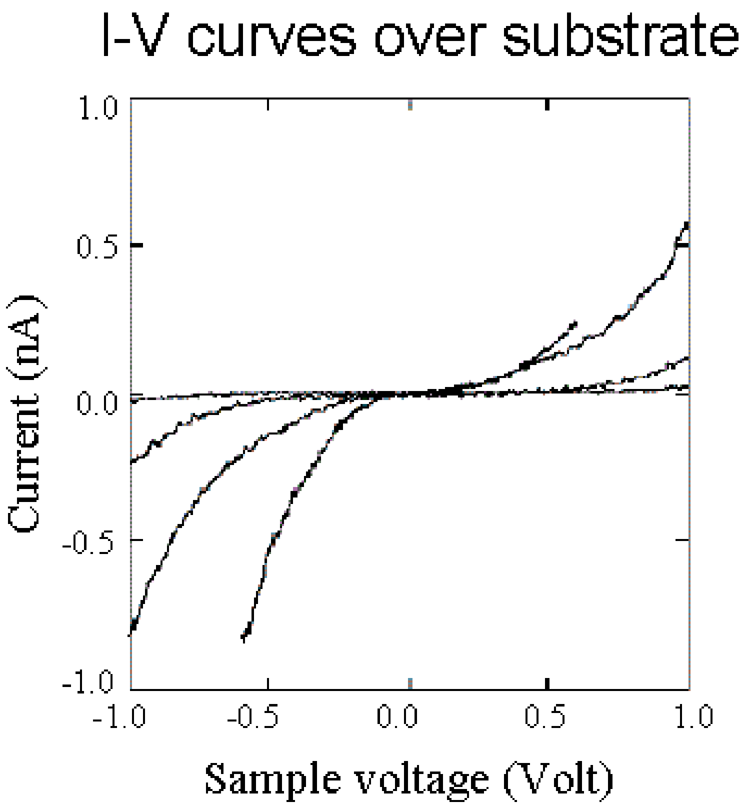
What else can we do?

- Rather than just sweeping across the surface with a set bias…

- STS sweeps the bias at a fixed x-y-z position

- Generates a "local" I-V curve, representing the integrated density of states at that position as a function of energy

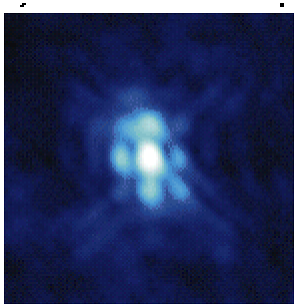
- The derivative of this I-V curve, dI/dV, can tell us even more…

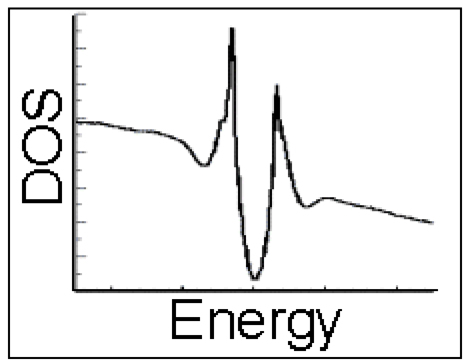


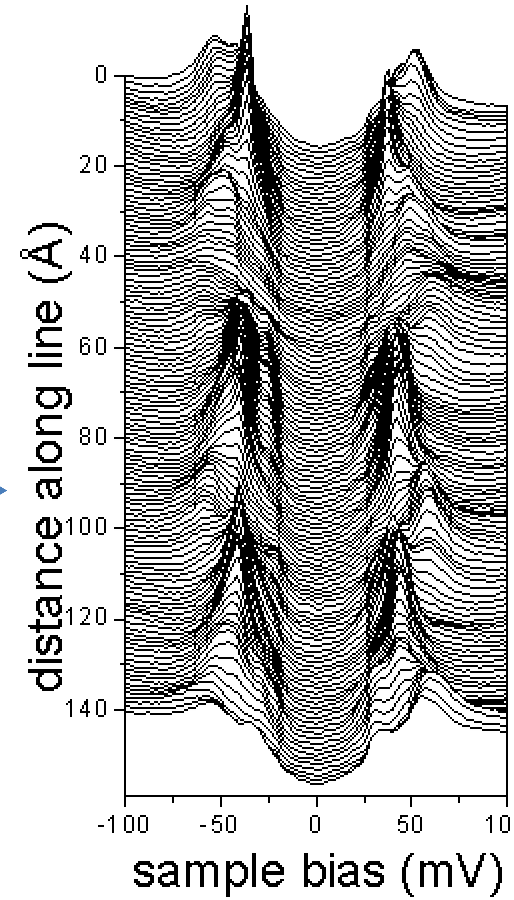
Scanning Tunneling Spectroscopy

- Instead of the integrated density of states, the dI/dV spectrum shows us the <u>actual</u> density of states at that location as a function of energy

- Can use the density of states data to create a "map" across a sample area at a chosen energy







Conclusion

- SPM covers a huge variety of more specific characterization methods
- Each one of these characterization methods is useful in and of itself

Whether it's C-AFM, STM, or some other method…

SPM is an <u>extremely</u> useful and powerful characterization tool

Light Beam Induced Current/Voltage

Guy Raz

ECE 4813 – Dr. Alan Doolittle

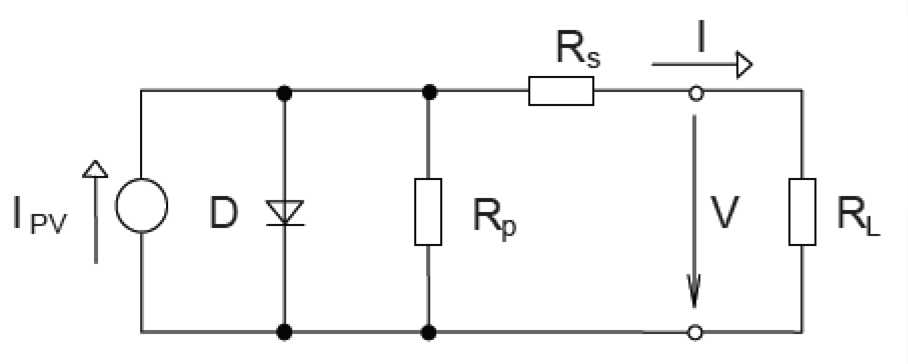
Fall 2011

Georgia Institute of Technology

Motivation

- The development and production of polycrystalline solar cells creates a necessity for analysis with high spatial resolution
- Contactless probes can be used to examine:
- EBIC - has been widely applied
- LBIC is more appropriate for solar cells
  - for study of defects
  - LBIV – measuring V_{OC} instead of J_{SC}

Solar Cell Modeling



- For a short circuit current (LBIC method) where V=0

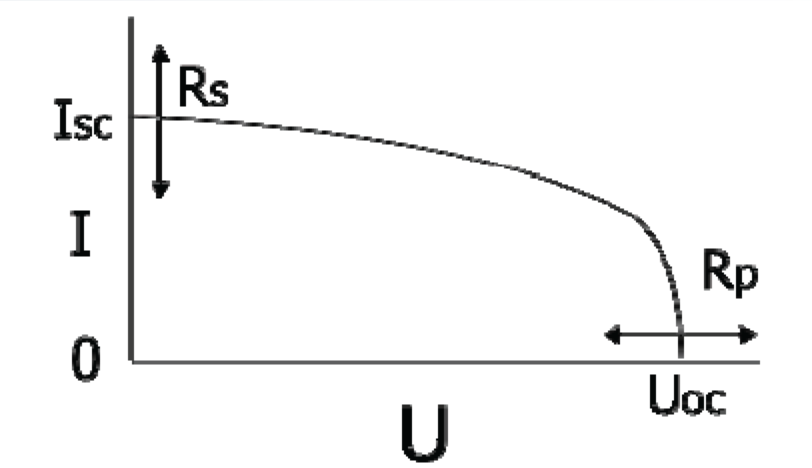
$$I_{sc} = AJ_{PV} - I_{01}[\exp(e\frac{R_s I}{kT}) - 1] - I_{02}[\exp(e\frac{R_s I}{2kT}) - 1] - \frac{R_s I}{R_p}$$

- For open circuit voltage (LBIV method)

$$V_{oc} = \frac{2kT}{e}\ln(\frac{-I_{02} + \sqrt{I_{02}{}^2 + 4I_{01}(I_{02} + I_{01} + AJ_{PV})}}{2I_{01}})$$

Figure and Equations from: Salinger, Benda & Machacek

Solar Cell Modeling (cont.)



- LBIC measurements are made around V=0
  - Slope is low, current changes slowly
- LBIV made around I = 0

Figure and Equations from: Salinger, Benda & Machacek

Introduction

- When a light beam strikes a semiconductor, it will generate electron-hole pairs (EHPs) within the beam's interaction volume.
- These EHPs will be separated by drift due to the internal electric field.
- The E/Hs can be collected at the contacts of the object.
- Amplifying and analyzing these measurements show variations in generation, drift and recombination which can be measured and displayed.

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Apparatus

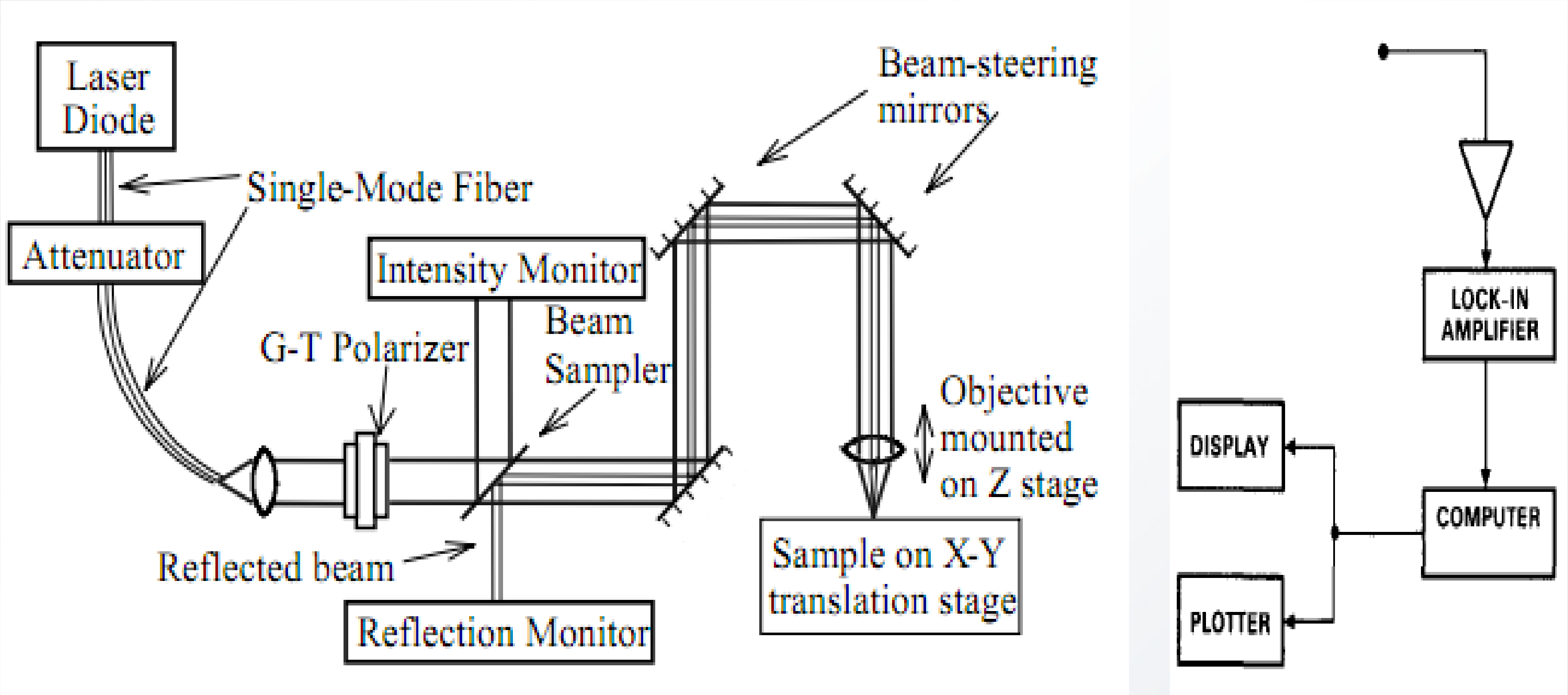

Figure from: Hiltner & Sires

Quantum Efficiency

- QE is the ratio of the number of charge carriers collected by the solar cell to the number of photons shinning on the solar cell.

$$IQE = \frac{1}{1-R}\frac{I_{sc}/e}{P_L/(h\cdot c/\lambda)}$$

- EQE
- I_{SC} is dependent on the amount of absorbed light
  - Corrected by first factor

Photon Penetration/Absorption

- Energy of a photon depend only on its wavelength by

$$E = \frac{h \cdot c}{\lambda}$$

- The depth of penetration depend on Energy (KeV) and material. (silicon example)

