Electron Diffraction and Imaging
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Lecture – 34
Revision of recent trends in microscopy

Welcome you all to this course on electron diffraction and imaging. We have almost come to the end of this lecture series. In the last few classes, we have been revising some of the topics which we have covered in detail in this course. So, what we have done essentially is we looked at what is the basis for our conventional transmission electron microscopy, the operating principle of the microscope; there are different modes of operation both in diffraction as well as imaging like parallel beam deflection; converging beam diffraction, Kikuchi diffraction.

Similarly, bright field dark field and weak beam imaging then we came to high resolution transmission electron microscopy that is what we revised in the last class.

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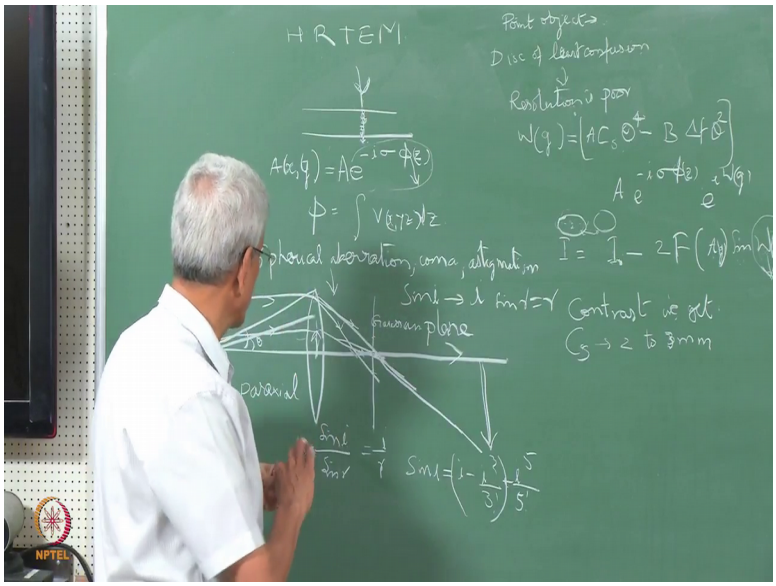

In that high resolution transmission, electron microscope essentially what we talked about was that we considered the electron beam as a plane wave which is entering into the sample. As the beam enters into the sample; as it passes through the different regions of that sample; what is essentially is being done is that it the potential of the atoms which

are there at different positions or the potential of the sample. It is trying to modify the phase of the wave phase of the incident wave. So, the wave which comes out of that sample at the back of it has got a phase which is different from that of the phase with which it has entered that sample this is called as the exit wave.

So, essentially what we considered we considered that sample as a function we can represent as a function A x and y; this is equal to e to the power of 5 some function sigma into phi of is r thickness. This phi is essentially the one which depends upon the potential. We wrote phi equal to integral of V x y z V z. So, I essentially; we considered this is a phase object some factor amplitude will come in the picture, but we assume that that remains the constant. Essentially the phase of the incident wave is getting modified as it passes through the sample. This is in an ideal case, but since we are using the lenses; what is essentially that objective lens does? It is the objective lens has got some operations which are associated with it. They are spherical aberration then comma astigmatism; all the rest are fit like being cushion point; cushion distortion curvature of the field. All these things happen; what is essentially important is the spherical aberration; what does spherical aberration does is that it essentially brings about a lot of changes.

Before we talk about what all; how it deteriorates the resolution; let us first talk about what is spherical aberration. So, in a lens; normally we assume that in a paraxial condition as the beam passes through that sample that is if the beam is coming from this point and the beam is coming from these points and all these points; we assume that at the back focal plane; all the rays which are parallel; they are all focused to a particular point and then they form an image. In the image plane and we consider this as a paraxial approximation. What is paraxial approximation is when the beam enters from here to the lens which has got a different refractive index it as undergoes refraction and that is given by the formula mu equals sin i by sin r.

Paraxial approximation means that if the value of i and r are small; you know from mathematics that sign i can be approximated to i sin r can be approximated to r then we can write it as i by r. This is the approximation which makes all the rays whether they are close to the optic axis or far away from the optic axis to be brought to a focus at the back focal plane at a particular point; what actually happens in reality is that the rays which are far away from the optic axis; the sin i that angle i and r which the beam makes with

respect to that lens is large because of which this approximation is not valid. So, in such a case, essentially if you wonder to use we have to; we know that expansion sin i equals i minus i cube by 3 factorial plus i to the power of 5 by 5 factorial this sort of expansion could be used.

So, essentially this is the sort of only the first 2 terms are taken this called us the third order correction when we do that what is essentially is going to happen if that the ray which is coming from here the ray which is close to the optic axis it is broad it cuts at the optic axis the way which is far away from it. It cuts somewhere here because of which what is essentially is going to happen is that far up rays which are emanating from a point we do not get a point image instead we get essentially yes product image in the Gaussian plane. This is Gaussian plane.

So, this approximation we call it as what is it? So far points object if the object is a point object at best; what we can get is one your disk of this confusion that is the size which we can get it. So, what is the effect of it this effect of this is the solution is poor resolution is poor or resolution is limited by that spherical aberration the next point is that. So, essentially what is going to happen is that for an object; which is point; essentially, we are going to get this where exactly that image of that object is going to be there; we do not know these are all the problems which we face in the case of a conventional electron microscopy.

