Electron Diffraction and Imaging

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Lecture – 22
Tutorial – 04
Correlation of Diffraction Spots to Microstructure

Welcome you all to this course on electron diffraction and imaging. In the last class we mentioned about the basic structure of the electron microscope how to get and bright field dark field and diffraction pattern. Today what will talk about is those patterns how we can use to correlate microstructure with diffraction pattern.

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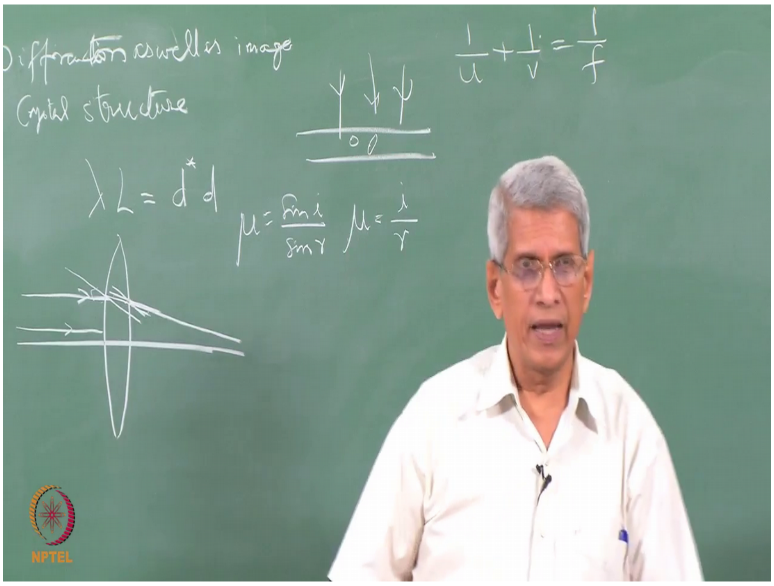

The greatest advantage of a micro electron microscope is that, we can get diffraction as well as image from the same region of the sample which we can obtain, that information could be used to correlate microstructure to the phase information how the different phases are oriented in the matrix, how to go about I will explain in a very brief way, but this takes a lot of time and one allot to gain a lot of experience.

So, what all if information which we can obtain or what all the applications one we can get information about the crystal structure, that is one information which we can get it from a microscope by analyzing the diffraction pattern which are taken in different orientations. Then another great advantage is that in a transmission electron microscope since electron beam we are using it we can focus it to a that beam to a very small area and then get information from even in a poly crystalline material from one single grain that is a single crystal information we can obtain.

The next is if there are different phases are there which has formed during solid state transformation, we wanted to find out how these phases are oriented in the matrix that information can be obtained that is what we called as orientation relationship between phases.

As such using the crystal structure we can identify using diffraction pattern suppose different phases are there, in the diffraction pattern taken from a region like suppose a sample we this is that sample and this the area that we miss falling suppose in this area two types of I am just showing with two different morphologies. But this could be two different phases which are present in that region then we can identify what all the phases which are there because the diffraction pattern from that area contains diffraction from the matrix diffraction from these particular phase diffraction from these phase. If you analyze this diffraction pattern then we get all the information about the phases and how this patterns are oriented with respect to other that gives information about the orientation relationship.

Then suppose in many cases not only it need not be a different phase, some case especially when non cubic phases form like an tetragonal phase forms in a FCC matrix then it can form with it is c axis along any one of the a axis or a b axis or a c axis of the cubic matrix; that means, the same phase, but in three orientations that will that can also be identified using diffraction pattern and combined use of diffraction and imaging.

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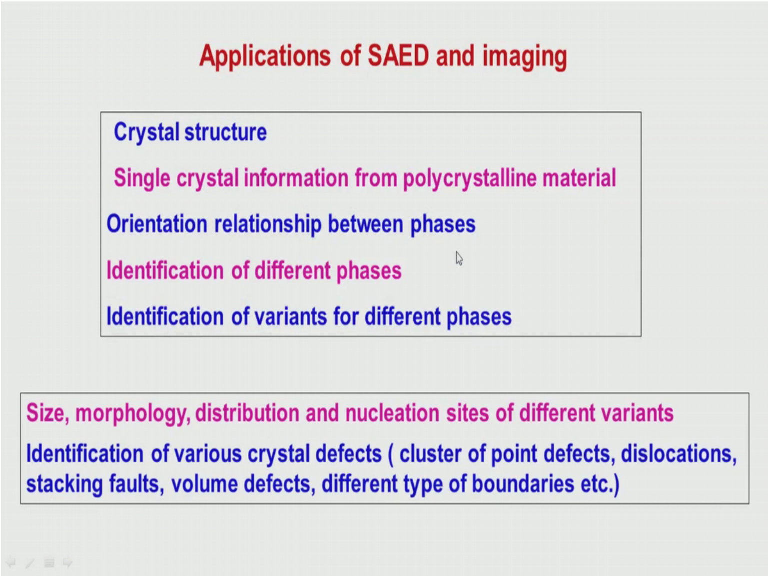

In addition to it in the image side what all information which we can get what is the size of the particles second phase particles which form, they are morphology, how they are distributed whether it is uniform and another is that whether what all the sites that which they can say particles are nucleated or whether they are formed at the grain boundary preferentially or whether they are formed at some sub boundaries or whether they have uniformly distributed all these sort of information which we can get.

In addition to it if defects are present in the material, we can get information about the various types of defects which are present in the sample, all these aspects about the finding out the size distribution the identification of the crystal especially the identification of the crystal defects that will be dealt with separately in detail in a another class. In today's class we are only going to talk about getting a selected area diffraction and how this simple bright field.

