

Course materials: http://in.materials.drexel.edu/blogs/515_experimental_techniques/

Experimental Techniques in Materials MATE 515

Physical Methods for Materials

Characterization:

Lecture 5 Cont (Ch 4)

Electron Microscopy: Interaction, Sample
Preparation , TEM

Instructor : Peter Finkel

MATE 515 Laboratory Demonstration Schedule

Facility	Supervisor	Location	Times /Date
OM	Superuser	MCF	Week 5 Oct 26 2 -4 pm
XRD	Zhorro Nikolov	MCF	Week 6 Oct 31
SEM/ESEM	Dee Breger	MCF	Week 7 Nov 6
WDS/EDS	Superuser	MCF	Week 8 Nov 14
OIM/EBSD	Superuser	MCF	Week 9 Nov 22
Magnetic	Peter Finkel	525 Bossone	Week 10 TBD

- Exact time of these demonstrations is to be confirmed one week in advance basis in agreement with the MCF staff for and will be offered at least 2 times /week for 3 groups.
- CORRECTION** : They are NOT optional, please make an effort to attend them, undoubtedly it will be helpful for your exams and, more importantly, your research.

HW 2 Q1

1. If the pattern had been obtained from a solid polycrystalline structure rather than from a powder sample, what would be relative intensities of X-ray diffraction lines then? Explain briefly your reasoning.
 - If texture present, preferable orientation ...intensities will be higher
 - Powder – less dense, intensity higher

HW2 Q2 Production of X-Ray

- The continuous spread of x-ray wavelengths is caused by the electron deceleration and this is called the continuous spectrum or bremsstrahlung or white spec

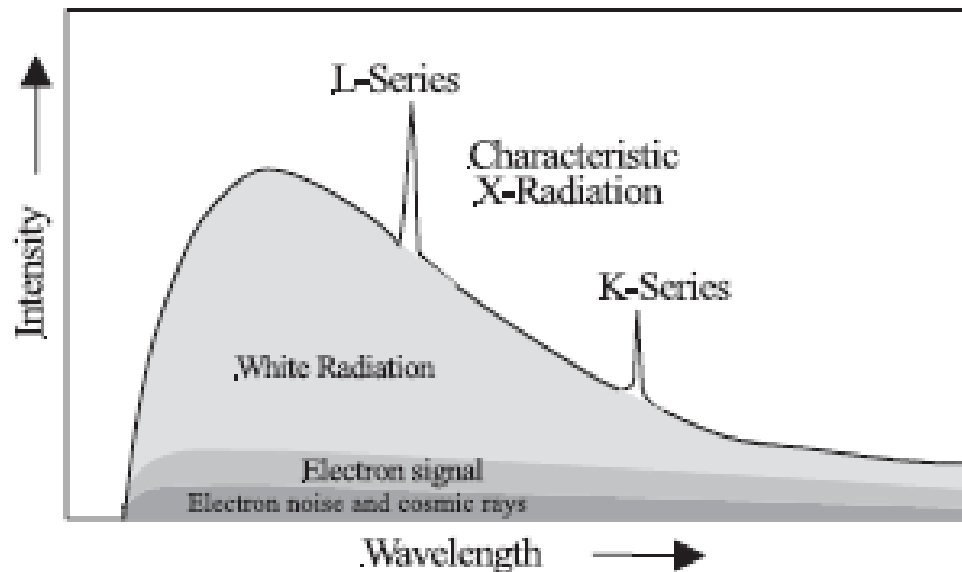


Figure 4.24. The characteristic X-ray spectrum from a metal illustrating K and L peaks superimposed on the continuous bremsstrahlung background.

