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Kanpur

NP-TEL
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on
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-06

by…
Prof. Krishanu Biswas &
Prof. N.P Gurao
Dept. Materials Science & Engineering

So in the last class we have discussed about the high resolution electron microscopic principles and I said that we will discuss about the MA simulation today as we know that high resolution electron microscope P gives you images of the atoms or columns to the resolution level of Angstrom or strong even it is possible to obtain resolution level of 0.8, 0.7 Angstrom in the microscopes available today the problem is that interpretation of such high resolution images are really difficult or daunting task.

Because image contrast can vary tactically depending on the focus as we know as we have I shown you that the focus is the most important factor in obtaining the high resolution images in the times in electron microscope?

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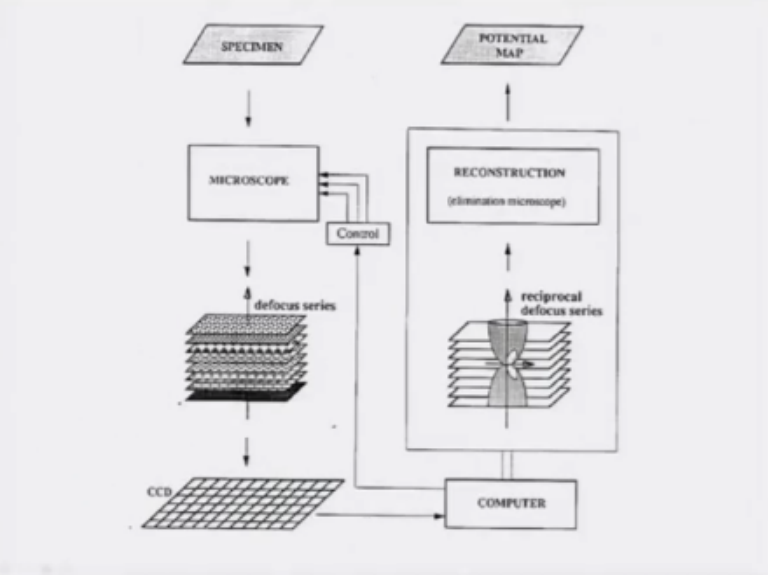

So therefore during the subsequent process after receive the images from the microscope we need to simulate it using the prescribe models available for the crystal structure of the material if it is a crystalline if it is a non crystalline material then we must have prescribed model available for the structure of the non crystalline material like amphora's or any other structure so during as you know during imaging process the electrons undergoes 3 distinct interactions.

If we have to list down in a very short form and each of this interactions can be simulated in a computer so first thing your first interaction electron undergoes is called the basically because of this dynamical scattering of the electrons in the sample or the specimen and this basically this can simulated the dynamical scattering is basically because electrons falls on the sample and then can get scatter in the different direction and this is basically it is a dynamical process.

So one needs to apply the dynamical diffractions theory to obtain the exits way functions of the electrons now this can be done using technique called multi slice method or in this method actually the specimen is sliced into many slices or actually specimen is basically taken into many different in a small slices and then this slices is basically taken at a normal to the electron beam I arrange the beam.

And then this after we obtain this slices we can simulate the electron interaction dynamic interaction of the electron with the material by using different methods the methods like reciprocatory reciprocal scape formalism or fast furious transformations or real space approach or block curious back pro is depending on how precisely want to remain the interactions now I do not have time to deal into talk into each of this techniques very precisely.

Because of the time constrain but now a day's any conversational text book on the transnational electron microscopy will provide you all the knowledge available for this techniques the most import technique which we day to day apply in the high resolution microscopy is called fast furious transformations in this technique we basically use the actual high resolution image which showing the collapse of the atoms as a real space image and then furious transform the this real space information and obtain the refraction pattern.

The compare the diffractions patterns will be the once which is can be derived from the structural model available provided we know this structure of the material very precisely and the comparing it we can get many dusting information regarding the interaction of electrons with the material and input to such a multi slice method obviously has to contain different parameters of the specimen of the object.

Like unit cell the position of the atoms and insert the unit cell the what is called terminal factor dual factor because of the privies the atoms are set the temperature increases and the orientation of the specimen or orientation of the crystal are the object as soon as the thickness so this itself is a basically not a easy situation as you understand there are so many parameters which goes into the simulations.

And if we do not have dusting information or dusting values or this parameters like thickness specimen the object orientations values of dual of factors very precisely for the material which is turning we do not get very in a good simulated picture now results of this calculation actually gives us the way function as we know wave function is these important result of this way function as they exist plane of the sample is obtained by using the multi slice method and in the second step once you obtain is wave functions.

The formation of the images in electron microscopic are the tendencies to microscope can be simulated using different equations which I have discussed this equations actually based on the transport functions so transfer functions actually are to be know a priory for a particular set of microscopes once you know the transfer functions and related equation how this the points in the pristine is getting transferred to image plane.

The we can basically obtain the instrumental factors in the image so therefore in this simulation second step simulation one is to provide all the information regarding the instrument the

microscope which you are using fro obtain high spectrum images and this included obviously the spherical operation constant they refocus as well when if it is possible to give the information regarding the currents of the different lenses.

Subsequent of the objective lens finally in the 3rd step electrons intensity of the electron beams which are coming at the image plane because of this interaction can be calculated by the squaring the wave function and this can be displayed like a monotone are halftone image basically halftone image on a high resolution screen on computer so these stay 2 steps are normally done now a day's in all kinds of computers available where the commercial software's actually allows you to run this different steps.