So, because of this aberration instead of a point object; we get a disk of this confusion, but fortunately we know that if we defocus that sample the defocus behaves in a wave where here in this case the ray is the refracted ray is brought also towards that optic axis defocus what it does? It is it is mostly away from it. So, these 2 act opposite to each other. So, this introduces; what is called yes; a net phase error. We call it to the optical part that phase error is one depends upon C S to the power of theta to a power of 4 e A minus B A constant. This is the delta f theta to a power of 2. This is the sort of a expression which we get it for w g.

This w g is essentially convoluted with the sample function. So, essentially A exponential minus i sigma phi is there. This is convoluted with e to the power of i w g; this is what happens. So, all the mathematics when we do it finally, what is essentially is going to happen is that with respect to the; you are very scattered beam.

Finally we will be getting A that intensity equals the 1 which corresponding to the intensity corresponding to a central beam then another factor which will come is to that Fourier transform of this function A will come and into a term which corresponds to this sin omega g which will come the essentially; what this is going to do is with respect to this will be A f g. So, with respect to every deflected wave; this contributes to the contrast depending upon the sign of this. This can either add to that contrast or it can subtract that contrast and the net effect of it will be that how many scattered or the diffracted beams can give rise to a contrast that is what it matters. How we look at it is that if suppose; this value of this term is very small in the sense that almost all the terms have got the same type of a sign; sign w g then what is essentially is going to happen is that all of them will try to add to the contrast or subtract the contrast. So, that in the high resolution image whatever is the image which we are getting it in this image the contrast is enhanced or the contrast is decreased.

So, essentially what is going to happen is that either its adding it or decreasing it whichever way it does it brings about a variation in contrast and that is a contrast which we observe as dottie contrast in the image which we have seen in many of these pictures then what is essentially going to happen is that when such a contrast which you are observing it suppose in some case that value of this we can see that this depends upon this value of theta what is this value of theta the theta is essentially with respect to a length what is the bi-factor beam or the scattered beam when it reaches lens what is the angle which it submits.

So, it all depends upon that depending upon that this value can change from if it is sin pi by 2 that w g becomes then this becomes one, otherwise if the sin changes value becomes larger than that the sin can change and when that changes for different values it will be that contrast which we get. We will not be able to explain all these things. We have discussed in the last class. So, essentially what is going to happen is this spherical aberration one it is the for a point object you get a disc of these confusion that brings about the resolution and that other effect of it is that this affects these decides what is going to be the contrast which we get.

So, the third part of it is that when this sort of for a point object, we get this sort of an image; we cannot precisely tell where exactly is the position of the atom from this image trying to find out where exactly a position of the atom in that sample it is going to be

very difficult these are all the problems which we encounter in a conventional high resolution electron microscopy and generally the value of C S that spherical aberration is about something like 2 to 3 millimeter.

Suppose we can make this spherical aberration extremely small how exactly it can be done as we have seen the rays which are far away from the optic axis they are brought to a focus before the back focal plane if we use some quadruple lenses essentially, what it does it is we have seen that these quadruple lens are used for a astigmatism correction that essentially what will do the rays which are far away from the optic axis are affected by that, but the rays which are closer to the optic axis; they are not affected as much because of this they can be used to correct and bring there is back to the back focal plane this is precisely what is being done when this C S is made small delta f also can be made small.

When the C S become 0 delta f can be made 0 then this phase error can be made into 0; that means, that in such a condition we are get in that case we are getting an ideal mic that is the aberration is not the spherical aberration is made 0 or the error which it introduces in the optical path that has been made 0 and because of which for a point object first the point image could be obtained. So, the solution is whatever is given by the Rayleigh criterion that sort of a resolution we should be able to obtain.

The next part of it essentially is going to happen is that this spot is now become a point or this. So, any small changes in the object there will be a relatively change in the image also from which we can measure the position of the atoms positions in the image could be measured accurately and the third is that all the terms are going to contribute to a contrast. So, we get a much better sharper contrast. These are all the 3 advantages and these 3 are utilized to get a lot of information about the sample.

Some images are being shown here these images I had already shown earlier, but what is essentially one has to see here is that one more point which we have to remember is that using these lenses the quadruple or hexapole or octupole lenses. We can make the spherical aberration not only 0; spherical aberration can be made negative also positive all such things are possible.

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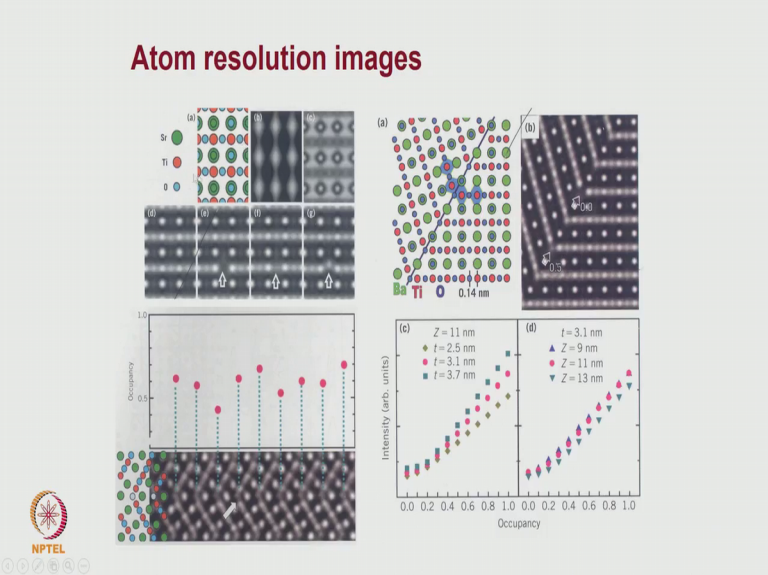

Using these condition essentially one thing which could be done is that now this is a sample where the image is taken a sample of zirconium titanium I said that strontium titanium you cannot zirconium its strontium titanate. In this particular case, if you look at it in some positions, there is an absence of titanium atom position is there right not titanium oxygen atom position that absence could be noticed and another one you can see that this atom has slightly shifted and; that means, that the position of the atoms the image could be measured very accurately especially here we know that this strontium titanate when it exhibits piezoelectric effect, it is in a tetragonal distortion is introduced to a crystal structure.

