Indian Institute of technology Madras
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NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Lecture-35

Materials Characterization

Fundamentals of Transmission Electron Microscope

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Hello everyone welcome to this material characterization course in the last class we started looking at the transmission electron microscopy I just gave a brief introduction about the, the technique as well as for the instruments and we realize that the transmission electron microscopy technique involves a lot of diffraction principles and it is good that we have the enough background about the diffraction in the x-ray diffraction section itself.

So I hope that you will be able to appreciate this electron diffraction in a similar way without any difficulty so let me continue the discussion on the instrumentation details about the TEM so we just started looking at the electron source and the lenses and then basic operations and the types of electron source and so on so in that we are we are also looking at some of the details about the other parts and in continuation that we will now look at some more details.

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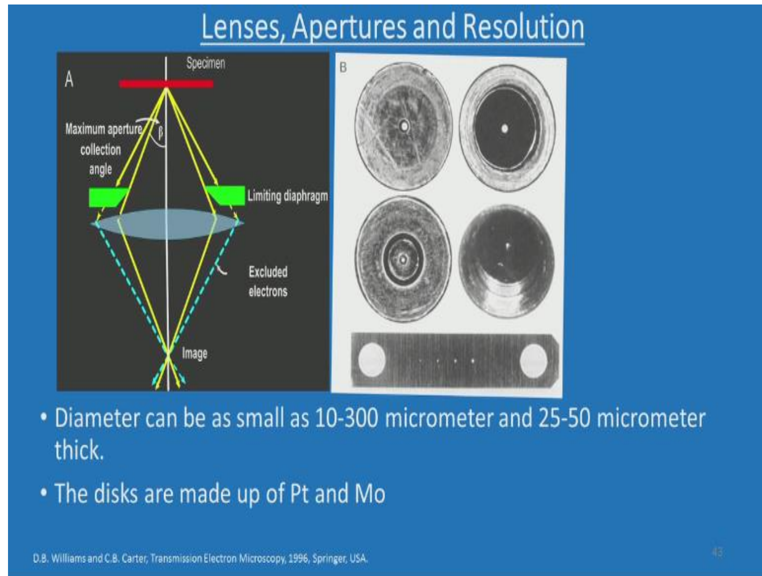

So we were just seen that the details of the electromagnetic lens and how it is functioning and all that and today we will discuss about the lenses apertures and their relation to the resolution so look at this take what is shown here is it is a there is a specimen there is a electron beam coming through and then you have the maximum aperture collection angle β and then you have the limiting diagram and then you have the lens and then image is converging so we are talking about this limiting diaphragm and the apertures.

So some of the apertures and the diagrams appear like this, this is an actual photograph from this textbook and you see that typically they are all metallic discs which where you have the perforation in the middle depending upon the size or the diameter requirement so typically the diameter can be as small as 10 to 30 micrometer and 25 to 50micrometer thick so this is the typical dimension of this kind of a purchase and diaphragms and the discs are made up of typically platinum and molybdenum.

So one of the objective aperture is shown here you can see that a small perforation it varies from model to model and we will look at the actual size of this strip there is an objective aperture when we look at the in an electron microscope operations I will just show you what is this for

different perforations will do to the image and image contrast and soon so this is the information about the lenses so what are they control what are the aperture and diaphragm functions to control the collection angles what is collection handle this is a β so they control that in objective lenses.

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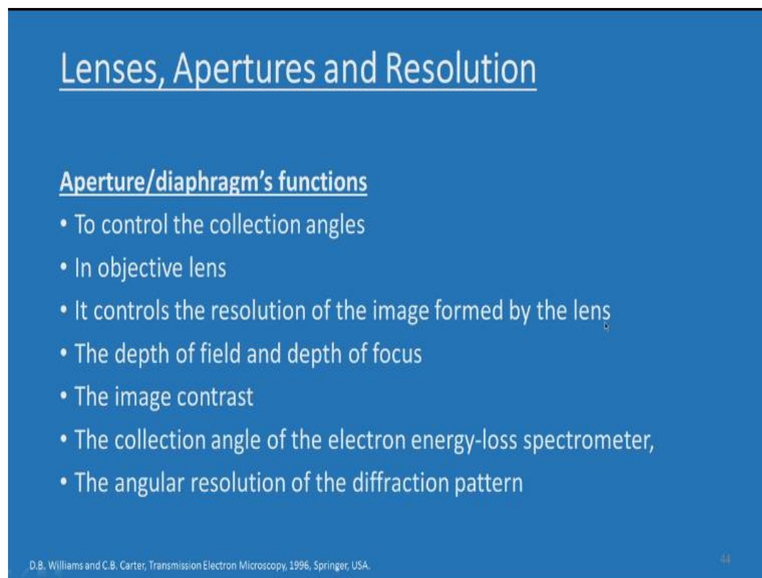

It controls the resolution of the image formed by the lens the depth of field and depth of focus also controlled by this the image contrast the collection angle of the electron energy loss spectrometer they are also it is being used the angular resolution of the diffraction pattern so the apertures and the diaphragms will have all this factors will be involved or it will influence the, the functions of all this parameters and also you have the various pumps and you have the diffusion pump and ion pumps and vacuum pump and so on.

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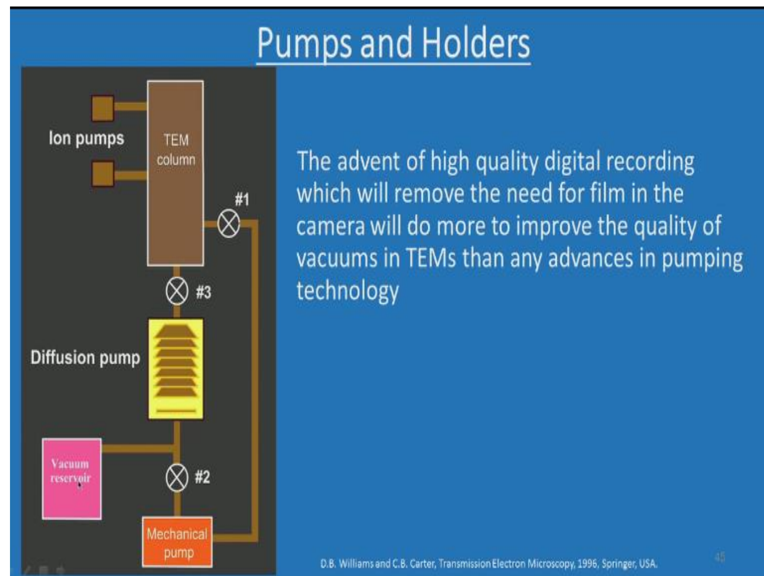

I will give the actual details when we when I show the laboratory demonstration and what they do is so just to give a general comment the advent of high quality digital recording which will remove the need for the film in the camera will do more to improve the quality of the vacuums in the team's than any advances, advances in pumping technology so today we be completely record all the information in a digital recording system and some of our microscopes still use the plate films.

