Techniques of Material Characterization
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Lecture – 26
Signal generation in SEM

Welcome everyone to this NPTEL online course on techniques of materials characterization we are now in 6th week module 6 and we will be discussing about scanning electron microscopy from now on and previously we have covered in the first few modules we have covered general concepts related to microscopy techniques like how resolution, what the definition of resolution then magnification, depth of field, depth of focus, various type of aberrations and so on which is very general to any kind of microscopy.

And then we discussed about optical microscopy and there we learned about various ways image can form in an optical microscope and then various modes of optical microscopes, fluorescent mode, interference contrast, phase contrast and so on and various components as well, objective lenses, condenser lens and so on and then we switch to transmission electron microscope.

And before starting transmission electron microscope actually we studied about some general concepts of electron microscopy like electron material, interaction, electron gun, electron lenses and scanning problem and so on. And then we studied about transmission electron microscopy where we saw that image formation, how the image forms, how the contrast forms in a transmission electron microscope. Various sources of contrast mass thickness contrast, compositional contrast and various modes, bright field, dark field and then HAADF mode, STEM mode all various things in imaging.

Then we discussed quite in length we discussed about electron diffraction. And in there we discussed about like how the electron diffraction basically happens, what is diffraction, Bragg's law, reciprocal lattice, Ewald sphere construction. And then how to index this diffraction patterns, what information we can get out of it, zone axis determination various things we discussed.

And now we are switching to scanning electron microscopy. And scanning electron microscopy is another very important topic or very important type of instrument that is there under big umbrella or under that universal umbrella of electron microscopes, scanning electron microscopy is one very important member of it and today we will discuss about signal generation in scanning electron microscopy.

How different type of signals are generated and then we will move to something like how detection happens with that signals.

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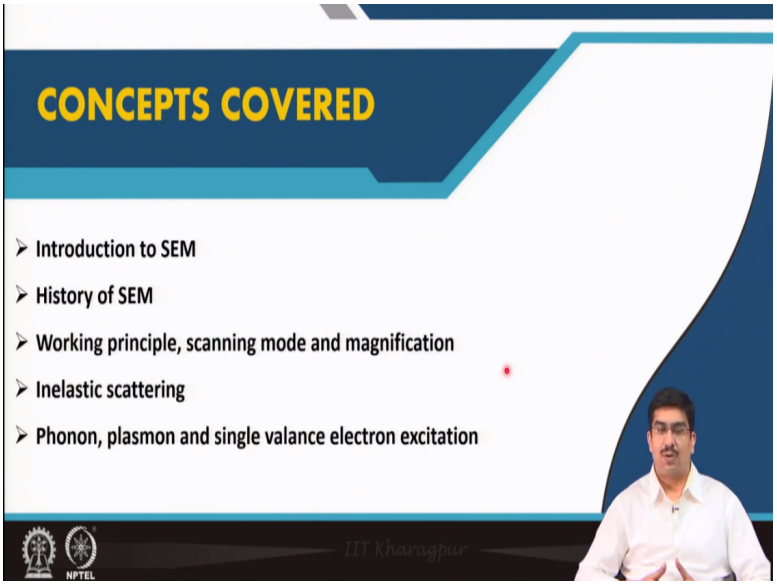

So, today we will start with a brief introduction to SEM and then a little bit of history of SEM. Of course, we will discuss about working principle, scanning mode as the name suggest it is a scanning electron microscopy. So, what exactly is the scanning mode and magnification how it is produced in a scanning electron microscope and then we will switch to signal generation by inelastic scattering then various type of inelastic scattering phonon, Plasmon, single valance electron.

And then in the last class we will discuss about various other type of signal generation through this inelastic scattering.

