Characterization of Construction Materials
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Lecture - 39
Scanning Electron Microscopes - Parts and Functioning – Part 2

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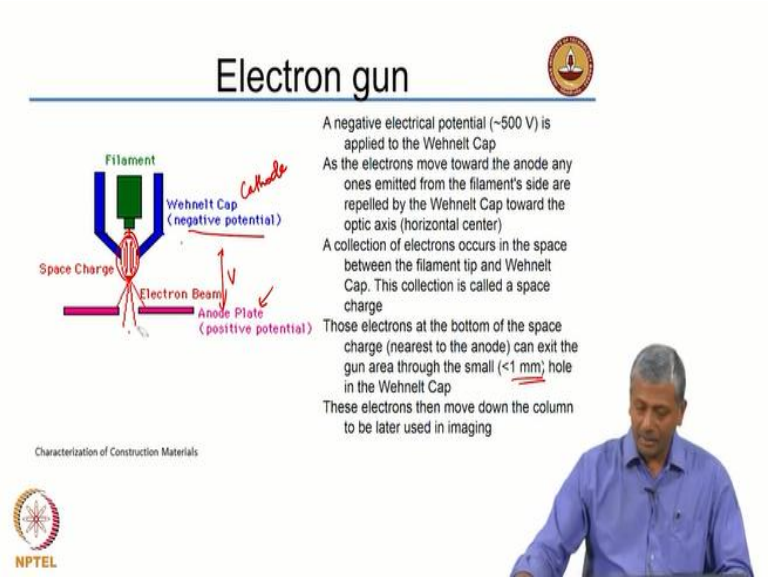

So, what is an electron gun? Electron gun basically, is similar to the tungsten filament that you have in your light bulb. So what happens in a light bulb, when you turn the switch on, it burns the tungsten film. The burning gives out electrons and electrons are basically ionizing the gas that is present inside the bulb, which leads to fluorescence, that causes the light to appear. So in this case, again, the filament could be something similar like a tungsten filament. So when you turn the instrument on and you apply the potential difference, the electrons from the tungsten filament start getting discharged and they are controlled through this Wehnelt Cap, which is at a negative potential, which is the cathode, towards this anode plate, which has a slit in the centre. So the potential difference is applied between the Wehnelt cap which is the cathode and the anode plate which is at a positive potential.

So, the electrons which are discharged from the filament move down from the Wehnelt cap into this slit which is between the anode plate and that is how they move down exactly along the SEM column. So, generally we apply a negative electrical potential to the Wehnelt cap.

As the electron move towards the anode, any ones that are emitted from the filament's side are repelled by the Wehnelt cap, because if you are on the side, the Wehnelt cap which is at a negative potential basically will repel them, because electrons are negatively charged. So, the cap will repel them to stay in the centre.

So what we will have is basically a space charge developing because all the electrons are basically collecting in that gap in the Wehnelt cap. And the electrons at the bottom of the space charge can exit the gun area through a very small hole of about 1 mm at the bottom of this Wehnelt cap. And then, these electrons then move down the column through the anode down the optic axis, and the higher the accelerating voltage, the faster they'll move down. So this is the electron gun, so for this we need to ensure that there is a good quality filament that you are using for imaging.