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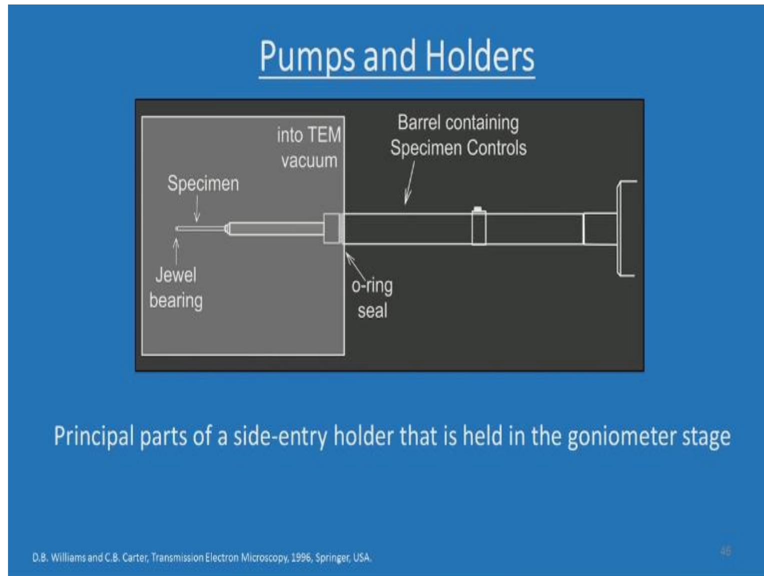

And we will look at the details when we go to the laboratory exercise and coming to the holders this is how the TM holder will look like you can see the details here so, so you this is a barrel containing specimen controls this is awaring seal and the, the section which is shown in the shaded box is under the vacuum and this is not under the vacuum.

So you can see that the specimen will somewhere sit here and then there is a bearing which will sense the I mean the positions of this specimens inside the column so this is the principal parts of a side entry holder that is held in agoniometer stage so the, the specimen is held in agoniometer stage in this orientation wherever we make a side entry holder and you can have different types of holders whether you can have a rotation folder where the sample is will rotate in this fashion and, and you have the heating holder where you can heat.

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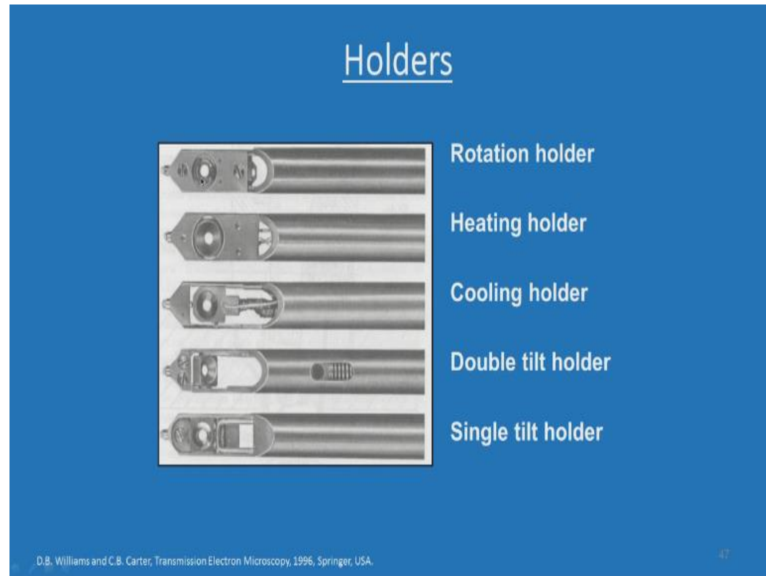

The sample locally to look at the phase transformations and thermal response of a material can be studied thoroughly and similarly a cooling holdup you can have a quite a bit of a heat treatment can be done and then you have a double tilt holder it means it will tilt in this axis as well as this axis so that is called a double tilt holder and typically most of the basic version of this equipment will have this single tilt holder which will rotate in this fashion in this axis this is an axis in this rotate way in this fashion so I will show in the lab all this holder show how they look like in I mean these are all photographs taken from this textbook.

But you can I can also show you in our laboratory how this holders are being used or typically you can if you look at the usage rotation folders they are being used to analyze some of the no orientation effects and then and then and crystallography symmetry operation which, which can be very effectively studied through this holder and then as I said for all the phase transformation studies it can be used and W tilt holder is used especially if you are interested in defect analysis are when you want to obtain a very specific contrast mechanisms are if you are interested in identifying a very specific location and these kind of holders are very useful.

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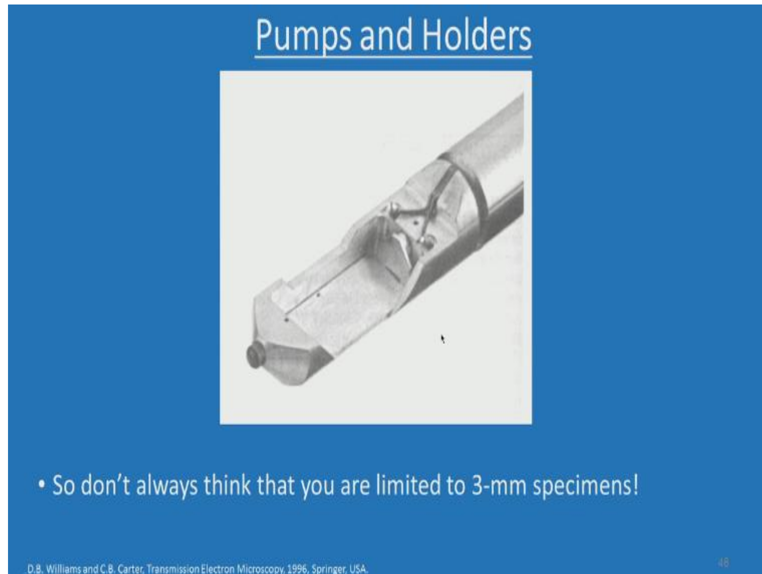

Especially if you want to do a typical analytical work you need a double tilt holder for a normal conventional imaging you can do with the single tilt holder and so on so this is a brief introduction about the holders and it is not that only a small sample size can be kept and then you can also have this kind of a big samples more than 3mm specimen just for an information and then you can also simultaneously load the samples in a multi sample holders in a in a inside the vacuum you can put it in a this kind of a multi sample holder also you can put it in the two slot holders and so on.

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How to "see" Electrons

- Our eyes cannot "see" electrons, we have to resort to the phenomenon of cathodoluminescence (CL) in order to provide an interface between electrons and our eyes.
- The CL process converts the energy of the electrons (cathode rays) to produce light (luminescence). As a result any electron display screen emits light in proportion to the intensity of electrons falling on it.
- The "fluorescent" screen is coated with a long-delay phosphor

D.B. Williams and C.B. Carter, Transmission Electron Microscopy, 1996, Springer, USA.

So these are all some of the varieties of folders which is available with each of the microscopes now how to see electrons see you have to remember that what we are seeing in an electron microscope is it is a beam of electrons our eyes cannot see electrons we have to resort to the phenomenon of cathodoluminescence in order to provide an interface between electrons and our eyes so the cathodoluminescence process converts the energy of the electrons that is cathode rays to produce lightluminescence as a result any electron display screen emits light in proportion to the intensity of electrons falling on it.

The fluorescent screen is coated with a long delay phosphor so what we have to appreciate here is be only past the electron beam and which comes out of the sample after the electron beam after comes out of the sample must have interacted with the specimen accordingly the intensity profile will change as I just mentioned in the yesterday's on animation I showed an electron beam passes through a sample the intensity will vary according to the specimen interaction and then that effect can be visualized only when you put it on the fluorescent screen which will again emitter right depending upon the, the amplitude of the light which, which it receives that contrast also will produce.