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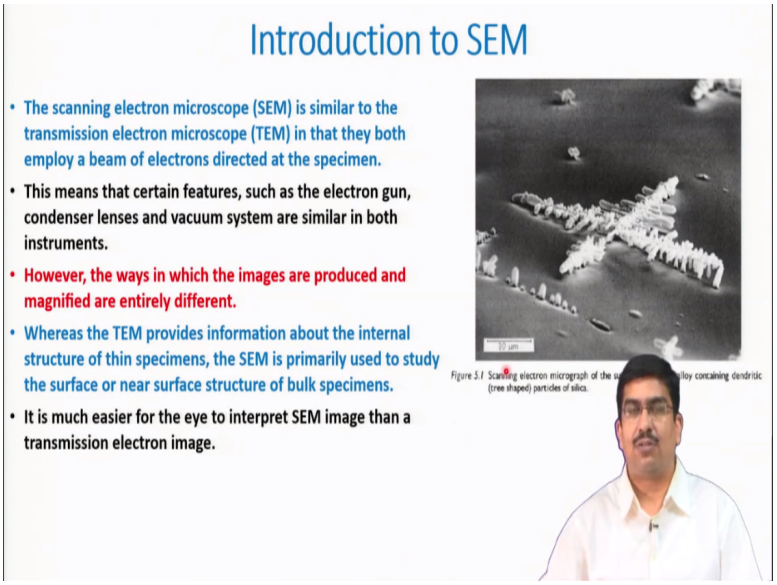

So, introduction to SEM, our introduction to scanning electron microscopy; the first thing is as we already understood scanning electron microscopy is another type of microscope, but in configuration wise many of the parts are very much similar to transmission electron microscope. So, basically these two are I can tell like this to our two almost equivalent in the way they work or in the configuration many of the components are pretty much the same.

And the way they are constructed that also same many of the principles are exactly the same, but obviously there are certain differences and those differences we will understand when we discuss about this SEM. Time and again we will draw analogy with transmission electron microscope and the differences with SEM. The main commonality between SEM and TEM is that they both of them they employ a beam of electron.

So, here the source signal is the same electron beam, detection signal may vary, the way detection happens may vary, but the source signal is an electron beam and here they are different from optical microscope where the source signal is a light. This means since electron is a source signal in this case both type of microscope SEM and TEM so certain features are common between them like electron gun.

So, just like TEM we have seen that electron guns can be either thermionic gun or can be field emission gun here also we have pretty much the same type of guns then the lenses electromagnetic lenses also are pretty much same at least the condenser lens part is pretty

much the same. Even that configuration of the electron guns everything is same, you have the anode plate, you have the acceleration of the electron that is generated out of the electron gun everything is same, condenser lens up to condenser lens basically it is the same.

The difference happens afterwards which we will discuss. The main difference between this scanning electron microscope and transmission electron microscope basically lies in the way the images are produced or rather the signals are produced, what images are produced means what develop this contrast, how the contrast is generated and that inherently related to how the signal is produced in these two types of electron microscopy that is quite different.

And of course how these signals are captured, how the magnification finally happens, how finally the image appears all of these things are different between these two. So, another very fundamental difference between transmission electron microscope and scanning electron microscope in that TEM basically provides information about the internal structure of a specimen.

That means here we are mostly concerned with electron transparent specimen. So, by configuration itself in TEM.

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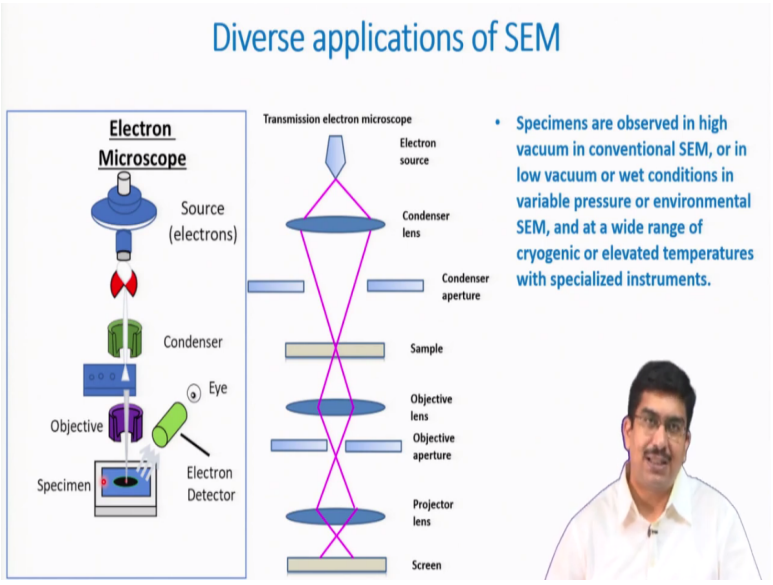

If we just see this configuration 2 of SEM and TEM so this side it is the scanning electron microscope and this side it is the transmission electron microscope. So, the first difference

that they happens other than the commonality as I said from source to condenser lens almost up to condenser less it is pretty much the same for both whether it is a TEM or whether it is a SEM that is quite the same.