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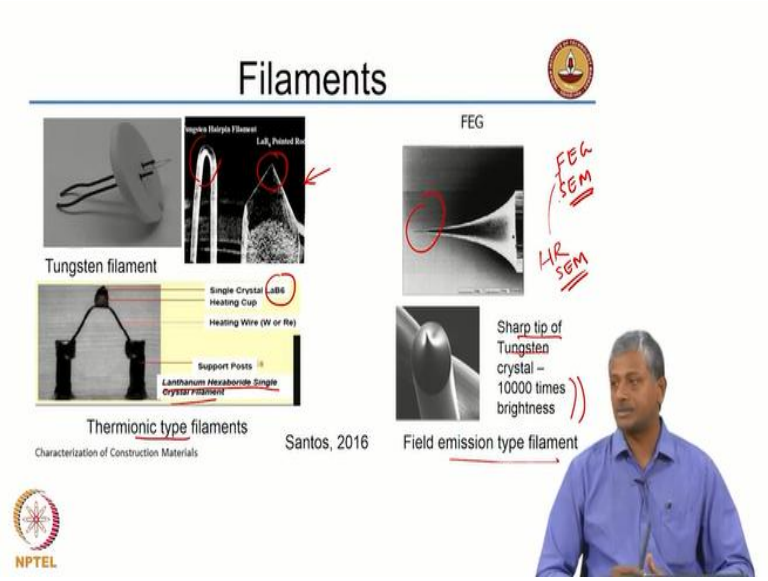

So types of filaments that are obtained can be either a tungsten filament, which is similar to the filament that you have in your electric bulb, or you can have thermionic type filaments, for example, the LaB6 - Lanthanum hexaboride single crystal filament. That is also a very common filament that is used and generally the kind of imaging capabilities provided by LaB6 filaments are much better as compared to your tungsten filaments. The only difficulty is LaB6 filaments will be much more expensive as compared to tungsten filaments.

So the tungsten filament as you can see here, it is actually a scanning electron microscope picture itself of the two filaments. So you have a hairpin-type tungsten filament here, and the pointed rod type LaB6 filament, LaB6 is nothing like Lanthanum hexaboride.

Now the other type of filament is field emission type filament. When you come across these SEMs which have been marked as FEG SEMs, that means the filament that is typical of most common SEMs like tungsten or lanthanum hexaboride is not what is present in this case. So here what happens is you have the tip of this tungsten crystal, which is capable of actually generating electrons which leads to about 10,000 times better brightness as compared to your typical tungsten filament. So again we are using tungsten in this case, but we machine it to such a sharp tip, that it is able to now discharge electrons which are able to give much greater brightness as compared to your regular filament. So when you talk about HR SEM, High Resolution Scanning Electron Microscopy, most HR SEMs are based on the field emission gun system, and not on the thermionic filaments.

What does Thermionic mean? Heat producing the discharge - so basically these are heated. Why does the heat appear? Because of charging the material, the heat appears, electrons are discharged, and then they come down the optic axis. But in the case of a field emission gun, you have a much better system, where you can actually generate electrons which create much greater brightness as compared to your regular LaB6 or tungsten filaments.

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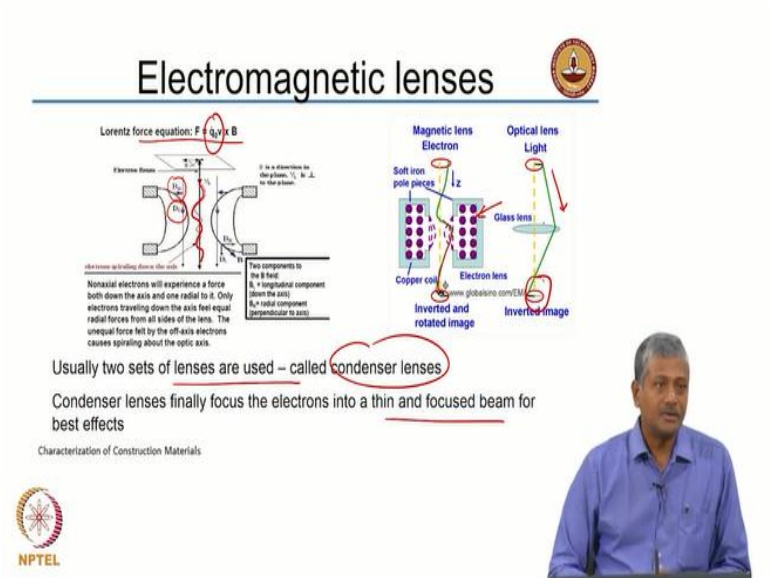

So once these electrons come down the optic axis, I said that you need to have these magnetic lenses to control the path of the electron so that they don't start straying out from central path, and move directly down the optic axis. So, from your high school physics, you may have come across this equation called Lorentz force equation and the idea is that as your magnetic

field is applied by coil around the electron beam the resultant field tends to push the electrons right down.

