Techniques of Materials Characterization
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Lecture - 35
Optics of SEM (Continued) and Analytical Detectors

Welcome everyone to the NPTEL online certification course on techniques of materials characterization, we are in seventh week and we are continuing with scanning electron microscope. So, we have a till now discussed about various basic components of SEM and mostly we discussed different detectors, secondary electron detector, backscattered electron detectors and so on.

And then we were for the last two lectures we were discussing about the optics. Part of an SEM, how the resolution is calculated in SEM, how depth of field is calculated in SEM, how those things are related to the probe size to the beam current and so on and so for. So, we will continue with that discussion a little more. And we will try to derive certain expressions of getting an ultimate resolution of an SEM, what should be the minimum probe size from practical considerations.

And then we will start discussing about some of the analytical detectors that is the composition of detectors.

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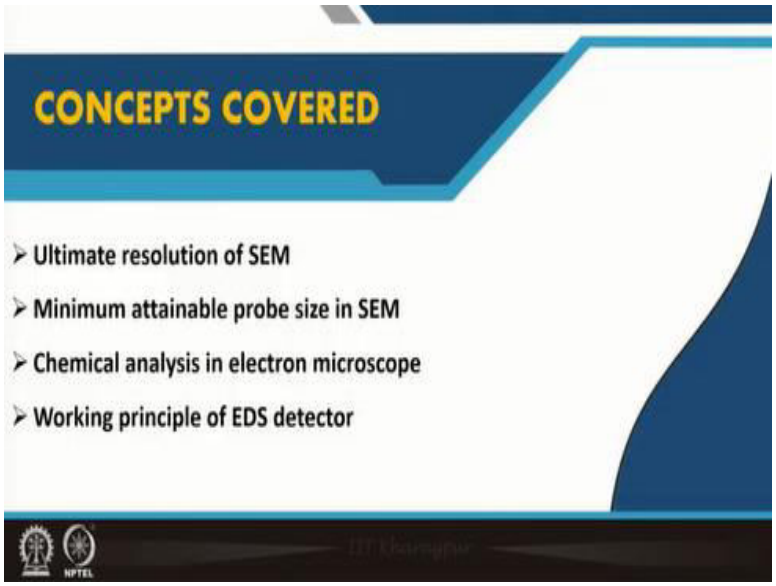

So, the concepts that will be covered here in this lecture will be ultimate resolution of SEM and then minimum attainable probe size in SEM for the best resolution. And then we will discuss about the chemical analysis in electron microscope and today we will just start about it and discuss about the EDS detector, working principle of EDS detector.

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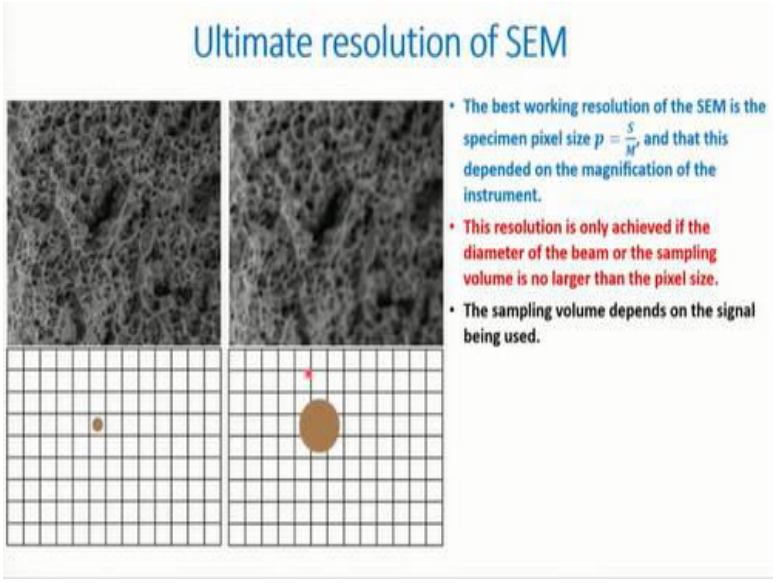

So, let us first discuss about the ultimate resolution of SEM and this we have discussed before the relationship between the pixels or size of the pixels in an SEM versus the resolution that we can achieve. And we said that for getting the best resolution or the most practical case will be when the spot size or the beam size or the beam diameter, probe diameter is of the order of the pixel size. So, that is how the resolution is related to pixels in an SEM.

