Nanotechnology Nanostructures and Nanomaterials: Characterization and Properties

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Lecture - 37
Transmission Electron Microscopy

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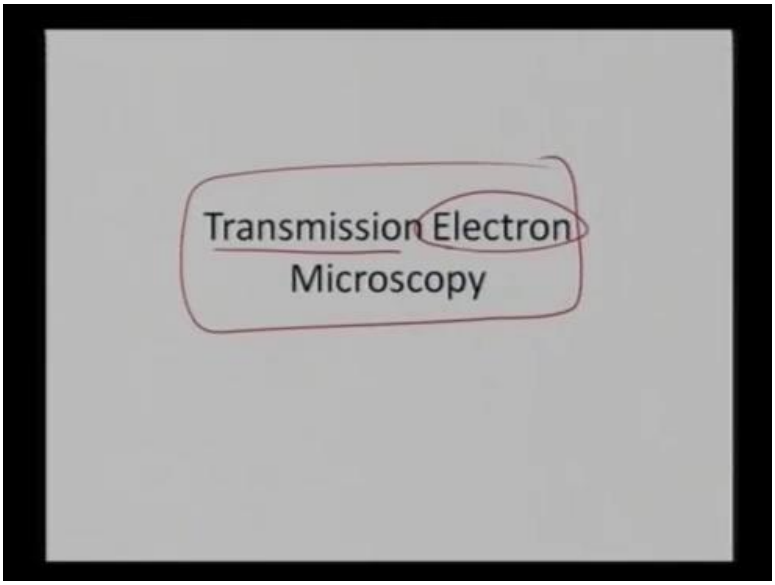

In this lecture will learn about transmission electron microscopy. As the term suggests that we are utilizing transmission mode of electrons to find out the features or see inside the material. So, in this part of the case we want the material to be transparent to electrons, so that electrons can when it gets transmitted through the material and provide some information about the material itself.

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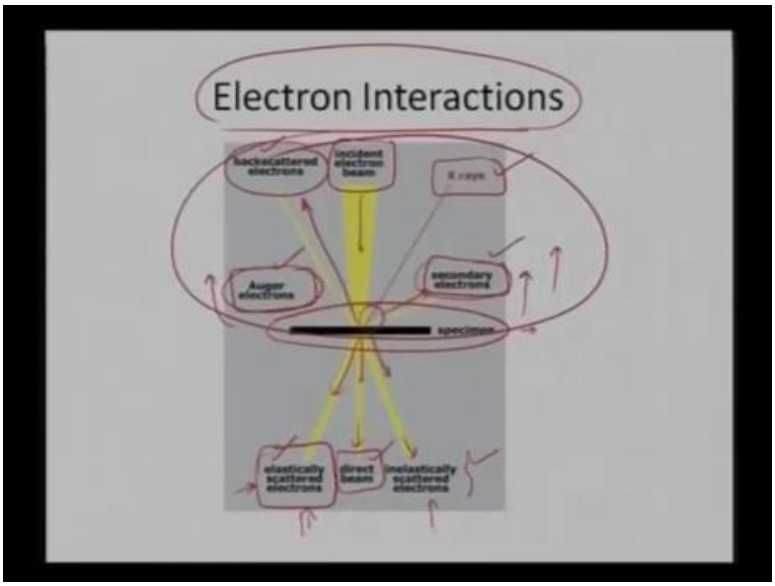

And the way the electrons interact with the material is, like if you have around here, we can get some electrons which have, which are inelastically scattered. And we can get them either as secondary electrons, we can also get them as auger electrons sometimes can also get elastically scattered back after the interaction with the material and those we can get them as a backscattered electrons, once we apply a some incident electron beam. So, once we are applying electron beam I can get some signal which are basically coming back, either a secondary electrons or as auger electrons or also a backscattered electrons.

They can also interact with the material to produce something called x rays, so we can see that these are all, these are all everything is happening when the electrons can be detected, all those signals coming detected back above the sample regime. They are not allowing the electrons to pass through the material. But if we have a material transparent enough, if we can attain a specimen which is thin enough and through which I, we can attain transparency for the electrons to pass through the material, what we can see is, we can get the incidental electron beam. We can let it pass through the energy should be high enough so that, it comes out as a direct beam or it can also get inelastically scattered.

We can also detect the inelastically scattered electrons, but we do not really need them why, because once we are supplying certain kind of energy to the, to the material. And if

the electron is undergoing some inelastic losses, so we do not know what is the input though, we know now what the output is we will not know what the input is, the input energy of the electrons. So, we will not know their wavelengths we will not know their overall functionality of how they are interacting with the material, but we are finally getting some signal. In some cases we can also tap those particular signals and we can, it might also help in analysis of the overall material.

The overall, the structure of the material or to track some sort of a crystallographic directions, but again we can get much more than formation from a material if we are, if we are, if the electrons are getting elastically scattered. So, if I let my incident electron beam to interact to the material and I let it elastically interact with the material, I basically detect what is, what the elastically scattered electrons are. So, I now I know what is my input energy, I know how they will interact with the material to give me a final elastic interaction after their interaction with the material elastically.

So, I know what is my output I know what is my input, I can get much more information from the material after this elastic interaction has occurred with the material so that, electron interactions they can be very complicated in nature. Starting from secondary electrons, backscattered electrons, auger electrons they can also start inducing some production of some x rays. And that is actually everything is going back to the above the sample, but if I, all the signals are not really getting transmitted through the material.

But if I let the electron beam be strong enough so that, I can get some signals which are letting which are basically been transmitted through the material, I can get them as either direct beam, inelastically scattered electron or also as inelastically scattered electrons. And mostly I utilize this as my overall feature of analysing a particular material, though I can also find some information from the inelastically scattered electrons as well. So, this basically comes under the transmission electron microscopy.

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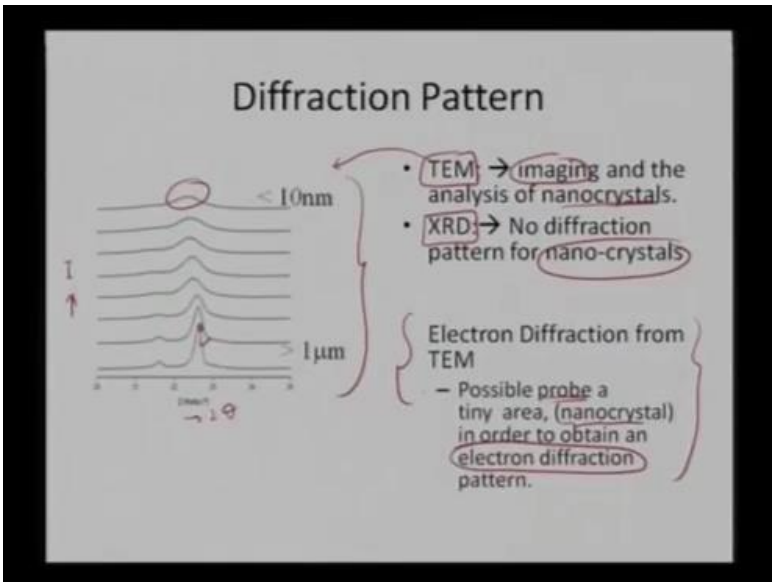

First of all what is the difference between my TEM and my XRD, TEM is the transmission electron microscopy, my XRD is the x ray diffraction. So generally, generally we see that for a micron size particle I always get some p. So, in this particular case I have my 2 theta in this particular case I have my intensity. So, for a Nano crystalline, for a, for a microcrystalline which has grain size greater than 1 micron, I will see some peak which is associated with a particular 2 theta value. So, I am getting certain intensity of the, from the crystal after, after it has diffracted. So, I can get some I am getting some information, but as soon as the material starts becoming much more Nano crystalline or some micron size then, what happens?

