THIN FILMS CHARACTERIZATION

(PART I)

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OUTLINE

- Thin film characterization
- Optical characterization
- Chemical bonds
- Electrical properties

THIN FILM CHARACTERIZATION

What do we want to know ?	How do we find this out ?
What does the sample look like ? •on a macroscopic scale •on a microscopic scale •on an atomic scale	•optical microscopy •scanning electron microscopy (SEM) •transmission electron microscopy (TEM) •scanning probe microscopies (STM, AFM ...)
What is the structure of the sample ? •internal structure •density •microscopic and atomic scales	•X-ray diffraction (XRD) •stylus profilometry •quartz crystal monitors (QCM) •ellipsometry •low energy electron diffraction (LEED) •reflection high energy electron diffraction (RHEED)
What is the sample made of ? •elemental composition •impurities •chemical states	•Auger Electron Spectroscopy (AES) •Energy Dispersive Analysis of X-rays (EDAX) •X-ray Photoelectron Spectroscopy (XPS) •Secondary Ion Mass Spectrometry (SIMS) •Rutherford Backscattering (RBS)
What are the optical properties of the sample ? •refractive index, absorption •dielectric properties •as a function of wavelength	•ellipsometry •Spectroscopic reflectometry
What are the electrical properties of the sample? •device properties - not covered here •material properties • resistance / conductance • capacitance	•resistance - four point probe
What are the magnetic properties of the sample ? •hysteresis loops	•magneto-optical Kerr effect (MOKE) •ferromagnetic resonance (FMR)
What are the mechanical properties of the sample ? •internal stress in films / substrates •friction •adhesion	•stress curvature measurements •pin on disk friction test •adhesion tests

THIN FILM CHARACTERIZATION

Characterization fundamentals

- Available probes:
  - light (electromagnetic radiation)
  - electrons
  - ions (and nuclei and protons)
  - neutrons
  - neutral atoms
  - "touching" the sample = forces

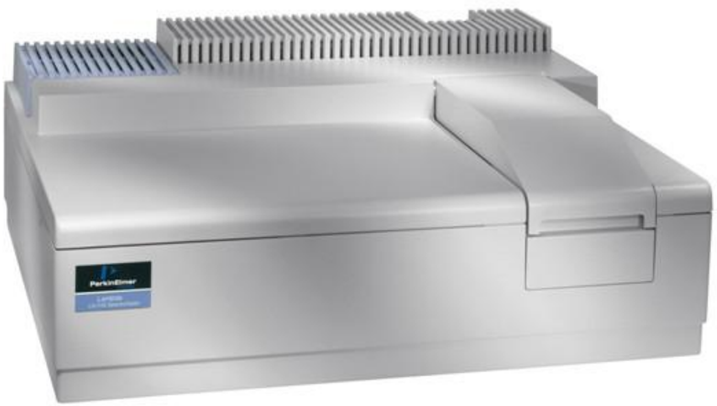
THIN FILM CHARACTERIZATION

Two general types of techniques

- "counting" techniques
  - How much ? or How many ?
    - intensity
    - force
    - numbers of particles
    - polarization
- "spectroscopy" techniques
  - Distribution by
    - energy
    - wavelength, frequency
    - mass
    - position
  - need to consider instrument sensitivity variations

OPTICAL CHARACTERIZATION

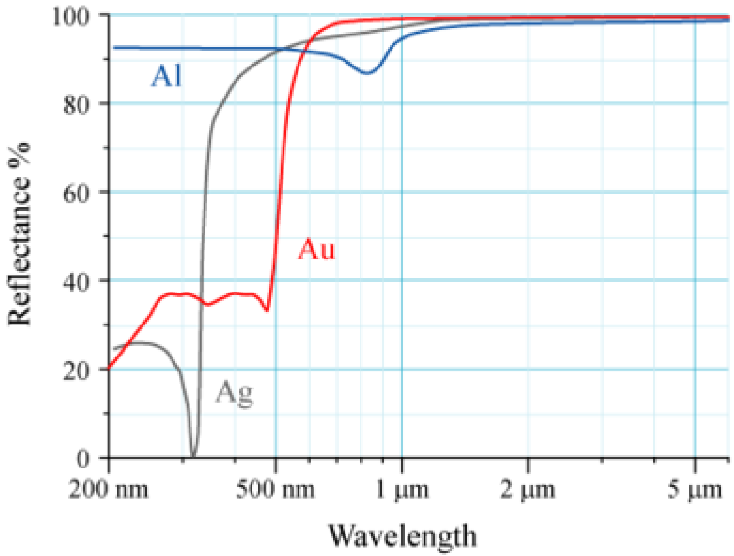
- Devices
  - Photospectrometer
  - Ellipsometer