This is not this is something special about the scanning mode because only the scanning mode has this pixel concept. If we consider some other mode of image formation, the direct mode for example, if we consider the transmission electron microscope, this is not what limiting the probes or limiting the resolution the pixels at least not the pixel. May be the; beam size that can be achieved or the semi angle of convergence by various lenses spherical aberrations.

All of these things are very important in direct imaging, those are important here also. But here more important or equally important, if not more is the pixel size. What is the pixel, specimen pixel size that we are having? And of course, the best working resolution is the specimen pixel size. We have already seen that if it is bigger than this that is if we are having a probe size which is much bigger than this pixel size then signals will be mixed.

And in that case, we will be losing the resolution it will be all blurry, the entire image will be very blurry. If it is smaller than the pixels then we will be having a chance of missing information and many times we will be also hitting the interfaces. So, signal again chances of missing the signals are high. So, the best situation is when the pixels are probe diameter is around that of the pixel size.

And then we will have a very good resolution, very nicely focused beam and very good resolution on this. Also, we have seen the pixel size how it is related to the specimen pixel size, how it is related to the display pixel size. And display pixel size for most of the cases display pixel size are constant. So, it is the magnification which basically determines the specimen pixel size, what will be the specimen pixel size.

Because this usually is around 100 micron obviously that can be changed but this usually is around 100 micron. You do not want to bring a different display in order to get a different magnification keeping the specimen pixel size constantly. In order to improve the resolution, you really do not want to bring in another display. You want to do everything in the SEM site itself rather than changing the display or the image capturing device.

So, usually this S remains constant and with change in magnification, the specimen pixel size changes. That means we also discussed about this effect of like the relationship between the pixel size and the resolution and the imaging. And what we discussed that the probe diameter basically should also be changed with magnification. This is the reason for in order to get the best resolution at high magnification where the pixel size is very small I need to suitably adjust the probe diameter as well.

And then we have seen that in order to adjust the probe diameter we have to also adjust the beam current because that is also inherently related. So, this resolution now as we understand this is related to the pixel size. And this resolution is achieved if the diameter of the beam or the probe diameter or rather the sampling volume is not larger than the pixel size. Now, what is the difference between the probe size and the sampling volume?

We just briefly discussed in the last class. But now, we are going into more discussion about this. The sampling volume depends on the signal that is used. This we have seen earlier also when we were discussing about the interaction volume.

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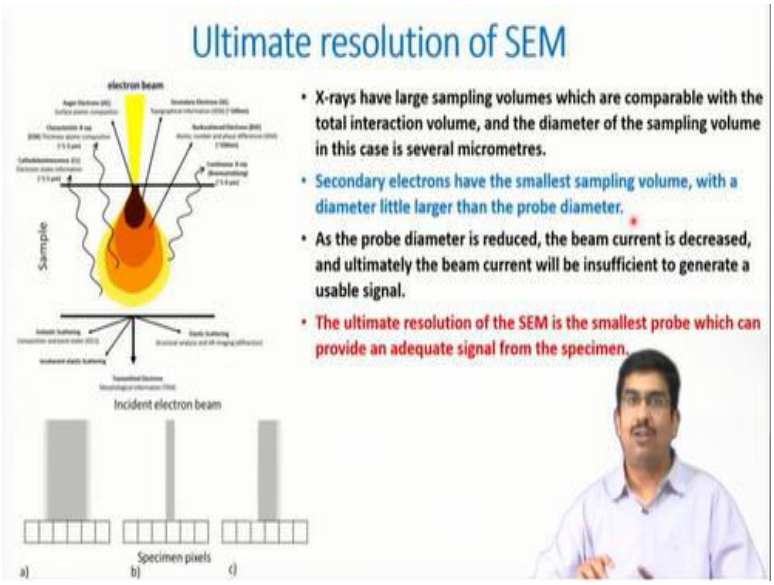

We saw that not every signal has the same kind of interaction volume and in turn they do not have the same kind of sampling volume. The area from which on the specimen surface, the area which that produces the signal and the depth from which the signal is coming these two things

basically determines the effectively the beam diameter. So, even if let us imagine that we are talking about the X ray signals.

