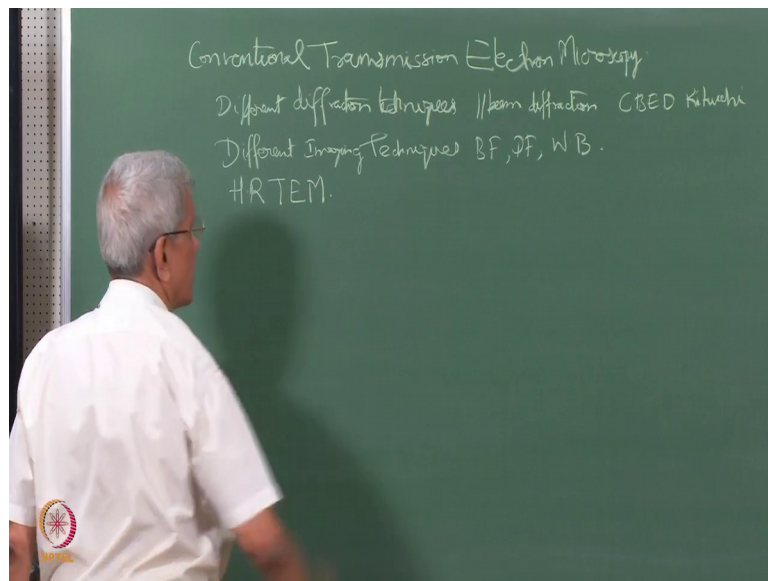


Electron Diffraction and Imaging
Prof. Sundaraman M
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

Lecture – 30
Recent trends

Welcome you all to this course on electron deflection and imaging, so far we have covered conventional transmission electron microscopy, microscopy.

(Refer Slide Time: 00:23)



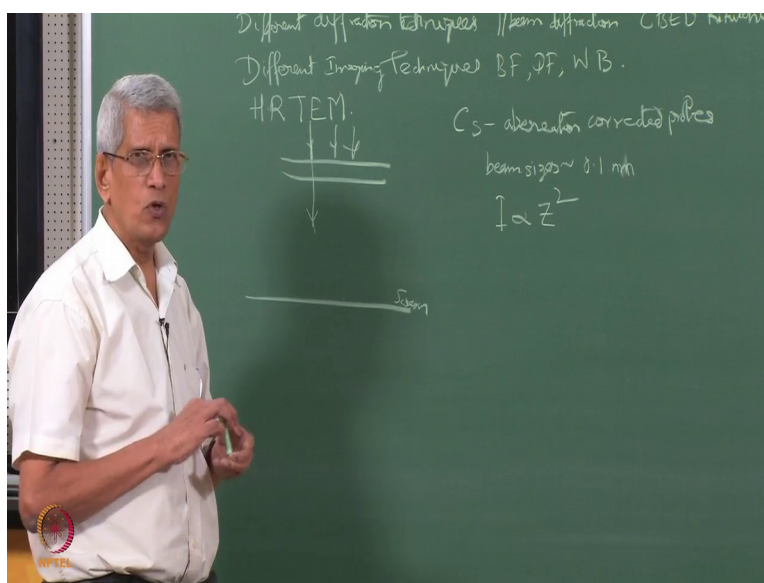
In this you have looked at different diffraction techniques as well as different imaging techniques. The diffraction techniques of essentially parallel beam diffraction con C B E D convergent beam diffraction and kikuchi diffraction which is nothing, but a divergent beam diffraction. Similarly, in the different imaging techniques we have covered bright field imaging, dark field imaging then weak beam imaging, then the other techniques which we have covered are high resolution transmission electron microscopy.

Similarly, a stenography projection which is required for a analyzing as well as interpreting relationship between different planes and directions in a crystals, all this aspects have been covered. Today what I thought is I will talk about some recent trends and new advances in electron microscopy which you should be aware off, what are the techniques which are available? One of the techniques which is available in an electron microscopy is called as an electron tomography. What is tomography? Tomography is

nothing, but getting a 3 dimensional image of a sample which you know that CAT scan which you call its computer aided tomography this is quite similar to that.

Because, normally what happens in an electron microscope is that, as there though the beam passes through the sample and through the full thickness the image which forms on the screen essentially a 2 dimensional projection of the 3 dimensional information which is available, Looking at that image taken in one direction we will not be able to make out what is the shape of this particle, these information we can get it using electron tomography this has lot of applications in macro molecular biology then in finding out the shape and size of second phase particles all these aspect which we will cover. The next one is which I wanted to talk about is Lorentz microscopy, what is Lorentz microscopy? This is a technique which is used to look at magnetic materials especially to find out how the magnetic domains are distributed these information which we can get it.

(Refer Slide Time: 04:44)



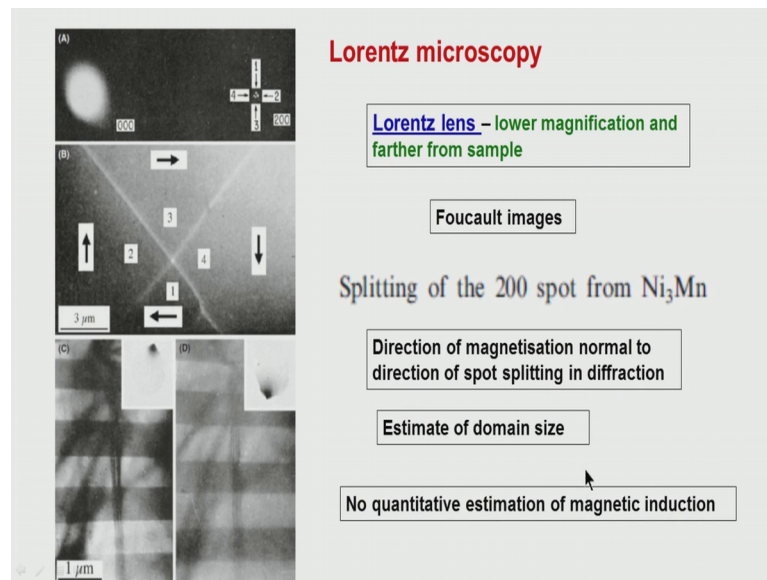
This we will cover, then the other one is scanning transmission electron microscopy this technique I had already covered and I had I told you also that using this scanning transmission electron microscopy we can, there is using the beam as fine as possible especially using C s r aberration corrected probes, corrected probes one can get beam sizes of the order of 0.1 nanometer with good beam current using any F E G s the a field emission guns that field amend and using this we can scan on that sample surface and the electrons the transmitted as well as the scattered electrons we can collected.

