Techniques of Material Characterization

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Lecture – 16
Electron-Material Interaction (contd.) & Image Formation and Contrast Generation

Welcome everyone to this NPTEL online certification course on Techniques of Materials Characterization. So, now we are in module 4 that is in week 4 and previously we have studied some general concepts of microscopy then we studied about optical microscopy in very great details and last week we spend a lot of time understanding the electron material interaction.

Various components of electron microscopy, general concepts of electron microscopy and so on and from this week onwards we will be moving into a particular technique in electron microscopy that is transmission electron microscopy, but before that I think last week we were left with very little portion about electron material interaction which we need to understand.

Before we move to transmission electron microscopy and that part also has its own importance for transmission electron microscopy and then we will move to the image formation and contrast generation mechanisms in transmission electron microscopy.

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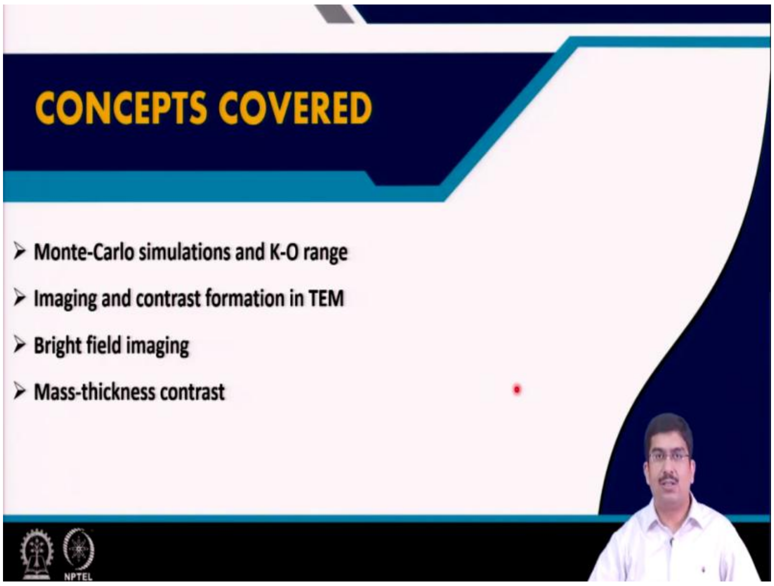

So, the concepts that will be covered the topics of this lecture will be as I said initially we will discuss about Monte Carlo simulation and K-O range and then we will move to imaging and contrast formation in TEM and we will be discussing then on bright field imaging and there we will be seeing at least one of the major contributor to contrast form generation that is mass thickness contrast.

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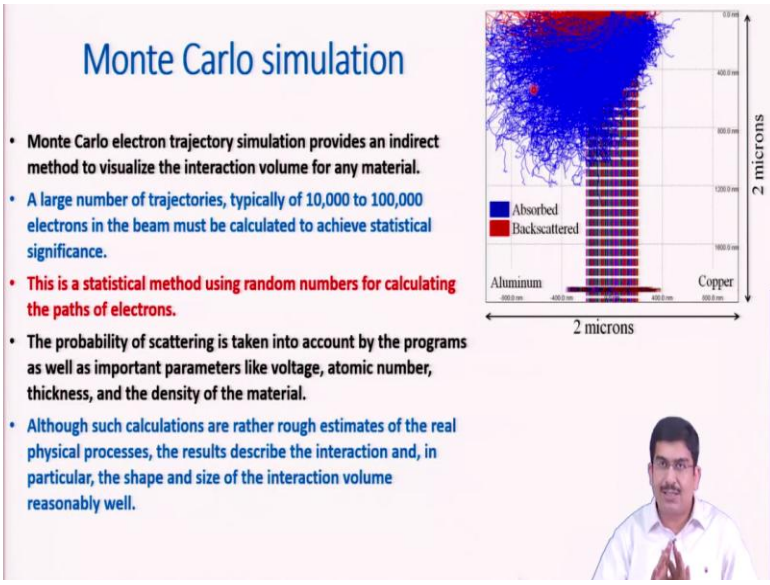

So, first let us finish the incomplete discussion about the electron material interaction. So, what were we stopped in the last class, last week was basically the difference between the plural scattering and multiple scattering. So, I was discussing about a Poisson's equation which was valid, which was giving the probability of having an electron material interaction when we have plural scattering.

