## Indian Institute of Technology Madras Presents

## NPTEL NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

## Lecture - 11 <u>Materials Characterization</u> <u>Fundamentals of Scanning Electron Microscopy</u>

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Hello welcome back to this material characterization course! In the last few classes we have just reviewed all these optical microscopy variants and it is working principles and live demonstration so on and now we will move on to the next domain of electron microscopes. Like I did in optical microscopy first let us review some of the fundamentals of electron optics which will be useful to understand the electron optical system as well as electron lenses design and its operation methods.

So far we have just looked at the light optical rules and then we will see how this light optical rules will be applicable to the electron optical system in this few lectures of fundamentals of electron optics we will try to build a background to appreciate the electron lenses and their application to electron optical system and then we will also review the aberrations which are encountered in this electron lenses and then how to correct them in order to obtain a better resolution of the microscopes. So with this intention in mind let us begin our fundamentals of electron optics lecture with few remarks.

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In fact the paths of electrons in an electric or magnetic field are identical to the ray paths which is associated with light where glass lenses are the refractive medium. In fact this approach was first made by some of the German scientists who applied this analogy of the light optical system to the dynamics of electron in the electron optical system. So in the case of an electric or magnetic field however the refractive index is at any point depends on the corresponding field strengths we will see how this is valid for the actual electron optical system. (Refer Slide Time: 03:20)



We will first discuss electrostatic lenses because the electrostatic lenses were the first used in the electron microscope and then their design and behavior were studied then only this was adopted to electromagnetic lenses; so let us review some of the primary features are the theoretical concepts underlying this electrostatic lenses.

So an electron beam passing from a region of low potential V 1 to higher potential V 2 is on acceleration observed to under acceleration as defined by Snell's law  $\sin R / \sin I$  equal to square root of V 1 /V 2 so we know that the Snell's law which we have reviewed in the fundamentals of optical microscopic system. So similar thing is obeyed by this electron optical system as well so this equation clearly mentions that this clearly demonstrate that your electron beam also undergo a refraction according to Snell's law.

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Look at this schematic where we are demonstrating the refraction and reflection of electron beam on encountering the region of potential difference you see these two diagrams first we will describe this first one look at this the electron beam is encountering the potential difference by this electrostatic lenses where V 1 is less than V 2 and then it undergoes refraction.

So where I is the angle of incidence R is the angle of refraction on the other hand if you see that this is electron beam encountering the two electrostatic lenses where the potential is reversed where v1 is greater than v2 then your electron beam undergo a reflection like this and then you have the refraction also taking place in this manner; we will see under what condition this two are happening.

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The electron beam on passing through a region of potential difference with v1 is greater than v2 experiences a retardation making an angle of refraction greater than angle of incidence; so this is what we have just seen so where I is very large then these two conditions are valid so for the refraction  $\sin R / \sin I = (V \ 1/V \ 2)^{0.5}$  where I  $<\sin^{-1} (V \ 1/V \ 2)^{0.5}$  for the reflection where R' = I where I  $> \sin^{-1} (V \ 1/V \ 2)^{0.5}$ .

Where R is the angle of reflection from the plane of potential zone we will go back and then see so the plane of potential zone which we referring is somewhere here and then you see that I is equal to R a when the reflection is considered. So with this we simply see that the electron beam exactly follows the rules of a light optical system and we will see what are the additional points we need to consider.

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And this schematic clearly shows that they the cylindrical electrostatic lens action. What you see is you see this electron beam coming and then the diverged beam is going through this the electrostatic field and then it is getting converged; so the converging action of this electrostatic lenses very clearly demonstrated in this schematic. So an electrostatic lens for v1 is less than V 2 is thus observed to act in an identical fashion to glass lenses with respect to the focusing action on a divergent electron beam so this is what is clearly demonstrated in this schematic.



Now as I just mentioned before the electrostatic lenses were the one first developed for the electron microscope and you can see in this schematic that it is exactly analogous to a glass lens system so you see where the light is coming and falling on this glass and then it is converged in the right hand side and here you have this electrostatic lenses here again the converging action is demonstrated in fact the focal length the front and back focal length of these two lenses. I mean in this each systems are equal and hence we will see that that lens equation is exactly valid in this electron optical system as well.