Wavelength (nm)	Penetration Depth (μm)
400	0.19
500	2.3
600	5.0
700	8.5
800	23
900	62
1000	470

http://micro.magnet.fsu.edu/primer/java/digitalimaging/ccd/quantum/

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Diffusion Length

- Photo-induced current decay is dependant on the relative thickness of sample
- It is necessary to consider two cases when analyzing LBIC intensity :
  - Thick Sample Cases ($W > 4L_b$)
  - Thin Sample cases ($W < 4L_b$)

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Sample Cases

- Thick Sample Cases ($W > 4L_b$)
  - EBIC intensity as a function of distance x from the barrier, in a semiconductor of semi-infinite thickness and far from the collector edge ($x \gg L_b$)

$$I(x) = C \exp(-\frac{x}{L_b}) x^{-n}$$

- Thin Sample Cases ($W < 4L_b$)
  - In thin samples, the influence of the front and rear surface recombination becomes very important

$$I(x) = I_0 \exp(-\frac{x}{L_{eff}})$$

Equations from: Sayad, Kaminski, Blanc, Nouiri & Lemiti

Surface Recombination Velocity

- If surface recombination velocities are negligible

  ($s_f = s_r = 0$)

  - $L_{eff} \approx L_b$
- However, if surface recombination velocities are large

  ($s_f = s_r = \infty$)

  - $\frac{1}{L_{eff}} = \sqrt{\frac{1}{{L_b}^2} + \frac{\pi^2}{W^2}}$
- If $s_f = 0$ and $s_r = \infty$, or the opposite

  - $\frac{1}{L_{eff}} = \sqrt{\frac{1}{{L_b}^2} + \frac{\pi^2}{4W^2}}$

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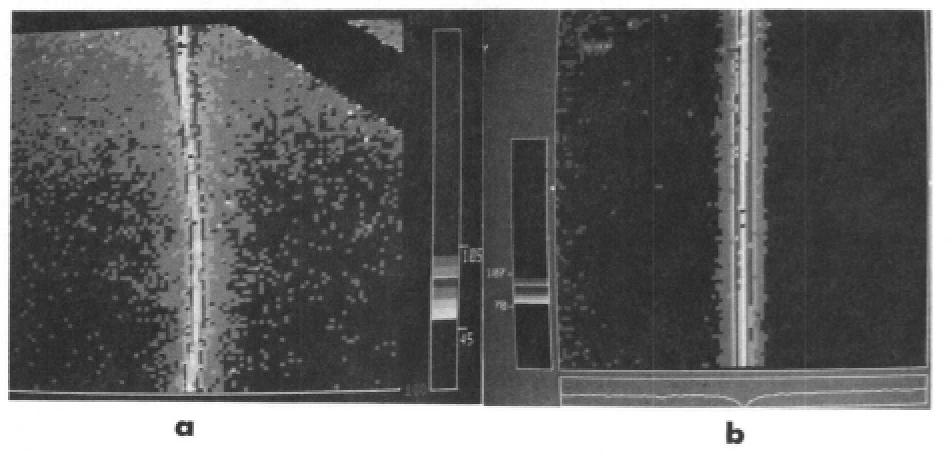
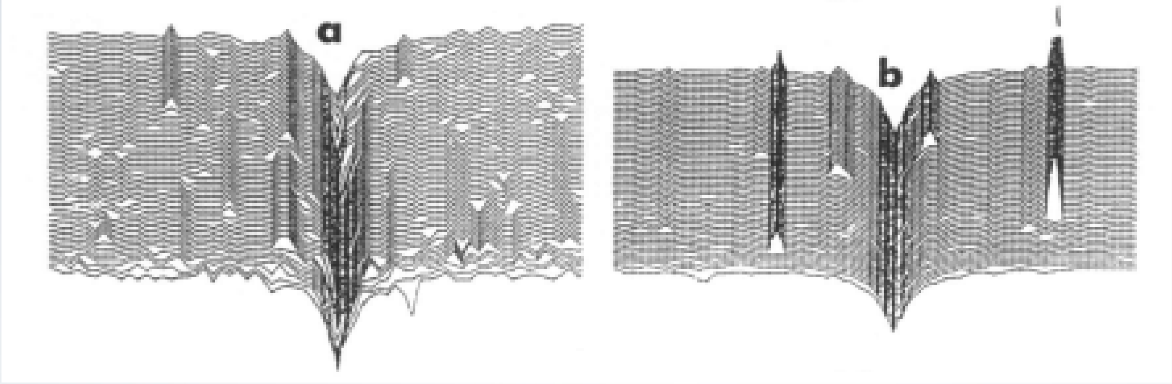
LBIC topography



- Imaging carried out using 100 line with 100 points each at a step of 10µm
  - Solar cell area is 1mm^2
- Minority carrier recombination is clearly evident
- Photocurrent reduced by 25% near grain boundary

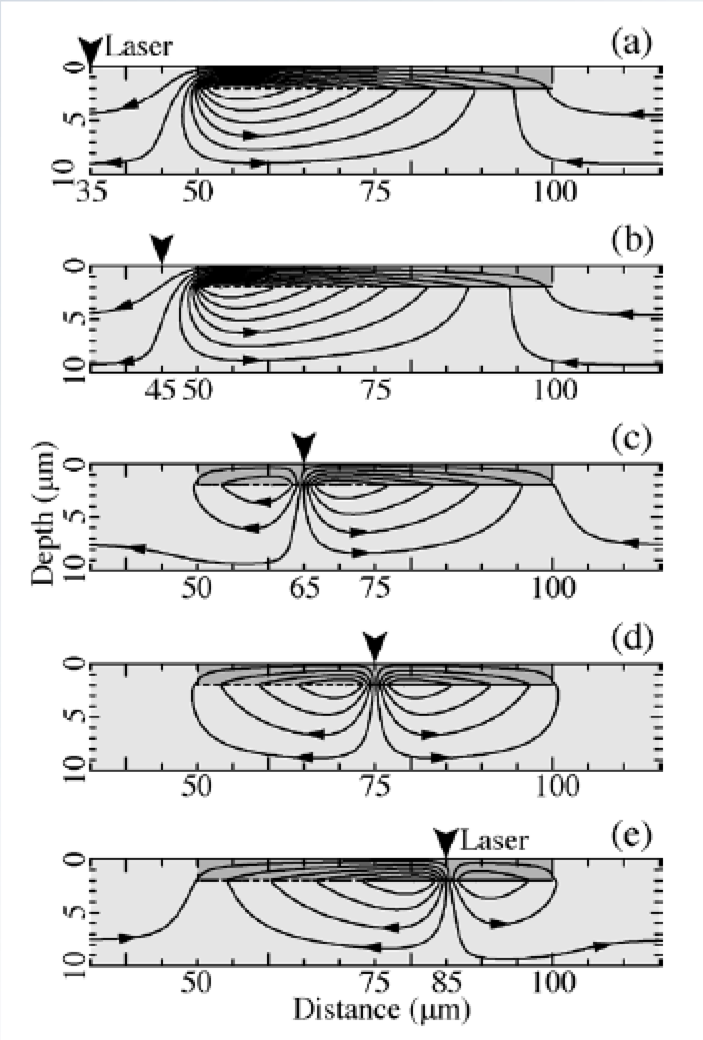
Figure from: Masri, Boyeaux, Kumar, Mayet & Laugier

LBIC topography (cont.)

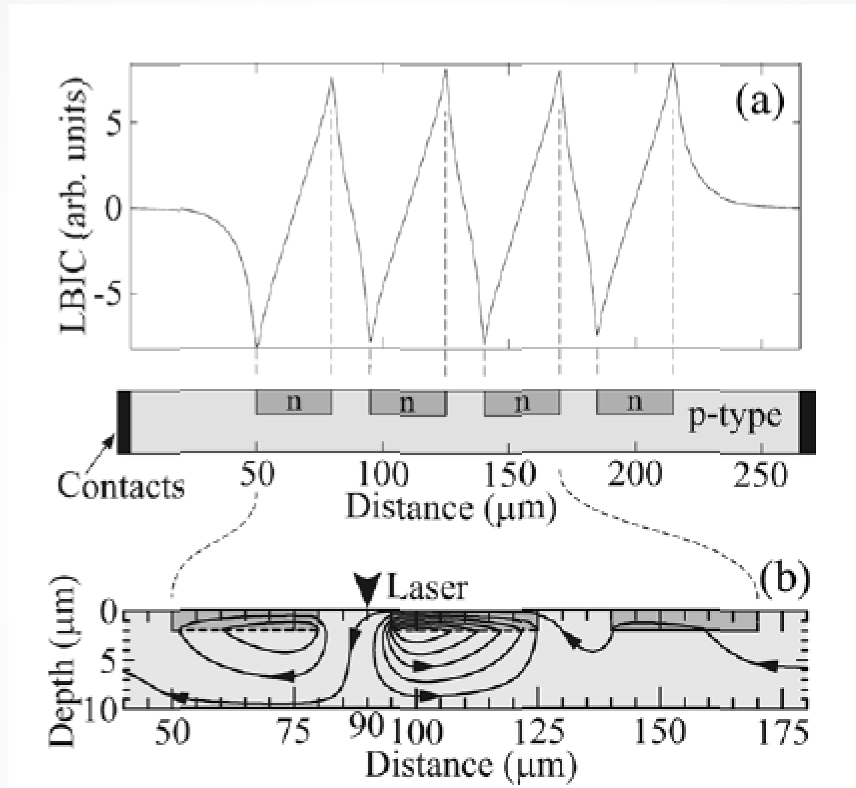


- Finding the surface recombination velocity we can find the diffusion length L_1.
- The background current density is given by
  - $I_0 = qN(1-R)\alpha L(1+\alpha L)^{-1}$
- We can solve for diffusion length L_2
- The observed difference between the two above Ls leads us to interface layers and/or the presence of impurities in the sample

Light Sweep



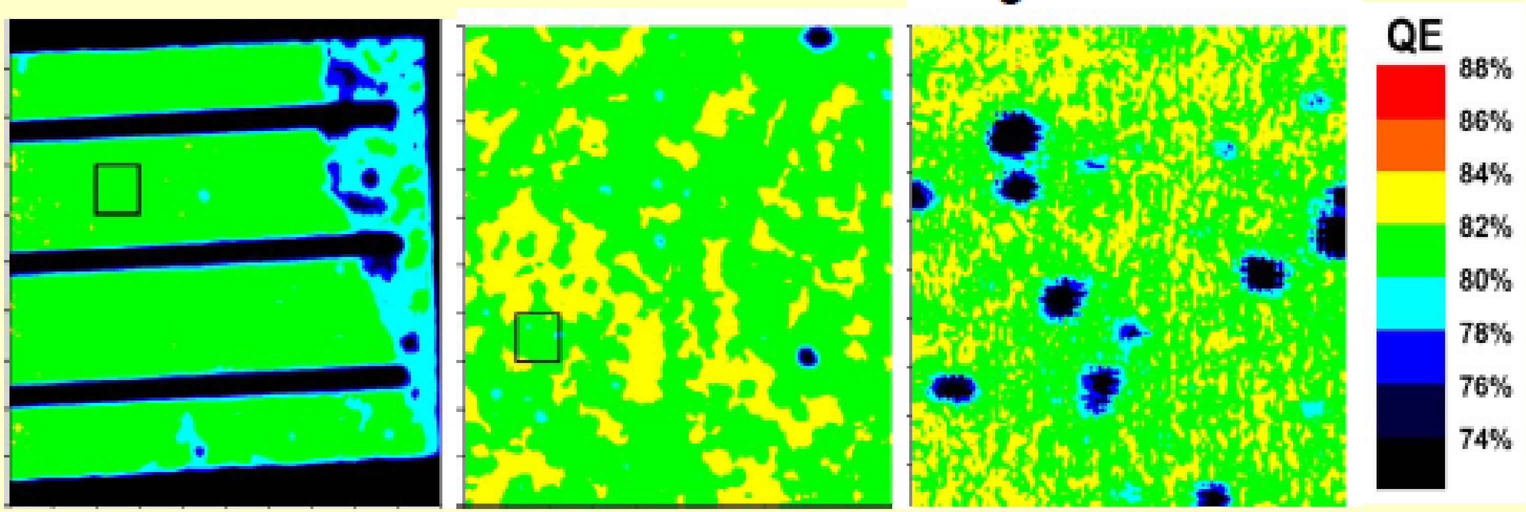
Laser
(a)
(b)
(c)
(d)
Laser
(e)
Depth (μm)
Distance (μm)



(a)
LBIC (arb. units)
n
p-type
Contacts
Distance (μm)
Laser
(b)
Depth (μm)
Distance (μm)

Resolution

Low Resolution | Mid-Resolution | High Resolution



5 x 5 mm field
100 micron spot

500 x 500 micron field
10 micron spot

50 x 50 micron field
1 micron spot

Notes: (1) LBIC QE very consistent with standard QE
(2) Standard conditions: zero bias, 1 sun, and 638 nm