So, single scattering then plural scattering and then multiple scattering and what I said is that in case of a transmission electron microscope we tried to restrict the interaction if the best is possibly in single scattering or at the most plural scattering so that most of the scatterings are elastic in nature and then that will just deflect the incoming electron signals it will not drag any energy out of them so not much inelastic scattering.

And that will help us to understand the contrast generation and that will help us to use those electrons for electron diffraction experiments because in elastic interaction since the energy is not changing, no secondary signals are generated those electrons are needed because there will be electrons of known wavelength. So, there wavelength will not change since their energy is not changing their wavelength also will not change only their phase will change their amplitude may also change.

So that we need elastic scattering mostly we intend to have elastic scattering in case of transmission electron microscopy specimen, but if the specimen is very thick then we discuss that there will be possibility for both elastic and inelastic scattering and in those cases the Poisson's equation which gives the probability for such scattering elastic and inelastic scattering to happen that fails that will no longer be able to predict the path of the electrons.

So, for that kind of situations what we need to utilize is Monte Carlo simulation and one such example is given here how does it look like. So, in this case all this lines that you are seeing this basically are trajectory of individual electrons which is undergoing elastic and inelastic scattering. So, the red and green are two different types of electrons do not get confused about that.

But basically all of this lines these are how electrons are travelling within a specimen. So, this one is the backscattered electron the red one is basically backscattered electrons. So, electrons which are scattering almost 180 degree and most often goes out of the specimen whereas this other electrons are stayed within the specimen itself because these are undergoing constantly inelastic scattering.

Elastic plus inelastic and in every inelastic scattering it is losing some part of its energy. So, ultimately they got stopped there, movement got stopped within the specimen. We can imagine that they are getting absorbed. So, this simulation basically stresses the path of every electron. So, in the beam whatever electron was there and what happens to them that is what this simulation techniques takes care.

And in that sense the biggest implication of Monte Carlo simulation is that we can finally get the interaction volume. We can simulate the entire interaction volume for any material from this Monte Carlo simulation method. Now, where from this name comes Monte Carlo, what does it do? So, I often ask this question to my students and very rarely I get an answer that what is Monte Carlo basically means.

And also I sometimes discuss with them that if you want to understand, if you want to have a primary idea about any scientific event then try to understand the name because this names for scientific events, scientific phenomena these are given with the purpose these are not natural names these are somebody has given this name and often these names carry the characteristics of that phenomena or that event or that process and so on.

So, Monte Carlo is something same thing. Monte Carlo is a place in Europe and there is a small country small nation is there called Monaco and it is between France and Italy and Monte Carlo is a capital. So, basically it is a very small one almost like size of some megacity, big city metro city kind of thing, but the point is the Monaco or Monte Carlo is famous for its casinos.

So, many people goes there for gambling and when you have gambling you can understand that gambling has typically a role for randomness. So, it is kind of in true sense it is kind of a luck, faith whatever you call it, but there is a typical element of randomness in the gambling method any kind of gambling. So, this Monte Carlo simulation is also something to do with the randomness of any event.

So, any kind of event which has a randomness inherent to it can be best simulated using this Monte Carlo simulations and this problem this typical problem tracing the electron trajectories Monte Carlo simulation therefore are very, very good because nobody knows how many elastic interaction will happen, what will be the scattering directions, how many inelastic scattering will happen, what energy will be lost in each of this methods so on and so forth.

So, there is a huge amount of randomness that is involved in the entire process and that is why this Monte Carlo simulations are used. So, very typical simulations, Monte Carlo simulations can have anywhere between 10,000 to 1 million electrons in the electron beam we can imagine and those beams, their paths, there trajectories are calculated here using certain random number which will try to simulate the probability of having elastic and inelastic scattering and the energy loss and all.

But in this process of course what is taken into account is the important parameters which influence this scattering or this interaction process. For example we have seen how accelerating voltage has the role in the interaction. So, if it is very high accelerating voltage then the interaction cross section is very low and so on and so forth then we have seen the possibility for atomic number.