That my XRD is not able to detect whether the, whether I have some crystallinity into the material, it starts showing broadening of the peak, because as soon as my grains are becoming finer and finer, I start getting broadening of the peak. So, the ((Refer Time: 05:09)) crystallite size can also be calculated from the broadening of the peak itself. So, but here itself I have not, I have, I do not know whether the broadening is either, because I have an amorphous material or the crystals have become Nano, Nano crystalline in nature. So, that complexity can be easily analysed by TEM and in TEM or the transmission electron microscopy, we can image and analyse all these Nano crystals.

So, what I can get it from the electron diffraction is, I can get a, I can prove a very small area that can even be a Nano crystal and I can obtain an electron diffraction pattern.

Because now my beam is much more refine, I am letting the electrons interact with the materials inside of an x ray, the x ray, x ray will interact with the cloud of an electron or a or an atom as a whole. But my electrons is so sensitive, my electrons are so sensitive that once I am sending an electron to interact with the material it gets affected even by, it gets affected even by a single electron or the positive charge, which will be in the nucleus.

So, I can see that I can get a very strong signal if I let my electron interact with the material and now I can point it towards single Nano crystal and that is what will give me out its diffraction pattern. So, ((Refer Time: 06:22)) overall pattern many many grains contribute to the overall diffraction field, but in my electron diffraction I can let a small beam of electrons to interact with the particular Nano grain and I can get a diffraction pattern out of it. So, that is a advantage of my transmission electron microscopy over the x ray diffraction.

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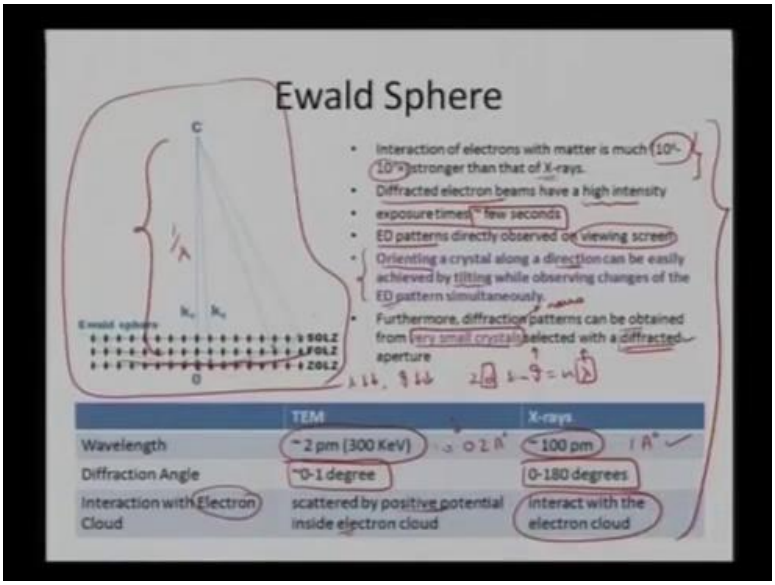

So, what is so beautiful about this transmission electron microscopy is, that electron interaction with the material is much more stronger. So, it is approximately 10 to the power 6 to 10 to the power 7 times stronger than those of x rays. So, eventually my diffracted electron will have a very high intensity, as one more part we can see out here is the Ewald which provides me the diffraction pattern the radius of the evolved sphere is given by 1 by lambda. So, coming back to it the wavelength in TEM is approximately to

3 Picometers whereas, in x rays is approximately 1 Armstrong or 100 picometers. So, in this case I have 0.02 Armstrong's and here I have around 1 Armstrong.

So, I can see that the wavelength part it is much much higher in x rays, but in TEM my wavelengths are very very smaller. So, eventually my Ewald sphere which is been forming, which is been formed so my 1 by lambda is very very, very very less. So, I get much bigger radius for the Ewald sphere, so that makes Ewald sphere much flatter. So, instead of touching, touching only few points now once my ((Refer Time: 07:57)) is becoming much more flatter, I can see that more number of points now start interacting with the Ewald sphere and the producer diffraction spot. So, coming back to it if we can orient to particular crystal for achieve a diffraction pattern, I can obtain all my diffractions within 0 to 1 degree.

Whereas, in for x ray diffraction I had to rotate my crystal from up from 0 to 180 degrees to get a particular diffraction, because we know that 2 d sin theta is equal to n lambda. So, as soon as I start reducing my lambda i can, for a particular interplanar spacing obviously, my theta is also getting increase with increase in the lambda. So, once I reduce my lambda to very large extent, my theta also will get diffracted, the diffracted spots will be very very near or they will be within a range of few 0 to 1 degree. So, that tells me that I can get all my diffraction patterns within a particular tilt of a particular, tilt of particular plane which is only 0 to 1 degree along the beam, that much parallel to the beam.

So, if I am, so my beam has to be approximately parallel, so there is only 0 to 1 degree. So, my beam can be much parallel to the particular oriented crystal and still it can produce a diffraction spot, it has to be within 0 to 1 degree and that also will give me a particular diffraction pattern. And one more thing about it, about it here is since the intensities are very very strong, because electron will interact very strongly with particular matter, it is 10 to the power 6 to 10 to the power 7 times stronger. So, what you have to do, my exposure time automatically reduces to only a few seconds. Whereas, for taking an XRD spectrum, I spend 40 minutes or 45 minutes or an hour or much more than that, to collect the spectrum of how my diffraction has really affected.

And also I limited to a certain range, may be its 0 to 180 degrees or 20 to 90 degree, so I have to limit my diffraction angle 2 theta value and then still it takes me couple of hours.

Whereas, diffraction through TEM through electron it is much more rapid I have to spend only 2 or 3 seconds to attain a spectrum and I will. So, since it is very very rapid, I can see my electron diffraction pattern, I can directly view it on a fluorescent screen or I can collect it on a particular detector. So, since my theta values are very very narrow 0 to 1 degree, I can orient my crystal along the beam direction and just by tilting it marginally I can also get an electron diffraction pattern.

So, that is a beauty of it I can get a particular image I can again orient my crystal only within the couple of degrees and I can still get a diffraction pattern and so I can get from diffraction pattern from a very small crystals, also can be obtained. Because my beam is very very sharp, very very intense. So, I can focus it to a very localized location or where something called Nano crystals, I can focus my beam into that those Nano crystals and I can still get a diffraction pattern by particularly allowing the beam to a particular diffraction, diffraction aperture. So, I can direct my beam to a diffraction aperture and I can still get a diffraction pattern from very very fine crystals.

As we can, as we know that the electron cloud is scattered by the positive potential which is there in the electron cloud. Whereas, x ay they interact with the whole of electron cloud. So, this is these are the overall differences once we go from x rays to TEM, that x rays will interact with the major part of the material whereas, electron beam is much more sharper much more intense. So, it will it generally provides you very drastic or very very high intensity information and instead of a focusing it to very large area, I can focus it to very fine area. And I can get diffraction pattern from even very fine grain or which can be a very fine crystal or a Nano crystal, I can get my diffraction pattern from very few seconds, I can view it on a directly on a screen as well, because it is so rapid.

And I can also achieve all the diffraction with a tilt of 0 to 1 degree, so those are the advantages of TEM and comparison to that of a x ray diffraction. And more over I, as we saw here that the Ewald sphere is, so is so flat that many of the points they coincide with the Ewald sphere the reciprocal point the reciprocal lattice point, they coincide with my Ewald sphere to give me a diffraction pattern.