So there is usually a tangential component and a radial component of the magnetic field, but using your right hand thumb rule, you know that when you are applying this field around the electron beam, it will tend to push the beam right down the optic axis. So again, those of you who are interested in the physics of this lens can go deeper into this subject. There is a lot of information present in several textbooks and on the internet. But the idea is that you are able to send the electron beam spiralling down the central axis, and because of the field, the electrons will be accelerated more and more down this optic axis. The electrons are getting accelerated more and more down the optic axis.

So, usually we use two sets of lenses and these two sets are called condenser lenses. Condenser lenses basically finally focus the electrons into a thin beam for the best effects. And this thin beam then goes down to the objective lens system, which we will discuss next. So here again in an optical lens, you have a glass lens and the light goes through the lens and creates a certain focused image, which is on the other side. In the case of an electron lens, you have the electrons which are going down the optic axis. The electron lenses are electromagnets, which are basically coils which apply a field which spirals the electrons down the optic axis to go right on top of the sample itself for imaging. So, again, the idea is to have a thin and focused beam of electrons right in the centre to produce the best imaging effects. So after the condenser lens systems cause the electrons to keep moving down, and see here (Lorentz force equation) that the velocity of movement of the electrons also depend on the amount of field that is getting applied. So again, the electrons go spiralling down the axis, and then go through the objective lens systems to the sample.

Lorentz force equation: $F = q_0 v \times B$

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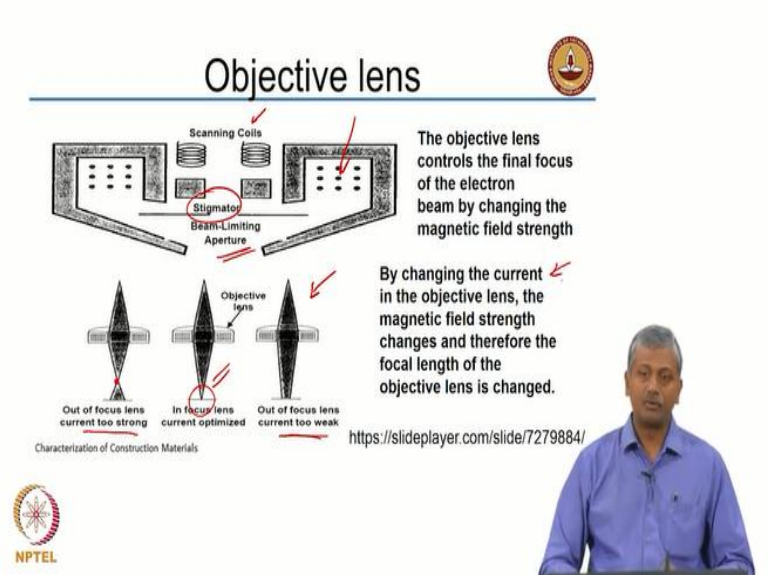

So what is the objective lens system? Once again, it has an electromagnetic coil on the outside which controls the level of magnetic field. It has also got scanning coils and a stigmator corrector. Please remember we talked about astigmatism earlier, which relates to the fact that you have two different focal lengths in the X and Y directions which causes the focus to be shifted or slanted towards one axis, rather than being equally aligned across both axes. So stigmation correction is also provided in the objective lens and there is a beam-limiting aperture which controls the width of the electron beam that goes down this axis.

So this is a simple schematic which is provided at the bottom. The objective lens system's main purpose is to ensure that you control the magnetic fields in such a way that the electron beam gets focused right on top of your sample or specimen, depending upon what you have. There'll also be this confusion when to call it a sample when to call it a specimen, please ensure that you understand the distinction.

