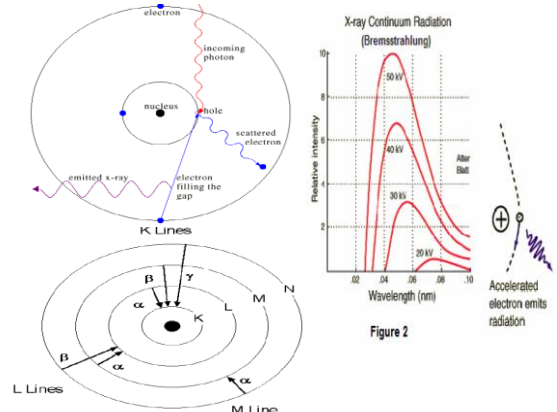
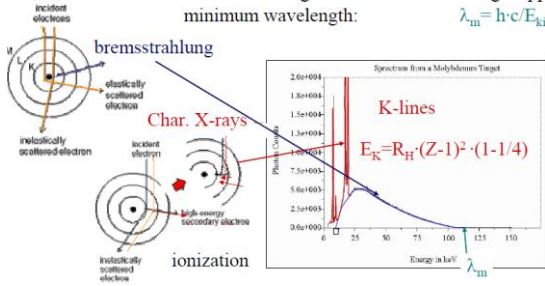


Physics of X-ray Sources

Char. X-Rays: $E_x = h\nu = hc/\lambda = \Delta E_{kin} = E_{kin}(i) - E_{kin}(f)$

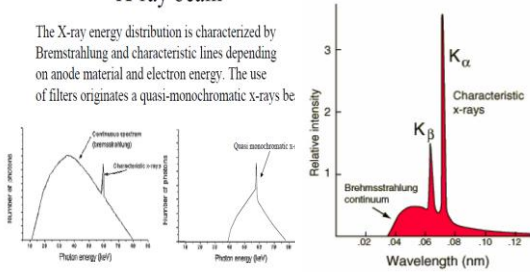
Bremsstrahlung: electrons being stopped;
 minimum wavelength: $\lambda_{min} = hc/E_{kin}(i)$



X-Rays

X-ray beam

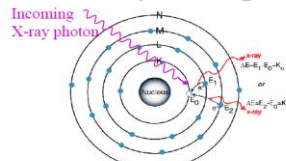
The X-ray energy distribution is characterized by Bremsstrahlung and characteristic lines depending on anode material and electron energy. The use of filters originates a quasi-monochromatic x-rays beam



Low energy x-rays are absorbed in filter material

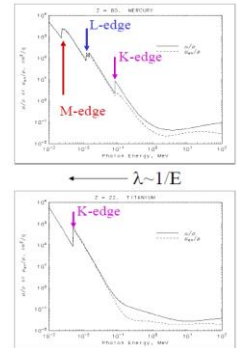
X-Rays

X-ray absorption mechanism



X-rays are increasingly absorbed in matter with decreasing energy
Absorption: $\mu \sim \sigma \sim E^{-3}$
 Absorption edges indicate the additional excitation of electron from an inner shell (M,L,K), which requires more energy.

As higher energy as less absorption



XRD

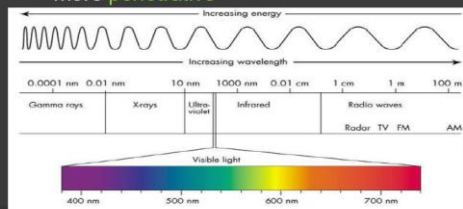
Introduction

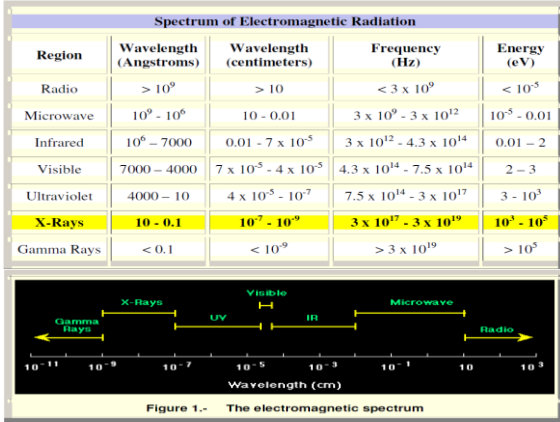
- **Motivation**
 - X-ray diffraction is used to obtain **structural information** about crystalline solids.
 - Useful in biochemistry to solve **the 3D structures** of complex bio-molecules.
 - Bridge between physics, chemistry, and biology.
- **X-ray diffraction is important for**
 - Solid-state physics
 - Biophysics
 - Medical physics
 - Chemistry and Biochemistry