And provide you actually the image which is can be compared with the actual image now in practice this is a actually routinely done in the even while doing the microcopy one can turn this things one can use this functions which are available in the software in the compute and compare and get information's or otherwise if the structure looks to be very complicated then one needs to go back to structure modify it and then obtain images which can be then compared.

But you know these simulations obviously are dependent on many input parameters as we have seen the most important input parameters arte the specimen and the microscope itself now as for the microscope is concerned one can actually give this input parameters very precisely there values of the CS or the western constant or even the focus values can give provided but as for the specimen is concerned.

This is very difficult provide the actual values of the input parameters like you know orientation of the sample the thickness of the sample where the image is taken and many was called other parameters so that is why many people actually what they do they initially obtained this information or measure the thing the thickness of the sample very precisely using Carmon electron diffraction technique or all of the technique available and also determine the orientation of the sample very precisely and use this is as input or précised values of these used as input to the simulation so that one can obtained as called good images for reasons you know this are sometimes available for reasons of sometime.

Now available to do the simulations if you are lucky enough we are having good microscopic team then you can guess this information very easily from your experiment own experiment or

form your curly experiments doing the whole set of terms like a microscopy and then you can run this simulations at the same time for the machine as I said it is possible to provide the precise information of specula water in constant are defocused values, but many times we do not relay on that the defocused values which are.

Provided where rather what we do is that we take a series of defocus images from the hallow set of microscope and then compare it with the simulated images this is, so therefore in a nut shell I could basically show you simple in a simulated picture in the slide where this is the specimen.

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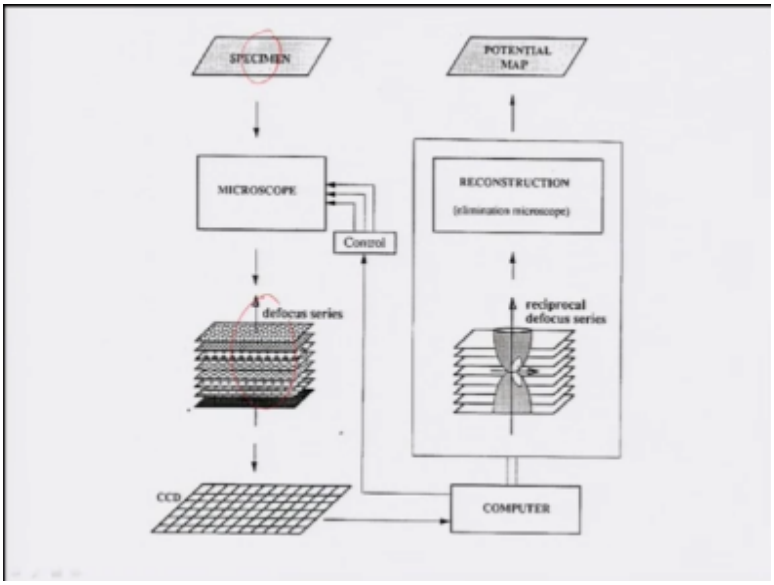

You see their electrons from the gun falls on the specimen after going to do different you know lenses and then interacts interaction basically dynamical in nature and then is basically passes through the other lenses in the microscope like intermediate lenses and finally obtain a image in a CCT camera and to make like this simpler we always get a defocus series of images where

defocus is varying of very what is called systematically and images are obtained as black and white dots as you seen.

In some of the pictures and this images where there front into the computer one this is in a front of the computer, computer has the software in belt and this software's are basically runs based on this three principles which are just now told you and then reconstruct the original image by comparing with the, the model have a level and finally it gives you a potential map, so that is actually the cycle in which we similar the images.

And obtained finally most comparable image what do you understand from this whole process because there are lot of input variables which can be changes at the few variables and also the compulsion of the whole technique tells you that it is not an easy one to basically get information out of halls in images that is why now -a - days people provide this images in the different papers of the books just for the sake of increasing the quality of the images many times we find that the exact information from this images.

Are lacking in the both in the papers and sometimes in the books also because of this problem of simulating this images and getting the quantity based on the machines, so obviously qualitatively one can explain the images using software but obtain the quantity information requires and another kind of simulations which are more complex and probably outs of this scope of this particular course, so I will not going to tell of this but just to give an idea that how complex is this you know simulations.

And just telling with this aspect that be careful while comparing the actual images were simulated once sometimes this may be miss leading so therefore you need to first run how to run obtain good image of the electron microscope like good ones in such a straight hand and then compare this images with the best possible software available in this world to extract quantity information about the microscopes, so this I close the hallows electron microscopy portion of this course and I move on to the next portion of this course that is called stem scanning transmission electron microscopy.

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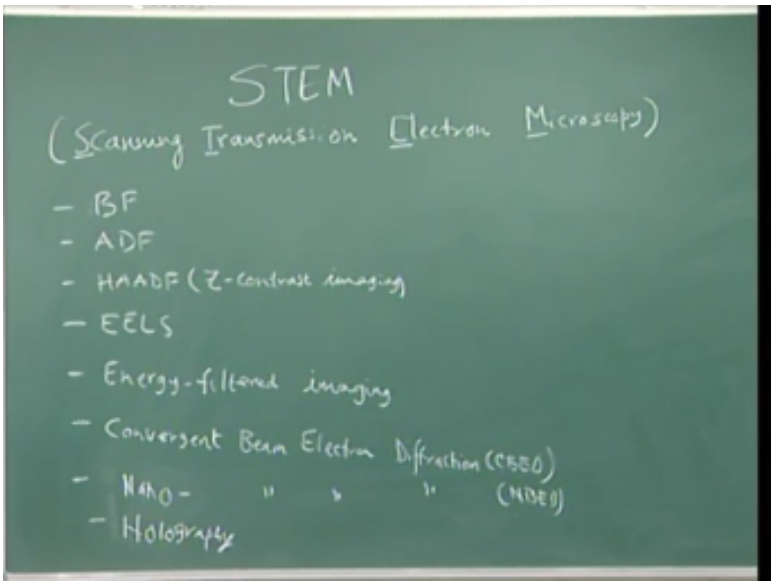

As you know scanning times electron microscopy has become an integral part of the microscope now- a- days all the modern day microscopes has scanning electron microscope facility scanning times electron facility this is has originated from work by Crowley crue and many others, and those who have tried very hard to use scanning terms electron microscopy the reasons scanning microcopies is important because of these aspects we can get information's for bide field and duck field images.