Afterwards, the biggest difference happens is that in this case in SEM what we use is a electron opaque specimen. So, we use a thick specimen where the electrons cannot penetrate whereas in transmission electron microscope as the name suggests we have a specimen which is electron transparent. So, electrons can pass through it. How it is passing through that is a different question. It is not only to do with the sample.

It is also to do with the beam in case of transmission electron microscope the beam the acceleration voltage is very high of the order of 100 to 300 kilo electron volts whereas in SEM we hardly use 10 to 30 electron volts or sometimes even lower than 10 kilo electron volts we use for SEM and again the sample is also thin down in this case TEM case sample is very, very thin.

And in specimen in SEM you do not have any such restriction like it is an opaque. So, you do not really have any restriction in the thickness whatever the specimen chamber, whatever the instrument allows you to put you put it. So, I have seen SEM where actually a real big component in a millimeter range components also can be put. So, these are special SEM made with a special purpose and all.

But basically in terms of sample what kind of a sample you can scan that is very versatile, SEM is very versatile whereas in transmission electron microscope you really have to make it all the way down to electron transparency that is one of the main difference and in the process what happens is that since in this case the sample the electron passes through the specimen.

So, the contrast is generated from this entire thickness. So, entire internal structure whatever is generating the contrast whether it is a mass thickness contrast, whether it is a compositional contrast, whether it is any other kind of contrast, diffraction contrast, whether it is a

dislocation contrast whatever it is that is throughout this specimen. So, the entire specimen is responsible for contrast generation.

So, if you remember the dislocation contrast that we discussed not necessary that dislocation has to be on the surface, the dislocation can be somewhere in within the specimen and still if the G cross B condition is satisfied the specimen will show that dislocation in the final image which forms here that is the difference. So, that means I am actually seeing an internal contrast or in other way round if I let us say if I have in the electron transparent specimen if I have something like a higher atomic number element or higher atomic number precipitate somewhere within this I should be able to see it in the final image.

So, it is completely showing the internal structure of any material of this sample. So, entire thickness whatever features or whatever mechanism is contributing to this contrast generation that is spread throughout the specimen whereas scanning electron microscope is very much related to the surface or a subsurface level. It depends on what kind of a signal you finally use for your imaging or your analytical studies, but more or less it is the surface phenomena.

All the surfaces, all the samples or all the signals are generated mainly from the surface and that is why what you are seeing is basically lies on the surface. So, that is the main difference one of the major difference between SEM and TEM the way the images are generated or the information those images they carry that is the first thing because of this since this SEM basically shows you the surface features or surface whatever the condition of the surface.

It is easier to interpret this SEM images than a transmission electron mechanism. There is other reason for that and we have discussed this in transmission electron microscope there are many different sources of contrast generation and since as I said it is throughout the specimen. So, it is very different to interpret or contrast generation mechanisms are difficult to interpret those images.

You have more than more than four, five different types of contrast formation mechanism and this is throughout the specimen. So, finally to interpret that image is really, really difficult and it is also very difficult to deconvolute those contrast formation mechanisms that is another

difficulty in case of transmission electron microscope whereas SEM the contrast generation mechanism are mostly we will discuss about this.

But contrast generation mechanisms are mostly one or two and that too you can deconvolute them and these are almost pure. So, finally what happens is that interpreting these images is pretty easy and if I have to draw an analogy what I can say is that SEM images basically the kind of features when you see a surface, when we see with a naked eye. So, exactly the similar kind of image we will see in SEM.

So, as if we are just seeing it with a naked eye just it is that features that features are getting magnified to us. Similar kind of images like a optical images in a reflection mode. So, you can imagine that in optical microscopy the reflection mode the kind of images that we see is pretty much similar to scanning electron microscope and the kind of images we seen the transmission mode is basically in optical microscope is similar to transmission electron microscope. So, these are very important differences between this.

And as an example you can see this image and this is pretty much like what we would have seen this feature if it exist in the millimeter range where we can see it with a naked eye. So, as if this feature exists in millimeter range and we are just seeing it only difference now we are magnifying it by the electron microscope.