XRD

What is X-ray

- Beams of electromagnetic radiation
 - * smaller wavelength than visible light,
 - * **higher** energy
 - * more **penetrative**





XRD

History of X-Ray Diffraction

- 1895 X-rays discovered by **Roentgen**
- 1914 First diffraction pattern of a crystal made by Knipping and von Laue
- 1915 Theory to determine crystal structure from diffraction pattern developed by Bragg.
- 1953 DNA structure solved by Watson and Crick
- Now Diffraction improved by computer technology; methods used to determine atomic structures and in medical applications

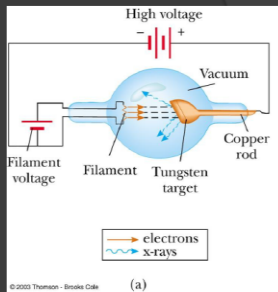


The first X-ray

XRD

X-ray Production

- When high energy **electrons** strike an **anode** in a sealed vacuum, **x-rays** are **generated**. Anodes are often made of copper, iron or molybdenum.
- X-rays are electromagnetic radiation.
- They have enough energy to cause ionization.



XRD

What is X-ray Diffraction (XRD)

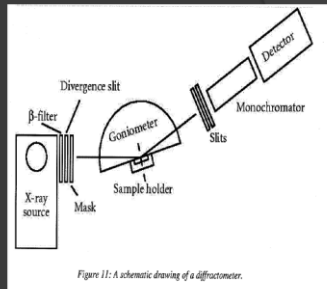
- Most useful in the characterisation of crystalline materials; **Ceramics, metals, intermetallics, minerals, inorganic compounds**
- rapid and nondestructive techniques
- Provide **information on unit cell** dimension



XRD

Components

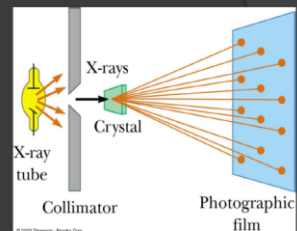
- X-ray source
- Device for restricting wavelength range "goniometer"
- Sample holder
- Radiation detector
- Signal processor and readout



XRD

How XRD works

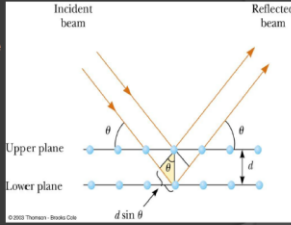
- A continuous beam of X-rays is incident on the crystal
- The diffracted radiation is **very intense** in certain directions
 - These directions correspond to constructive interference from waves reflected from the layers of the crystal
- The diffraction pattern is detected by photographic film



XRD

How Diffraction Works: Bragg's Law

- The beam reflected from the lower surface travels farther than the one reflected from the upper surface
- If the path difference equals some integral multiple of the wavelength, constructive interference occurs
- Bragg's Law gives the conditions for constructive interference



$$\lambda = 2d \sin \theta$$

XRD

How Diffraction Works: Schematic

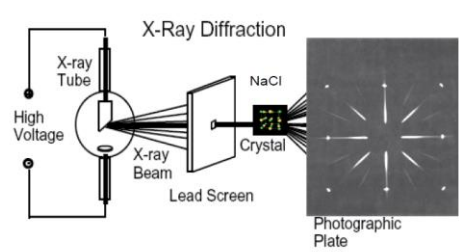
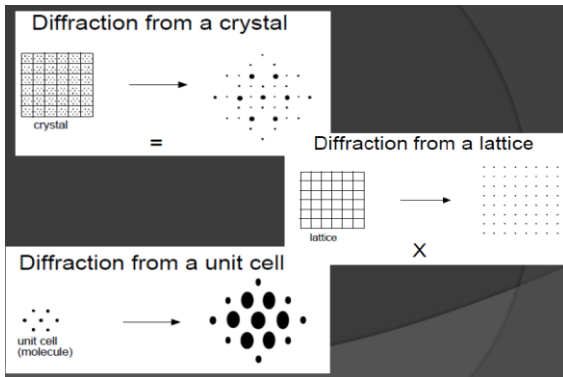


