Non - Metallic Materials
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Module - 09
Characterization of structure, composition, and microstructure of non - metallic materials
Lecture - 48
Optical and scanning electron microscopy

Welcome to my course Non-Metallic Materials. And today we are in module number-9, Characterization of structure, composition and microstructure of non – metallic materials. And we are in lecture number-48, Optical and scanning electron microscopy will be introduced.

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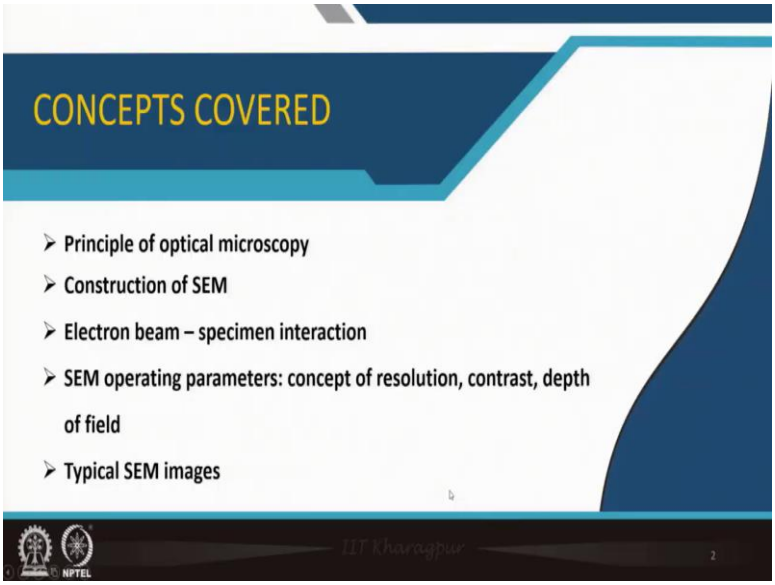

So, first we will very briefly touch the principle of optical microscopy. Most of you are familiar with it.

And more focus will be given on the construction aspect of scanning electron microscopy, scanning electron microscope. Specifically, we will introduce the electron beam specimen interaction, and what is the operating parameters for the SEM to get a good micro structure, what is the concept of resolution, contrast, depth of field. And finally, some typical SEM image will be illustrated.

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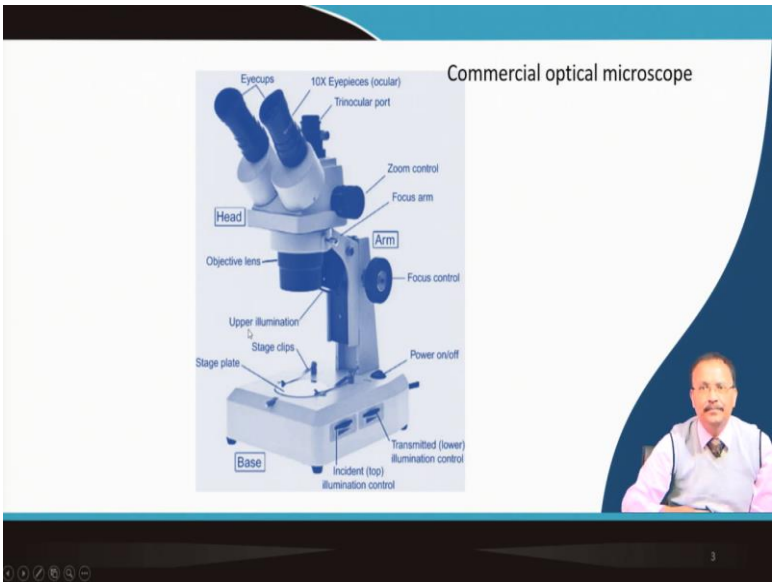

So, this is the photograph of a typical commercial microscope. And you know that various parts are this is the eyepiece where from the sample is basically seen, the magnified image of the sample. And illumination is also there to see the sample surface. And the focus control is basically done from here. So, it controls the length and focus the image.

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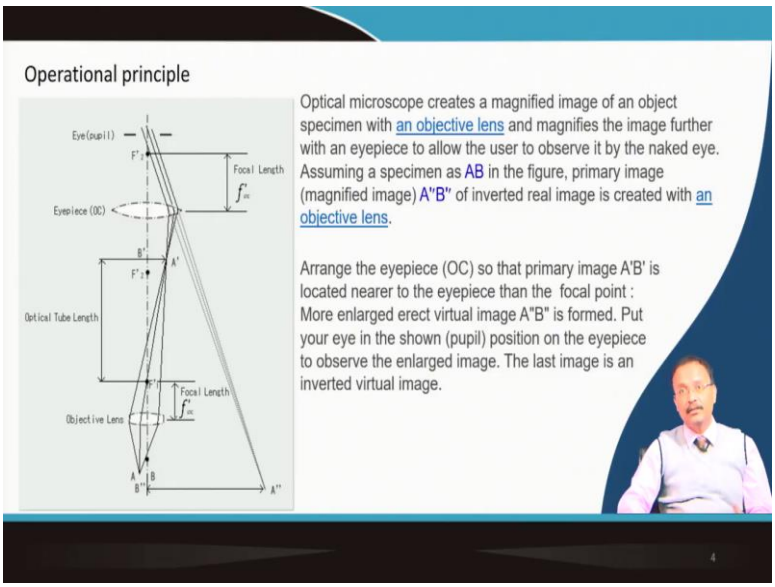