The best one is seen in the case of backscattered electrons as we already discussed backscattered electron or backscattering happens when electrons are scattered by positively charged nucleus and that has a big effect atomic number has a big effect on the trajectory and the number of such backscattering events then of course the thickness of the material, density of the material that means basically those mass thickness and all whatever we have studied.

So, all of these things is taken into account in case of Monte Carlo simulation and then finally it gives this kind of a shape and from this what we can get is the shape and size of the interaction volume. Of course, even using this many number of electrons large number of electrons taking into account all different kinds of effects and try to trace back the trajectories of the electrons using random numbers and so on.

Still this process is a very rough estimate of what really happens in case of elements of higher atomic number and we have no other way to trace it experimentally except for believing this whatever simulation, whatever interaction volume we are getting.

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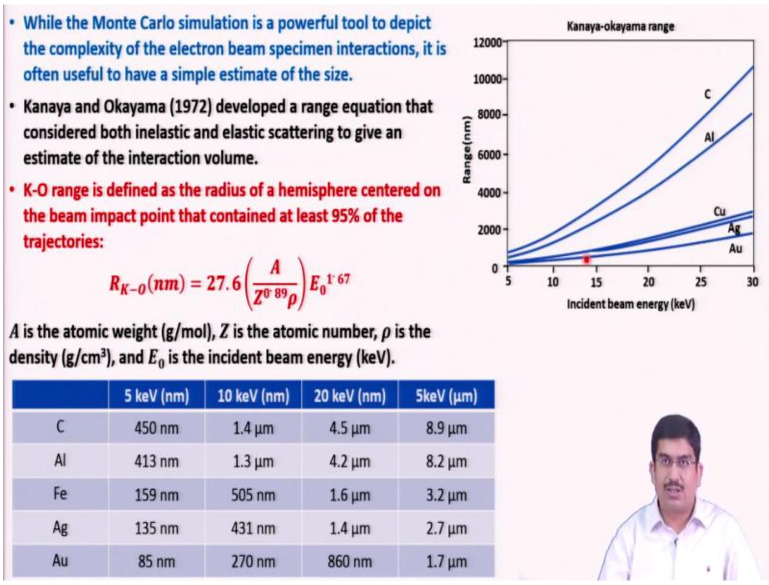

Now the Monte Carlo simulation is definitely a powerful tool to depict this electron beam specimen interaction process and it often from here we can get the shape and size of the interaction volume and as I already told in the last week, last classes that the shape and size of this interaction volume is very important because that is what finally is deciding or that is also responsible in deciding the resolution that you finally you are getting out of that process particularly in a scanning process.

When you have successive pixels and basically the distance between the pixels or step size is determined by the interaction volume size. So, interaction volume size is very important point here. Now, what happens is Monte Carlo simulation from there we can get an idea about the shape and size the way we discussed in the last class that we can determine the maximum depth also the width at maximum the maximum width here.

But this is often not often or not a very scientific methods of getting this sizes. So, in order to solve this two Japanese scientist in 1972 they proposed Kanaya and Okayama they proposed a equation range equation that consider both elastic and inelastic scattering and what they imagine is that this interaction volume as a hemisphere. So, they imagine that this is a hemispherical shape.

And then from that hemispherical shape they find out the radius of that hemisphere. The specialty of that hemisphere is that it should contain at least 95% of all the interactions that is the criteria, that is the cut off. So, wherever that happens the hemisphere extends up to that from a Monte Carlo simulation and then from there we can find out this equivalent radius. So, now this equivalent radius or range K-O range that is how it is known.

And it is usually given in nanometers. So, this is a very complex function of atomic number and accelerating voltage plus you have this atomic weight as well in this entire equation and that is how you can calculate from Monte Carlo simulations you can calculate the K-O range or rather the size of this interaction volume one single size not two sizes, not depth and width separately.

But one single size that can tell you what is the size of this entire interaction volume. So, if you now look at how this K-O range or this K-O size or K-O range varies. So, as I said this is a very complex function of course it is inversely related to the atomic number that is what you can see here also that when you look for one single let us say we are going to 20 kV which is typical for most of the SEMs.