So if the magnetic field is too strong, or current is too strong, generated by the objective lens, your image will become out of focus because the convergence happens much before the sample stage. In the case of a properly focused system, your focus is directly on the sample stage. Whereas, if you have a weak system then your focus is not converging on top of the sample but it is going beyond. So by changing the current of the objective lens we are changing the magnetic field strength, and therefore we are affecting the focal length of the objective lens.

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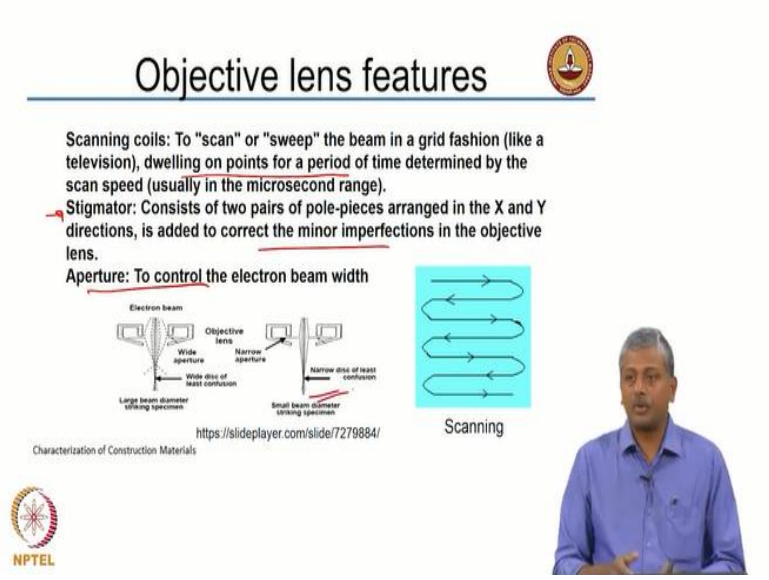

So again in scanning coils as we said earlier, we need to scan or sweep the beam in a grid like fashion, just like in the older televisions - the cathode ray tube televisions. So, while the scanning is going on, each point that is intercepted by a beam or where the beam falls is dwelled on for some time. Of course this is only a matter of a few microseconds so we do not actually get an estimate of that, but each point of being dwelled on. Otherwise what will happen is, you will not get any interaction from that point. So you need to get some interaction so each point is dwelled on for some time, depending upon the scan speed - how fast you are scanning, or how slow you're scanning. So, what would be the effect of scan speed on the kind of details that you get for an object? If you scan very fast, you will miss features on the sample. If you scan very slow, you will be able to collect more and more features on the sample or specimen.

The stigmator basically consists of two pairs of pole-pieces. The idea is to adjust the strength in such a way that the focus along the X and the Y planes is made equal. So, they are again magnets which are ensuring that the focus along the X and Y planes is equated. So again, we need to use a stigmator all the time, in the case of a scanning electron microscope to ensure that we do not get images that are skewed to one side. I will show you later some examples of images that are having this issue of astigmatism.

The aperture of course controls electron beam width. The smaller the beam width, the better will be the resolution. So we are able to actually get smaller features much more clearly by a limited beam width.