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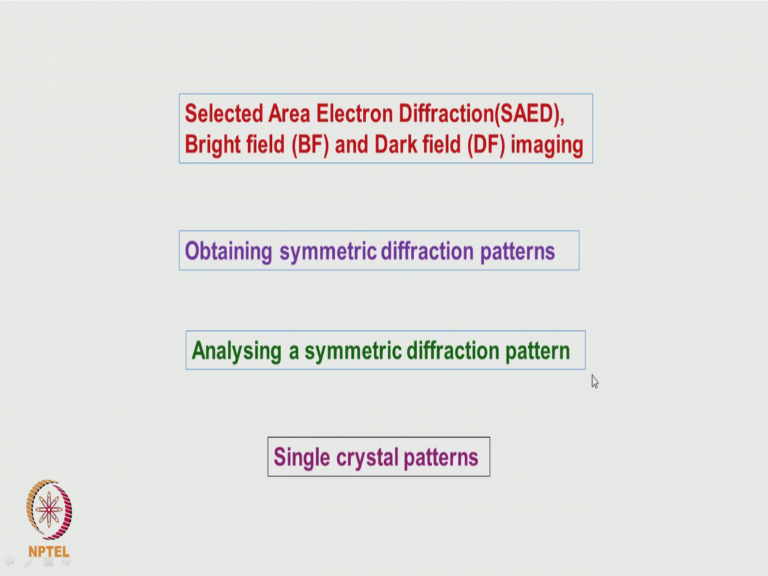

And dark field technique which we have mentioned in the last class that itself can give lot of information about the second phase particles most of the work essentially bulk of the microscopy work is done using these three methods of imaging.

What is the first in step which is we have to follow is, one obtain symmetric diffraction pattern from the sample that is the first step in doing analysis that is once we have a sample in the microstructure that beam is there, the sample can be tilted if a sample is there like this the sample can be tilted in different orientations whichever orientation we want it, and then get a symmetric pattern. Once we have got a symmetric pattern we should be able to analyze this pattern that is an another part of it which comes what I will talk about today is only with respect to new single crystal patterns, I will not go into anything else other than that.

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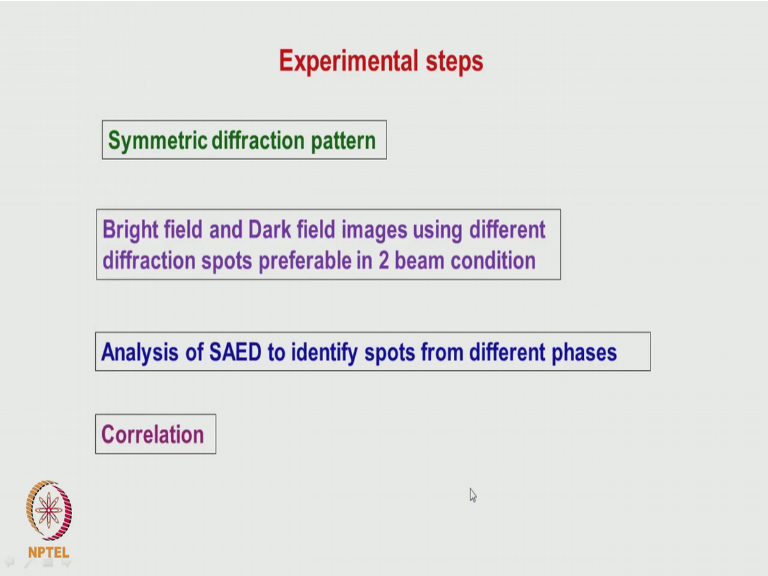

The experimental step is that first we obtain a diffraction pattern, once you have got a symmetric diffraction pattern then next step which has to be followed is that if you contain if that the diffraction pattern contains different spots, use all the spots to get images of the sample using the undiffracted spot which is the transmitted spot which we call it as a bright field image, and using all other reflections which are there also try to get the dark field the dark field images.

Quiet often what is happens is that you may take some few patterns afterwards you realize that I should have imaged it with the particular diffractions part which is important. Because if you have a lot of experience then when you look at sample in the microscope you understand what is necessary when you do not know when you are a beginner it is better to take that information. So, you do not lose anything, you can do an analysis later may be you might have got taken more patterns what is necessary that is what you think that never happens. From my experience I can tell you that take as much pattern as possible, and that helps really in the analysis in confirming a lot of hypothesis with which you start.

The next step is that you analyze diffraction pattern to identify the spots; because from a single phase we get a pattern it is essentially a two dimensional periodic patterns which we get it, as a reciprocal lattice section which we are getting it, is easy to analyze it and find out which crystal structure, but when we have patterns from other phases first thing which we have to then pattern looks like random distribution of lot of spots, first thing which we have to identify is that what all the types of periodicity periodic type of two dimensional lattices which are present there. Once we identify that is the first step in an analysis of the diffraction pattern; then once that has been done now it has to be correlated to each type of patterns from which phase which it has to come that is the next step and there are many softwares are available with which we can do these analysis.

But I am not going to talk about the software because first one should learn how to do it. So, that whether the software does it because software like a black box, whether it gives the correct information or not one should be able to cross check.

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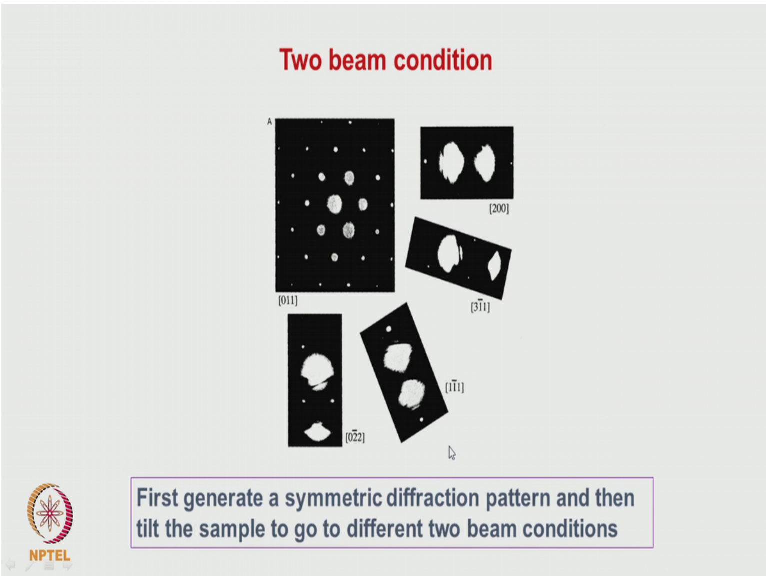

So, I mentioned about the first step is to get a symmetric diffraction pattern, this symmetric diffraction pattern we can get it in a microscope by tilting that sample observing it on that screen and. So, that we get spots which are of uniform intensity how to get this pattern using kukuchi pattern all these things are being covered in a different class we assume that we have for this lecture we assume that we have got a symmetric diffraction pattern ok.