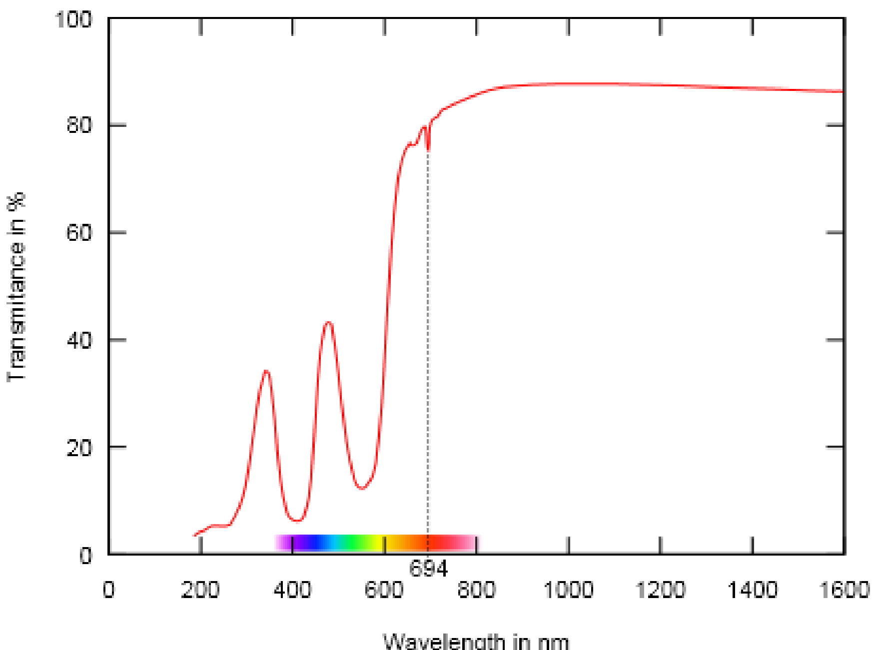


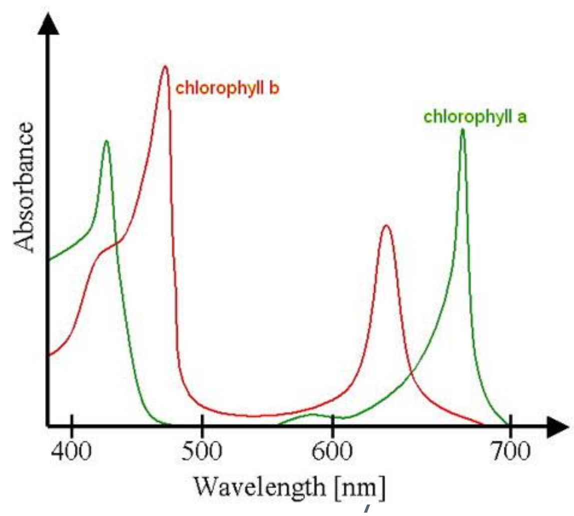
Other methods: synchrotron,

OPTICAL CHARACTERIZATION

- What can be measured
  - Reflectance (*R*)
  - Transmitance (*T*)
  - Absorbance (*A*)

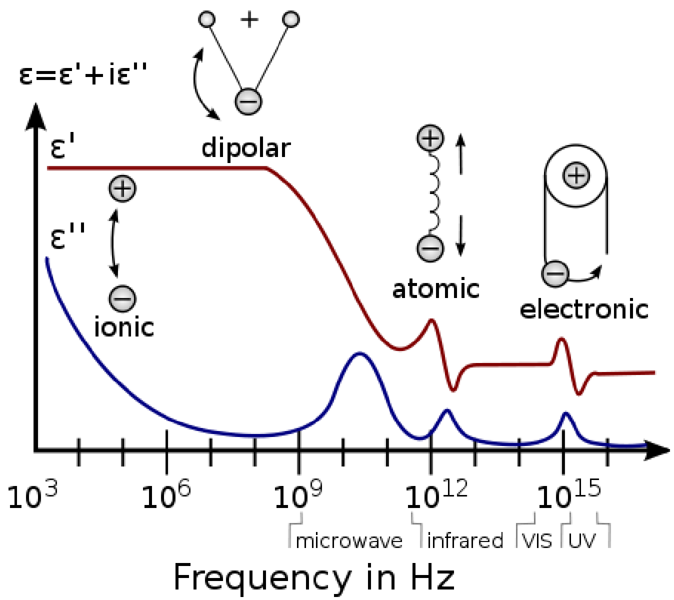


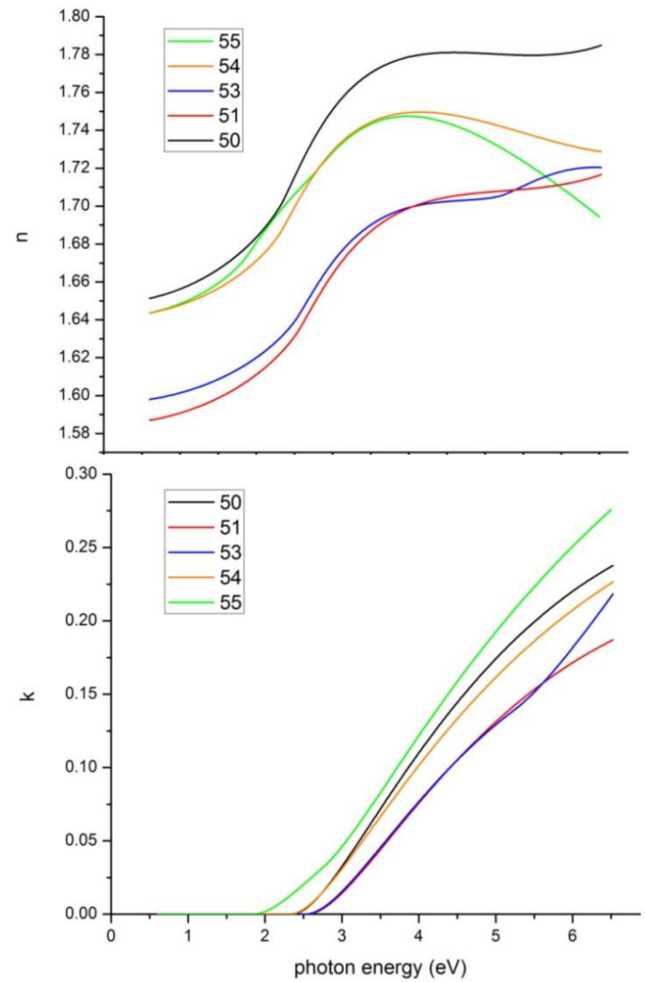




OPTICAL CHARACTERIZATION

- Dielectric (ξ)/optical properties of materials (n,k)
- Thickness of thin films (d)
- porosity and roughness