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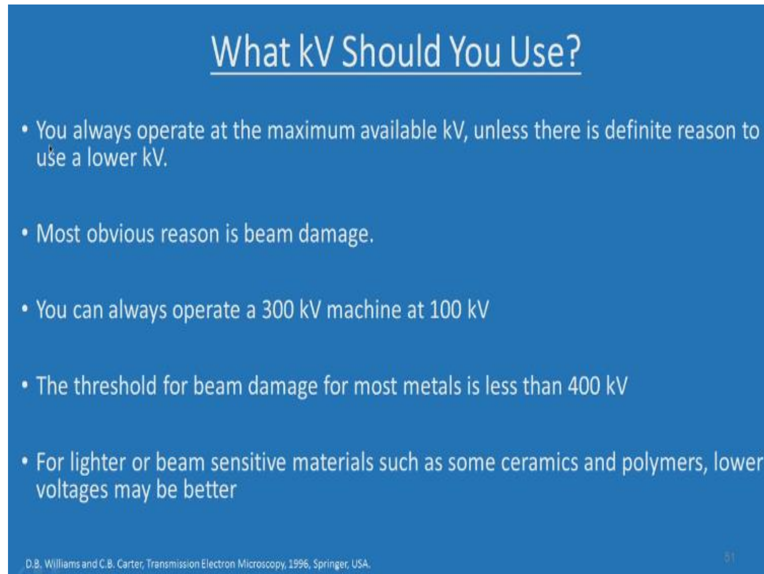

We will look at the much more detail about this how the image formation in, in much more detail when we discuss the TM imaging section so this is one to give you an idea how the electrons are electron beams are made into an image and we should know what kb should you use so we have a varieties of microscope with different voltages of operating voltages so you always operate at the maximum available kb unless there is a definite reason to use a lower kv and most obvious reason is the beam damage if you recall.

I just showed in the beginning of the TM lecture I showed some of the images where the specimen get affected by the radiation damage and I also cautioned you that we should not just misinterpret those kind of a damages as a characteristic of material so we have to be very careful with the that is why the appropriate kv need to be chosen if it is a metal or if it is a ceramic if it is a polymer or biological samples you need to choose an appropriate voltage to examine the samples.

We will talk about it much more detail when we when we go to the sample preparation section so you can always operate a 300 kb machine at 100 kv the threshold for a beam damage for most metals is less than 400 kv for lighteror beam sensitive material such as some ceramics and

polymers lower voltage maybe better so this, this comes by inexperienced it varies with samples to sample.

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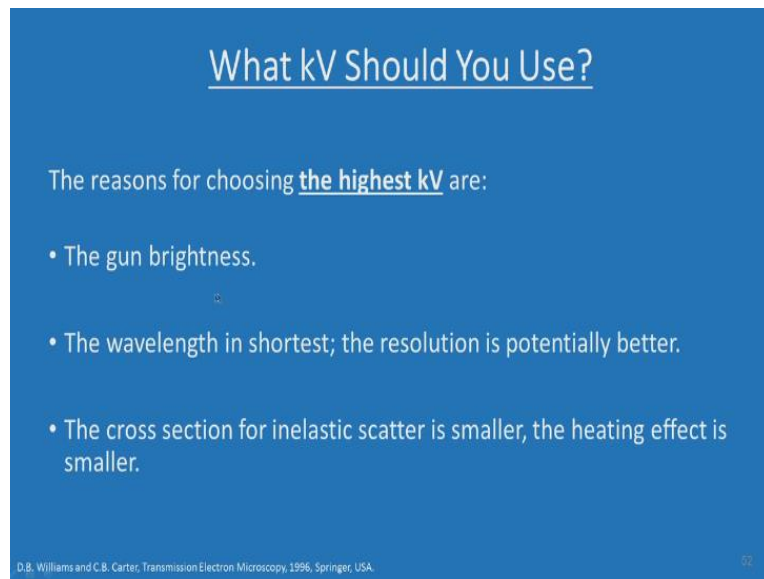

So one has to really judge this with the previous experience and so on so why do we always prefer a highest kv so there are some of the reasons because the higher the voltage operating voltage or accelerating voltage that done brightness is better we get the better run by the and as we all know that from the broadly relation what we have seen yesterday the wavelength is wavelength in shortest many when you kv is higher the resolution is potentially better the cross section for inelastic scattering smaller and the heating effect is smaller.

You see we have to remember that when the heating of the sample also will affect your you know characterization purpose because that will that should not induce a new effect into your material or you should not transform ever material from the initial stage to some other stage so the heating is very important so you have to be very careful about the, the type of kV you choose before you operate the I mean before you put your sample inside the TEM column to analyze it.

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Contamination

- Vacuum can be a source of contamination. **Particularly residual hydrocarbons from the pump oil** which crack under the electron beam.
- **Carbonaceous material** then deposits on your thinned specimen, making it difficult to do sensible HR imaging or microanalysis

D.B. Williams and C.B. Carter, Transmission Electron Microscopy, 1996, Springer, USA.

So these are some of the general information about the kV then we will talk about very important aspect of again a specimen electrode electron beam interaction that is contamination vacuum can be a source of contamination particularly residual hydrocarbon from the pump oil which cracked under the electron beam carbons material then deposits on your thin specimen making it difficult to do a sensible high resolution imaging or microanalysis.

So this we have to be very careful and any material which is being coated on your sample and then whatever you will see as a new feature may not be belong to your specimen at all so we have to be careful about this so that is that is exactly the contamination is always avoided contamination also occurs through lock with the specimen it can be minimized by heating the sample above100 degree in a heating or cooling the specimen to a liquid nitrogen temperatures polymers and biological samples.

We can easily introduce hydrocarbon contaminants as the out gas in the vacuum so it is sensible to cool the specimen when you cool the specimen it attracts water vapor which condenses as ice on the surface so here again the some of the basic idea of contamination is given but then you have to take a lot more care while preparing the sample for a tem analysis these are all general

guidelines within the column but even before even putting the sample inside the TM column you need to be very extra careful which some of the aspects.

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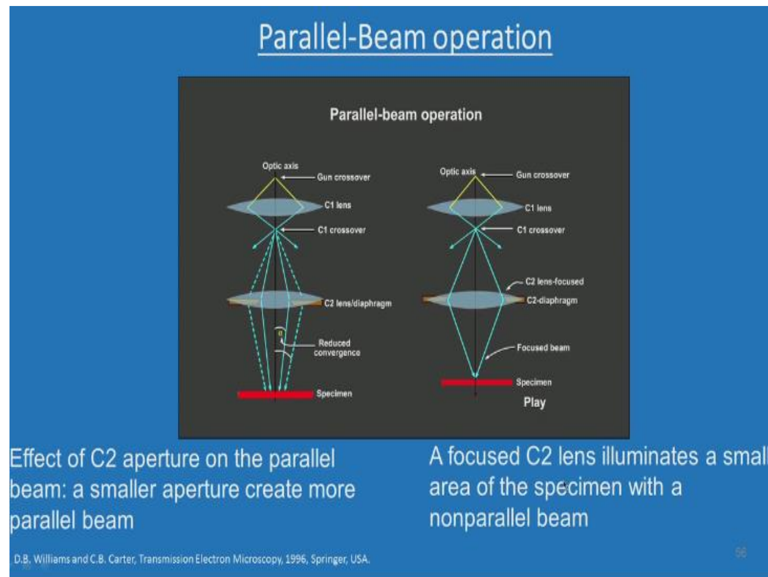

We will discuss when you prepare when we go to the sample preparation class I will just show you some of the live examples now we will just go to the some of the basic you know instrumental details and it is instrumental operation and what you are now seeing is one of the schematic which shows the operation that is called a parallel beam operation the first one that somatic shown here is you see that you have the c1 lens see one cross over and c2 lens and then it is falling on the specimen.