Now, once this pattern has been obtained you can tilt the beam a little bit so that only two spots are essentially strong this is called as two beam condition. So, if you make two beam two spots only strong that is the central spot as well as the one of the diffracted spots using these central spots now you try to image take a bright field and using this diffraction spots you take the dark field image. Both images are necessary then you take with respect to may be instead of taking them you take with respect to these two, then also take a bright field under that you will think that what is it going to make because when you tilt as sample little bit the contrast can change drastically. As we have discussed in the earlier class the diffraction contrast strongly depends upon the orientation of the sample with respect to a beam not only that even if it is a slight tilt is taking place the contrast can change drastically. Because we know that that exact bragg condition maximum peak intensity, if you tilt a little bit drops that will be reflected in the image also.

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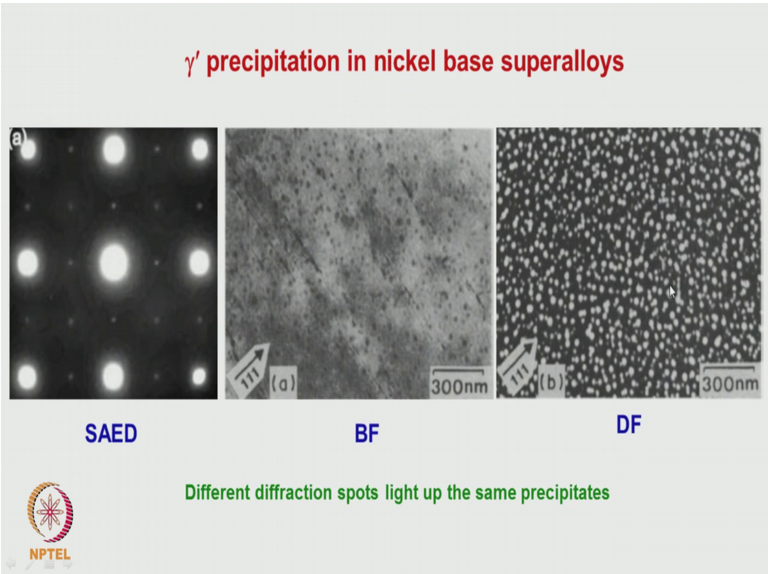

So, like this you can go two different two beam conditions and take images ok.

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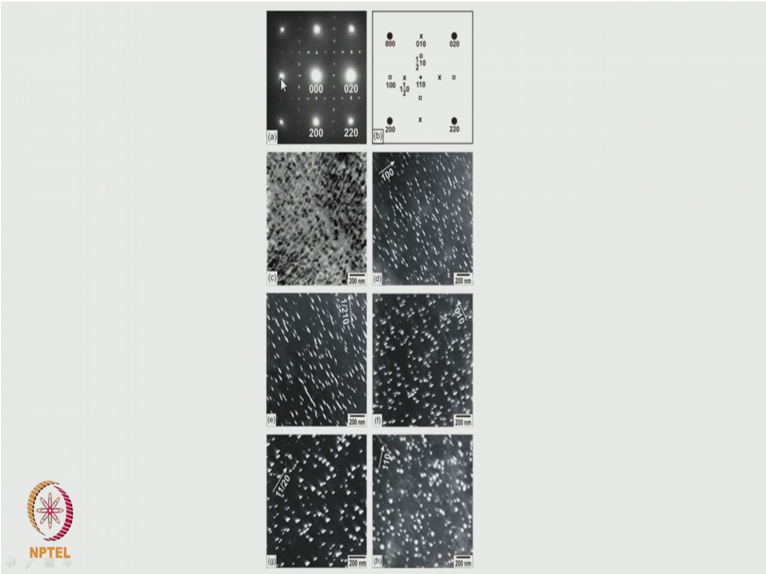

So, what I am showing it here is just an example of a diffraction pattern which has been taken. Why I am showing this picture is essentially to know that in this you forget about these pictures you just concentrate on this diffraction pattern, this diffraction pattern contains lot of spots some spots are very strong some spots are weak. What I have done it is go on to two beam condition put an aperture around the central spot and imaged it that is where I get this bright field picture.

Then what I have done is I have taken with respect to these reflection put an aperture around it and imaged it in the dark field I get some ellipse idol shape second phase particles could be seen. Then I put an aperture around this one and try to image it that is what this 0 1 0, yeah this is first it is taken with this 1 1 0 0 this what it has come then with respect to 0 1 0 I image it this reflection has come. Then I put around these particular one or you can say that equivalent to this one, then I get one of this images then with respect to an another one like this I get this image then I put this around this central one I get this image this sort of images which had gone.

But now if I look at these one information becomes very obvious from this, if I take it with respect to this 1 0 0 as well as with respect to one half 1 0, half 1 0 essentially comes like this half 1 0 this is half 1 0 this reflection. This reflection and this reflection if I take it is essentially the same particles which are there. So, both of them belong to those two spots are there they are coming from different spots from the same diffraction pattern from the same phase that is what you can identify; that is why I mention that you using all the spots take images otherwise you never get the actual information which were looking for. Wealth of information is covered, but you should not be stingy in taking micrographs liberally and similarly with this central one if I take it I can see it here with respect to this part there is 0 1 0 as well as this 1 1 0 0 as well as the one half 0 if I take it, you can see that here again it is that same type.

So, this is that information which comes out of it you understand that that is why you mention that you have to take spot diffraction from whatever the spots which you can see closest to this, similarly you can take with respect to this fundamental reflection because when we look at it obviously, this periodicity is very clear. So, we can take with respect to the this reflection this reflection and this reflection if we take it all other reflections are essentially a comes from the periodicity of the lattice.

So, essentially we have covered most of the reflection. So, this way we will have complete information about the various phase and the structure which we have got. I will come back to this again because this has more information at this stage I do not want to talk about it. This is an another example that is a much more complicated one why I took that picture is to tell that from two different spots if you image quiet often you find that that the same precipitates guts.