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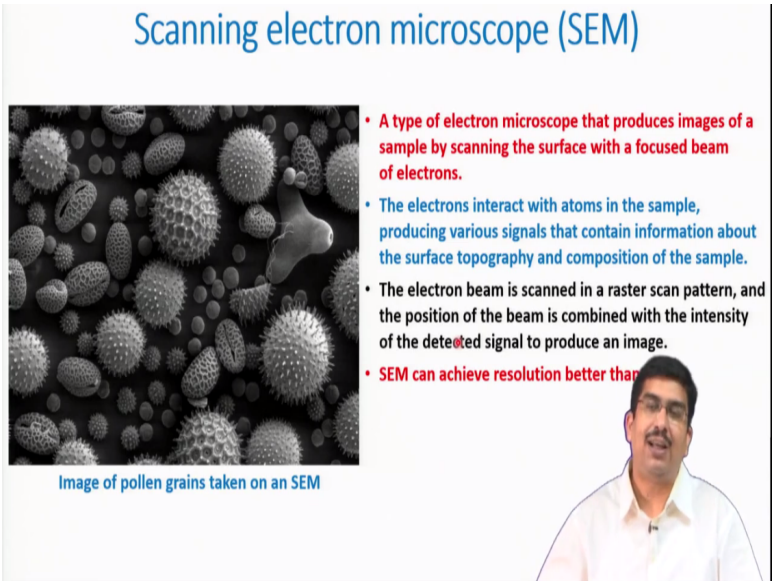

Again one more example you can see that what kind of images it can produce this scanning electron microscope pretty much the surface features that you are seeing, you are not able to see what is lying within. So, this is an image from a pollen grain and looks pretty much like viruses and these days it is almost you know this kind of structure shown for viruses as well, but this is an image from a pollen grains which also looks like the same.

Mother Nature makes it almost the similar way and you do not see anything inside basically. What you are seeing the features which are present on the outer surface. So, this is pretty much the surface phenomena and in this case the images are produced by scanning the surface with a focused beam of electron that is another difference. In case of a transmission electron microscope this is like a direct beam.

So you are having either you take a direct beam to generate a bright field image or you take the diffracted beam to generate a dark field image it is like one single beam. It is a direct image one time the entire fluorescence screen is illuminated, contrast is generated you are seeing that. It is not any scanning. In case of scanning electron microscopy basically you scan point by point a focused beam of electron is produced.

And then you scan it point by point over the entire sample. So, this image if you take this image this image is not one single or not caused by one single electron beam. It is caused by a beam which is moving point by point. First it is moving possibly like this then it is moving like this then it is moving like this so on and so forth.

It is not one single beam that is why usually the beam size the spot size of SEM is much smaller than that of transmission electron microscope which is forming actually a much bigger because you have to I mean obviously it depends on magnification as well, but more or less the beam which you use for SEM is very, very small and in this case again electron interacts just like transmission electron microscopy.

Unlike transmission microscopy of course here the contrast is mostly produced by secondary ways or when the beam interacts with the specimen there is certain different type of signals that is produced we will discuss about that. There are certain different type of signals are

produced compared to the primary beam and those signals used for final image formation whereas in transmission electron microscope you use basically the primary beam at least if you are in bright field mode you use the primary beam.

And depending on the sample, depending on the mass thickness contrast composition and so on whatever changes happen in the direct beam, primary beam that generates the contrast finally. This is another big difference between these two mode and SEM as I already said in this case this is electron beam is made to scan, raster the beam and of course these days modern days many a times this is also a notion that transmission electron microscope can give you very good resolution almost atomic level.

We have seen that also in HAADF mode, STEM mode and so on. Scanning electron microscope if it is suitably made and if it is suitably all the adjustments are done with a lenses and with scanning it can also give you resolution around one nanometer of the same level resolution is possible with modern day scanning electron microscopes very regularly. In fact in STEM mode when we were discussing about STEM modes in TEM where the TEM beam was made to raster the specimen in order to improve this resolution.

So, that scanning mode inherently have a resolution advantage because here you are using a very small beam and that beam you are made it to raster so your beam size is less and that inherently increases the resolution we will come to that. Another big difference between scanning electron microscope and transmission electron microscope is the diversity of SEM. So, this is a very unique point and because of that why I bring this issue is that every year when I have this in my department when I offer this course I used to get a lot of people.

