Indian Institute of technology Madras
Presents

NPTEL
NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Lecture-40
Materials Characterization
Fundamentals of Transmission Electron Microscope

Dr. S. Sankaran
Associate Professor
Department of Metallurgical and Materials Engineering
IIT Madras
Email: ssankaran@iitm.ac.in

Hello everyone welcome to our transmission electron microscopy laboratory today I am going to talk about transmission electron microscope.

(Refer Slide Time: 00:22)

let us hear what you are seeing is a 120kv transmission electron microscope in the transmission electron microscope so we are using an electron beam to form the image because of the shorter wavelength nature of the electrons you are getting a high resolution in this transmission electron microscope for this purpose only we are going to electron microscope this part in this transmission electron microscope is a gun part in this gun we have cathode assembly and then a node is there the here cathode is a filament we are using lanthanum exuberant and tungsten filament.

(Refer Slide Time: 01:10)

By heating the filament we are getting a bunches of electron these electrons are oscillated from the gun part to the anode okay.

(Refer Slide Time: 01:14)

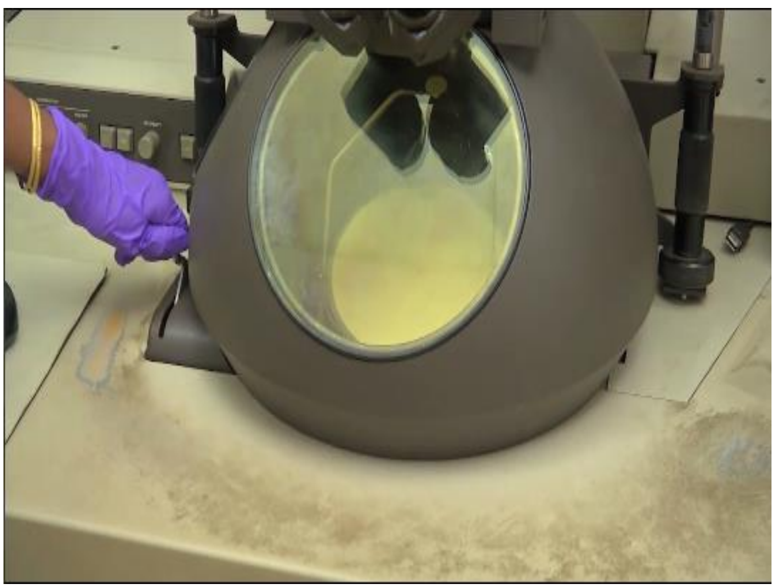
So from my anode it has to pass through the column to the bottom of the column okay so for that this electron should be were oscillated with a high speed because the electrons are pausing through the column in the column is under very high back up otherwise it will interact with the air molecule there should be a scattering effect to avoid that the whole column is under high ultra-high vacuum.

So from this here the top is cathode assembly is a the bottom is an anode from this far to here we have lenses are there the lenses are electromagnetic lenses so by applying a current you can change the strength of the electromagnetic field by change changing the strength of the electromagnetic lenses you can form the image and magnify the image okay, so in this particular CM total transmission electron microscope they have introduced 4 elimination lenses that is condenser one condenser to mini condenser and condenser objective lens.

Then from here to here all image formation lenses and magnification lenses are there so from this part we have an objective lens and then intermediate lens and diffraction lens these three will form the image as well as the diffraction whenever the electron beam hit the specimen you will get so many signals some of the electrons will pass through the specimen some of them will get scattered to a particular direction when it satisfies the black condition.

So that is called electron diffraction this will come from here to here by using this intermediate and diffraction lenses this image and diffraction is forming in different planes that is always the diffraction is forming in the back focal plane of the objective lens and then image is forming in the different plane so if you want to project the diffraction pattern you have to project the back focal plane of the objective lens by using a projector one and two lenses, okay if you want to see the image plane you have to project the image plane and then you will get the image in the bottom.

(Refer Slide Time: 03:28)



So here what you are seeing is a screen this is the fluorescent screen if you see this there is a disk in that that is the fluorescent screen so you cannot able to see the electron image with your eye so for that this electron should hit the spell fluorescent screen immediately it will glow then you will see the image details okay.

(Refer Slide Time: 03:48)

So from this we can capture the image which is here we have a CCD camera with this so that the image you can see in the monitor apart from this magnetic lenses the electron beam should pass through different approaches some of them are fixed at aperture some of them are changeable aperture you can mechanically change the aperture so here this part is a condenser aperture part we can change the size of the condenser aperture by using this condenser aperture you can reduce the beam diameter okay.