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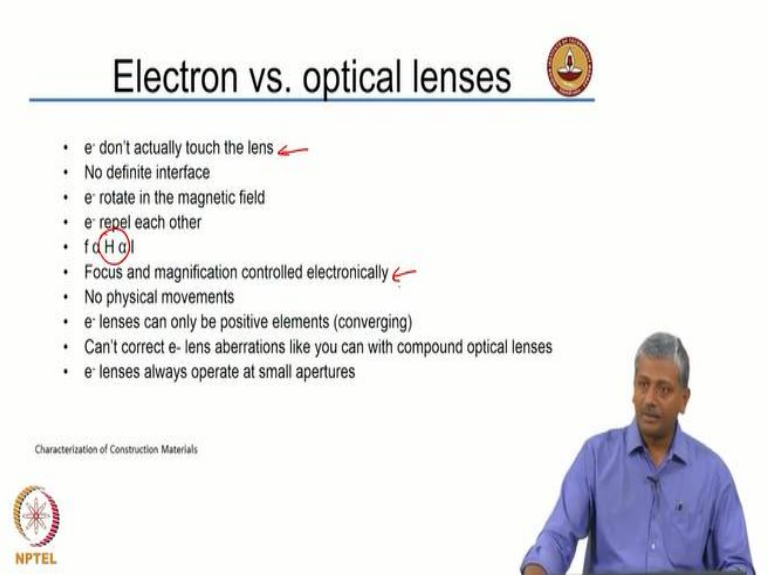

So, what is the distinction between electron lenses and optical lenses?

- Electrons do not actually touch the lens. The lens is nothing but the electromagnetic coil, which ensures that the electrons keep going down the central axis. In the case of an optical lens, obviously the light rays are passing through the lens.
- There is no definite interface - electrons rotate in the magnetic field, we actually get a spiralling sort of an action. Light rays on the other hand are going parallel.
- Electrons repel each other, light does not have that issue - the particles of light do not repel each other
- The focal length is directly a function of the intensity of the magnetic field and Focus and magnification are controlled electronically in the case of electron lenses. In the case of optical lenses we control the focus by moving the stage up or down. Even in an electron lens, or in a SEM system that can still be done.

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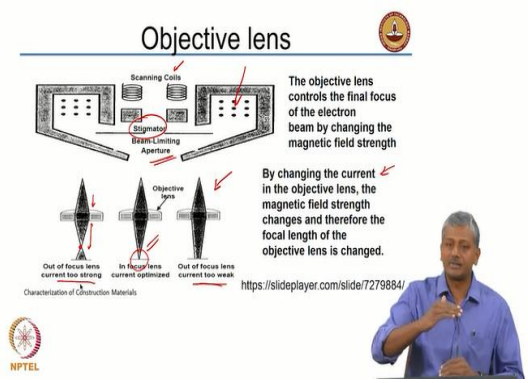

We saw here, for this strength of the objective lens, we can then shorten the working distance to bring the specimen into focus. Instead of adjusting the magnetic field of the objective lens, we can simply change the distance between the sample and the objective lens system to bring it into focus, in which case, what will happen is, we will have a very strong current, and that will help us get interactions much better with respect to your X-rays that are coming out of the samples. So we will talk later about when to use one particular system over the other, for example, when to use a shorter working distance and when to use a longer working distance.

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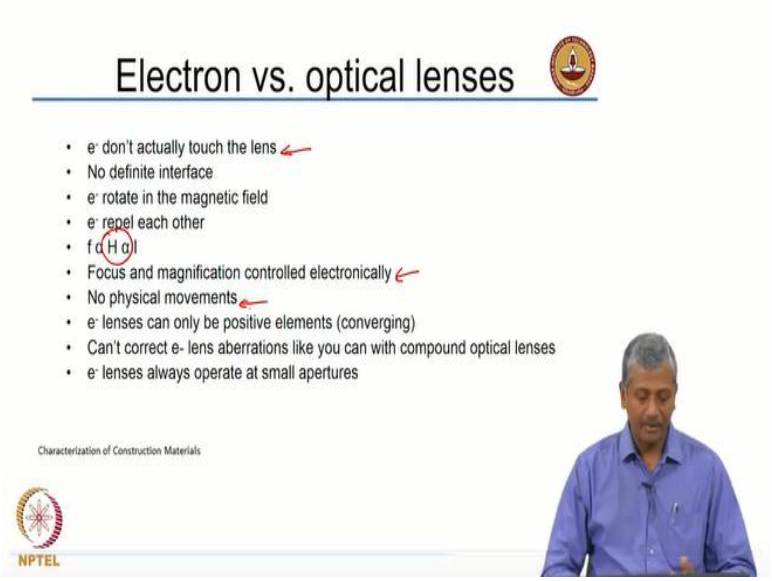

