Chapter 5 Electron Diffraction

5.1 Production of electron beams

5.2 Electron Diffraction

5.3 Indexing Electron Diffraction Patterns

5.4 Type of Electron Diffraction

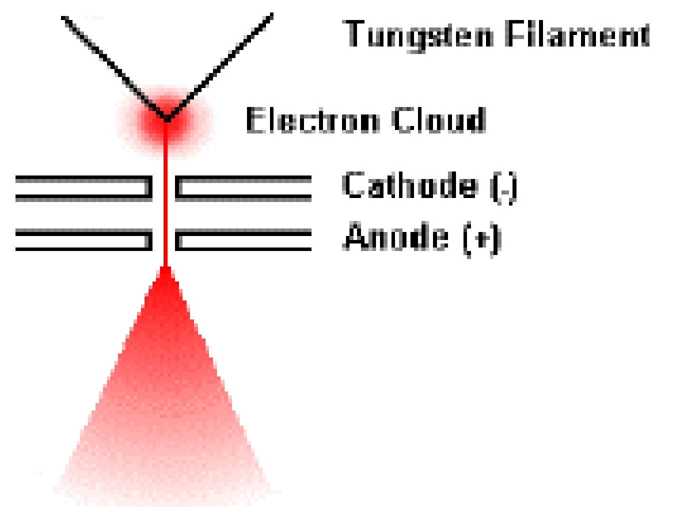
Learning outcomes :

By the end of this topic, student will be able to do the following:

- Define and explain the duality properties of the electron.
- Identify and explain the production of electron.
- Define the electron scattering/diffraction.
- Write down the type of electron diffraction.
- Identify and draw the diagram of each electron diffraction method.
- Explain and show the basic working principle for each electron diffraction method.
- Identify the diffraction pattern for each method.
- Explain the electron diffraction pattern.
- Relate the pattern with the crystal system.
- Identify the crystal system using electron diffraction method.

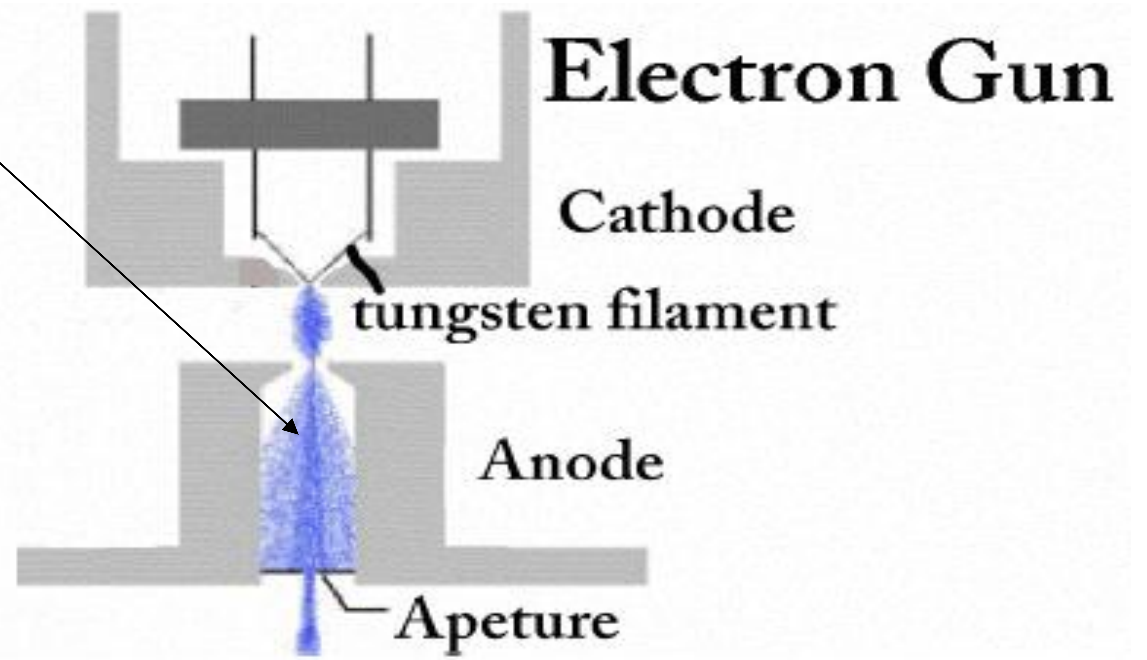
5.1 Production of electron beams

- An electron (beams) gun is used to produce a stream of electrons with a well defined kinetic energy for diffraction or other purposes.
- E-beams are mostly produced by ***thermionic emission*** from a heated cathode.
- An electron gun consist of two parts :
  - A heated filament,
  - The accelerating region, which is bounded by two electrodes, known as the cathode and the anode.



- The filament consists of a piece of wire, commonly made of a refractory material such as tungsten, which is heated by an electric current
- They drift through a small hole in the cathode, into a region where there is an electric field, which accelerates them across the gap to the anode.

They then pass through a hole in the anode, with a final energy which is determined by the applied voltage.