So which has got some the semi aperture angle is there α on the right-hand head side you see that the beam is made parallel y parallel because some of your you know diffraction experiments we when you do only with the parallel beam you get will be able to focus the diffraction spot to the sharpest as possible so parallel beam operation is important in a menu when you do a diffraction analysis so for that you need to get this condition.

So this is done by the insertion of another objective lens where the which is the French focal plane of the turbulence you have the another objective lenses their upper objective lens so which is being made being made into parallel and the same line you can see another schematic where it shows the how the convergence angle control the parallel beam operation so for example you see that effect of c2 aperture on the parallel beam a smaller the aperture create more parallel beam.

You can see that smaller the aperture create more parallel bring you can see that α you can have a different α will give you a different kind of a converging beam and then what you have to understand here is the, the nomenclature for the, the lens is c1 and c2 are pertaining to a particular system you can have condenser lens one condenser lens too and then objective lens one something like that so do not worry about this designation of the lenses but it is you have to just see that what the operation of that particular lens and what is the effect of the effect of that particular lens our diaphragm on the probing electromagnetic radiation that is all.

We are interested and then you can see that a focused see two lines eliminates a small area of specimen with their nonparallel beam suppose if you have the focused lens then you will have a focused beam here, here the c2 lens is focused and this is a c2 diaphragm then you are able to get the focused and nonparallel beam so just give you an idea inside the column what kind of beam designation or what kind of beam conditions under which they the specimens are examined.

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The converging beam is a probe we use such a probe when we want to localize the signals coming from the specimen as in microanalysis or a converging beam also known as a micro or nano diffraction this is again a very important operations of diffraction suppose if you are wanted to obtain a diffraction information from the very localized region for example a small second phase particle you just want a diffraction from only from that particle then you need this converging beam operation I will show you the actual diffraction pattern when we discuss the diffraction in tem in much more detail in the following section.

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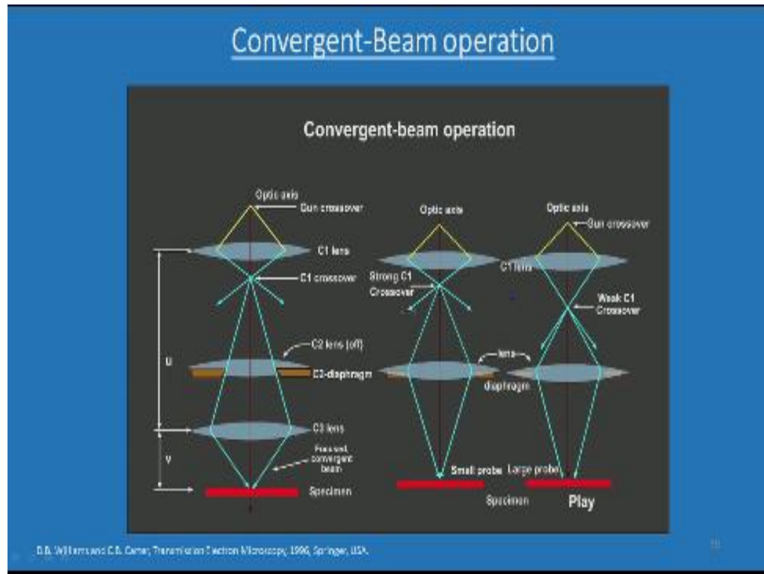

So how the converging beam operation is done this which is being shown as a schematic with the in the ray diagram here. You can see that this left hand head shows how the converging beam are obtained and here also you can see that how a small probe and a large probe size are controlled by this lens system just give you an idea so you should have the large you by V ratio which promotes the converging beam of the probe so this is just shown with the two condenser lenses one is a strong p1 cross over use a small probe and other is a beak C1 crossover will give a large probe. So how to get the microprobe or a nano probe is it, because the controlling the c1crossover.

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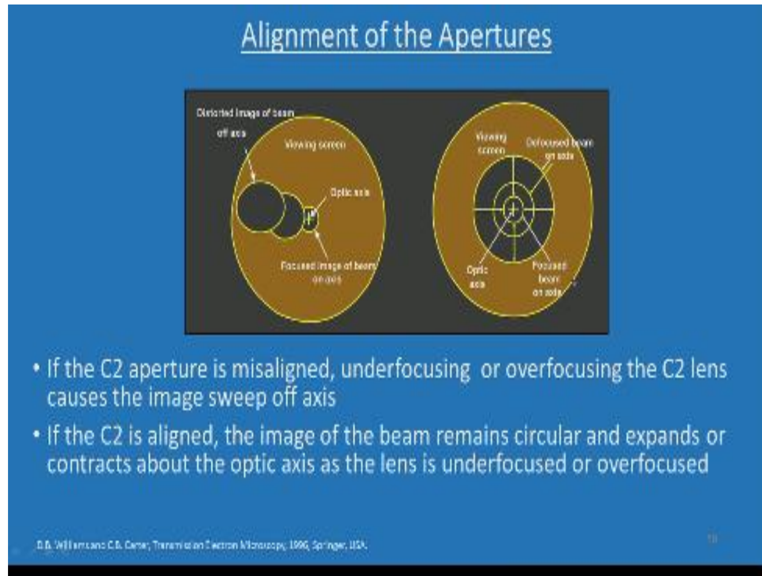

Now we will just look at some of the alignments of the aperture so we have just seen what types of apertures and then if they are not aligned properly what is the issue so these are the some of the schematic which shows that so you can see that now a distorted image of a beam of axis and this is an optic axis this is a viewing screen and the focused image of the beam on axis and then when it is distorted you will see at this kind of a configuration of the beam on the screen so here.

Here if everything is focused here you can carry the focused beam on the axis and the D focused beam on the axis on the this is on the vm scream so these two explains how a beam supposed to appear like ideally on the fluorescent screen when you do a lineman setting so this is just give you an idea what is that alignment is about so if the c2 aperture is misaligned under focusing our or focusing the see two lines causes the image sweep of axis.

So it could be under focusing our over focusing from the sea to lens if the c2lens is aligned the image of the beam remains circular and expands or contracts about the optic axis as the lenses under focused are over focused so it is completely aligned but you can just under focused and war focus it will just open and close in a symmetric manner so then we can make sure that the

beam is aligned in the column then we are ready to go so these operations. We will see it in the laboratory.