So, even if we think that our probe diameter is this much only this, the signal is actually coming from a much larger area both on the surface as well as in the depth in the volume. So, this 3D volume the interaction volume of this X ray signal is much bigger compared to the beam diameter and also the sampling volume on the sampling area from which the signals are coming is of the order of several micrometres.

They are comparable with the total interaction volume the sample volume. So, that means, in case of X ray signal if we want to get best resolution out of an X ray signal, we cannot work with a very fine pixel size and then adjusting the beam according to the pixel size that we choose. So, what does it mean? Understand this very correctly. Here the effective beam size that we need to consider is basically the size of this interaction volume.

And not the probe diameter that we earlier calculated, the d_0. Basically, we calculated $d = d_1$ in to (v_2/u_2) and then we get the relationship in terms of the crossover and so on. That is not effectively now for an X ray signal that is not the size of the beam that we need to consider. The size of the beam is basically the size of this interaction volume or the sampling volume. So, our pixel size in order to get the best resolution no mixing of samples.

So, in order to achieve this kind of a condition where the size of the pixel is matching the size of the sampling volume we should consider and we should try to select our pixel sizes as per the size of this interaction volume, not the beam diameter that is the point with X rays. Now if we consider secondary electrons, we are in a better situation. Because in secondary electrons, the sampling volume if we just consider SE1 signals, at least then the sampling volume is very very small.

And of the order of the beam size itself. So, the interaction volume is very close to the surface, it is not going deep within and the sampling area also is in and around this beam which is falling. So, for them effectively we can achieve a very good resolution even with a very small pixel size.

So, that is a point. So, this interaction volume or sampling volume for SE signals are very small. And that is why it will be much easy to achieve smaller pixel size even if we use a smaller pixel size.

We go at a very high magnification, we use a very small pixel size we can still achieve a very good resolution because the interaction volume of secondary electrons are very very low. So, from that side onwards, Backscattered electrons are somewhere in between they are having bigger interaction volume compared to the SE electrons but of course they are not as big as the X-ray. That is why backscattered electrons have at least a better spatial resolution than X-ray signals.

And that is why in X-rays, we will discuss about this possibly a little bit when we discuss about the working principle of detectors. But more or less when we use the X-ray signals, we do not try to go to higher magnification. Because in that case what will happen the beam interaction volume will be larger than the pixel size. And also remember one thing that in order to get in order to achieve a detectable signal the beam current also is important.

So, if we reduce somehow by some means, if we reduce the beam diameter, we also need to assure that we have to get enough signal out of it. So, this is a very big problem for X-ray signal. So, as the probe diameter is reduced the beam current will be also decreased and ultimately the beam current will be insufficient to generate any X-ray signal at all in the first place. And detector always needs some finite amount of detector also has its resolution in terms of detecting.

How much energy difference or how much wavelength difference it can have in the X-rays. So, considering that we need to have a considerable signal strength for this X-ray signals. And that is why we cannot go beyond a certain amount or certain beam diameter, less than that it is not practicable to go. So, the ultimate resolution in the SEM we can think of as the smallest probe which can provide an adequate signal from the specimen.

And this smallest probe should be calculated considering the entire sample volume and not the theoretical beam diameter that I can achieve by adjusting lenses or the apertures. So, that is how

these things are related and that is what is dictating the ultimate resolution of the SEM, of any SEM.