And this can be illustrated by this ray diagram. So, basically the image of a sample that is magnified and you are seeing the magnified image through this eyepiece. So, the lenses

are used for this magnification purpose, which are optical in nature. So, objective lens is important here. And basically, you can control the focal length, and inverted magnified image is shown, so that is a very-well known principle of the and optical microscope.

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But in principle the scanning electron microscope is very different, here you are not using the optical radiation. So, the lambda is very, very small as compared to the optical radiation. So, you can see the magnified image of your sample using this scanning electron microscope.

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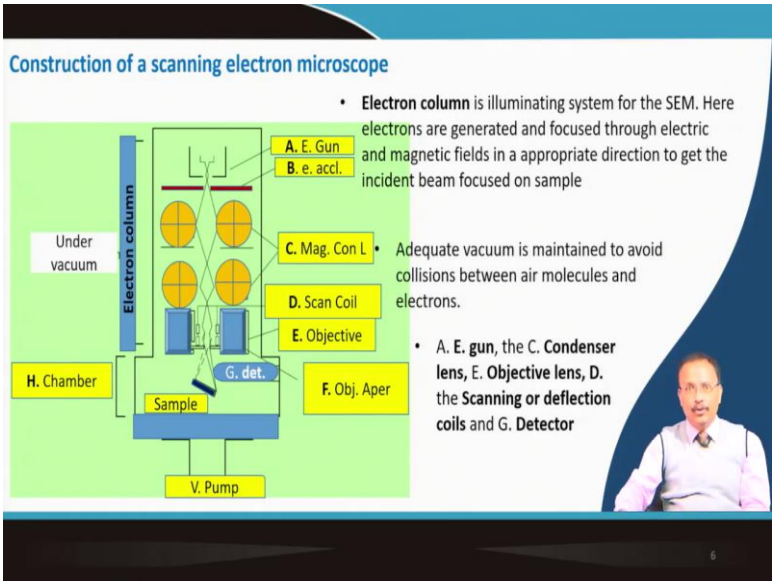

So, basically the scanning electron microscopy this is operated under vacuum. So, vacuum is used; otherwise the electron which is generated from this electron gun which is shown by this, this thing. So, this gun this electron will get scattered. So, you will have to focus this electron back to the sample. So, not really the lens made by glass is used. We use electromagnetic type of lens here in order to focus this electron beam progressively to the sample surface.

So, electron column this whole column is a vacuum, vacuum, so that the this electrons does not get scattered.

And this is basically focused by this electric and magnetic field of appropriate direction to get the incident being focused on the sample. So, there is a scan coil here which basically control the scanning of the focused electron beam on top of the substrate. So, sample surface it is basically scanned, and then this electron the secondary electron that is used for the imaging purpose.

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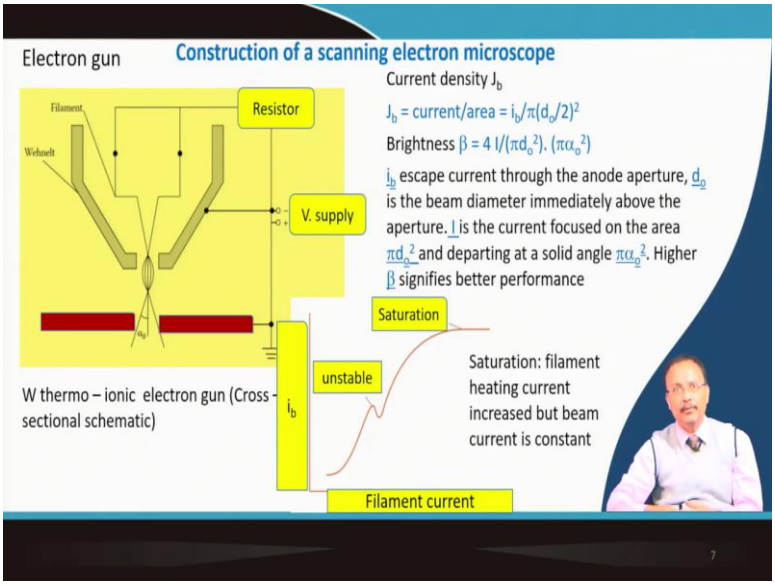

So, if you see the individual component now, first is this electron gun where electrons are generated by thermo ionic emission. So, tungsten filament is used, and filament is heated and electron is generated.