What I am going to show in this schematic is : you see these are all some of the electrostatic lens design for the cathode lens microscope and the what you are seeing is a uni potential electrostatic lenses for a fixed focal length. In this schematic it is clearly shown this is for a fixed focal length I can play this schematic for you just to have a better capture of the concept you see that electrostatic lens and then the electron beam is forming entering into this electrostatic field and then and you see that f focal length is fixed in this situation.

And in the second case it is a variable focal length where you have the a combination of electrostatic lenses for a different field strength you can also vary this focal length F 1 and F 2 you can see that the first one coming through this F 1 point is lying are meeting at A 1 and B 1 in the image plane and then the beam passing through F 2 is falling on the image plane at the point A 2 and B 2. so you have the variable focal length electro optical system is demonstrated and what you see in the right hand side is a simple light table analog.

I just want to make sure that the electron optical system is exactly what we have in a light optical analog you should not get confused just because we are replacing this light I mean light optical system where we use a glass lens as the refractive medium instead of this refractive medium in an electron optical system you have electrostatic lens. So I hope this schematic gives you a nice

comparison between this light optical system as well as the electron optical system where the electrostatic lenses are used or the cathode lens designs are adopted.

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The electrostatic lenses we just discussed about where the electrostatic unimposing potential electron lenses the most useful for the incorporation into a general electron optical system since it is essentially analogous in function to a single converging glass lens in a light optical system this is what just we have seen what is uni potential lens in any potential lens the image.

And the object regions of the lens are at the same potential with the consequence that the refractive index is constant. So, as I just mentioned that the front and the back focal plane a focal length are I would say that the focal length in the front and back focal plane are same so the focal length F is related to the object image geometry.

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In the form 1/F=1/P+1/Q so the refractive power of the unipotential lenses expressed by approximately 1/F=3/16 ( $\int^{z_1}z_0$  times (Vc by Vo)<sup>2</sup> dz so which is a function of the field strength so I think with this few introduction to the electrostatic lenses we will now look at how the electromagnetic lenses are being developed into the modern electron microscopes since electrostatic lenses are analogous to the optical system the same electrostatic lenses also are I would say the electrostatic lens design is adapted to electromagnetic lens. Let us see how it goes.

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# Eucommentals of Electron Optics (Theory of electromagnetic lenses) (Theory of electromagnetic lenses) Electromagnetic lenses are analogous to the unipotential electrostatic lenses, which are fundamentally analogous to a glass converging lens in a light optical system • The action of magnetic field on electrons is that any deflection the electron experiences is proportional to its charge and mass. • The Magnetic field exerts a force on a moving electron in a direction normal both the field and the propagation direction of the electron.

The electromagnetic lenses are analogous to the unique potential electrostatic lenses which are fundamentally analogous to glass converging lens in a light optical system so what that we have to now understand is what this the additional magnetic field does to the electron path or beam of electrons so let us see the action of magnetic field on electrons is that any deflection the electron experiences is proportional to it is charge and mass

The magnetic field exerts a force on a moving electron in a direction normal to both the field and the propagation direction of the electron so what you have to understand here is the magnetic field is going to produce an additional force in a direction normal to both the propagation and field direction of the electron; so it is perpendicular to both.

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So this is demonstrated in this schematic you see this : is a typical cylindrical type electromagnetic Lenz action it is a cross section where you have all the circular slots where a soft iron coil is being wound like this; and this is the electron beam getting into this a core of the lens and then you see the field which is being generated and then you see all the electron beam is converging; so the magnetic field produces a force normal to this field direction as well as the propagation of the electron. So that means perpendicular to this direction so that produces a field like this and which will have a kind of a cylindrical shape with the radius R we will see how this is perceive.

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Thus a magnetic field acting in a direction parallel to an electron beam will not affect it while a field normal to the beam will cause it to describe a circle with the radius given by Ro = (1/B)  $(2mv_o / E)_{0.5}$ ; where Ro is in centimeters for V not the acceleration potential inwards and B is the magnetic field strength in Gauss; in effect the electron in a uniform magnetic field will describe a helical path; please make a note of this in a uniform magnetic field describe a helical path with a radial extent limited by Ro. So what you have to remember is this is R where you have the circular beam our field is represented around this region.