A lot of research scholars, lot of master students I used to get them from various diverse departments like civil engineering, like mechanical engineering, like biology department, like Zoology department I used to get them. And when they come and when I ask them that why you are attending them this course on materials characterization? One answer I get sir I have to use this technique and when I ask them that what kind of a technique you are planning to use or you think you are going to use? Invariably I get two answers.

One is scanning electron microscope, one is x-ray both of this I rarely hear anybody saying anybody saying that I need to use the transmission electron microscope. So, transmission electron microscope is pretty much a specialized technique which is used mostly at this still this time which is mostly used by material scientist, which is not so widespread for other disciplines like biological there is a problem I discussed.

The problem with the biological specimen is that transmission electron microscope it is such a high energy beam is used 100 KV, 300 KV beam is used that the carbonaceous material those live specimens cannot sustain that. They will burnt off and we will see that a lot of heat is generated in that process, but in scanning electron microscope since we are using very low energy electron beam it is still possible to scan live specimens or a carbonaceous material so that is one reason why SEM is much more popular in various other disciplines that is A.

Second thing which is very good with SEM is that many of the manipulations can happen in this specimen chamber. So, what we can have is we can either have a high vacuum in a conventional SEM or a low vacuum even wet condition, variable pressure, environmental conditions everything we can have it in SEM, the way we want. In TEM, doing this is very difficult, Cryo TEMs are there of course where we can cool the specimen up to that level and we can even use it for like biological samples or carbonaceous material we can try to see them under TEM that is available.

It is extremely difficult and not so popular whereas these cases having an environmental controlled SEM specimen chamber or SEM holder is very easily available, commercially available first thing and not very costlier. The main reason for this or again you can have even elevated temperature also you can bring here. In these days you can even do some mechanical testing under this SEM chamber.

Such things it is very difficult to do in a transmission electron microscope to heat the specimen, to check in situ testing in the transmission electron microscope is much more difficult than to do it in scanning electron microscope. The main problem which according to me or when my students ask me that why it is so problematic? The main issue here is the simplicity of this scanning electron microscope.

Whereas if you see this transmission electron microscope and this is a very simplified diagram, but here itself you have so many different lenses and so many different type of lens configuration you have a screen, you bring lot of detector here so it is very complicated design. So, instrumentation part is very difficult that is one thing in transmission electron microscope.

So, to make this part work with anything, any changes in this sample here in the sample chamber anything, any environment you create, you try to heat them, you try to deform them anything you do here it is very difficult to accommodate and adjust that with this other parts whereas in scanning electron microscope it is pretty simple. So, after this specimen after the electron beam heats the specimen the signal generation you only have this detector, electron detectors to take care there is nothing in between.

This entire other part is basically just the electron beam comes and heats the specimen up to there that is it. So, if you try to change or try to create an environment or try to do any kind of in situ studies here it is possible in SEM without affecting the other part or electron generation part, electron focusing part, electromagnetic lens and all without affecting that part too much you can separate them out that is first thing.

Second thing I also used to tell people that in this transmission mode not only electron beams you have to bother about this part you have also bother about this part when the beam passes through the specimen. So, that means it is really difficult you have very little space available here to create any kind of environment or to do any kind of in situ testing. Here it is much more easier because this side the back side you do not have anything.

So, you can make it as big as possible and remember the depth of field and other things in scanning electron microscope basically depth of focus also is very high. So, where exactly you are detecting this does not make much of a difference you can really make it specimen chamber quite, quite large and the way you want you can do it. So, that is why the diverse this SEM is having very diverse application.

And of course it is also as I said the instrumentation is much simpler it cost less and maintenance is also quite less. So, all of this together putting together the scanning electron microscope is a much popular technique than transmission electron microscope. Of course, transmission electron microscope the biggest benefit it gives is the kind of resolution it can achieve, the magnification that you can achieve and so on and so forth.

So, it is s specialized equipment which is mostly still now used by material scientist whereas scanning electron microscope is more like a popular kind of electron microscope.