So, the current density which is the current generated by the relevant area that is given by this relation where i b is the beam current, and d 0 is the diameter of the beam that is

getting generated, so that one is important, the current density is important. And next one is the brightness of the beam which is defined by this relation where you can see that this brightness is related to the current I which is focused on the area of this pi d 0. So, this area, where it is focused so that I is used for the brightness.

And here you can see that the beam basically just above this aperture, it try to get delocalized and defocus, and it leaves this region with a solid angle. And this area is pi alpha 0 square. So, this solid angle area is pi alpha 0 square. So, it depends on this area this pi d 0 square and pi alpha 0 square, both these areas they are important in order to determine the brightness of the beam.

So, if you plot the beam current with the filament current, because you are applying current to generate this electron, so that plot looks something like this. So, you have a saturation limit where you can even if you increase the filament current, the beam current will not increase more. So, you operate in the saturation limit where the beam current is quite stable in nature.

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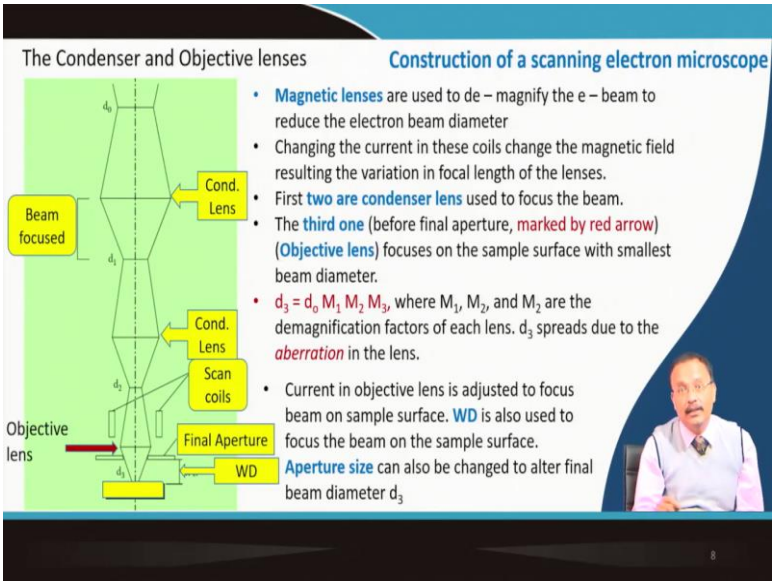

So, again we will look back this whole column. And you can see that magnetic lenses are used in order to demagnify the electron beam to reduce the electron beam diameter. So, wherever this tries this after getting out from this electron source, whenever it tries to get diffused, so the idea of using this condenser lens is to again put it back to the focus, focus condition.

So, multiple this lenses are used. So, the first two lens as you can see here and here – they are the condenser lens. And the third one that is used it is the objective lens. And that objective lens is used to focus this beam onto the sample surface, the substrate surface.

So, if you have the demagnification parameter M 1, M 2, and in third case it is M 3, then basically you can see the diameter of the beam that is falling onto the substrate. And there are aberration inside the electron beam because of slight variation in its lambda. And this, due to this aberration, this is not really a very sharp focus a small focus, but there is a spread in this electron beam finally at the surface.

So, you can use you see that whenever this condenser lenses are used, these three lenses are used; first two are condenser, and this one is the objective. Apart from that, there are two scanning coil because as I have said that it is not a single point which is important, but this electron needs to scan the surface in a raster area. So, this scanning coils are used for that purpose.

So, that is the final aperture which you can control the size of the aperture you can control which will take this scan beam on top of the surface. And this focusing is also important. The beam diameter you can change by controlling this working distance between this aperture and this substrate. So, you can keep top and up and bottom, and that is also used to alter the final beam diameter which is denoted as d 3.

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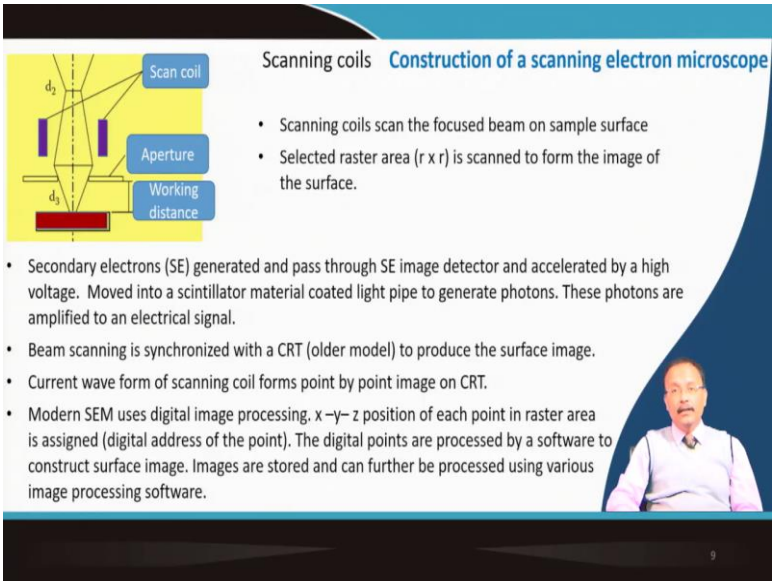

So, this idea of the scanning coil here is shown in the violet color that scanning coil scans the focus beam on the sample surface, and you can select a raster area r by r square on the sample surface, and which is basically translated to the image. So, secondary electron is there used to generate the image.