So now we will see how the other parameters are getting affected the refractive power of the electromagnetic lens is given by  $1/F = (0.022/ \text{ Vo}) \int_{Z_0}^{Z_1} H^2 dZ$ . Where Vo is the potential through which the electrons converging on the lengths have been accelerated and H is the magnetic field strength on the z axis in gauss .So the field strength is related to the physical design of the lens coil by  $4\pi \text{NI} / 10 = \int_{Z_0}^{Z_1} H^2 dZ$ ; Zo equal to - infinity to ZI put infinity H DZ from which we can observe that the lens power is proportional not only to the number of turns n of the conductor and the current flow I but also to the extent of the field region.

So now it is very clear from this expression you can understand this I go back to this you can understand the typical electromagnetic lens and the number of coils which is being used to produce this magnetic field in this kind of a slotting system is going to be also a function of u are the magnetic field strength. So you are from henceforth in an electron microscope you are going to use only these kind of lenses electromagnetic lenses instead of what we have seen already the optical and all.

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So now I will just play some of the schematic where we will demonstrate the electromagnetic system I want you to go through this carefully and then see what you observe then I will explain one by one you see that this is a object Oa okay; so I hope what all of you would have seen this schematic once I will replay this, you observe it again okay. What I'm going to describe from this slide is the primary difference between the glass lens optical system or electrostatics system to the electromagnetic system.

In in a light optical system you see that your image inversion takes place here also you can see that way the object is inverted and it is not just inverted even what inversion takes place at 180 plus or minus  $\phi$ 1 you have the additional rotation takes place here and if you have their double lenses then it is further rotated back to a B but then you see that in the additional rotation is added that is  $\phi$ 1+or- $\phi$ 2.

So this is the primary difference between the light optical system or electrostatic system with electromagnetic system you have image rotation takes place we will see the consequence and importance of this image rotation when we deal with electron I mean transmission electron microscopy which I will deal with later. So carefully if you see the next schematic the animation clearly showed that you see that the first lenses has same strength as the previous one so it has undergone a inversion plus rotation.

But the second lens there is a difference I hope you will be able to appreciate this you see that the number of lines have come down that indicates the field strength has come down; so you see the similar reaction takes place here that means this rotation also will come down. So if you look at the third schematic you see that inversion plus rotation takes place and I have the second lens the completely the field is absent and you see that there is no additional rotation that is the  $\phi 2$  is 0 the  $\phi 1$  which is generated by the first lens remains the in the image plane. You see that this is a object Oa okay, so I hope what all of you would have seen this schematic once I will replay this you observe it again.

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Okay so what I am going to describe from this slide is the primary difference between the glass lens optical system or electrostatics system to the electromagnetic system. In a light optical system you see that your image inversion takes place here also you can.

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See that way the object is inverted and it is not just inverted even what inversion takes place at 180 plus or minus you have the additional rotation takes place here and if you have their double lenses then it is further rotated back to AB but then you see that in the additional rotation is added that is plus 2 so this is the primary difference between the light optical system or electrostatic system with electromagnetic system you have image rotation takes place.

We will see the consequence and importance of this image rotation when we deal with electron I mean transmission electron microscopy which I will deal with later so carefully if you see the next schematic the animation clearly showed that you see that the first cleanses has same strength as the previous one so it has undergone a inversion plus rotation but the second lens there is difference.

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So this particular schematic and with the animation clearly demonstrates the primary difference between electron optical system our electromagnetic lens system with the light optical system this is the only difference you can fat all if you want to make between these two systems otherwise rest all this same.

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Now we will also look at another schematic where you see the clear animation shows that electron optical system where you have the electron source usually it's a filament and then you have the condenser lens and then you have a specimen and you have objective lens and then some of the additional intermediate lenses and then projector lenses and finally the image you see that a similar analog of optical system is also shown.

You can see that animation very nicely shown so that except the lengths electromagnetic lens action or you can see all this corresponding components of the electron sorry optical system corresponding to the light optical system you can see that condenser lens which here it is used to regulate the light and here also it is being used to regulate the electron beam and convert them onto specimen that is the primary action and here also the objective lens will focus the light to the image plane the same action is done here the objective lens and then these two additional apertures also helps.