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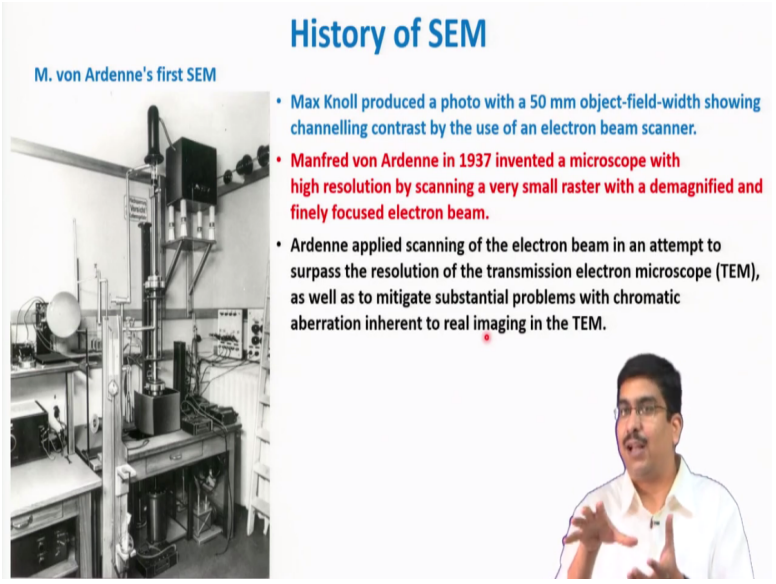

So, history of scanning electron microscope we already discussed once, but at that time we were discussing history of electron microscopy in general. So, basically scanning electron microscope people were trying it almost of the same time when Ernst Ruska and Max Knoll they were working with their first prototype of transmission electron microscope. Since that time itself people were trying to create this scanning mode.

But it was very difficult at that time with TEM that was a high energy electron beam to make it scan that is the problem, why they were looking to scan it? Two reasons number one preparing a transmission electron, transparent specimen is very, very difficult and there are other issues with it, various other issues like you have image sources and so on. If time permits maybe we can have discussion on that someday.

Electron specimen preparation for TEM it is really difficult. So, most often, but preparing an opaque specimen and most of the material naturally occurring materials are electron opaque. So, to do electron microscopy for opaque specimen you need to generate like you have to make that beam to scan and to raster so that you can get really very high resolution. This similar kinds of resolution which you can get with transmission electron microscope.

So, this is why people were looking for this to develop the scanning mode and the first person to shows it is Manfred von Ardenne and he invented a microscope with a very high resolution by scanning over a small raster with a de-magnified and finally focused electron beam very fine electron beam we focused it on the specimen and then he made it to scan and this is possible because electrons as we already discussed they are charged particles.

And you can deflect them by using a electromagnetic lens these days. So, altogether this is how the scanning electron microscope was basically came into picture and this entire of course the effort started with a transmission electron microscope and slowly to overcome certain difficulties of transmission electron microscope for example Ardenne main reason to go for a scanning mode was that he wanted to mitigate the problems with chromatic aberration inherent chromatic aberration in TEM.

Chromatic aberration means already we discussed that it is basically something to do with the variation in wavelength with this transmission electron microscope at that time field emission guns were not available. So, to do with this to remove the chromatic aberration was very difficult in TEM to get such resolution whereas if you are finally focusing a beam and made it raster you can basically remove, you can use a sort of an aperture.

And you can restrict yourself, you can restrict beam spherical aberration you can reduce, you can restrict the beam close to the optical axis many a things you can do if you made the beam to scan.

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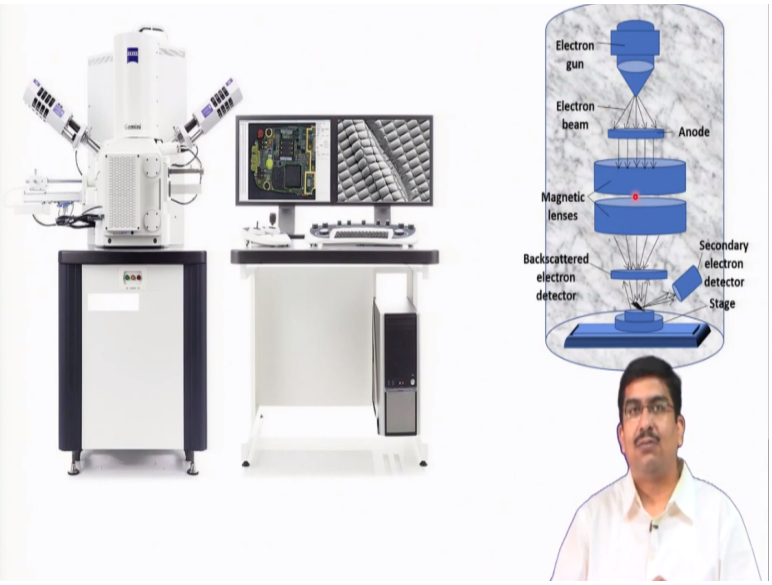

So, from this first prototype of SEM from this now we have come up all the way up to here this is the similar kind of electron microscope which is there at IIT, Kharagpur, Material Science Department and this is made by ZEISS and this has a Gemini column and so on. So, this is kind of a image that is very similar to the one that we have at our department and you can see one thing that what I was telling.