So, the shift of the atom positions could be measured accurately not only that using this we can find out; how many atoms are present along each of this column as the beam passes through; if some atoms are absent; there will be a variation in the contrast all these information could be obtained very clearly. So, in this particular case also, we can see that here that absence of a atoms is there all these aspects could be imaged and we can give correct explanations for it and using these images we could find. We could get the exit wave function that is as I mentioned here as the electron beam enters passes through it; only the phase has been modified. So, the wave as it comes out of it; it has a different phase compared to what it has been it has initially entered and this field is called as the exit wave essentially what we considered as image is nothing, but that of the

exit wave function that is what we are getting it in all the high resolution images this is the great advantage.

So, all these aspects; we have covered in detail essentially what we have for as I mentioned earlier and also in the earlier classes; the most important thing is that the resolution point to point resolution is improved. The consequence of this is that the position of atoms in the image could be measured quite accurately. The third is that almost all the atoms that is normally in a conventional high resolution images; if a heavy element is present. In the presence of the heavy element the light elements their images, we do not see essentially, it is the heavy element which contributes to the data contrast whereas, in these cases from this example can see the strontium which is a heavy element and an oxygen which is a very light element all could be imaged this is the greatest advantage of a high resolution microscopy.

So, this is one of the recent advances which has come from the beginning of this century this is being used to get information about the samples which was not possible like for example, we can get information conventionally when we talk of electron crystallography or crystallography we essentially assume an ideal unit cell were all atom positions are occupied, but from this technique are using C S charactered microscope we can get information about the defective crystal structure or the real crystal structure and this could be used to find out; how the band structure changes. All the electronic property changes in real samples all those informations could be obtained recent technique which is being extensively used in electron microscopy is called as that the electron tomography.

Tomography is a word which you have heard and you know that computer aided tomography are cat scan which is used in medical field to get 3 dimensional image of that objects; either it can be done using X-Rays or it can be done using ultrasound. Various ways in which one it is being done similar why it is being done because in a 2 dimensional projection; we do not get exact morphology of that sample for example, in a tem if you look at.

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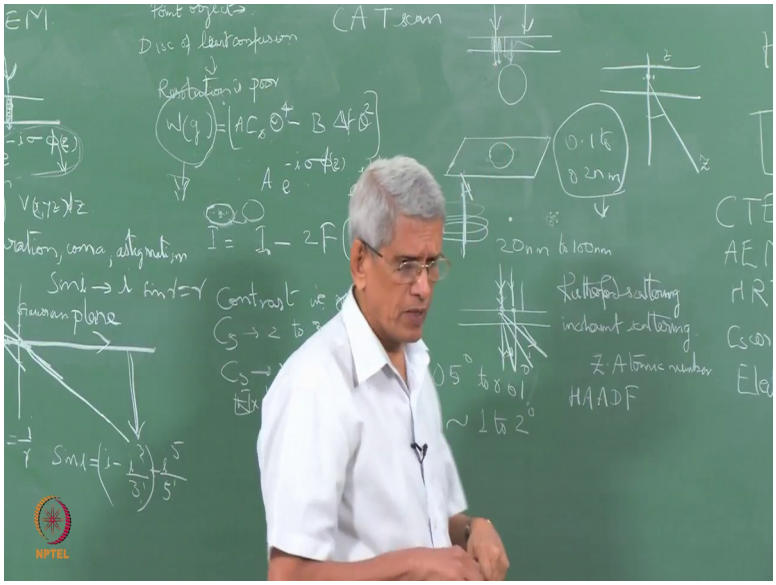

It is a sample as the beam passes through the sample the projected image which we get it on the screen that is in till it is only a projection of the morphology which we give for example, suppose we take a thin disk and the disk surface is parallel to the beam direction then what is going to happen is that that image will appear as just as a thick line.

By looking at this image, we will not know what is the morphology of that sample; if it tilt it by a small angle then we know that this image will just change become an elliptical one; you can depending upon which direction we tilted finally, when its tilted to ninety degree this will become at a circular one. So, this is a very simple case which we can consider especially in biological samples like many of the molecules are proteins structure. So, many biological systems when one looks at it they are very complicated complex morphologies which they have in such cases to get the true morphology which is very important for many applications and understanding is or can be obtained if we tilt that sample in 2 perpendicular axes by some small degree maybe normally what is done by about one degree or 2 degrees.

It is tilted and the image is being taken and then all these images are stretched because when these images are being taken that contrast will be continuously changing, then there are some softwares which are returned with which one can construct a 3 dimensional image of the object which we can reconstruct that tells what exactly is the

morphology which these different viruses or bacterias or various proteins which they look at the microscope can the same thing has been used especially in material science also to find out the morphology of the different type of precipitates which form during phase transformation.