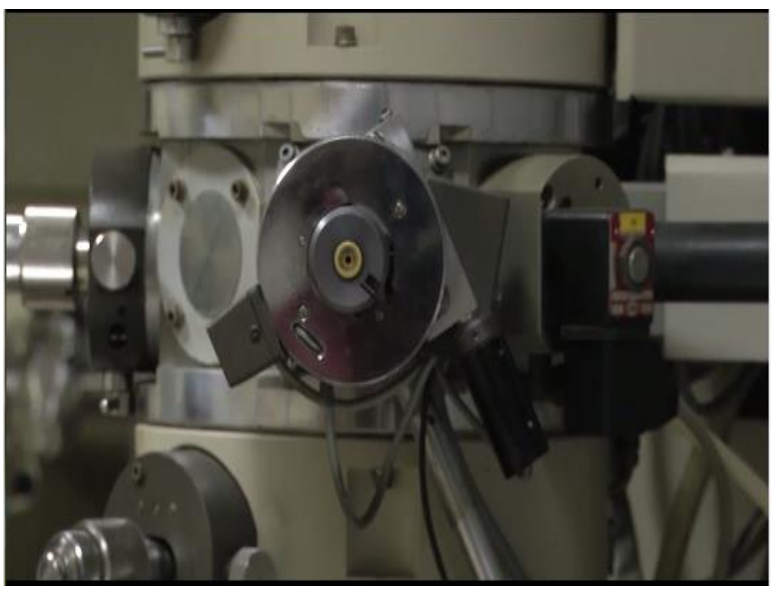
But then this is an objective aperture nothing but we used to call this as a contrast aperture we used to change the contrast of the images this is called selected area diffraction aperture by using this you select the area of the specimen and then you can get the diffraction of this particular specimen, so by using this transfer electron microscope we can get a structural information the slow graphic information as well as the chemical information okay the chemical information you can achieve by energy dispersive x-ray analyzer.

Okay by using a characteristic extra energy you can detect the elements present in that how you are getting is when the electrons with the specimen the x-rays will come out from the specimen okay there is each element has a characteristic extra energy that can be detected by silicon lithium detector so silicon lithium there is a single crystal silicon on that lithium is doped the TM is a very light element.

So it will be very mobile in nature to hold it in the silicon crystal it should be cooled with liquid nitrogen temperature okay so from this is a very important point in the transmission electron microscope the electron beam should oscillator heated by with the filament the electrons will bunches of electrons will come out and then it has to come through the lenses through this upper share and then it has to pass through the specimen.

Okay the beam should pass through the specimen this portion is a specimen inserting portion this part is called a goniometer.

(Refer Slide Time: 06:02)



So if you see this is the specimen holder because the electrons is passing through the specimen your specimen thickness is very important you have to use a ultra-thin specimen okay so for the different methods are there to prepare the specimen so your specimen should not exceed 100 to 150 nano meter thickness other ways the electron will stop here itself okay to avoid that you have to make the specimen to 100 to 150 nanometer thickness by different methods for a conductive material we have to go for electrolysis method and then for nonconductive material we used to go for ion milling by ionization process will turn the specimen for your powder sample.

We used to disperse the powder in on the slough grid that is called carbon coated copper grits okay so this whole instrument because we are using an electromagnetic says the whole is get heated up to avoid the heating up it will be circulated with 120 degrees chilled water we have a chiller in that okay so this here I told this whole system is under high vacuum now I am going to show you the control controls of this transmission electron microscope see here the knobs are there this now basic intensity knob you can converge and diverge the beam electron beam.

Okay these two knobs or for tilting the specimen in both the direction by goniometer tails as well as the holder tilt say this is the specimen holder for transmission electron microscope.

(Refer Slide Time: 07:35)

What you are seeing is a single tilt low background holder this is a double tilt Holden so by using this holder we can rotate the specimen but that is a goniometer rotation and then you can tilt the specimen in the easy access direction okay the tilting of the specimen angle is shown here this is a heating holder.

(Refer Slide Time: 07:58)

by using this holder you can heat the specimen inside the microscope you can continuously observe what is the heat treatment effect in this is a training holder you can give this you can strain the sample inside the microscope okay well observe a observing you can strain the sample and see the effect of that straining so we have two screws are there you loosen the screw and then you have to put the specimen here and then you can proceed.

