Indian Institute of Technology
Kanpur

NP-TEL
National Programme
on
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-05

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So in the first class of hydrolyzing electro-microscopy I have discussed you the basic fundamentals of idle electron microscopy and we are going to continue from the class which where we ended in the last class so as I say so new that the contrast transfer function which is known as the transfer function in a hydrolyzing microscope is basically a very complex function.

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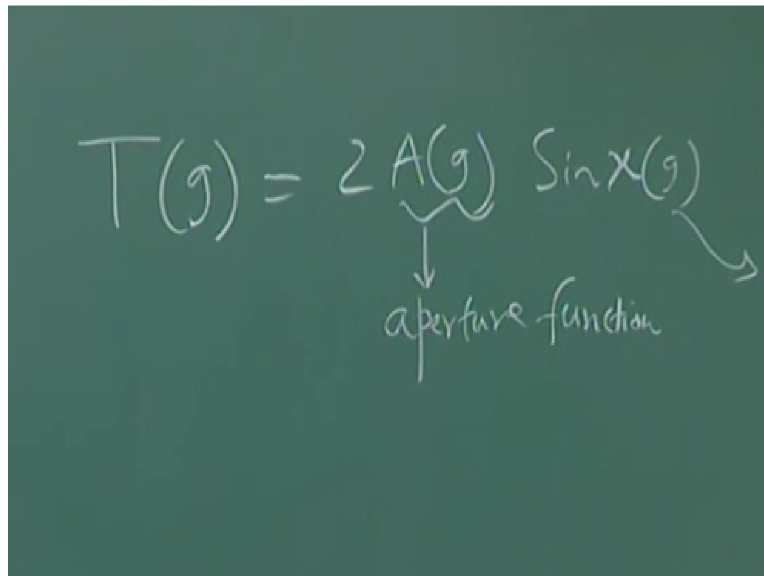

And it can be written like the one I showed you transfer function is equal to 2 where the first factor is as a function of g is basically the aperture function and this actually is a strong function

of the g and χ is basically the angular factor which comes into play so from this function we have seen that how the χ actually plays a very important role and finally we derive the optimum our values of the χ the one which I can write like this is depends on defocus and trail as depends on the C_S or the aberration spherical aberration constant.

So therefore the factors which control the contrast transfer function basically at the defocus and the spherical aberration constant of a microscope obviously λ goes into the picture because λ is the wavelength of the electron beam and g is the special frequency which is inverse of λ and so therefore defocus and the spherical aberration constant demarcates the values of χ and χ I can be as a function of g as we have discussed here square with little bit defocus and the fourth power for the C_S.

So we know that for a particular microscope C_S is a constant C_S values never changes for a particular microscope but obviously it can vary from microscope to microscope also it can be changed if we change the orbital length of a microscope, so knowing that Cs is fixed by a particular microscope the free parameter which can play the important role is d δF or the defocus and that is where we actually shown you how this can be determined using different values of CS and different values of defocus how this function can change.

So we discuss this factor in detail and find out what the optimum focus limit for high lesion microscopy so this is the slide where I am showing you the actual or ideal transfer function when a microscope lens is a perfect as you see that they know microscope is a basically a perfect one because there will lens defects like farcical aberration and other factors astigmatism and as well as this Quantico aberration so Quantico aberration can is very difficult to correct.

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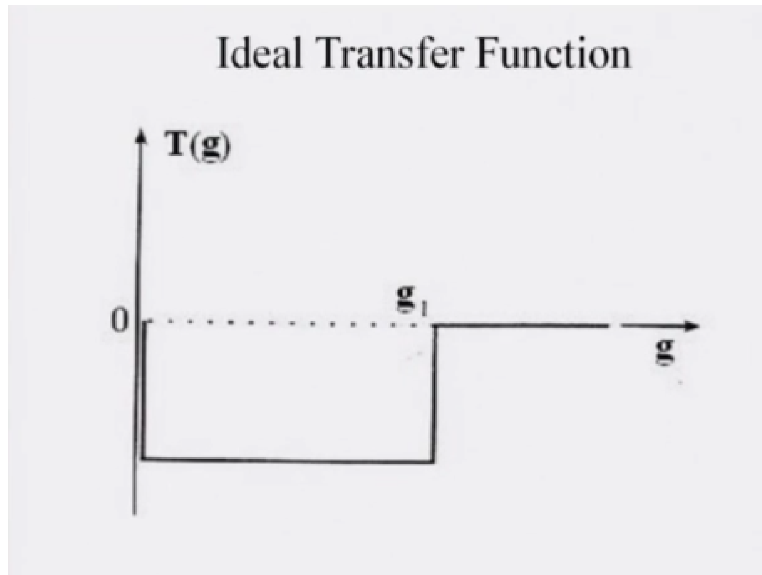

And because of the aspects that the beams which are falling on the sample may have different energies but estimation can be 100% corrected on the other hand the spherical aberration which is a important factor going into this equation is very difficult to correct but off light it has been found that spherical aberration constant can spherical aberration can be easily corrected in electron microscope and can be used.

So this is the idea of spherical aberration the transfer function which is shown here so obviously an ideal transfer functions the as a function of g the obvious.

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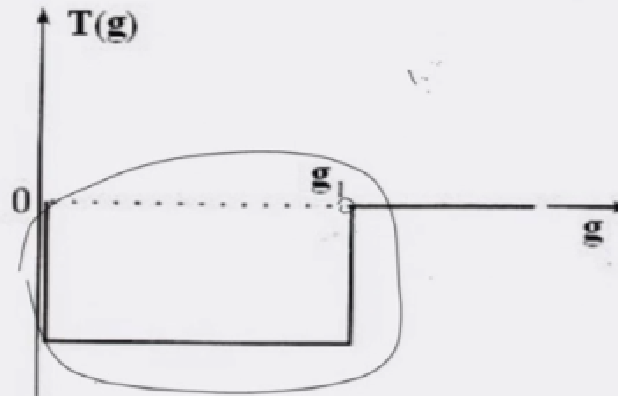

The value of 0 in the transfer function should be as small as possible that number of 0 here is this and this is for an ideal microscope which is not possible to achieve at any cost so in a real microscope the transfer function so the actual the here I am showing you the ideal transfer function for any transmission electron microscope so the you can see that the transfer function varies starts from 0 it goes down and finally it is basically a square function reaching 0 again at certain value of g which is at g.