- In the case of an electron lens there are obviously no physical movements. The lens system is perfectly stable at a point. But in an objective lens system, you generally tend to move the distance between the sample and the lens often.
- Electron lenses can only be positive elements, in other words that, they are converging systems which make sure that the electrons always stay away from the lens.
- We cannot correct electron lens aberrations like you can with compound optical lenses. So if you have an eye defect, all you do is add one more lens in a spectacles or eyeglasses to ensure that you correct those defects, and in electron lens you cannot do that.
- Generally we operate with very small apertures with respect to electron lenses.

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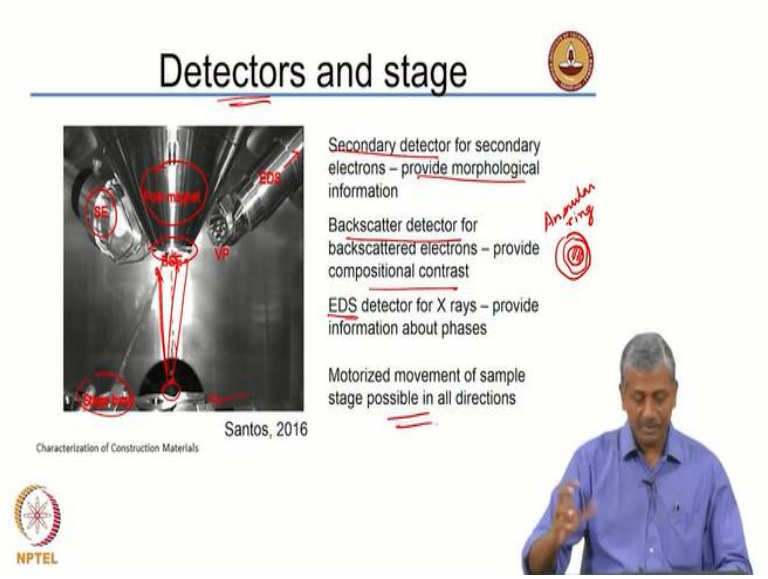

So let us look at what happens to the electron beam once it interacts with the specimen - that is more important for us. What are the detectors and how are they actually positioned around the sample stage? So here, this is the sample stage base (labelled in figure). This is where you are actually trying to keep your sample of specimen as the case may be. That is your pole magnet, or the objective lens system, which is ensuring the electron beams, come right down this axis, interact with the sample, and then the interactions need to be captured by what are called 'Detectors'. The detectors are of three types typically:

1. Secondary Electron Detector – SE or Secondary Detector - which generally are used for providing morphological information. For example if you want to understand the crystal shapes, sizes of features and so on and so forth, we will get surface related information by using the secondary electron imaging.
2. On the other hand, the Backscatter Detector is providing compositional contrast. So backscatter detectors are placed right around the objective system. Of course, you can't completely block the path of electrons, so usually the backscatter detector will be annular. So this will be the gap in-between and that will be the detector on the outside, so the gap allows the main electron beam to come right down and providing this annular ring around the gap ensures that all the beams that are going right on top after elastic rebound from the sample are getting captured by the backscatter detector. So that is an annular ring.
3. EDS detector or Energy Dispersive Spectroscopy detector is basically for X-rays that are coming out of your sample. Now we talked earlier about X ray fluorescence, which is when you have an incoming source of X-rays which interacts with the

sample and generates X-rays from the atoms of the sample, because of interactions that happen. That is called fluorescence. So in this case, the electron beam has a very high energy and when it interacts with the inertial electrons of the sample atoms, there will also be X-rays that are generated. These generated X-rays are then captured by what is known as an EDS detector or Energy Dispersive Spectroscopy detector.

Of course the motorized movement of sample stage is possible in all three directions, and sometimes even allows for rotation to happen.