Another one which is used quite frequently is to find out the magnetic domains because we know that in an electron microscope most of the samples which we are looking at are non magnetic samples. If the sample is magnetic, there is a problem. What is the problem? The lens itself which we are using it in an electron microscope in the normal case optical lenses are glass; whereas, in the case of an electron microscope, the lenses which we use are essentially electromagnetic lenses in an electromagnetic lens.

The essentially a magnetic field which is being my current is passed through a coil which generates a magnetic field. This is the coil current; the magnetic field which is generated with the north and the south pole along that axis and as that electron beam travels in the same direction one this optic axis its un-deviated, but if the beam is inclined with respect to the axis of that field then what is going to happen is the Lorentz force will be acting on it this force is what is responsible for the beam to spiral and give and that is use to generate this our construct the a convex lenses.

If the sample itself where magnetic field is different in different directions; then what will be the effect of that? Normally as the beam passes through that sample, the scattering is taking place within the material depending upon the planes which are in claimed with respect to a beam direction whether the sides face black condition or not; in addition to it, when the magnetic field is present in this; that will be then at generating a Lorentz force on the electrons and this will be changing the direction of the scattered being or the diffracted electron beam.

The consequence of this scattering is essentially going to be that if we get a diffraction spot, this is the central spot and we get a diffraction spot here in a non magnetic sample. Suppose the sample undergoes from magnetization when we are viewing it at lower the temperature, then this part will be essentially split into few that depends upon the type of domains and the direction of the magnetization. Now we can put aperture on each of this part and then try to image the magnetic domains this is exactly what is being shown in this here you can see that depending upon the domains the diffractors spot has been split

into 4; using this we can image the domain boundaries and using and this images are called Foucault images.

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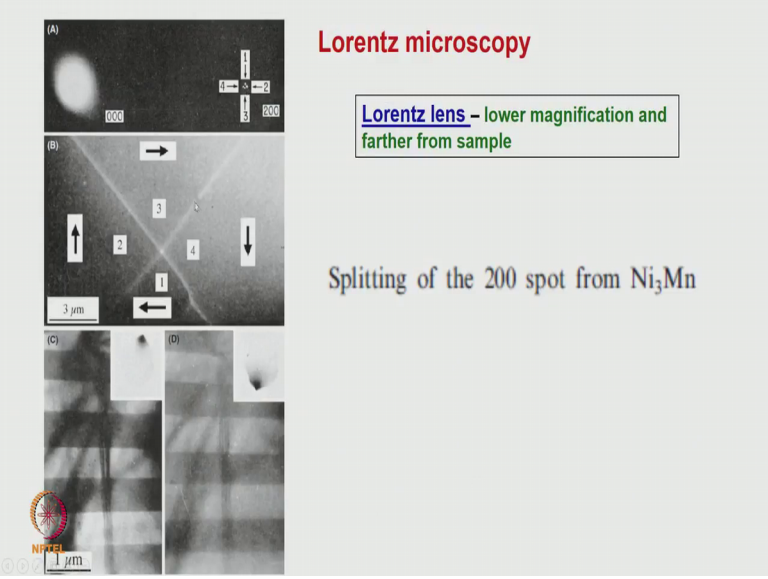

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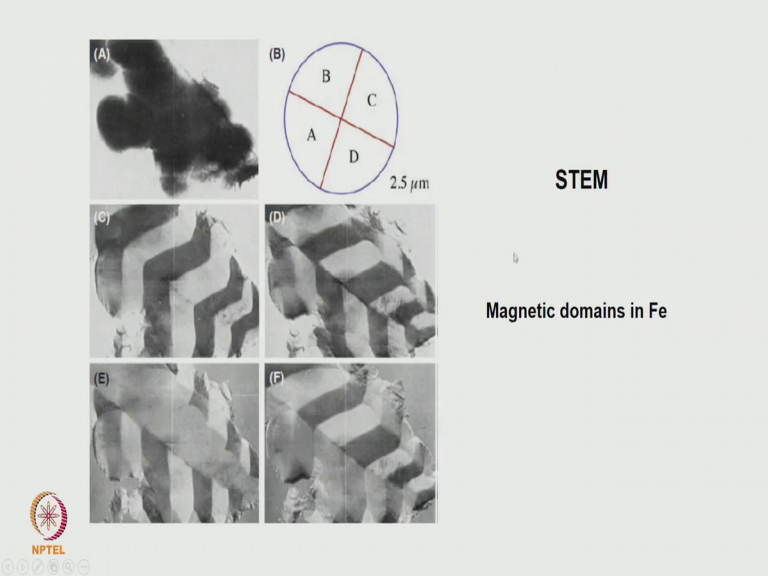

But essentially what is important is that in this sort of Lorentz microscopy; we can find out; how the magnetic domains are distributed. Like in this example, but we cannot get any information about the intensity of the magnetic field and the same Lorentz microscopy done in a scanning transmission electron microscope also this is another technique here also one can image the domains quite nicely because so far whatever we

have covered was conventional transmission electron microscope where the beam is falling onto the sample and the beam which is scattered; they are scattered in different directions because of the crystal energy of the material and this scattered rays are brought to a focus at the back focal plane where the diffraction pattern we get it and in the image plane we get the image.