So, you do not know whether each the particular super lattice reflection which you see is belonging to the matri the one particular phase or an another phase that also with an example I will come. Here it is a gamma frame precipitation a nickel base super alloy if you look at these diffraction pattern you have a strong spots plus some weak spots are also there, then this is the bright field picture which is taken with respect to putting an aperture around it when I put an aperture around this reflection and try to image it I get a dark field picture. In this one I am not showing all the pictures you believe me when I put an aperture around this reflection or around this reflection, I find that the same precipitates is getting image; that means, that all this spots coming from the same the second phase.

Now, the next question comes is that this is logically we have argued out now how to identify how to index this diffraction pattern that is what we will talk about it. You see this pattern this pattern, you can see this parts first thing which I can see it is that with respect to a brights parts I can see a periodic lattice there then I had talked about to how to index a diffraction pattern I had mentioned that by measuring this because most of the time in an electron microscopy we come with an information about the type of phases which are present and we use microscope to find out how this phases are distributed because x ray diffraction does not give any information about it, x ray diffraction tells these are all the phases which are present.

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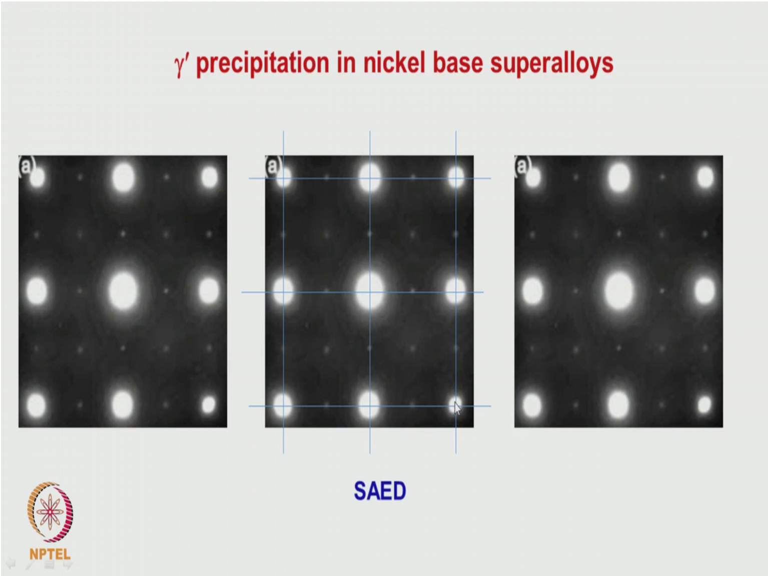

So, we have some idea about it, but we wanted to find out that how these phase are distributed can we identify that from an electron micro from an electron microscope.

So, now if you look here this pattern we can identify them, this is a periodic pattern and that is what I had shown with respect to putting a grid around it. This grid what essentially it tells it is a two dimensional grid by measuring these distance measuring these distance and measuring these distance.

Using this formula which I had mentioned earlier lambda into l equals d star into d where d is the spacing in the real lattice and d is the spacing which we are seeing for the different g vectors that is equal lambda is equal to wavelength of the radiation, l is the camera length which we call it as a camera constant. Using this formula we can find out since we know the crystal structure we can find out what the spots corresponds to each of the spot.

Then I also I mentioned that using stereographic projection you can index them without any ambiguity, all the spots could be indexed correctly. Once such an indexing has been done for this we have to use this sort of a trying to identify first how the periodicity is there in the lattice that is the first term there is this is one which is periodic then there are other spots are there, what is the periodicity which is associated with that. So, for that if I look for I can make out that this one, this one, this one, this forms as smaller bit this is what essentially ok.

Now, you can make out that these parts are all forms another periodic lattice. So, essentially we have two types of two dimensional periodic lattices are there. So, it could mean that one this pattern could be a super lattice reflection corresponding to the same phase or because the two choices now arises. The second is that it could be a entirely a different phase also in this particular case it is essentially a gamma frame precipitates since it has been analyzed I can tell it, but otherwise this is with that choice you have to go these are all the two possibilities now we have to find out which is what it is this is where imaging helps simultaneous taking of the image is important because by aperture around it, we are able to identify what is the type of whether it is a second phase particle is present or not all this information suppose it is ordered single crystal second phase one will the same area will get image, correct.

So, that we can get information by not on just not by taking diffraction pattern, by taking dark field images; but by just taking images you cannot get any information you have to take diffraction pattern corresponding diffraction pattern forms the basis of taking images not the other way round is it clear. So, now, we can identify two phases and in these as I showed in the previous slide like here. So, we can find out the distribution of the phase one particular precipitate is also there, this the crystal structure we have some information from a x r d and all with which we can analyze it and tell that. So, we know how the disti how were they are distributed, they are distributed uniformly in the matrix all this information which we have.

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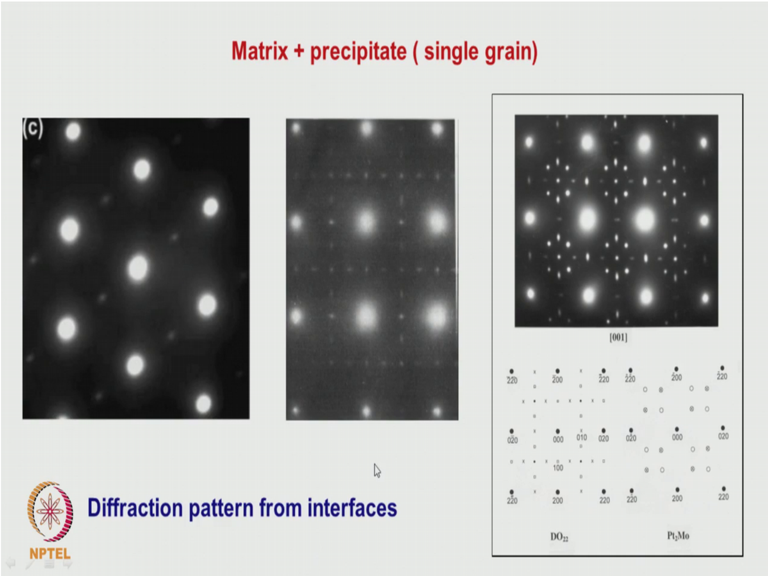

This is another pattern from that same sample, this you can do as an exercise and try to index it and find out this is one of the simplest one where only a single phase is there, now will come back again to the diffraction pattern which I had shown it is a little bit more complex. How we had gone about to analyze it? When you look from a visual observation we can make out that this bright spots all form a periodic lattice one should always remember that how many two dimensional periodic lattices which we can get it? It is nothing, but five either it is a square, is it a rectangle or rhombus, parallelogram or a hexagon only these five types of two dimensional lattices we can have which exhibits a.