CHEMICAL BONDS

- Chemical compounds are formed by the joining of two or more atoms. A stable compound occurs when the total energy of the combination has lower energy than the separated atoms.

- The bond is caused by the electromagnetic force attraction between opposite charges, either between electrons and nuclei, or as the result of a dipole attraction.

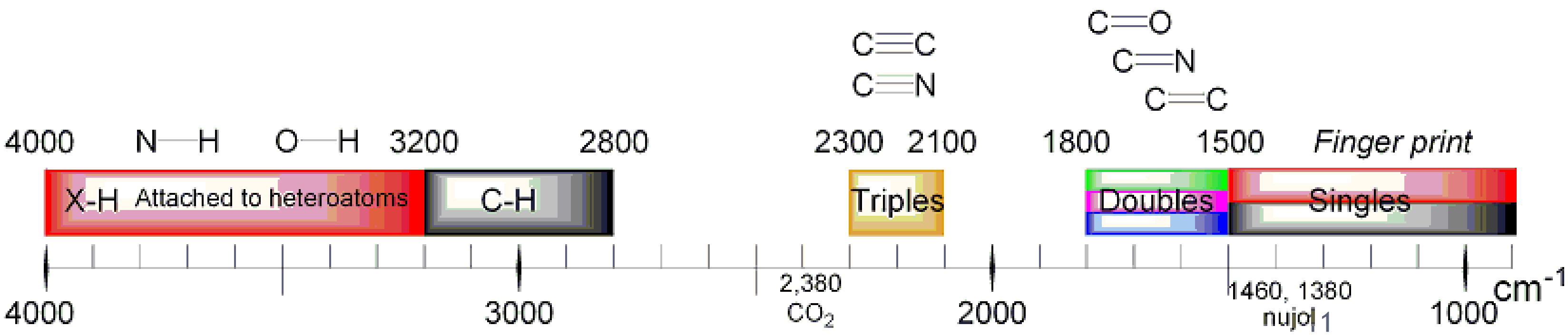
CHEMICAL BONDS

- Devices
  - FT-IR
  - XPS
  - Raman

Other methods: NMR. Auger, EELS

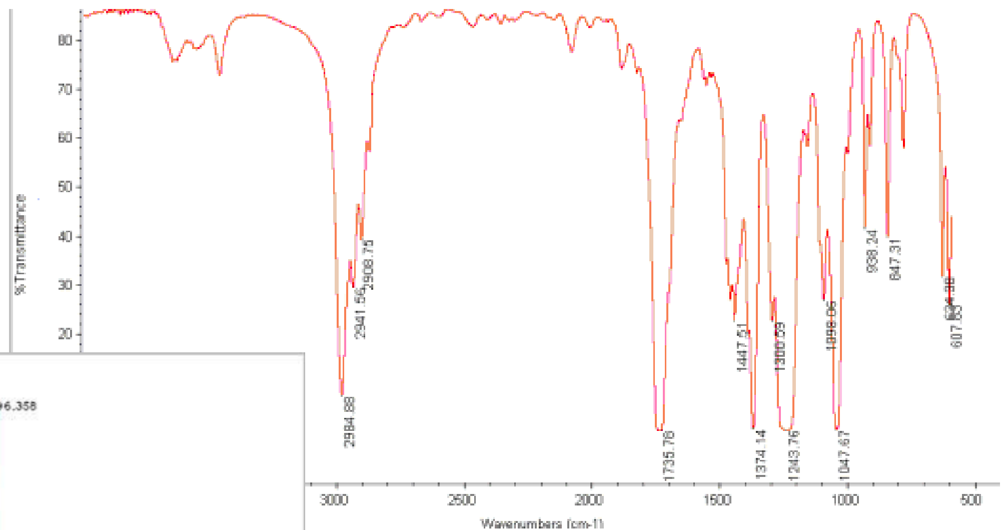
CHEMICAL BONDS - FT-IR