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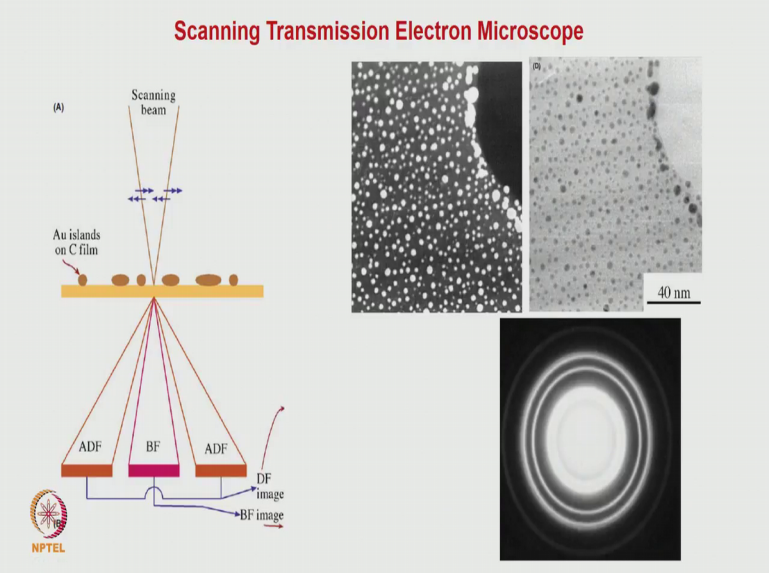

Another way in which we can do the same images which we can obtain other images of the sample is that if we make the beam into a very fine beam; this is possible because of the advent of FEG; that is field emission gun which gives very good brightness and since the operations could be corrected, we can focus the beam into a very fine point of the size of around 0.2 to 0.1 nanometer and this beam can be made to scan on the surface of the sample as the beam hits at this particular point; the beam is scattered and the transmitted beam and that a diffracted beam will be coming.

These beams could be separately collected using detectors and effectively that if we scan the beam from point to point and the transmitted beam is collected by this detector depending upon the; how much is the scattering which is taking place in other directions there will be a variation in the intensity of the beam which come outs this will give rise to a contrast like in an SEM. We can get an image and with very high resolution this is what is done in a scanning transmission electron microscope and that diffraction. If we

collect the images which are coming to this annular detector which is being used, then this will give rise to a dark field image of the sample.

Some of the examples is shown. This is a bright field image; the sample and this is the dark field image. This is the diffraction pattern which has been obtained at the advantage of scanning and transmission electron microscope is that in the normal sample as we had seen that contrast arises because of a bending; there is a bending of that sample is going to be there, then that gives rise to some dark contrast all those things does not affect the contrast in scanning transmission electron microscope.

So, we are able to get relatively a much better uniform image and especially when we wanted to find out the particle sizes and all this image could be used to get correct information the details of it I have covered earlier. So, I will not go into it. Another important information which we can get it in this microscope is that when that beam is falling on that sample and we are making it move from one region to another; as it moves from one region shifts to an another region, we are able to collect a diffraction pattern from each of this region and another from this region like from every point where the beam is falling; we can collect an independent diffraction pattern. If we analyze the diffraction pattern, we can get information about the orientation of the beam the orientation in the sample that if from region to region; if there is a variation in the orientation of that sample is going to be there that will be done. So, this is called as the orientation imaging microscopy.

This is similar to the EBSD image which is get obtained in the reflected mode in scanning electron microscope; in this also; one can get an angular resolution of about something like 0.5 degree to 0.01 degree and then the area which we scan could be some micrometer square to millimeter square and the beam sizes could be the lateral resolution could be from something like 20 nanometer to 100 nanometer. With these resolution, we can get information about from region to region; if there is a small mis-orientation of about point 5 degrees that could be easily measured. It is an elastic scattering, but what is essentially is going to happen is that in this scattering; this is its essentially an incoherent scattering and more than that the intensity of the beam in specific direction depends upon he said that is the atomic number of the element.

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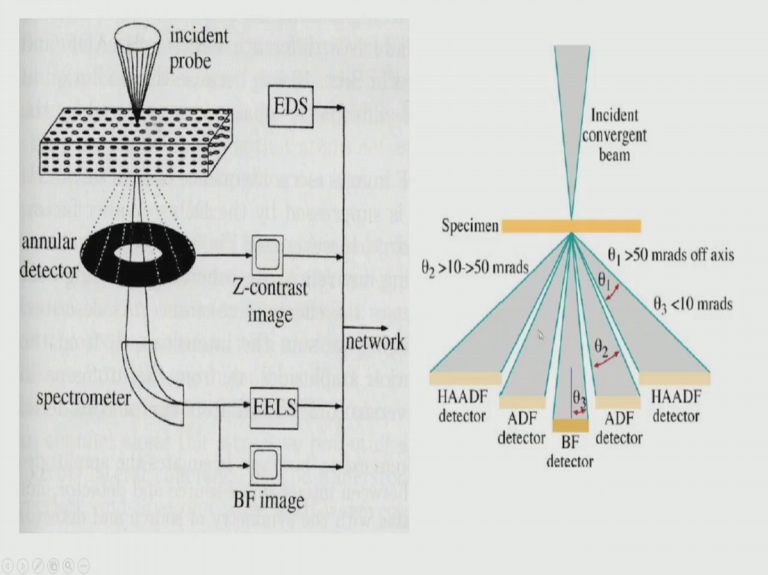

Suppose we assume that I think with this slide which we can see it here is that there is essentially as it is being mentioned the bright field detector could be used to get information about the bright field image. Annular detector, dart field detector could be used to get this information and this the influence of the effect the Rutherford scattering that could be collected by what is called as a HAADF detector that is high angle annular dark field detector. This essentially happens when the scattering angle is more than 50 milligrams or more in this case suppose; we assume that in that sample from specifically this region some element is present surrounded by a yellowish of the element and in this large angle which it makes with respect to it from this element; the intensity of the signal which is coming is going to be large which is depend upon this; whereas, the element which is there the intensity of the signal which is going to come from here in this direction is depends upon this.