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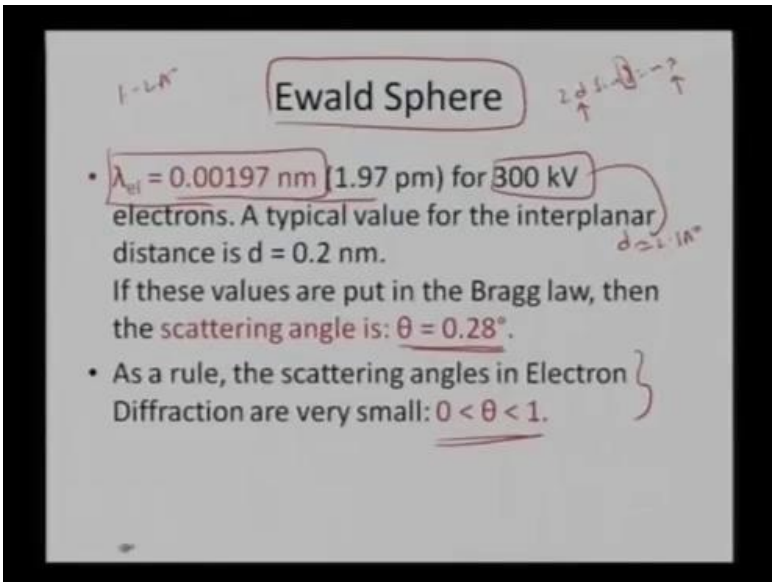

And as we see here, the evolved sphere it comes out to be approximately 1.97 picometers, once I have my energy of 300 k v electrons. So, particularly coming back to the d value which is approximately 2 to 3 Armstrong for a, for a particular crystals. If I put these values in my Bragg's equation 2 d sin theta is equal to n lambda. So, my lambda is known, now my theta is d is also known, because d is approximately 2 to 3 Armstrong's. And I can see that, the theta value comes out to be 0.28 degrees only. So, generally as a rule the scattering angles in the electrons diffractions are very very small, they vary between 0 and 1 degree.

So, I can see that my Ewalds will become very, very much flatter, because my lambda value is very, very, very, very less and compared to that of lambda value of a of an XRD beam. So, in my XRD I had used a wavelength of approximately 1 to 2 Armstrong's or, so which actually narrows down to less than, it becomes around 2 picometer for 300 k v electrons and that basically brings down the scattering angle to around 0.28 degrees. So, eventually my scattering angles are very, very small in electron transmission microscopy.

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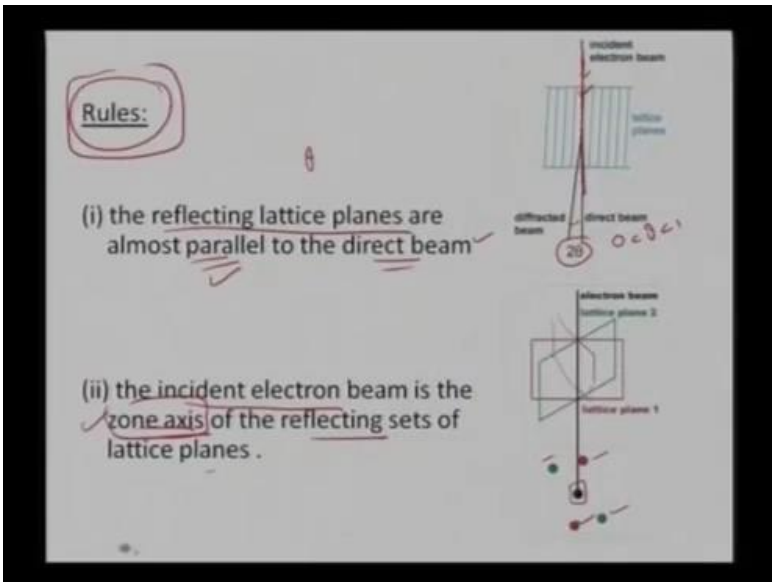

So, there are certain rules I have my incident electron beam, it interacts with the lattice planes and it gives me a diffracted beam at an angle of 2 theta and since my theta value is very very low, the reflecting planes are almost parallel to the direct beam. So, that is what we can see, that my beam which is been getting diffracted it is tilted only at very fine angles of 0 to 1 degree. So, they are almost parallel to the direct beam and secondly the incident electron beam, the beam which is basically falling on to the particular to interact with the particular lattice plane, becomes the zone axis of the reflecting set of lattice plane.

Because, it is approximately perpendicular to the plane which is basically it is interacting. So, the normal to it will be perpendicular to that and my electron beam is again perpendicular to the normal of the particular plane. So, my incident beam, incident electron beam becomes the zone axis, so this becomes the zone axis in terms of defining all the other diffraction planes. So that is the, those are certain rules, which are being followed out here, that my reflecting plane is parallel to the direct beam and secondly is my incident electron beam becomes the zone axis of the reflecting set of lattice planes. So, because there are so many lattice planes so those become, those, my incident beam becomes the zone axis for all such planes.

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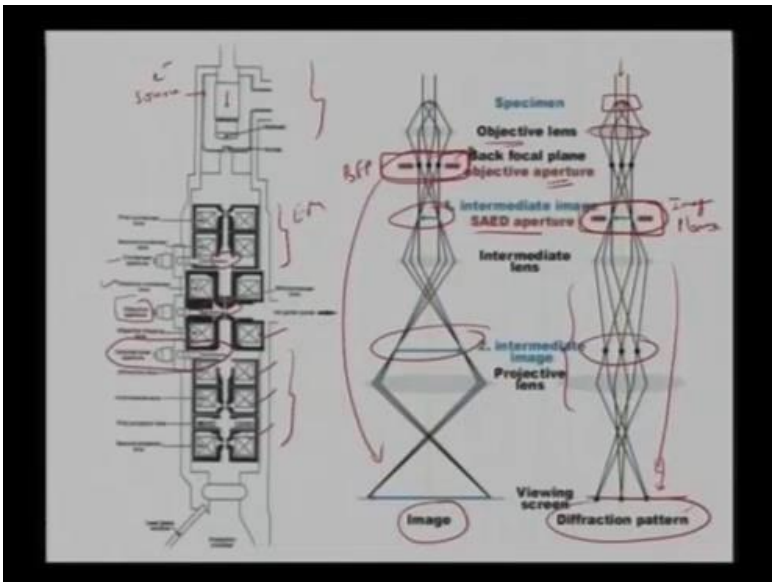

This is the overall construct of particular TEM, transmission electron microscope that initially I have a source for the electron, source an electron source. Here I generate my all the electrons then, I have set out magnetic electromagnetic lenses, we have certain condenser lenses generally 3 to 4 condenser lenses are utilized. Then, I have my condenser aperture along this particular part, so it is required for the alignment part and then, I have my objective aperture, objective, objective condenser lenses out here. And then, I have objective aperture out here and this is what decides the overall resolution of my TEM.

Then again I have some objective lenses, then again I have selected area aperture and then, again I have some diffraction lenses, I have intermediate lenses, I have projector lenses and I keep my sample actually in between, which I will come to in the next slide. But this is the old concept of a TEM that I have my electron source, a set of electron magnetic lenses, plus the condenser lenses and then my objective, objective lenses and then the projector lenses, to finally get an image. So, I can see out here if my, if I keep my specimen at this particular location I have the, I have, I have it already passed through the condenser lens.