High angle duck field high angular duck field images which can gives us jet contrast we can also what is called we integrate this stand with yields energy filter are energy electron and the loss spectroscopy which can be used to obtain energy filter images one can also integrate this with convergent electron beam or nano electron beam diffractions and then one can actually use the stem to get hallow graphic images, so I will not be discuss all of them in this course but I will try to give an idea how steam works.

And stem can be used to obtain different sets of information's as I said the concept of scanning turns electron microscopy is start a new one it has been employed by crue way long back now who introduce this whole concept into the electron microscopic community and he actually used first time showed that first times scanning electron microscopy unit used most they prelims in guns so in a scanning terms electron microscopic images what we basically do is like this we have a basically a fix source FEG source.

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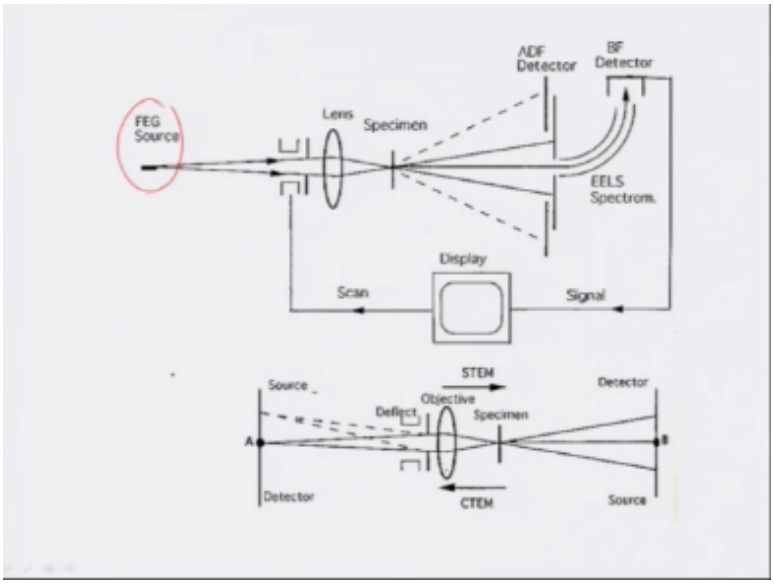

Or FEG source what we can say there is a very high brightness and very small slangy spread this FEG source can be focused by using the objective lenses on the specimen obviously the FEG source is focused on the specimen by using these this kind of objective lenses the beam will be de magnified electron beam and basically with this beam is called convergent beam and once the convergent is falls in a sample or the specimen it diffracts and gives you diffraction disk this diffraction disk can be either.

Transmits to termite electron beam are diffracted beams now one can actually use different detectors they allow the specimen to obtain the images using this or this is called either transmitted beam or the diffracted beams that is the basically idea so it is shown very nicely here in the second picture where you can see this is source electron beam which is then it is can be scanned or what the sample there by using a deflector which is their normal scanning electron microscope.

And this images fall this beam is falling on the objective lenses which is focus is or de magnified the electron beam to a very small size beam or convergent electron beam on the specimen and then specimen diffracts and we can get different cancel information's which are listed there, so this is basically the idea now has you understand that you know as the convergent beams falls in the specimen and some part of this convergent beams will be diffracted and some part will be transmitted.

So if one collect this diffracted the bean intensity use a detector one can form duck film images or if one collect these transmitted beam using a detector one can actually displays as a bade film images so and then once this beams scan so the sample and this whole imaging can goes on as a speed of aster it is just like a scanning electron microscope and scanning electron microscope and scanning electron microscope the raster scans of the electron beam and signals like back scared electron or the second electrons or whatever are the signals generated this signals are then used to display the image on the computer scale. Same thing can be done here also as the beam scan over the sample the diffracted of the transmitter beam can be either of them can be taken by the detector and we can we did not plotted on the computer to up to an image.

So the first you understand the intensities of the diffracted beams or the transmitter beam which is passing to the sample it is totally depended on the initial intensity of the convergent beam, which is falling on the specimen. So that is why in this kind of stain configuration one uses something known as FEG or what is called as fill eviction guns result is very simple the fill eviction guns intensities of the electron beams are very high almost three to four what are men should have then the normal lab six the implements or the Tungsten harp implements.

So these beams the which are high energy or high intensity beams are there can be de-magnified to about nowadays one nanometer or in a les in many cases one can go down to 0.5nm also, they magnified and to get a very fine probe or very good intensity are polling on a sample. So that the diffracted beam which is coming or the beam which are coming out on the exits specialist specimen can have separation intensity to get information's regarding the sample.

So that is why FEG sources are normal used as I give an idea the if I take FEG fill eviction guns source one nanometer beam as a current of 0.5nams, so that is why with FEG bright and dark fill STEM angers can be easily required within few seconds or even under TV scan rate that is what

you want we want the beam to scan on the sample and then image to be displayed at the same scan rate.