So, because of the variation in this since the intensity depends upon that gives rise to a contrast this contrast could be use to get information about how different elements are distributed in that sample not only that and since I mentioned that the beam in scanning transmission electron microscope can be focused 2.1; 2.2 nanometer which is almost of the order of the atomic resolution we can make the beam scan across column by column of the atom. So, as the beam passes through here if the atom is going to be there then the transmitted direction intensity will be less and in the in between region the intensity will

be more essentially here sort of a lattice resolution image which we can obtain that is what is being shown here.

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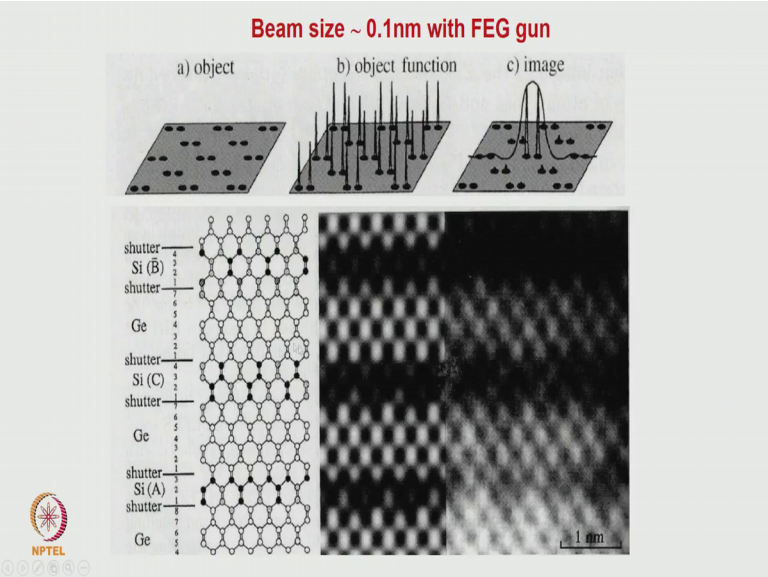

In this specific case, it is essentially a multi layered sample which contains silicon germanium. Silicon germanium like that it is happening and in this case, you know that the silicon and germanium has got different atomic number because of which we can see that the contrast; there is a variation is there. So, essentially what is being done is that using this Rutherford scattering or you seeing this high angle annular dark field detector; we are able to get image at which region which type of elements are distributed, but still we should have some idea of the type of elements because this can only tell that depending upon the intensity of the signal which is coming or the intensity of the image we can tell whether it is a high inset or a low inset element, but to find out which is the particular type of an element we may have to use either EELs or EDs using that we can find out which specific element is responsible for it.

So, essentially using a combination of various techniques, we can get an information about how elements are distributed; not only how the elements are distributed; we can get information about this distribution almost on an atomic scale that is along the column; how the variation is going to take place from region to region and an atomic resolution distribution of elements could be obtained, but using this; still we have not reached a stage where we can find out at which position in the sample that atom is sitting

for example, in this particular case if we see here we can notice it that in this position; this regions are bright which corresponds to antimony which present along maybe in this column which is sitting there that is what that information.

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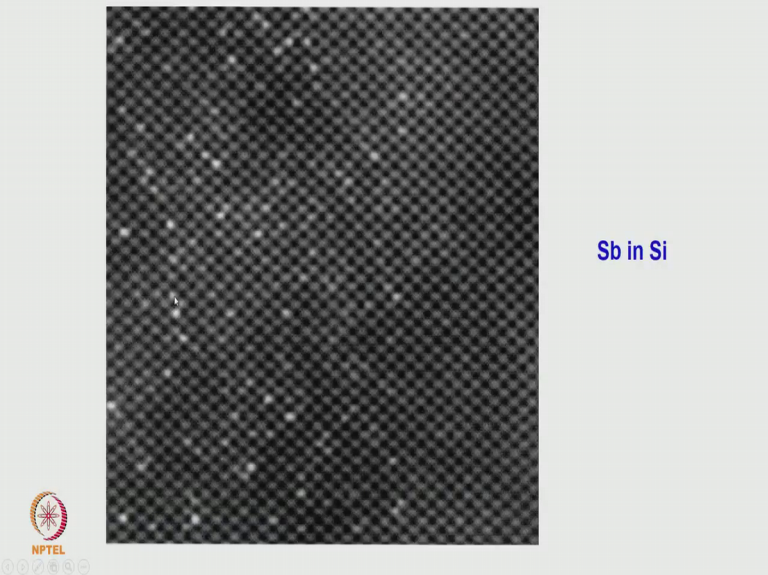

But in that column whether the antimony is sitting here on in this region or in this region that is going to be extremely difficult, but in the lateral resolution or the lateral space of that sample; we can find out in which specific location these atoms are distributed that information; we can obtain; another important technique which has come is what is called as the electron holography because as in this class, I started with talking about high resolution microscopy. So, that using high resolution microscopy, we can find out distribution of that atom positions that is what our aim is why do we wanted to do that? This is to find out which positions atoms are occupying in the unit cell in the crystal; we wanted to get that information.