Figures from: Sites & Nagle

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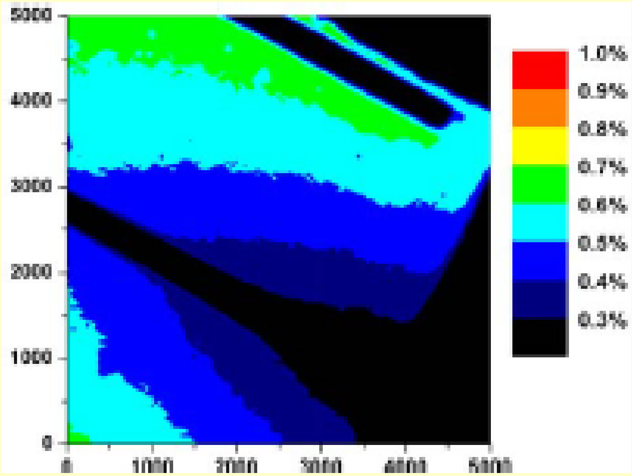
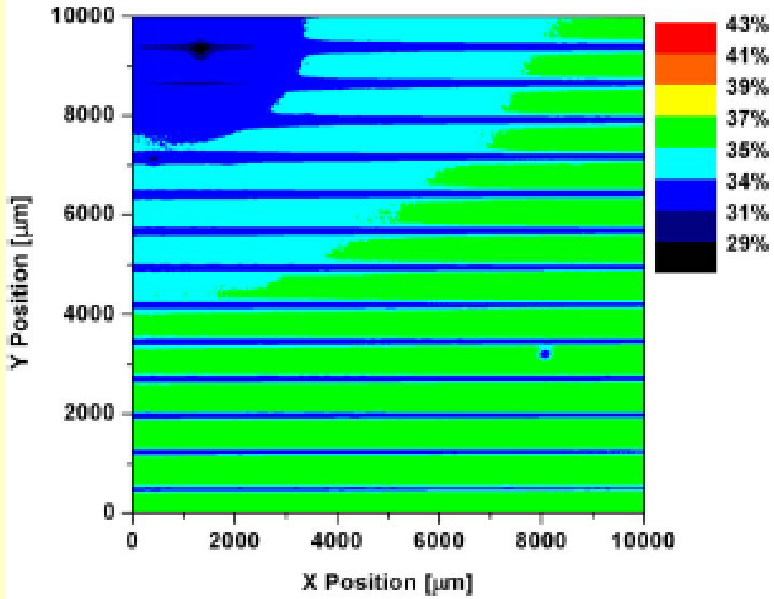
Defect Detection

CIGS cell with grid finger edge short. Very low QE.



Shunt is located very near grid line.

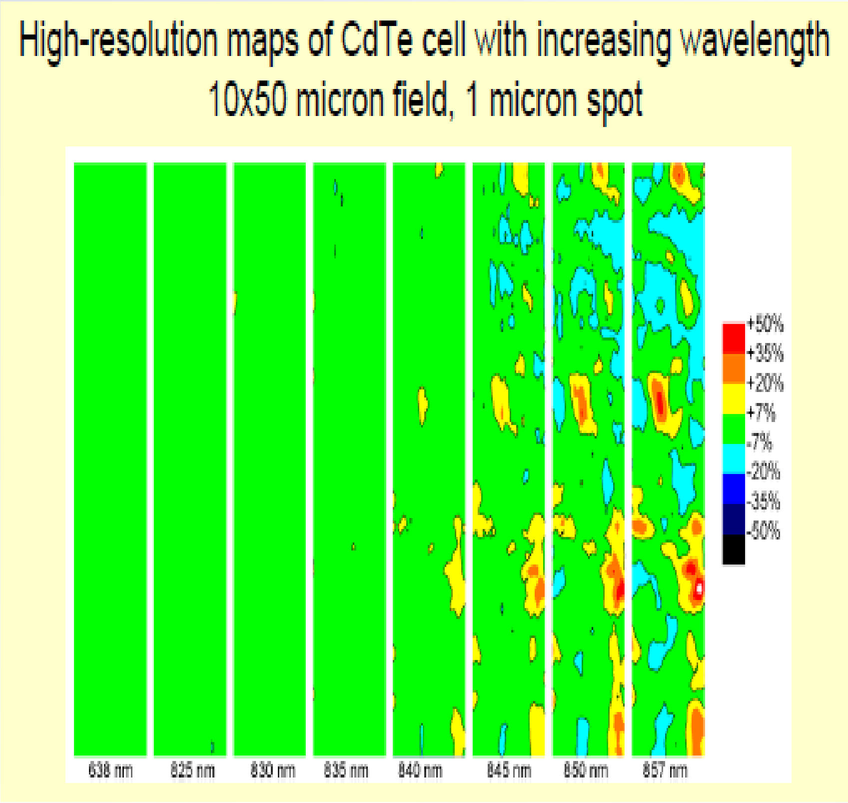


Shunt affects large area: field is 1 cm^2

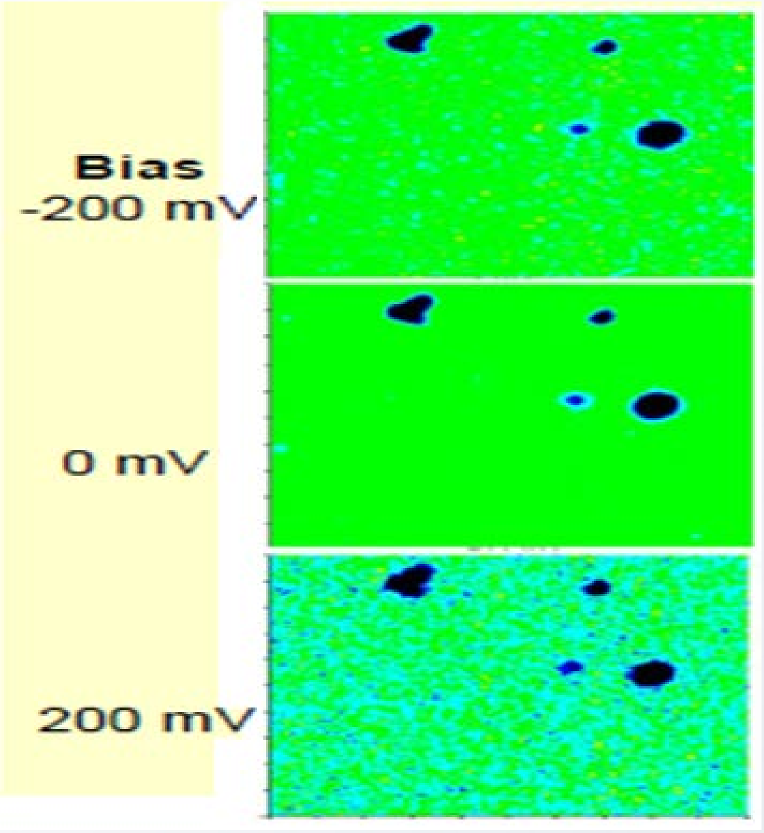
Figures from: Sites & Nagle

LBIC Variations

Wavelength Variation



Bias Variation



Figures from: Sites & Nagle

Quiz

1. What does LBIC stand for?

2. How is light generated for the LBIC apparatus?

3. What is the limit for defining a thick/thin sample?
   (W >/< ___)

4. Which would have a greater affect on the I_{sc}? R_s or R_p

5. What is the highest resolution that can be seen with LBIC?

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Vibrating Sample Magnetometer

Brooks Tellekamp

ECE 4813

November 2011

Georgia Institute of Technology

Outline

- Overview of Magnetic Properties
- Units
- Basic Magnetic Relations
- History
- VSM Basics
- Mechanical Design
- Properties of VSM

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Overview of Magnetic Properties

- B = Magnetic Flux Density or Magnetic Induction
- H = Magnetic Field (typically applied to a sample)
- m = Magnetic Dipole Moment
- M = Magnetization
- μ = Magnetic Permeability
  - Permeability of free space $\mu_0 = 4\pi \times 10^{-7} \frac{V \cdot S}{A \cdot m}$ (SI)
- χ_m = Magnetic Susceptibility

Units

- Gaussian units – a physical system for electromagnetic units based in CGS (centimeter-gram-second) base units

Unit	SI	CGS	Conversion
B	Tesla	Gauss	1T=10,000G
H	$\frac{A}{m}$	Oersted (Oe)	$\frac{A}{m} = \frac{4\pi}{1000}\text{Oe}$
m	$A \cdot m^2$	$emu(\frac{erg}{G})$	$A \cdot m^2 = 1000emu$
M	$\frac{A}{m} = \frac{J}{T}$	$\frac{emu}{cm^3}$	$\frac{A}{m} = .001\frac{emu}{cm^3}$
µ	$\frac{H}{m}$	Unitless	$\frac{\mu}{\mu_0} = \mu^G$
χ_m	Unitless	Unitless	$\chi_m{}^{SI} = 4\pi\chi_m{}^{G}$

Overview of Magnetic Relations

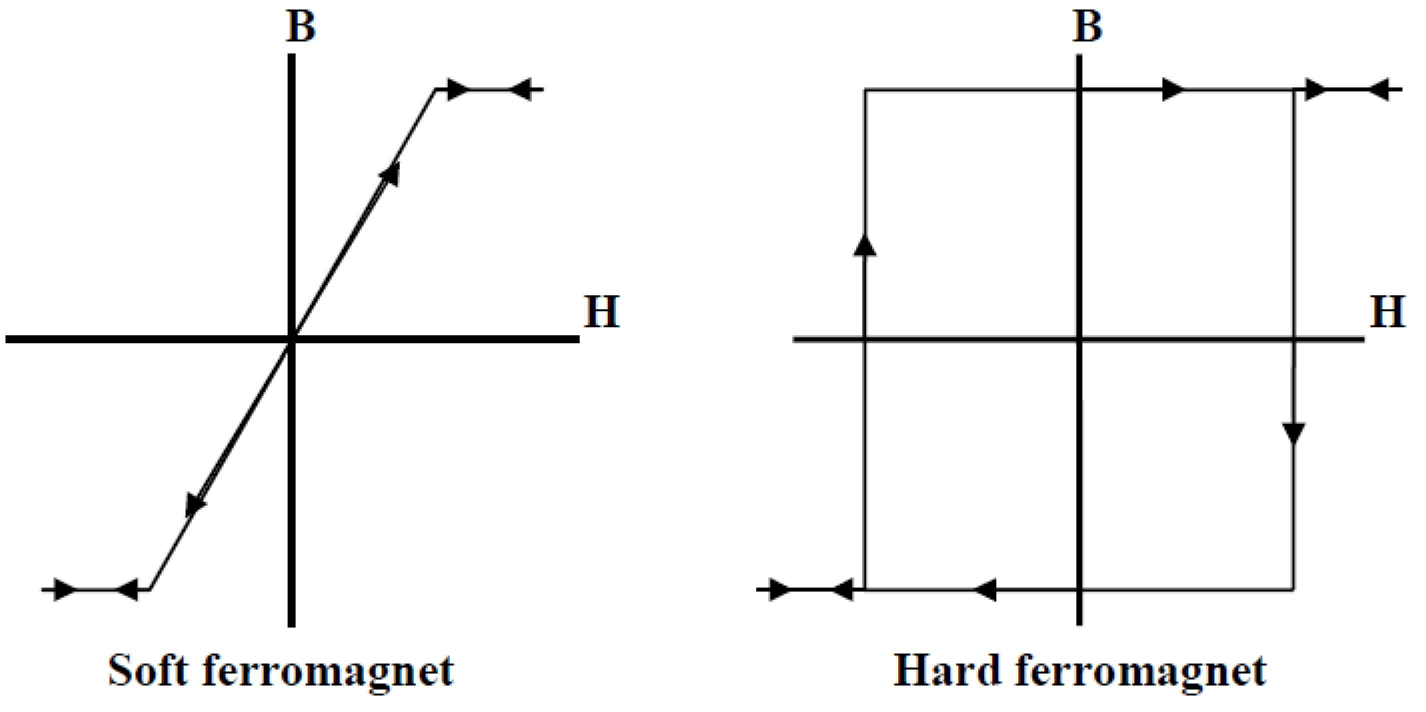
SI	CGS
$B = \mu H = \mu_0(H + M)$ $M = \chi_m H$ $\mu = \mu_0(1 + \chi_m)$	$B = \mu H = H + 4\pi M$ $M = \chi_m H$ $\mu = 1 + 4\pi\chi_m$

Where $\frac{\mu}{\mu_0} = \mu_r =$ relative permeability (material dependant)

And $M = nm$ where $n = \frac{N}{V}$ (number of acting moments per unit volume)

Hysteresis

- Ferromagnetic materials retain magnetic orientation
- Ferromagnetic materials exhibit different curves for directional field sweeps (+ to -, or - to +)



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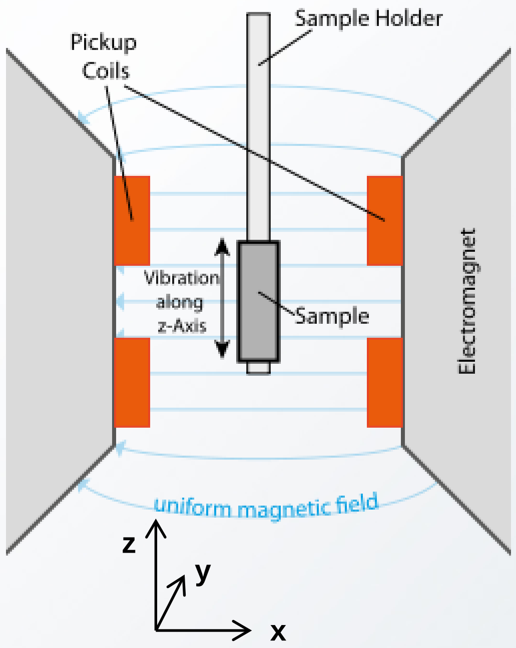
History