So, when we have typically 20 kV electrons and with increase in atomic number you have a decrease in the size of this interaction volume. So that is very much also obvious from this relationship. Again, if you go for any single element let us say we take Iron and we see that the accelerating voltage with increase in accelerating voltage this range the size of the interaction volume also increases.

So, this is how one single number also able to tell us the effect of this atomic number and accelerating voltage, but if you look at this exponents they are very complex ones and ultimately that is why this range is a very complex function this is how it varies this is something that experimentally is determined not experimentally I mean through simulations you can determine this range and you can see that it is a very complex function of both this parameters.

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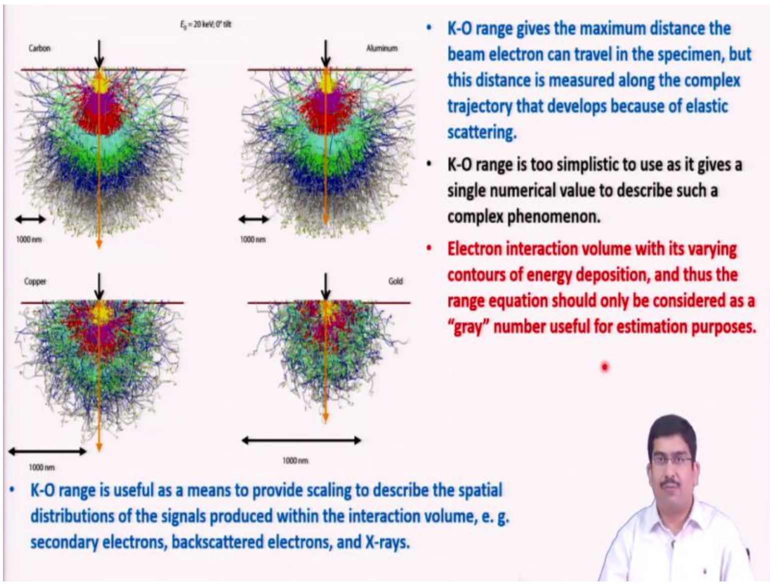

Now these are some other Monte Carlo simulations for various elements carbon, aluminum, copper, gold so on and so forth. So, you can imagine that the size or that the atomic number is increasing from carbon to aluminum, to copper to gold. Correspondingly, you can see that this interaction volume, the shape of the interaction volume is also somehow changing. Here it is really like a pear shape.

Whereas if you go to a heavier element like copper or gold you really have almost like a real hemispherical interaction volume which you can determine from this Monte Carlo simulation as well. So, the shape definitely changes, but ultimately you can imagine everything to be like a hemisphere equivalent and you can calculate K-O range and this K-O range of course gives the maximum distance the beam electron can travel in the specimen.

So, that is the maximum depth also in some sense that this is how this much the electrons can travel at least 95% of them can travel up to this much. Also this distance takes care of elastic, inelastic interaction all of them. The problem with K-O range is that it is too simplistic to use. It is even simplistic than to use two different sizes, the width and the maximum depth and maximum width even simpler than that.

So, it is of course it is giving us one single number which is easier to use, but this introduces a lot of error. So, K-O range should be used with lot of caution and this should be just a number for comparison, most of the cases it should be just a number used for comparison. If you try to use this number for calculating the resolutions, the way I said that size of the interaction volume most often you will be ending up in wrong calculations for resolutions.

So, it is just a size you can just a number that you can use for comparison of various elements and they are comparatively which one should have a higher interaction volume so on and so forth, but still it is good to have a rough estimation of this interaction volume so that is the main importance of K-O range.