So, depending upon the interaction which takes place there will be a variation and this can be used since the beam size is very small we can almost get an atomic resolution and interpretation of these images are very easy also, relatively easy compared to the image which we get. Interpretation of images in high resolution transmission electron microscopy. This aspect I will not go into detail which I have already discussed at sufficient, using this same facility in the scanning transmission electron microscope using annular apertures there is an aperture which collects information at angles which are greater than, maybe 3 degrees one can get electrons which are essentially not a backscattering it is essentially Rutherford scattering.

So, these are all in coherent scattering and the intensity of the scattered radiation is proportional to z^2 at the atomic number that is, intensity I is proportional to the z square and here again since we use a beam which is very fine we can get atomic resolution information about the disposition of atoms in that sample, this aspect also has been already covered another is C_s, corrected high resolution that is aberration corrected the transmission electron microscope which also has been covered. I talked about what is the advantage of using this microscope how this gets information about the atomic; how this microscope could be used to get information about the atomic structure of the samples not only the ideal that not only ideal structure, but also the different structure.

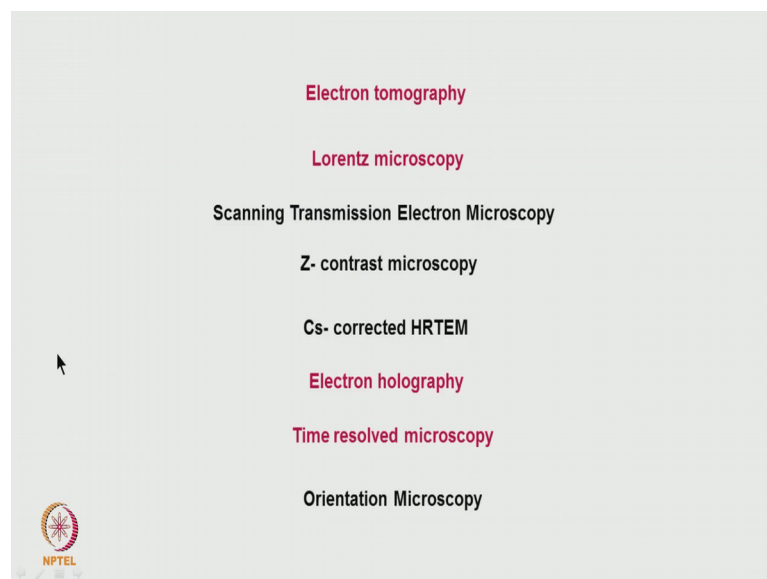
Another technique which is very important which is being, which is finding prominence is electron holography this is a technique which, with which we can get information about not only the phase, not only that I am from the image which we get we can get information about the phase cells (Refer Time: 08:09) that amplitude. So, that generating atomic structures from this information is relatively straight forward, this we will talk about it, the another type of microscopy which I will talk about is time resolved microscopy are dynamic electron microscopy or we can call it as a pulse the electron microscopy. This also has got a lot of applications which we will talk about orientation microscopy is one like in SEM you know about EBSD is used to get information about how different grains are oriented ok.

(Refer Slide Time: 08:58)



Similar information could be obtained in a transmission electron microscope also and this aspect I have already covered, first we will talk about the electron tomography, what is electron tomography?

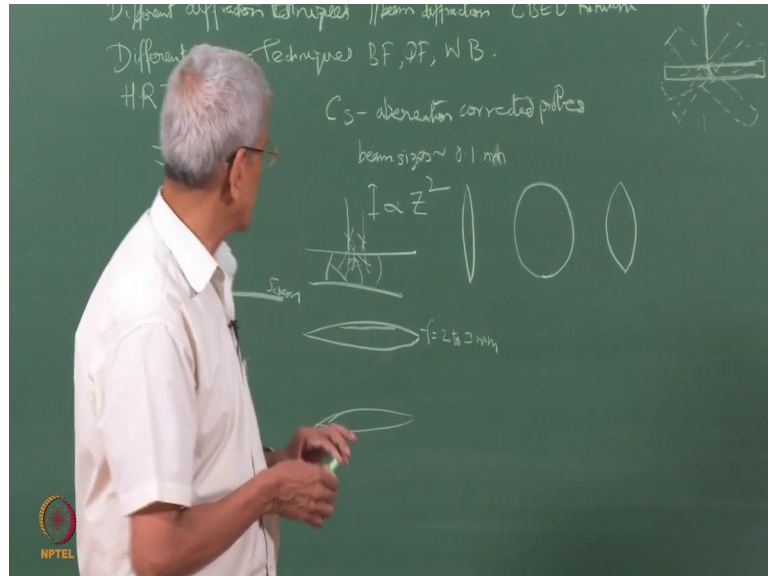
(Refer Slide Time: 09:04)



As I mentioned it is we can get 3 (Refer Time: 09:13) dimensional information about as sampled, how exactly it is implemented in an electron microscope as I mentioned earlier, in an electron microscope we get at 2 dimensional projection of the 3 dimensional

information. So, we do not get correct information about the size of the objects for example, suppose we have a ellipsoidal precipitate is there.

(Refer Slide Time: 09:40)



So, if you looked at this cross section in this direction it looks like an ellipse.