X-ray production

- When the x-ray tube voltage is increased, high energy electrons are produced. When they give up all their energy at once to the atom when they hit the target, sharp intensity maxima appear at certain wavelengths superimposed on the continuous spectrum. These are called characteristic lines. They arise because the transferred electron energy ejects an inner shell electron from in to as x

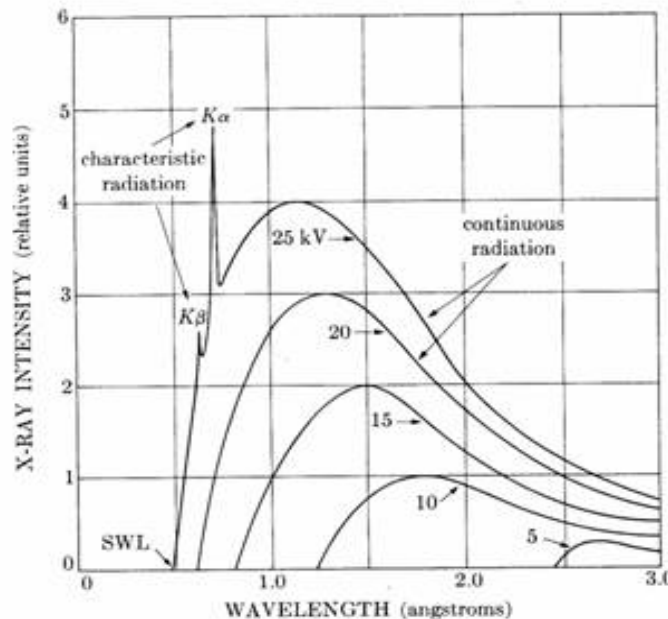


Fig. 1-4 X-ray spectrum of molybdenum as a function of applied voltage (schematic). Line widths not to scale.

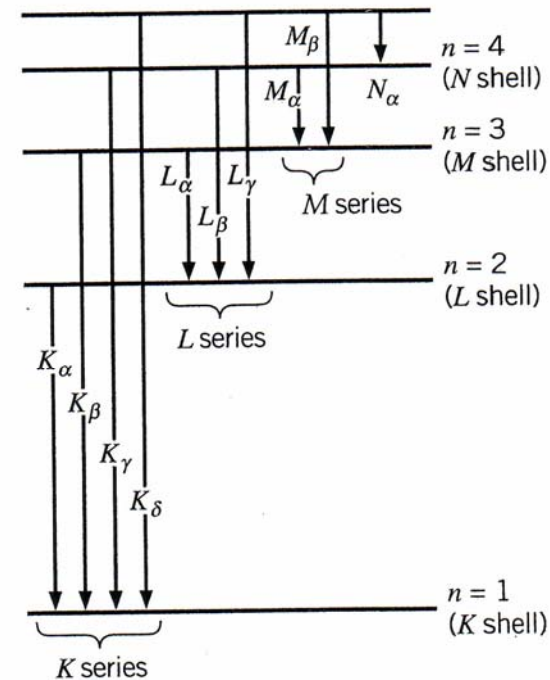


FIGURE 8.7 X-ray series.

HW2 Q5

Discuss how you would measure the following, including the x-ray wavelength (or spectrum) you would use, the measurement parameters, and likely data for a successful experiment.

Determine the crystal structure of an unknown crystal.

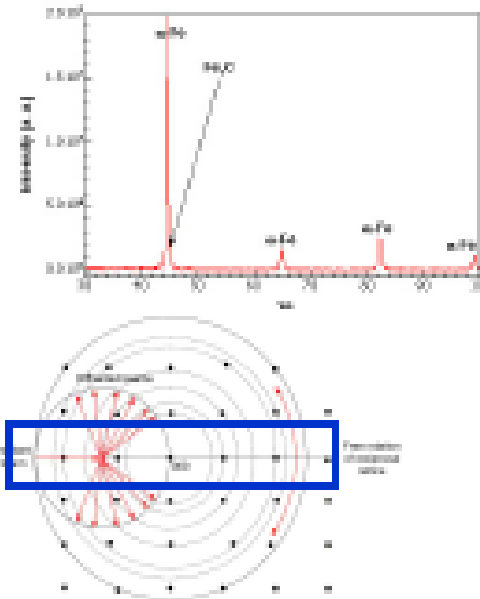
Monochromatic, Theta/2theta measurement, compare peak positions and intensities to jcpds file.