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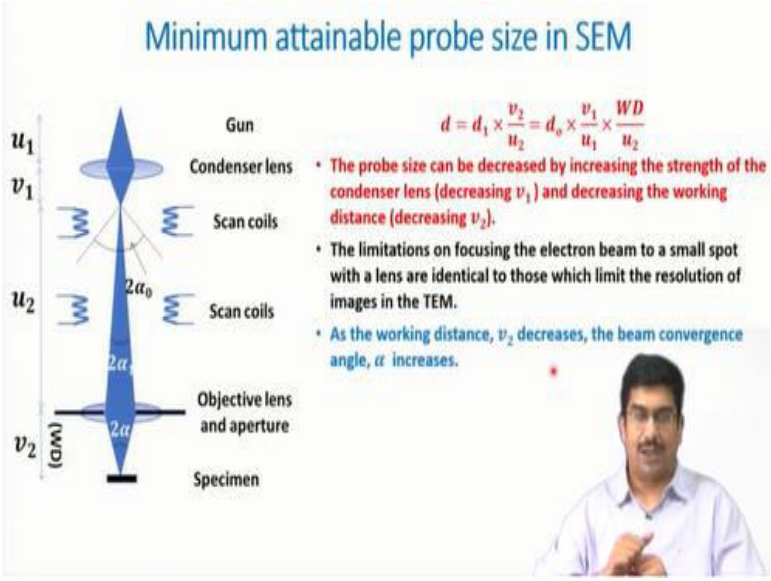

Now, let us discuss what is this minimum attainable probe size? And again, considering this is a theoretical consideration. So, we are just considering that what beam and this is valid only when the beam diameter and the sampling volume are of the same order. If this is order of magnitude different than these calculations are not valid like in X-ray signal. So, basically what we are imagining that here the signal is mostly SE signal.

And the interaction volume or sampling volume is within the diameter or order of magnitude within the diameter of the beam. So, in that case, let us consider what should be the minimum attainable probe size. So, we know this relationship by now, we have seen this many a times and we know this relation that the final beam diameter here d depends on these distances, the working distance finally depends on this v_2 that is this working distance.

And it depends on the u_2 that is this image distance between the condenser lens where the condenser lens focuses the beam from there all the way to the objective lens. And then we have a few other things like the d_0 is the crossover size and this gun here and u_1 is again the distance between the crossover and this condenser lens and v_1 is the strength of this condenser lens. So, that where the condenser lens is basically converging all the electrons.

So, from this we have also seen that the probe size, final probe size the d here in the specimen. The final probe size can be decreased by two ways, we can do two things to decrease it. Number one, we can either increase the strength of the condenser lens that is we can reduce its focal length, we can ask or we can make the electrons to converge at a much smaller distance from the condenser lens or in other words by decreasing v_1 here.

From this relationship either by decreasing v_1 by increasing the strength of the condenser lens. Or otherwise, we can also decrease this working distance we can try to bring the specimen close to the objective lenses as close as possible. So, in that case v_2 will decrease. Now, the point is if we do this adjustment now, we are not discussing about this adjustment we are already I have discussed how what is the problem of working too much with this condenser lenses and so on.

We are discussing about this, if we try to change the working distance what will be the problem. And in nature these problems will be pretty much similar to the kind of problem we face in case of transmission electron microscope when we consider the resolution from diffraction effects. So, we have seen that we calculated the diffraction Rayleigh's criteria and all those things from there we have calculated certain diffraction related minimum resolution.

And then we have seen that how in order to achieve that, how we are sort of compromising with something like spherical aberration. Because spherical aberration has the same effect α semi angle which reduces the diffraction related resolution is making the spherical aberration related resolution worse because these two goes in opposite direction. Here also what we will see that these two things are in nature the problem is pretty much same.

Like as we decrease the working distance here. That means we are taking the specimen closer to the objective lens we are decreasing v_2. The beam convergence angle now this α this semi angle of beam convergence is increasing. So, if we are taking without changing the objective lens too much, I mean objective lens as I already said the purpose of the objective lens is to focus this beam on the specimen irrespective of the working distance.

Whatever be the working distance the objective lens must focus this beam on the specimen itself. If it is not focused defocus then of course and it is beyond the limit of the depth of field then of course it will be out of focus, the features will be out of focus it will not be of any use. So, the beam will be optimally focused on the specimen surface always. If that is true then if we decrease the working distance what will happen is that the beam convergence angle will increase.