So, that is exactly because for periodic lattices these. So, all the diffraction patterns are in a reciprocal lattice unit cell we are taking some cut section. So, they can exhibit only these five types, but from analyzing this I am going back and find out what the crystal structure is a daunting task. So, this is a square pattern that is the first information because that part I will not go into from these we can say that this has fourfold symmetry. We can from measuring the lattice parameter we can identify what all this spots which correspond to once this spots have been identified what this corresponds to we if we take a cross product of two of this parts, that will give you raise to that will give information about the it is own axis.

Now, the same methodology which we this is an another one because in these particular case different variance and two phases are present, because since it has been analyzed, but it takes years works to do all these it is not c c one class one hour I will be talking about it today, and this is an another diffraction pattern where two types of phases are present where the patterns we could see them distinctly here it could not be I will come it.

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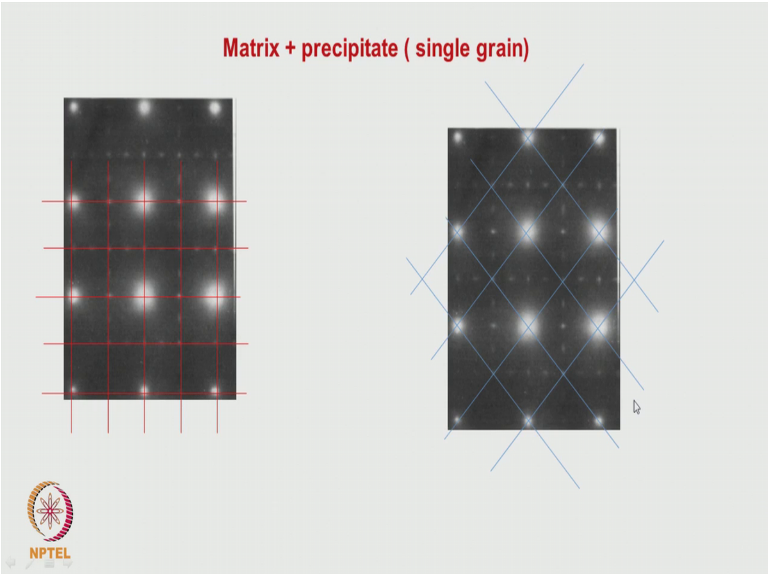

The first thing which I can do it is that, here again the same pattern I take it I can put make a this is a periodic fact pattern. So, I am just showing a grid to identify them if you this is the matrix one here there are some spots which come in between like this, this is one type of a pattern which I can identify at this corresponds gamma prime also which we had seen just now that type of pattern could be seen.

Now, this is that same pattern is there any type other type of a periodicity which is there. If I look from here to this one this one this one this particular one and the look at the streaking is that same in these ones correct, this forms a parallelogram which repeats itself like this that you can see it. Now no just come back to it here what I have done it is in these particular pattern I am just taking this central spot this one this central spot here this forms a grid this again because these are all circular spots they form a grid like this, this is one type of a pattern which is there that is what now you can see it.

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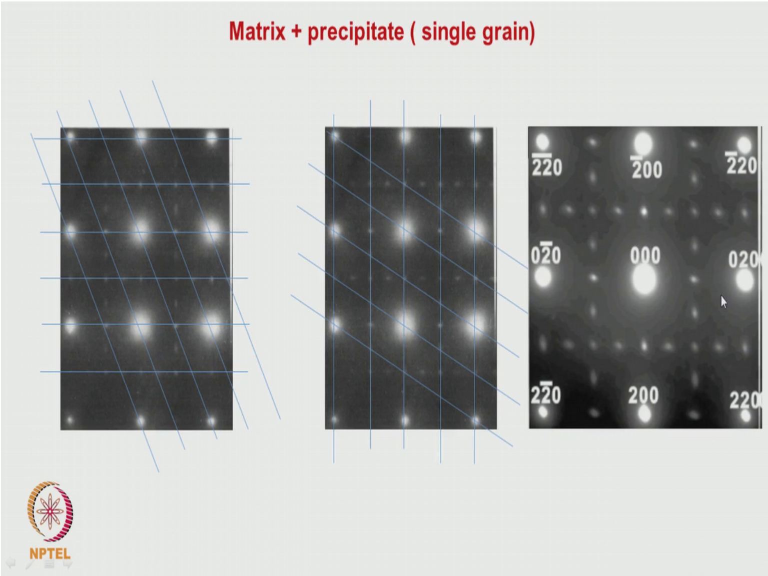

Now, in that same pattern whether any other pattern is there if I look from here to here this one spot which is straight, the similar type of a spot which is here this is a forms a parallelogram this parallelogram repeats itself again correct. So, this is one type of a grid which forms a two dimensional pattern which is there. So, we have seen one type of a pattern which is a square, there is an another type of a pattern we can see it here. In this one again if you look from here from here to here this one and this one these four together form an another rectangle this also is repeating. Now I put this, so that means, that this diffraction pattern contains essentially looks like 3 plus 4 types of period reciprocal lattice sections which are super imposed on one another that is when the diffraction pattern has been taken that is the first information which we require.

So, now you we have all the diffractions spots how these periodically arranged that information which we have got it correct? Otherwise if you look at the pattern like this apart from these central spots you do not see any periodicity the first information is to get that. Once this information has been obtained if you have taken a dark field using these reflections, that helps in identifying whether this spots corresponding to one particular second phase particle or it could so, happen that the other choice is that two second phase particles by chance depending upon that orientation, those spots are coming at the spots are coming at the same position.