If I look at this precipitate from this direction this will essentially look like a circle, depending upon which direction I look at that sample I get different information in between directions, if I look at it this will be essentially the size will be looking like this.

So, if we take one picture and we get a size if it is like a circle or if it like an ellipse we may not be able to talk anything about what is actually if the shape of that sample, this information is really necessary when we wanted to find out the interaction of precipitates with dislocations and the separation between the particles. All this information 3 dimensional information about the size distribution, size shape as well as the distribution is very much necessary, to get this information what is done in an electron microscope is essentially we have a sample this sample is put on holder nowadays they call it is a tomographic holders, special holders are available are essentially what it is you keep a sample like this make the beam pass through that sample then tiled the sample gradually over different orientation like in both this directions making sure that the beam falls exactly at that same area and collect all the images which are formed for different tills.

What is essentially going to happen is that depending upon if a precipitate is here, depending upon the tilting of that sample that precipitate also will change its orientation with respect to incident beam. So, that the shape of the particle which we are getting it, as well as the contrast also will vary both these information which we have to use that is using image processing technique we can stitch all these images together and construct a 3 dimensional image of that sample this is a lot of computational work which is essential in image processing using this we can get information about how this particle looks like. So, that otherwise looking at one image we will be getting an information about the particles whose morphology is not correct.

So, this can be this sort of work can be done in both it bright field as well as in dark field, doing it on an optical scale with a light scattering it is easy whereas, in an electron microscopy you know that especially we are dealing with diffraction contrast depending upon the different orientations and if we tilt slightly the contrast varies, considerably. So, one has to take a lot of care to see to it that the same diffraction spot is used, you respect here what the tilt which we have used to collect that image, where exactly this becomes it is a necessary is that when you deal with lot of big macromolecules when we are looking at it what is the shape of this molecules that if you wanted to find out there this electron tomography helps a lot. This is own technique which is being which is extremely, which is becoming extremely popular and it is being used also. The other technique which is being used very frequently is Lorentz microscopy, what is Lorentz microscopy? The term comes from that Lorentz force that is if a magnetic field is applied we know that magnetic materials they feel a force that force is called as the Lorentz force that is why it is called. We know that when we with a conventional electron microscope when we wanted to look at that sample, the lenses which we use especially the objective lens ok.

All the lenses are electromagnetic lenses, because the lenses are electromagnetic lenses if the sample is conducting, but it is non magnetic then the magnetic field does not affect any of the contrast from the sampled, because that magnetic field is essentially used to make the beam pass through that the beam which is passing through that sampled which get scattered in different directions they are being focused to a point and this acts a like a simple lens, what is essentially going to happen when the sample itself is magnetic is that as the electron beam enters into the sample the magnetic field can vary locally at different points that sample suppose, different domains are there magnetic domains are

there depending upon the domains the mag electron can interact with the magnetic field, a Lorentz force we will act on it and it will just shift the beam in different directions.

This sort of shift what is going to happen is that the team which has already been aligned, now the whole alignment as go sorry this is one of the problem with magnetic materials. So, looking at the magnetic materials and getting microstructure information about it is extremely difficult, but it can still be done if we use very small mass of the magnetic material and some precautions have to be taken that the sample is sandwich between 2 grids are it is groove to a grid. So, that it does not fly of otherwise it creates a lot of problem in their microscope, but the same magnetic behavior of the sample itself could be used to get information about the distribution of the domains in that sample, this microscopy is called as the Lorentz microscopy. In this what is being done is that it normally the objective lens which we use they give a quite high magnification about 50 times, and most of the time if you there sample is immersed within that for the lunch which we use if you look at the focal length it is about 2 to 3 millimeter and then the sample is almost kept within that, it is which is call that it is immersed within that field itself.

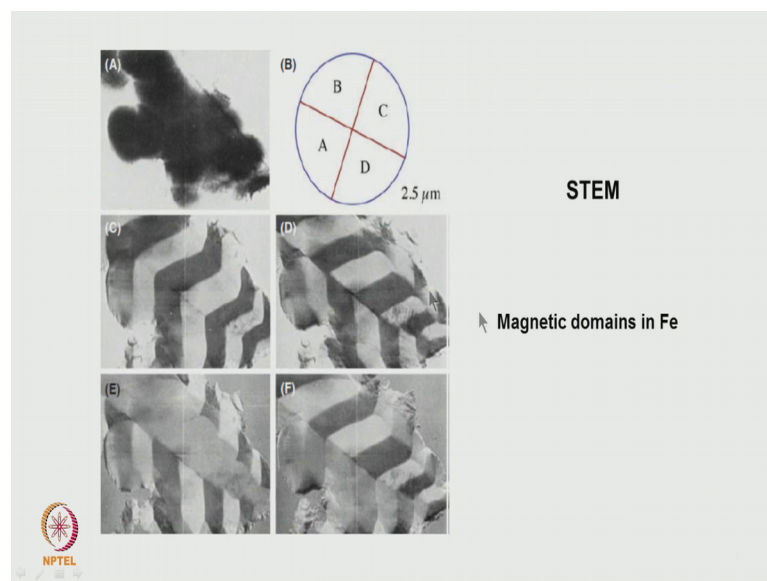
So, the strong magnetic field it sees a so, if you wanted to use the Lorentz there is a magnetic property and to get information about the distribution of the magnetic domains, one thing which one can do is essentially use a low magnification and use a lens who strength is very small or in a microscope what is done is there that in something the objective lens itself can be switched off and the intermediate lens which is far away there can be used to form the image a low magnification image by using that, the effect of the local magnetic field how there is, there is how this objective lens magnetic field is going to affect that optical alignment that can be reduced considerably, but apart from that what is it which is going to happen with respective a sample, of the electron beam enters into the sample depending upon the direction of the magnetic field the electron will be just scattered in slightly different directions, that is the path of their electron will be changed because of this suppose we try to get a diffraction pattern for that sample.