Determine the crystal structure of a bunch of particles.

Monochromatic, selected area diffraction.

Determine the coefficient of thermal expansion of the material in a single crystal.

Monochromatic, theta/2theta as a f(temp), calculate d at each temperature.



$$\Delta d = \frac{n\lambda}{\sin \theta' - \sin \theta_0}$$

Outline Electron Microscopy

Introduction

Review

- Principles:
- Lenses, defects and resolution
- Comparison of SEM and TEM
- Electron/Sample interactions
 - Scattering
 - Diffraction

Review - EM

- **Transmission: TEM (Included HIGH Voltage, HVEM and High Resolution, HREM)**
- **Scanning: SEM**
- **Combined: STEM**
- **Analytical: AEM (X-ray and Electron Energy Loss Spectroscopy, EELS)**

Review Common Features

- **Beam of electrons (@1keV- 5 MeV) with short wavelengths: $p = mv$, $mv^2/2 = eV$**

$$\lambda = h/p = h / (2m eV)^{1/2} \approx 1.226 / (V^*)^{1/2}$$

(nm)

$$V^* = V [1 + eV/2m(0)c^2]$$

V^* effective voltage corrected for relativity, $m_e(0)$ is the "rest mass" of the electron 9.11×10^{-31} kg.

- **1keV $\lambda = 39\text{nm}$,**
- **10keV $\lambda = 12\text{nm}$,**
- **100keV $\lambda = 3.7\text{nm}$,**
- **1 MeV $\lambda = 0.87\text{nm}$**
- **10MeV $\rightarrow \lambda = 0.12\text{nm}$.**

Common Features EM

- **"High" Vacuum** (10^{-6} torr (mm of Hg) $\approx 10^{-9}$ atm)
- **Magnetic (convex) lenses**
- **In TEM, the same principles are used as for OM - Small region illuminated by radiation and magnified image formed on a screen in the microscope.**
- **In SEM, Focused beam hits sample - different signals produced (secondary/Auger/ primary electrons) X-rays, etc. Beam Scanned over sample, signal detected, amplified and used to modulate CRT scanning, synchronised with e beam scan. (Same scan generator)**

Introduction

Transmission Electron Microscope (TEM) versus Transmission Optical Microscope (TOM)

	TEM	TOM
Source	100-400 kV electron gun high current densities: $5 \times 10^4 \text{ Am}^{-2}$ for tungsten filament $1 \times 10^6 \text{ Am}^{-2}$ for field-emission source	Light source
Condenser lens	Electromagnetic lens, focus adjusted by controlling the lens current	Glass lens, focus adjusted by lens position
Specimen stage	Allows for specimen tilt as well as some z-adjustment	Allows for specimen tilt as well as some z-adjustment
Objective lens	Fine focusing of the image by adjusting the lens current	Fine focusing by adjusting the position of the specimen and the objective lens
Final imaging system	Employs electromagnetic lenses to produce image on a fluorescent screen	Eye piece forming image for direct viewing
Recording system	Computer monitor or TV	Normal viewing or photographic films
Experimental set-up in	Vacuum, better than 10^{-6} torr	Air, at atmospheric pressure

Common Parts

- **Common parts of the systems (TEM/SEM):**

i) **Vacuum. Conventionally two pumps:**

Rotary Pump - RP 1 atm \rightarrow 0.01-10⁻³torr (1-10⁻¹Pa.)