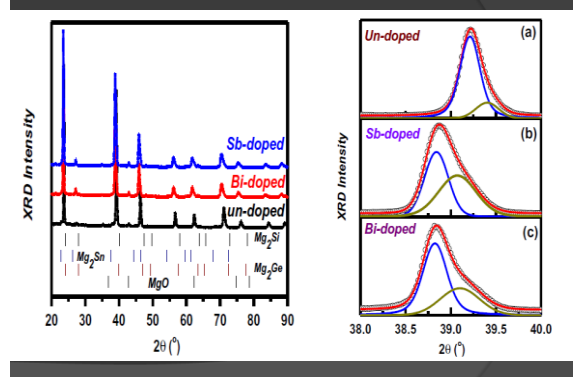
Figure 2. A schematic of X-ray diffraction.

<http://mrsec.wisc.edu/edetc/modules/xray/x-raystm.pdf>

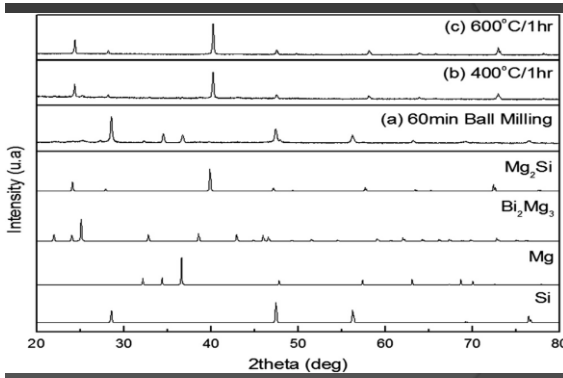
XRD



XRD



XRD



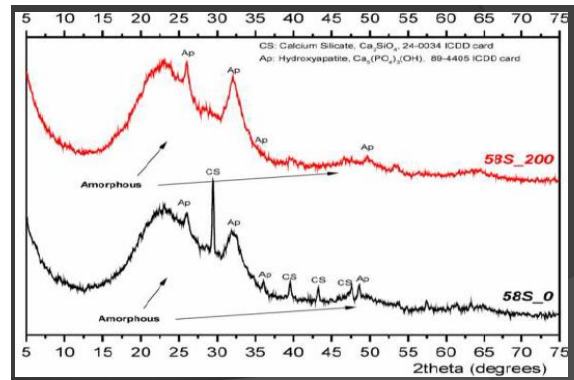
XRD

Single Crystal X-ray Diffraction

- Used to determine
 - crystal structure
 - orientation
 - degree of crystalline perfection/imperfections (twinning, mozaicity, etc.)
- Sample is illuminated with monochromatic radiation
 - Easier to index and solve the crystal structure because it diffraction peak is uniquely resolved

XRD**X-ray Powder Diffraction**

- More appropriately called polycrystalline X-ray diffraction, because it can also be used for sintered samples, metal foils, coatings and films, finished parts, etc.
- Used to determine
 - phase composition (commonly called phase ID)- what phases are present?
 - quantitative phase analysis- how much of each phase is present?
 - unit cell lattice parameters, crystal structure
 - average crystallite size of nanocrystalline samples
 - crystallite microstrain and texture
 - residual stress (really residual strain)

XRD**XRD****Applications of X-Ray Diffraction**

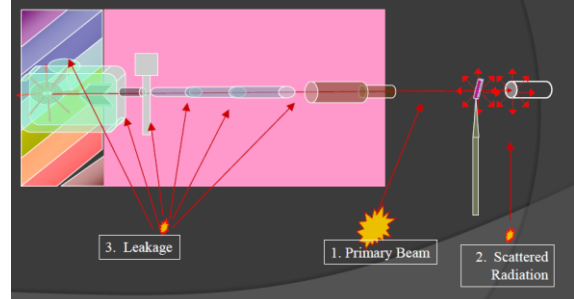
- Determination of **Crystal structure**
- Phase identification / transition
- Grain size / micro-strain**
- Texture/stress** (i.e. **polymer** , fiber)
- Determination of **thin film composition**
- Industry Identification of **archeological materials**

XRD**Advantages of XRD**

- Fast** identification of materials,
- Easy** sample preparation,
- Computer-aided** material identification,
- Large library** of known crystalline structures.