And so as you know that more 0 means more less number of information going into the going through to the from the example to the image so that is why the Ideal transfer function will have only one 0 here and all these information's which are here are basically at low special frequency are transferred to the image from the sample so which is this kind of situation is they have never daily achieved because all these microscopes will have defects and the defects change this transfer function drastically.

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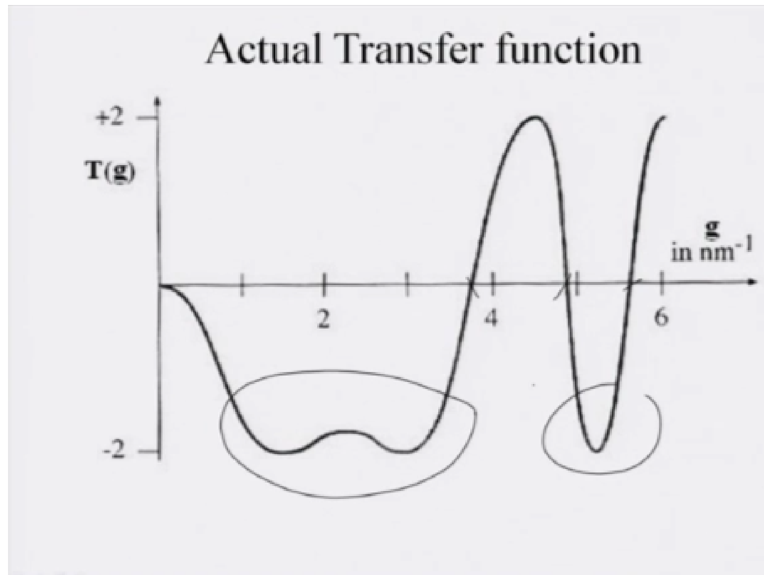

And I am showing you one such picture where the actual transfer function is shown T(g) as a function of g here and T(g) values can actually can be varying from +2 to -2 but what you can see is that there are a lot of variation not only number of 0's have increased in this transfer function but also the special frequencies depending facing frequencies the values are changing so there are higher order values there are lower values of the transfer functions.

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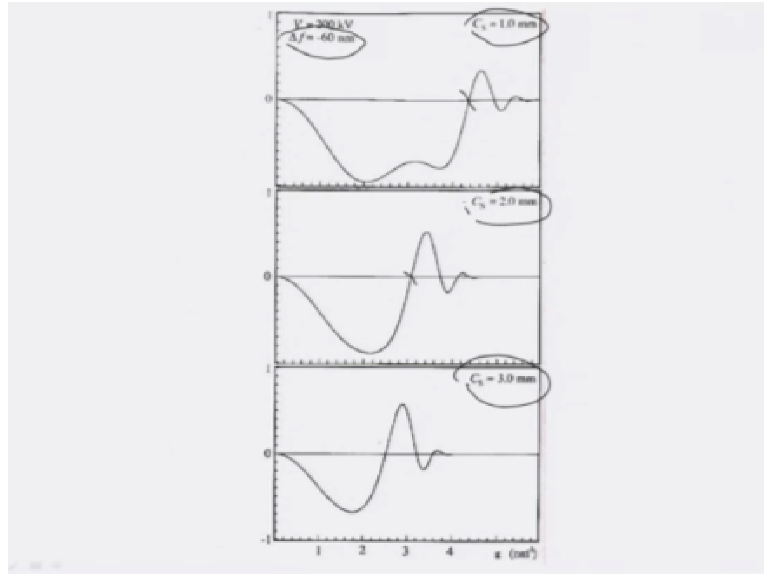

Now, one can actually see the effect of transfer function effectively space spherical aberration constant is transfer function this is what is shown here which I have shown in the last class itself if I change spherical aberration constant CS from 1 to 3 millimeter the transfer function changes drastically for one basically the transfer function has a very large with before the 1st zero comes here.

And again as Cs has increases this 0 is slowly moving to the left side a lower value of g and that from less number of spherical aberration the official frequency can be transferred in the actual microscope and this is done at a fixed value of defocus - 60 nanometers. So once we optimize the Cs value okay here it is seen that optimizations here value is basically 1.

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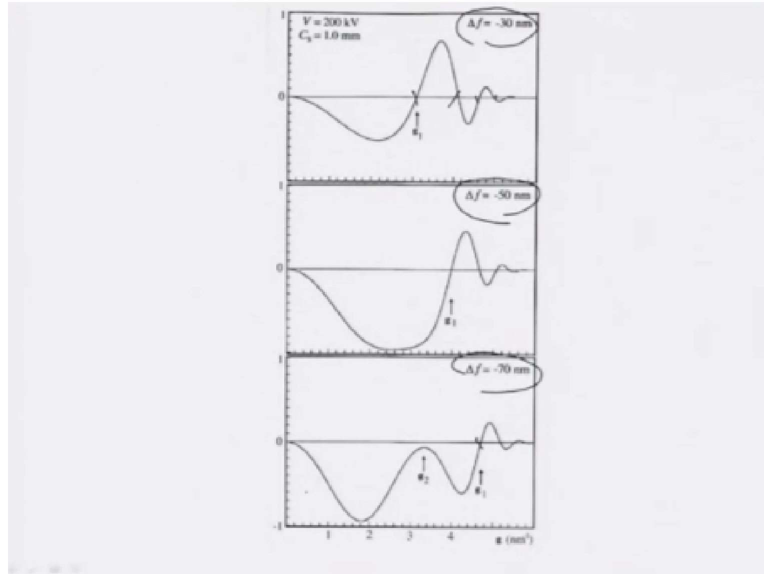

And then we can look at what happens basically as a function of the defocus as a function of Δ F so a Δ f is varied here for 200 kilo microscope with CS 1 millimeter from point - 30 nanometers to - 70 nanometers as you vary from - 30 to - 70 the profile or the task function profile is a function of g is changing drastically for very low value of Δf that is - 30 the g1 comes at a frequency like this and there are several 0 here after this g1 and as you go from – 30 to -50 the g ones values shift to the right side that be more information's where lowest spectra frequency ranges can be transferred to the image plane.