The experiment this is a multiple specimen holder by using this holder you can load the specimen three specimen together and then you can view the specimen one by one this is a single tilt holder which I have loaded a aluminum foil see here you can see the specimen in this here the goniometer is there so I am going to insert this specimen so because I disturb the vacuum here chamber so now it is evacuating this specimen chamber this chamber is a specimen chamber if you observe it so it is now the pump is running.

So now we have got evacuated I am inserting the specimen holder inside the microscope these are the controls here see this is the layout of a vacuum system we have a rotary pump oil diffusion pump and get Ryan pump okay all our microprocessor controlled everything is isolated with pneumatic valves the pneumatic valves we are using a tie our compressor to control this pneumatic valves okay if you see this portion is a control panel okay so you go here and then you see this is a high tension knob whenever you want to work in the microscope you have to switch on the high tension.

So the selection of high tension is very important it depends on your specimen so in here in this on 20 carries the maximum voltage you can use it that is an oscillating voltage so the depends on the kb what you are using your resolution is determined okay then this part I have already selected a 120 kb then what you have to do you have the heat the filament so thermionic emission okay.

(Refer Slide Time: 10:16)

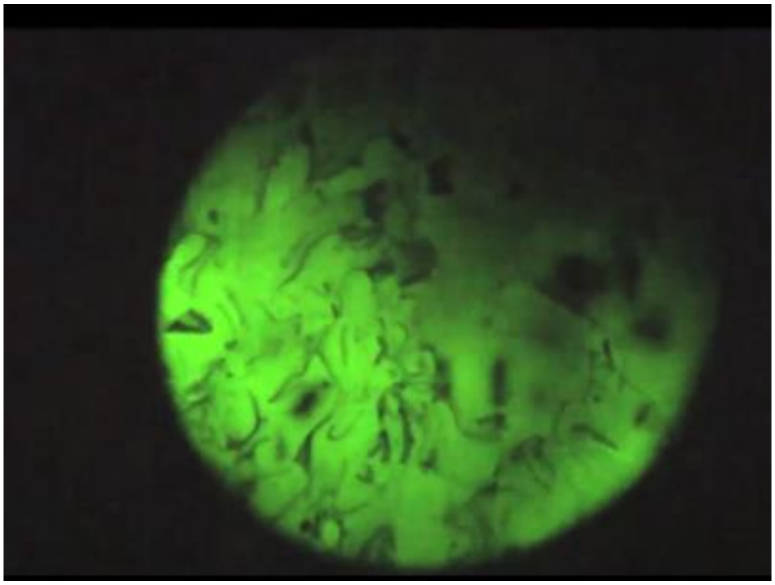
So what I am doing is I am heating the filamentous II there is just I am heating the filament you have to wait for until it is get a maximum electrons oh okay then from here you see here so many knobs are there so this is for magnification knob you can in this at cm tall microscope we can go to 8 lakhs20,000 magnification so this is a diffraction knob when you want to project a diffraction pattern from that a back focal plane you will switch on you just press this you will come to the diffraction mode these two or the beam shifting XY direction we can check the beam from X Y direction then this knob is a focusing knob this is a step size this is sine focus.

You can change our course for coarse and fine you can make it so this post portion is called a multi-function knob this can be used for different purpose if you have an astigmatism problem you press this pattern stigmatize button you can adjust with this you can adjust the stigmatism correction so this is called a dark field image that I will show you in the monitor how the dark field image is coming though by pressing this you can tilt your gun slightly.

To the diffracted beam okay this is called an alignment because you have a very linear column you have to align the beam for that different alignment procedures are there starting with gun and then after that column and then everything from here to here so many lenses you can adjust the current and then you can align it and make her proper beam should hit your specimen.