Normally in a non magnetic material we will be getting one spot because that electron path is affected by the Lorentz force generated by this magnetic field depending, upon which directions the fields are there the single diffraction spot we will split into multiple spots, that is what essentially is being shown in this case there is 1 2 3 4 spots are there

and if you put apertures around all these spots together and try to form an image then only the magnetic domain boundary becomes bright and the other regions are, this way we can identify where the domain boundary is or we can put an objective aperture and try it to choose only each of these spots and try to generate images. When we do this images essentially give the distribution of the magnetic domains this is in the case of a nickel (Refer Time: 19:59 so this images are called as Foucault images).

Generally the direction of magnetization is normal to the direction of the spot splitting in the diffraction pattern, what all the uses this can be used to find out the size looking at these sort of micrographs, we can find out the domain size information we can get it, but from these images we cannot get any information about the magnetic field itself that is magnetic induction that information cannot be (Refer Time: 20:40) no quantitative estimation can be done from Lorentz microscope, there is what we talked about is in a conventional transmission electron microscope.

(Refer Slide Time: 21:02)

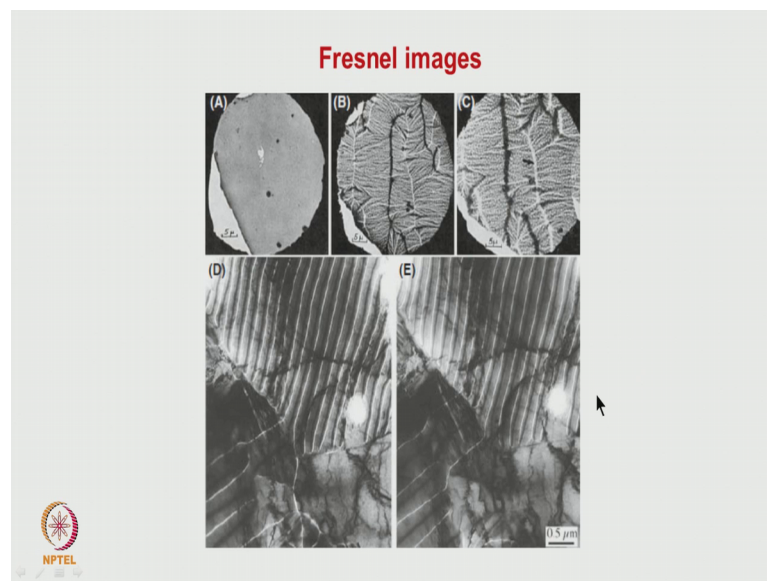


In a scanning transmission electron microscope also we can make the beam scan on that sample surface depending upon the magnetic, the direction of the magnetic field in different domains the electrons will be electron path will be changed accordingly ok.

So, they will be coming in different directions when they come out of that sample surface, the detector which is used below in an STEM either we can use a single detector to collect or we can split the detector into few quadrants, each quadrant can collect that

electrons which has been scattered in different directions. If this information which we collect it we can use each one of them to form an image we know the electrons which are scattered in each direction from that we can get how the magnetic domains are distributed we can add subtract information from one quadrant to the other. So, that we can manipulate their image contrast and I get information about the distribution of that magnetic domains.

(Refer Slide Time: 22:12)

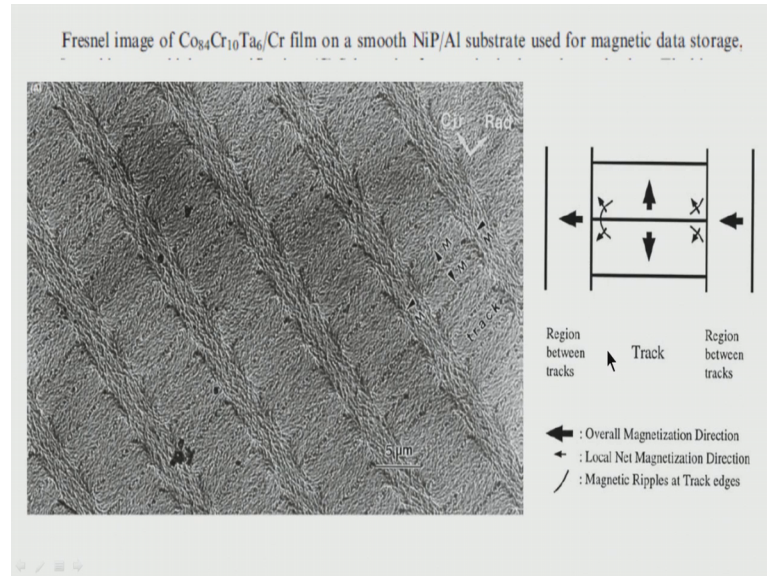


This is an example which is for magnetic domains in iron these sorts of images are called us Foucault images ok.

There is an another way in which we can get a get information about magnetic domains this is using Fresnel image, these are called as Fresnel images. What is essentially done is that we know that if we change the focus a little bit they are want the sample which contains the hole we know that around the hole (Refer Time: 22:41) we get some fringe pattern, alternate bright and dark fringe patterns which are getting it. These fringe pattern comes from Fresnel diffraction which is taking place because from one side to another side, there is a potential variation is there this information itself could be used and we know that in a conventional microscope when it is in perfect focus the contrast of the images a beam, this is an example of a sampled where you can see that where it is in a perfect focus we do not see any image, but when the sample is magnetic especially magnetic domains are there from one side to other side if we go the magnetic potential