But as we increase the value Δf display - 70 nanometers you can see that g1 actually never reaches the 0 value or 0 axis value and g2 rather so g1 actually is coming at a higher value but G 2 is another kind of spikes come into picture which is giving or restricting the transfer function to a much lower value of g which can be transferred to the actual image so these are the two effects of Cs and Δf then for a given microscope Cs values are known how do I optimize the Δf values.

And this is basically done by a scientist called Auto scissor in 1949 and that is what we will do so as you can understand that the presence of 0 in the what is called in these transport functions are basically tells you the gaps in the output signal correct because these zeros means they had giving no output signal as far as the transfer of the actual things in the sample to the image plane is concerned. So that means these frequencies can be thought of filter out so obviously based on function will be one in which there are least number of zeros the one I have.

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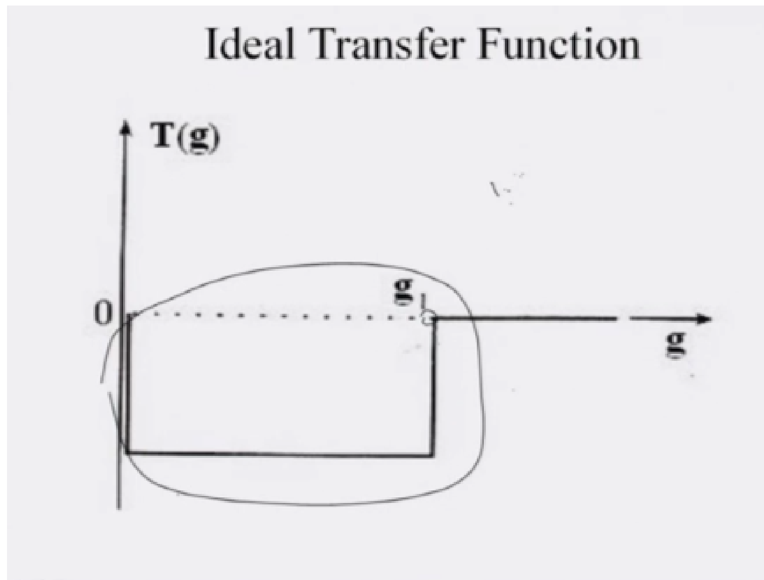

So new one slide back here is basically the ideal transfer function where there are only one zero there is only one zero.

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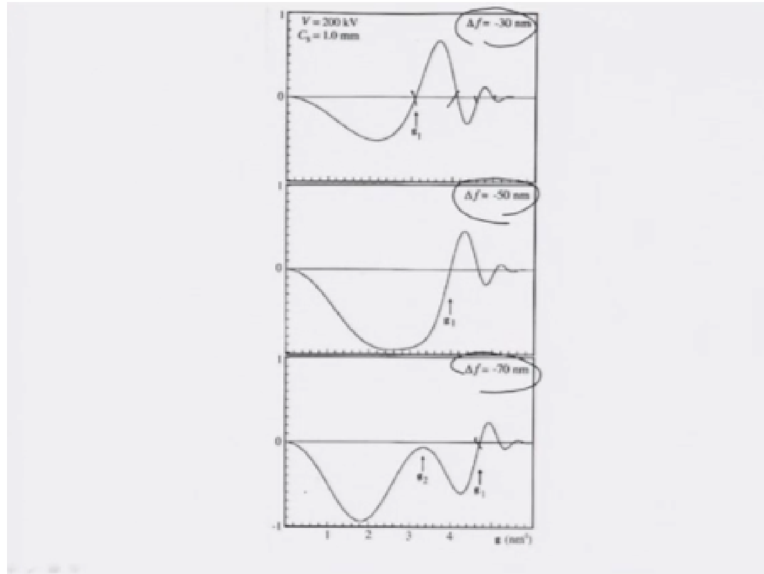

So therefore best one will be the one you where there will be least number of zeros in the transfer function so that the best resolution is possible so in what Scheer has done Scheer are two Scheer along back in 1949.

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He has basically described he has basically described the optimization of the target the focus or de focus in a particular microscope what is said that the transfer function can be optimized by balancing the effect of spherical aberration against a particular value of defocus so that means if I need to get a particular value of defocus where the effect of Cs can be balanced this ΔF is known as say defocus which is given by this formula -1.2 Cs $\lambda^{1/2}$ and many times we write this equation as -1.2 Sch Z where Sch Z is known as given the Scheer focus defocus Cs $\lambda^{1/2.}$

So what you can see clearly from this picture is that the Scheer focus is basically tells us that all the beams will have at the defocus all the beams will have nearly constant fades out to these first 0 a fast crossover in the 0 g axis that is what is shown.

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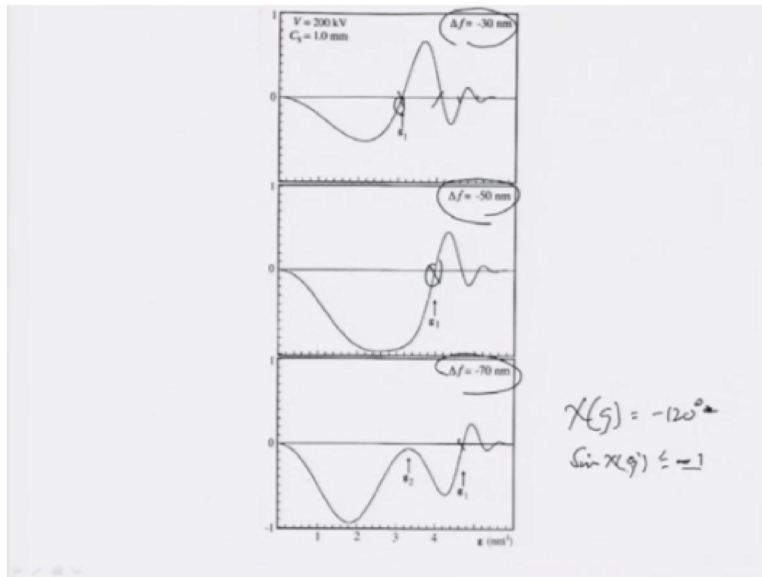

Here this is the first cause of where 0 defocus 0 g axis so therefore all these beams or the electron beams will be nearly constant phase out to this and this is what basically required to obtain the best possible did not the resolution our information in it electron microscopes this performance the best performance obviously will be expected from microscope at values first value of first course of our value on the 0 g of the z-axis unless until obviously we do lot of image processing later on that is we improve the contrast by doing some other means.