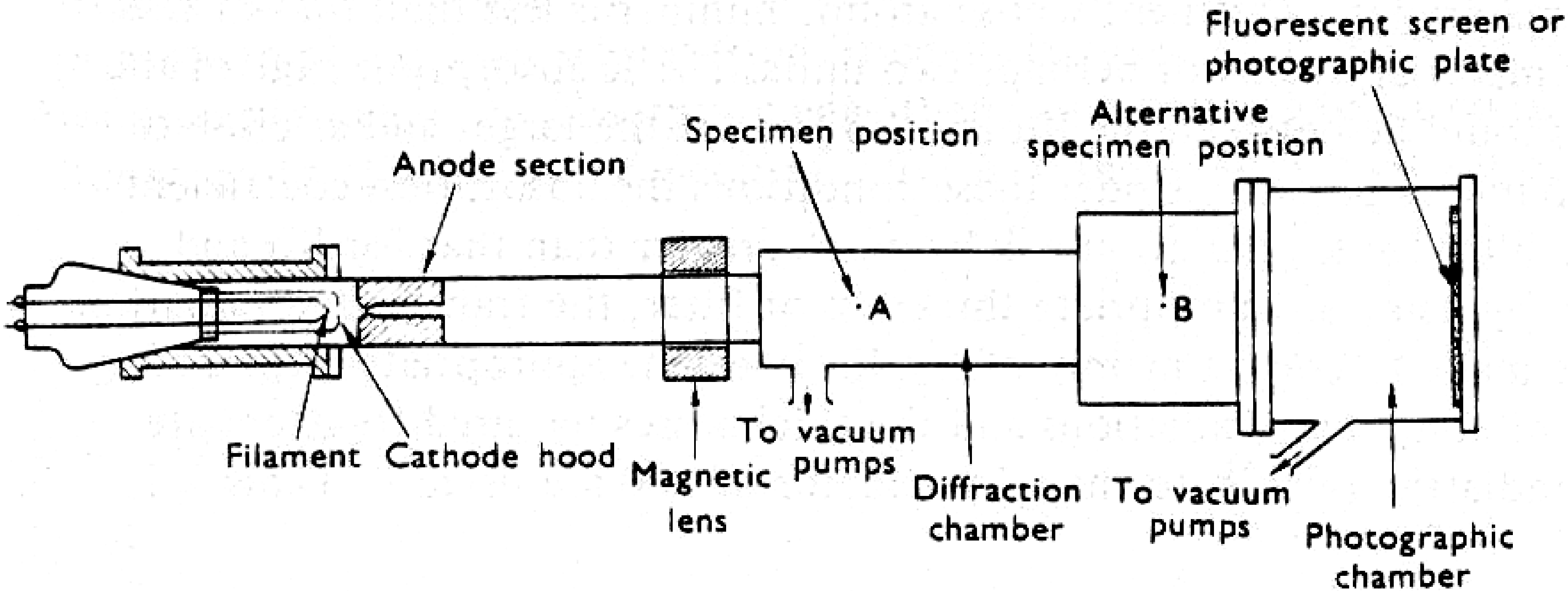
- ***Thermionic emission*** is a process by which some of the electrons inside a piece of metal can '*boil away*', that is leave the surface of the metal into the surrounding space.

- Inside a metal the electrons are not stationary, but are constantly moving, with an average speed which is controlled by the temperature of the metal.

- Since the speed of the electrons increases with temperature, the number of electrons with sufficient energy to escape also increases with temperature, in fact exponentially.

- At room temperature (300 K) the number is very small, but if the wire is heated to 1000K the number of electrons escaping is dramatically increased.

- The electron gun are very much the same as those of an electron microscope.
- In fact, many electron microscopes are designed so that they can also be used for obtained diffraction photographs.

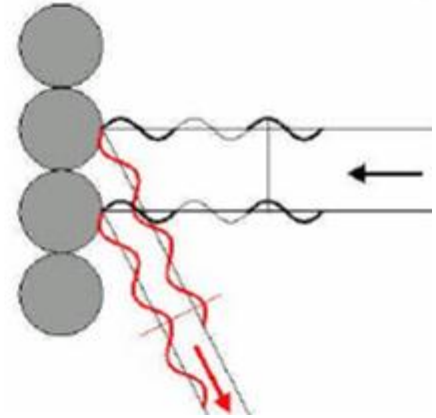


- Electron with the energies used in diffraction are very strongly absorbed in matter.
- Specimen which are to be used in transmission must therefore be in the form of thin film (few hundreds Angstrom).
- Hence, if thicker specimens is used, only the surface layers of the specimen contribute to the diffraction pattern.
- The small penetration of low-energy electron into a crystal is exploited in the technique commonly know as **LEED** (low-energy electron diffraction).

LEED

- This techniques is used to study the structure of surface using electron in the energy range 10-500 eV.
- The penetration of such electrons into a solid is limited to between 3 to 10 Å (one to three atomic layer).
- The scattering depend very largely on the 2-D structure of the solid’s surface.
- Electron scattered in reflection from the surface of the sample are detected either by a Faraday collector or a fluorescent screen.