As we are moving it upper and upper this alpha angle will increase that is what intentionally also from our intuition also, we are able to see that we are taking a closure. And now for everything remains the same this angle is getting is increasing, alpha is increasing.

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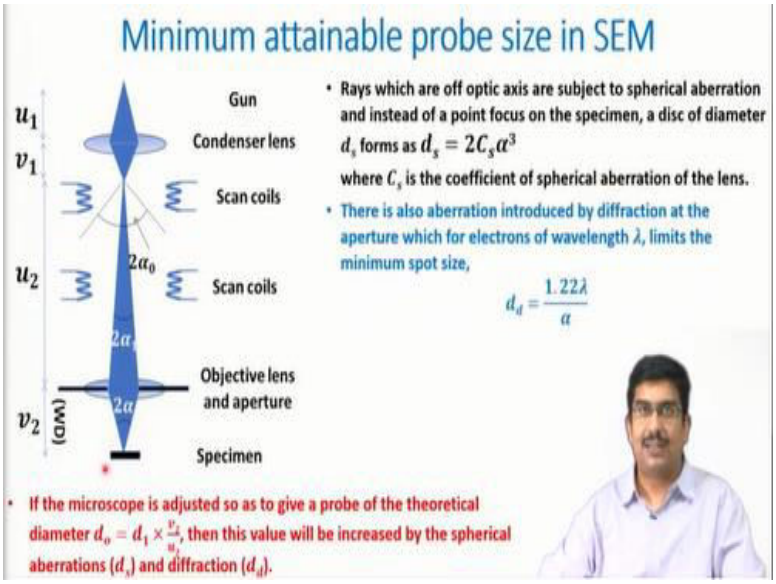

So, if alpha is increasing now that means the rays which are off optic axis like the convergence angle is increasing. So, the rays which are far off from the optic axis, they will start causing a spherical aberration. So, aperture is not it is like increasing alpha it means is like taking out the aperture. So, aperture is increasing. So, we are allowing more and more rays which are off optic axis which are diverging.

So, increasing the convergence angle means we are allowing more of a diverging electron and that will increase the spherical aberration. And what it will do again the same thing, instead of a point focus on the specimen, a disk will form. And the disk of this place diameter list focus that

disk the diameter of the disk will be if we consider d_s it will be $2C_s \times$ (alpha) to the power 3. This is the same spherical aberration where C_s is the coefficient of spherical aberration.

And we have discussed about this in the general concepts of microscopy as well as during discussion in TEM. So, if convergence angle is increased in order to reduce or in order to improve the beam diameter simply from this calculator using the lenses or aperture in an effort to increase the beam diameter by reducing the working distance, what we are seeing is that spherical aberration is increasing now.

The second point will be of course this aperture and remember, we have discussed this in terms of transmission electron microscope. Whenever you have an aperture, you will be having a diffraction effect. That diffraction effect will also limit the resolution and also it will extend or it will also cause the beam to sort of increase the size of the beam basically, if you can consider in it that way. So, the diffraction effect from the apertures will also increase the size of the beam.

In a similar way they did it for like in a transmission electron microscope. So, they are the diffraction they are the beam size was solely determined by the diffraction effect first and then the spherical aberration, all other aberrations was started coming. Here, we have to consider three different effects. First, the beam size that is inherently we obtained because of the presence of these lenses that is the first one.

Then how much this beam diameter inherently whatever the beam diameter that we are going to get because of the lenses, how much it is changing due to the diffraction effect from this aperture system in the objective lens plus how much decrease or how much degradation in the beam size or what the beam size how it is increasing how much it will increase because of the spherical aberration. These two effects we have to consider now.

So, if we consider that the microscope is set for giving a probe size minimum probe size, d_0 where d_0 again from the same equation. We can instead of d_0 writing here we have written this as d_0 just in order to differentiate. So, d_0 is now we know the $d_1 \times (v_2 / u_2)$. So, then d_1, d_0 whatever d_0 we are supposed to get because of the lenses because of the presence of this condenser lens.

And objective lens whatever d_0 whatever the beam size we are supposed to get here that value will increase by spherical aberration d_s and the diffraction related effect d_d, these are the two things.