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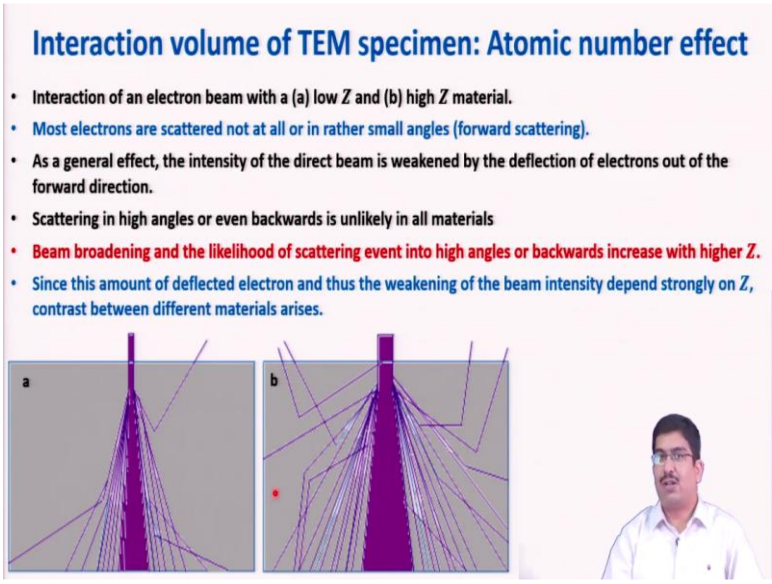

Now, let us see for a TEM specimen how this Monte Carlo simulations are useful in order to understand various effect of various parameters which we were already discussed when we were discussing about interaction cross section, mean free path and all we have seen the effect of accelerating voltage and the effect of atomic number. Now, let us see the same thing, but by doing real Monte Carlo simulation.

So, this is to Monte Carlo simulations for TEM specimen and of course you can see that the electrons are entering from this side and going out through this direction. So, basically this is a transparent electron transparent specimen and in some cases also you have a little bit of backscattering happening here the electrons just go out of the same surface through which they enter.

So, this one is of some low Z value, this one is high atomic number value. Most electrons of course has not scattered at all or scattered by a little amount that is the first observation for both kind of specimens only a little much. So, most of the electrons, incoming electron just go away through following the direct path some of them gets a little higher angle scatter and backscattering is really, really very less.

Also what we can see hit here that the amount of backscattering or amount of backscattered electron basically increases with atomic number this is for a high atomic number specimen and this case the number of backscattered electrons is much higher or higher than this one that is first. Second observation of course the high angle scattering and the beam broadening both of this is much higher for higher atomic number element.

So, ultimately what it shows this Monte Carlo situation the same thing that we were discussing earlier that this high atomic number elements is affecting the beam broadening and the yield of backscattered electrons and yield of high angle scattered electron all of this things are increasing for this atomic number with increase in atomic number compared to this element.

Now what we will be discussing in the subsequent session as a source of contrast generation so this difference which is coming in just because of the atomic number difference can be used. So, if I have these two elements next to each other in a specimen then I can very well distinguish them just if I capture either backscattered electrons or if I capture even this direct electrons or high angle scattered electrons. I can very well make a difference between them how and what we will discuss just in a moment.

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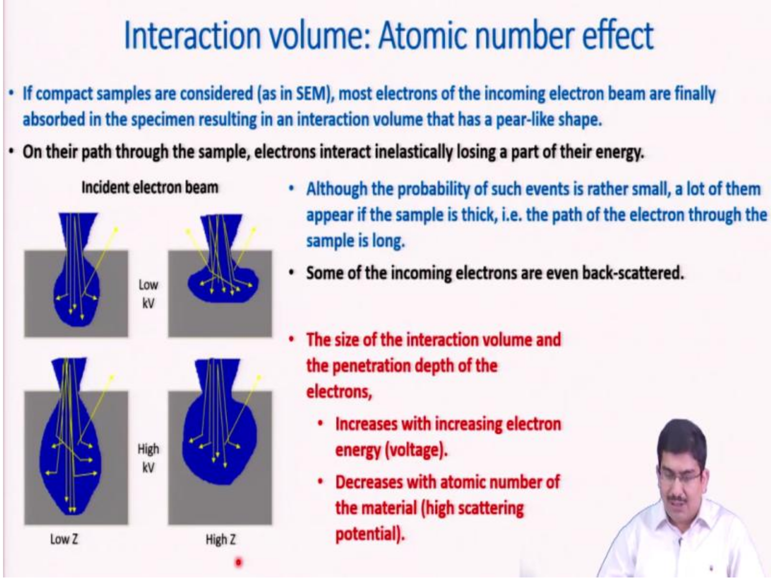

The second thing what we can see again a combined effect of atomic number and acceleration voltage. So, we have two situations again, but now this time it is in SEM not any longer we are not any longer in TEM that is why we will see that first of all this interaction volume is confined within the specimen it is not reaching to the other side that means it is an electron opaque specimen in scanning electron microscopy SEM.