LEED system



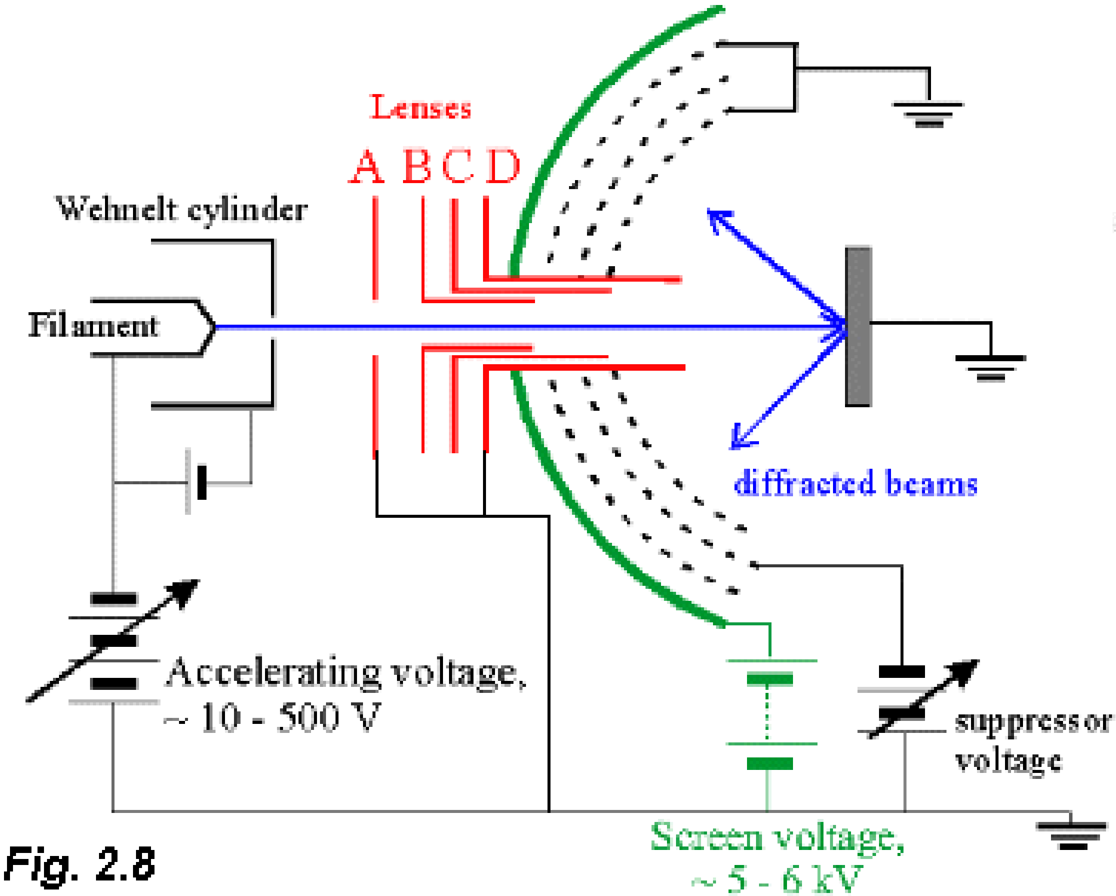
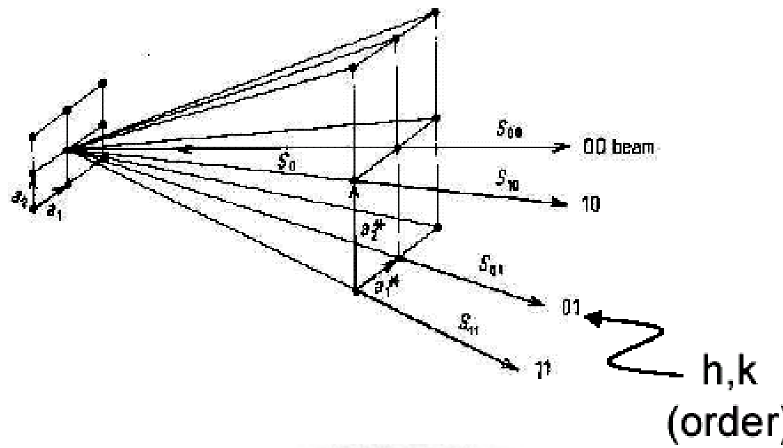


Fig. 2.8



5.2 Electron diffraction

- **Electron diffraction** is the phenomenon associated with interference that occur when electrons are scattered by atom to form diffraction patterns.

- The electrons are not only behavior as particles but as waves.

- Range of energy (E=eV) :
  - – Low energy (5 eV – 500 eV)
  - – Medium energy (500 eV – 5 keV)
  - – High energy (5 – 500 keV)

Duality of electron

- In1924,De Broglie claimed that *all* matter has a wave-like nature; he related wavelength, λ, and momentum, *p*:

$$\lambda = \frac{h}{p}$$

- He included the particle-wave property duality theory of matter, based on the work of Einstein (E=hν) and Planck.