XRD**Safety in XRD**

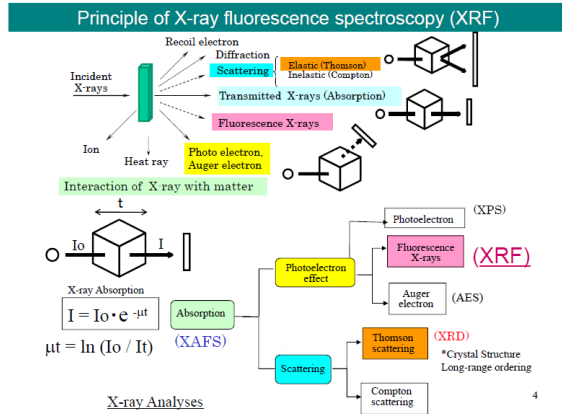
- Exposure types**
 - Short-term high-dose
 - Long-term low-dose
- Invisible, odorless, colorless** (most exposures undetectable)
- Lab users must understand radiation safety issues and pass an exam to use lab
- Safeguards present in lab do not substitute for knowledge and following safe procedures

XRD**What are the dangerous areas?**

XRD

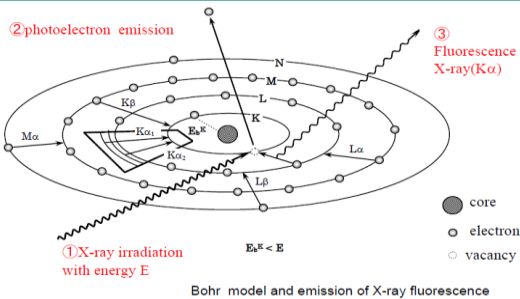
Summary & Conclusion

- X-ray diffraction is a technique for analyzing structures of biological molecules
- X-ray beam hits a crystal, scattering the beam in a manner characterized by the atomic structure
- Even complex structures can be analyzed by x-ray diffraction, such as DNA and proteins
- This will provide useful in the future for combining knowledge from physics, chemistry, and biology



XRF

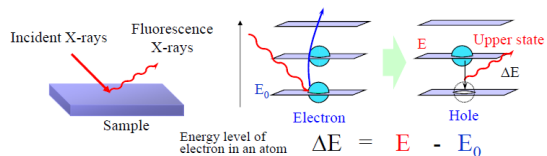
Principle of X-ray fluorescence spectroscopy (XRF)



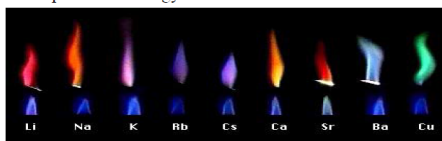
X-ray energy $E >$ Binding energy E_b

XRF

Principle of X-ray fluorescence spectroscopy (XRF)



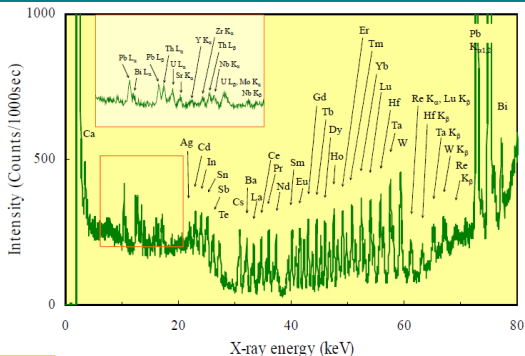
ΔE is equal to the energy difference between the two electronic state



ex) Flame reaction The color (energy) is unique to element

XRF

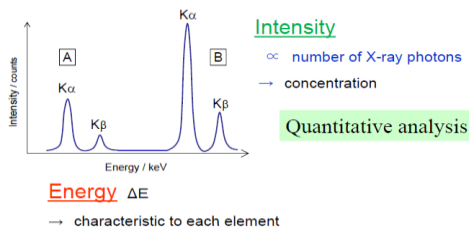
Principle of X-ray fluorescence spectroscopy (XRF)



XRF

Principle of X-ray fluorescence spectroscopy (XRF)

Measuring energy and intensity of XRF signal

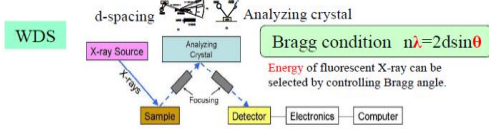


Qualitative analysis
 Chemical Composition Analyses

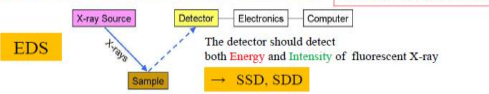
XRF

How to measure E and I of the fluorescence X-rays.