But generally what happens is that when we get the image; we know that the amplitude essentially is a complex function and because of that the size i star the information about the phase factor is completely lost. So, what does not, but what we have done it is that in a high resolution microscope. This phase information; we have tried to convert this into a contrast. So, if we can try to quantify the contrast then we can get some information about the phase of the in wave exit wave for that what it has to be done is that we have to assume a model of that sample and then go about and do it and many images have to be

taken in a higher conventional high resolution image and then we have to do an image simulation then only we can get the information about the crystal structure.

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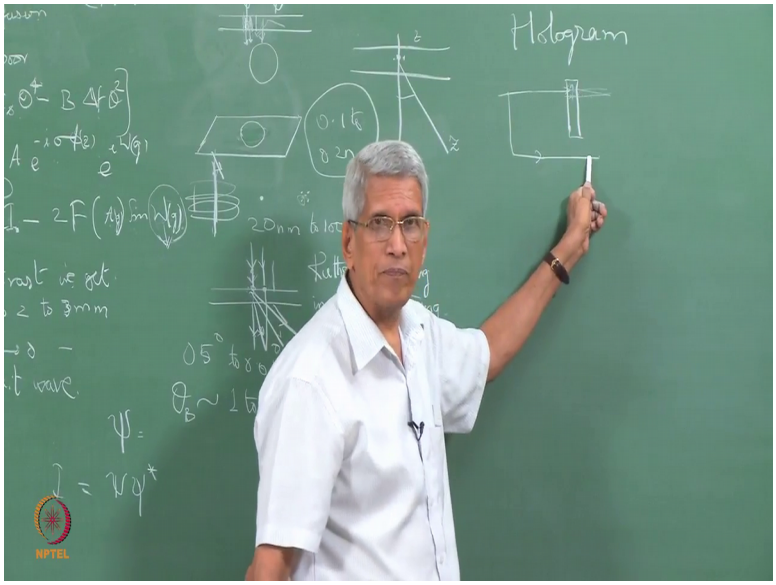

The same information can be obtained in a much more elegant manner using electron holography. Hologram is a technique which has been invented in 1950s for which Gabor got the Nobel Peace. What is the principle of a hologram? Essentially as a coherent monochromatic beam passes through a sample; suppose it is a sample which is there as the beam passes through the sample; what is essentially is going to happen is that depending upon the refractive index of the material; in case some phase difference will be introduced as the beam passes through different regions; different phase differences could be introduced in different regions and the rays which come out will all be coming out with different phase different-different phase.

If this beam is split into 2 and this is called as the reference beam; if we allow these 2 beams to interfere then what is going to happen is that the interference pattern which we get it or that is called as the hologram that contains information about the phase that is the contrast which is going to arise depends upon what is the sort of a phase difference which has come at different region with respect to the reference beam this is how a hologram is made.

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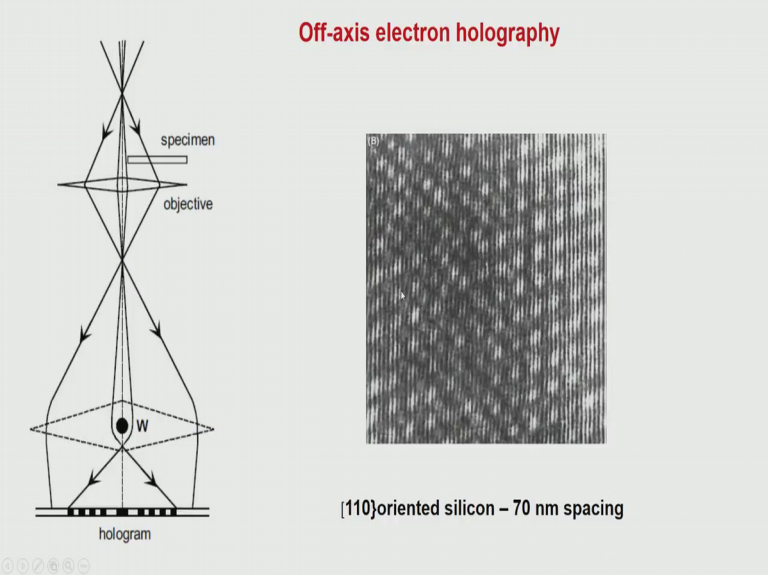

The same principle is used in a electron microscopy; also there are many types of holograms which are being made. They are called the on axis hologram off axis hologram. There are various types are there essentially, what is being done in an electron microscope is that that beam is made to part of the beam is made to pass through that sample and different regions that the beam passes through as we have seen that exit wave. The phase has changes depending upon the local variation in the composition of that sample and this is the reference beam with which this is being made to interfere split into 2 and interference is done and then this is being made to interfere with the a reference beam then the type of an interference pattern which we get it this is called as the hologram.

This pattern from region to region as the contrast varies that can be related to the type of phase which has been introduced to the incident beam at different regions of that sample. This sort of phase difference which we talked about in the case of a sample could be introduced by either the electrostatic potential variation in electrostatic potential or in the magnetic potential.