So, we have this low atomic number element and this high atomic number element these two are there. Now again first observation is this most of the electrons are getting observed within here. So, both elastic and inelastic scattering is happening and the electrons are losing their energy slowly because of the inelastic scattering and finally getting absorbed here itself. Now, what we can also see is that some of the electrons definitely are getting backscattered from here.

But the important point to consider that this and one more interesting point is that the shape of this interaction volume almost remains constant in all the cases it is always almost like a pear shape. So, that is not changing much. What is changing is basically for example the depth of penetration if you just check this depth of penetration. So, this depth of penetration is in increasing with increasing electron energy.

So, if you take the same material, in this case same atomic number material then with increasing electron energy or accelerating voltage the depth of this or even the size width also for the interaction volume is increasing same thing for this high atomic number material, but in this case the difference is not that pronounced. Also if you consider let us say between these two situations and these two situations of course this size of penetration depth will decrease with higher and higher with atomic number.

So, as the atomic number increases the depth penetration depth will also decrease for this interaction volume. So, with this we are stopping here our discussion about the interaction volume or interaction cross section all of this discussion electron material interaction we stop it here and we continue now our discussion about image formation in a transmission electron microscope.

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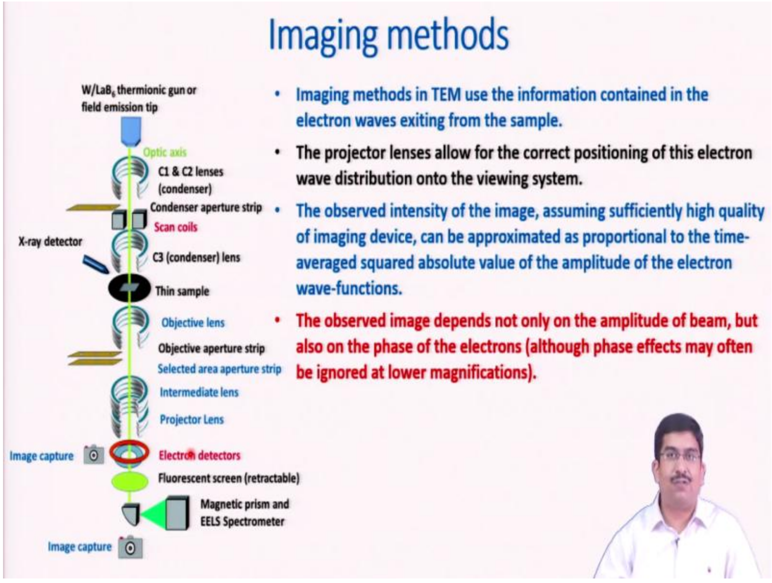

So, again the same schematic that we have already seen basically showing a cross section a schematic cross section of a transmission electron microscope. So, first you have the electron gun then you have the condenser lenses, condenser aperture you have a thin specimen, very thin electron transparent specimen, you have the objective lenses, objective apertures and finally you have some projector lenses and you have at the end you have a fluorescent screen.

So, this is very unique for TEM transmission electron microscope this detect (()) (23:21). We will discuss a little more when we discuss about scanning electron microscope we will discuss more in details about this detectors, but for now for electron microscope or for transmission electron microscope just understand that this is a fluorescent screen and we have already discussed about the fluorescence properties.

In this case what happens the material when an electron hits this material then it emits a light and since it is a fluorescent screen their emission is immediately decayed as soon as the beam stop electron hitting stopped immediately the light will stop and that intensity of the light is directly proportional to the number of electrons hitting that screen. So, here the contrast generation on those screen first of all the contrast generation is sensitive to the number of electrons that is hitting at any given location.

That is how first thing image forms on this fluorescent screen and of course the same screen can be recorded, same image can be recorded using any image capture device CCD camera or CMOS camera you can have and there can be many other different type of detectors on the path we are not going to that. So, basically here in transmission electron microscope the image formation is happening from the electrons which are coming out of the specimen.