(a) Wavelength dispersive spectroscopy



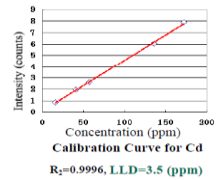
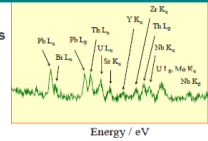
(b) Energy dispersive spectroscopy



XRF

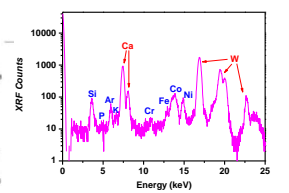
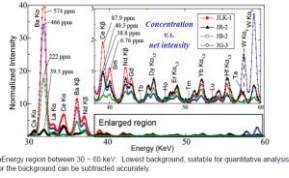
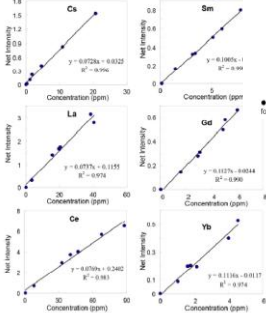
Procedure of X-ray fluorescence Analysis (XRF)

1. Check the chemical composition for samples
 - Qualitative Analysis
2. Select the best condition for XRF analyses
 - Combination of X-ray source, Detector, measurement time, etc.
3. Make calibration curve from standards
4. Calculate elemental concentration for the sample from the peak intensity
 - Quantitative Analysis



XRF

Calibration curves: 8 rock reference samples



Probes used

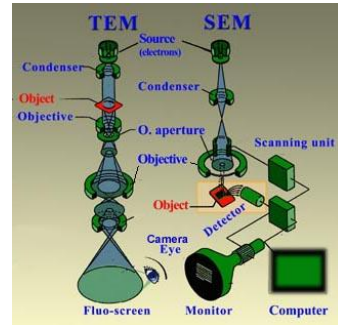
- Visible light
 - Optical microscopy (OM)
- X-ray
 - X-ray diffraction (XD)
 - X-ray photo electron spectroscopy (XPS)
- Neutron
 - Neutron diffraction (ND)
- Ion
 - Secondary ion mass spectrometry (SIMS)
 - Cleaning and thinning samples
- Electron
 - Scanning electron microscopy (SEM)
 - Transmission electron microscopy (TEM)
 - Electron holography (EH)
 - Electron diffraction (ED)
 - Electron energy loss spectroscopy (EELS)
 - Energy dispersive x-ray spectroscopy (EDS)
 - Auger electron spectroscopy (AES)

Electron Microscopy

Introduction and History

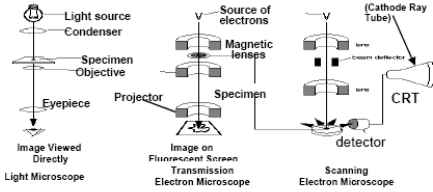
- o Electron microscopes are scientific instruments that use a beam of energetic electrons to examine objects on a very fine scale.
- o Electron microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light.
- o In the early 1930's this theoretical limit had been reached and there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus, mitochondria...etc.).
- o This required 10,000x plus magnification which was not possible using current optical microscopes.

SEM - TEM



SEM - TEM

Comparison of OM, TEM and SEM



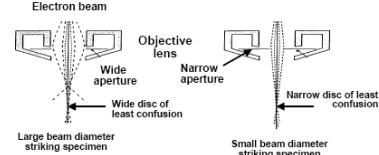
Principal features of an optical microscope, a transmission electron microscope and a scanning electron microscope, drawn to emphasize the similarities of overall design.

SEM - TEM

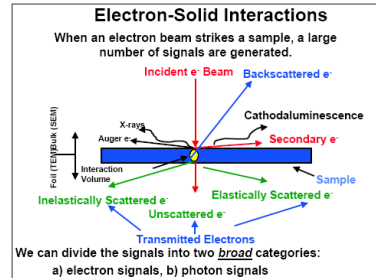
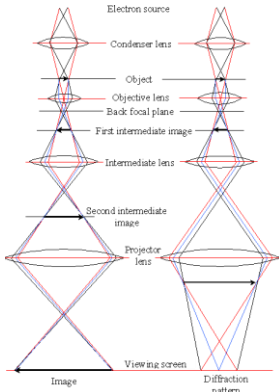
Electromagnetic Lenses