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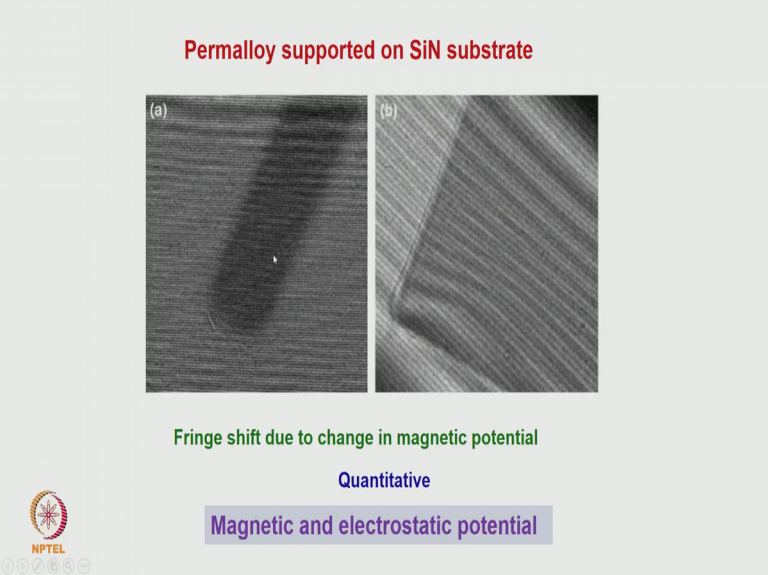

So, using and not only that this contrast which we are getting it are the variation in intensity could be quantified and that could be related to the magnitude of the electrostatic or the magnetic potential. So, as I mentioned earlier in the Lorentz microscopy, we can find out the magnetic domains, but we can find out the intensity of the magnetic field and the direction, but in the case of electron holography, we can get not only we can get the domain information, but also the direction in which the electrostatic field are the magnetic means that is in the case of a magnetic material it is a magnetic domain information.

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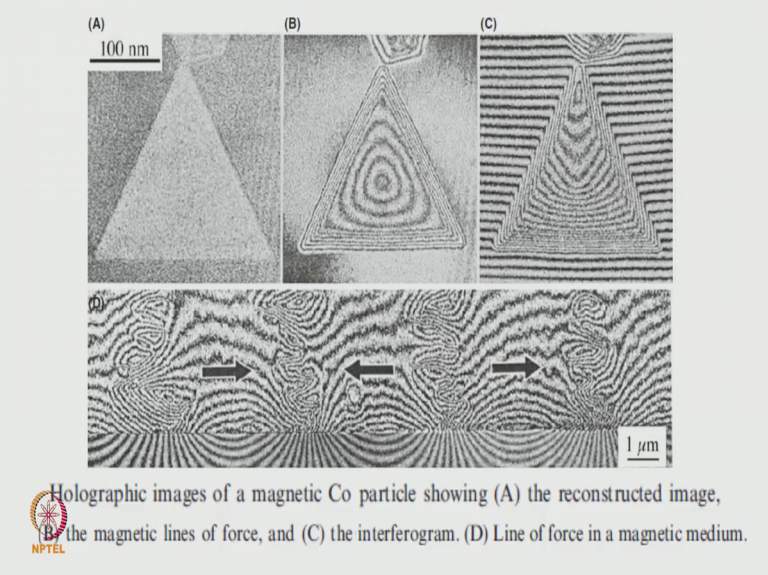

Holographic images of a magnetic Co particle showing (A) the reconstructed image, (B) the magnetic lines of force, and (C) the interferogram. (D) Line of force in a magnetic medium.

In the case of a piezoelectric material or ferroelectric material there we can get information about the how the domain such distributed also how the intensity changes all that information which we can obtain; this is essentially a holographic image from a magnetic cobalt particle; this I had shown earlier. Just I am repeating it again to just make sure that you can recall what has been described at that time and here you can see that this sort of fringe patterns which are coming and another is interference patterns which are coming. This is the reconstructed image and from this we can make out; how the magnetic field is distributed within that sample as well as the outside of that sample how the affinity is all those information could be obtained. This is an image which is taken from lines of force in a magnetic medium.

Especially when a lot of work is being done on memory materials, the electron holography technique in TEM is extremely useful to find out; how the different domains are distributed and this can be used to increase the capacity of that memory material for those applications. Electron holographic can be used if you look at a microscope; we have various types of microscope. We have conventional transmission electron microscope, then analytical electron microscope, then high resolution transmission electron microscope, then we have C S character microscopes which are very common now, then microscopes which have it electron holograph. These are independent microscopes which are optimized for getting the best information. These sort of microscopes are available. Other than that the recent develop in the development in the

last maybe 5 to 10 years is in; what is called as a pulsed electron microscope because what happens in all these microscopes? The electron beam is continuously falling on that sample surface and as the beam passes through many of these; samples are electron sensitive and high energy electron beam can damage the sample especially in the biological samples.

In such cases, by the time we examine that sample and image the information which we are getting is the about the sample which has already been damaged. To avoid this; what is being done is that the incident electron beam itself can be pulsed monochromatic beam is pulsed with at a particular frequency. So, that of a very small pulse duration; may be nanosecond pulse duration as the beam passes through the sample you record both the image and the diffraction pattern and then as progressively as the beam passes through that sample; we can get information about how the electron beam is modifying the sample; that information could be obtained. This has a lot of great advantage in biological samples; not only that we can get information about how the electron beam is interacting with that sample; that information also could be obtained from those images.

So, these are all the development; essentially, all these developments have taken place because as the beam passes through the sample, different type of interaction the incident beam has with the sample. So, one has to identify what is the way in which is interacting with that sample on that basis or one can create a particular set to collect that information and that information like you know collector and that scattered information will be able to give some specific information about that samples and this is all left to you people how you wanted to take this field to different heights.

So, essentially what I have done is reviewed the different types of microscope or whatever I have taken. So far, in this class, including a conventional microscopy high resolution microscopy also the recent trends in various electron microscope recent trends in the transmission electron microscope I will stop here now.

Thank you.