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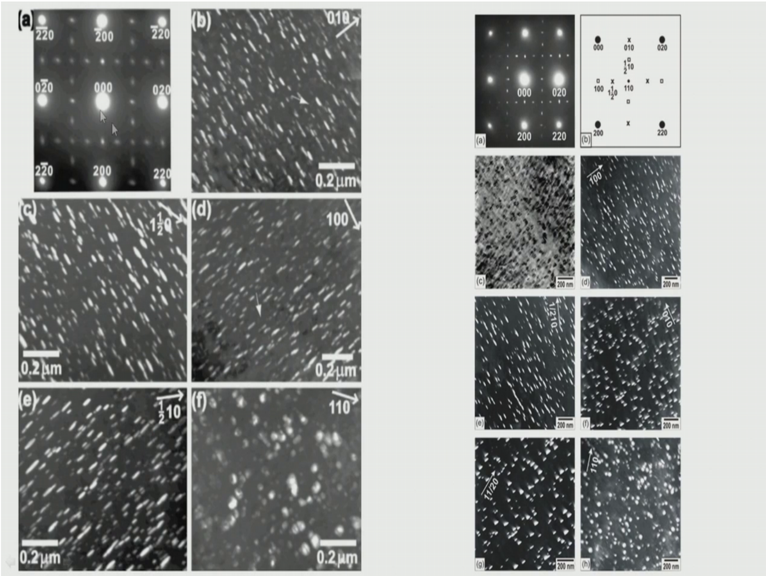

So, this can also be identified that we can do it only if we take a corresponding image, that is what essentially is being shown in alloy inconel 7on 8. In this if you see it I think here that indexing yeah it is correct you look at these 0 1 0. 0 1 0 is a essentially this reflection with which when I image it this is how this precipitates are aligned with respect to 1 0 0 when I tried to image it. So, 0 1 0 then if a image with one half 0 1 half 0 is this is there this is one half 0 these two streaking is in the same direction. So, maybe they are coming from the same precipitate.

When I put an aperture around tilt and try to image it, and when I put an aperture around this and try to image it, the difference which essentially what happens is that here if you look carefully in addition to this spots which are streak there are many circular particles are there; that means, that two morphologies, which we can see of a precipitate then the question is that if it is a particular orientation of a precipitate you cannot have a different morphology it has to be because in a single grain we are looking at it has it should have be the same morphology. In this case only option which we have is that this diffraction spot 0 1 0 is coming from two precipitates or diffraction spots of two of them are going you understand, this is that complexity which can come in a microscopy analysis that same thing happens here with 1 0 0.

So, try to image with as many reflections as possible, now we are able to differentiate and tell that this spots this one, this one and this one and these three reflections that is 1 0

0, 0 1 0 and 1 1 0 they match with both this phase one is called gamma prime and another one is called gamma double prime. But if I take with one half 0 those reflections come only corresponding to the precipitate particles, and now if you see it this is where you can see this these spots corresponds to one variant, this corresponds to another variant, and this one corresponds to the third variant plus this one if you take it these central spots correspond to both the variants.

All this information now we can correlate because first using these methods we are able to identify how many types of periodic two dimensional patterns are there, and then we have imaged all the reflections and with we are able to get these information. This is in an another alloy system where that is this is an another (Refer Time: 30:05) nickel super alloy inconel 625 where you get the same type of a precipitate gamma double prime is there we do not know whether gamma prime is there or not. So, how do we identify because analysis of this pattern tells that gamma prime and gamma double prime, the super lattice reflections of gamma prime coincide with that of gamma double prime, but gamma double prime has got extra reflections.

So, again we did a similar analysis, but when we look from 1 0 0 as well as one half 0, we find that only that ellipsoidal precipitates are getting image this gives an indication that this alloy contains only gamma double prime and not gamma prime. This is how one can get information this I have taken it because it is some personal choice from my work experience I have taken that that I thought this explains how to go about and analyze complex diffraction pattern and get useful information about the microstructure by analyzing this diffraction pattern.

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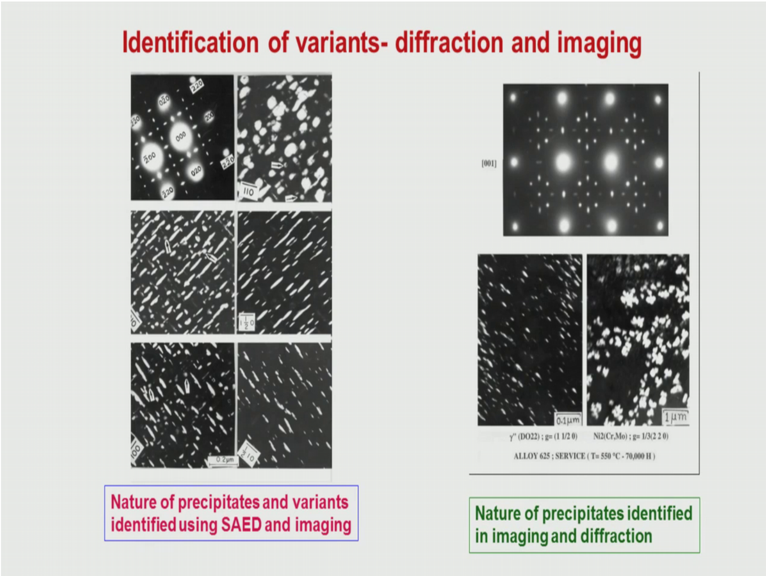

And in these particular picture if you see it you can now here see that with the 1 0 0 type the circular particles to morphologies could be seen which I had explained earlier. This again if you see it in these particular one there similar type of a gamma double prime is there in addition to it I have one which spot corresponding to here to another four spots are coming.

If I look the periodicity if I see this one I draw it around like this I can join it like this, this I can just go on this is an another one and then with respect to this one and then with respect to this one, with respect to this one I can join them essentially for the new phase which is there two types of possibly because the pattern looks similar, but they are oriented in a different way. That means, that since the pattern is similar we can tell that it is a different variant of the same phase which is there in the matrix that is what with a dark field image we have identified. Here I have not shown all the there are twelve variance are there which has been done, but here I am just showing only one particular one you understand that. This is the way one has to go about to and do analysis to get information about the various phases which are present in the sample.