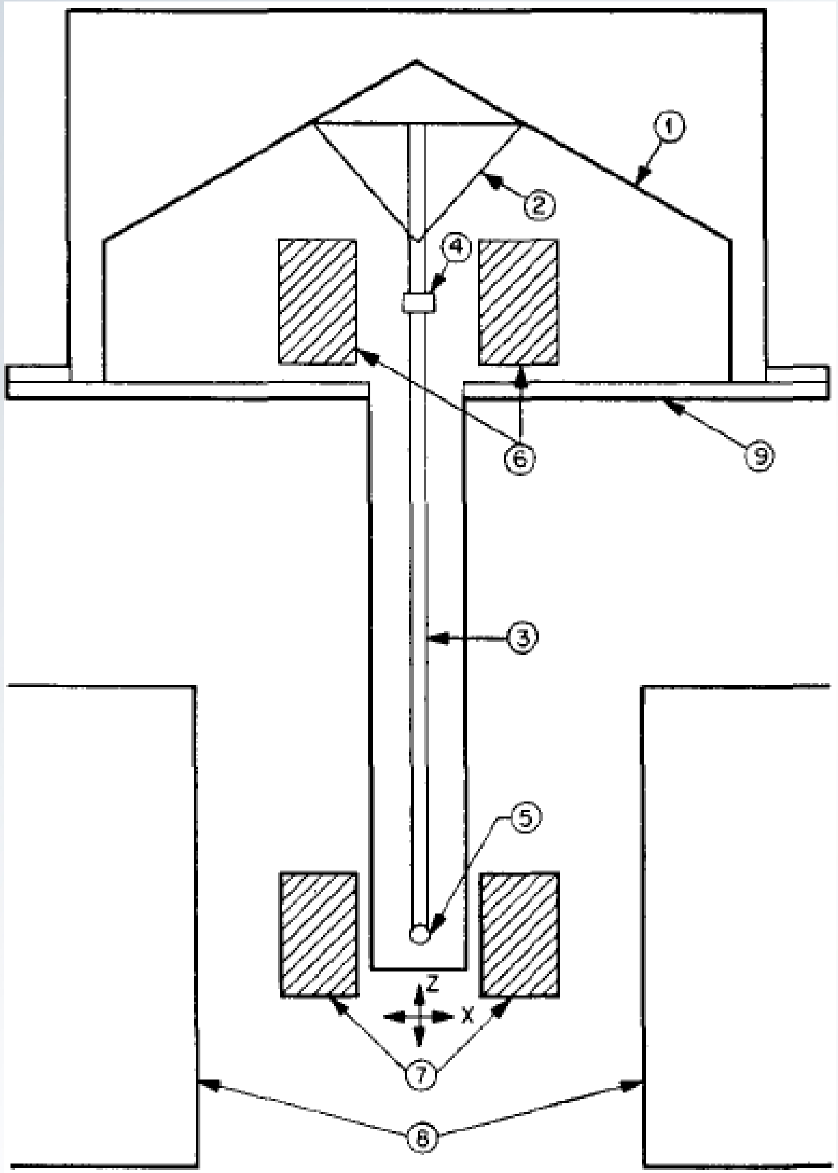
- Developed in the late 1950’s
- No good way to measure magnetic moments without considerable prior knowledge of material properties
- Force methods are very sensitive and require a field gradient
- Other specific techniques existed, but were not adaptable to many material classes
- Vibrating coil technique used a coil with the detection axis parallel to the applied field
  - Idea modified by Dr. Simon Foner of MIT to vibrate the sample and use a coil perpendicular to the applied field

VSM Basics

- In a uniform magnetic field, a ferromagnetic sample is vibrated along the z axis
- The dipole field induces a current in the pickup coils, which is proportional to the magnetic moment
- Susceptibility is obtained as the slope of the M-H Curve
- Permeability is obtained as the slope of the B-H Curve



Mechanical Design



1) Loudspeaker Transducer
2) Paper Cup Support
3) Sample Holder “Straw”
4) Reference Sample
5) Sample
6) Reference Coils
7) Pickup Coils
8) Magnets
9) Housing

Frequency Invariance

- Reference sample attached to sample holder
  - High coercivity material
- Identical coil arrangement to pickup coils
- Loudspeaker vibrates the sample and reference sample at the same frequency
- Phase and Amplitude of coil voltages are directly related via the magnetic moment of the sample

Time-Varying Dipole Field

Fixed Dipole Scalar Potential

$$\varphi = \frac{Mx}{r^3}$$

Time variant field

$\varphi_1 e^{j\omega t}$ where

$$\varphi_1 = -a\frac{d\varphi}{dZ} = a\frac{MxZ}{r^5}$$

Where the flux pattern is

$$-\nabla\varphi_1$$

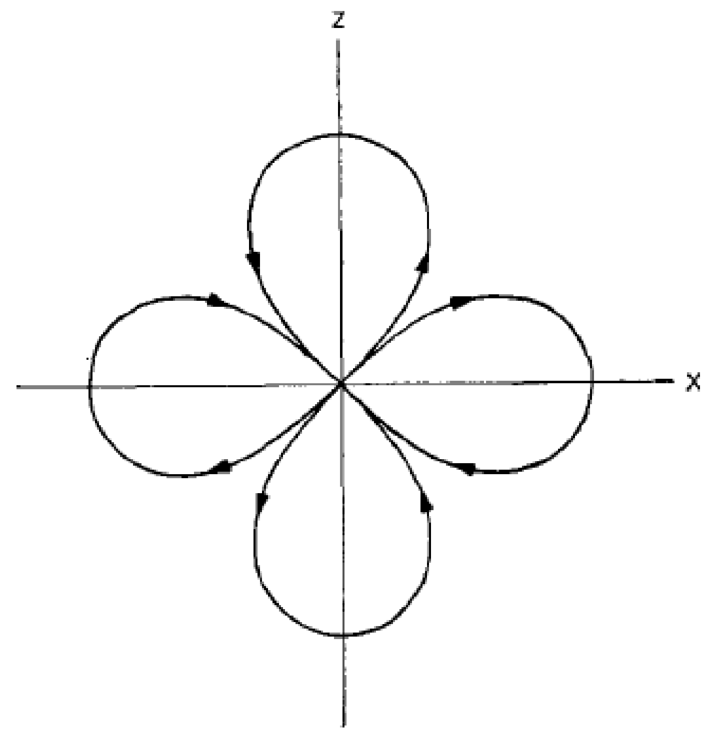


FIG. 4. Time varying part of dipole field in X-Z plane for vibration parallel to Z and dipole moment parallel to X.

The pattern allows for a variety of coil arrangements where the coil axis is along a flux line.

Circuitry

- Many options for output signal measurement
  - Always a temperature controlled resistor in series with the pickup coils
    - Voltage drop is proportional to magnetic moment, m
  - Lock-in Amplifier to compare reference signal and sample signal.
  - Null Amplifier from a calibrated diode bridge
  - Reference signal is controlled with a potentiometer for precise voltage division to balance with the sample output

Sensitivity

- Sensitivity depends on coil geometry
- With a 2 vertical coil method
  - Susceptibility changes of $5x10^{-10}$ can be measured
  - Magnetic moment changes of $5x10^{-6}$ emu
  - Average stability of balanced signals is 1 part in 10,000

Calibration

- Can be calibrated by 2 methods
  - Using a sample of known magnetic properties and mass
    - Usually 8mg of pure Nickel (high coercivity)
  - For weakly magnetic samples of obscure shape
    - First measure the sample in vacuum
    - Then measure in pure O_2 gas (well known susceptibility)
    - The difference of the two gives the susceptibility of the sample, which is used to calibrate that specific shape

Demagnitizing Factor

- Calibration is used to determine the Demagnitizing Factor, γ
- Once γ and m are determined, the BH curve can be extracted

$$M = 4\pi \frac{m}{V}$$

$$H_{internal} = H_{applied} - 4\pi\gamma M$$

$$B = H_{internal} + 4\pi M = H_{applied} + 4\pi M(1-\gamma)$$

Sample γ values…
Sphere: $\gamma = \frac{4\pi}{3}$,
Infinite plane: $\gamma = 4\pi$,
Cylinder: $\gamma = 2\pi$
Other shapes are well documented

Note: SI equations only, CGS equations vary

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Measurements

- Low-Conductivity Materials
  - Spherical or ellipsoid samples are preferred
  - Cubic crystals should be oriented 110 perpendicular to the z-axis
- High Conductivity Materials
  - Demagnetization corrections are not necessary
- Paramagnetic Samples
  - VSM can measure the magnetic field created by paramagnetic materials by the average value over the sample volume

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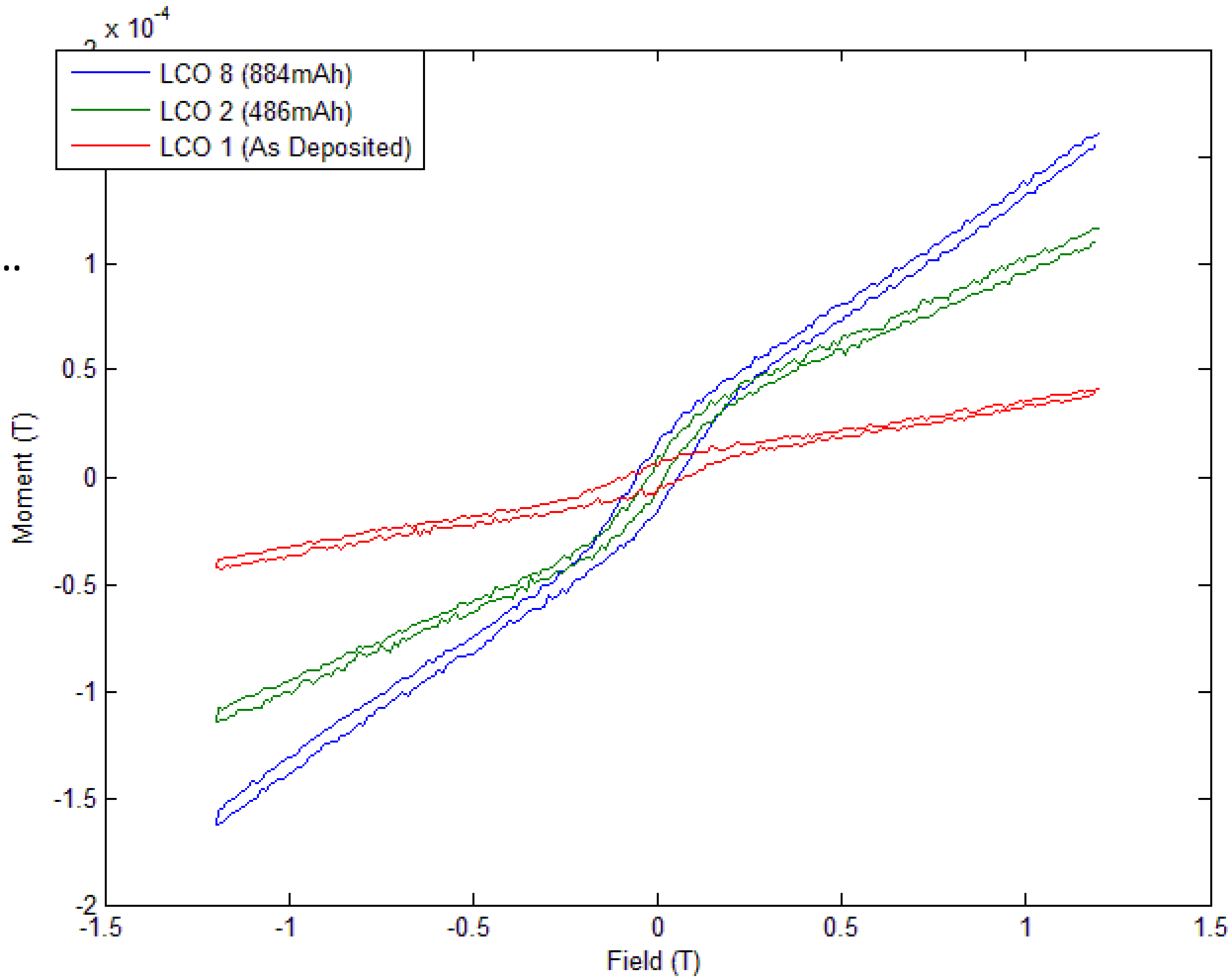
Sample Data

Actually emu…



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Transmission Electron Microscopy (TEM)

Jevon Raghubir

ECE-4813

Fall 2011

Dr. Alan Doolittle

Georgia Institute of Technology®

Why TEM?