So, initially this secondary electron is passed through a secondary electron image detector, and they are thereby I mean in in there it is accelerated at high voltage, and moves to a scintillator material which is coated on a light pipe. And basically, after this scintillation of the electron when it hits the surface of it that produces photon. And this photons are basically amplified to get a electrical signal. So, secondary electron that is basically converted to electrical signal.

Now this beam scanning is synchronized with a cathode ray tube. So, in an older variation of the SEM, they used to use a cathode ray tube where this beam scanning is synchronized with this CRT to produce a surface image.

And this imaging technique is slightly complicated, I am not going into the details, but the idea is to replicate the surface of the particular sample as an image. So, it is a current wave from the scanning coil, it forms a point by point image to a CRT. And you can basically control this raster size to get the area of your interest that is magnified in the cathode ray tube.

But the modern SEM, they do not use the cathode ray tube, but in instead they use this a digital image processing technique where the x – y – z of each point in three dimension that is defined in the raster area. And we define it as a digital address of that point. And this digital points are processed by a software internally to control to construct the surface image. And this image can be stored, and can be also further processed by a suitable image processing software.

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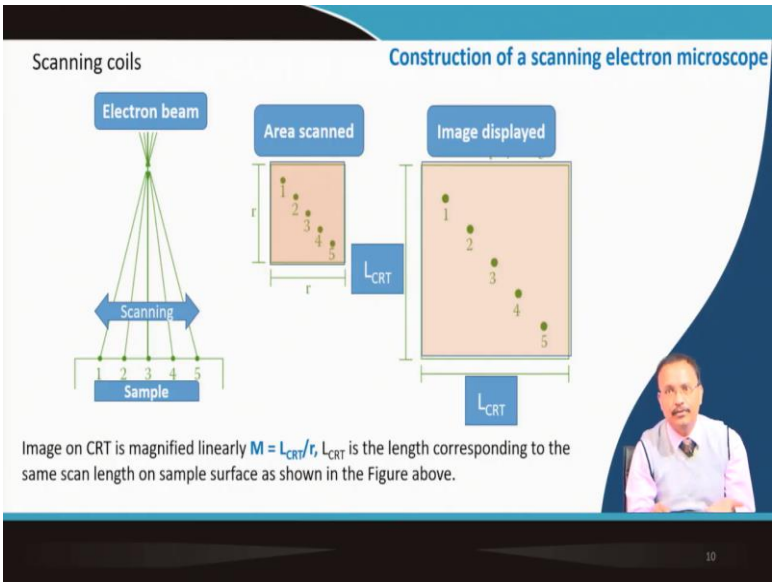

So, this is the principle whatever I discussed that is defined here. So, here as you can see that this is a slight spread takes place here of the beam diameter. You remember that d 0 and alpha 0 the solid angle it forms. And basically that forms this electron beam which is scanned at different points here.

So, on the sample surface, the beam scans. So, the area scan is the raster area 1, 2, 3, 4, 5, this points are scanned, and then it is magnified and shown into the CRT display. So, it is magnified here. So, the magnification is given by the length of the CRT this region divided by the raster area. So, it is much more magnified in this in this kind of image formation as well. So, that is the principle of the scanning coil and to generate the image of the surface that you are examining.

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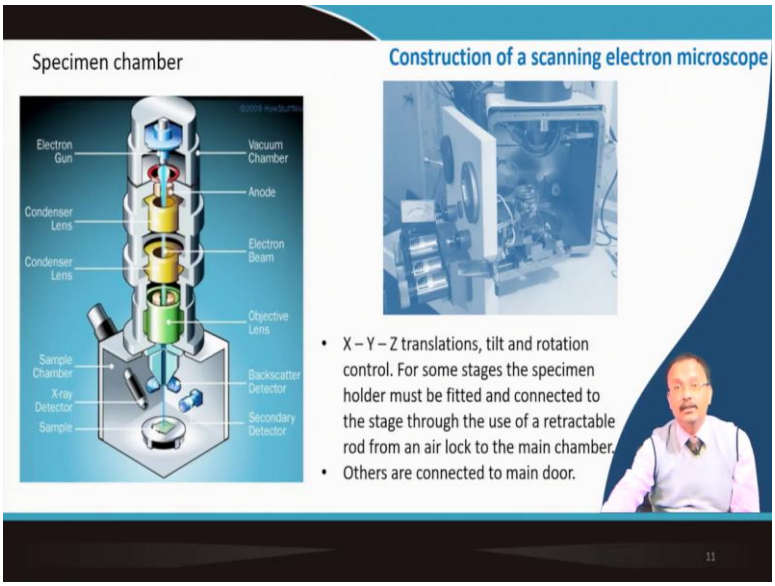

So, the construction is something similar to this where you can see all the parts the cross section of an SEM. So, this is the electron beam here. Your sample is here. Then you have your objective lens, and you have the other lenses to focus the electron beam the condenser lenses. And just after the electron beam generation, you have a anode also here to attract this electron, so that is this purpose.