- De Broglie's formula was confirmed three years later by guiding a beam of electrons through a crystalline grid and observing the predicted interference patterns

- According to quantum theory, any particles moving with momentum mv has a wavelength

$$\lambda = \frac{h}{mv}$$

where h – Planck's constant.

- Therefore, $v = \frac{p}{m}$

- Therefore, the kinetic energy is given by

$$E = \frac{1}{2}mv^2 = \frac{p^2}{2m} = eV$$

- The technique is only used on crystal samples that have a regularly spaced atomic lattice.

- Electron diffraction has been used for phase identification, structure and symmetry determination, lattice parameter measurement.

- There are three types of electron diffraction:
  - Low Energy Electron Diffraction (LEED)
  - Transmission High Energy Electron Diffraction (THEED)
  - Reflection High Energy Electron Diffraction (RHEED).

- Most electron diffraction is performed with high energy electrons whose wavelengths are orders of magnitude smaller than the interplanar spacing in most crystals.

- For example, for 100 keV electrons $\lambda < 3.7 \times 10^{-12}$ m. Typical lattice parameters for crystals are around 0.3 nm.

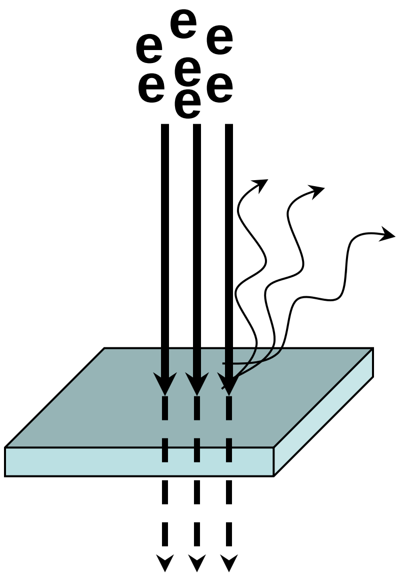
- Electrons are charged, light particles and their penetration into solids is very limited.

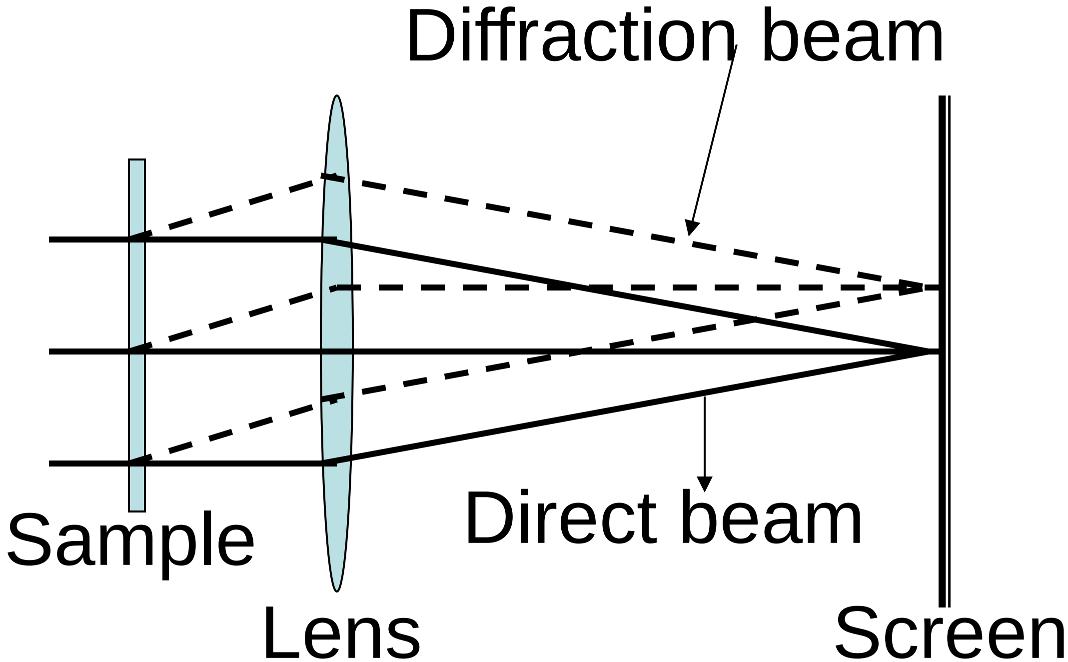
- They lose energy by ineleastic scattering or are scattered elastically (diffraction).

- Therefore, both LEED and RHEED are considered to be surface characterization techniques.

- THEED is limited to specimens less than 1mm thick.

- THEED is usually carried out in a transmission electron microscope (TEM).