So to do that you need the TV scan rate and to get a TV scan rate you need to have a substance intensity of the beam, so as I have discussed with this slide this looks like a very simple there are lot of attachment involve which I have removed for the sake of simplicity of the images. But essentially the components in the scanning electron microscopy is exist amole a same axis conventional transmission electro microscope instrument and not much difference is there so except there is a scanning quail which can make the beam scan on the sample like in a raster mode which just like a scanning electro microscope.

But you know there are practically there are lots of advantages using this scanning turn's electro microscopy. So the real advantage is that the dark fill images can be obtain with a very high collection efficiency in stem as compare to the normal dark fill images in scanning transmission electron microscope this is mainly because that they all these scatter electrons outside the incident beams spot can be collected in a stem mode and once is collect all the scatter electrons diffracted beams the intensity of the diffracted beam is sufficiently high. So that it can be within give as a very good collection efficiency that is one of the beigest advantages.

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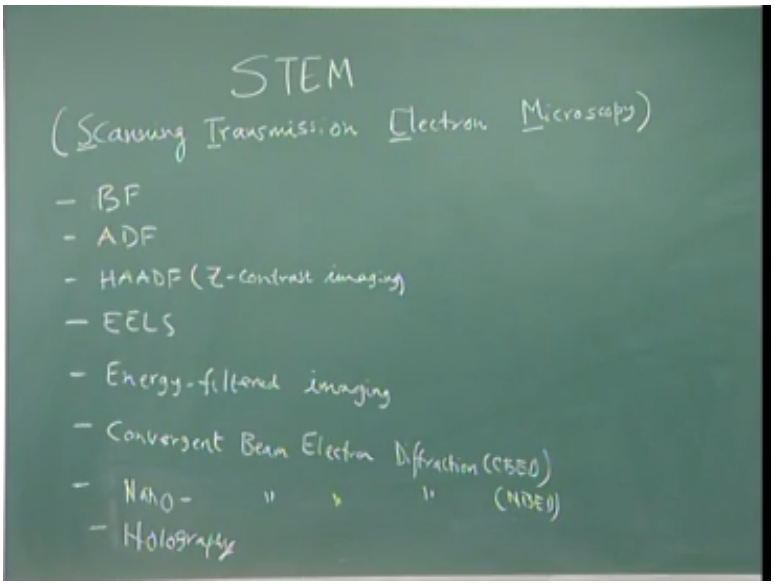

And that is why almost half of the imaging detectors in the stem mode at dark fill like low dark fill high angle and load dark fill is actually totally used to obtain all kinds of different future I going to discuss one by one. Not only that is the biggest improvement advantage not only that another important advantage is that in the stem in a conventional electron microscopes or the convention TM normally a two dimensional detectors suggest photographic plates or a CCT camera is use to record intensities that all image points which are in parallel like as the beams goes parallel and falls on the screen the images are recorded.

But in stem image information's produced the serial form like a time dependent voltage or current variation for many years in fact this gives stem this unique possibility of all line image processing to manipulate the image contrast for special used or purposes. And now with you have the CCT cameras have level of high quality high relations and so therefore this gives us the serial read out or online image process is I will set ion conventional team also have a stem or the stem there are many other possibilities exist.

The same stem detectors can be are the stem actually several detector can be used simultaneously to produce images on the different signals coming out from the sample. So there are varieties of these stem detectors available.

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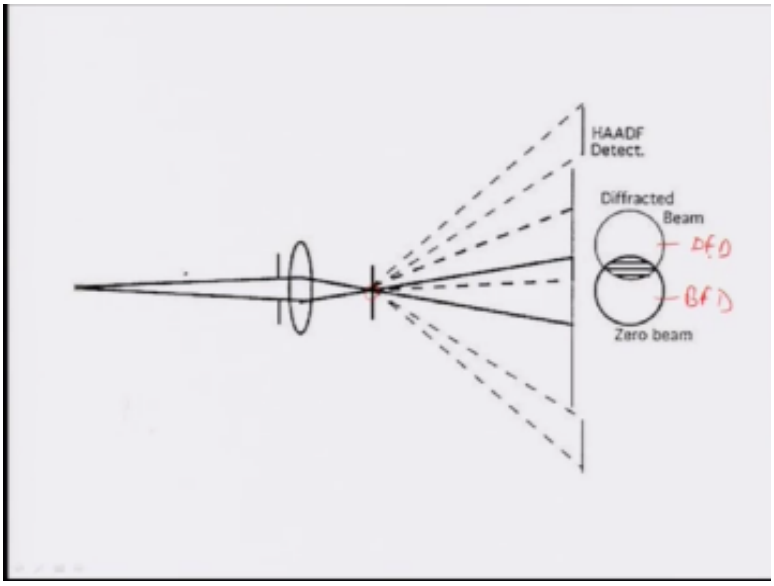

And which I can show you here or I have listed down there variety of stem detectors available to obtain different signals other that this white fill dark fill, so as this beam falls in a SAM specimen it is one part of a beam is getting diffracted that is why it is the big this diffracted disk is shown here other part is equal 0 beam which is not overall going suppose to be undergoing not on any diffraction as fast scatter beam, so we can put a BED and we can put a DFD and obtain conventional back fill dark fill images just like that.

As a collection efficiently high fill dark fill images qualities better even many cases the resolution also better because the beam which is basically coming out after diffractions they contain the information which having very high resolutions not only that one can actually use integrate as I said the stem with in a electron loss spectra meter which can not only allow you micro analysis or the specimen or very small area to detect elemental presents or the state of different elements present in the sample or specimen in a small area but also it allows you to from the images with electron that are lost particular moment of energy.