The interesting part is that in this chamber, where your sample is fixed you have various types of other detectors for example, we have a back scatter detector, we have a secondary ion detector that is for the imaging purpose, the back scatter is forming this annular detector across the incident beam. We have a X-ray detector also inside the sample chamber, so not only the image, but lot of other things that you can do. And it is the actual photograph of from taken from an SEM.

And you can see that the sample change of the sample this is in modern SEM that is there in the door itself the sample changing is done the lock system in in the door. Earlier in the older version, there used to be a retractable rod from an air lock to maintain the chamber-chamber vacuum and that has been replaced now. So, now, each time you will have to put the sample back, and again the vacuum needs to be put on.

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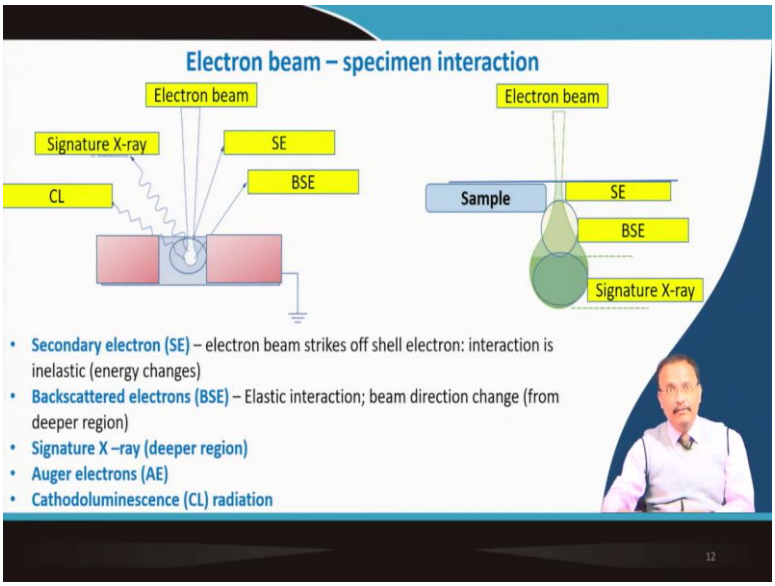

So, when the electron beam strikes the specimen, various types of things happen within the specimen. As you can see that initially the electron beam strikes off one of the shell electron, and this interaction is elastic the energy changes, and those electron we call secondary electron.

Other than that there could be elastic interaction this beam that is back scattered basically from the neutron, and proton in the nucleus. So, it is backscattered. So, energy is not changed. So, it is an elastic interaction. And once it strikes it, it can generate characteristics X-ray from the material. And auger electron that also can be generated; and apart from that, cathode illuminations radiation is also operative.

Now, I will just go into the details in a while, but you can see here that the secondary electron is basically generated from a very small surface near the surface of the sample. So, this is your sample.

So, very near to the surface, you have secondary electron generation. Backscattered electron is coping coming from a relatively deep region of the sample. And the signature X-ray is coming from this region of the sample. So, it depends on the depth of the sample to generate this all types of other kind of radiation which is so important to characterize the material surface.

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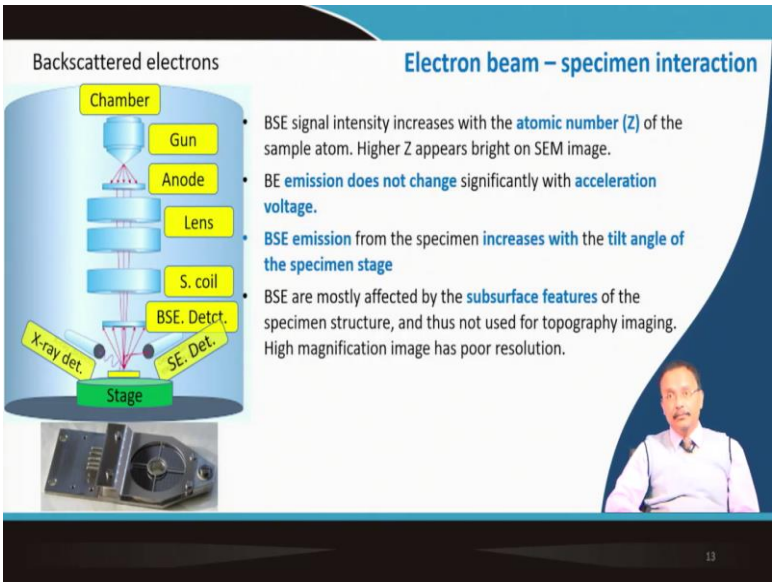

So, the back scattered electron that actually the intensity of that kind of radiation that increases with the atomic number of the sample atom. So, the atoms present in the sample the atomic number higher the atomic number, it will appear bright in the image.