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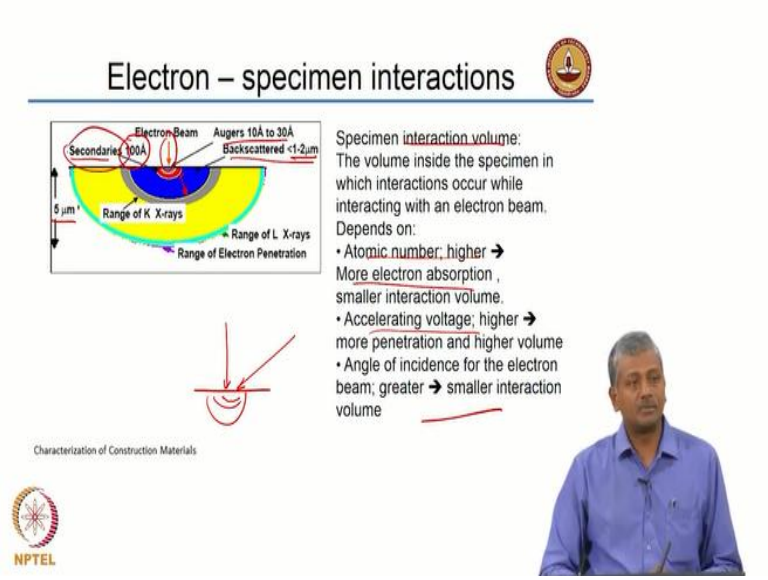

So what are these attractions which produce information about the material - that is again aspect of interest, as far as understanding the sample is concerned. So you have your primary electron beam which hits the surface. From the very near surface zones, it creates what are known as Auger electrons between 10 to 30 Å. So that means within the depth of 10 to 30 Å, we are talking about atomic or subatomic scales there. Within that depth it creates these Auger electrons. So actually if you read the literature, there is enough work that has also been performed on Auger electron spectroscopy. But collecting these electrons becomes very difficult.

The next set of electrons that is produced is from a slightly greater depth of about 100 Å or 10 nm into the sample. So again very much limited to the surface and those are the secondary electrons.

Still further deeper, up to about 1 to 2 µm, you collect what are known as backscattered electrons. Now, if your sample is extremely dense, what will happen? The

electron beam that comes in, will lose its energy trying to penetrate the sample, because there will be lot of absorption and scattering. But if the sample is not very dense, it will not absorb. But it will tend to simply scatter the electron beam. So we collect more backscatter in this case. So, the backscattered electrons are obtained from a depth of about 1 to 2 μm in sample.

Whereas your X-rays are typically from a much greater depth about 5 μm into the sample. So again, please remember, we are still scratching the surface of the sample or specimen. You have broken down your meter-sized objects into already centimetre or millimetre sized objects to keep it under the microscope. But when you are actually imaging, you are only trying to get information from the first few microns on the surface. So that is why you need to be careful with the way that you interpret the images, like what are these images actually representing.

The specimen interaction volume is nothing but the volume inside the specimen in which interactions will occur with the incoming or incident electron beam. Obviously it depends on atomic number. The higher the atomic number that means the phase that we are observing is more or more dense or the material we are observing is more and more dense, as a result of which there will be more electron absorption and a smaller interaction volume.

On the other hand, if the accelerating voltage is higher, then there will be more penetration and greater interaction volume. So that is why I said earlier, that when you are having ceramic systems or concrete-based systems, you need to have a higher accelerating voltage to ensure that you are able to penetrate deep enough to get a representative understanding of the sample. Whereas in biological systems we choose lower accelerating voltages, because you can easily get penetration into those samples.

Angle of incidence - If you have a greater angle of incidence, for example, if this is your sample, and the incident angle is like this (say 45°), obviously we are not going to be able to get a lot more penetration into this system. If you have a perfectly straight electron beam, your penetration into the sample will be much greater. So, if the angle of incidence is greater, you reduce the amount of interaction volume that your sample has.