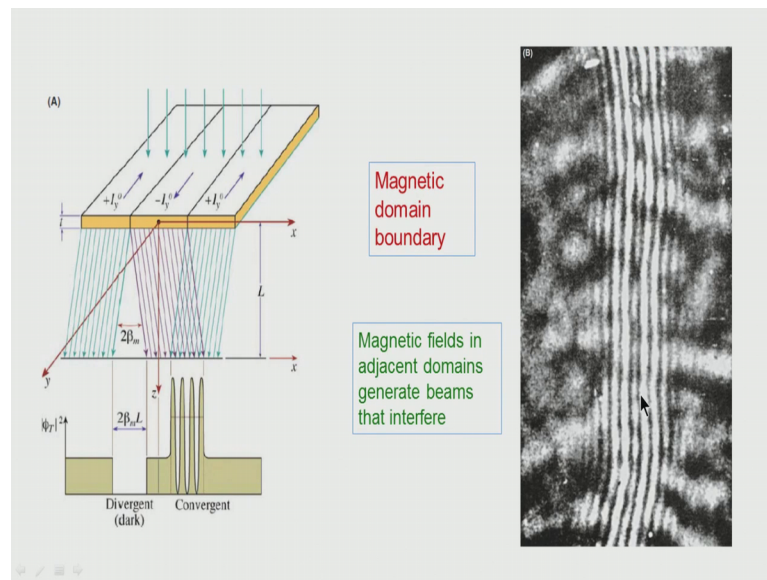
there is going to be a sudden change is going to be there, because of which if you defocus a little bit we will be getting a bright and the dark contrast ok

(Refer Slide Time: 23:43)

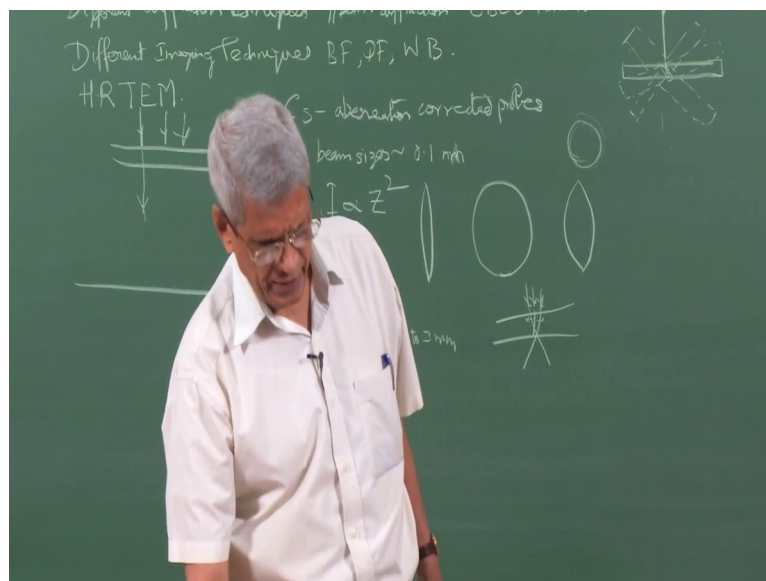


This contrast itself could be used to identify the magnetic domains. That is exactly what is being done in this particular case. In fact, the same final images could be used to obtain distribution of the domains etcetera, this is one such example which is taken from the literature this is on a magnetic data storage device where how on the tip the magnetic particles are distributed and not only that these are also magnetized some data is being stored because of that when it is a slight defocus condition when we get it because of the Fresnel kx diffraction we are able to see a contrast of this domain.

(Refer Slide Time: 24:29)



(Refer Slide Time: 24:42)

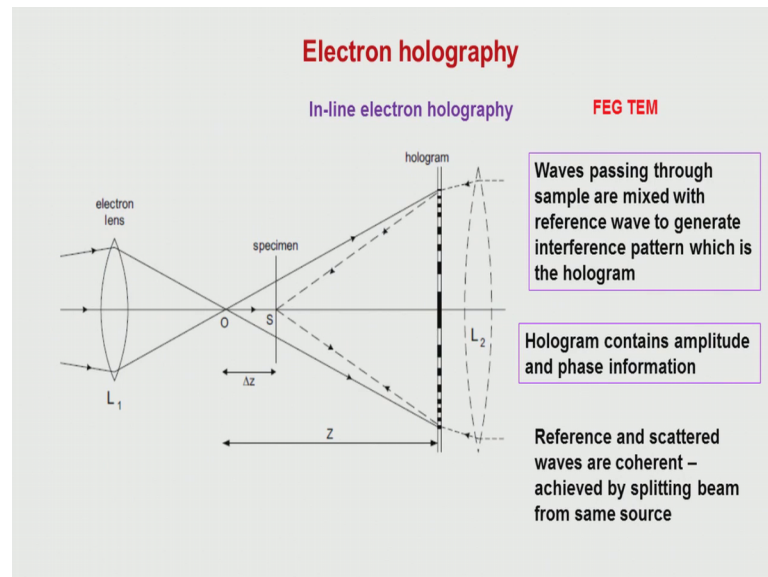


Then another way in which a magnetic domain boundary can be get information about it this is that, Suppose we cancel this is the sampled across these boundary that magnetic domains are oriented in 2 different directions because and when that electron beam is coming like this.

These orientations can make the beam in one direction go like this and the another direction shift the beam like this and these beams can also interfere and this will give raise to an in difference with scattered this is the bi prism Fresnel bi prism and with

which also the domain boundary could be image, this is just an in difference contrast. So, there are all the various ways in which we can get information about the magnetic domains.

(Refer Slide Time: 25:26)



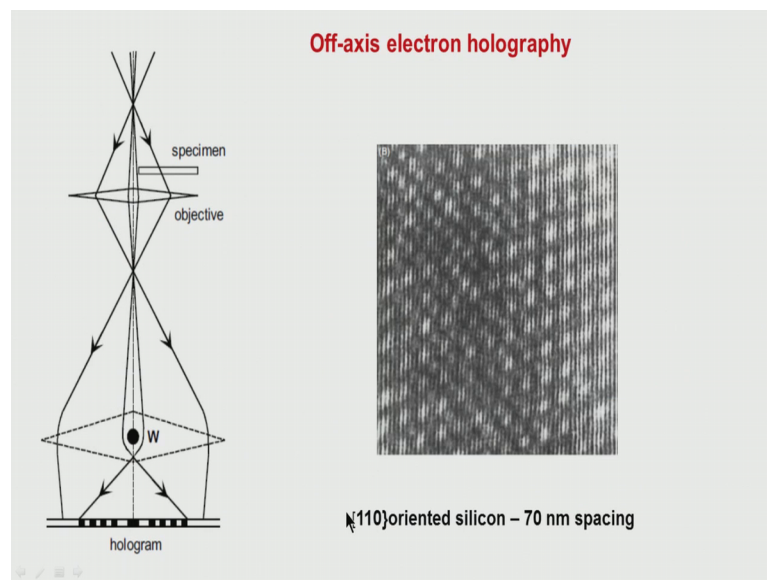
Now, what we will do it is that other techniques we have already covered. So, now, we will talk about electron holography I mentioned that electron holography is one of very important technique, because using this technique we can get information about the phase as well as the amplitude of that sample.