So, this back scattered type of emission that not that does not change very significantly with the acceleration voltage, because you remember that you accelerate the electron from this place to focus in this region. So, this acceleration potential, acceleration voltage that does not change much the backscattered emission.

Backscattered emission from the specimen that increases with the tilt angle of the specimen stage. So, sometimes this specimen stage is tilted. And with the tilt angle, the emission is grossly enhanced.

And usually the backscattered is most affected by the subsurface feature because as I told that this BSE – backscattered electron is coming from certain depth of the sample. So, it does not get affected by the topography of the surface image. So, if you want to take a back scattered in image form, then usually this images are not of that high intensity as compared to the secondary electrons.

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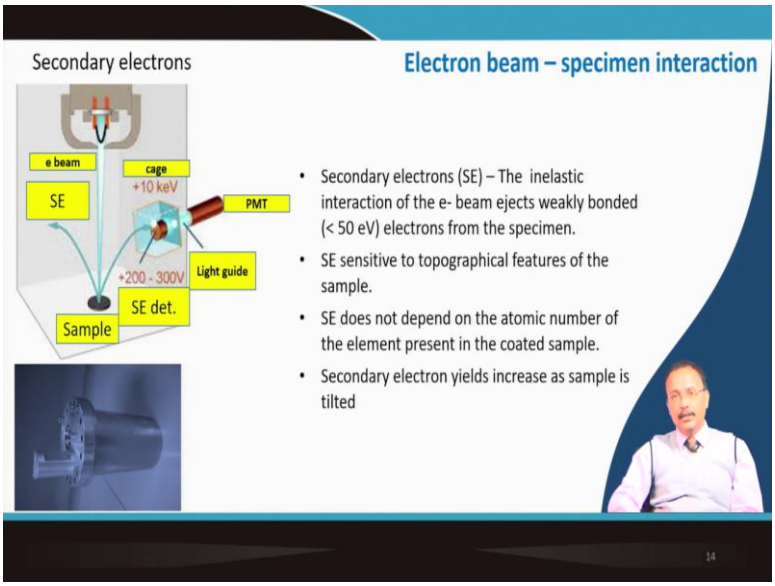

So, secondary electron as you can see that this is inelastic interaction as I have told. And this electron beam ejects weakly bonded weakly bonded electrons from the specimen, typical energy is less than 50 electron volt. And this secondary electron is they are very sensitive to the topographical features.

So, for getting the surface image this secondary electrons they are used. Since I have already told that it is first introduced into this cage by applying a positive potential. And then there is a light guide where the scintillator coating is there, and followed by a photo multiplier tube to enhance the signal. Finally, it is transport into a cathode ray tube or some other digital storage device.

So, this secondary electron does not depend on the atomic number of the element present in the coated sample. Sample surface also needs to be coated if it is insulating sample because as you can understand electron is showering on top of it. So, there will be charge accumulation, and image will not be seen clearly.

So, you need to coat with a thin coating of metal under of the conducting samples like ceramics in order to see a good image. And like your back scattered electron, secondary electron yields also is increased if the sample is slightly tilted. So, there are mechanisms to change the sample angle, there is a mechanism to change the substrate the working distance of the sample as I have already shown in my earlier slide.

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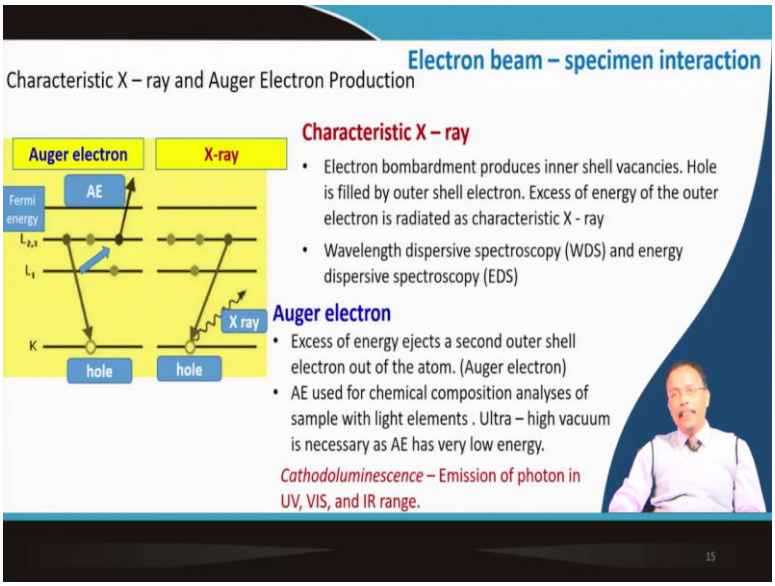

So, characteristic X-ray, they are also generated. And the formation of the X-ray is very straightforward because you are bombarding with electron of high energy. So, inertial vacancies are created. And some other electron from one of these shells, they will drop here from this region to the whole region and X-ray is generated. So, this X-ray is used to characterize the type of the element present inside the material. So, therefore, we have termed it as characteristic X-ray.