The Objective Lens - Aperture

- Since the electrons coming from the electron gun have spread in kinetic energies and directions of movement, they may not be focused to the same plane to form a sharp spot.
- By inserting an aperture, the stray electrons are blocked and the remaining narrow beam will come to a narrow "Disc of Least Confusion"



SEM - TEM



SEM - scanning electron microscopy

$$\lambda = h / (2m_{\text{electron}}qV_0 + q^2V_0^2/c^2)^{1/2}$$

Effects of increasing voltage in electron gun:

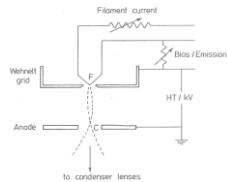
Resolution increased (λ decreased)

Penetration increases

Specimen charging increases (insulators)

Specimen damage increases

Image contrast decreases



SEM - scanning electron microscopy

Scanning electron microscopy is used for inspecting topographies of specimens at very high magnifications using a piece of equipment called the scanning electron microscope. SEM magnifications can go to more than 300,000 X but most semiconductor manufacturing applications require magnifications of less than 3,000 X only. SEM inspection is often used in the analysis of die/package cracks and fracture surfaces, bond failures, and physical defects on the die or package surface.

During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attracted and collected by a positively biased grid or detector, and then translated into a signal.

To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, and translated into images of the topography being inspected. Finally, the image is shown on a CRT.

SEM - scanning electron microscopy

- The energy of the primary electrons determines the quantity of secondary electrons collected during inspection. The emission of secondary electrons from the specimen increases as the energy of the primary electron beam increases, until a certain limit is reached. Beyond this limit, the collected secondary electrons diminish as the energy of the primary beam is increased, because the primary beam is already activating electrons deep below the surface of the specimen. Electrons coming from such depths usually recombine before reaching the surface for emission.
- Aside from secondary electrons, the primary electron beam results in the emission of backscattered (or reflected) electrons from the specimen. Backscattered electrons possess more energy than secondary electrons, and have a definite direction. As such, they can not be collected by a secondary electron detector, unless the detector is directly in their path of travel. All emissions above 50 eV are considered to be backscattered electrons.

SEM - scanning electron microscopy

- Backscattered electron imaging is useful in distinguishing one material from another, since the yield of the collected backscattered electrons increases monotonically with the specimen's atomic number. Backscatter imaging can distinguish elements with atomic number differences of at least 3, i.e., materials with atomic number differences of at least 3 would appear with good contrast on the image. For example, inspecting the remaining Au on an Al bond pad after its Au ball bond has lifted off would be easier using backscatter imaging, since the Au islets would stand out from the Al background.
- A SEM may be equipped with an EDX analysis system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen.

SEM - scanning electron microscopy

1.1 Characteristic Information: SEM

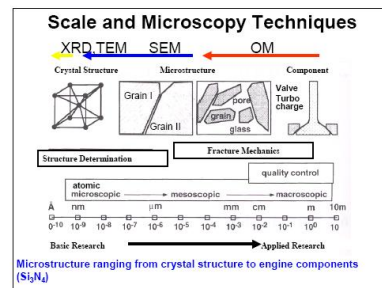
Topography
The surface features of an object or "how it looks"; its texture; direct relation between these features and materials properties

Morphology
The shape and size of the particles making up the object; direct relation between these structures and materials properties

Composition
The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties

Crystallographic Information
How the atoms are arranged in the object; direct relation between these arrangements and material properties

SEM - scanning electron microscopy



SEM - scanning electron microscopy

Advantages of Using SEM over OM

Mag	Depth of Field	Resolution
OM: 4x – 1400x	0.5mm	~ 0.2mm
SEM: 10x – 500Kx	30mm	1.5nm

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample.

The combination of higher magnification, larger depth of field, greater resolution, compositional and crystallographic information makes the SEM one of the most heavily used instruments in academic/national lab research areas and industry.

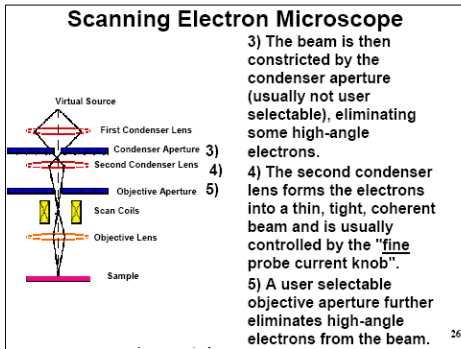
SEM - scanning electron microscopy

Scanning Electron Microscope

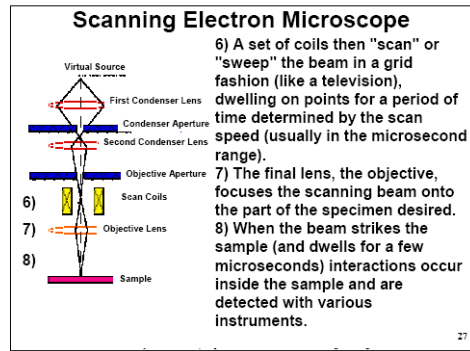
1) The "Virtual Source" at the top represents the electron gun, producing a stream of monochromatic electrons.