How exactly it is electron holography is done, there are many versions of it is called as an in line electron holography half axis electron holography there are many types are there, this is essentially a schematic diagram of an in line electron holography . So, what is holography essentially this was discovered by Grabber wave back in fifties, essentially if a coherent beam passes through a sample that is a beam be split into 2, 1 part passes through that sample and the amplitude of the wave which comes out with their some phase changes we will be occurring in that sample and if you mix with that reference wave when these 2 beams are coherent beams, they will give rise to a sort of an interference fringe contrast ok.

This interference fringe contrast contains information about the phase as well as amplitude of that sample. If we use either that same beam or an another beam we allow it to pass through the image as well as mix it with the reference beam, we will be able to

get information about the that is we will be able to form from the hologram the image of that sample and that image is essentially form because of the phase information which is available. The same thing can be done now if you have an, a hologram which is there we can use computers to do all these job get all the information can be obtained. So, what is essentially done if that this is a electron lens which focuses the sample, which focuses the electron beam right in front of that sampled and the beam is passing through and some of the electrons are passing through the sample which is scattered from here and this is the one which is assuming that is reference beam. These 2 interfere together and then gives rise to a contrast this is the contrast which has information about the phase as well as the amplitude.

(Refer Slide Time: 28:20)

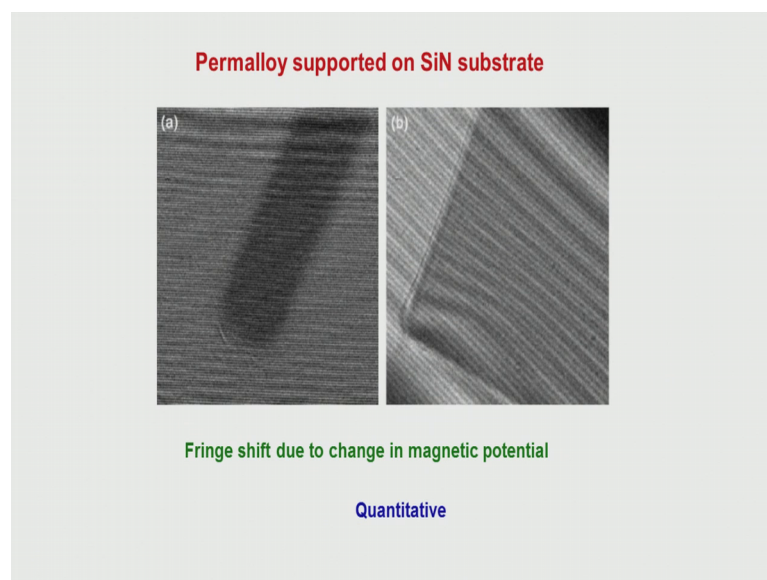


So for we talked about essentially the holography which is in line holography, off line holography is essentially which is very similar to the Fresnel bi prism which be explained in one of the earlier classes. Here what is being done is that this is the specimen through which the part of the electronic beam passes through and this is the reference beam and the all beam in passes through here we have a Fresnel bi prism is there which is splitting the. So, what is Fresnel bi prism here what we do essentially, this is like a small glass coated with the conducting material and to which if you apply a voltage this will be reflect, scattering the electrons in different directions and the this is the direct beam which interferes with the beam which is scattered through the sampled and this will give a holographic image which it will be creating. This holo this is 1 such

holographic image which is obtained from a silicon is shown 1 1 0 oriented silicon is being zone.

But here if you look at it there are 2 types of fringes which we can see, there is 1 fringe contrast which is larger spacing and another is a fringe contrast which is a fine spacing, the fine spacing own corresponds to the conventional of high resolution microscopy and the larger on which appears they have about 70 nanometer spacing these are all the ones which can be used to get information about the phase as well as the amplitude distribution in the sample, that is are in effect we can give raise get the structural information about that sampled from this hologram.

(Refer Slide Time: 30:17)

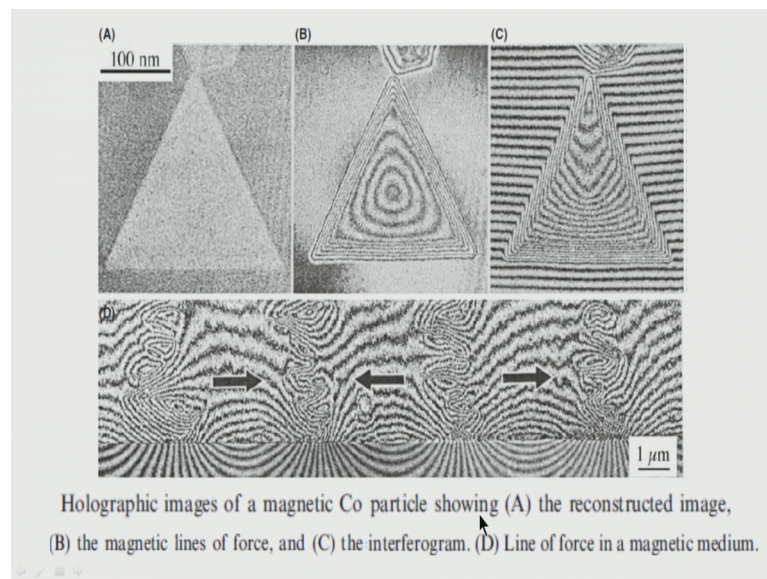


Another important thing which you should understand here is that, in this particular case the sample is not a magnetic material it could be any sample. So, the information which we give it is how the interaction with the electrostatic potential in that sample the beam interacts that is mixed with a reference beam and that is how we generate hologram.