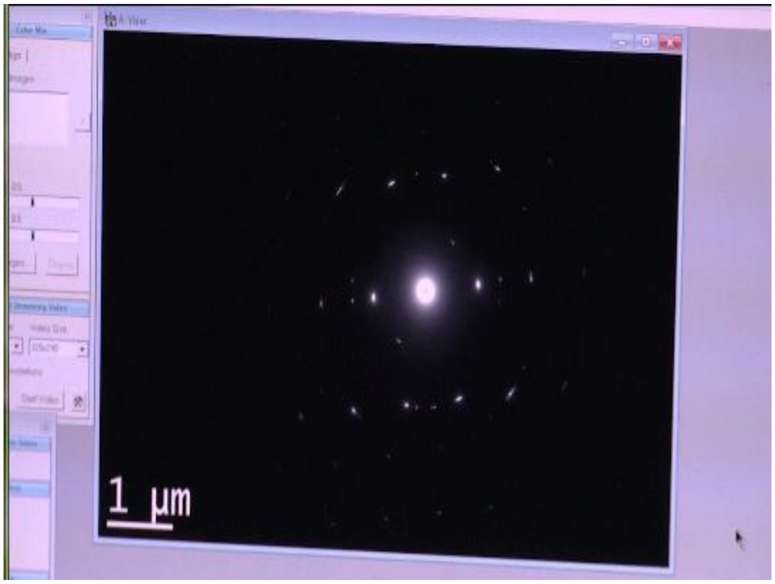
I have already heated the filament so the electrons are coming over from the filament by heating the filament that is oscillated by height near high tension 120kv then it will come towards the anode okay here all the lenses are positively charged so the attraction is towards the lenses and then you are seeing the image here now in this now already I have loaded an aluminum foil I am just going to show this aluminum foil really.

(Refer Slide Time: 12:29)



The images from the aluminum foil you are seeing the images on the door essence cream then that will be seen in the monitor this is a objective of aperture I think maybe we used to call this as a contrast aperture what you are seeing this images without aperture when I insert the aperture you will see the difference see the contrast difference in this what you are seeing is a image of where aluminum foil okay then with this unit you can get the crystallographic information this is the selected area diffraction aperture see if we insert you can select the area from the image.

(Refer Slide Time: 13:12)



I have reduced the aperture size then again you can go to still slower aperture okay so the purpose of this aperture is you can select the area and go for diffraction this is a diffraction now if you trust this selected the area will give a diffraction pattern this is a diffraction pattern from the selected area when you select the transmitted beam to form the image that we used to call as a bright field image to form the image that is called dark field image I am using the transmitted beam to form the image this is called bright field image this is slightly out.

I am using the focusing knob to focus that be nothing but I am adjusting the objective lens current OC by using this translator control now moving to the moving in the y-direction the other side control is for X direction this area is darker some of this area is lighter so well de electron beam passing through the specimen some of them will get scattered nothing but get diffracted so those areas are less electrons hit that hit on that place here more electrons are hitting on the specimen okay because of that it is looks darker this is brighter in color okay so this is why this if you take a diffraction from this area you will get a exact orientation so this is in orientation this is in another orientation no I am again coming to the diffraction mode, so the previous one I believe that image is formed by the transmitted beam.

So by using an objective aperture I am going to select this be okay so now I am going to select any one diffracted spot if I say this image some areas are getting highlighted this is darker in color ok so what is happening is these areas are correspond to that particular plane particular diffraction pattern okay this will represent the particular diffraction spot okay so if you have any defect or anything so we can find out the particular plane where the defectors say by indexing the action spot okay this image is called dark field image.

Okay when you use the diffracted spot to form the image that is called dark field image if you use a transmitted beam to form the image that is called bright field image say some collection of x-rays are here this corresponds to energy versus intensity okay so what I am going to do is I am going to find out the elements what is that element see it belongs to aluminum so I am going to do this aluminum okay it is a compare commercially pure aluminum and then some copper unless this is a copper pick a point.

So cropper you add it okay so you see here now we can see this raising a you have to give sometime for the collection okay and then I am going to stop it then you quantitative both the thing you can do it here so I am going to quantification I am going to the quantification of this elements present now you see this is a weight percentage and then atomic percentage both are you are getting from this EDX analysis energy-dispersive x-ray analysis.

(Refer Slide Time: 17:29)

This unit is there 200 kv technique unit okay this is also a transmission electron microscope working in 200 kv that is the only difference from the CM 12 so because using a 200 kv as an oscillating voltage your resolution is more higher okay because of the higher voltage you're getting a shorter wavelength then the resolution is very short that is point 2nanometer level you can resolve the specimen okay so the same working principle is same here same electron gun is there the emitter is a lanthanum except alright.

(Refer Slide Time: 18:00)

Then by hitting the filament if you will get an electrons that will be oscillated by in high kv200kv then there is an anode then through that the electrons is passing through different lenses or lenses are electromagnetic lenses apart from the TC type of condenser aperture objective aperture and then selected in a diffraction aperture all this thing electron beam is passing through these approaches so this portion is a specimen chamber same single tool to holder I have loaded here so the electron beam should pass through the specimen and then we will view the specimen in the fluorescent screen okay.