– 1 atm (760torr = 1 bar = 0.101 MPa = 10⁵ Pa)

– 1 torr (1mm of Hg) \approx 10⁻²Pa

Diffusion Pump DP 10⁻³ to 10⁻¹¹ torr (10⁻² to 10⁻⁹Pa)

Low vapor pressure liquid. (Usually Silicon Oil)

Common Parts - Vacuum

- **In conventional "High" Vacuum:**
 - **Hard rubber "O -ring "seals.**
 - **Silicon (low VP) Vacuum grease**
 - **"Pirani" Thermal Conductivity, low vacuum, gauge**
 - **"Penning" Ionization, high vacuum, gauge.**

Common Part - Vacuum

- **Diffusion pump must be first evacuated before heating - also must be water, or in some cases air, cooled.**
- **Pump out D.P. with the "Backing" R.P. , heat D.P. and close backing valve. "Rough" pump the apparatus until it reaches safe level $\approx 10^{-3}$ torr. Close roughing valve, reopen backing valve and open baffle valve.**
- **Baffle valve should be cooled with Liquid nitrogen to stop oil back streaming into chamber.**

Vacuum issues (cont)

- **Ultra High Vacuum.** $> 10^{-10}$ torr.
Surface Studies.
- **At 10^{-6} torr, a surface atom is struck by a gas atom every second.**
- **At 10^{-10} torr, a surface atom is struck by a gas atom every hour.**
- **Use Ion, cryo or titanium sublimation pumps.**
- **Metal Seals (not reusable) and no grease.**
- **Metal "Bellow" Seals for motion.**

Vacuum issues – cont.

- Apparatus must be "baked" at $\approx 200^{\circ}\text{C}$ overnight to get rid of adsorbed gases before use.
- "Clean" or "Dirty" vacuum. Hydrocarbons from oil and human fingers is polymerised under electron beam.
- After short period of high mag. scanning, lower the mag. - black square shows where you have been.
- Under stationary beam either a "ring" is formed or sometimes a spot - a "witches hat". Oil arriving by surface diffusion is polymerised before or when it reaches the center of beam.
- Do not handle samples with fingers. Wash with solvent and use Plastic Gloves.
- Ideally do not put polymer samples in the TEM, they decompose under the beam and dirty the chamber.

Electron Microscopy

- Major components are the electron column consisting of an electron gun and the electron lenses, and the control console consisting of a cathode ray tube viewing screen and the scanning and control electronics for the electron beam.
- The three dimensional appearance of the image is due to the large depth of field.
- A scanning electron microscope (SEM) employs a probe lens to focus the electron beam into a fine probe and scanning coils are used to scan the probe over the sample.
- Resolution of the SEM is controlled by the probe lens. It is the inelastically scattered electrons that provide information. Usually < 30 keV electrons are used in SEM.
- In optical microscopy and TEM, information is collected continuously over the full field of view (from all image points simultaneously) and focused by suitable lenses to form a magnified image. In SEM, information is collected sequentially.

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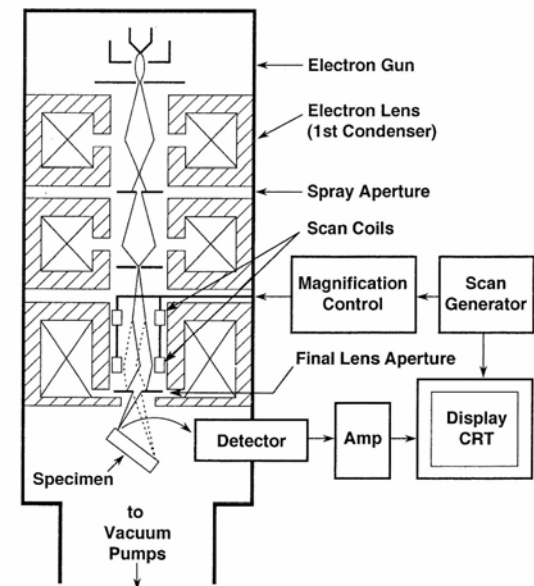


Figure 2.2. Schematic drawing of the electron column showing the electron gun, lenses, the deflection system, and the electron detector.