We will look at the details when we look at when we deal with this especially the transmission electron microscope and for the for the introduction I just want you to have a feel of these two system in comparison so that you don't have to feel anything confusing they are all the same whatever we have just looked at in the light optical system as far as we the instrument details are concerned or the ray diagram is concerned.

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First we will look at the electron gun you see that this is a typical schematic of electron gun design you have the filament and then you have the cylinders called a Burnett cylinder the grid gap is I mean the filament itself a cathode and then you have the anode then you see that the field strength is a kind of a converging; this is done by a negative bias given to this between filament and this anode which will not only accelerate the beam and also concentrate the beam to this region. We will see the importance of this in due course I just want to introduce this in the beginning like this.

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So the filaments usually operated about 100 to 1000 volts less negative than the grid cap with the anode and the ground potential, so this is the bias which I talked about so filament is operated at one hundred two thousand volts less negative than the grid cap. This arrangement improves the stability of the emission stream and because of the bias aids in concentration of the electron beam.

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And if you look at the function of the condenser lenses it serves to regulate the intensity of the electron beam in an optical system also converts the beam onto the specimen object of particular interest. The effective focal length is determined by the expression of the form

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Fc =  $\zeta$  c stand for condenser and then Vo is a potential / Nc<sup>2</sup> and Ic<sup>2</sup> all C stands for the a condenser this is a focal length of the condenser lens where  $\zeta$  c the condenser lens form factor is the geometric parameter and Nc equal to number of terms of conductor in the condenser coil system now you will understand what I mean by the condenser coil you have seen that cross-section of the electron optical electromagnetic lenses so you will be able to relate it very quickly.

So Vo is the acceleration potential of electron beaming waltz Ic is a condenser current in amperes; so it is clearly understand by this expression this focal length of this electromagnetic lens is related to these many parameters.

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And then if you look at the function of objective lens in an electron optical system especially in a transmission mode performs the same function associated with the class objective lens in your light optical system focusing the electron beam to your final area of a solution this objective lens is very different from the other lenses primarily in terms of the more constricted field parameters necessity by a shorter focal length through the concentration of magnetic field strength on the axis of the system. So the objective lens has a slightly different role in order to bring the shorter focal length so obviously.

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The design will be slightly different you can see that it is slightly bigger even if you go back to the schematic diagram we have shown always the objective lenses shown much bigger than be the condenser and other intermediate lenses because of this special action of this objective lens. So we will see that the focal length is defined in an equation of the same form  $Fo = (\zeta o^* V_0 / Ni)^2$ .

So where  $\zeta$  objective is objective lens form factor n is number of turns in lens coil V<sub>0</sub> is acceleration accelerating potential I is objective lens current so you can see that nicely drawn the schematic you can see that there is an additional hardware which is used called pole piece. This is an this is used to focus all this electron beam in the column and this whole piece is completely magnetized during the operation and you see that the electron field our electromagnetic field current speed strength is focused using this two pole pieces. These pole pieces are used in all the lenses whether it is condenser as well as objective and other lenses.

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Now we will just see what are the types of electron guns it's just an introduction we will see the details of functions much more all the details we will see when we actually look at the system but I just want to introduce this types of electron guns so to provide a stable beam of electrons of adjustable energy you have turbine an emissions they are also called emitters.

Example tungsten and lanthanum hexboric lab 6 it is being also called a lab 6 or lanthanum hexode or it and these two are a thermionic emitters and then you have another type called field emission guns which has got three variants cold field emission tip thermal field emission tip hot key field emission tip.

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So what are the general characteristics of electron gun the important parameters for any electron gun are the amount of current it produces and the stability of the that current. The current emitted from the filament is called emitted current  $i_e$  the portion of electron current that leaves the gun through the hole in anode is called a beam  $i_b$  current. At each lens and the aperture along the column the beam current becomes smaller and it is several orders of magnitude smaller when it is measured at the specimen as the probe current  $i_p$ .

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How this gun performance is estimated. So electron emission current brightness lifetime source size energy spread and stability. You will appreciate all these parameters when we actually look at the operation of the electron microscope and the some of the application we take-up and then we'll explain the each parameter how it affects the resolution and the brightness and so on; another important parameter is brightness is the most important of all this because image quality at high magnification is almost entirely dependent on this parameter.