Now interaction volume is critical because you need to image the material and understand clearly what phases are present inside the system. But at the same time, depending

upon what you would like to do, you can work with lesser, or more interaction volume. So for example, I told you earlier about the fact that if you want to collect the morphological information, surface information you do not need to penetrate deep down. So you can actually work with smaller accelerating voltages, so that you are able to collect more secondaries emanating from the top 100 Å or 0.01 µm on the surface.

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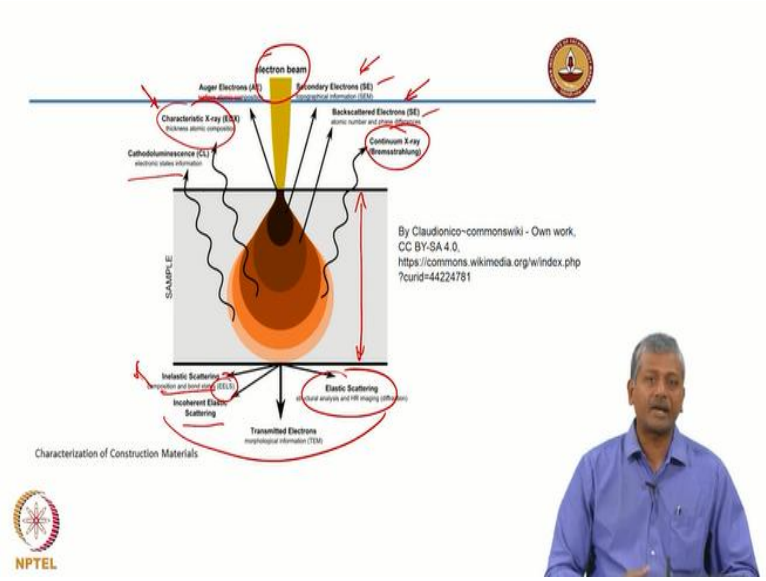

So again just to put this in a different perspective, you have the central electron beam, which is interacting with the sample and then you have different depths of interaction for different types of species that are coming out. For example, you have Auger electrons, Secondary electrons, and Backscattered electrons. You may have two types of X-rays: Characterisitc and Continuous X-rays. When you did your X-ray diffraction chapter, you would have learned about Continuous X-rays and Characteristic X-rays. Characteristic X-rays are the ones which are representative of the elemental species or crystalline species that are present in the sample. So in this case, obviously we are detecting the elemental composition by using X-ray analysis. Now you can also get some level of cathodoluminescence, which basically is some sort of a light fluorescence that happens in the system. But again, we are not particularly looking at that aspect as far as our system is concerned.

Now of course, in all the cases, there will obviously be some level of transmission of the electrons also, since these are very high speed electrons, and depending on the density of the material as well, if it is not dense enough to completely absorb all the electrons, some of it will actually get transmitted. You may have some elastic scattering which does not come straight up. Reflection of the electron right back on top is also one type of scattering, in

which the angle of scattering is almost 0 or 180°, depending on which way we talk about it. Exactly opposite from the incident beam you get the backscatter, but you may also get other angles of scatter, because if it hits any nucleus or any atom, the scatter could be in multiple directions. So that is basically elastic and inelastic scattering can actually happen. So in fact, this inelastic scattering that happens from atoms of the direct electron beam leads to another type of spectroscopy called EELS. You can also get incoherent scattering which basically means that you are losing your electrons in the dense atomic structure of the material.

So a lot of interactions are possible. You need to ensure that you understand what type of interactions give you what sort of information. So for our discussions we will look primarily at Secondary Electrons, Backscattered Electrons, and your Characteristic X-ray emanating from the sample after the electron beam has bombarded the sample.

So we will stop with this for this lecture and we will continue and try and understand in the next lecture as to how the detectors are able to capture these escaping electrons and what sort of interactions create these secondary and backscattered electrons and X rays. Thank you.