- Limited image resolution in light microscopes
- Smallest distance that can be resolved by VLM:

$$\delta = \frac{0.61\lambda}{NA}$$

- Around 300nm for green light ($\lambda = 550\text{nm}$) w/ $NA=1$
- That is 1000 atom diameters
- Need to image details all the way down to the atomic level
  - Solution: TEM

Electron Wavelength

$$\lambda = \frac{h}{\left[2m_0E(1+\frac{E}{2m_0c^2}\right]^{1/2}}$$

- At high energies electrons approach the speed of light
- Relativistic effect must be taken into account

Accelerating voltage (kV)	Non-relativistic wavelength (nm)	Relativistic wavelength (nm)	Mass ($\times m_0$)	Velocity ($\times 10^8$ m/s)
100	0.00386	0.00370	1.196	1.644
120	0.00352	0.00335	1.235	1.759
200	0.00273	0.00251	1.391	2.086
300	0.00223	0.00197	1.587	2.330
400	0.00193	0.00164	1.783	2.484
1000	0.00122	0.00087	2.957	2.823

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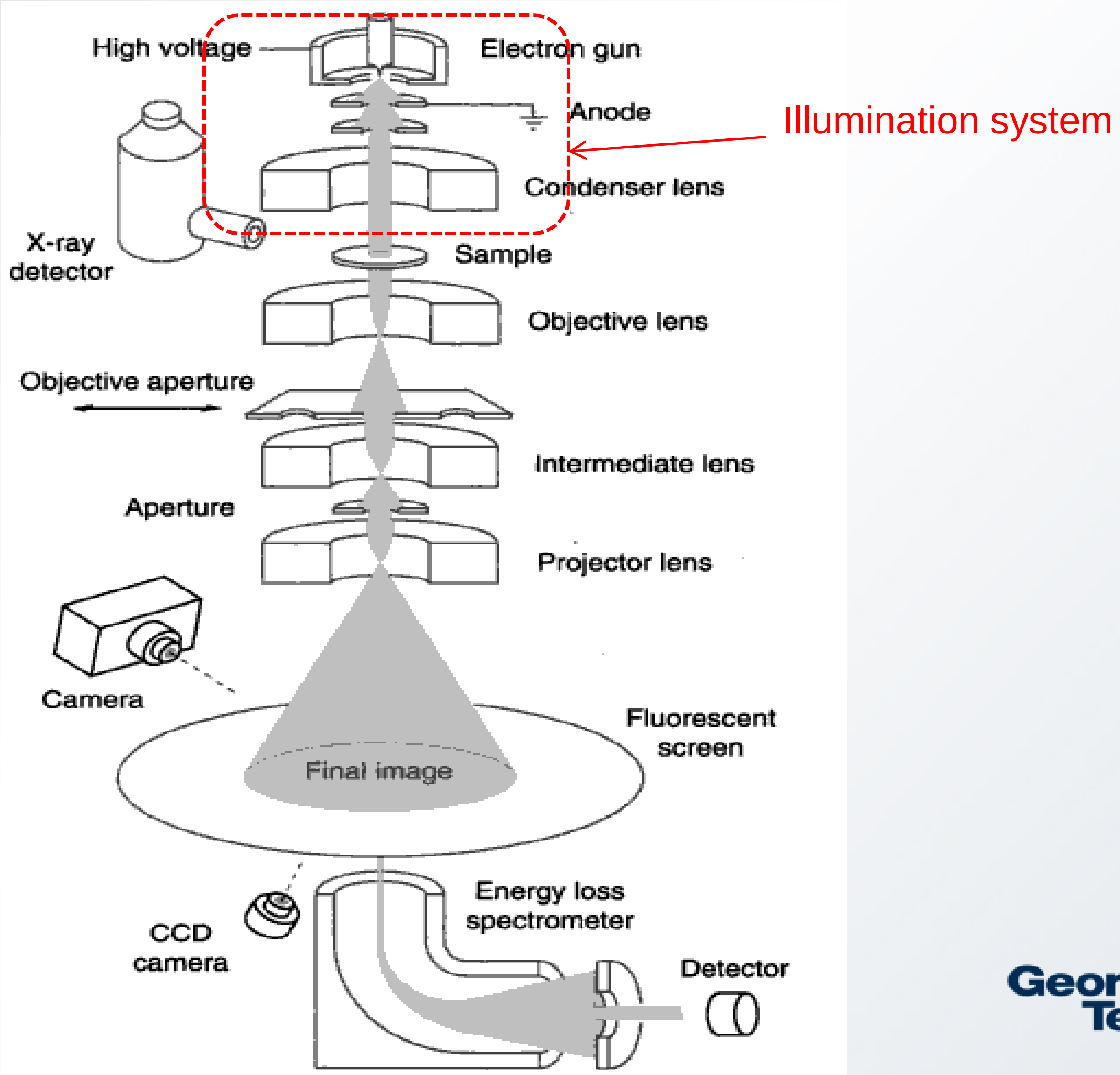
Schematic of TEM

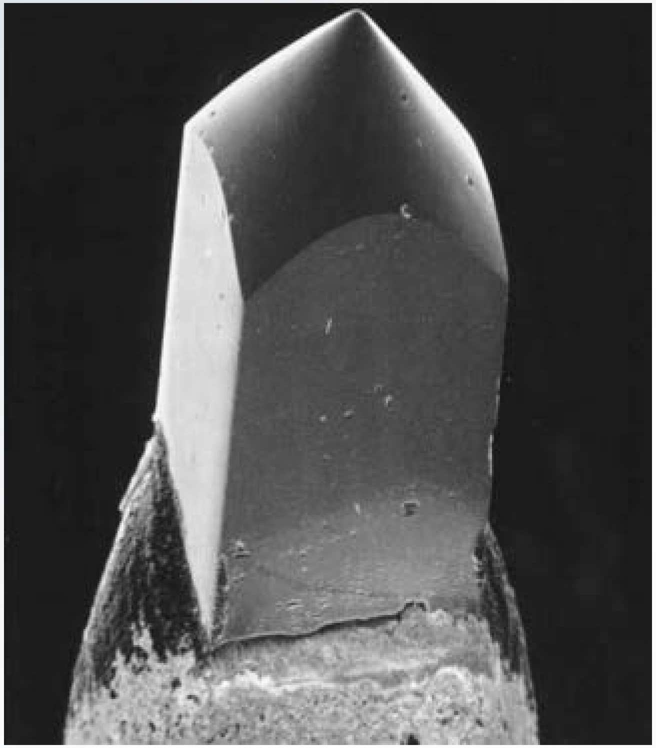


Illumination System (Electron Emission)

- Field Emission
  - Uses large electric fields at sharp points
  - Electrons tunnel out of source
  - Source: tungsten wire
- Thermionic Emission
  - Uses heat
  - Electrons gain enough energy to overcome natural barrier Φ
  - Sources:
    - Tungsten filaments
    - LaB6 crystals

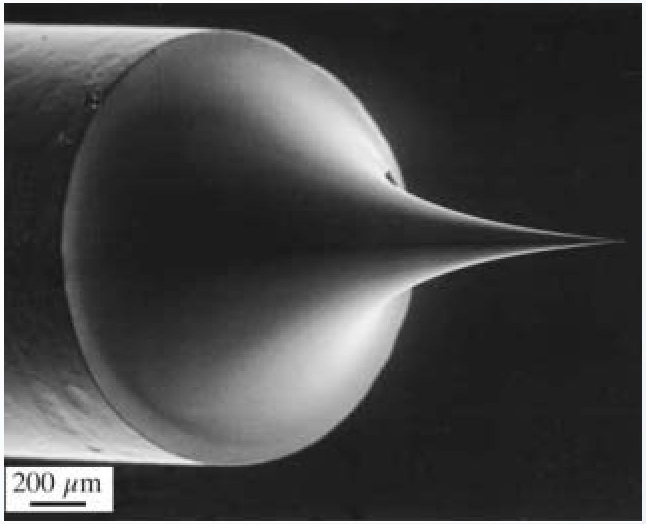
Electron Sources

Thermionic emission source



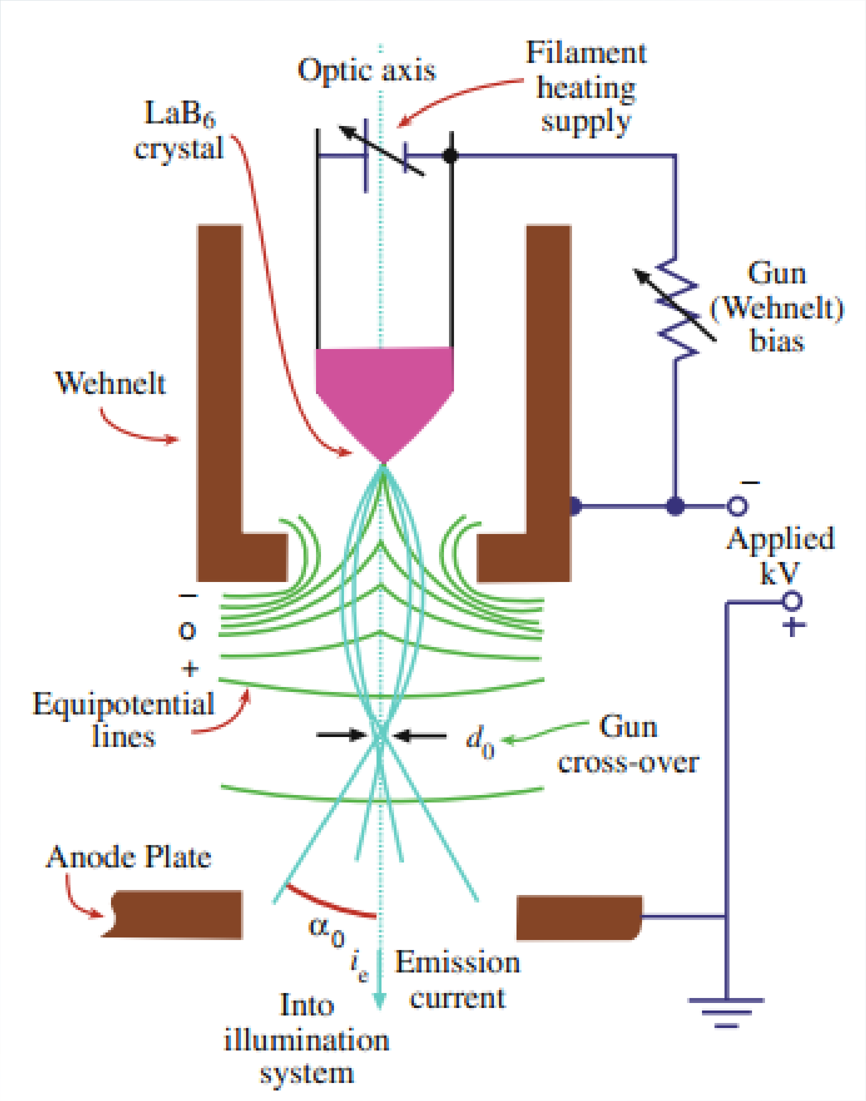
LaB6 crystal

Field emission source



Tungsten needle

Illumination System (Electron Gun)



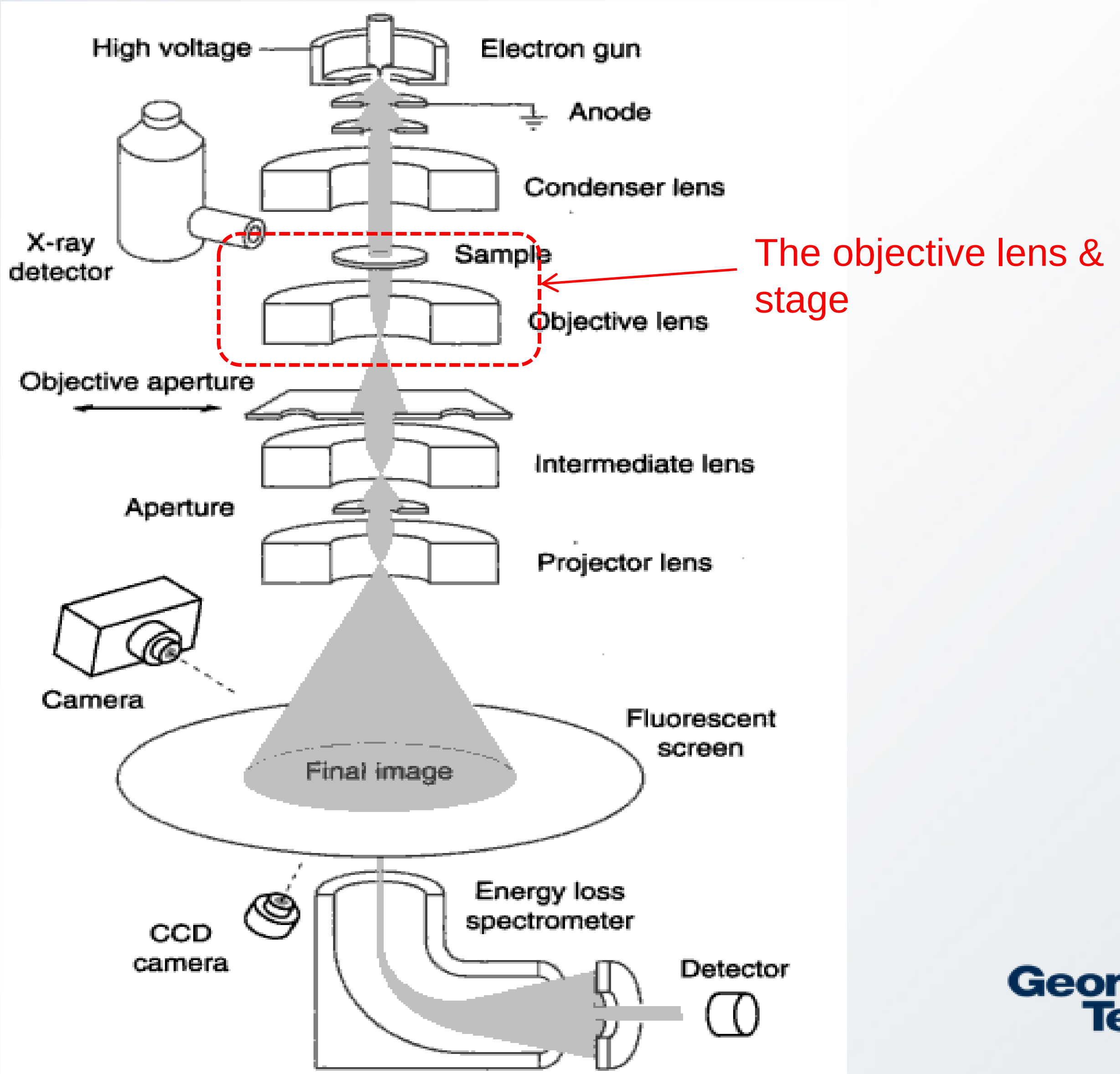
Thermionic electron gun

Illumination System (Condenser Lenses)