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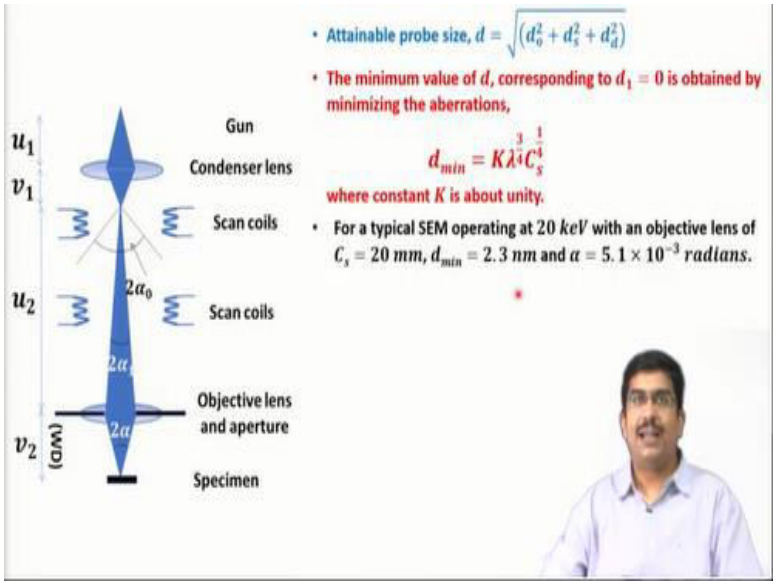

So, finally, the attainable probe size that will get here d that is given by this formula root over (d_0 square + d_s square + d_d square). So, this is how the final probe size is calculated here considering diffraction effects and aberration effect. So, this is the practically this is what the beam size we get from certain microscope configuration. This is what will determine what is the minimum beam size or what is the maximum or whatever the minimum beam diameter that we can achieve.

Now, if we want to calculate the minimum value of this d where we corresponds to $d_1 = 0$ sorry this should be d_0. So, this d_0 means basically from the lens system what is the beam diameter that we are going to achieve if we set that even to 0. Then for minimizing all other factors diffraction related factors and aberration related factors the final beam size that we get we d_0 is set to 0.

Remember and then minimizing these two factors only diffraction and spherical aberration the final beam size that we are going to get is of this much K lambda (3/ 4) C_s to the power (1/ 4). This is exactly again the same beam size the same resolution that we have calculated during the

our discussion on general concepts of microscopy. There also we said this is the maximum or this is a minimum resolution that can be achieved in any electron microscope.

We are deriving the same thing now for a SEM. But the consideration here is that the beam is perfectly focused on a point, the beam does not have any diameter at all on the specimen surface which is again not practical. So, beam will definitely have a certain finite diameter and on top of that, we will have certain increase in the diameter of the beam due to the spherical aberration and the diffraction that is it.

If we now put some typical values here like we imagine that accelerating voltage is 20 keV that means the lambda we calculate the lambda for the electrons from this accelerating voltage. And we use an objective lens with this spherical aberration coefficient of 20 millimetre then the d minimum that is a minimum attainable probe size will be around 2.3 nanometre. And alpha value that will come from this same calculation we can find out the alpha value.

And that will come around 5.1×10 to the power - 3 radians. So, this is the kind of values we will be able to achieve in a typical SEM around 2 to 3 nanometre of probe diameter considering a very optimized condition almost a point focus, very minimal spherical aberration, very minimal diffraction effect and so on. We can achieve 2 to 3 nanometre diameter it is possible in an SEM.