What all the limitations in doing a microscopy analysis. If you are looking for statistical distribution of different phase and all then and volume which we are analyzing it is extremely small. So, it is gives a poor statistics, but for that s c m is better to get information, but s c m is not able to differentiate various phases and get that information that can be obtained from here, the strength of the microscopy is that you can identify how different phases are present and how they are distributed.

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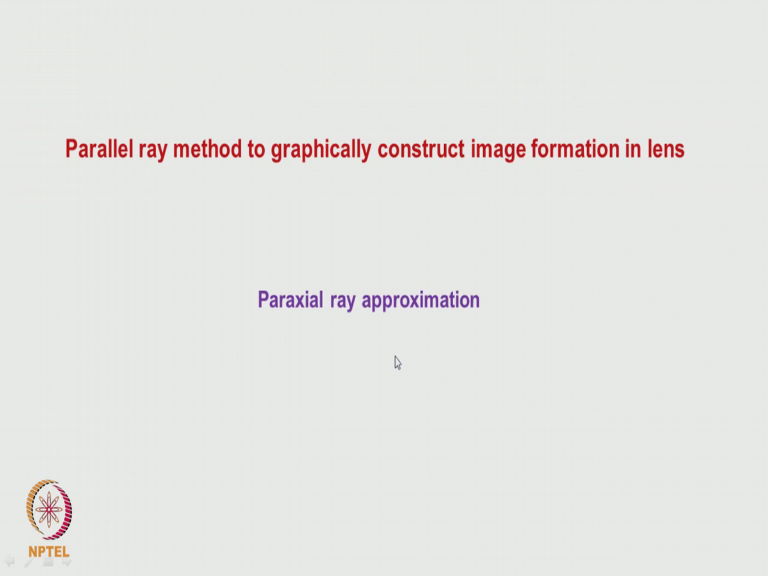

What position how where they are nucleated these are all the information which it can give. In a conventional transmission electron microscope what we have considered is only just a bright field dark field and selected area diffraction pattern.

Now, we will go to the paraxial ray method to graphically construct image formation in lens because in the last class I mentioned that the ray diagram can be drawn to find out first thing which one should know is that what is paraxial approximation.

A lens it is an object where when the parallel ray comes essentially at this particular point reflection takes place correct, then again here I can draw a surface normal again refraction will take place from here into this one and finally, refraction to the surfaces are takes place.

So, if we wanted to strictly find out the draw the ray diagram graphically we have to use the Snell’s law. The Snell’s law essentially says mu equals sin i by sin r where i is the incident angle r is the refracted angle correct. You assume the case of the ray which is passing through the center here the angle which is the rays meets incident and this one is both are 0 and if I consider a ray which is very close here then also I and r becomes very small in such a case we know that sin i and sin r can be approximated to I and r ok.

So, mu becomes i by r we can take it, this is what is done in geometrical optics to draw all the ray diagrams which we have studied what information which this can give is that this will at least tell you if you keep an object at a particular place, if we know what is the focal length of the lens what is the position at which we will be getting the image. Though the image at the center may be alright, but away from it will blur that is what it happens if we use a magnifying glass to we look at it only at the central region is bright other regions blurring comes which you might have noticed already ok.

So, this is the formula which we use it if we use this formula when we know the lens that is 1 by u plus 1 by v equals 1 by f where u is the distance from the lens to object and v is the distance from object lens to the image v and here u will come in this case and f is the focal length of the lens. So, using this formula we keep an object at a particular distance you can find out where exactly the image has to come that is very clear. In an electron microscope we get two types of information diffraction information and image information and both of them are occurring at different places or for that matter for a lens when we consider.

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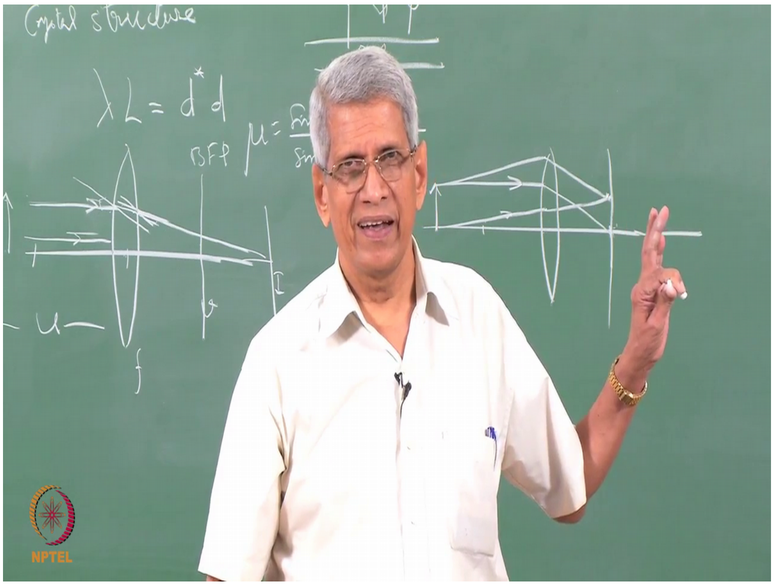

When we say that focal length is f inherently there is one plane which is at a distance f is going to be there on either side and this is which I have taken a convex lens, which of relevance to an electron microscope.

In this particular one at this distance and this plane is defined as a back focal plane, in the back focal plane what happens all the rays which are emitted from the sample in a particular direction, they are all brought to of focus that is if I will draw it again object, the ray which is parallel to the optical like here another ray we can consider it these two rays will be focused at this particular point these two rays are focused at point. So, this is the back focal plane. So, in the back focal plane we get both the get that diffraction image information and then in the image plane we get a image information which we get it correct.