So, before this we were discussing we were kept ourselves confined up to here. So, we have first discussed here the electron gun and then we discussed about lenses, scanning coil etcetera. So, we were almost of the cases we were in the upper side. Now we are discussing and also we have discussed the electron material interaction here. Now we are discussing what happens afterwards.

So, let us image that I have let us say a coherent electron source (()) (25:13) or I have tungsten whatever thermo ionic gun, field emission gun whatever I have that is giving me electrons and more or less they are coherent electrons. those electrons are finally coming and hitting the specimen. Some electron material interaction happens I try to keep it as much elastic interaction as possible as much I try to keep it within the domain or within the plural scattering phenomena and then the electrons are coming out.

Now, from there our discussions starts. So, this electrons which are coming out they carries the information basically which will help in forming the image that means the electrons which are coming out from the specimen the information they carries about the contrast depending on the specimen surface. specimen feature depending upon the electron material interaction.

They will different locations will or the electrons coming out from different locations will carry different kind of information and that will finally produce the contrast over here in the fluorescent screen. So, now what we have to understand is basically what information this electrons are carrying and how this electron information are differing that is it. So, first of all in TEM mostly as I said that we tried to keep it restricted within elastic scattering.

So, basically the wavelength does not change, what it changes most often is the phase. Amplitude also most often amplitude does not change or at times the amplitude also changes, but more or less it is the phase change which always will occur for this electron due to the scattering. If I am talking about pure elastic scattering then more or less always there will be a phase change amplitude change may happen, may not happen.

And the intensity what we can imagine that this imaging device in the fluorescent screen, the intensity will be a time average square absolute value of the amplitude of electron wave functions. The electrons which are coming out of the specimen and finally hitting this fluorescent screen a time averaged squared so amplitude square basically amplitude square is intensity.

So, the amplitude square of this electron wave functions that will produce the ultimate intensity there and this final image the contrast that will form here will depend both on the amplitude and the phase of the electrons particularly the phase effects sometimes is ignored for lower magnification cases or for thicker specimens the amplitude or it is mostly dominated by the amplitude effect the phase effect is not that much phase contrast.

But for a thinner specimen at higher magnification because they are thinner specimen, higher magnification, thinner specimen means mostly it is elastic scattering where phase changes more obvious than amplitude change. So, for those cases we must consider the change in phase in the electrons and that how finally will affect the intensity here after superposition after interference.

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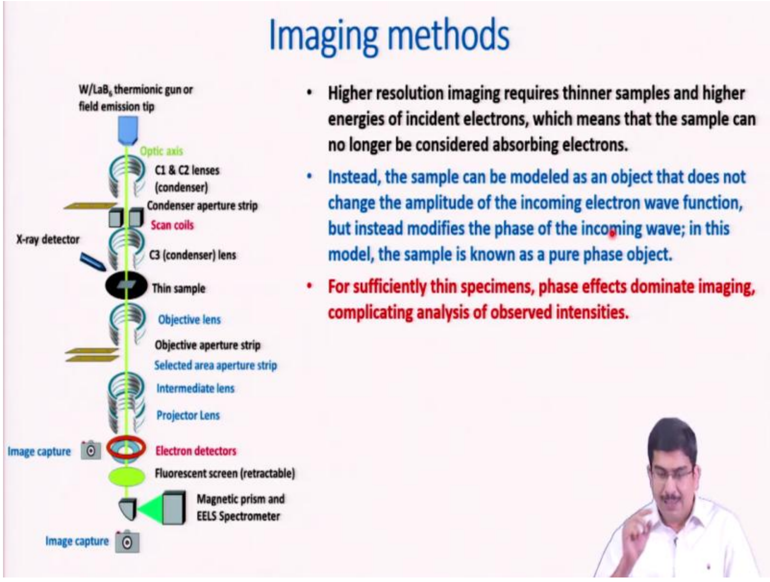

So, again what we can imagine that definitely thinner samples what I was saying the thinner specimens and higher energy means the samples will be like what we can imagine that the samples are not absorbing electrons. So, there is no change in their energy and it is purely elastic scattering and only phase changes happening not much of amplitude change. So, amplitude change is mostly happening when there is an inelastic scattering that is what changes the wavelength, that is what changes the energy amplitude mostly.