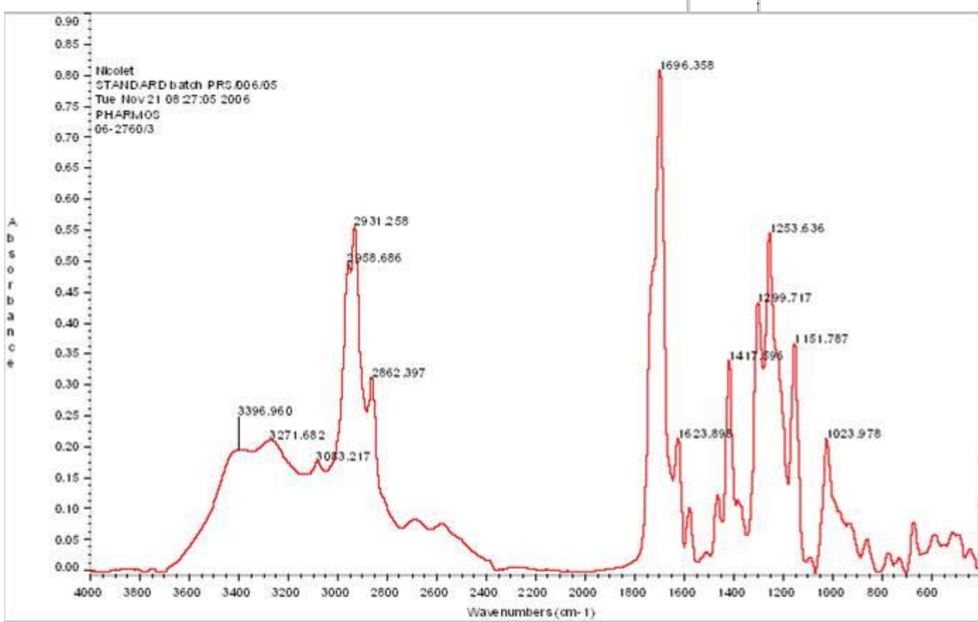
- It can be used to identify and study chemicals bonds by using IR to excite the structure of the sample
  - 14000–4000 cm−1 (0.8–2.5 μm wavelength) can excite vibrations
  - 4000–400 cm^{-1} (2.5–25 μm) may be used to study the vibrations and rotational-vibrational
  - 400–10 cm^{-1} (25–1000 μm), has low energy and may be used for rotational structure



CHEMICAL BONDS – FT-IR

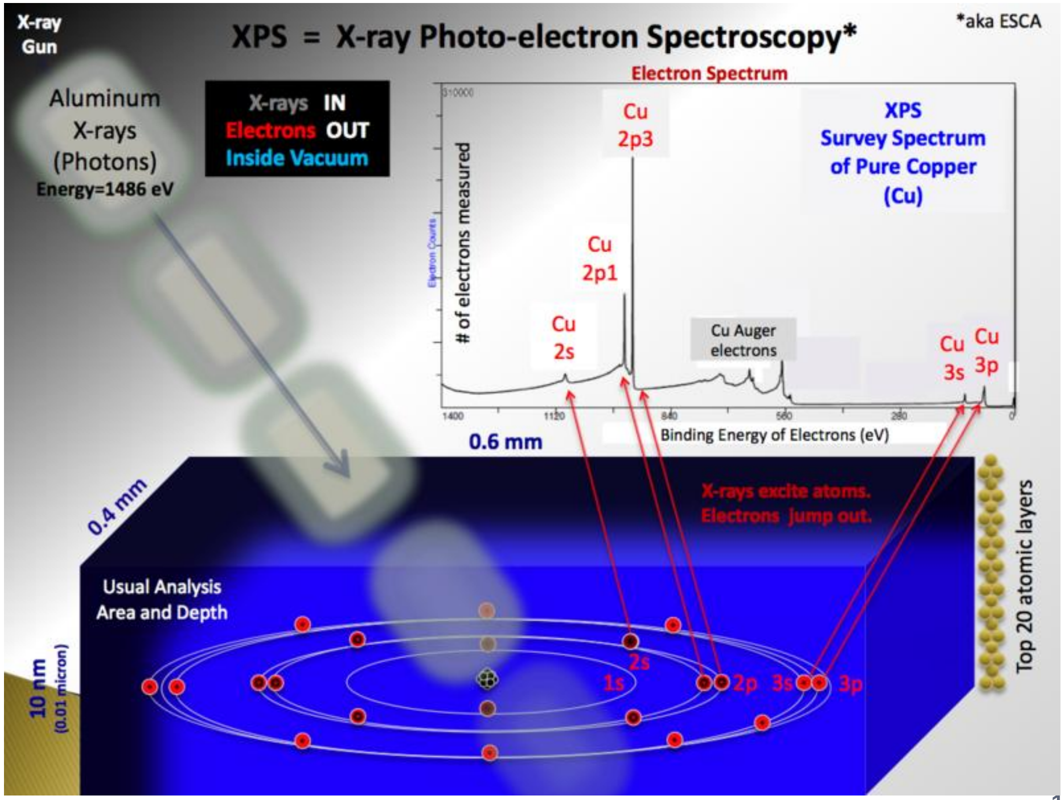
- The FR-IR can measure in two modes:
  - Transmitance
  - Absorbance