Electron Sources

1. Thermionic emission; f is the work function:

$$f = E_v - E_f \text{ (eV)}$$

- Tungsten (W) hairpin. Directly heated by dc current
- LaB6. Indirectly heated

$$J = A T^2 \exp(-f/kT)$$

2. Field emission - very sharp point (need not be heated)

3. Photo-emission. Here the sample is the electron emitter. In the PEEM, different parts of the sample with different values of f give, when illuminated by UV light (often plus heat), different currents which are imaged.

- All require STABLE high voltage source.
- (Expensive. Cost rises with keV).

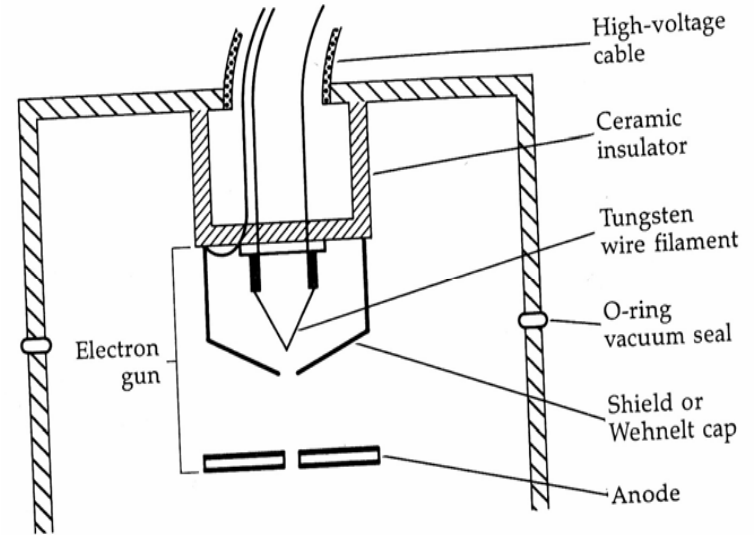
Electron gun

- The filament is resistively heated to 2000 – 2700K by applying a high voltage and a small amount of current to a point that valence electrons are released from its tip in what is called a space charge cloud. The amount of energy needed to cause electrons to leave the filament is called the **work function**.
- The electrons are released in all directions. The Wehnelt cap/grid has a slight negative potential (charge) - or excess of electrons - to create negative lines of force that focus the emitted electrons and control their emission.
- The filament or **cathode** is supplied with a high negative voltage, e.g. -20,000 volts. The **anode**, a metal plate with a hole in it, is at ground potential (0 volts) and is greatly positive with respect to the cathode. This potential difference accelerates the electrons toward the anode.

Electron gun

- Along the route from the cathode to the anode, the paths of the individual electrons cross each other. This is called the point of **crossover** with diameter d_0 . It is considered the **real source** in the electron gun, and its image is projected onto the specimen surface. After the crossover, the electron beam diverges with a divergence angle α_0 . The condenser and objective lenses then produce a demagnified image of the crossover on the specimen with an image diameter d_p .

- The **anode** has a hole in it. This hole allows only a fraction of the electrons to continue down the column toward the lenses. The remaining electrons are collected on the anode and returned via the ground to the voltage supply.



Electron Sources

- Hot Cathode Guns: Tungsten filaments and Lab-six – LaB6 crystals, producing thermionic emission of electrons.
- Thermionic emission formula:
 - Current density $I = AT^2 \exp(-E/kT)$
 - A = Richardson's constant depending on the source material,
 - T = emission temperature K (C+273),
 - E = work function or the energy required to escape from the filament into the vacuum,
 - k = Boltzman's constant.

- **Tungsten:**

- stable beam current
- short life
- large tip
- large emitting area (probe diameter)
- low brightness
- high work function
- high evaporation rate
- lower vacuum (10⁻⁶ Torr)
- low resolution
- 2700 K

- **LaB₆:**

- stable beam current
- 10 times longer life
- smaller tip
- smaller emitting area (probe diameter)
- higher brightness (10 times higher current)
- lower work function
- medium evaporation rate
- higher vacuum (10⁻⁷ Torr)
- higher resolution (due to thinner beam)
- 1700 K

Electron Sources (cont.)