But this is what is obtained for a particular for a particular machine with a given value of Cs now how does this formula arrive that can be basically done by using simply several aspects so what you can see from this from this picture is that from this what is call for the slide is that the closest we can get to the idle card ideal car which are shown is basically corresponding to Σ corresponds to this value the $\chi(g) = -120^0$ and that correspond to sin $\chi(g) = -1$.

So that means this is the obviously maximum value or minimum value of sin possible but the sin is a bounded function between 0 and 1 or -1 and 1 basically depends on angle so - 1 is the minimum value of the 6 sin is possible and this is the gives you the best optimal possible the value of transfer function now let us see how this can be basically derived using this formula which I have already given you for χ and then for χ we have seen that the χ given depends on the defocus and also the spherical aberration constant.

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So this is $=\pi \Delta fg^2\ 6\ \lambda g^2 + ½\ \pi Cs\ \lambda^3 q^4$ so if I basically minimize this function at a particular value of g I can get basically the Scheer focus so let us do that if I minimize this I get very simple equation $2\ \pi\Delta f\lambda g$ + again $2\pi Cs\ \lambda^3 q^3$ and this obviously has to be 0 so we can write down this essence this + $Cs\ \lambda^2 g^2$ so that can be easily seen so when χ will be -120 so we can obtain from this equation that this is $-2\ \pi/3 = \pi\Delta f\ \lambda g^2\ + ½\ \pi\ CS\ \lambda qg^4$ and combining these two equations one can obviously obtain a simple mass simplified expression up size of focus or the focus that is f SCH = $-4\ /\ 3\ CS\ \lambda^{-} Cs\ 4/3\ Cs\ \lambda^{1/1/2}$

So that becomes 1.155 Cs $\lambda^{1/2}$ so that can be easily derived by this way just by putting the derivative first derivative of χ respect to g to be 0 for a particular value of χ there is 120^0 because at 120 χ = -1 so by using this equations we can derive that says our optimum schees focus become 1.155 Cs $\lambda^{1/2}$ which is very close to 1.2 and this by finding in this calculation obviously one can arrive at the same set of focus and which is basically nothing but that balancing act which you do of Cs by putting the focus value at a particular number.

And this is routinely down in a microscope nowadays where the optimum image is obtained at a particular value of Schees focus now obviously one can go ahead and even go to the next step that is the first crossover is by this the next cross over can also be calculated and cross over as it basically comes at a particular g value.

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The next cross over can come at a particular g value given by this is 1.51 Cs - 1 / 4 λ - 3 by 4 okay and this cross over is very important in a sense that at this defocus at the basically said that the focus which corresponding to this value of g will give us a resolution limit of the microscopes and this resolution limits can be obtained by sticking inverse of this which 0.66 Cs λ q $^{1/4}$ so this gives us the optimum resolution limit or the actually relation limit for any time selector microscopes.

So by knowing this number C_S of the objective lens and λ we can basically get that that is why you understand now why people use minimum goals electron microscopes because λ can be extremely exceedingly reduced by using higher and higher accelerating voltage from 100 to 200 300 people have gone up to 1.5, 1500 actually kilovolts so by and obviously another way of improving resolution is by correcting the Cs which you are going to see today when the end of the lecture how we can do that.

So this actually puts as the limits of resolution this what this is not the limits of the information limit is much actually higher value than the resolution limits in microscopes okay information sorry much lower value than the initial microscope information can be achieved even much lower in fact there are report the 1970s a detail of 0.66 Armstrong was actually done or achieved and.

When the interpreted resolution was about 3.3 Armstrong so you can see that that actually it details an image that mean that you can gain in useful information’s about the microstructure so

there is a distinct difference between the solution limit and the information limits as far as the microscope is concerned well so therefore these two function one is as Sch and other one is that $\Delta f\ Sch = 1.2\ Cs\ \lambda^{1/2}$ these two are the master equations in the high resolution electron microscopy.

They actually gives us give us the limits for any uses in the high resolution electron microscope and this was due to auto Sch and other 1 the first equation is basically developed by glaser so therefore and these two people are these two scientists are called the pioneers of the high resolution electron microscopy for developing this concept in the highly resolution microscopes so that is actually sets the tone of the highly electron microscopes.

So in many students so many users have misconception that high listening to microscopy means just getting a lattice fringe are just getting some information on the on the computer screen all on the imaging Screen where you can see the atomic planes okay but that is not the case there are a lot of many interesting aspects one is to know now as we have discussed here or we have shown you that the transfer functions we normally do not take any the things which are beyond is 1st 0.

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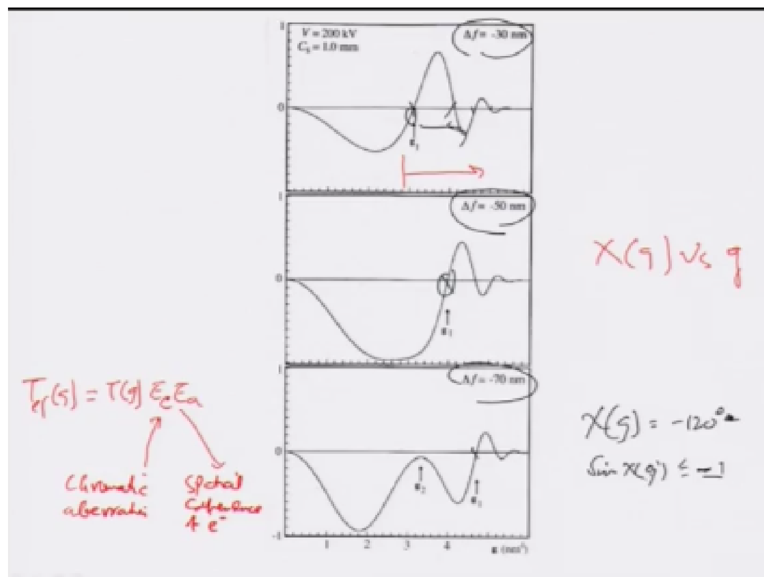

Something which is basically coming beyond this number or being the g1 value okay which is basically of no use to us but why it is so whether this is really true or not that is what it gives us

the new concept called envelop damping functions so plots on this T(g) as a χ g whatever χ g is also related to T(g) χg vs g as you can see here can they be extended from this 1st 0 to the other house that is the question we need to ask ourselves because as you understand if we can extend this value to the higher g numbers and g is basically spectral frequency.