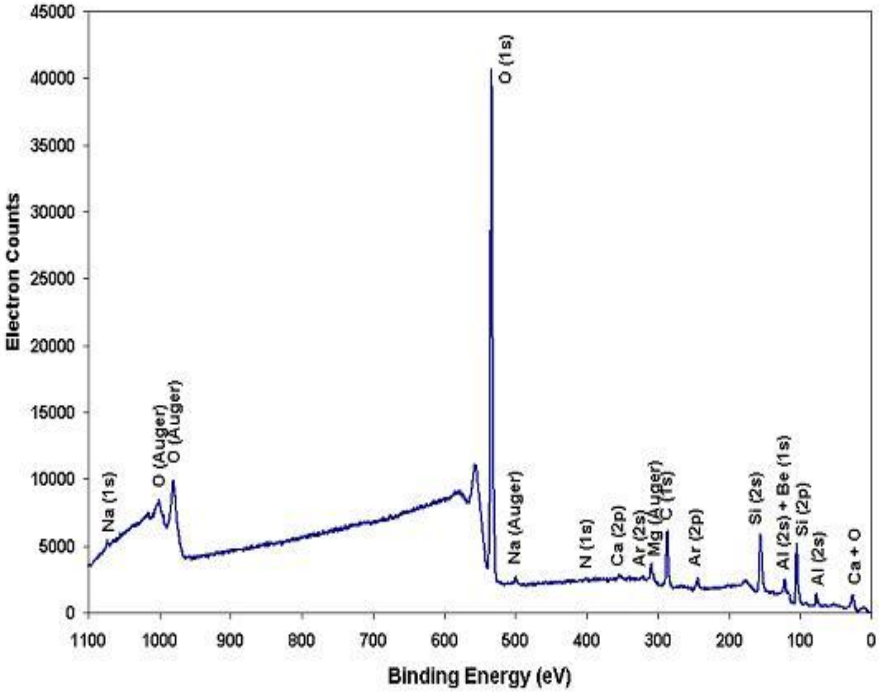
CHEMICAL BONDS – XPS

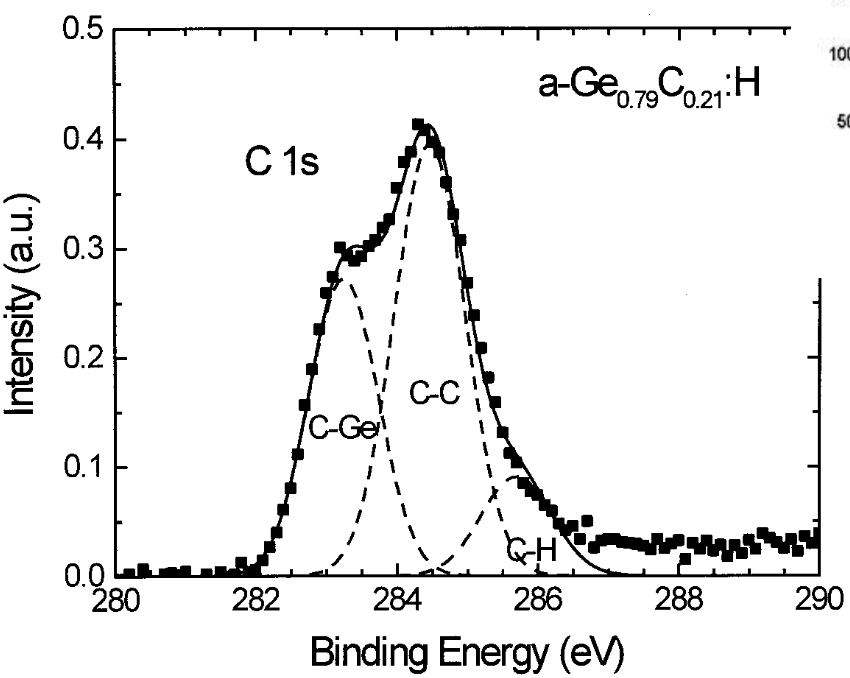
- Irradiation of a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape



CHEMICAL BONDS – XPS

- Two modes:
  - Mapping (survey)
  - Peak focus



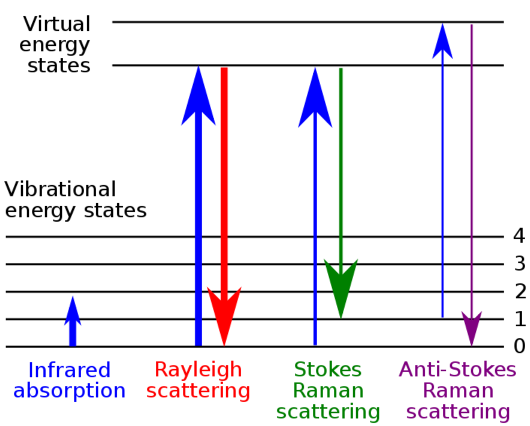


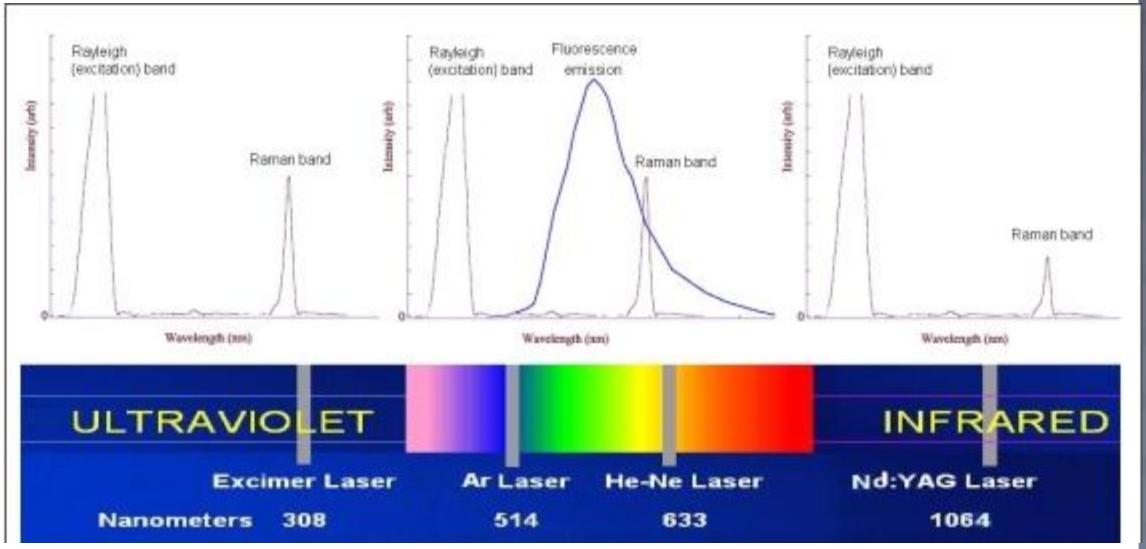
CHEMICAL BONDS – XPS

- Samples:
  - solids - metals, semiconductors, insulators
  - samples must be clean
  - no high vapor pressure materials
- Limitations:
  - does not detect H or He
  - detection limit of about 1 at %
  - sampling region diameter: 30 microns - 10 mm
  - requires ultra-high vacuum
  - beam damage on some samples
- **X-ray Photoelectron process**
  - X-ray incident on material with energy = hv
  - Excites electrons to be ejected from atoms
  - Electrons close enough to surface (30Å) can escape from the material
  - electron kinetic energy = hv - binding energy
- **Chemical State Information**
  - small differences in binding energy can be detected
  - allows identification of different chemical states
    - elemental Si has a different binding energy than Si in SiO_2
    - C bonded to C has a different binding energy than C double bonded to O
  - curve fitting of data
- **Angle resolved XPS**
  - depth profiling of near surface region can be done by rotating the sample limited mean free path changes depth from which photoelectrons are detected
  - depth profiling of surface region (100 Å) by angle resolved XPS
  - depth profiling using Ar ion sputtering

CHEMICAL BONDS – RAMAN

- Raman spectroscopy is used to study vibrational, rotational, and other low-frequency modes in a system and provides a fingerprint by which the molecule can be identified





CHEMICAL BONDS – RAMAN

- Two modes:
  - Mapping (survey)
  - Peak focus

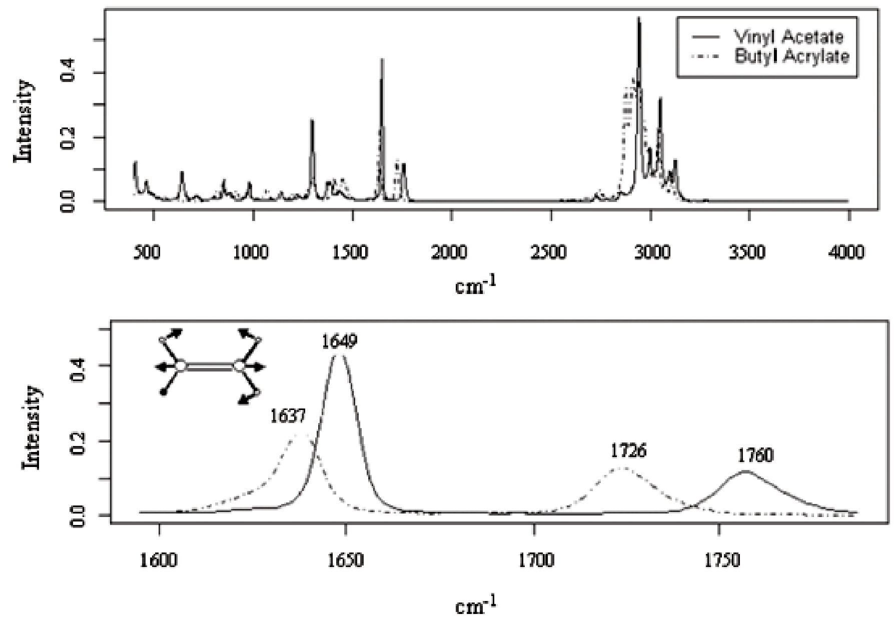
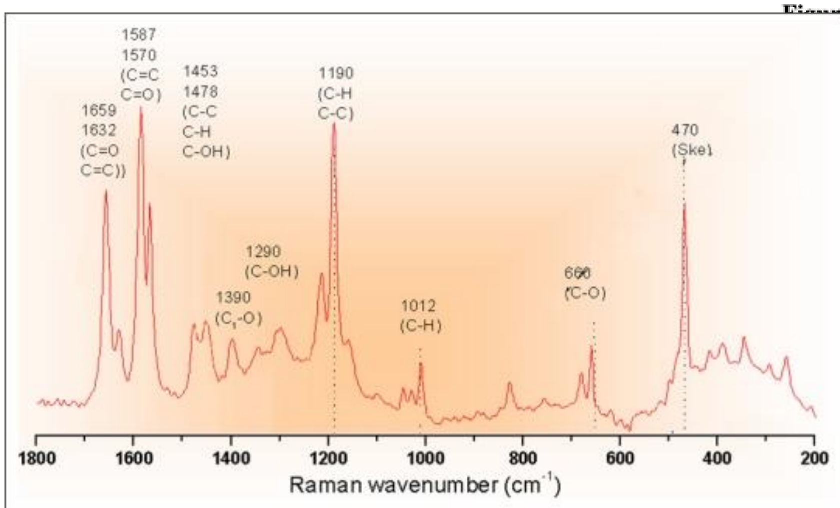


Figure 1: Raman Spectra of vinyl acetate and butyl acrylate. At the top: the spectral region between 400 and 4000 cm^{-1}; on the bottom: the bands due to C=C stretching (1637 and 1649 cm^{-1}) and C=O stretching (1726 and 1760 cm^{-1}).