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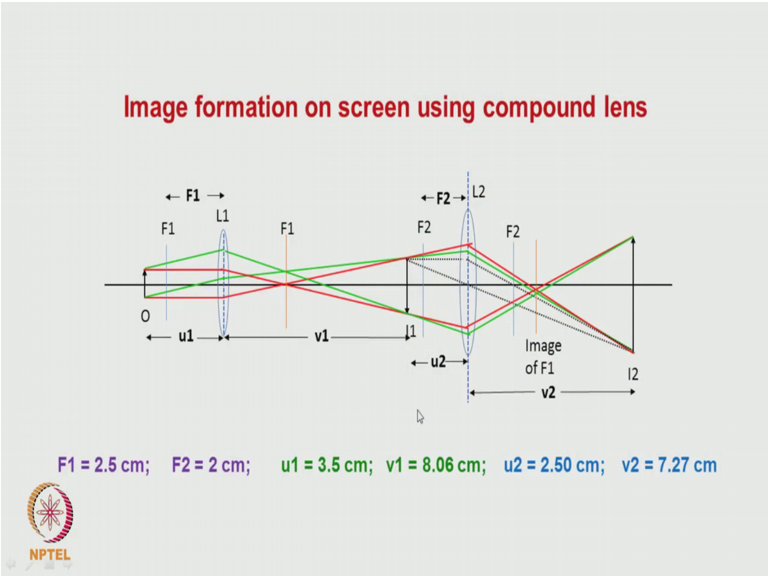

So, how to draw the ray diagram? With this way we can draw the ray diagram and what is image plane image plane is one which all the rays which are emitted from the sample in different directions.

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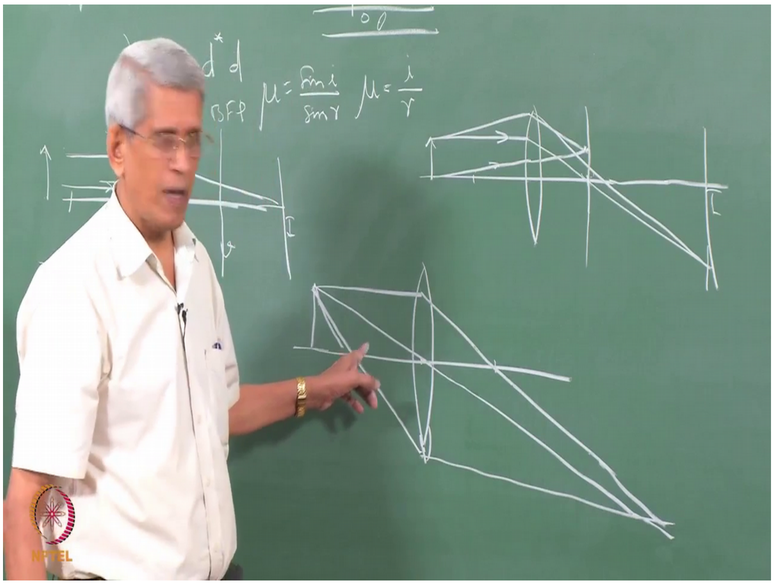

They will all be brought to a focus at a particular that is this ray. So, all of them will be coming this is what how we define as an image plane. So, this is how draw the ray because the first we know the object at what position it is kept and with respect to the lens then we can find out where the image plane is going to be there.

Now, using this paraxial approximation using this condition that rays parallel to optic axis they are all focused back focal plane, and what is this parallel ray diagram. Parallel ray diagram is essentially to find out this information you take a lens we know that a ray which is parallel to the optic axis passes through the back focal plane correct, and a ray which is passes through the center of the lens from the particular point that goes undeviated correct. And the third is this is an another focal plane the ray which passes through the this particular plane when it comes and meets the lens this will become parallel.

So, three rays which are emanating from that sample parallel, they have not drawn it correctly this as to be a parallel one all of them will be meeting it at that point is the image plane. So, image plane can be found out using the formula or graphically this way also we can do it. So, essentially what is done here is that when we do that this is the back focal plane, this is where you see that the rays the green rays are the rays which are emanated from sample at different points, but in the same direction that green as well as the red they are focused at these point, and the rays which are emanating the green and red are coming from the same point and they are all focused to a point this is what the image plane is this we can calculate it.

Suppose there is an another lens which is kept there, if this lens has a focal length such that this is where exactly it is then we know what is u 2 you know what is f the focal length if you know of this lens then we can identify where exactly is that image will come. Then how to draw this is that again use the same parallel ray graphical method with which I have taken one, I can immediately now able to identify where the image has to come and then this rays which are joining here and diverging and meeting the lens, I can draw them and this is how the ray diagram can be drawn. This is for an image here I have a taken a focal length f 1 equals to 2.5 another is f 2, 2 centimeter this is u 1 and these distances I had given with which it has been done.

Suppose I wanted the diffraction pattern to come on this screen what should I do? I mentioned in the class that all this lenses which convex lenses by changing the lens current we can change the focal length this lenses are kept at the same position this is exactly what is being shown here now you see this. The lens is here the focal length has become four point three four there it was two with this when I have done it and now use the paraxial because this is the object now for this lens. So, a ray which is coming from here this is the way it will come and join, and the ray because and the ray which is coming from here that will come and join here.

So, now we have drawn where exactly this rays have to form that image, and now you see that all the red ones which are coming from, they get focused here other one will get focused here by changing the focal length. Now this has been used as a because this dash lines are the ones which is essentially the parallel ray method which I am using to identify the image plane because here what I have done is the between the object like in a microscope object is kept at a particular position in the sample, and the image is taken on that screen. So, that distance is fixed. Now by this lens focal length remains that same like in an objective lens only this length a focal length of this lens is being changed this is exactly what is done in a microscope to get a diffraction pattern.

Now, we can see that using this method you can draw the ray diagrams. I expect all of you to learn this because in an electron microscope basically when you wanted to understand at different places we form images one should be able to draw ray diagrams to identify what all the positions in the optical column where different type of images form. This is just like a tutorial I had shown, but essentially if you draw it yourself taking different focal lengths then only you will understand it I may give as an exercise for you to solve, but I expect you to work it out yourself.

This I consider it is that one should understand; normally what most of the students do is that they have in memory how this diagrams are there, but if you tell that this is the particular length as a focal length draw the ray diagram nobody knows how to draw it. One should not be in such a position that is why I took this as a sort of a tutorial to just inform you how one should go about and draw ray diagram for diffraction as well as image to come on that screen. We will stop here now.

Thank you.