- Parallel electron beam
  - TEM imaging
  - Selected-area diffraction (SAD)
- Convergent beam
  - STEM
  - AEM

- Dependent on mode of operation

Schematic of TEM



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Objective Lens (Cs Correction)

- Instrumental resolution is limited primarily by spherical aberration of the objective lens

HRTEM Images

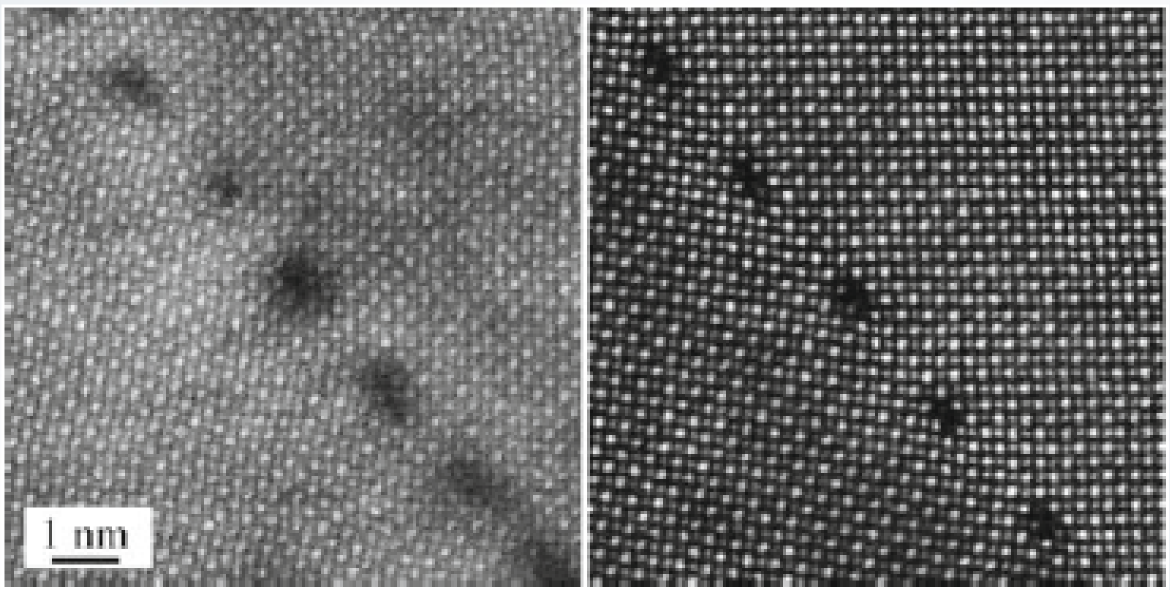
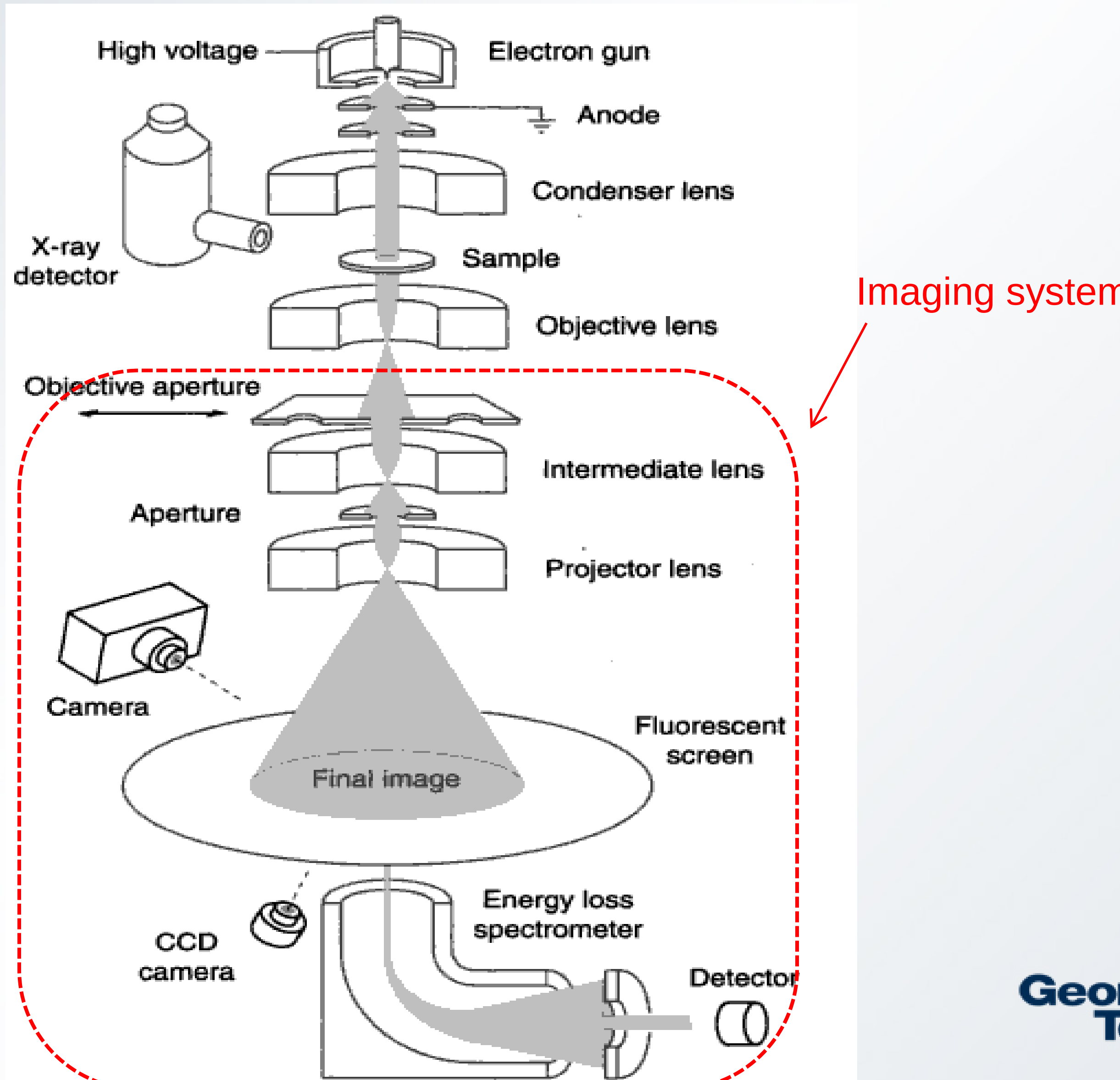


Image w/o Cs correction   Image w/ Cs correction

Schematic of TEM



Imaging System

- Post-specimen lenses
  - Magnify signal transferred by objective lens
    - Diffraction pattern
    - Image

- Viewing images and DPs
  - Fluorescent screen
  - Photographic film
  - CCD camera

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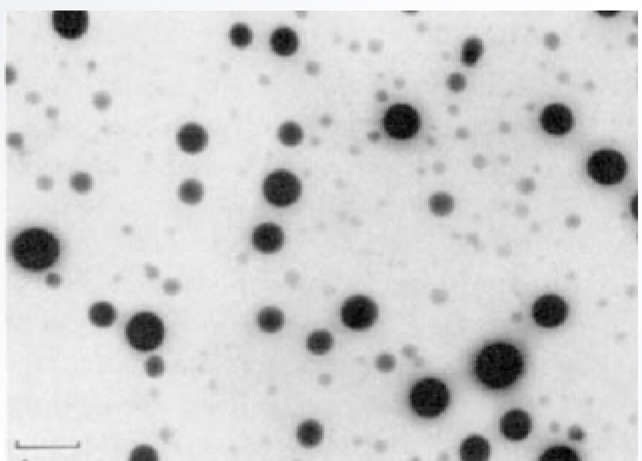
TEM Modes of Operation

- Diffraction mode
- Image mode
  - Bright-field microscopy
    - Block all diffracted beams and pass only transmitted electron beam
  - Dark-field microscopy
    - Allows diffracted beams and block transmitted electron beam
  - High-resolution electron microscopy
    - Admits transmitted beam and at least one diffracted beam

Example Images

DP from a single crystal Fe thin film



BF TEM image of a specimen

TEM Uses

- Crystal structure
- Lattice repeat distance
- Specimen shape
- Analytical measurements
  - Chemical information
- Study of defects
- Failure analysis

Advantages

- High lateral spatial resolution compared to other type of microscopes
- High quality and detailed images
- Can produce wide range of secondary signals
  - Backscattered electrons, auger electrons, characteristics x-rays, elastically and inelastically electrons, etc.
- Wide range of applications that can be utilized in a variety of different scientific, educational and industrial fields

Disadvantages

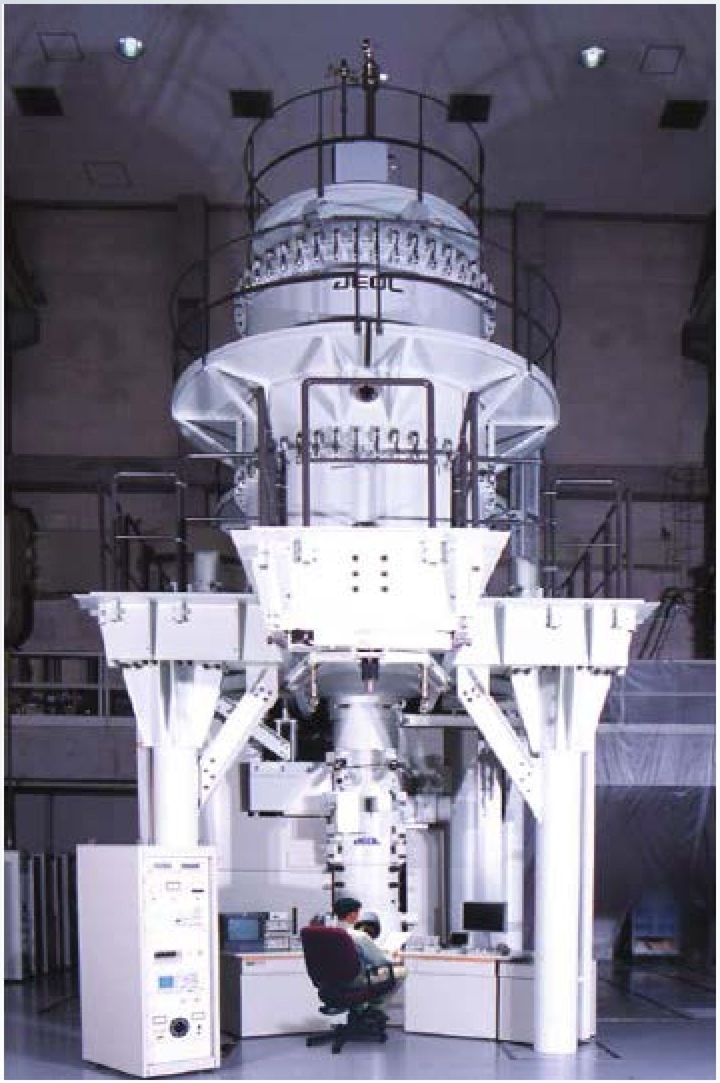
- Limited depth resolution
  - Gives 2D images for 3D specimens
- Specimen preparation
  - Thinning procedure can affect both their structure and chemistry of specimen
  - Time consuming
- Cost
  - About $5 for each eV