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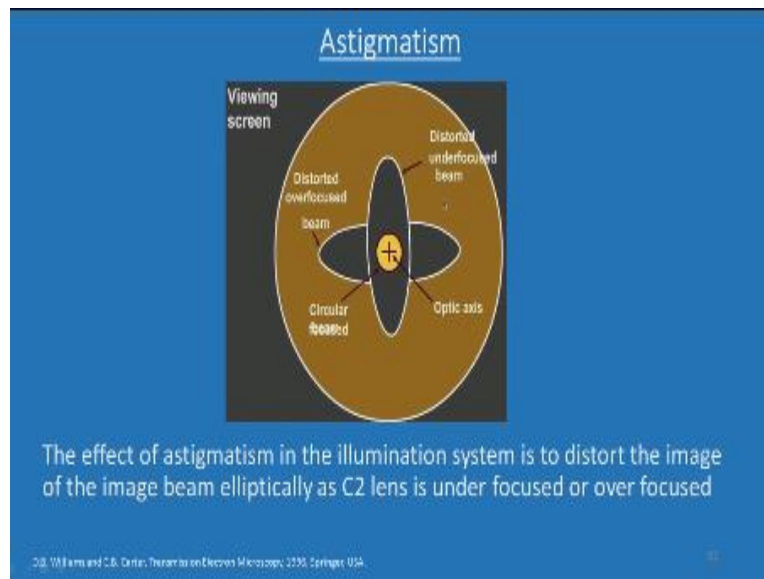

And very importantly how do we identify whether we are beam is having astigmatism so suppose this is a shape of your beam on the fluorescent screen then you, you can simply say that there is some issue. So you have this distorted and folk under focus beam and this is a distorted over focused beam and this is supposed to be a circularly focused beam should appear like this and if it deviates from this circularity and then it shows a oval shape like this either it could be under focused beam or it could be a war focused beam you have to cut it for the astigmatism.

So the effect of astigmatism in the illumination system is to distort the image of the image beam ellipses elliptically as see two lenses under focused are over focused so this is one aspect we have two very important aspect of alignment and this is one of the major part of the operation.

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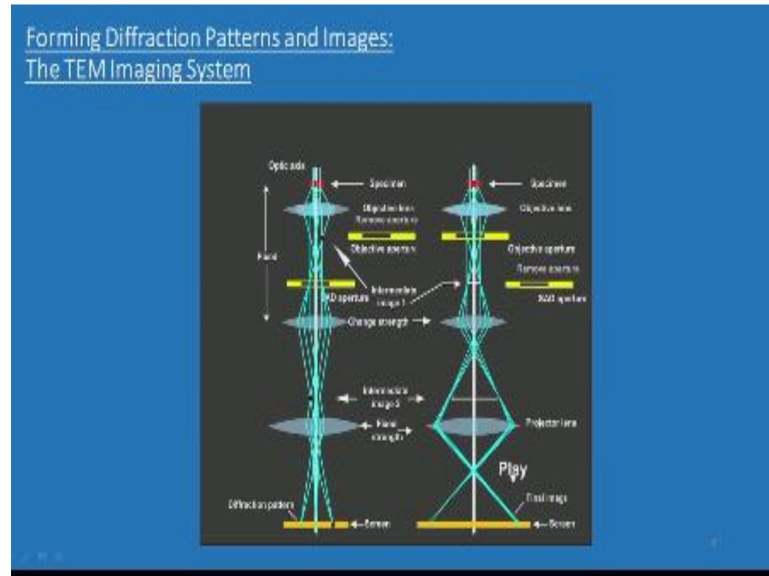

So we will now go to the imaging system and I will just play some of the schematic ray diagram which will show what kind of imaging operation one need to carry outing a TM so what you are seeing here is the first one is where diffraction beam will be formed the second one is image will be formed.

So I will you use you see that this is specimen and there is an objective lens and you have objective aperture and then assay aperture and then you have the other intermediate lens and then finally it reaches the screen. So what you are to appreciate is when you want to record a diffraction pattern what are you supposed to do so I will play this schematic again

You just observe so I have the objective lens this is an optic axis so you have objective aperture and a Sadie aperture suppose if I want tore cord SI d then objective of pressure should be removed from the optic axis that is what shown in the schematic suppose if you want to record image your essay d aperture should be out of the optic axis.

So you can see that correspondingly how this you know the intermediate image is being furthered demagnetized and then magnified and finally it reaches the screen and you can see that

how the back focal plane again get projected and then how the image formation maggots so the two important a physical operation here one is forming a diffraction pattern one is forming an email a basic thing. We will talk about actual image formation in and another better example this is one to show that what kind of physical operation one need to do when you do an image.

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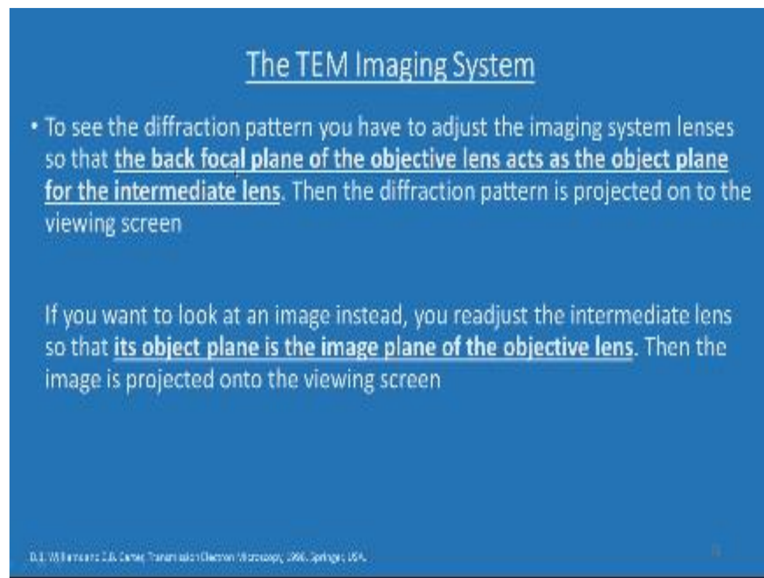

So this is the description of what I just shown to see the diffraction pattern you have to adjust the imaging system lenses so that a back focal plane of the objective lens acts as the object plane for the intermediate lens then the diffraction pattern is projected on the viewing screen let us go and see so this is the back focal plane which of the objective lens will act as an image for the intermediate lens.

Then the diffraction pattern is projected on the viewing screen if you want look at an image instead you read just the intermediate lens so that it its object plane is the image plane of the objective lens then the image is projected under the viewing screen like what I have just sho

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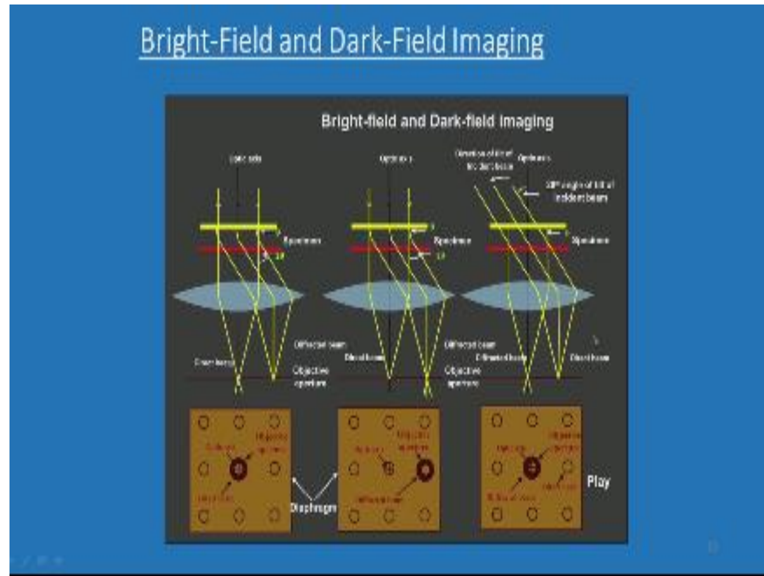

So another important imaging system in a TM is producing a bright field or a dark field we have seen that in the optical system itself so you know the basic principle of obtaining a bright field and dark field so in TM it is it is the same thing but the schematic clearly illustrates that how the bright field and a dark field imaging are realized so you have this specimen this is an optic axis and then you have the whatever you get on the screen is given on the 2dprojection here.