We have to understand that hills basically operates on the energy lose of the electrons, so as the electrons comes out of the sample exit phase they carry lose information because there electrons are underground different kinds among to energy lose and this is nothing but because of the scattering of the electrons and the specimen. And so therefore we can starve this information's by trapping those electrons which are underground lose of energy because of illustration scattering and this lose can be characterize depending on the Togo element present in the sample.

So one can actually use a certain kind of technique in which a specific energy levels can be used to obtain image, and these are all energy filter imaging and so therefore the what is call as the image is which corresponding to electrons that are lost a particular amount of energy can be obtain and to give an idea.

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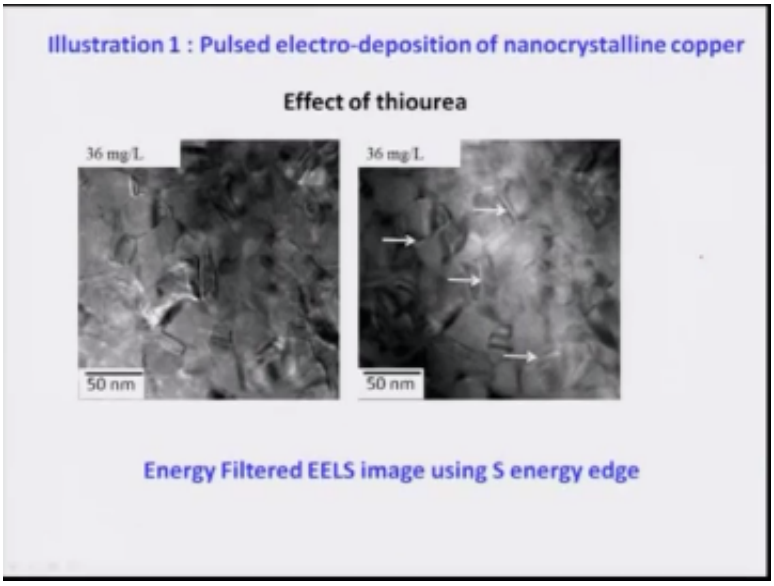

This is the illustration which I have show in the past lecture for the nano-crystal in copper falls electro deposited using thyoria, so it is dimmed or thought a thyoria act as a gain refiner by priming the gain boundaries of the copper. But there are no work or no experimental evidence available showing that thyoria only goes exactly sitting at the periphery of the copper grains there in electro deposition of the copper. So to show that one can actually use so what is known as another filter imaging using else as I said that EELS can be used to filter or the two images or to obtain images for the electrons which have lost a particular amount of energy.

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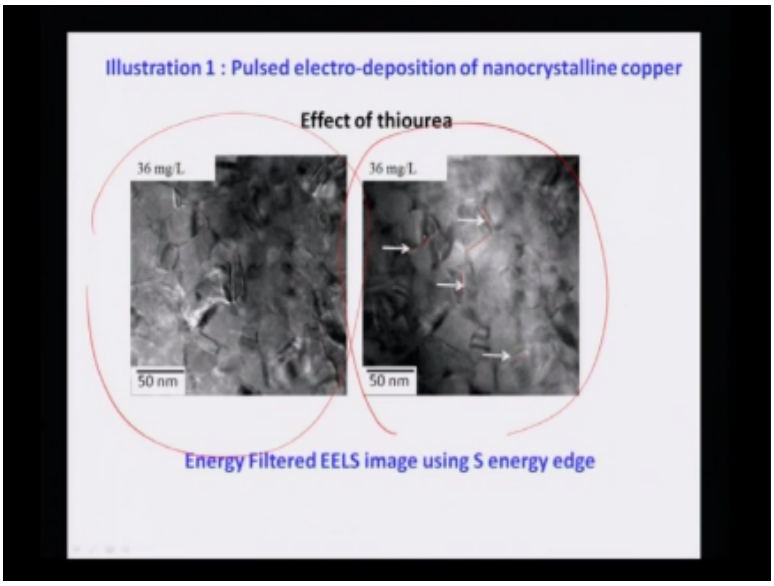

This is the left side of the picture is basically showing you the normal bilateral image and right side of the picture is taken using sulphur energy edge that means the electrons which are lost energy corresponding to sulphur energy edge in the EELS and that can be used to obtain the bi field or image and there we can see this bright lines and the boundaries many places, this bright line signifies the presence of the sulphur.

And as I told you even in the first lecture the idea is basically a molecules which contains sulphur or the nitrogen hydrogen and carbon and sulphur is the distinct part of the molecule, so the presence of the sulphur means presence of the thyroid. So by doing this kind of exercise one can actually obtain images top the resolution of the electro the microscope prior we can show that where a particular element is present.

So therefore this are now does it routinely infinite catalyst which is very important field of researches now days, one can actually use this energy filter images to clearly signify while a particular element is present or not. So other than that the STEM mode one of the EELS the most important attachment in microscope, which many microscope as, the micro9scope which I showed you, in our campus do not have this EELS but I have shown you where the EELS can be attached at the bottom of the microscope.

And the signal can be processes using computer, also images can be formed in a STEM mode by using the low energy, secondary electrons or oxy electrons or the catalyst x rays are there. So serial nature of this image signals provides basically the possibility of the quantifications

basically and then the information can be correlated with a specimen composition and as well as the morphology.