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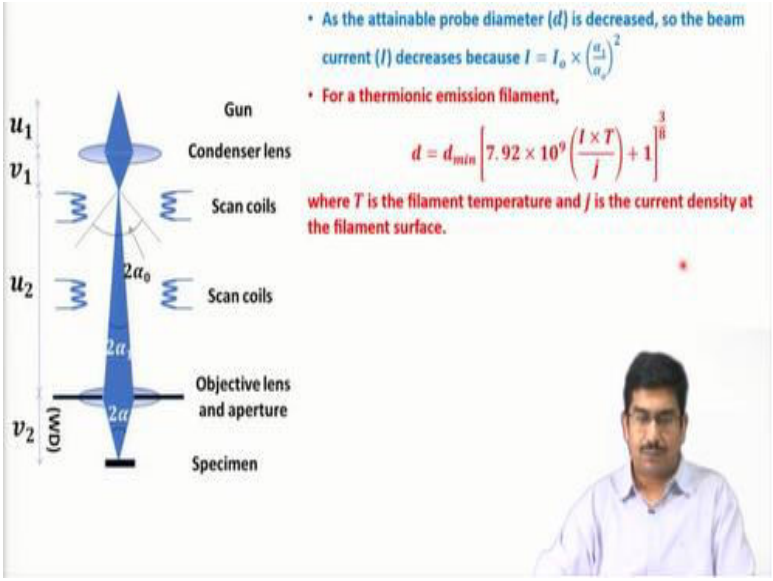

Now, the point is that is not all; that is not the end of the story. As I already said in order to get an ultimate resolution, so ultimate resolution will be that minimum probe size where I will get some finite amount of signal as well, being current as well. So, if the beam current and we will make this relationship even further. We will take it further and we will see that when we will discuss in the next module when we will discuss about imaging.

We will find out that what is the; relationship between the beam current and the signal that is generated out of that beam current. So, that is also important, we will move another step there. So, just you might just understand this that in order to attain this minimum probe diameter d, the beam current also decreases. And it is decreases because of this relationship $I = I_0 \times \{(alpha)1/(alpha)0\}$ square.

So, if here, alpha 1, alpha 0 all of these things are decreasing and then in order to get a minimum probe diameter, the beam diameter if we decrease the probe diameter will also decrease the current. And if we use a thermionic emission filament then to get something of this minimum beam current, this is how the probe current depends on the beam current and as well as the minimum probe size.

That is attainable plus some other factors which are basically like from the filament. So, this is how in a thermionic emission filament. The maximum or the minimum probe gets modified because of the requirement of a minimum attainable current. And that current has to be generated. Remember, the current definition of current means, it is the number of electrons which are present within the beam.

So, in order to get that much of current in the first place generated by the thermionic emission filament generated by the gun itself, the beam size gets modified further in this case at least. You know and that is another advantage why we tend to use the FEG sources. Because they are we do not need to worry too much about the electron size we can go for a finer beam size. So, it is not only like in a transmission electron microscope we have seen use of FEG.

How it is helping in improving the resolution, because there we were able to minimize the aberration. Particularly the spherical aberration we were able to minimize the spherical aberration by chopping of most of the beams which are off optic axis, very fine aperture we were able to use. Here in this case in SEM, not only that, it also since in SEM it is not direct imaging.

It is always remember that in SEM the imaging is done by signals which are produced due to inelastic scattering mostly. So, the beam current is pretty important here. In case of TEM, the lambda value was important, spherical aberration was important, the beam current was always quite a lot in order to generate a certain a good amount of contrast in the final image and that is a direct image. Here it is a pixel by pixel image.

So, the specimen in order to generate a good contrast it also needs that the specimen generate a good amount of signal and for that the beam current is pretty important. So, this source is also important in the process that it should have enough adequate beam current, it should generate adequate beam current in the first place. So, that even if the probe diameter is reduced in order to get the best resolution, the beam current should still be enough.

And that is ensured only in field emission gun, thermionic emission guns are not that efficient in order to give a very high beam current in the first place. So, that is another point to remember about why we want to use even field emission gun for scanning electron microscope.