2) The stream is condensed by the first condenser lens (usually controlled by the "coarse probe current knob"). This lens is used to both form the beam and limit the amount of current in the beam. It works in conjunction with the condenser aperture to eliminate the high-angle electrons from the beam.

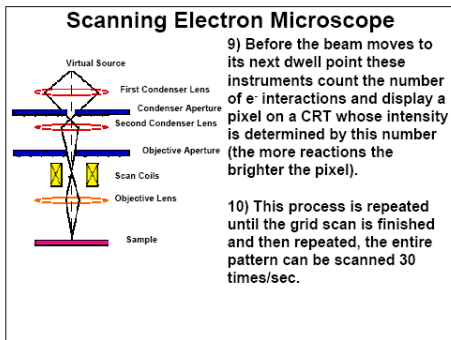
SEM - scanning electron microscopy



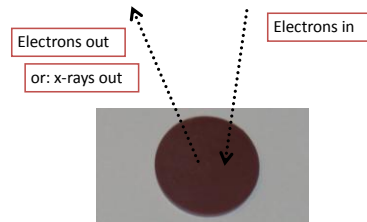
SEM - scanning electron microscopy



SEM - scanning electron microscopy



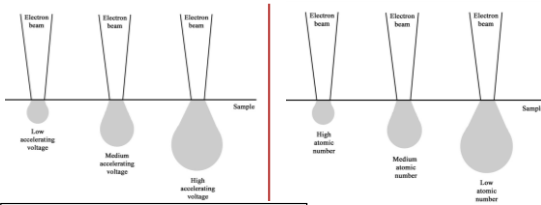
How do we get an image?



- In brief: we shoot high-energy electrons and analyze the outgoing electrons/x-rays

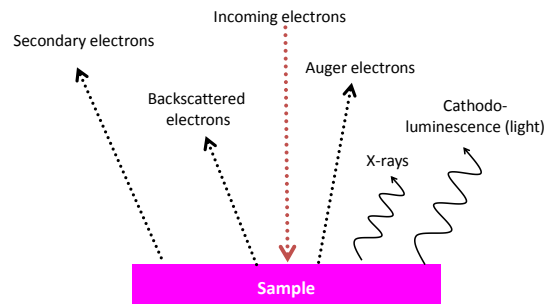
Electron beam-sample interactions

- The incident electron beam is scattered in the sample, both elastically and inelastically
- This gives rise to various signals that we can detect (more on that on next slide)
- Interaction volume increases with increasing acceleration voltage and decreases with increasing atomic number

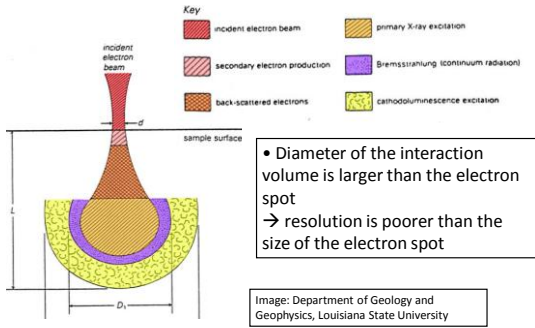


Images: Smith College Northampton, Massachusetts

Signals from the sample

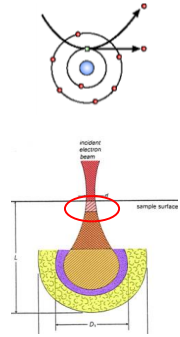


Where does the signals come from?



Secondary electrons (SE)

- Generated from the collision between the incoming electrons and the loosely bonded outer electrons
- Low energy electrons (~10-50 eV)
- Only SE generated close to surface escape (topographic information is obtained)
- Number of SE is greater than the number of incoming electrons
- We differentiate between SE1 and SE2



Why do we need vacuum?