So the same way there is a CCD camera is there you will capture the image in the CCD camera and then you can see this in the monitor okay some controls are different here I will just want to show you that so here the left side control panel is for this there is a knob this is africa are controlling the intensity and then there is a stigma to control in control is there is a multi-function knob that is as in cm12 we can do there is astigmatism correction and then you can check the beam everything you can done with this and then there is a track ball by using this you can share move the intensity of the beam XY direction there is a tilt control that is by tilting is this is a goniometer total condition.

Now this is a holder tilt knob so these things are optional you can select l1 l2 l3 alone is 14 l2 is reset focus and then l3 spot size reducing the spot size that is so this all are this is an optional by clicking it you can select whatever you want in this okay you see here all these things are intensity zoom all these things are they you can select or whatever you want this is a multifunction in X direction then if you want to increase the magnification you have to use this knob to increase the magnification the same way course and fine focusing knob and then we when you press this will go to the dark field imaging if you want to go to the diffraction you can press this knob all this bobler and your synthetic focus is there so for a while tilting your area of interest should not go out of the optical axis.

For that you have to do the eccentric correction for this we are using these two knocks this one is also optional r1, r2, r3 you can select according to that so r1 is for screen dim we have selected or two is where α hablar are three is for sports size that is beam diameter reducing you can go to a spot 12 like that increasing order so increasing the diameter you have to go to the reducing order.

(Refer Slide Time: 20:55)

This is a vacuum layout of this microscope there is a priori pump and then I GP pump to pump to I GPS are there because it is a very big column and then we have oiled efficient pump is there so this is 10 power minus seven to eight are you can achieve from this for thing after getting the vacuum status you will see the values numerical values here we know it should come to gun chair should come nearly 23 then we have already selected high tension to 200 kv this is the 200 kv electron microscope.

So in 200 kv only you will get a maximum resolution then after that I have to heat the filament just pressing this knob now the filament is getting heated up ok you have to wait for some time there is the way have already given sometime delay so it is getting heated up okay they are here if you see this, this is a magnification in at present I am in 2lakhs 50,000 magnification there is a spot size all these data sorry given here by using this now we can change all these okay.

(Refer Slide Time: 22:07)

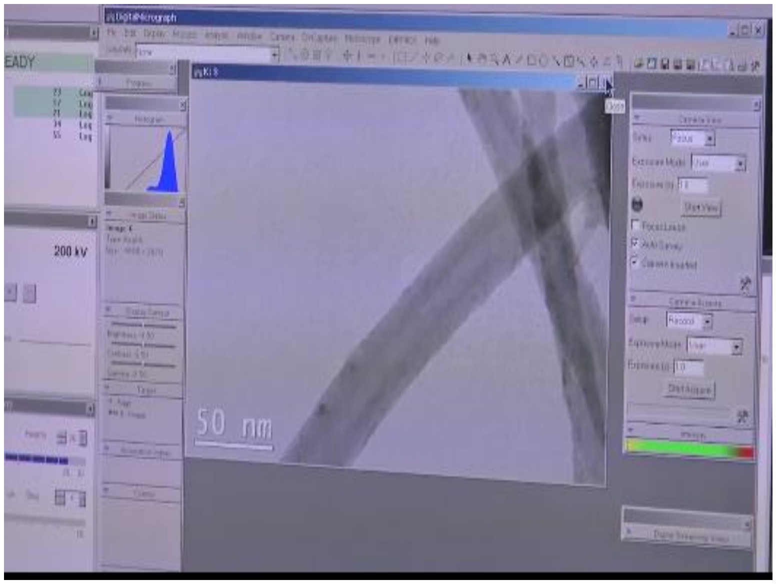
We have to wait for some time to get the filament heat what you are seeing is the image of your specimen so this is the folder sample say I have mentioned in cm12 there is a copper mesh on that there is a fine coating of a carbon film that is a few nano meter thickness on the carbon film we disperse the powder if you see this there is lines are there that is the mesh image okay that is a shadow of the mesh.

So in between you will see the squares inside the square you are seeing something some black black things or they know that is your specimen say this is a joystick you can move the specimen from X Y direction okay by using the joystick you select the specimen place okay so by editing by moving this X Direction y direction like this you can move all the in all direction ok this is the joy stick see now I am selecting this area okay right.