So, I can see that my specimen is at particular location and then I have my objective lens which basically gathers information from, gathers the information from the particular specimen. So, electron beam comes interacts with the specimen and that information is

been collected by the objective lens, these are not really some tangible or something like optical or they are not like optical lenses. They are more of an electromagnetic lens, so there is nothing hard as such as we see in the optical microscopy, that we have really glass or lenses which are guiding the light. But in this particular case we are having the electromagnetic lenses, which are guiding the electrons.

So, once I collect the information, so I can see I have my objective lenses which are gathering the information from the specimen, after the electron beam is interacted with the specimen. Then I can see I have an intermediate which is called intermediate diffraction pattern, which where what I call back focal plane arise from my intermediate diffraction pattern or later on I can also form an intermediate image plane. So, this is an image plane and this is my back focal plane, so if I keep my aperture at the back focal plane finally what I get is an image. And if I, if I keep the aperture at the image plane finally what I get is the diffraction pattern.

So, I can see that I have forming my intermediate image at certain location, so I can have aperture, something is called objective aperture or something called back focal plane objective aperture. So, here I am forming my diffraction pattern and I keep and if I keep my aperture at this point of location, I can get bright field or dark field image. On at the second location where I have my SAED aperture, here I am forming my intermediate image and if I keep my aperture out here, what I get is a diffraction pattern. So, these two are more complementary kind of features, which I can really tap and I can, I can form an intermediate image as well.

So, I can form my intermediate image or intermediate diffraction pattern to finally get an image or a diffraction pattern. My viewing screen can be the fluorescent screen, on which the electrons can interact and they can come and fall and it can also be CCD camera. So, I can see that I have a particular specimen I let the electron beam pass through the condenser lenses, when it interacts with the particular material, it passes through, and then I have a set of a set of objective lens, which gather the information. And lets it through a certain apertures, it can be at the back focal plane or it can also be at the image plane and depending on where I choose my aperture, I can get, if I keep my aperture at the back focal plane, I get something which, some image.

If I keep my aperture at the image plane, I get a diffraction pattern and in between, I can have some intermediate lenses for basically magnifying a particular image or particular image. So, that is what I can see in this particular construct or the ray diagram.

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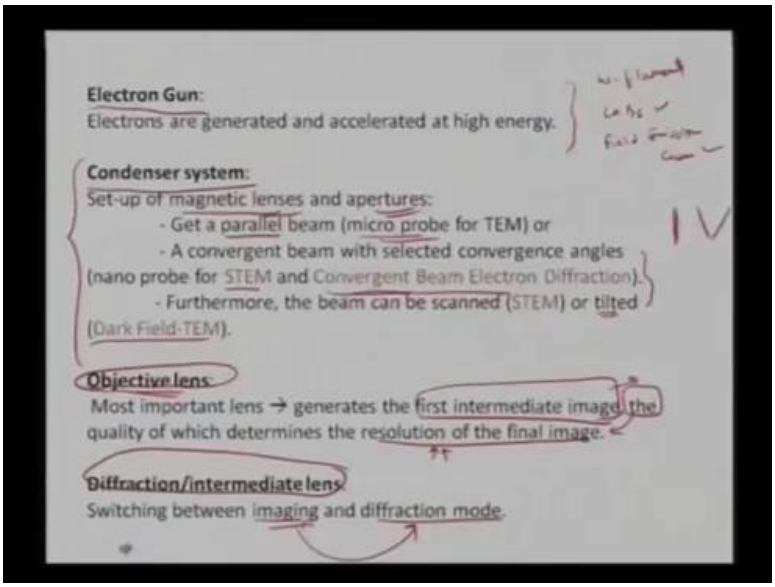

So, it is essentially to construct TEM, my first requirement is the electron gun, because I need to generate the electrons at some point so that, I can let it pass through the, through the specimen. So, I need some source of electron gun, so electrons are basically generated out here and they are after that they have to be accelerated so that, at very high energy so that, they can come and interact with the material. So, I have some sources of electrons those that can be either tungsten filament or LAB6 filament or even the field emission gun. So, I can get something called tungsten filament, it can also be LAB6 filament or it can also be field emission gun and depending on that actually tungsten filament is the low cost, but lower emission source.

LAB6 can improve the intensity of the electron similarly, field emission gun can also enhance the intensity of electrons to very drastic, very high extent and the beam size also reduces to very fine. So, there certain ways we can neutralize all those sources of electrons to generate the electrons so that they can come and react with the particular specimen. So, once the electrons have been generated I need to get a parallel beam of that so that, I can accelerate them. So, they have to be accelerated by note and after that

they are basically parallel, they are made parallel so that, they, we can get a parallel beam or they have to be made to fall on a, on a very fine beam.

So, there is some set of magnetic lenses and certain aperture which will allow me to basically condense the beam, make it parallel and then basically I can, I can focus it further for imaging part. So, I can get a parallel beam so I can make it like a more like a microprobe. So, I can get a microprobe beam of electrons, again I can also allow it to get convergent, so I consider instead of getting a parallel beam, I can get a converge get it, convergent beam for certain applications. So, for probing I need it like for scanning tunnelling electrons microscopy, I want a Nano probe. Whereas, for getting a lattice fringe imaging or getting more diffractions patterns, I can more diffraction interaction of more ((Refer Time: 21:42)) zones at particular location I can also utilize my convergent beam electron diffraction.

So, there are certain ways I can utilize either to achieve a parallel beam or a convergent beam. So, for that I need a condenser lenses which can really divert my electron beam, so if I can direct my electron beam, I can control the electron beam, I can get the information what I am looking for. Additionally, I can also tilt my electron beam to get a dark field imaging, so dark field transmission electron microscope imaging can also be attained, once I have control over my condenser lenses so that, I can guide my electron beam. So, that is the importance of the condenser lenses systems, electromagnetic lens systems and finally, the objective lens is decides the overall resolution of the final image.

And objective lens is one of the most important lenses, because it is generating the first intermediate image and the quality of that will be essential to get the overall resolution. So, once I am able to control the beam that is good enough, but objective lens is a one which will collect then for mission for the specimen. And since it is creating the first intermediate image, this is highly, this is highly, very critical factor in deciding the resolution of the final image. And in between I can have diffraction or intermediate lenses, either to get a diffraction mode or an imaging mode, because they are being formed at a different locations. So, I need to have two apertures or lenses which will guide me in terms of either switching or imaging to diffraction mode or being able to select a particular aperture.

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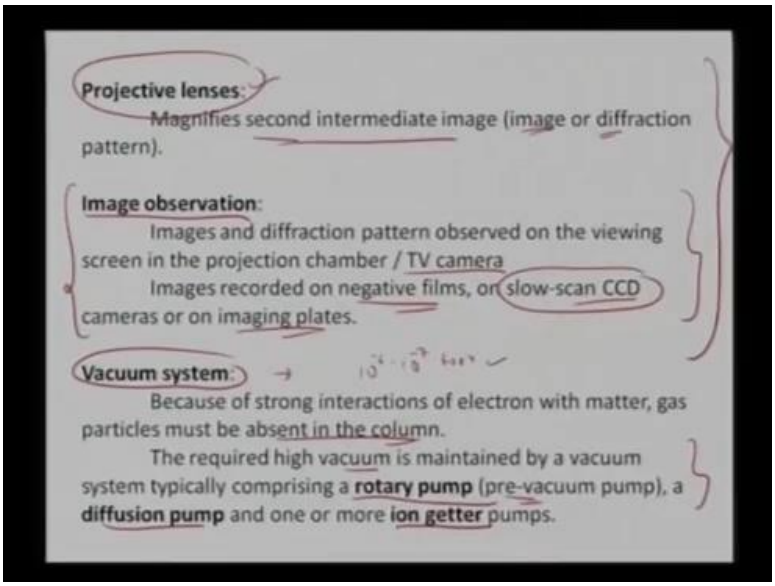

And there can be as well as some projective lenses, they will magnify the second intermediate image, they can be either image or it can also be the diffraction pattern and. So, projective lenses they help in the magnification of the second intermediate image, once I have found my image and I have basically magnified it, I also need to see image. Because, I cannot see the electrons, so I need to see electrons how they have, how they have interacted with the material. So, either I can view them on a fluorescent screen or I can project them on a TV camera or I can also record it on either on negative film or I can also record it on a slow scan CCD camera.