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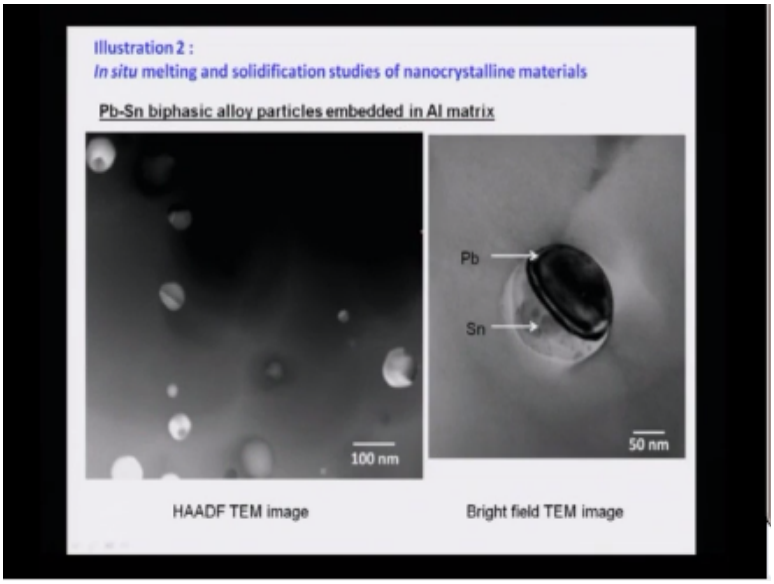

So another important detector is basically added.

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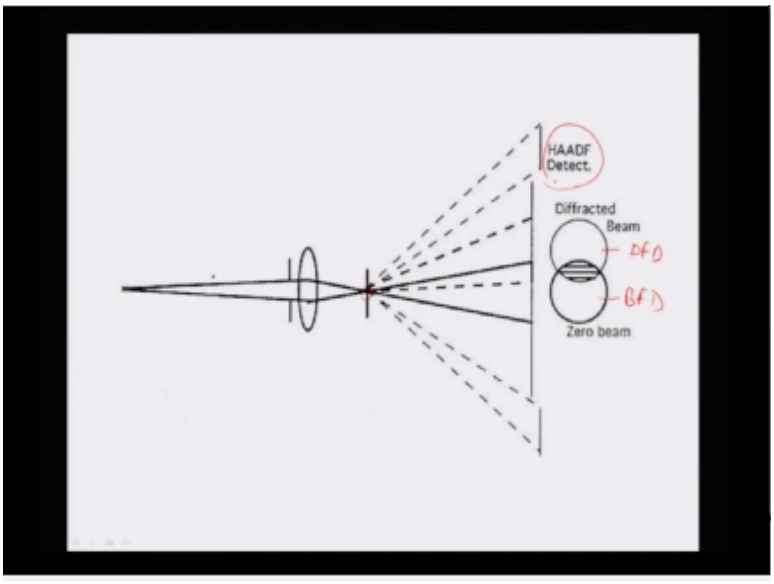

In to the microscope or into the stem is known as HAADF high angular detector, this was basically done or developed by and they have seen that if we use angular detector, angular detector means, it is like this.

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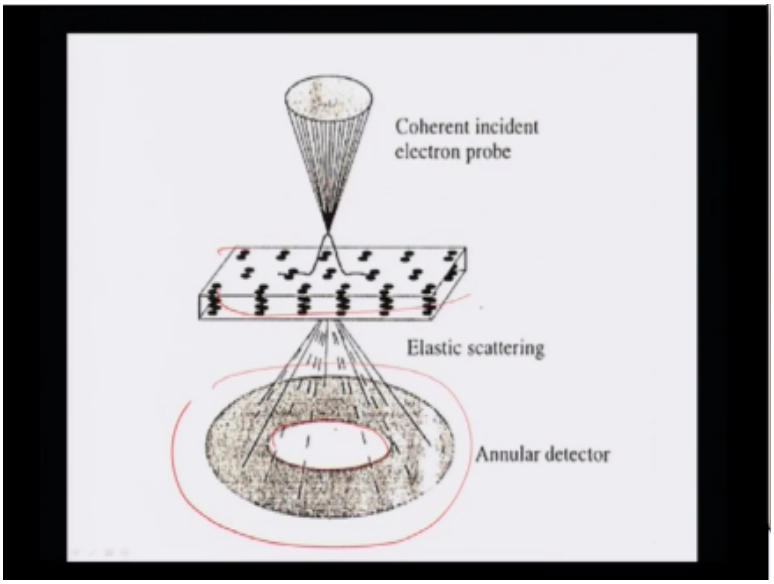

Suppose this is the specimen here as you can see and this is my converge beam coming from this objective lens, STEM and then obviously electrons are undergoing diffractions. See if you use a detector which is angular type, so what actually happens is that we can collect the diffraction information in a disc okay surrounding by this central circle and this is what is called as the angular detector in fact the reasons for it, which I will tell you in few minutes time.

So if we can put these detectors at a very high angle electron diffraction you have to remember the scattering happens at the small angle may be 1 or red or actually micro radians are few 1 or 2 micro radians like that. The high angle means of that nature, not that several couple of 20, 30, 40 radians or so no. so therefore these detectors can be placed just like one source here, at a little distance on the transmitted electron beam or the zero beam.

And they contain information regarding the atomic number contrast, it has been shown by curator the basically the intensity of the image which forms while the detectors is proportional to $J^{3/2}$ where z is basically the atomic number of the element present in the specimen. So therefore this detectors if we collect the information and display will tell us chemically composition present in the microscope.

This is very clear and this is routinely done nowadays and the microscope which I showed you as the hardy detector, takes the signal which are coming out or coming from the specimen at an little larger angle than the commonly defect beam in a angular detector and displace on the computer screen. So to see you once that image here this one I have shown you in very first lecture of this course this is basically a picture taken in a hardy detector in a microscope.