Therefore we may be able to achieve much better information in the real space because g is in the reciprocal space so if g higher energy means lower value of x or y or z real space variables so if we can extend this g to the higher values and include them in to transfer functions in a electron microscope we will be able to achieve much at a higher resolution and much higher information limits also can be achieved.

So if that is the case is it possible answer is no we are we will and we do not use this higher you know cross over or we do not extend the values of the g beyond this fast cross over normally why this is mainly because of the damping effects I think because of the other issues like this special coherency of the electron beams and also romantic aberrations so which normally comes into play when spherical aberration is corrected and these are basically due to special coherency means as the electrons Falls on a sample or basically if you look at the electron beam how small it verities even it is one nanometer of 0.5 nanometer whether the special coherency of the electron beam exists or not it is very difficult to ensure even using the fake guns.

So because of these resolution is hampered or Which of these higher the g values can never be accessed second one is the commodity cavitations although we can use a FeSch the real emission guns to reduce the beamed energy spread to even very low values still it is very difficult to get 0 chromatic aberrations or it is actually not yet possible to correct the commodity aberration to that level.

So because of presence of both the commodity aberration and special coherency problems this kind of these frequencies can ever be actually used to get information from the sample now obviously the exactly mathematically this can be derived and mathematical envelope are form of this envelope functions K is very complex and one can actually simply write that T effective function as a function of g = T (g)EcEa Ec is basically due to chromatic aberration and Ea is due to special coherence of electron beam.

And once you put this envelope function into the transfer functions they are coming as a multipliers as you can clearly see and this basically gives us limitation of using this higher values of G into the transfer function this effectively means the envelop function is basically acting as a virtual aperture in the back pocket plane of the objective lens the goal is whatever the value of the defocus that is what is the actual meaning of that.

So that means physically if we at all put an aperture in the in the scroll in the back focal plane to remove this unwanted noise this must be less than size R that the virtual aperture present because of this general angular functions, so that is why you know presence of this virtual person means higher-order pass bands Hara Department means these pass bands higher order values and never accessible it cannot be accessible because of presence of these envelopes that is why we I say that this is nothing.

But n roll up limping damping functions these they these envelopes are actually these functions frequency is getting a damped out because of presence of the envelopes due to kinetic evolution and spiracle cohesive and this is what can be.

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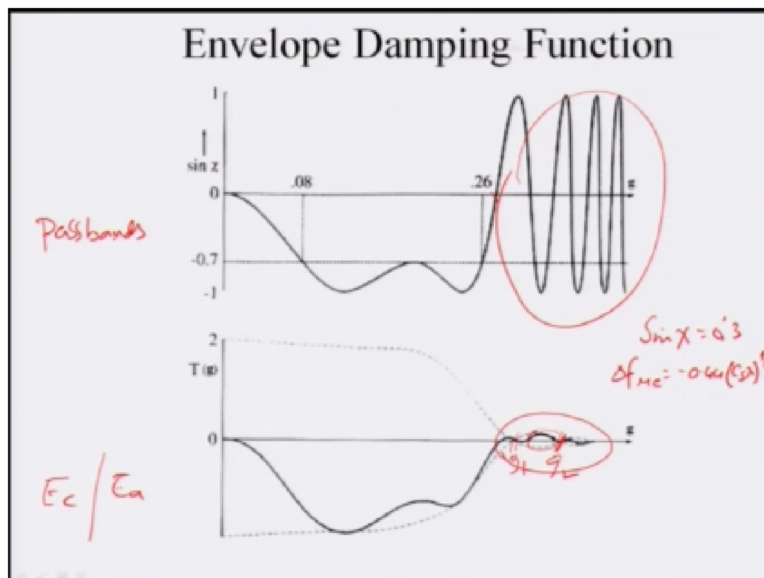

So it is on there so as you see here this is the science I as a function of G so there are many, such higher frequencies or higher daddy values which gets damped out because of the presence of this envelops due to UC and Ec so effect of EC and Ea is to damp out this higher frequency so you cannot access them, so whatever may be the situation as you can clearly see that there are all the

G is more than higher than the g1but this beyond that the values of the TG is very small and whether this can actually affect the solution or the information.

On the microscope is needs to be analyzed and it has been seen that it can be it can affect so that is what although resolution limits will be given by the first crossover but the information limits can be higher information limit can be given by this point G2 which is at a higher speak in see higher special frequency, so that is why in many microscopic images highlights of microscope means nowadays whatever you see all the dilution is about say 0.8 Armstrong for the title microscope 0.5.

Man struck for the commodity carburetor microscopes but information can be actually picometer levels, so we can achieve information's much higher require a much higher resolution levels are much higher value better values than the resolution level give them the microscopes that is what is possible in the microscope because of this the these cut presents of the small you know higher order G values and in the in the transfer functions, so these all give us some extra information now my knowing all these aspects.

How actually in a real microscope is changed our operate the microscopes because as you see the focus of a strong role to play in getting the actual hydrogen images because Δ F what you have seen is a very important aspects and we have to go to the surgery focus limit to obtain the best resolution image, so how do I achieve that Δ F said in a real microscope that can be done in many ways obviously first thing one can do is that I know for example worst case of town, so transfer function is that when the transfer is minimized that is you just start for defocusing the orbital lens in such a way that you do not get see any contours on the screen on the computer skin are on the basically flows and skin.

So if you do not see that that becomes your best from that is start actually changing the focus value and reached a defocused value, so this minimum value here as you can see because corresponding to this number of science I and this number of Sciences happens to be 0.3 so therefore minimum contrast the minimum contest for the orbital and focus length is given by – 0.4 CS γ to the 4 how this can be calculated obviously in an actual microscope knowing the γ and the CS.

But in real microscopic stands you do not need to do you can simply change the objective focus in such way that you do not see on the screen anything that is becomes you are the minimum contrast in a microscope and from there you start changing the focal length and as you change the focal length the abducting microscope you will be it should be able to reach the say the focus otherwise, what can people do is that in some same cases transfer function K actually settings result setting of transformation are also used.

one such is basically aspect is to use something known as pass band or use larger window in the transfer function okay, so that we can allow this higher special frequencies to contribute the image how it is done obviously you as you see from this figure.