So, these are required for the image observations, because we cannot really see the electrons. So, we can we have to let it interact with the fluorescent screen or see it on a TV camera or I can also get it on a negative film or I can also capture it on a slow scan CCD camera. So, it can also be on an imaging plate. So, these are certain ways through which I can observe my particular image and all these things are, all the setup of TEM involves interaction of electrons with the matter the. So, the travelling of electron is highly necessary and that can happen, because I am also looking for achieving information which is elastically, which is elastically scattered electron.

So, I need to avoid any interaction of electron with the atmosphere, so for that I definitely need something called as vacuum system. Because I want to, I want the electrons to pass through the beam, through the particular, to the particular channel

without interacting with anything else any of the atmosphere. So, we interact with the atmosphere it is basically losing its energy, so that become inelastic interaction. So, to allow, to disallow any interaction of any matter or with the gas. So they, the gas particle should be absent in the column, so for that I definitely need a vacuum system and here I require very high vacuum which is 10 to the power 6 to 10 to the power 7 minus 6 to the power minus 7 tor.

So that part, that much, that much vacuum I require, require 10 to the power 6 to 10 to the power 7 minus 7 tor, I require for the creating of vacuum. So, there is this is very high vacuum which is been utilized out here. So, for this I am require, I require very high vacuum which is 10 to the power minus 6 to 10 to the power minus tor so that, electrons can continue without interaction with the nearby gases. So, I can get an information which is truly from the interaction with the specimen and I can achieve my vacuum by utilizing a pre-rotary pump, which is some sort of a pre-vacuum pump and later on I can go for a diffusion pump or a ion getter pump to get such a high vacuum.

So, that is what is highly required for a TEM imaging. So, here we see that we require a projective lenses for magnifying the second intermediate image and then for visualizing the image, I need to have some sort of a TV camera or a CCD camera or I can also record it on a imaging plate or I can also have it locate on a negative film. And since everything is happening with the electron, the electrons need not get interacted with the nearby gases. So, I need to allow a vacuum to be there, so that, so I get the information only which is truly from the material. So, vacuum to the order of 10 to the power minus 6 to 10 to the power minus 7 is required and which I can attain either by utilizing of pre-vacuum rotary pump or backing it up with diffusion from border or the ion getter pump, which becomes the main source for creating the vacuum.

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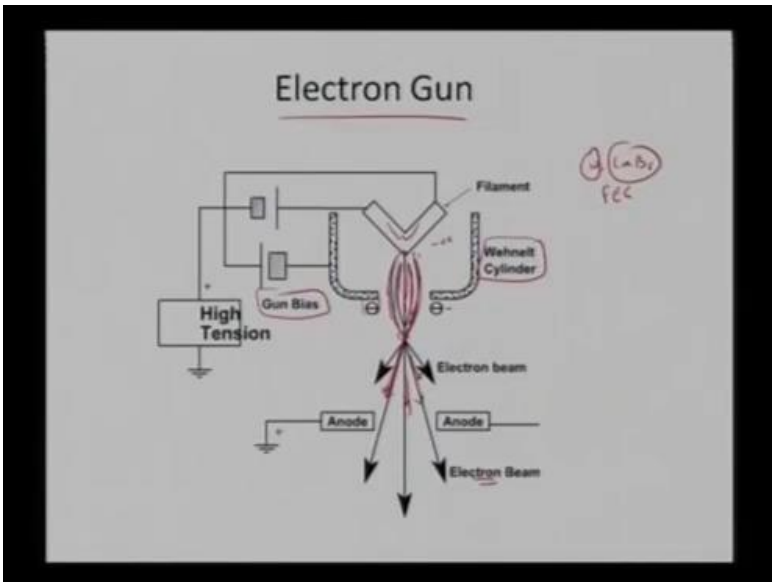

So, coming back to the picturesque of the electron gun I have my filament this can be as a tungsten filament or the LAB6 filament. So I this is, this becomes my source of electrons, I apply certain bias to it, I apply certain bias to it and I generate electrons. Once electrons are been generated, my wehneit cylinder it is, it has a negative potential. So, once the electrons are been generated it is now it will it is not basically focusing the electrons to some particular spot. So, that is what is been given done by the negative potential out here, I am generating my electrons, electrons are negatively charged particles and they are they now get focus or they get repelled by the negative bias and from here, they are not accelerated.

Because as the as I know, I apply certain potential, positive potential. So, now I help the electrons to accelerate to finally get an electron beam. So, in this particular case I can utilize tungsten or LAB6 filament or even field emission gun as my electron source, I apply certain negative bias to basically construct my electron beam. Later on I can create some positive potential so that, electrons can get attracted and they can get accelerated so finally, I can get my electron beam.

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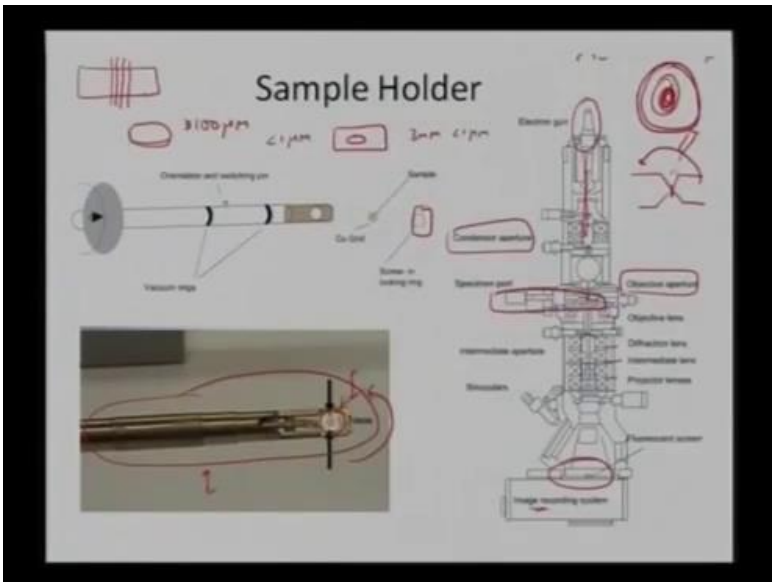

And the sample preparation it, it is very, very highly critical, because I need to make my sample truly transparent to electrons and this is how overall construct of TEM looks like. I have my electron gun to supply me the electrons then, I have setup condenser, apertures and condenser lenses, which basically focus the beam or control the beam. I can get either parallel or a convergent beam then here is my specimen port, where I keep my specimen then, I have certain objective aperture which collect the light from the, from the specimen. I have objective lenses again, I have some diffraction lenses or intermediate lenses and again some projector lenses to finally be able to observe it on a fluorescent screen or on image recording system.