Types of Transmission Electron Microscopes

- HRTEM (High-resolution)
- HVTEM (High-voltage)
  - Can be damaging to specimen
  - Huge
- IVTEM (Intermediate voltage)
- STEM (Scanning)
- AEM (Analytic)

Example TEMs

HVTEM



HRTEM

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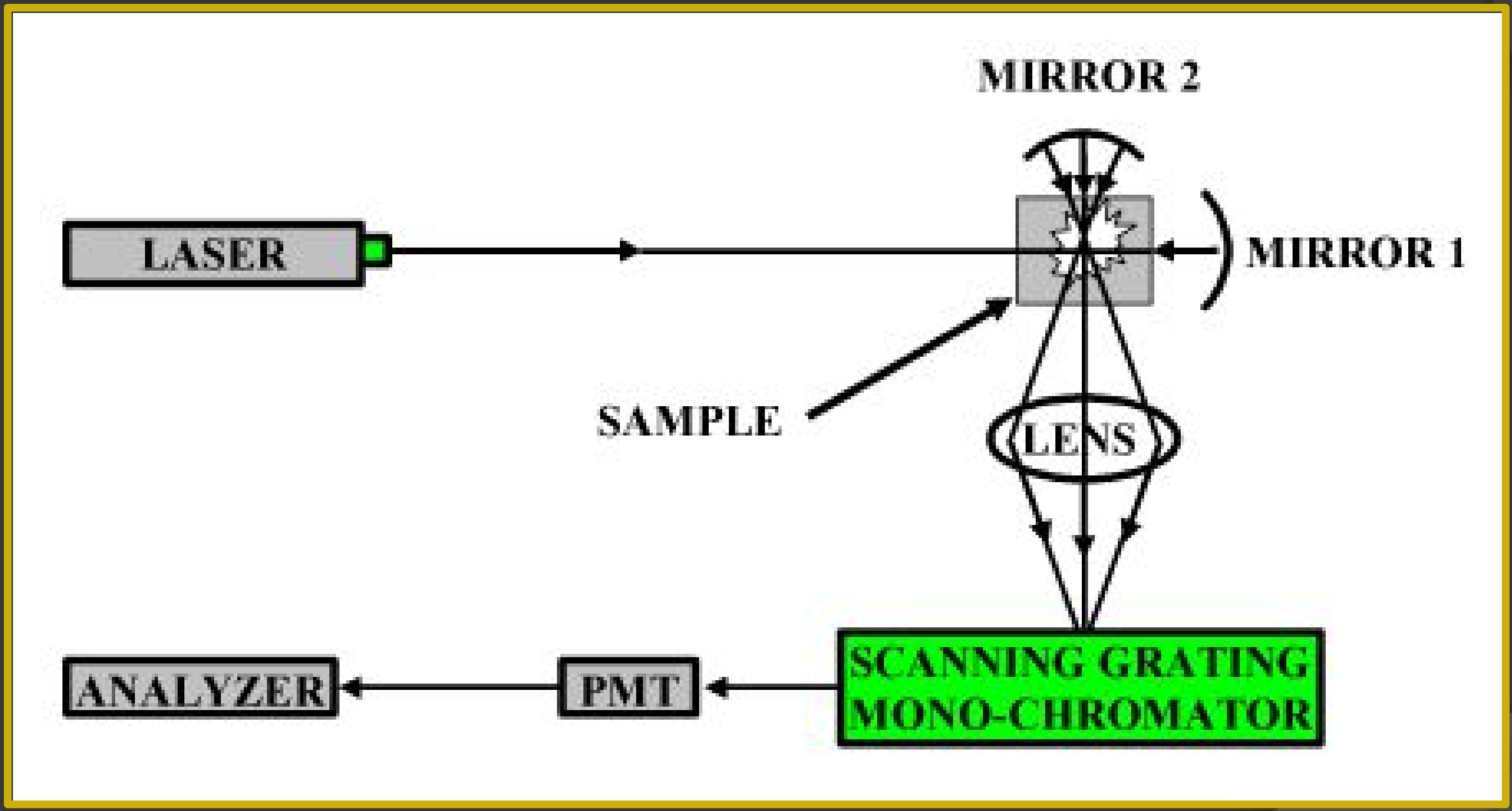
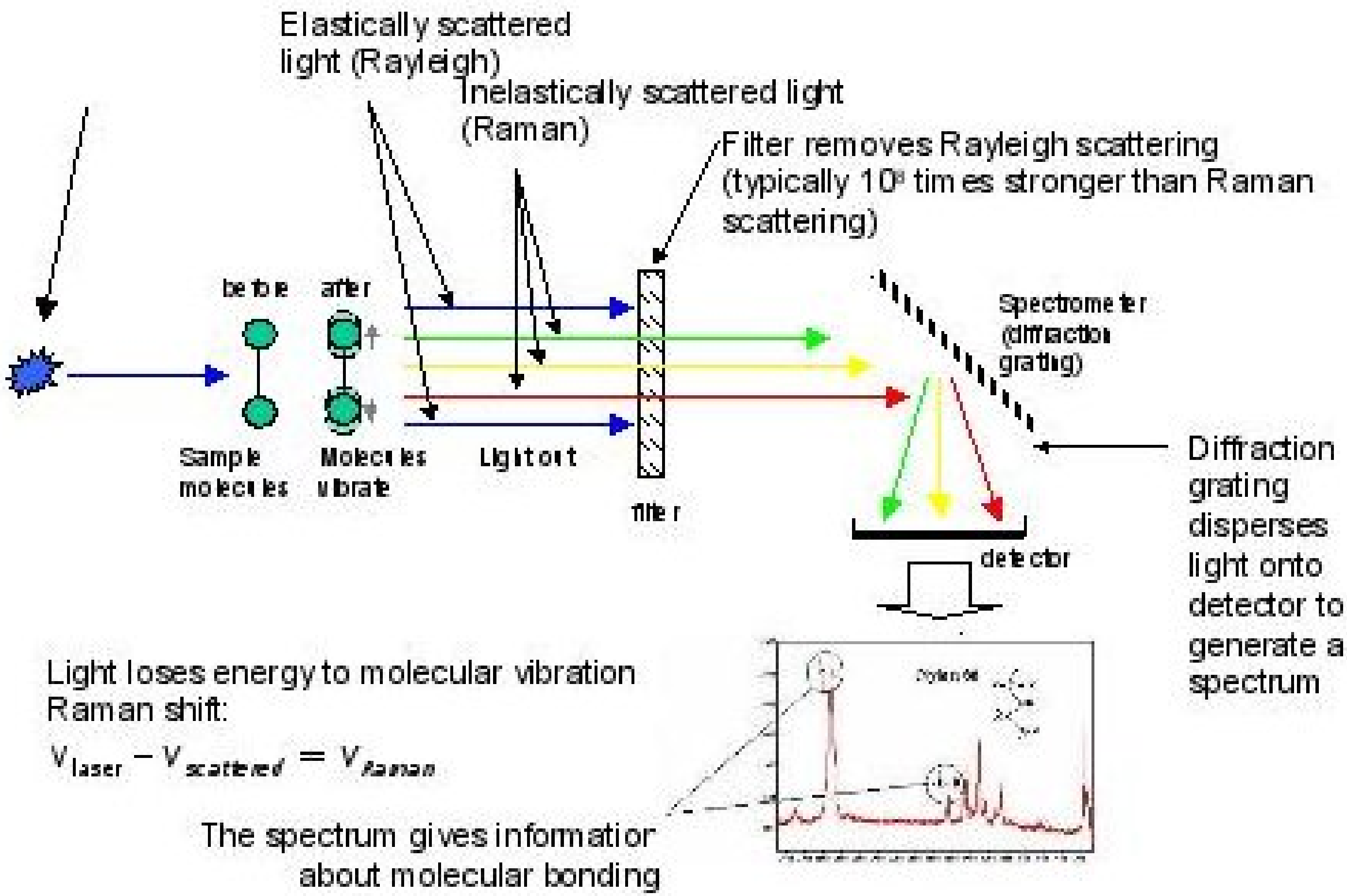
Maruchan
Raman
Spectroscopy
By Alex Walker

Raman Spectroscopy

- Based on the effects of Raman effect, first reported in 1928
- This is a vibrational spectroscopic technique that can detect both organic and inorganic species and measure the crystallinity of solids
- Advantages:
  - Free from charging effects
  - Sensitive to strain

Raman Spectroscopy




Elastically scattered light (Rayleigh)
Inelastically scattered light (Raman)
Filter removes Rayleigh scattering (typically 10⁸ times stronger than Raman scattering)
before
after
Sample molecules
Molecules vibrate
Light out
filter
Spectrometer (diffraction grating)
Diffraction grating disperses light onto detector to generate a spectrum
detector
Light loses energy to molecular vibration
Raman shift:
$V_{laser} - V_{scattered} = V_{Raman}$
The spectrum gives information about molecular bonding

Types of Scattering

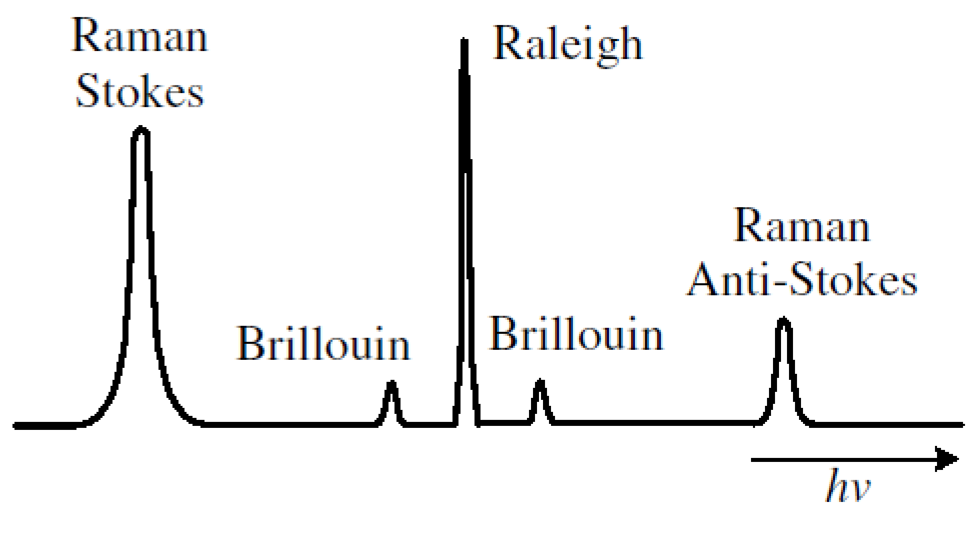
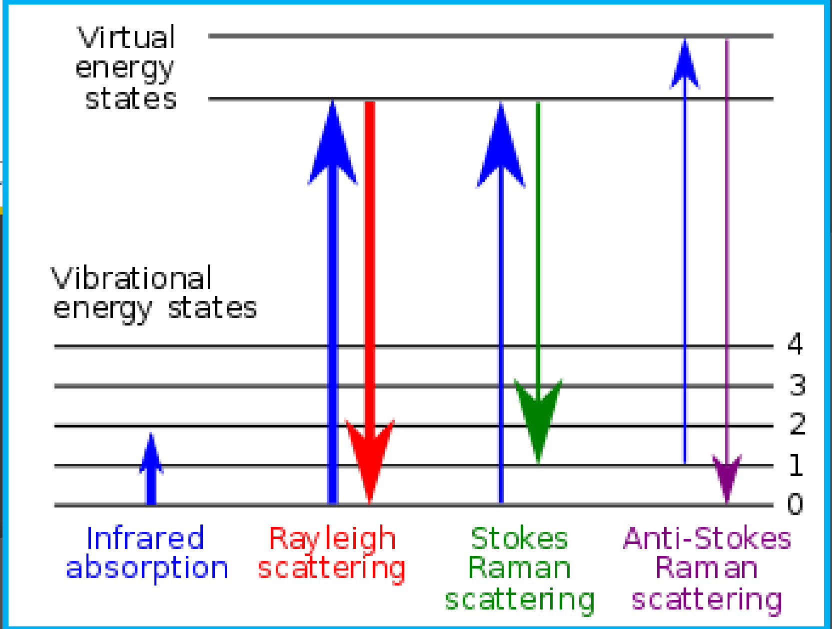


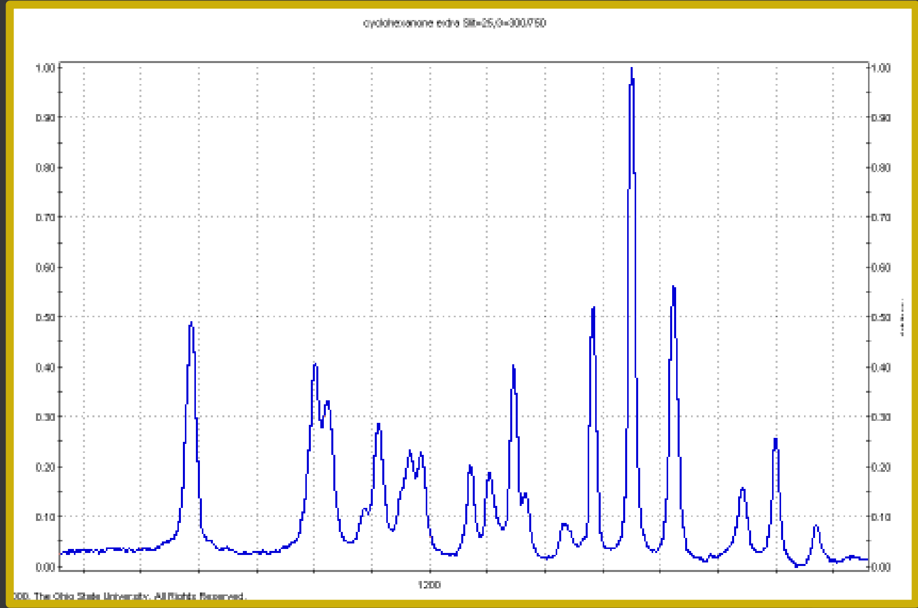
Fig. 10.31 Energy distribution of scattered light