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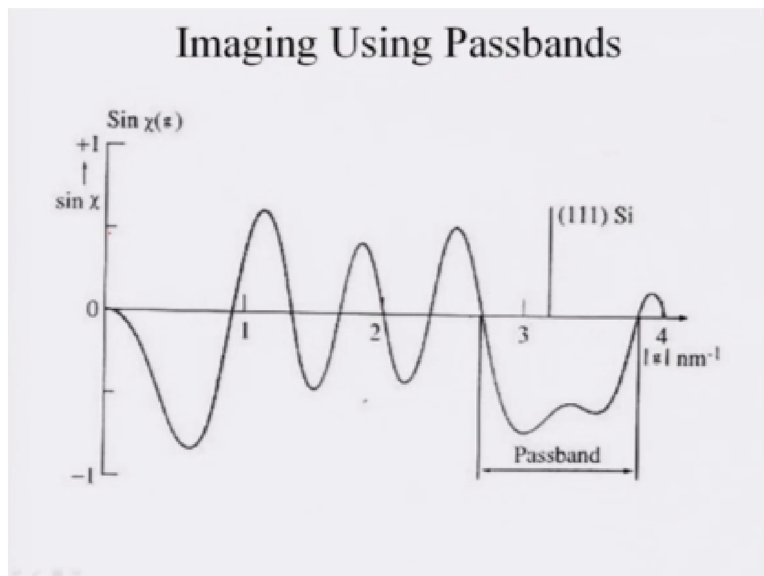

I clearly from the next figure which I am showing here from this figure very clear you can see is that that this requires that Si to become and obviously Sin means this value of site size depending on the σ Si and also obviously depends on the CSM ΔF.

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This requests I to be constant and also D side to be constant and D Si DG to be very small one such example is shown here for silicon as you can see the sign size varying from – m1 + 1 as a function of g here g is the special frequency and what you can see is there many pass bands and if I select a pass band which corresponds the one-one-one reflection of silicon and what you can see is that I can really get to see that side remains almost constant for this from this part to this part of g and D Si by DT is also very small that means it is all nonzero.

But it is very small value so because this idea is also very small and size the many constants, so that we can use this higher-order passing bands and normally this kind of Hydra passing bands or critically and one can actually obtained such equation like this Δ FP n = 8 n + 3 / 2 Cs γ all half so when equal n = 0 that become the fast this value here okay, so and that becomes nothing but 3 / 2 Cs $^{1/2}$ 3 /2 is 1.5 and if you take root of that becomes 1.15, so that is because that becomes actually the same as has a focus.

For n =0 so this technique gives you the access to the high especially frequencies the air you can see here higher critical frequencies thus the fine and details in a real space as I said higher value of g means photos in the real space and this price basically is that only price rupees there, now there are many zeros 1 2 1 2 3 4 5 extra zeroes are than the first zero so because of this more

number of 0s and 0s corresponding to know how to signal and therefore there will be transferred the g mod 0s means lower pass frequencies will give you at modulus and less information.

Transport transfer function will be heavily affected that is the only problem but this is used widely to basically select the focus in our highly spectral images in many microscopes, so therefore an nut shell I could say that in the in the lecture for largest last lecture in this lecture I have given you very what is called bleep picture of the transfer function, how the transfer function actually varies as a function of Δ F CS and obviously facial frequency G and how we can use it.

In different ways to obtain the optimum focus values area are met by balancing the C_S and this all are done routinely nowadays by computer okay, so we do not need to do yourselves many times what people are many of the users to is there that instead of trying to obtain the optimum the focus we take through focus images that is we take images at different level of focus and then we see which is the optimum focus or which is the basically correspond to say their focus that can be easily done routinely.

Now-a-days using these the digital cameras where we do not need to spend money to graylag rather gap image just like earlier days you used to spend if the what is called camera plates where each camera produced two cores about hundreds rupees, so in a normal imaging technique where you use the digital micro, so digital imaging process you do not rather actually need to spend money for more images or less images you take, so that is so nowadays one can easily take three focus images.

Where at least twenty minutes as a function of focus can be taken and then from there one can actually decide which is the optimum focus image, but that is actually a trial-and-error method which normally people use but for the beginners actually this is the best method possible in fact when I started using a chat TM I did the same way taking through focus images from the almost in flames and then find out which is the optimum focus, well that is basically the state of art till 1999 or 2000 from there a lot of changes happen in the microscope I listen to microscopy. E Especially people have started correcting the CS as you have seen CS plays a major role in deciding the resolution as well also the focus, so if we can able to correct the C_S then obviously we can get better resolution better this information in the hallucinatory media's and that is what has been done by scientists called Eros in age rose in 1990 he proposed that it is possible to

correct the CS value in an electron microscope, okay by using a set of spherical lenses and extra poles and this was a major discovery major discovery in a field of electron microscopy.

As we know in a normal camera we can basically correct the spherical aberrations by using a set of lenses okay by basically diverging converging lens type we can basically do this changeover so in a nutshell this is what I would like to tell you about highly senior microscopy and I have basically started from a normal camera, and how the transfer function can change in electron microscopes what are the factors controlling the transfer functions and how to actually optimize them now as I have says shown you several lectures several slides.

So actually in the last lecture in the today's lecture also that she is the spherical aberration constant plays a very important role in deciding the resolution of the times electron microscope rather to break the resolution limit of tangency electron microscope requires Cs to be corrected and this started in1990 by the classic paper published by H those from an optic as you know that in a normal camera we can correct the spherical aberration by putting a set of converging diverging labs.

Now an electron microscope this can be done by using set of spherical and exit polls that is what is shown in the slide.