So this is the optic axis and this is also a zero-order zero- order beam that is transmitted beam and then you have a diffracted spot around this so when you want to do a bright field image the objective aperture just stays on this transmitted beam and then you will get an image but on the other hand if you are interested in taking a dark field you can see that your aperture is moved to from the optic axis of transmitted beam to one of the diffracted beam.

So you can do this dark field imaging on any using any one of this diffracted spot of your interest so each one will give a different information from a different set of planes from the specimen so based on that interest you can do a bright field or dark field and so on so normally this is done through a beam tilt operation beam is tilted rather than an objective is aperture is just brought

down to this part a beam tilt operation will make the beam the diffracted beam come to the center and then aperture is being put on that then you will get the dark field image.

So that is the primary information from this slide is you should get an idea a transmitted spot is being used to form a bright field image any one of the diffracted spot is used to form a dark field image so that is the information from this slide.

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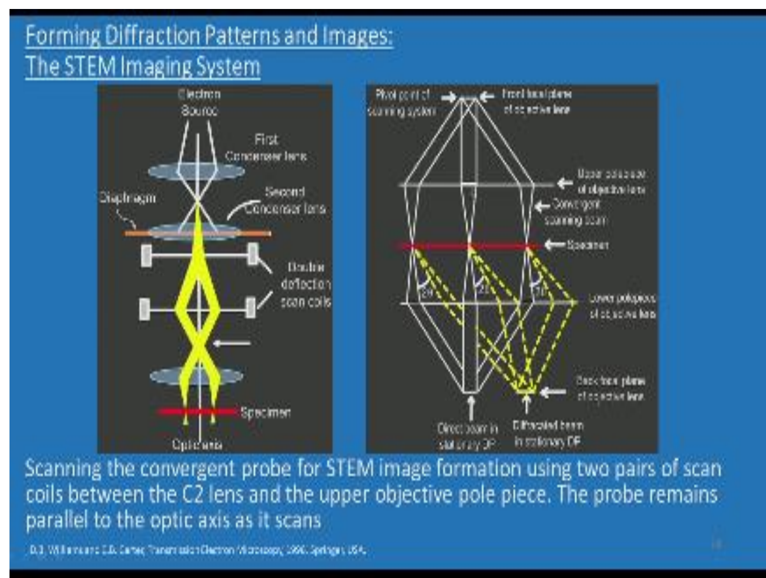

Now we will look at the other important imaging system in a TM called stem scanning transmission electron microscopy imaging so you have the go through this schematic you have the electron source and then you have the condenser lens please recall what we have discussed in a scanning electron microscopy lecture where we had the deflection scan coils so similar setup is here which will manipulate the electron beam according to the scanning action.

We have very we have gone through the scanning action in much more detailing the scanning electron microscopy you just recall those aspects similarly beam will scan through the sample on the complete surface the only difference between the scanning electron microscopy and this is here that your specimen is transparent but then the scanning action is the same.

So you get the signals again you have the detector and then which will attack the signal and so on they have to rest all rest all the operations are seen except that the specimen is transparent here so that is one point the right hand side schematic shows that how exactly the you know the ray diagram looks like.

So you have the pivot point of a scanning system French focal plane of an objective lens here and then you have an upper pole piece of an objective lens and then you have the convergence scanning beam which scans on the specimen like this and then you have the lower pole piece of objective lens where you can see the direct beaming a stationary diffraction pattern and a diffracted beam in a stationary diffraction pattern.

So you can see that this is falling on a back focal plane of an objective lens so you have the scanning the converging probe for a stem image formation using two pairs of scanned coils between the c2 lenses and the upper of the two pole piece so this is the exact location of the your scan coils between c2 lens and upper objective pole piece the probe remains a parallel to the optic axis as it scans.

So this is a simple way of putting this transmission as well as scanning system together and you have to just you know you can appreciate that the operations are similar to as the scanning electron microscopy with regard to scanning action is concerned but the difference is this specimen is here at transparent electron transparent specimen. We are in a seem you have only the surface which is B I mean you gives out all these signals after the scanning so that is one point you have to remember.

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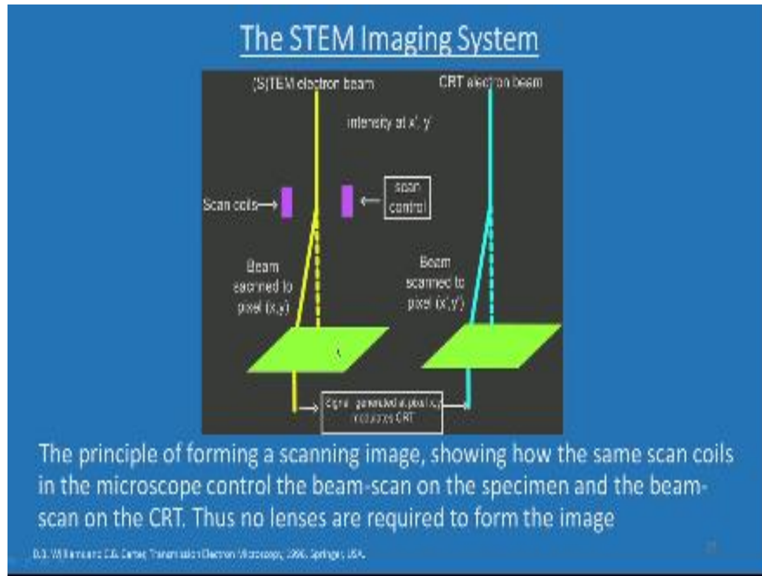

So here a principle of forming a scanning image showing how the same scan coils in the microscope controls the being scanned on the specimen and the Bing scan on the snow lenses are required to form the image so it is similar to scanning electron microscopy as I told you before so how the image formation occurs in a scanning electron microscopy is demonstrated in this so you have already and knowledge on this so I will skip this.

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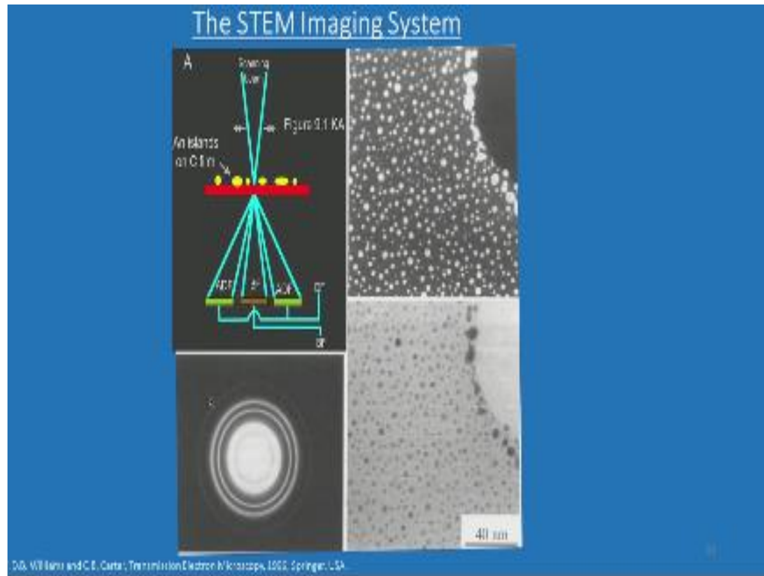

So typically you have the what kind of an image will see through a STEM imaging this is taken from this text book again so you have the bright field image and this is a dark field image this is called no another dark field image and this is a typical diffraction pattern what you have is you suppose if you have the specimen and this is a scanning beam and you have the bright field I mean image can be formed from this transmitted one and this is a diffracted beam.