Raman Shift

$$\Delta w = \left(\frac{1}{\lambda_0} - \frac{1}{\lambda_1}\right)$$

$$\Delta w(\mathrm{cm}^{-1}) = \left(\frac{1}{\lambda_0(\mathrm{nm})} - \frac{1}{\lambda_1(\mathrm{nm})}\right) \times 10^7 \frac{(\mathrm{nm})}{(\mathrm{cm})}$$



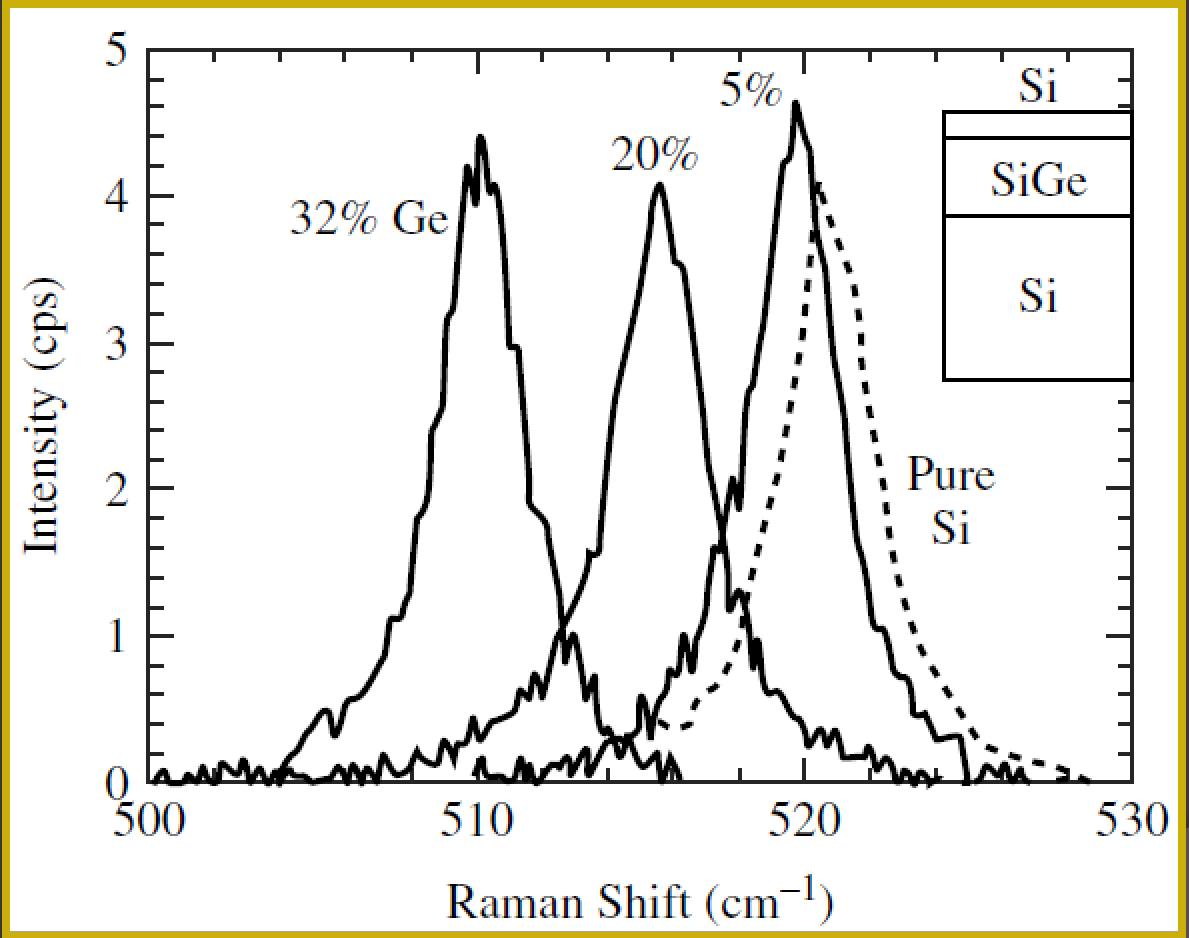
Raman Spectrum of cyclohexanone

$\Delta w = Raman\ shift\ expressed\ as\ a\ wavenumber$

Δλo = excitation wavelength

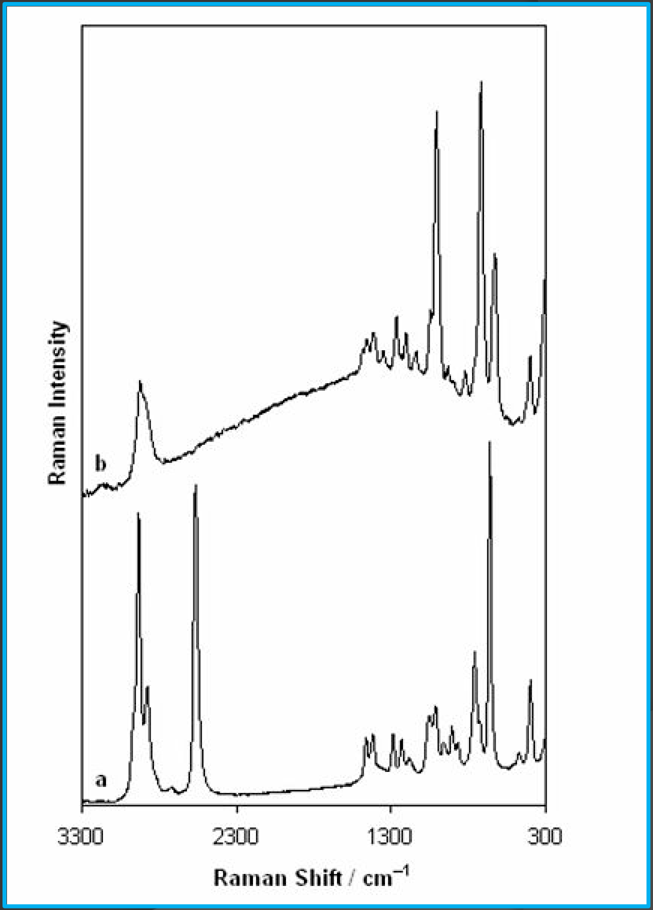
λ1 = Raman spectrum wavelength

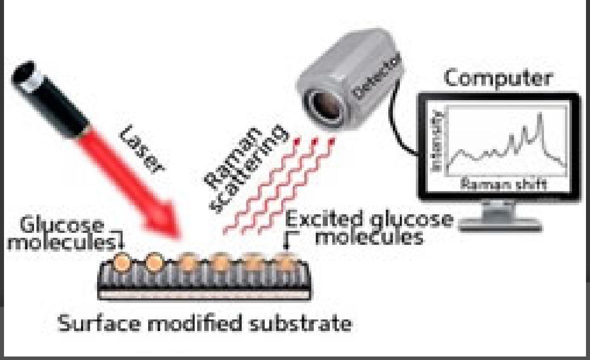
Raman Spectroscopy



Variations of Raman Spectroscopy

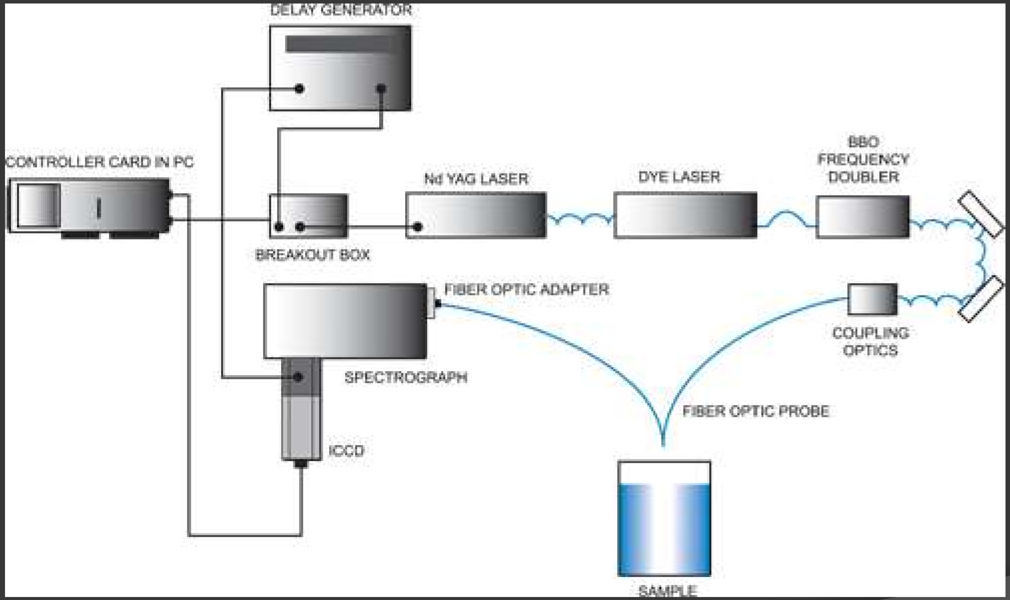
- Surface Enhanced Raman Spectroscopy
  - surface-sensitive technique that enhances Raman scattering by molecules absorbed on rough metal surfaces .

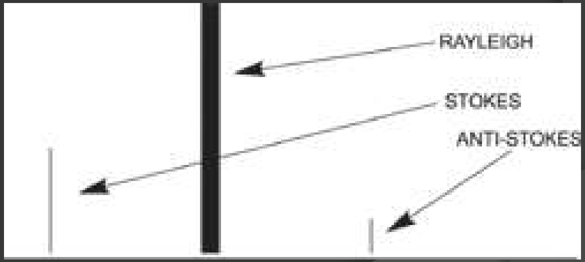




Variations of Raman Spectroscopy

- Resonance Raman Spectroscopy
  - Uses IR spectrum to identify unknown substances, measure the energy required to change the vibrational state of a chemical compound, and bioinorganic materials.

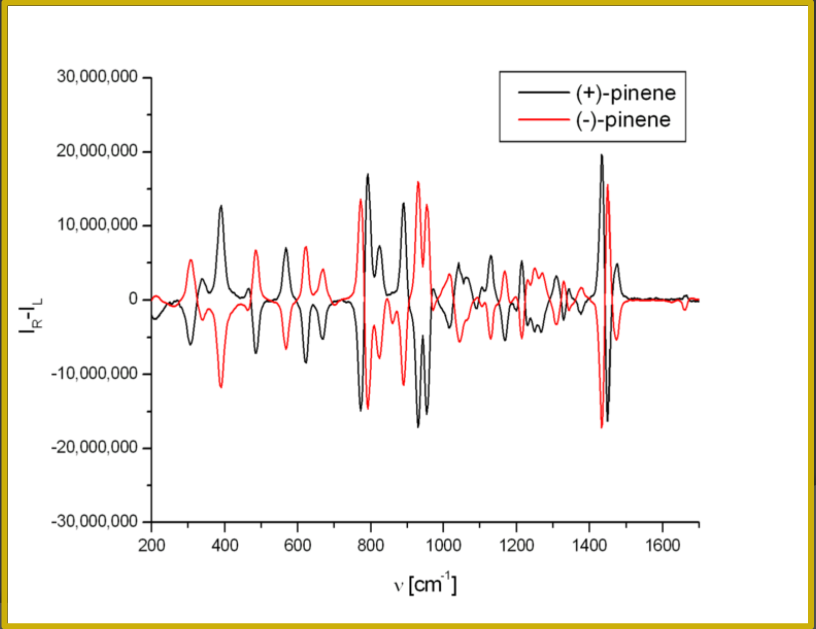




Ultraviolet Resonance Raman Spectroscopy

Variations of Raman Spectroscopy

- Raman Optical Activity
  - reliant on the difference in intensity of Raman scattered right and left circularly polarised light due to molecular chirality.



Resources

- http://www.fdmspectra.com/fdm_raman_organics.htm
- http://en.wikipedia.org/wiki/Raman_spectroscopy
- http://en.wikipedia.org/wiki/Surface_Enhanced_Raman_Spectroscopy
- http://en.wikipedia.org/wiki/Resonance_Raman_spectroscopy
- http://en.wikipedia.org/wiki/Raman_optical_activity
- http://en.wikipedia.org/wiki/Raman_optical_activity
- http://www.nano.org.uk/news/1368/
- http://www.andor.com/learning/applications/Raman_Spectroscopy/