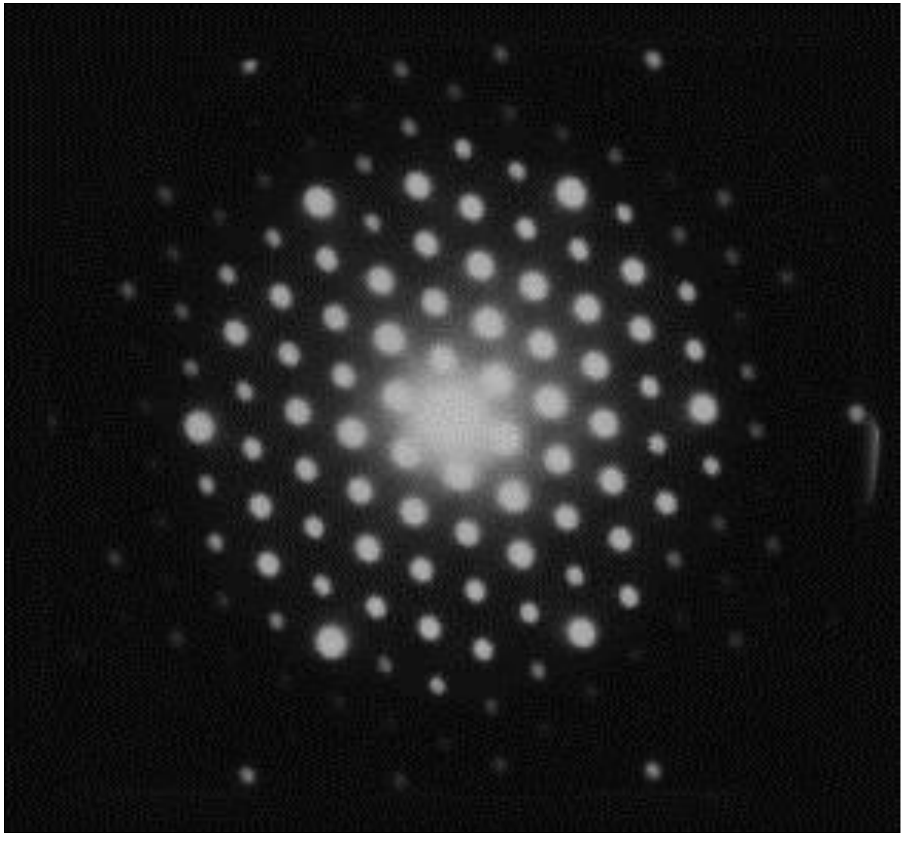
- Since λ is very small, Bragg angles are also small, so the Bragg Law can be simplified to:

$$\lambda = 2d\theta$$

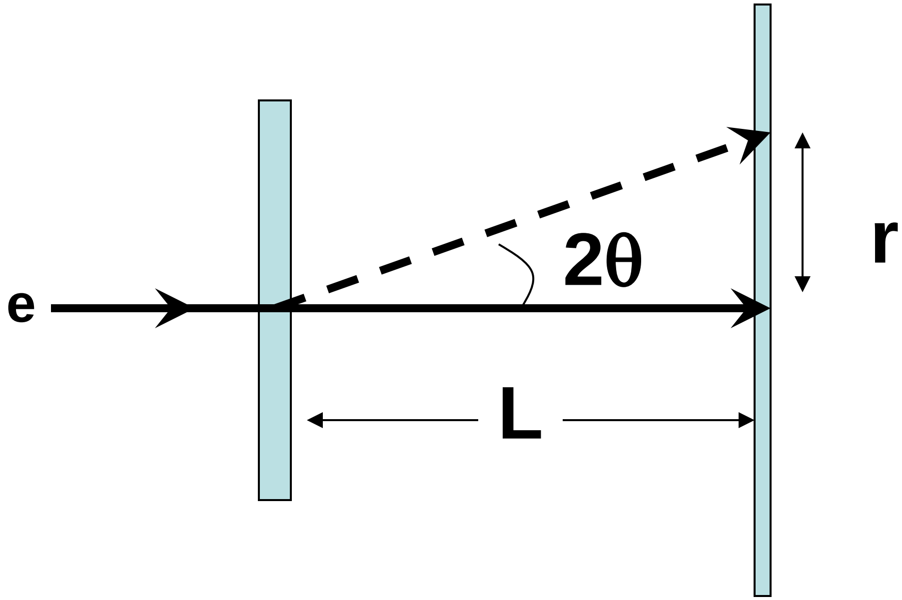
- The diameter of the Ewald sphere is very large compared to the size of the unit cell in the reciprocal lattice

- Lenses is used to focus the diffraction pattern and to change the camera length, which is equivalent to moving the film in an x-ray experiment.

- A typical electron diffraction pattern for a crystalline specimen is shown here.



Geometry of an electron diffraction experiment0



From the diagram :

$$\frac{r}{L} = 2\theta$$

Bragg Law for small angles approximates to:

$$\lambda = 2d\theta$$

Therefore, $\frac{r}{L} = \frac{\lambda}{d}$ or $r = \lambda L \times \frac{1}{d}$

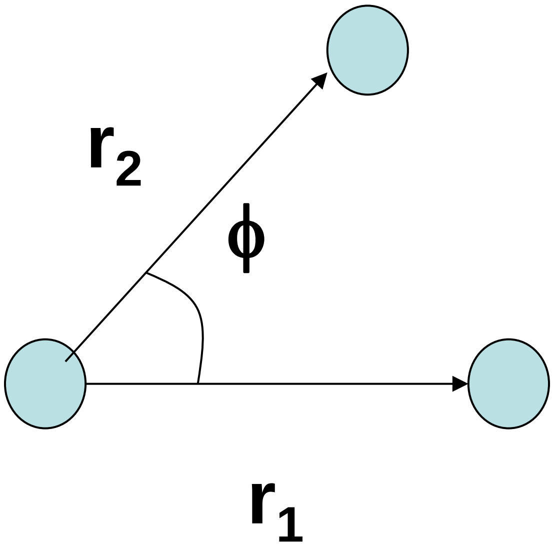
The distance, *r*, of a diffraction spot from the direct beam spot on the diffraction pattern, varies inversely with the spacing of the planes, *d*, that generate that spot.