- W hairpin;
- $T = 2700\text{K}$, $J = 1.75 \text{ amps /cm}^2$ at 25keV , $a \approx 5 \times 10^{-3}$ radians.
- Typical lifetime 50hrs. $\text{Vac} < 10^{-5}$ torr
- Brightness: $\beta \approx 3 \times 10^5 \text{ amps/cm/steradian}$
- Pointed W filament: $\beta \approx 10^6$ Lifetime 20hrs.
- LaB₆: $T = 1850\text{K}$, $\beta \approx 6 \times 10^6$ Lifetime 300-1000hrs.
Higher vacuum (10^{-8} torr) Higher cost.
- Cold Field Emission. Field V/r. $\beta \approx 10^9$
Lifetime vacuum dependent but needs 10^{-10} torr.
Very expensive. (Also current limited)
- Higher brightness mainly by reduced spot size at **crossover**.

EM intro

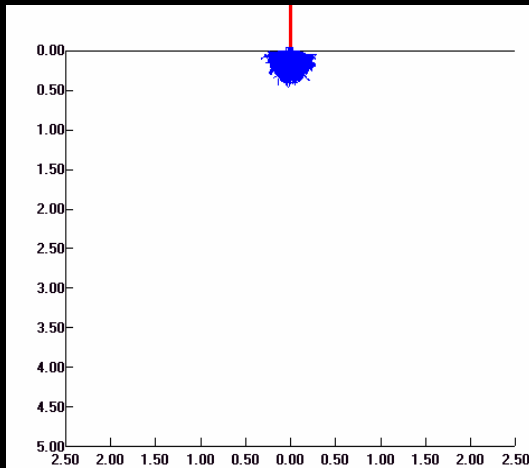
- [MATE 515 Brandon Ch 4.pdf](#)
- See PDF attached separately

Electron Microscopy

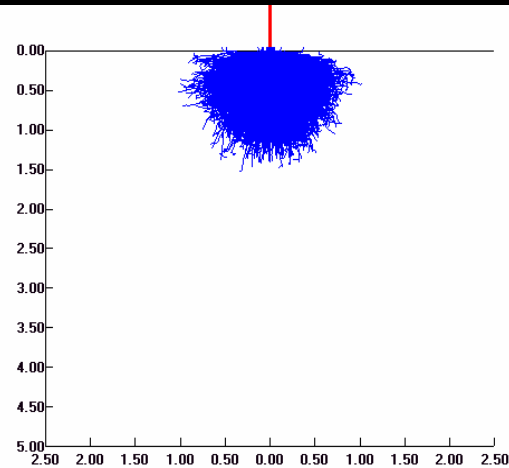
- TEM
 - Specimen preparation.
 - Contrast mechanisms.
 - Introduction to SEM

SEM

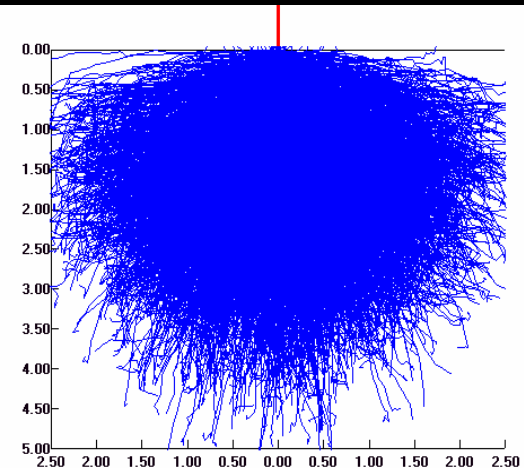
- •Electron-beam specimen interactions
- •Resolution
- •Imaging
 - •back-scattered electrons
 - •secondary electrons
 - •other imaging modes
- •Specimen preparation



5 kV



10 kV



20 kV

When a focused nanometer sized electron beam enters a specimen, it interacts with a larger volume in micrometer dimensions.