You have a BF is called annular dark fig that means suppose if you have this is the diffraction pattern a ring pattern in another ring will collect complete the dark field intensity and completely block the transmitted intensity then you get this kind of very interesting image that is called annular dark and then you have the right field which is formed with this image in this system.

Please remember a scanning transmission electron microscopy is very specifically used for a chemical analysis very useful in chemical analysis and microanalysis are the elemental mapping across the particular location of the material feature and mostly the probe which is being used in the stem is much more smaller as compared to normal TM operation.

We will just show you some of the demonstrations about this also so as an introduction to this course you should know what are all the possible techniques possible in a transmission electron microscopy in that respect only I just brought the brought the introductory slides like this then we will get into this basic idea when we actually operate the TM in the lab.

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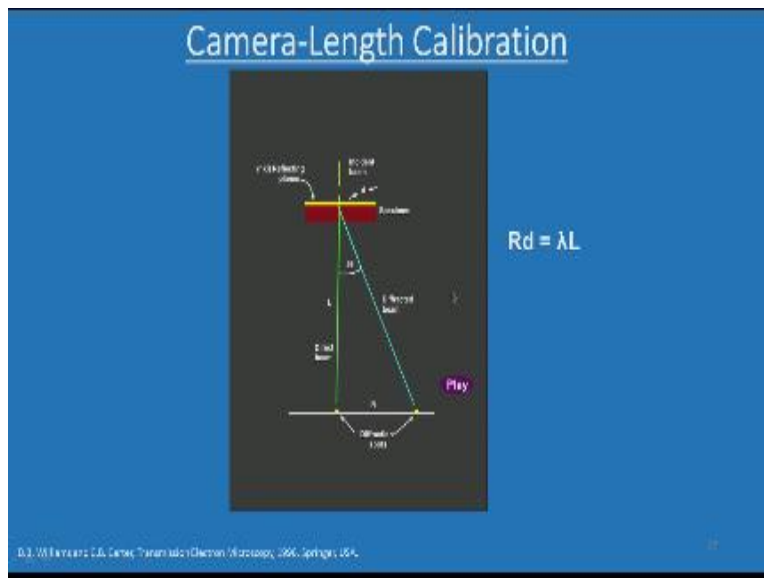

So now the another important aspect of TM operation as a camera length calibration the schematic which shows a basic geometry geometrical relation between the distance between the specimen and the screen and then and what you can do with that camera geometry so let us see that this is a specimen this is an incident beam suppose this is the back focal plane here and this is a diffracted beam this is 2θ and this distance is l and then this distance is r this is from the transmitted beam to one of the diffracted beam the distance is r then the relationship what we have is our dis equal to l lab l lλ.

So this is a very important relation we will we will look at that derivation when we look at the diffraction of the diffraction phenomena and TM in much more detail but this relation is a very important relation in identifying our indexing the diffraction pattern and the λ is kept constant always that is why it is called it a camera constant λ a camera constant and this requires some

calibration for before you do any analysis your camera length need to be calibrated with a standard sample and then it has to be fixed. So then only you can use this relation for any of your diffraction analysis and so on we will do a demonstration or we will work out some examples also

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Comparison of Experimentally Measured Camera Length (and Camera Constant) with the Digital Readout for a Philips EM400T Operating at 120 kV (λ=0.0335Å)

Camera length Setting	Digital readout (mm)	Measured camera length, L (mm)	Camera constant L (mmλÅ)
1	150	270	9.04
2	210	283	9.47
3	290	365	12.22
4	400	482	16.14
5	575	546	18.28
6	800	779	26.08
7	1150	1084	36.29
8	1600	1530	51.22
9	2300	2180	72.99
10	3200	3411	114.20

D.B. Williams and C.B. Carter, Transmission Electron Microscopy, 1996, Springer, USA.

And this is the typical table 1 generate after the calibration of the camera constant and I leave it there we will use this parameter when we do a diffraction analysis you will appreciate this importance of camera constant why I am showing this slide because when you when you decide to use this kind of an analysis like if you want to use a camera length and then if then it has to be calibrated.

First of all you have to check whatever it is showing in the display in a TM display whatever the camera considered it shows we have to we should not finally take it up we have to check whether it is calibrated or not then only you can take that value and use it for the diffraction analysis later so that for that information only I brought this.

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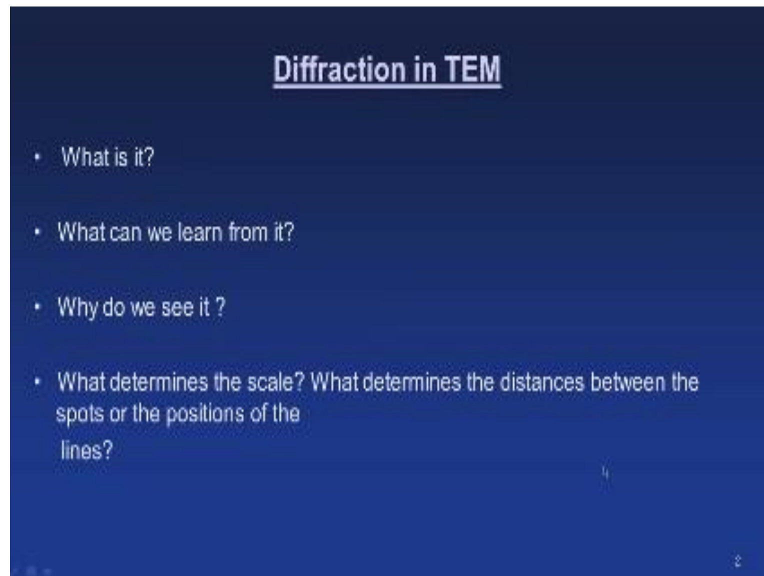

Now I slowly move on to the other important aspect in TM that is a diffraction in tem before I get into this in a very exhaustive topic in TM I would like to just recall some of the principles we discussed about diffraction in x-ray course are an x-ray diffraction lectures see the there is no difference in those principles for whether in terms of you know diffraction condition we talked about a reciprocal lattice we talked about a bolts fear concepts and then all this phenomenon whatever we discussed they are all going to be the same in this electron microscopy.

As well and then another important thing is though you have to just find out what is the fundamental difference between the electron diffraction and an x-ray diffraction you yourself will no doubt that only the wavelength is the difference because with increasing the accelerating voltage we will be able to control the λ that is what we have seen in the beginning of these course so through a B blog list relation and in x-ray we have a much higher wavelength.