- Chemical (corrosion!!) and thermal stability is necessary for a well-functioning filament (gun pressure)
 - A field emission gun requires ~ 10⁻¹⁰ Torr
 - LaB₆: ~ 10⁻⁶ Torr
- The signal electrons must travel from the sample to the detector (chamber pressure)
 - Vacuum requirements is dependant of the type of detector

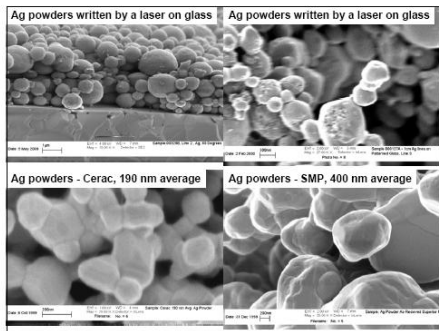
SEM - scanning electron microscopy

Specimen

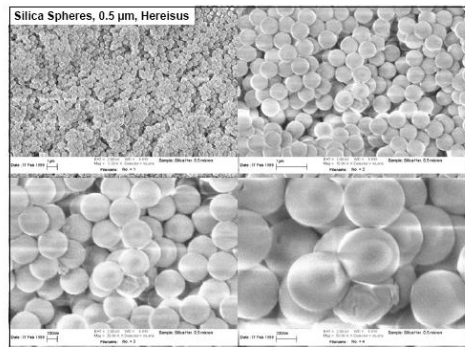
Can examine
fracture surfaces
electronic devices
fibers
coatings
particles
etc.



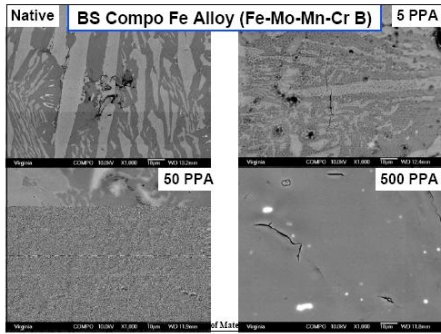
SEM - scanning electron microscopy



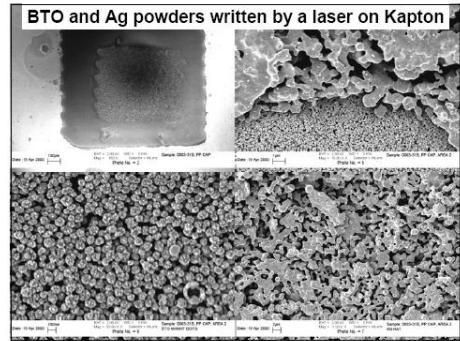
SEM - scanning electron microscopy



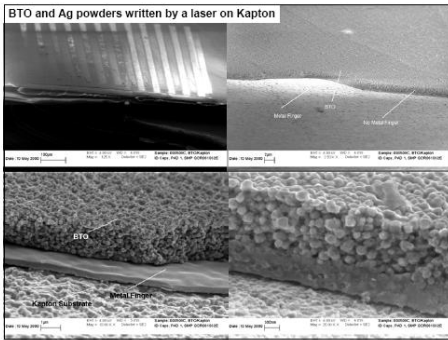
SEM - scanning electron microscopy



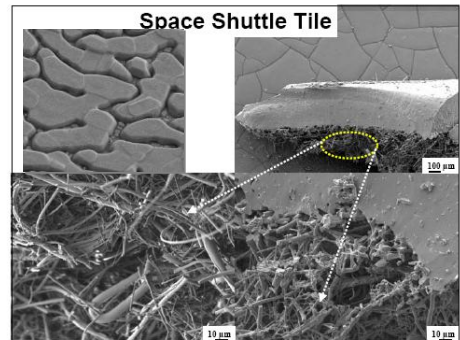
SEM - scanning electron microscopy



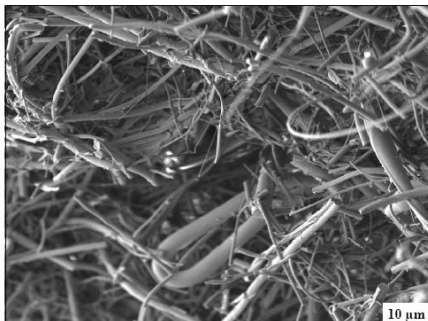
SEM - scanning electron microscopy



SEM - scanning electron microscopy



SEM - scanning electron microscopy



SEM - scanning electron microscopy