If the sample is a magnetic material then what is essentially going to happen and this is a particular case where a perm alloy is simply supported on a silicon nitrate substrate. Here again we can see that the holographic image, there is some bending of this are bands are there these fringe shifts are taking place because of the magnetic nature of the material or the how the magnetic domains are distributed, what is important which one should realize is that the Lorentz microscopy we used to get information about that distribution

of magnetic domains, in that case I mention that we cannot get any quantitative information about the magnetic field. Whereas, in this particular case we can get information about how the domains are distributed, not only about how the domains are distributed in that sample we can also quantitatively get information about the magnetic induction in that sample, that is a great advantage of using the essentially we can say that the magnetic potential variation in that sample that information could be obtained using the electron holography.

(Refer Slide Time: 32:09)



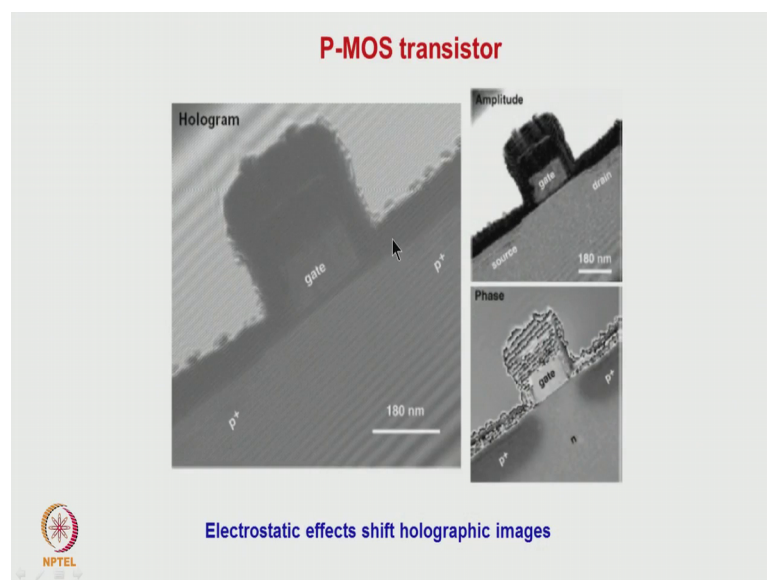
This is essentially taken from the literature essentially a holographic image of the magnetic cobalt particle, this is a reconstructed image of that particle which we see this is how we it looks like, in a conventional microscopy field look at it we do not get much information, but if we use a holographic electron holography technique which is attached to a microscope then we can get information about how the magnetic field lines are distributed in and around that sample and see if the end of from image which we get it of the sa sampled and this is for a single particle and here essentially is in a magnetic medium, how the magnetic field the the domains are oriented, how the magnetic walls are oriented all this information which we can get it.

Similarly, suppose we take a superconducting magnet thin field superconducting magnets, we wanted to know how the field aligns penetrate through that sampled as we fringe the temperature why this experiments we could get and the information about how

the how much of the magnetic induction is getting passing through that sample. penetrating there superconducting sample this information could be obtained using this technique that is we can directly image and quantify it also this is one of the greatest advantages of using electron holography, but the microscope to use this technique the first thing which we require is that, the wavelength of the radiation should be some value that is for the microscope, but what is essentially important is that the energies spread should be as little as possible then only we can get reliable interference pattern that is; that means, that field emission gun with filter if we use it, we can reduce that effect of the chromatic aberration and that microscope path difference of the electron is changed into a phase changes or the phase change is converted into the path change ok.

In all these cases the beam has to be as monochromatic as possible for all this experimental work to be done. So, to ensure the monochromaticity that we have to make the line with as small as possible the energy spread of the beam can be reduced only if you use field emission and that you if we use a filter the energy spread as I mentioned the earlier the last class it could be made as small as about some 0.2 electron volt, this is an another example where it is a hologram of a p mos transistor which has been taken.

(Refer Slide Time: 35:32)

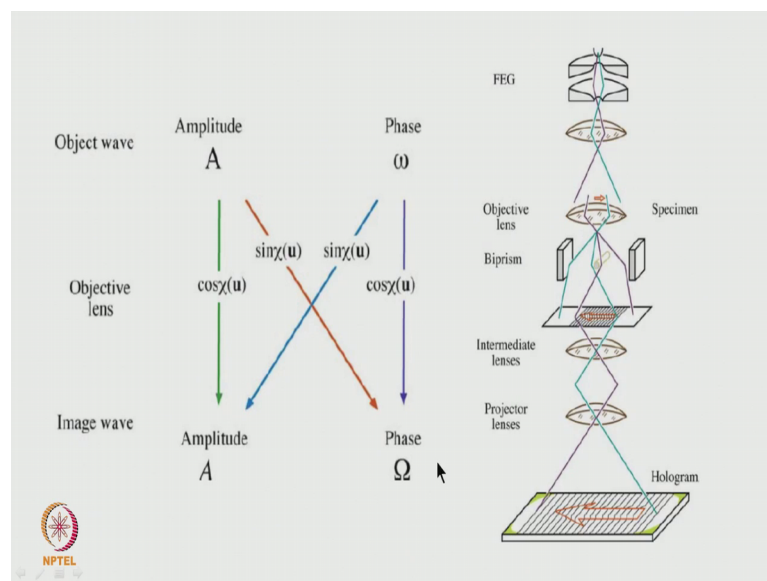


This is the holographic image, then how do we get an information from the hologram because we use a reference beam though this hologram has been generated using an electron microscope to reconstruct the sample information we can use any beam it could

be a laser beam which we could use it or we can use a computer to use some mathematical processing to get this images.