And this spectroscopy is known as either wavelength dispersive spectroscopy where the wavelength of the generated X-ray is of prime concern, or it could be energy dispersive spectroscopy that is termed as EDS. So, this X-ray is actually used for chemical analysis. There is another type of electron that is generated which is not through creation of X-ray, but this energy is spent to knock out other electron.

So, excess energy ejects a second outer cell electron out of this atom. And this, this electron is called auger electron. The pronunciation is auger. And this is also used for chemical composition analysis, particularly of lighter element. And this auger electron is having they are having very low energy.

Therefore, very, very high vacuum needs to be maintained for this detector. So, this is used for the chemical composition analysis. Apart from that, cathode illuminations is the emission of other types of photon like UV visible in higher range also the emission takes

place, they can also be used. So, depending on all these features, you have various types of detector present in the sample surface.

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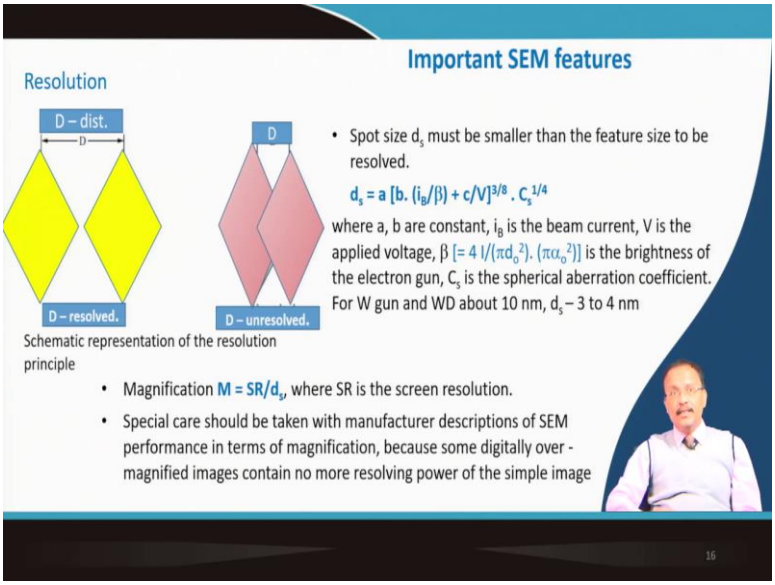

Now, the very important SEM feature is the resolution. So, resolution for example, in this figure, you can see that this distance you can clearly see. Distance between this two specific point, you can easily see. But here you cannot see; it is blurred. You cannot very easily resolve it. So, here the distance is unresolved.

So, the spot size which falls on the sample surface must be smaller than the feature size of the image. So, then only you can you can resolve it. So, the spot size is depending on basically the beam current and the acceleration voltage this b. And brightness as I have already illustrated that is given by this relation. So, that also is important for getting a good resolution.

And C s is defined as the spherical aberration, spherical aberration coefficient. So, the actual resolution for a SEM that also depends on the working distance. So, working distance, if you keep about 10 nanometer, then for a usual beam current acceleration potential and spherical aberration you can put this value to estimate what is the minimum size that you can differentiate usually it is 3 to 4 nanometer. And this should not be confused with the magnification.

So, magnification is just the screen resolution of the secondary electron. So, screen resolution divided by this spot size, so that gives you the actual magnification. So, this, this it should be properly understood that it is not the magnification which is important. You can magnify the image many time, but that does not mean that its resolution is more. So, resolution is particularly dependent on the brightness and the beam current, and also the spherical aberration of the lens involved.

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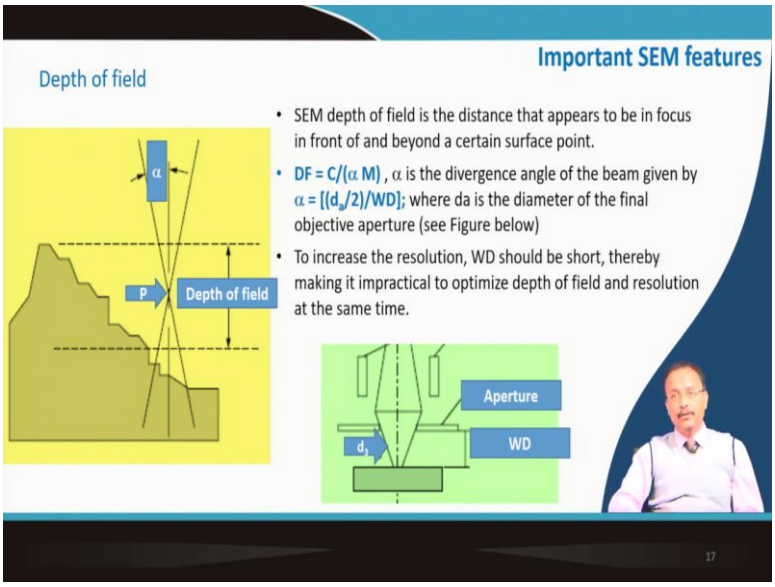

The other important thing is the depth of field. So, what is the distance in jet direction that remains under focus? So, that depth of field that basically appears to be in focus. So, it does not matter whether it is here or here. So, throughout this region these features are equally focused and that is also important for SEM. Although it is a surface feature that is important. But certainly, what type of depth that you can get the resolution along with the depth of field is also equally important.