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## **Electron Gun Characteristics**

### Brightness

- Electron-optic brightness β involves not only the beam current, but also the cross-sectional area of the beam d and the angular spread α of the electrons at various points in the column
- · Brightness is defined as the beam current per unit area per solid angle



So we have a definition for this brightness electron optic brightness Beta involves not only the beam current but also the cross-sectional area of the beam D and the angular spread alpha of the electrons at various points in the column brightness is defined as the beam current per unit area per solid angle which is represented by this equation  $\beta$  =current/area solid angle which is nothing but  $i_p/i_{pd}^2$  by n times i $\alpha b^2$  which is can be written like 4  $i_p/\pi^2 d^2$ .

T-square and alpha v square so where the P stands for probe current we will see the importance of all these parameters as and when we relate the we relate to the microscopic operations well as the image quality and aberrations and so on so these are all very important parameters to remember.

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This is another schematic which is just part of our this is from the another textbook we have taken you can see the similar filament and done design and we have already seen the action of the gun and so on.

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So a high voltage is placed between the filament and anode modified by the potential on the Burnet which acts to focus the electrons into the crossover with the diameter  $d_0$  and the diverge angle  $\phi_0$ , so these two just I want to show  $d_0$  and the  $\phi$ .

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# Electron sources

A high voltage is placed between the filament and the anode, modified by a
potential on the Wehnelt which acts to focus the electrons into a crossover with
diameter d<sub>0</sub> and divergence angle q<sub>0</sub>



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So these two are controlled by this lens design in order to focus the electron beam and this is the image of the tungsten harping the tip of attraction hairpin filament and the distribution of electrons when the filament is under saturated and misaligned under saturated and aligned and saturated. So this is one of the thermionic source and these images are two different conditions and this is under saturated and misaligned and you have under saturated and aligned and you have completely saturated.

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So you will understand all this when we go to the operation of the microscope especially in a transmission mode this is just for an introduction. The another thermionic emission filamentous lanthanum hexaboric crystal and the electron distribution when the sources under saturated and aligned and do is saturated this is for you were just an introduction of the electron gun source.

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The next superior electron gun sources as I mentioned it is a field emission source so where you can see that the field emission tip and you have this subsequent down. I know design and the electron path from the field emission source showing you how a fine crossover is formed by two anodes acting as an electromagnetic lenses so you see that this is very fine and you can also see this a photograph how sharp the tip is so that is why you are able to produce a very fine crossover of the electron beam and the action of the anode oneness to provide the extraction of extraction voltage to pull the electrons out of the tip and no two axial rates the electrons to 100 kVare mode.

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Source	Brightness (A/cm² sr)	Lifetime (h)	Source size	Energy spread ∆E (eV)	Beam current stabilit (%/h)
				4.2	4
Tungsten hairpir	10°	40-100	30-100 µm	1-3	
LaB₅	10 <sup>5</sup>	200-1000	5-50 μm	1-2	1
Field emission					
Cold	10 <sup>8</sup>	>1000	<5nm	0.3	5
Thermal	101	>1000	<5nm	1	5
Schottky	10 <sup>8</sup>	>1000	15-30 nm	0.3-1.0	

So we will look at the parameters are much more details about this field emission gun as we go along and these are some of the gun characteristics you can look at it. Please remember the microscope performance is related to this electron gun source and we will also see how it is but for the introduction I just want you to have a some basic knowledge about this electron gun characteristics you see the source your function harp in lapse-filled emission cold thermal and short key and in terms of brightness as I mentioned it is one of the primary requirement of the electron gun.

And its lifetime source size energy spread and then bream beam current stability you can see that the field emission sorry the field emission guns have superiority over this thermionic emission emitters in terms of brightness as well as lifetime also in the probe size this is very important you see that thermionic sources you can go up to 3200 microns lab six can go up to five250 microns and here we are talking about less than five nanometers.

You will all appreciate the importance of the probe diameter when we discuss the operation as well as image forming capability of different microscopes we will discuss and this is how the field emission than a superior because it is able to form a very fine crossover less than five nanometers and then also you see that energy spread is also very small compared to the thermionic sources also you see the stability is also much higher. So with this I would like to conclude this lecture and in when we come to the next lecture we will discuss another important aspect of this electron lenses are electromagnetic glimpses namely the aberrations the aberrations and its effect on resolution or limiting resolution these aspects we will see in the next class. Thank you.

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