Phase changes purely happening when elastic interaction happens that is what we intend to do in a TEM. So, for TEM specimens we mostly get a phase contrast rather than an amplitude contrast particularly for very thin specimens.

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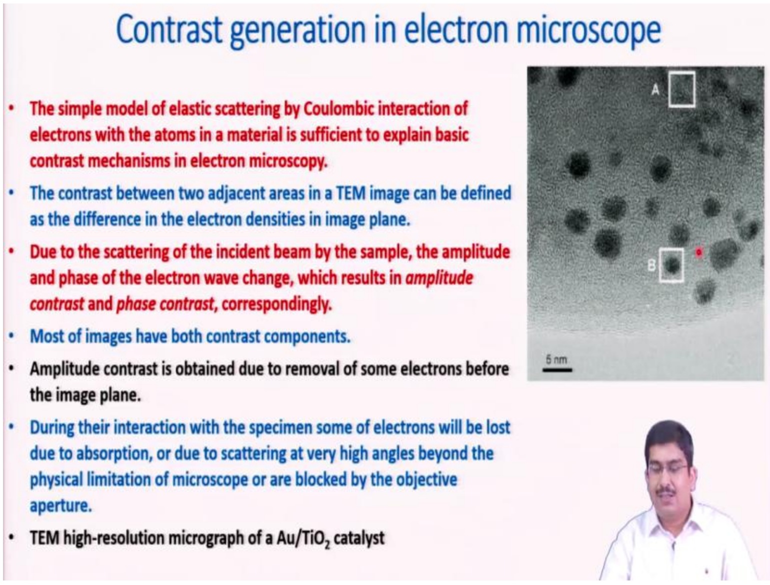

So, finally of course contrast generation in electron microscopes as I said it is a function of how many electrons are finally hitting and plus the electrons which are coming out what is their amplitude, what is their phase all of these things will finally dictate the contrast generation on the fluorescent screen or for that matter on the recording device that I use. So, this in order to understand this contrast generation we can simply consider the coulombic interaction that we were discussing when we were discussing about electron material interaction.

It was mostly based on the coulombic interaction between the incoming electrons and the electron cloud plus the positively charged nucleus. So that concept is quite okay for a rough or for the preliminary understanding about the contrast generation in a electron microscopy. So, as I already said if I consider this image which is basically a high resolution TEM image for two regions.

One is gold, one is TiO_2 or rutile catalyst. So, it is basically the number of electrons hitting. So, these are definitely from this two different regions. At this time I am not saying which one is gold, which one is TiO_2 that we will discuss later, but definitely because of the presence of these two elements or these two different phases which has completely different density, completely different atomic numbers.

The electrons which are coming out of that will be completely different in nature. So, when they hit the fluorescent screen they will produce a completely different contrast in the final image and most often as I said that because of the scattering the incidence beam of the sample the amplitude and phase changes and phase changes more obvious so finally it results in a amplitude contrast and phase contrast.

Amplitude contrast of course is achieved because of the removal of some electrons from the imaging planes because in the interference if some electrons are removed if I just take the direct beam for example we will discuss more about this. So, if I just think that I am capturing direct beam for imaging and some part of the beam is removed because of the scattering.

So, then finally when it hits this fluorescent screen number of electrons will be less and that will produce a lesser intensity because the amplitude of those final amplitude which is just a linear summation you can imagine that will be less. So, that is a pure amplitude contrast. The same thing will happen because of the phase as well because this electron which will finally interact due to scattering again they will have some different type of phases.

And that can lead to some interference and some places there will be change in intensity as well. So, because of this amplitude and phase difference I will be finally having this kind of a contrast difference and remember contrast is all about difference. So, these two different phases will give me two different type of electrons, two different intensity in the final fluorescent screen and that will give you contrast as well.

So, we will stop here today and we will continue about the contrast generation electron microscope more discussion we will continue it in the next class. This discussion we will be having in next session. Thank you.