That it is here this entire thing is actually the specimen chamber this entire one and the beam the electron beam the gun, the condenser lenses, the anode everything basically this entire part is contained within this and this is one of the best commercially available SEM scanning electron microscope that is available right now in market even with that the size of this entire beam or where the beam is generated and beam is made to focus on the specimen.

So, this entire instrumentation part is really small and really simple, much simpler than the transmission electron microscope where the beam if you remember the column itself was really big and huge and that is why you can do a lot of things here with this specimen chamber, you can create different type of an environment, you can attach different type of detectors in this.

And various different modes you can do in this SEM scanning electron microscope. Here itself it shows that you have backscattered electron detector, you have secondary electron detector and so on and so forth. So, even the magnetic lenses are much simpler here less in

number and the maintenance of this scanning electron microscope is also much simpler because of this instrumentation, less amount of instrumentation here.

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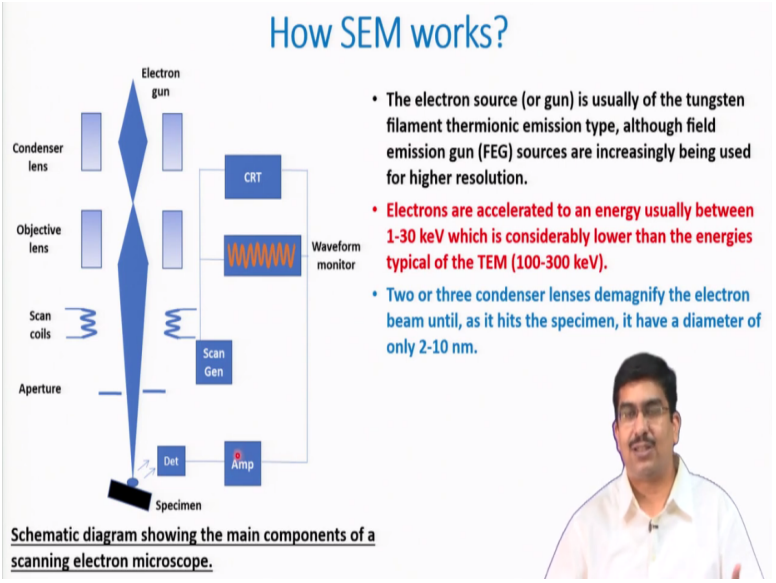

So, how the; electrons, scanning electron microscopes basically work. So, we have the electron source or gun here exactly same like transmission electron microscope. We can either have thermionic guns, but these days if you want to have very high amount of resolution it is better to use field emission gun basically the reason is the same, field emission gun can give you a much more coherent electron beam, much higher brightness that means a number of these electrons in the beam will be much higher so on and so forth.

So, basically the same kind of advantage which is there in transmission electron microscope which you utilize there, same advantages of field emission you use it here also then what you do is that now you accelerate this beam here, but at a much lower accelerating voltage, the anode that accelerates this electron beam after this crossover that is much less here around 1 to 30 KV you can vary depending on the specimen.

You can vary it usually for metals and non-metals samples it is 20 to 30 KV and for biological samples it is less than 10 that is usually that is what people used to do, but of course you can adjust this as per your convenience and as per your requirement, but definitely this is much lower than the energies which are typical for TEM where it is almost 100 to 300 KV 200 KV TEM is a most popular I think and frequently available.

And then what you have the is the condenser lens, you have objective lens and all and this basically de-magnify the electron beam until when it heats the specimen diameter is usually 2 to 10 nanometer of that sort of diameter the final beam is heating here and afterwards you have this scanning coils which made this beam to raster over this entire specimen and then you have detectors we will learn about the detectors not now, but in later classes.

So, you have the detector and you have this detection signal you convert it to current signal and from that you basically generate a display. How the display generation has happened we will study possibly all of these in the next class and all this inelastic scattering and other things everything we will discuss in the next class. So, good bye.