So, that is the overall construct of my transmission electron microscope, but again here the very much requirement is of the sample to which transparent to the electrons. So, generally we have some sort of holders which can either provide me a tilt. In case when I require it can be up down tilt, it can be side tilt or it can also be a rotation which can, which, which I can get from the holder itself. So, this is what it is out here that I keep my sample on a particular copper grid and it has some locking ring, on which I keep my sample which is approximately 3 millimeter in diameter. So, I can keep my, I can make a very thin film which is approximately 3 millimeter in diameter, but which is a, which the centre part of that is transparent to electrons.

I can also have some powders, which are very fine enough and those are, those are basically transparent to electrons, but to prepare a sample it is very very critical to attain, to attain transparent transparency to electrons. What I can do, since my samples will be mostly bulk in nature, so even to select a particular piece of sample from a bulk is very, very, very challenging. Because first of all my sample has to be representative of the overall bulk structure, what I have intent to look at. In say, for an example I am excluding a particular rod, so the structure at the surface will undergo much more shear. So, the grain requirement might occur much more at the surface whereas, the grains may remain unaltered at the core.

So, if I am taking my sample only from the surface and I say it to be representative of what is happening in the core of that particular rod, I am totally wrong. So, I need to carefully select my sample as such and then, be able to relate it to the particular surface or particular area where I have taken my sample from. So, I might require sample either from the surface as well as from the core to be able to say what is happening at the surface and what is happening inside the core of that particular rod. So, once I want my sample to be representative of the particular bulk material, so I will take so many samples like from the surface as well as the core, because what I will seeing TEM is only a very finer, very, very small regime of the overall bulk.

So, I need to very carefully select the region or the sample which is truly, truly representative of my bulk material. So, in order to make if my, in order to make my material, very very fine or transparent to electrons, initially my samples may not be transparent to electrons. So, what I will do, I will first of all I will section my material into very fine slices which is, which might be approximately a millimeter or less than that or may be couple of microns, as fine as I can cut them through a saw. Then, I start thinning my sample from say less than, from more than which is greater than may be approximately 100 micrometer in thickness, I start thinning it down to less than 1 micrometer.

So, once I have a particular disc a particular sample which is less than 1 micrometer, I will punch out a small regime which is approximately 3 millimeter in diameter and its thickness will be less than 1 micrometer. So, I will attain a disc which is approximately 1 micron and then less than 1 micro, 1 micrometer thin. Once I have this particular disc, I start thinning it mechanically, so I start thinning it down mechanically and I can, I can

keep mechanically thin, thinning it till it has reached much lesser than approximately 200 nanometres.

I even as fine as I can go, so what all I have is thin disc which is approximately about 100 to 200 nanometres in thickness. If the sample is conducting, I can apply some corrosive or some area which can start heating away my, this particular material. So, what I can get is, I can start throwing some media it this is a large view, I can start throwing in some media from both the sides until, it start heating over the material and it is a very fine hole in the centre of the disc. So, what I eventually get is, what I eventually get is more like this, I get a material which has a very fine hole in the centre. So this hole, this particular point is nothing but a hole and just the area, nearby this hole is not transparent to electrons.

So, ideally if I see I actually more like this it will be transparent to electrons. So, this is what I will really targeting a to get a material which is transparent to electrons or I can also utilize something called ion beam milling, this is for conducting samples I can use something called jet, twin jet polishing. I am sending two jets on each, one on each side to start heating away the material and make it transparent to the electrons at this, this centre. Once I create the hole, the centre the part near the hole is transparent to the electrons. So seeing it from the top view I can also, this particular thing will look more like this, I have a disc which is a hole in the centre.

So, that particular hole just a near nearby the particular hole is now transparent to electrons. So that, I can create by twin jet polishing or alternately I can also supply some ion beam, I will take an source of organ, I can start throwing my ions on the sample and I start rotating my sample. So, I can create the similar way I can create a hole in the centre and on the region near the particular hole is again transparent to the electrons. So, I can look around in this particular regime to learn more about the sample itself. So, there are certain and certain ways, so my overall disc diameter remains around 3 millimeter, but the central part is now, it has a certain hole and the area nearby the hole is now transparent to the electrons.

And I utilize that particular area to be observed or to be analysed under transmission electron microscope. So, I can see how critical particular sample preparation is rather to make the sample big enough, so that I can handle it at the same time some part of it or

the centre part of it has to be transparent to electrons. So, I can see or visualize or observe or analyse the region in that particular regime. So, that is what is the overall thing about the sample preparation in the transmission electron microscope and once my sample is ready I insert it in the particular tem holder or the transmission electron microscope holder, so I can observe it further.

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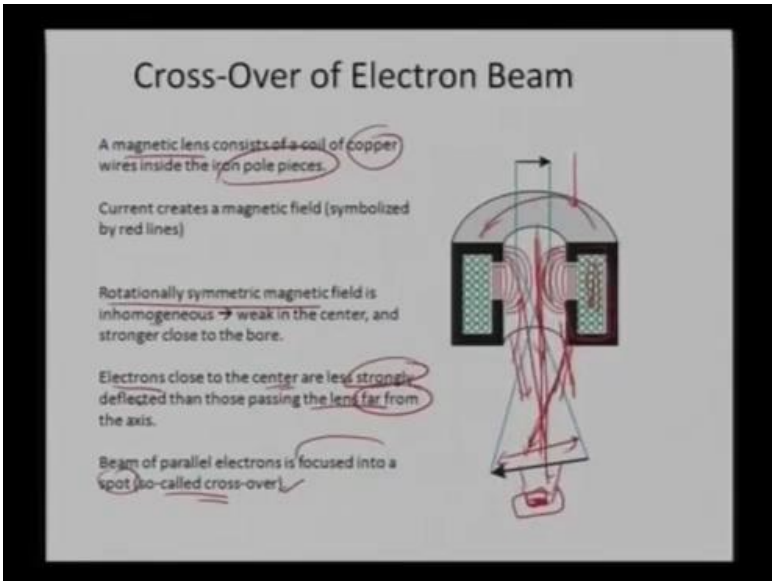

And how the overall magnetic lens looks like is, I have a magnetic lens which consists of copper wires and with some iron pole pieces. So, what I am seeing is copper wires which are out these, out these and these are nothing but the pole pieces of iron and once I supply some electric current to it, it will start creating a magnetic field. So I will get, I will get some magnetic field which are represented by these red lines and. So, what I can do? I can get a rotationally symmetric magnetic field, but this is again inhomogeneous, because I will get a weak field in the centre and very strong field in the, on the sides near the pole pieces.

So, what I can see is if my electron is passing through it, electron will get, electron will basically get strongly deflected once it, once it undergoes a very high field. So, once an electron is traversing along this and near the pole piece it will get diffracted very to very, very sharply. Whereas, an electron getting passing in the centre, will be deflected to a large to a lesser extent. So, I can see that they were undergoing a kind of a cross over, so they may not get focus at this same point so that, that actually bridges the results that the

electrons which are close to centre, the less strongly deflected. Then, those passing the lens from the far from the axis, so far from the axis they get diffracted very quickly.

Whereas, in the centre they keep going too much farther extent and they get deflected to a small extent. So, instead of getting a parallel beam, so we can try to focus all this, all this into a spot and this spot now becomes the, it is called, so called crossover. Because, I have a particular regime and instead of spot it is more like a regime on which my electrons are basically being targeted or being focused at. So, I get instead of getting fine very fine spot, I get a kind of a spot or a regime, which is called a crossover. And that happens, because I have my electromagnetic lenses and they create a stronger field near the pole piece and the weak field is much weaker at the centre part.

So, electrons which are travelling at the centre, they get diffracted to the smaller extent as compared to the near the pole pieces. So, I can get some instead of getting very fine or single spot, I get some sort of a regime and that is called a crossover of the electron beam.