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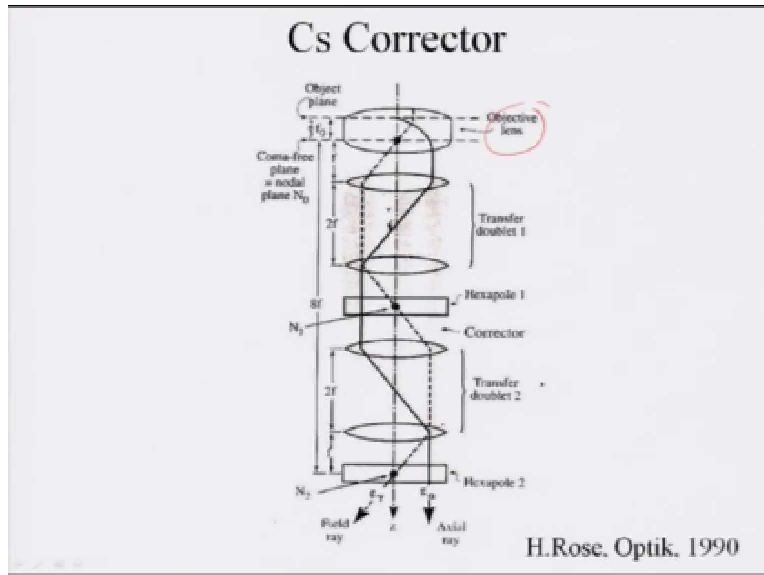

So as you can see this is the standard optic objective lens this one the standard objective lenses and the electron beams from there is passes through a set of spherical lens and except all lengths and by this when it passes through all this actually accept all lens and do not affect the paraxial path of the Rex okay, but it corrects this aberration in fact this has been reported that the it is possible to actually have pretty clever some constant negative and by getting that one can actually break the dilution barrier of one Armstrong.

And this is what has been done later on by many scientist okay and by publish a neutral bond and others in the Anschluss Crescent of electron microscope in Germany they have been the successful in making certain character possible and by putting characters below the Italians and out the orbital lens where both of the was called probe and the image characters can be inserted and such insertion of the characters leads to tremendous change of resolution, so as you can see the CS is corrected the resolution of the microscope will be dependent.

On only on the ecchymotic aberration so therefore resolution is basically can be written as when CH is corrected is like this Δe / E and γ Cc this is the comment equation to the power half, so when CS is corrected and Δa is basically 0.3and 200-level microscope and who can see we can clearly see these years will be approximately 0.8 in fact if Cc is corrected or the command character which is going on, now and in the team project in the US it is possible to reach when cc's corrected also DCs and Cc is possible to reach 0.28 Armstrong, so you can imagine that by using all kind of characters.

We can actually reach a limit of point three approximately point three Armstrong which is very close to these smallest atomic size the atomic diameter of carbon atom is 0.8 and 9 Armstrong, so atom in diameter of hydrogen atom is 0.5 mm stuff, so we will be able to reach resolution limit of less than the size of an had an atom that is the dream which is once needs to cherish, but obviously such a dream will come at a very high cost maybe once there I know that a C_S character microscope.

Cost about several million dollars actually about 10 to 12 million dollars so once see SNCC characters votes are actually inserted in a times electron microscope length of the microscope will be three storey building and such a microscope operating such a microscope requires help of computers without computers one cannot actually operate such a microscope well this is all are getting done and in fact CS corrective microscopes are now available in India IIT Kanpur is also going to get to see sky to microscope very soon I to Madras.

And many other places like TI for Mumbai and the Jain CSL Bangalore has also got see scattering microscopes and soon there will be many users using CS character microscopes and maybe in future in India will have Cc character microscopes, where we will be able to braid a solution very at two point three arms down then we can see whatever you want to see where the atoms appendicitis can be easily matched and as I have shown you that in the lot of lectures.

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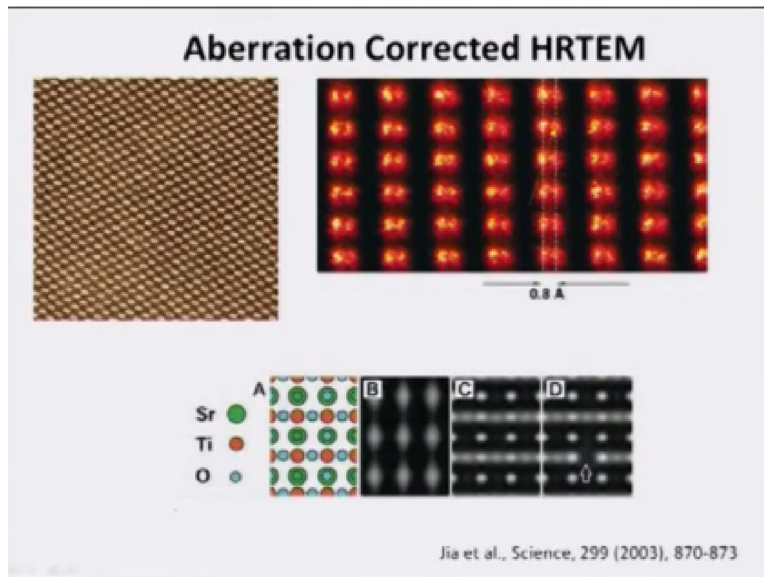

In the first class that if you have Aberration corrected character here the emails shows that change as you can see if we correct see as you can clearly see the dumbbells silicon numbers, so that means we can clearly achieve resolution of 0.8A which is some abstract resolution and from 0.8 2.3 will take us to a different domain of world or picture, so by using simply series character microscope as I shown you in the first in the first lecture HRTEM even we can image the oxygen atoms in the stands GM title net.

It has been reported by Jia et al science 2003 and now it is a routine things people can actually image oxygen nitrogen carbon atoms even which are very small using because solution limits are reached that is why the 0.8 is very easy to achieve.

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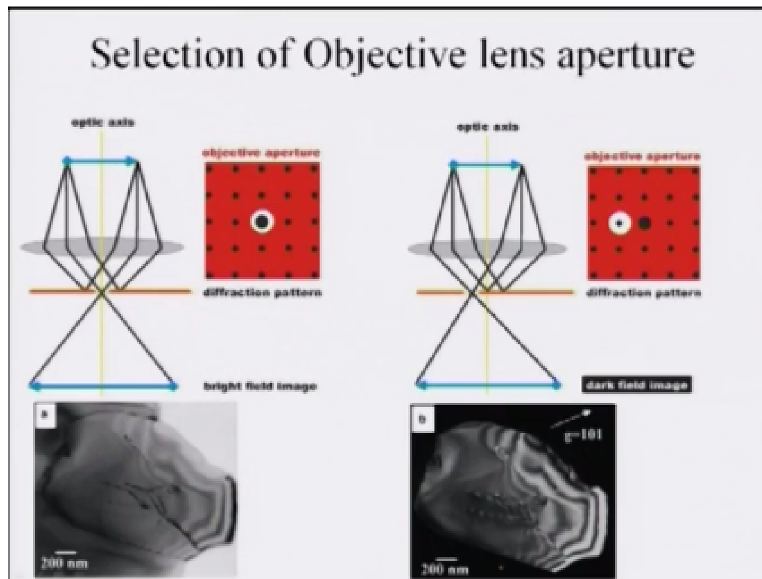

Now I do not want to go into all kinds of numbers but let me show you how in 1970s people used to do electron microscope.