So what I am going to do is see I have selected by see this by moving this here and then here very wet wherever you want you select ah by using this joystick ok the whole screen you can move like this like this okay right now by using this l1 button I am ripping the screen then you will see their image here this is a CNT powder carbon nano tubes powder so what you have to do know you have to select any one particle by increasing the magnification.

You see I am increasing continuously, continuously like this in this position you have to insert this aperture objective aperture to get a better contrast then you further increases you select any one fiber and you will see the difference that is what I am going to do is by using this no increase grace further increase further this is a carbon nano tube the distance is 200 nanometer this is a magnification of this carbon nano tubes I have encouraged it and focused it I got this you see here then I will go for further magnification okay say here further resolution nothing.

(Refer Slide Time: 24:38)

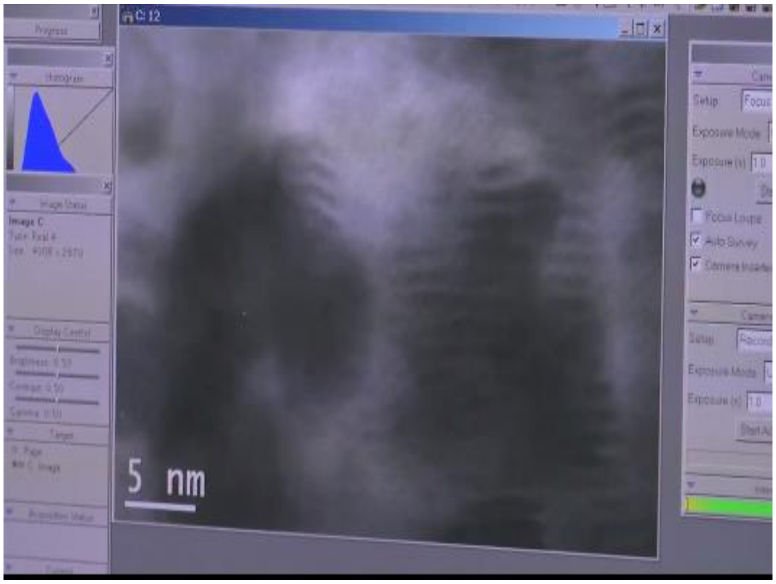


But further reduces resolution your this distance is 200 nanometer you will see the increasing the resolution this is 50nanometer okay we'll see this, this is a carbon nano tube and then when I am going further to that you see that this is a 5 nanometer image see here this is a fine animated image what you are seeing is a lattice fringes okay.

So this is a carbon nano tube so the distance between these two lines point three four angstrom so you because we are using a 200 kv because the resolution is high that is shorter the wavelength higher the resolution okay because of the higher voltage so this is a carbon lattice ok then if you see further see here this is again interest in high, high magnification then again come down so this is slightly lower magnification this is an for 490xkx nothing but this distance is 10nanometer ok then if you see here see this same 10 nanometer area.

You can see this ok so here this is a lattice imaging this is the advantage of this 200kv transmission electron microscope even if you go to 300 kb you can get even in the order of point 1 nanometer level even further with the Fair field emission gun you can get the atomic resolution is possible okay I am showing it this is again 7 700c again I am showing okay this is scene 700 I just want to show some of the photographs which I way have already taken with different samples ok just I am going to show you this one see here you will see this, this okay because it's a two-phase alloy sample okay this a minute we'll see the lattice of this tool addresses .

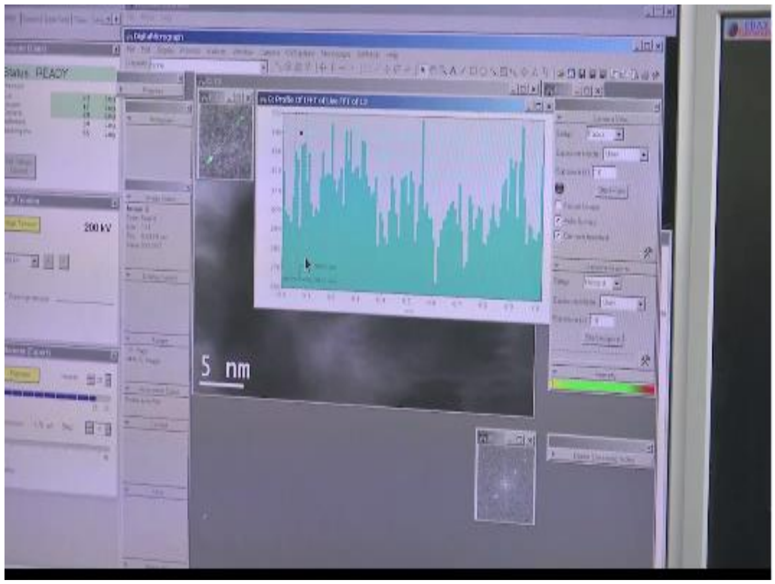
(Refer Slide Time: 27:02)