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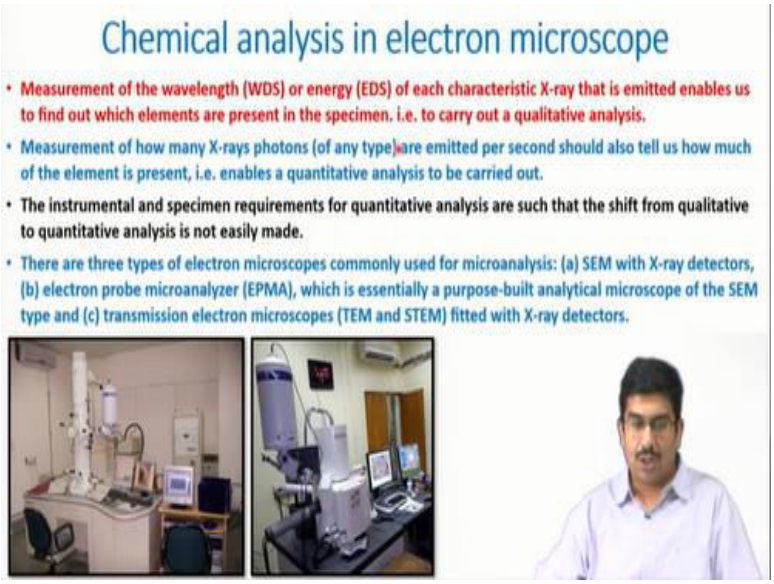

So, from here we will just quickly start the discussion on chemical analysis in electron microscope. So, in electron microscope the chemical analysis can be done in two different ways. Basically, either we can measure the wavelength that is called wavelength dispersive spectroscopy or the energy of the X-ray signal characteristic extra signal which is called the energy dispersive spectroscopy.

So, either we can measure the wavelength or energy of the characteristic X-ray which is emitted from the specimen due to the electron specimen interaction. We have learned about it the secondary effects, the inertial excitation and how it is leading to some secondary effects characteristic X-ray generation and auger electron generation and so on. So, either we can measure the wavelength or energy of that X-ray and from that we can find out which element is present in the specimen.

So, this is a qualitative information we are just able to ascertain the element or identify the element present in the specimen that is one way. The second one what we can do is that we can measure the how many number of photons of any type are coming X ray photons are emitted per second or the rate of X ray coming out from the specimen. And then, by proper calibration, we can find out which how much of that particular element.

So, we can identify the nature of the element by either measuring the wavelength or energy of the characteristic X-ray. So, we know exactly whether it is copper, it is aluminium, it is iron, it is lithium and sodium whatever is there plus if we measure in the detector, we can measure the number of X-ray photons coming per second or the rate of X-ray photons which are coming up and then we can back calculate.

We can have calibration, properly calibrated system we can back calculate and find out how much of what is the amount of that element present within that specimen. The point is the qualitative doing a qualitative analysis is not that difficult, but doing a quantitative analysis is very very difficult. So, this shift from qualitative to quantitative, qualitative to quantitative analysis is very very quiet difficult.

Because it involves that calibration step in between and the calibration study is not so easy. We will not go into how the calibrations are done and so on because that will open a big that itself will be almost one more module, we will not go into that. But we will simply discuss about different types of detectors I know a little bit of out there working principle. The calibration part called Z calibration and all that is a little difficult one to do.

So, basically there are three types of electron microscopes commonly used for this analysis chemical analysis. One is SEM with an X-ray detector whether it is like EDS detector most often is the EDS detector that you normally use to see whether you see a transmission electron microscope it comes with an EDS detector. Basically, this is the detector and this is a storage tank for liquid nitrogen, we will come into that.

So, this is the detector which can be attached to a transmission electron microscope or a STEM system as well. And same detector same kind of a EDS detector can be attached to an scanning electron microscope as well. And then there are another type of very specialized electron microscopes that is called electron probe micro analyser or EPMA system which is a purpose built analytical microscope of the SEM.

Basically, it is an SEM except that the probe size is much bigger in order to ensure adequate signal and so on. So, it is a little purpose built and particularly meant for chemical analysis and quantitative analysis most importantly quantitative analysis. And usually carries an wavelength dispersive spectroscopes plus sometimes energy dispersive spectroscope or most often energy dispersive spectroscope plus WDS.

So, WDS and EDS both detectors are usually present in EPMA whereas, normally in normal SEM and normal TEM we usually sees EDS detector more commonly than WDS detector. And these are the three different types of electron microscope generally used for micro analysis. So, we will stop here and in the next lecture we will be covering this EDS detector and WDS detector. So, we will stop here. Bye.