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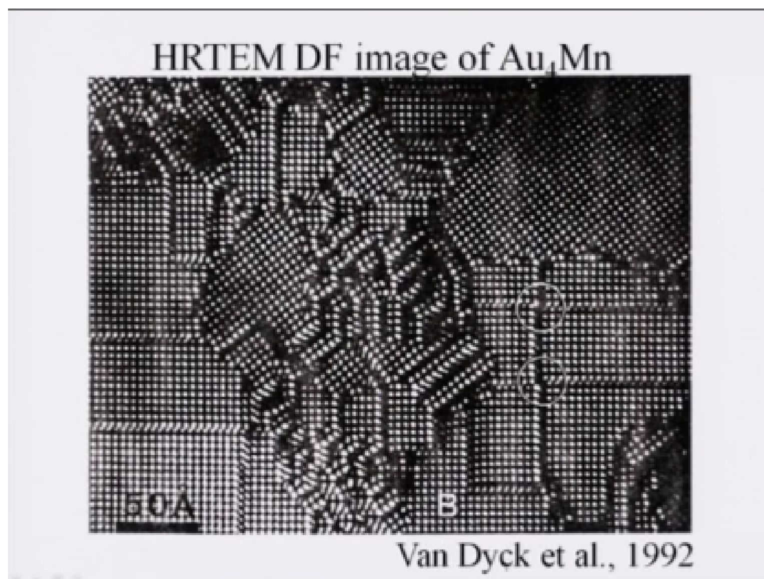

And get highly no my microscope this was is taken from on the panda I take it all of the group of a melling's and the plant and Alou in Belgium long back it is done in 1992 even before the characters have come into picture this is a 30 M dark field image of a you for mmm what you can see here all the black and white dots of atoms which you do not know which one is what but you can see the Antipater when boundary is marked, by these white circles also you can see lot of distortions huge number of distortion.

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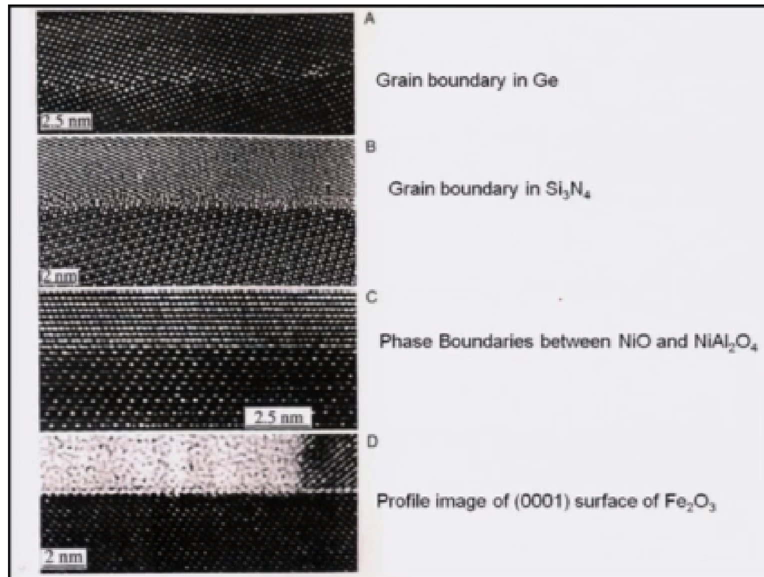

In the picture next one is taken from a set of pictures by many authors one of some opposites our so we can actually image gain bounded germanium you can clearly see it consisting of the dislocations at different intervals each film one has one dislocation slowing again boundary courses to go parallel set of dislocations you can see that gain boundary of SN 3 SI 3 and for silicon nitride has amorphous layer on the a very small thickness about say 0.5 amps from 0.5nanometers sorry and then phase boundaries between an IO and Iin 204 nickel aluminide which is spinel.

Is very flat and very sharp last one d is basically profile image all along 0:01surface of hematite where you can see the black and white dots of iron this was used to be the case in 1990s till1990s bus advent of CS character microscope has made she'd lot of changes, and it was possible now to pin point which item is part and to resolution of a level less than am strong, so therefore basically this all nice and fine you can easily routinely get this in C_S character microscopes, but the real problem comes how to interpret the images.

Or how to really you know the get the information from the images that is where the real challenge as i said the real handicap hydrogen to microscopy is to interpret the images our interpretation of my images requires simulations simulation needs lot of you know a priori knowledge as per the crystal structure of the material is concerned adding positions are concerned also interaction of the electrons with the sample how this interaction or interaction is

dynamic as we have seen, so in the next class i will just describe they saw the simulation techniques and then I will move on to much advanced techniques.

Like stem or scan scanning transmission electron microscopy where we can obtained even the normal routinely microscopes cannot even my electron microscope, like the one which are shown in new you in the beginning of this course, it is possible to attain achieve different other kinds of information like jet contrast the item number contrast information or even you can actually take hydrogen images using these same features to obtain or to see the heavier atoms presence in a particular material which these are all discussions we will do in the next class.

Acknowledgement
Ministry of Human Resources & Development

Prof. Phalguni Gupta
Co-ordinator, NPTEL IIT Kanpur

Satyaki Roy
Co Co-ordinator, NPTEL IIT Kanpur

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
Jai Singh
Sweety Kanaujia
Aradhana Singh
Sweta
Preeti Sachan
Ashutosh Gairola
Dilip Katiyar
Ashutosh Kumar

Light & Sound
Sharwan
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Production Crew
Bhadra Rao
Puneet Kumar Bajpai
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