So, here if you see it this is what it is a p plus region this is the gate and this image as I mentioned that we can get information about the amplitude, that is that amplitude information about what is the mass which is elements which are remaining all those information which we can the amplitude information of the phase and this is the phase information also of the phases we could get, this darker region shows that that phase is quite different compared to that of the same region and this also shows that there are some variations in their phase and this a periodic variations of the phase is taking place. All this information's we can get in (Refer Time: 36:47) that is not only for magnetic material the magnetic potentials the electrostatic effects also bring about changes in the holographic images that gets inform a that gives information about the electrostatic potentials that information also could be obtained using electronic hologram.

(Refer Slide Time: 37:11)



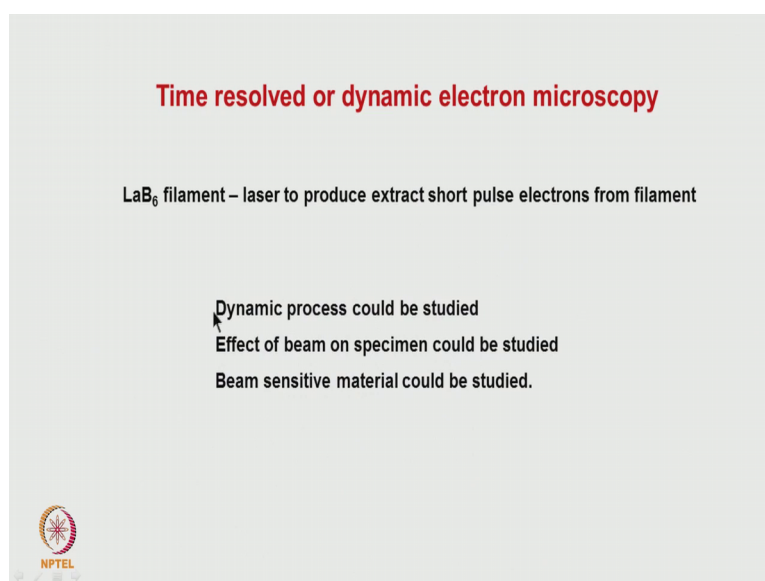
Though in principle when we talk about it this looks like, like a very simple technique, but exciting information requires lot of deep understanding of the physics behind this processes lot of detail software. So, I have been returned I can should understand what all phenomena's which look at to get that information, but what is the advantage essentially is that from one micrograph which has been generator one hologram we can get complete information about the phase and that temperature. Whereas, in a high

resolution microscopy as I mentioned to get correct structure information we may have to make the contrast matching for at least a few images which are taken for different defocus conditions ok.

Now, I will talk about the other technique the time resolved a dynamic electron microscopy, the problem in a conventional electron microscope is that since the electron beam is falling on to the samples of us, if it is a metallic sampled and if the beam incorrectly is relatively small the beam passes through it and without making, without generating much damage within that sample. Whereas, in the case of biological samples when that electron beam passes through that samples if it is focused then they can damage the sample during observation itself the time which it takes for this dimension take place could be so small that before we complete the, the examination of that sampled the sampled might have been completely damaged.

So, this is a very serious problem in many microscopes.

(Refer Slide Time: 39:01)



So, how this could be studied, that is that which was thought is that if we make the electron beam pulse the electron beam if you use it we make the beam pulse very small pulses are being made this pulses are some few nanosecond separation between them and femtosecond the duration, this pulses are generated using an LaB₆ filament, but in a normal microscopy LaB₆ is heated and the electrons are generated instead in this one what is being done it is a laser pulse is allowed to fall and there LaB₆, some ultra violet

laser radiation and that generates photoelectrons since this photoelectrons are coming in out this (Refer Time: 39:53) narrows spread in energy and short pulses they come out of that (Refer Time: 39:57) then they are accelerated to high energy and there allow to pass through the sample .

So, all the information about the interaction of the beam with the sampled for each of the pulse is collected. So, this way we can get information suppose some chemical process is going on we wanted to find out how actually the one phase is forming by an interaction, there is in some gas shall we can do it some gas reactions of that. So, how the chemical reaction takes place to form a phase all this information could be studied in situ that is why this is called the say time we solved electron microscopy and then beam sensitive material where this here we can controlled. So, that since short pulses we are using it we can get information about the sample from each of the pulses and in all these cases what has to be done is that we do not photograph that image essentially that the electron beam may is pass it through the sampled, it is collected using a slow scan CCD camera and then this has to be that digital data has to be process to get information about how the changes are occurring within this sample ok.

So, it is a very important technique with which since that femtosecond, nanosecond duration pulses with femtosecond pulse with which we are using it. So, this is of the order of dimensions where the atom motion takes place within that sampled because of which in many areas like catalysis or in chemical reactions, how atoms move from one side to another, how that interaction takes place all this information could be studied this is a very powerful technique and the this technique has started coming a.

These are all the trends which I wanted to talk about, but what is essentially important is that it is always come out because people have seen some limitations when you start using the microscope you see what you can get from the sample, one has to use an imagination to decide how to conduct an experiment. So, that some changes may have to be made to a microscope or some small special holders or set up has to be made. So, that the type of experiment which you wanted to get, to do, to get the information which you are looking for that could be performed.

So, the old microscopy field for that matter I can either field, is an evolving field a lot of new, new ideas are being used to develop new types of microscopes. To get best use of

that all this equipment you want has to apply one's own mind. So, essentially the equipment can give some raw data how to analyze and interpret is how we look at the data which you have got and how determine we are to get the best out of it that is way we have to get that information. So, essentially in this lecture I have talked about the various techniques which are available, that is the new modern trends which I have thought about it. May be in future you find that many more new techniques, new powerful techniques will come up in the electron microscopy. I will stop here.

Thank you.