Where there is a fake gun, so as you see this is the lot of large number of nano particles present in this specimen and this nano particles embedded in a matrix it can be basically written as aluminum matrix and this nano particles are let in and as you can clearly see it that in a high angle dark image, we can clearly see this particles having showing distinctly two phase contrast.

One is rare solution or TEM solution, so as you know that aluminum having very low atomic number, so therefore the z^{th} contrast because of aluminum having very low on the other hand LED and TEM having very atomic number so the z contrast will be very high but they will be distinctly different z contrast variation from the TEM and the LED which is seen from the different positions of the particles.

As I said that the bark of the particle as you seen in the right hand side of picture is TEN or the cap is basically LED and LED having the high atomic number then the TEN it will be looking brighter in the images. So this is again taken from our own work so one can actually use this particular technique this kind of information. This is exactly equivalent to the scanning electronic image and microscope.

And we routinely do in scanning electronic microscope to obtain the z contrast images so that we can see different phases very clearly. Exactly same thing can be done in the STEM but at the resolution of highly electron microscope. Now one of the biggest follow out of these particular hardy detectors is this, we can use this hardy detector to obtain highly electron micrographs okay.

How to do it I will just explain you within few minutes time, it is like this suppose you have the thin sample a specimen and you are using the STEM mode, so that the converging beam is focused on to the particular column of atoms and obviously as I said you using fake good fake guns one can actually covert the electron waves to one nanometer or in point 5 nanometer level. So if this small beam or very high intensity falls on the columns of the atoms.

And then this columns of atoms contain different atoms or atomic numbers, some of them are like aluminum which is very low, some of them let us contains some other very heavy lead may be high atomic numbers. So obviously the atoms which are heavy they will diffract strongly than the atoms which are having low atomics numbers so if we take or collect this information in harder detectors in a high resolution mode.

We can clearly depict that which atom is what whether it is a aluminum atom or it is holmium atom or it is basically lead atom one can clearly show on the microscope. So this is another important thing which normally people will do and if we collect this images in a HADDF mode or HAADF detectors serially that one can build the whole structure of the crystal slow by slowly one by one well nowadays you see titan who do not need to do it because the titan basically one can get distinct contrast from different atoms like one can gone tone trust from I have shown you example of stone titan it in last lecture most titanium and oxygen differently so you do not need actually hard detect on that way.

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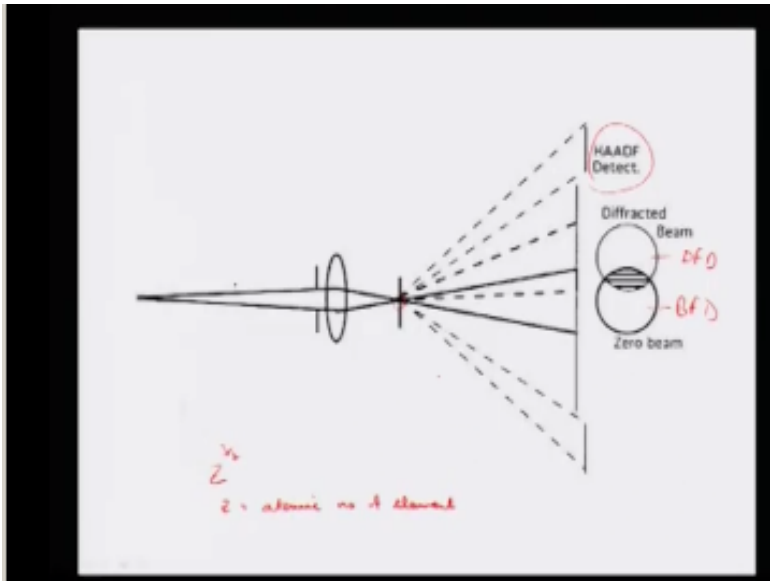

So this is another advantage of the stem, the stem actually as changed the whole color of the electron microscope so much now as I said that the one of the advantage of this higher detectors to obtain z contact images but it has its own you know limitations also limitation in the sense that it has to be properly sample as to properly oriented and many is not fond that very signal are depended on sample thickness the sample thickness is high.

The hard disc sequences are very scoot the sample thing is small and the signals are bad so that is why one needs to use very high density beam at the signal of the sample that is to focus with signals very properly the sample and for that intensities of the highly detected term or the theme reasoned sampled can be comparable another important technique which is attached to the stem is the converging electro diffraction.

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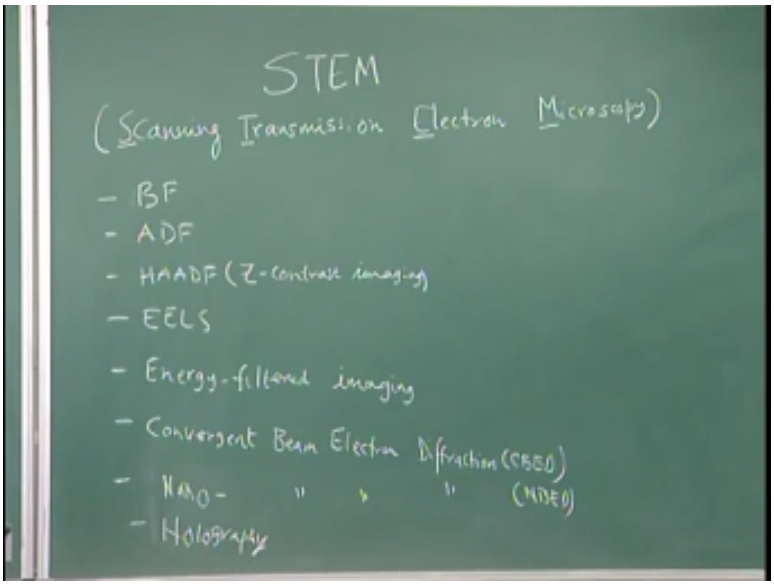