5.3 Indexing Electron Diffraction pattern

- Choose one spot to be the origin

- Measure the spacing of 1st spot, r_1 and 2nd spot, r_2.

- Measure the angle between the spots, ϕ.



- Prepare a table giving the ratios of the spacings of permitted diffraction planes in the known structure

- Take the measured ratio r_1/r_2 and locate a value close to this in the table.

- Assign the more widely-spaced plane (usually with lower indices) to the shorter *r* value.

- Calculate the angle between pair of planes of the type you have indexed.

- If the experimental angle, ϕ , agrees with one of the possible values - accept the indexing. If not, revisit the table and select another possible pair of planes.

- Finish indexing the pattern by vector addition.

Interplanar spacing tables

- From early studies, interplanar spacing, *d,* for a cubic structure with lattice parameter, *a,* is given by:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

- Therefore, the ratio of interplanar spacings between two different planes, d_1 and d_2 is:

$$\frac{d_1}{d_2} = \frac{\sqrt{h_1^{\,2} + k_1^{\,2} + l_1^{\,2}}}{\sqrt{h_2^{\,2} + k_2^{\,2} + l_2^{\,2}}}$$

FCC

allowed reflections only from all even or all odd *h*,*k*,*l*

		d_1					
d_2	hkl	{111}	{200}	{220}	{311}	{222}	{400}
	{111}	1					2.31
	{200}	1.5	1				
	{220}	0.61	0.71	1			
	{311}	0.52			1		
	{222}					1	
	{400}						1

BCC

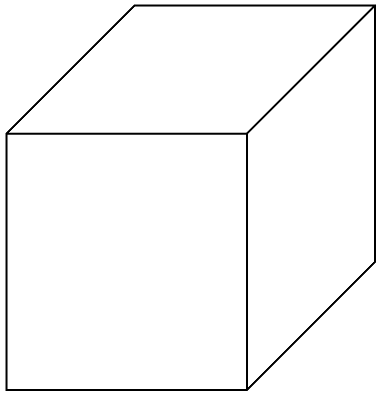
allowed reflections only from *h*+*k*+*l* = even

		d_1					
d_2	hkl	{110}	{200}	{211}	{220}	{310}	{222}
	{110}	1					
	{200}		1			1.58	
	{211}		0.82	1			
	{220}				1		
	{310}					1	
	{222}						1

Calculating the angle between two planes

- For cubic crystals, the angle, ϕ between two planes, $(h_1\ k_1\ l_1)$ and $(h_2\ k_2\ l_2)$ is given by:

$$Cos\phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{h_1^{\ 2} + k_1^{\ 2} + l_1^{\ 2}}\sqrt{h_2^{\ 2} + k_2^{\ 2} + l_2^{\ 2}}}$$

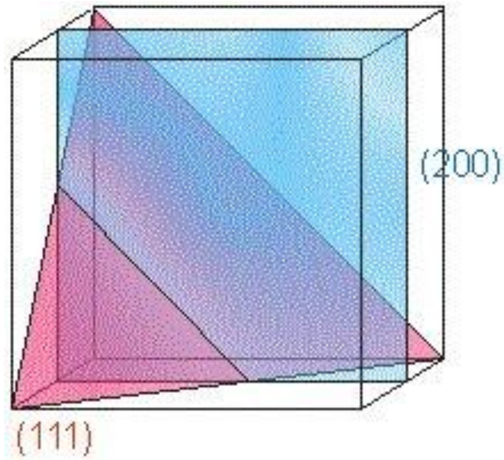


Example:

Calculate the angle between the (111) and (200) planes.

From equation above,

$$Cos\phi = \frac{(1\times 2)+(1\times 0)+(1\times 0)}{\sqrt{1+1+1}.\sqrt{4+0+0}}$$