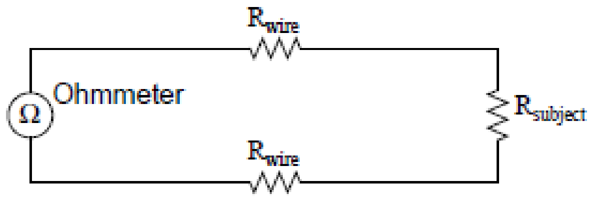


ELECTRICAL AND MAGNETIC PROPERTIES

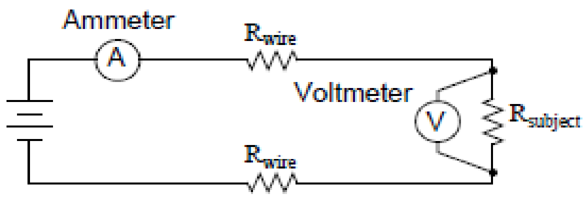
- Resistivity
- Conductivity (over frequency)
- Magnetic characterization

RESISTIVITY

- Standard measurement R=I/V



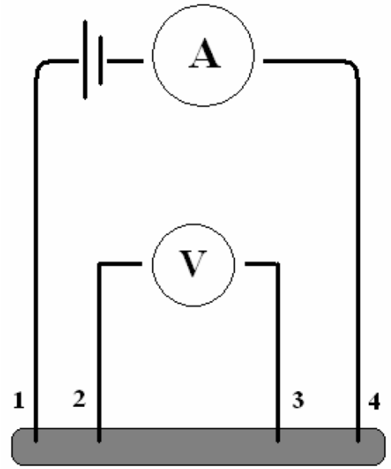
Ohmmeter indicates $R_{wire} + R_{subject} + R_{wire}$



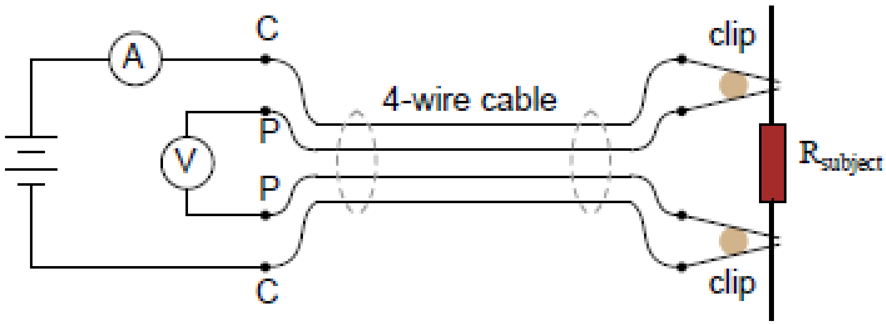
$$R_{subject} = \frac{\text{Voltmeter indication}}{\text{Ammeter indication}}$$

RESISTIVITY

- Four-terminal sensing (Kelvin sensing or 4T) is electrical impedance measuring technique



Four-point measurement of resistance between voltage sense connections 2 and 3. Current is supplied via force connections 1 and 4.

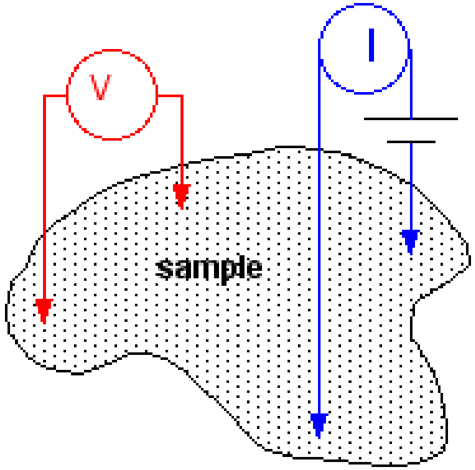


$$R_{subject} = \frac{\text{Voltmeter indication}}{\text{Ammeter indication}}$$

- eliminates resistance of wires
- still have contact resistance
- need to know spacing of inner probes to convert to resistivity
- careful not to puncture film with probes

RESISTIVITY

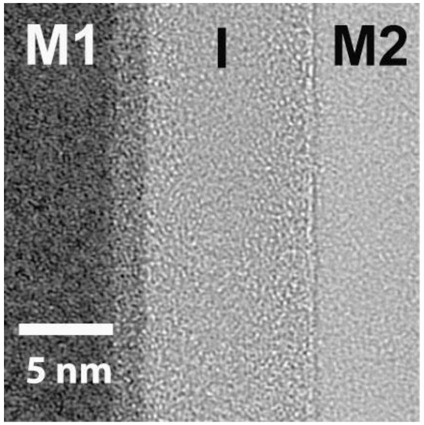
- Van der Pauw method

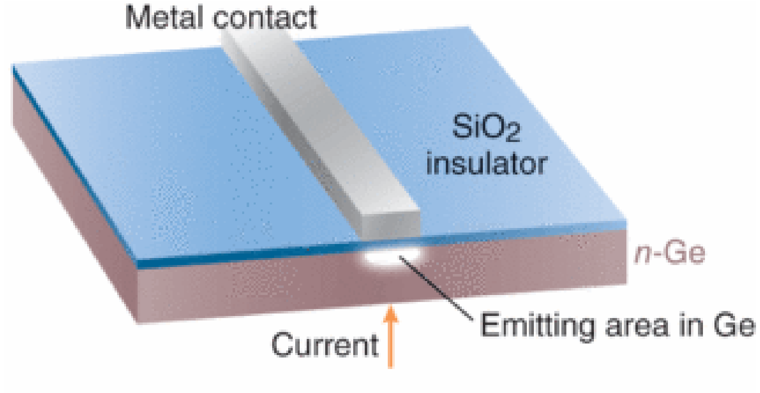


- placement of measurement points is arbitrary
- more mathematical analysis
- need two measurements with contacts alternated