We can say you can able to see it okay then we go here we see this, this is a lattice imaging okay so in this by using this we have we have 50 facility in this, this is called an FFT facility go to live f F F of T so you have to select any one area by putting an alt and this you have to select this Square and then select the area like this okay sorry it was not clear okay then go to process when you go to live effect f of t will get something here it is almost identical your diffraction pattern okay so this distance you can easily find out even if you if you move this you will see the difference in the diffraction pattern this is see it is in two phases or they presume it will see to learn two spots will be there here so if you go here also you can see this okay.

So if you go to this go to f of T then go 10 verses see that if distance between these two lines okay there is a fine lines so when we have a profile is there okay. So when you measure from this to this line okay you are getting a graph of this then you will see the spacing of this each line the small lines are there no so you will see the spacing of these lines so you can do like this you can find out so it should be see here you have to select properly otherwise you will not get the proper result.

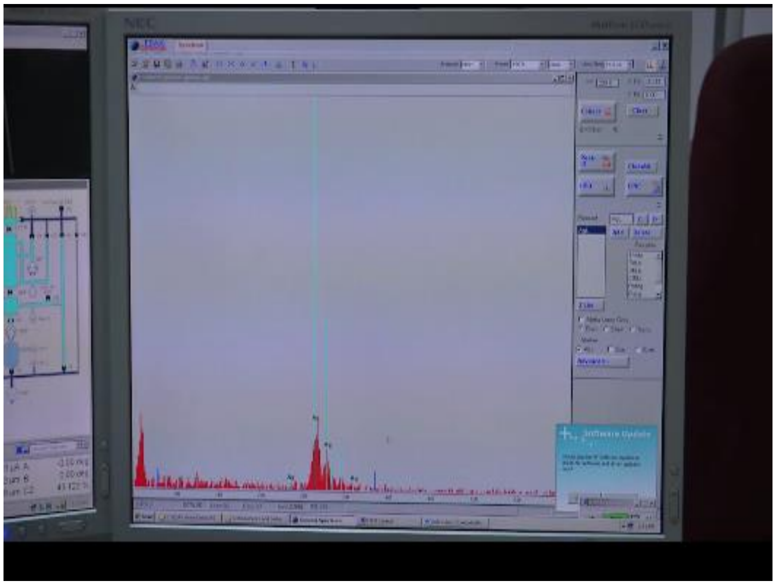
(Refer Slide Time: 28:40)



So you have to select this see this is she here from here to here so we are getting this, this is the value of Phi where you prop you have to select properly this is your resolution of your spacing see here it has given here okay. So from here to here if you observe I there but it is very fine so because of that we cannot able to find out because till 2 nanometer only we can easily resolve it because it is lesser than that it is a in diems silver alloy something they have the one brought it.

So this is the resolution you can find out by using this F of T option this is taken from our microscope this is a fine animator that is a maximum resolution you can get it even lesser than point2 nanometer we are resolving here this is the advantage of 200 kv electron microscope if you go to 300 kb even one angstrom resolution is possible.

(Refer Slide Time: 29:36)



This is an energy display with dispersive x-ray analyzer as per in cm tall the same configuration is there we have a same silicon lithium detector to detect the x-rays that by using a characteristic extra energy, energy via ray we are detecting the elements so the spectrum develop this belongs to silver okay so then apart from that some of the elements are there here we have to locate it some copper is there here see copper is a so you load it okay.

By using this you can quantify it they are qualitatively as well as quantitatively you can achieve the results by using a energy dispersive x-ray analyzer so you have to go for quantification you will get these things so this presence of elements element are silver, silver is there copper is there then you can quantitatively you can I find out the elements present in the particular sample. Thank you.

IIT Madras Production

Funded by

Department of Higher Education

Ministry of Human Resource Development

Government of India