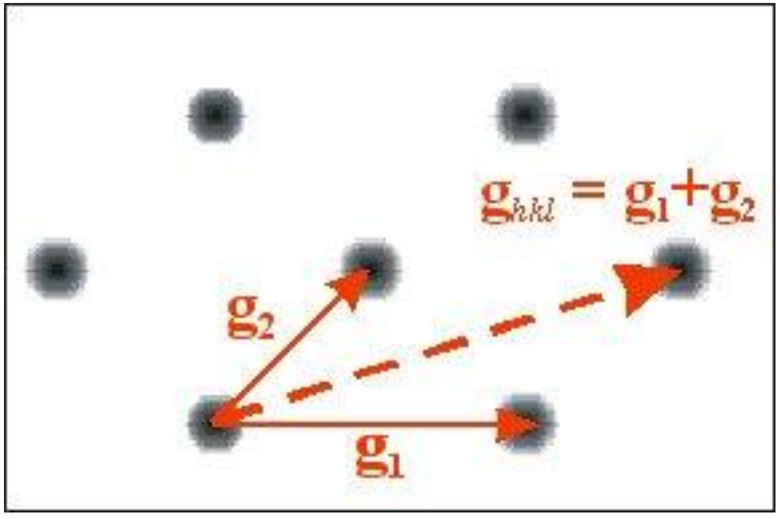


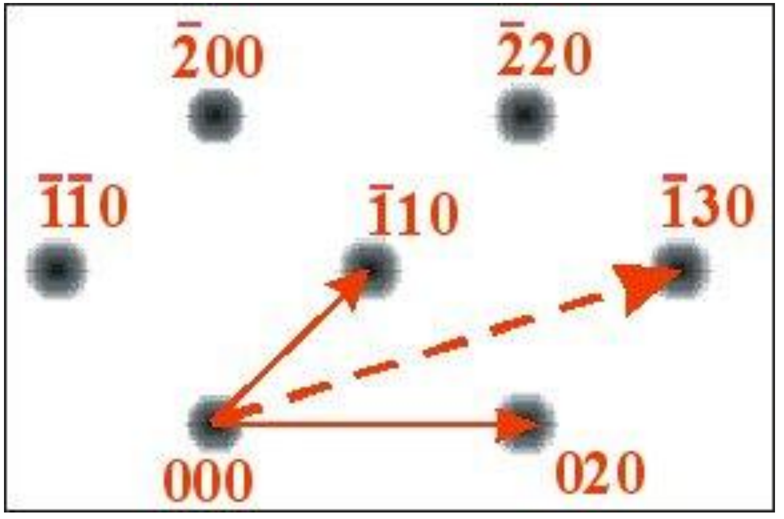
$$Cos\phi = \frac{1}{\sqrt{3}}$$

Therefore, ϕ = 54.75

Indexing Electron Diffraction Patterns

- If we know the index for two diffraction spots it is possible to index the rest of the spots by using vector addition.
- Every diffraction spot can be reached by a combination of these two vectors.





Why XRD is difficult to applied to surface ?

- X-ray photons have micron-scale penetration depths into solids which means that X-ray diffraction is not an inherently surface-sensitive technique.
- Low energy electrons, however, have penetration depths (mean free paths) of the order of a few lattice spacings of the solid and are therefore more suited to surface diffraction.

5.4 Type of Electron Diffraction

- Electron diffraction is frequently used to study the crystal structure of solids.
- Experiments are usually performed in a transmission electron microscope (TEM), or a scanning electron microscope (SEM) as electron backscatter diffraction.
- The periodic structure of a crystalline solid acts as a diffraction grating, scattering the electrons in a predictable manner.
- Sometime, electron diffraction is incorporate in other system to determine in-situ crystal structure.
- Two common type of electron diffraction:
  - Low energy electron diffraction
  - Reflection High Energy Electron Diffraction