And the nano electron diffraction specifically the nano diffraction is nowadays widely used we know that many techniques allows us to form grains or the particles which is very small suppose less than 5 nanometers so in those cases it is very difficult to obtain the diffraction in formation using conversion diffraction like the diffraction particle so one is to use they use the decimate or the converge electron deflection or specifically nano wave related diffraction to give you a example

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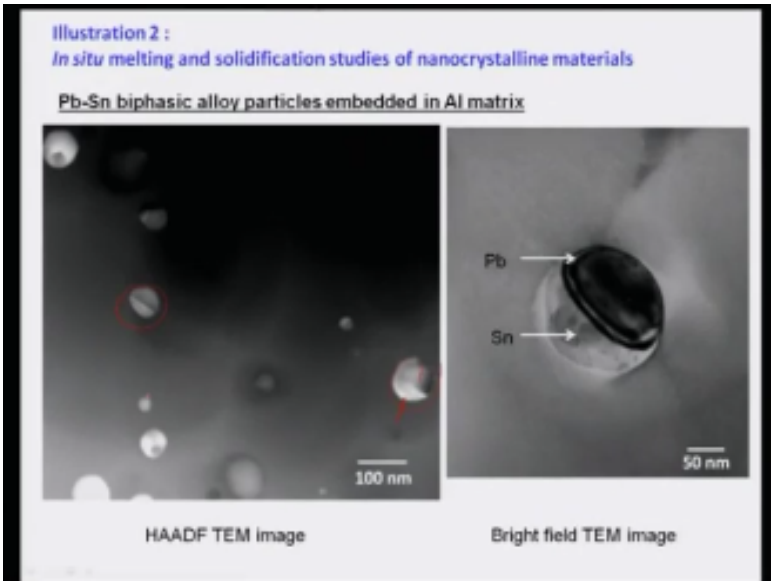

Suppose there is a particle layer small which are market there in the image size is approximately enhanced so to obtain diffraction permission function is small particle one needs to use a very fine probe and that kind of fine probe can be obtained in the stem mode so that the beam which is which is highly conversion side of the beam of the order of suppose three nanometers can be allowed to fall on this particle and then diffraction can be collected.

This is now understand routine in the fake guns one has to understand that the generalize electro diffraction depends on the available intensities of the insert beam because the sample is particle is not so thin the diffracted beam will not be enough to be recorded by the recording device so that is why many cases what is done is that the beam are used to fake game very high density and allow to fall on the specimen and then diffracted wings is collected one of the advantage of this is that.

One can actually obtained in the information for the large number of particles by this way and then get full scale diffraction information to the particles so as you see I am going to discuss about haplography here if this is a big subject as you see that using all this kinds of detectors in the scanning task microscopic one can obtain information and then compressing normal white field and dark field z context images combinations by yields and energy filter images by yields as well as diffraction information by monograph diffraction.

So this actually since to like that if this all kinds of information and advantages are many obviously advantages are many whether disadvantages so disadvantage is that image extend

more is manner so therefore it has practical problems it is like recording the images sometimes can be long and that is why in that time long recurring time sometimes this can shift there will be images not only that this also possible that there is taking the images.

They remember the current of the beam electron beam which is falling from the stem detector of the objective lens and the grate as a function of time so this can actually give stick images many times in stem those of few where used the stem to find if your microscope is not functioning at the optimal level it will get sticky images in the compress in not only that if we use a very high the converges high current electron beam is can damage.

The sample many times you will find if you do the electro deflection on a particle after taking the diffraction pattern particle is basically changed so the sample damage is really high so more so one has to be very careful about the particles senility of the specimen towards electro wave if the specimen various sense of the electro wave once is not used stem in a fake is gone rather once should use fake in the lab six parameter on the inter electron normally low.

So basically we uses of the stem depends on the particular type of the material, the material is good and stable and we go ahead with normal microscope stem configuration and optimal configuration the material is been sensitive we cannot do it so you have to change it to the normal microscope so in a national I have discussed with you the higher microscope stem in the three four lecture in the next lecture I am going to show you two important things.

And incentive from the microscopic latterly I will discuss and do some estimate discuss about the linear detector which is normally attached in the microscope nowadays in front new concept as come where super is used so using one can shower later microscope this is there is titan so I am going to discuss about that and then I will move on to the standing microscope where different kind of scanning of microscopic techniques like the technology different diffraction patterns or insecurely can be used to obtain last few information.

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Prof. Phalguni Gupta
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Camera
Ram Chandra
Dilip Tripathi
Padam Shukla
Manoj Shrivastava
Sanjay Mishra

Editing
Ashish Singh
Badal Pradhan
Tapobrata Das
Shubham Rawat
Shikha Gupta
Pradeep Kumar
K.K Mishra
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Sweety Kanaujia
Aradhana Singh
Sweta
Preeti Sachan
Ashutosh Gairola
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Ashutosh Kumar

Light & Sound
Sharwan
Hari Ram

Production Crew
Bhadra Rao
Puneet Kumar Bajpai
Priyanka Singh

Office
Lalty Dutta
Ajay Kanaujia
Shivendra Kumar Tiwari
Saurabh Shukla

Direction
Sanjay Pal

Production Manager
Bharat Lal

an IIT Kanpur Production