CONDUCTIVITY (OVER FREQUENCY)

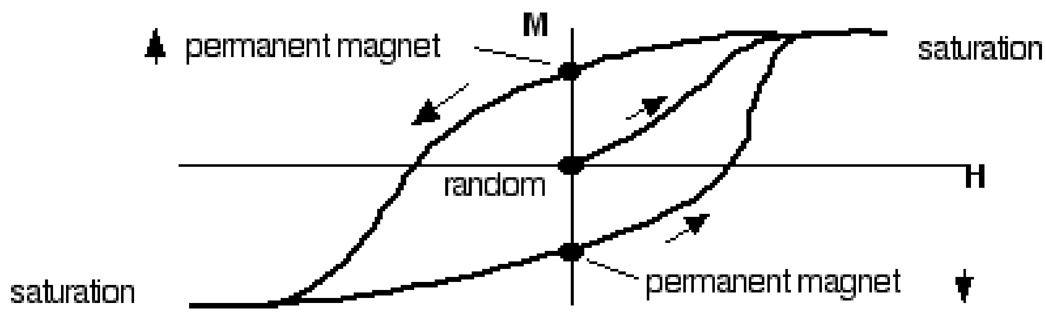
- Measures the conductivity (in respect to frequency)
- Two modes:
  - MIM (Metal Insulator Metal)
  - MIS (Metal Insulator Semiconductor)





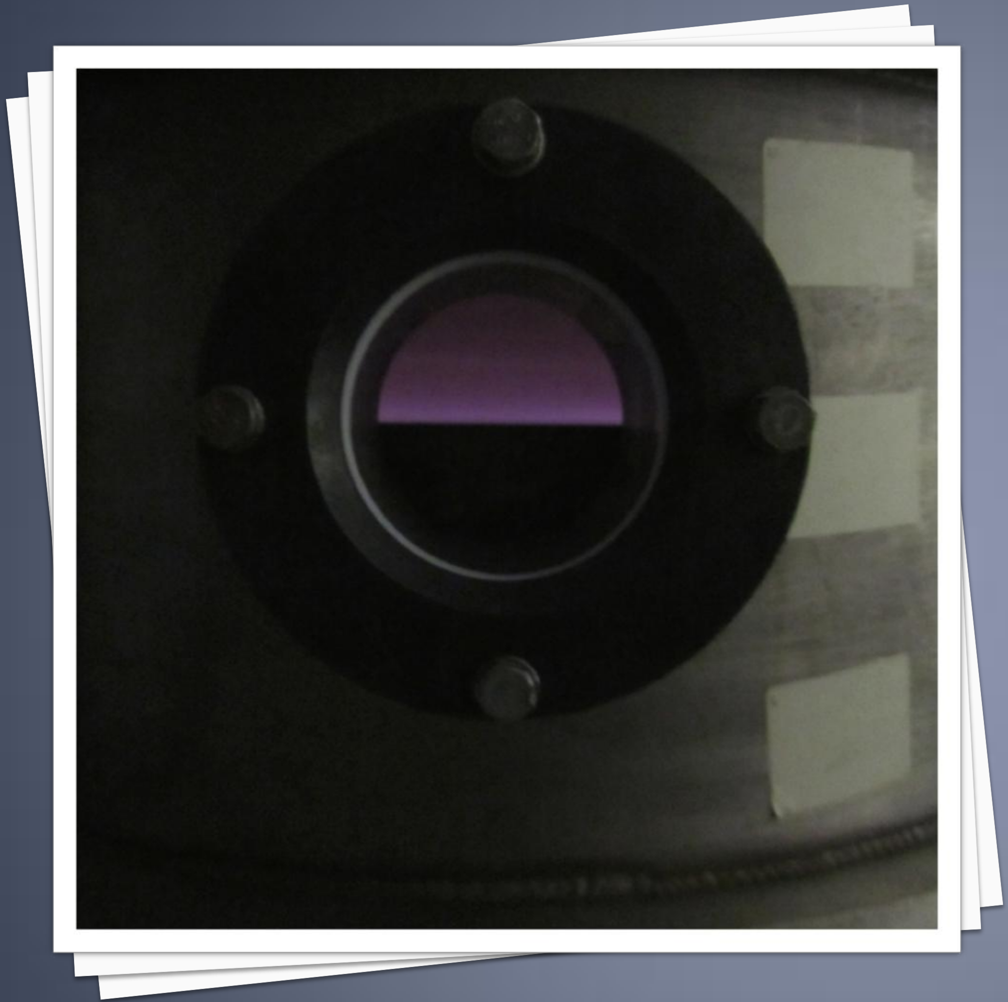
MAGNETIC CHARACTERIZATION

- Magnetic properties of thin films can often be described by a hysteresis curve:



Magneto-optical Kerr effect (MOKE)

- ellipsometry + a magnetic field on the sample magnetic materials cause an additional rotation of the polarization of reflected light.
- sensitive to thin films



THANK YOU FOR ATTENTION