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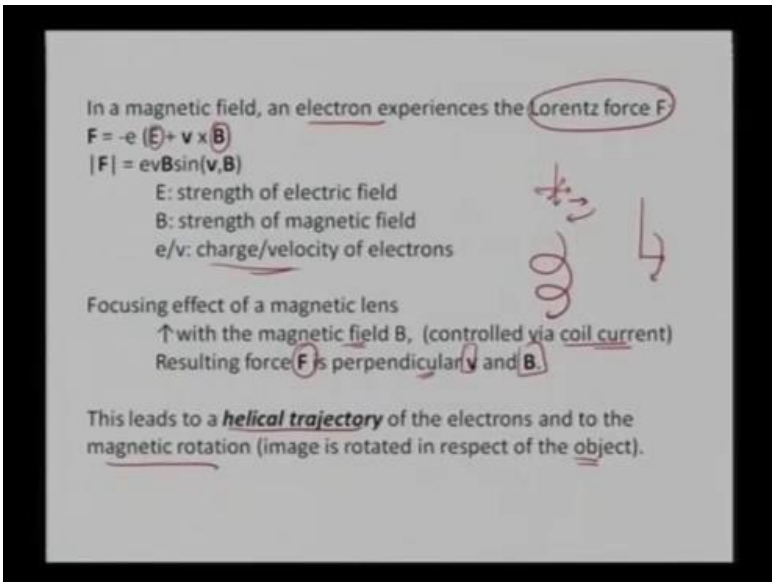

And since it is the magnetic field which is being applied, so my electrons, so the electrons will experience the Lorentz force. And since Lorentz force is the component of the electric field, as well as the magnetic field and it also depends on the charge by velocity ratio of the electrons. So, basically this particular part is more like this, that I am applying, I am letting the electrons get accelerate, I am applying a electric field, I am

applying a magnetic field. So, ideally with the magnetic field which is being controlled by coil current, it results in some sort of force, which is perpendicular both to velocity part as well as the magnetic part.

So, what happens that basically, that basically is trying to pull the electron in the same time it is to push the electron to certain direction. So, that creates a helical trajectory, because my electron is travelling like this and I, at the same time I want to pull it or I am also trying to push it, depending on the kind of field apply. So my, so my magnetic lenses they are, they are resulting some force which are perpendicular to v. So, I have some forces which are perpendicular to v as well as my b. So, I can get a more like a helical trajectory, so electrons will traverse more like an ((Refer Time: 39:13)) once they are traversing in the magnetic field.

So, I can see that the magnetic rotation is caused with respect to the object, so that basically is very essential, because depending on the magnetic field I am applying, it will tend to rotate my image. Because, electrons the way they are flowing if they are very stronger field they might to, they might get deflected to a larger extent. If I am applying a very smaller electric magnetic field, they might get deflected to a lesser extent and that is nothing but my magnification. Because I am applying field very strongly, I am allowing to form a very bigger image so my image can also get rotated once I am applying a certain field.

So, this is where on the very important points on TEM that the electrons are traversing in helical trajectory, that depends on the magnetic field which is being applied. Because, that magnetic field is strongly creates a force, which is perpendicular to both the velocity and the b part of it, the magnetic field part of it. So, that eventually forms a helical trajectory and then, it creates some sort of a rotation of the object itself.

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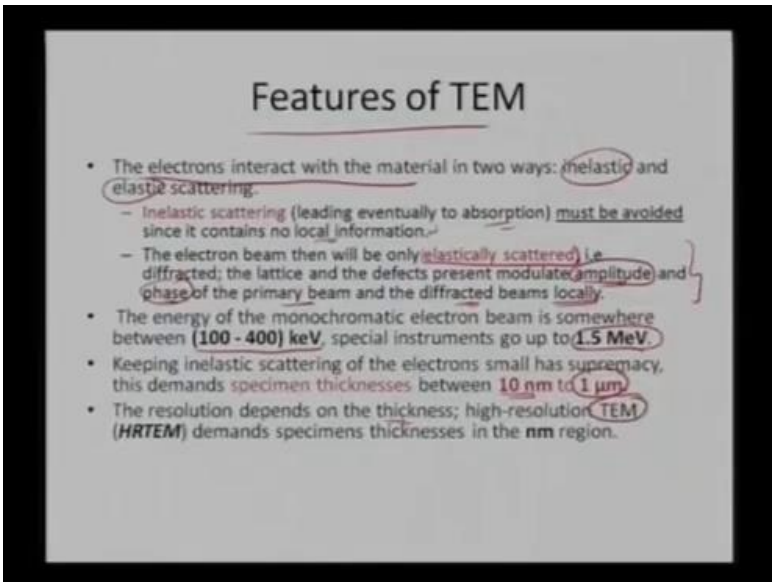

So, the features of TEM when they basically involve like this, that my electrons are interacting with the material either elastically or inelastically, but I want to avoid the inelastic scattering. Because it is not containing any information, it is similar to like leading to absorption, because I have, I am applying certain electron beam, I know it is energy, I know what are its features and it starts getting scattered in inelastically. I do not know how, what kind of losses it has gone through, once it has interacted inelastically and after its diffraction with the material I get some, I am getting some information, but I do not know how it is generated, what are the incident energy of the electrons. So, I have always need to avoid what is happening inelastically, because it would not would not contain any local information.

But if I am letting it elastically interact or elastic diffraction can occur then, basically I can also moderate its either amplitude or its phase with the primary beam. So, I can get both the information from either defects or the lattices, from that I can what is it kind of amplitude change or what is it kind of phase with respect to the primary beam, primary beam. And, so I can get the information I can extract the information from the diffracted beams what is happening locally. So, there is a advantage of my elastic scattering of my material I can avoid, so I tend to, I need to avoid the inelastic scattering part of it.

And again the energy which is base basically, what I am utilizing in the TEM is approximately 100 to 400 kv to result me Ewald sphere, which is inverse radius of 2

picometers and these phase, we can go energy as high as 1.5 mega ev. In that, in that particular cases I can have sample which is even more than a micrometer in thickness. So, that part basically decides the overall energy, which can pass through the material. So, conventionally or these we generally utilize energy which is approximately 100 to 400 kv and that is in a getting a giving us a good information, but instruments as high as 1.5 m e v, m e v are available which can penetrate down to penetrate into a material, which is approximately more than 1 micrometer in thickness.

But generally thickness of material has to be approximately 10 nanometre or may be the the lesser the better and again my resolution part depends on the thickness. And again, if you want to get a high resolution TEN imaging and that basically for that particularly we need to have, thin the specimen to be thin enough approximately nanometre in thickness. So, I can extract much more information in terms of its lattice fringes or high resolution grain boundary imaging and so on. So, overall which is of TEM are that I need to get, I need to avoid the inelastic scattering, I need to get the inelastic scattering and from that I can get, extract then from which can either from the lattices or for the defects. And then, I can measure the amplitude or the phase and I can tell what is happening locally in the particular material.

The overall energy what I the electron beam energy what is been interacted with the material, that generally is approximately 100 to 400 kv and for that we need to have a specimen thin, approximately 10 nanometre. And then these days some other instruments are also available which can increase the energy to 1.5 m e v and for that my sample size can be as thick as 1 micron. And for high resolution imaging I definitely need to have a material which is thin enough less than couple of nanometres.