Low energy electron diffraction : 1

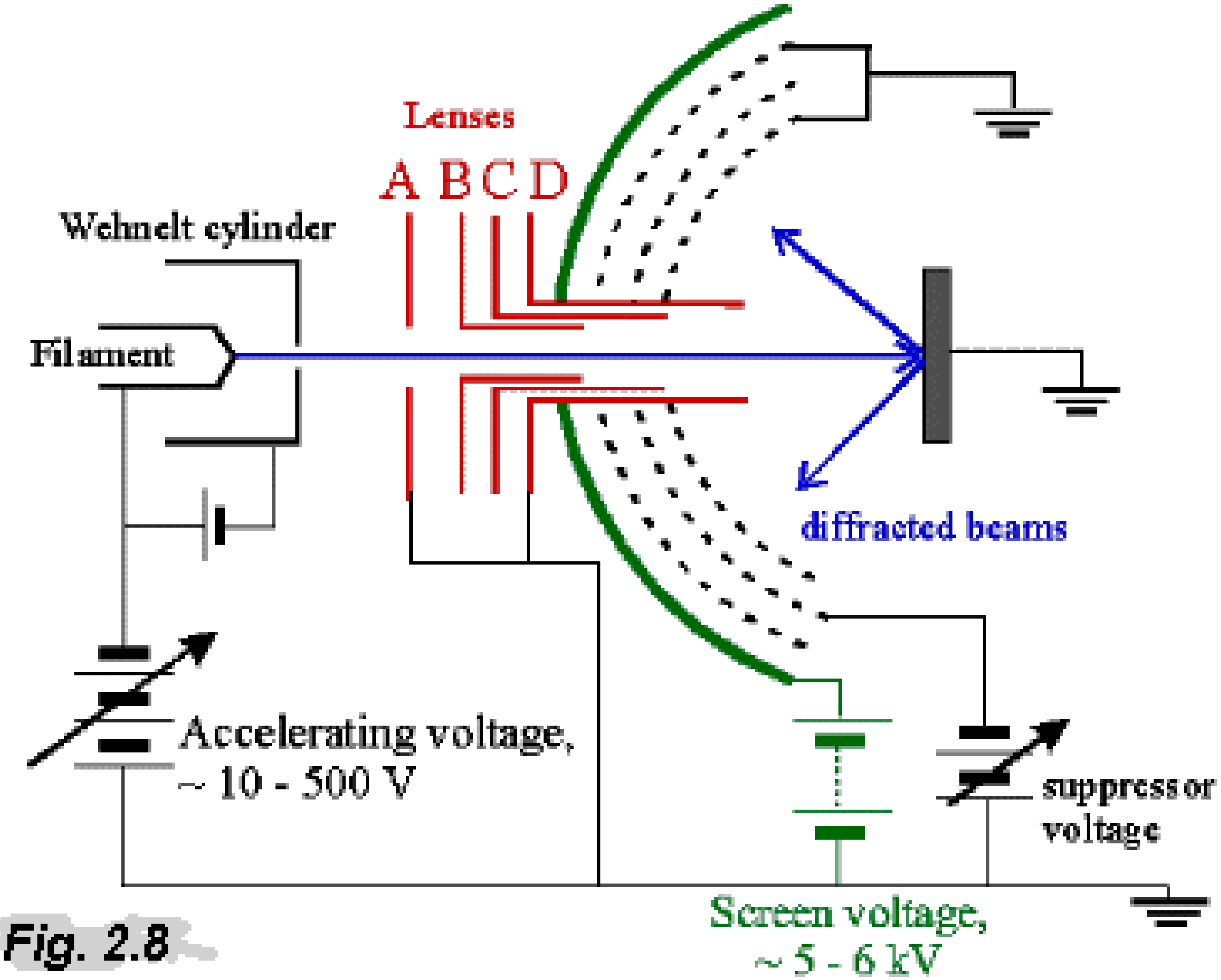


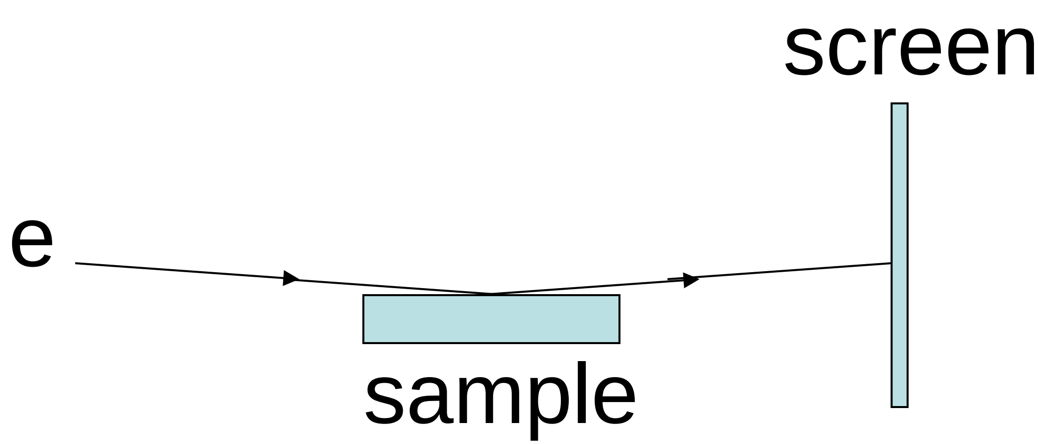
Fig. 2.8

- Electrons are thermionically emitted by the heated filament.
- The energy with which the electrons are incident on the sample is determined by the acceleration voltage (i.e. the voltage between the filament and apertures A and D).
- Apertures B and C are used to focus the electron beam.
- The incident electrons are backscattered from the sample surface and strike the fluorescent screen to create the diffraction pattern.
- The screen is at a high positive potential with respect to the sample surface so that the low energy, elastically scattered electrons are accelerated onto the screen.

- If the electrons is accelerated through a potential of 30 to 500 volts (i.e., their kinetic energy is around 30 to 500 eV), their wavelength is 2.2Å to 0.5 Å.
- This fits nicely into the range of distances between atoms in solids and can therefore strongly diffract from them.
- The recording and analysis of the diffraction pattern can tell us the arrangement of the atoms on the surface.

Reflection High Energy Electron Diffraction

- RHEED performed at glancing angles of reflection using electrons in the 10 - 100KeV range.



- The sample can be rotated about its normal axis so that the electron beam is incident along specific crystallographic directions on the surface

What advantages does RHEED offer over LEED ?

- The geometry of the experiment allows much better access to the sample during observation of the diffraction pattern.

- This is particularly important if it is desired to make observations of the surface structure during growth of a surface film by evaporation from sources located normal to the sample surface.

- Experiments have shown that it is possible to monitor the atomic layer-by-atomic layer growth of epitaxial films by monitoring oscillations in the intensity of the diffracted beams in the RHEED pattern.

- By using RHEED it is therefore possible to measure, and hence also to control, atomic layer growth rates in Molecular Beam Epitaxy (MBE) growth of electronic device structures.