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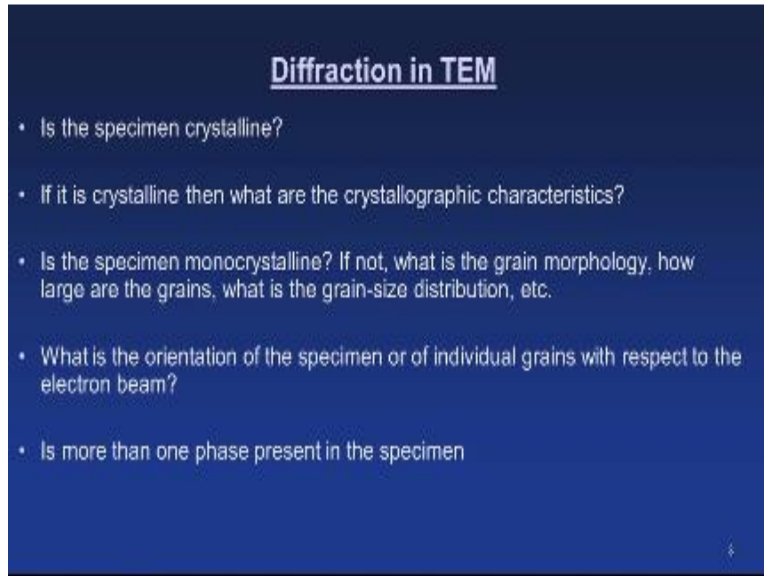

So that is one basic fundamental difference otherwise all the diffraction phenomena are same so with that background you will be able to appreciate some of the concepts in TM or so I will be moving little bit faster in this lectures I do not spend much time because the most of the concepts are same but I will spend much more time in some of the important diffraction experiments like converging beam electron diffraction or you have the kikuchi pattern and so on which are very important diffraction experiments in transmission electron microscope.

I will spend little more time on that otherwise they are all the same so if you look at the diffraction pattern in tem you have to just ask few questions suppose if you ask what is it and what can be learned from it why do we see it what determines the scale what determined the distance between the spots are the positions of the lines. So something like this if you try to answer these four or five questions you will you will see that surprisingly you will get lot of information about the specimen characteristics.

So some of the related questions is the specimen crystalline if it is crystalline then what are the crystallographic characteristics is the specimen mono crystalline if not what is that grain morphology how large are the grains what is the grain size distribution electron what is the

orientation of the specimen or of individual grains with respect to electron beam is more than one phase present in the specimen so some of these basic questions related to what i mean I have put some fundamental questions in the previous slide they are all related and.

You will be able to get all these information is from the diffraction so you have to remember in a transmission electron microscopy diffraction is most important in fact the whole microscopy operates with the I mean operation lies mostly on the principle of diffraction so you have to give an importance to the diffraction analysis when you really want to use this or exploit this TM.
So I will I will talk about the importance and much more when me I mean I actually give a practical example as well.

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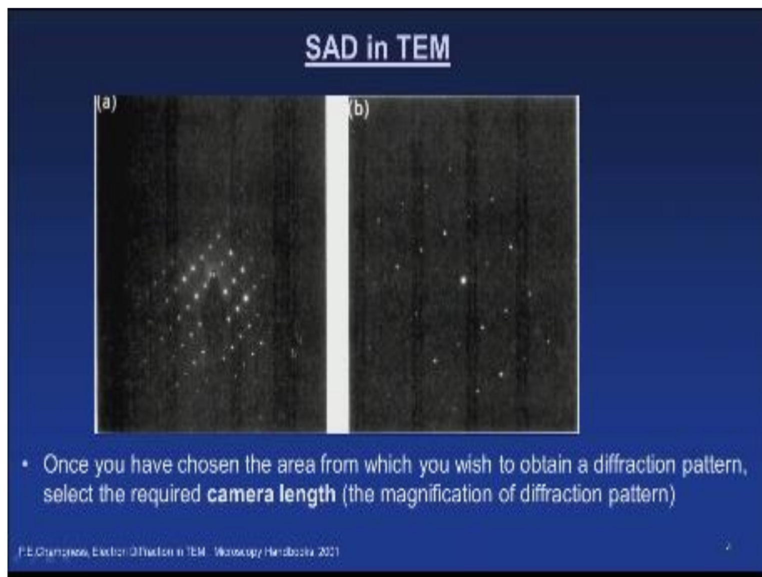

First look at the typical is sad in TM that means selected area diffraction in TM so what is shown in this photograph so this is a typical selected area diffraction taken from this textbook once you have chosen the area from which you wish to obtain a diffraction pattern select the required camera length that is the magnification of the diffraction pattern so the camera length is also called you know where whenever you turn this magnification knob in a diffraction mode that camera will I mean the camera length will increase our decrease.

So this typical diffraction pattern is taken at two different camera length you can see that this is the orientation of the two patterns are same but only thing is you can see that the distance between the spots are bigger that means this diffraction pattern is taken it one camera length and this diffraction pattern is taken at much a little more higher camera length so that is what it is.

So you have to choose a camera length of your interest that is what it is shown here and then I as I said before we have before we do all this and that your camera length need to be calibrated and then you choose appropriate camera length to do the experiments.

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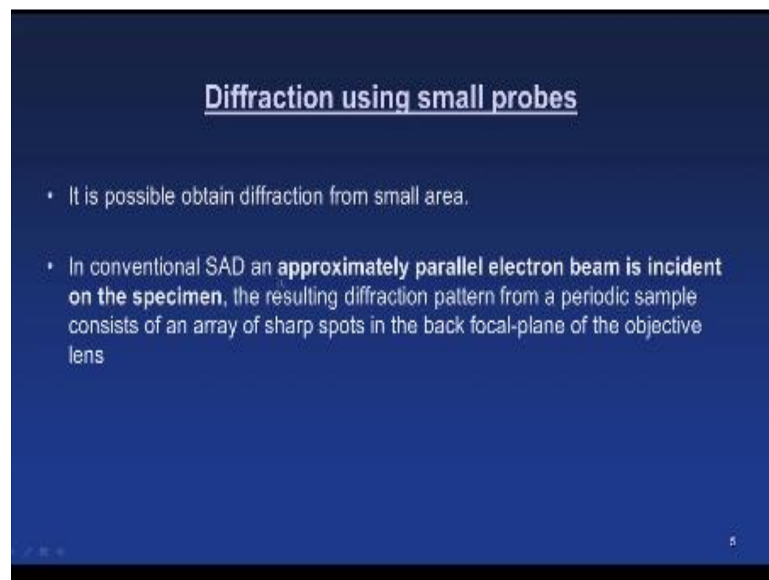

So diffraction using small probes it is possible to obtain diffraction from small area in conventional selected area diffraction and approximately parallel electron beam is incident on the specimen the resulting diffraction pattern from as periodic sample consists of an array of sharp spots in the back focal plane of the objective lens so as Introduced using that converging beam electron diffraction I said we will be able to do a microprobe or a nano probe.

That means you can make the probe into a micro micron size diameter or a nano size diameter we will also see the meaning of what I am saying now how what is the meaning of I am seeing a micro micron-sized a beam on the screen or at nano meter size screen and beam on the screen what is the physical meaning of it that we will see but then we can obtain a diffraction information using this either a microprobe or a nano probe from a localized region of our interest.

That is what we are discussing here in a conventional SI d as i just mentioned if when i when i talked about converging beam or a parallel beam I said that a parallel beam operation is important to in order to get a sharp diffracted spot so that is what is shown here in a conventional said so a parallel electron beam is incident a specimen where you get a very sharp pattern.

Whereas in a in a small probe a converging beam has to be used so we will continue this discussion on the diffraction and its effectiveness and also what are the details you are going to get it is very powerful a tool or I would say that the powerful technique to obtain a most information about the material characteristics we will continue in the next class thank you.

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