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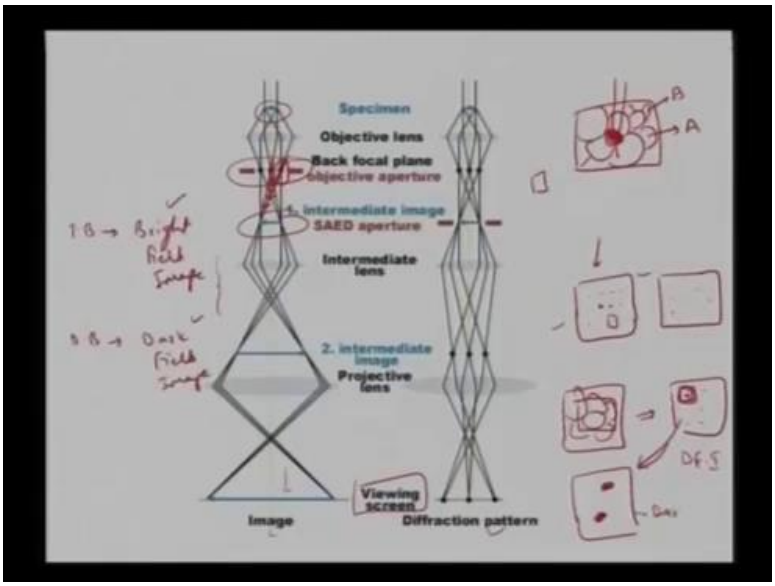

So, coming back to the ray diagram, I can see that my specimen lies here and then I have my back focal back, focal plane then I have my intermediate images which is forming. Then, I have some intermediate lenses and then finally, I get my image on the either as an image or as a diffraction pattern I can, I have a, I can have a screen. So, that is what is required out here that, I can selectively take a particular beam, in back focal plane I can allow only the transparent beam to pass through or I can also the diffractive beam to pass through. So, if I am allowing only my transparent beam to pass through what I get is something called bright image, bright field image or if I let only for the diffraction beams to pass through, diffraction beam to pass through, what I get is something called dark field image.

So, if I use a particular aperture, back focal plane aperture through which I am letting only my transparent beam to pass through, I get something called bright field image. If I let only one of the diffractive beams to pass through, what I get is a dark field image and conversely I can look at the certain particular area. If I, if I have particular microstructure in the in the bright field imaging say, I had a particular kind of a particular images which is formed in the bright field image and if I want to, want to see what say this these can be of different phases this can be phase a this can be phase b. And to confirm that, I need to get it diffraction pattern, because diffraction pattern is arriving from the elastic, elastic interaction of the beam with the material.

So, if want to see what is this particular phase say I want to see what this particular phase is, so I can let my beam concentrate on this particular part and it can give me a diffraction pattern. I can get a particular diffraction pattern, that will be consistent to the kind of orientation. So, I can again take my particular sample to get a different orientation, because within a tilt of few degrees I can get diffraction pattern of any different planes. So, I can get diffraction pattern depending on the orientation of this particular crystal. So, if I can align my beam according to this particular phase I can get some diffraction pattern.

So I can, I can do that that part as well or I can go back to it more like this, that I can select one of the diffracted diffracting spots. If I take an area and I can take as bigger area instead of a small focused regime, I can have an aperture, which can accommodate more number of, more number of grains out there. If I choose this much area which has more number of grains and I am getting some diffraction spots. Alternately I can choose a particular diffraction spot, I put my aperture here and then I can again come back and see that this diffraction spot is resulting, because of which particular grain. So, which all those particular grains which are contributing to diffraction spot will start appearing brighter and this thing is called dark field image.

Because, I am letting only one of the diffraction spot to pass through and only certain areas which are contributing to my, this of my diffraction spots, because they are oriented favourably. They will start appearing bright and rest of the field will be nothing but. So, I have a dark field but, my features are bright which are resulting this particular diffraction spot and this thing is called dark field imaging. So, that part I can achieve with the transmission electron microscope, I can either get an image or I can also get information about a particular crystal through its diffraction. So, that is the work capability of my transmission electron microscope.

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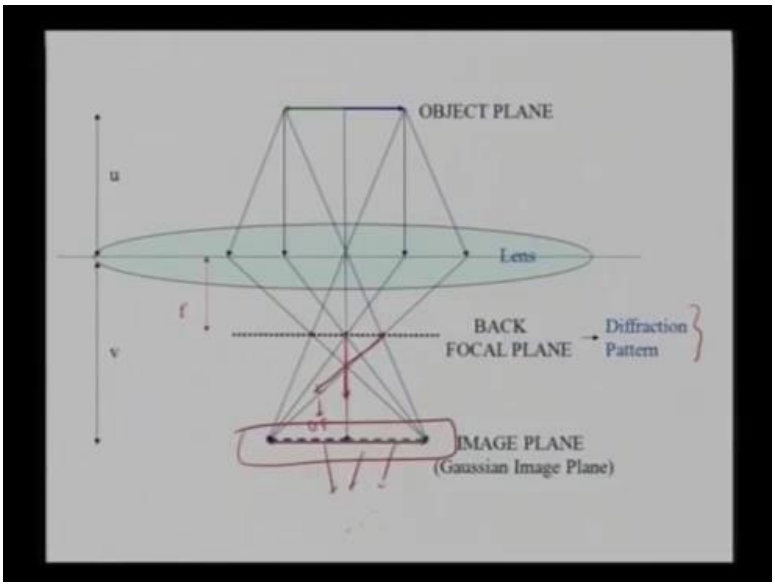

So, eventually what we can see, I have my back focal plane this gives me diffraction pattern out here and if I keep my aperture somewhere, I can get either a bright full image. If I let my transparent beam pass through or if I let my diffraction beam pass through, I can get some image and that will be the dark field image or if I can put my aperture at this particular location, which is nothing but a Gaussian image plane and that will eventually form a diffraction pattern. So, I can see that an transmission electron microscopy I am capturing the beam which is passing through the material. So, my particular specimen or my sample is to be transparent to the electrons.

So, if I am using something which is much thicker through which electrons can pass through, I will not get any information. So, the information like secondary electrons backscattered electrons they have to be collected back like not to the material, but back on the surface of the particular material of the sample. But in transmission electron microscopy I make this sample thinner, so my electrons can pass through and they can interact with the material elastically and then I get the information. So, for the diffraction the place have to be almost parallel to the incident beam and then, they get diffracted within the regime of 0 to 1 degree.

So, I can see that for a for a particular plane to align I have to tilt it to marginal extent, so I can align it and I can get a diffraction pattern from that particular plain and again the zone axis becomes the incident beam itself. The direction of incident beam becomes the

zone axis of the plane, which are diffracting, which are getting which are basically diffracted in the electron beam. So, I can see that the zone axis of that the incidental beam itself becomes a zone axis and then again the sample preparation is very very critical. Because, for electrons to pass through they need to be, they need to basically interact with the material and the sample itself should be thin, that the energy that is supplied to the electron is enough for it to come out of the material.

That is, so that is the requirement of that and again my Ewald's have become so huge in comparison to the x that of which is constructed in the x ray diffraction, that now my Ewald sphere can touch more number of points in a reciprocal lattice spacing. So, more number of planes basically produce a diffraction pattern and now I can analyse the diffraction pattern to come out with what is the whirl material of, what, how what, how the basically, what, how what is overall phase present out there in my particular material. At the same time, I can get the get an image, I can see the crystalline nature of the material, I can also focus a Nano crystalline grain itself and I can get some information out of it or I can do a vice versa.

Then from a particular diffraction pattern, I can look which all grains are oriented favourably to my incident beam. So, I can see this kind of things in the transmission electron microscope and there is much more to be explored, which I will basically cover in part two and I end my this particular lecture here.

Thanks a lot.