The simple equation that defines the depth of field is dependent on the divergent angle that I have shown that this angle solid angle which the beam diverges and condenser lens basically make it focus, so that is important. As you can see depth of field will increase if you have reduced divergence.

And the divergence is also related to the aperture the final aperture of the objective beam. You, you remember that on top of the sample we have a adjustable aperture, so that is defined as da. So, it depends basically on that aperture and the working distance

which can be manually controlled. And here I have shown it. You see this is your sample. And this is the aperture the control level aperture and this is the scanning coil. So, this beam is coming and focused here. So, you can control this aperture and you can also control this WD.

So, basically alpha, this alpha is controlled by this to parameter. And eventually this alpha will also control the depth of field. So, to increase the resolution, working distance should be short. So, smaller working distance should be there, so that the beam diameter is as small as possible.

So, it is basically impractical to optimize the depth of field and the resolution both at the same time because of this obvious reason. In one case, it should be pretty short. And then if it is very, very short distance, it does not really make sense of a very high depth of field. So, these two are not really complementary in nature.

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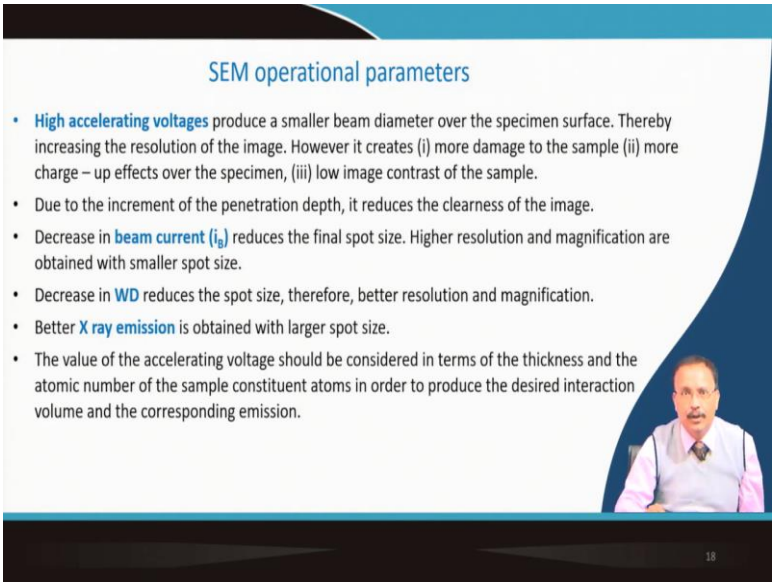

So, the operation principle of the SEM is of course the acceleration voltage, so that reduce a smaller beam diameter on the specimen surface. So, that introduce induce the good resolution of the image. But in fact, higher potential it damages the sample, and it creates more charge effect on the specimen.

So, the specimen is not clearly visible in the magnified condition. And contrast is also low for the for the image if you increase the high acceleration voltage to accelerate the electron from the electron beam generator. ah

Due to the increment of penetration depth, it basically reduces the clearness of the image. Now, you can control the beam current. So, if you reduce the beam current that reduces the final spot size. So, higher resolution and magnification are obtained if the spot size is smaller. So, the beam current you can keep low for a high resolution image. Working distance also reduces the spot size. So, for better resolution, you reduce the spot size as well.

Better X-ray emission is possible with a larger spot size because you are seeing a larger cross section of the sample. So, if you want to analyze your sample composition, then your spot size will have to be increased.

And for that working distance should be larger or acceleration potential should be small in order to get generate X-ray which is relevant for sample composition analysis. So, acceleration potential should be considered in terms of the thickness and atomic number of the sample constituent atoms to produce the desired interaction volume and corresponding emission. So, acceleration potential is an important parameter to think about.

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These are typical image taken from different sources. You can see with back scattered electron. You can very well characterize this phase how they are distributed the brighter one with higher atomic number phases as compared to the darker one. And this is a typical secondary electron image.

And it shows a grain growth, but at the same time a densified microstructure. This is a typical example of EDS spectra of a particular sample. You can do the atomic percent analysis of a typical sample. And this is a high resolution image. It is taken from some of our research work. And here a carbon coating on a battery a cathode material has been shown, and how clearly it is resolved in a SEM.

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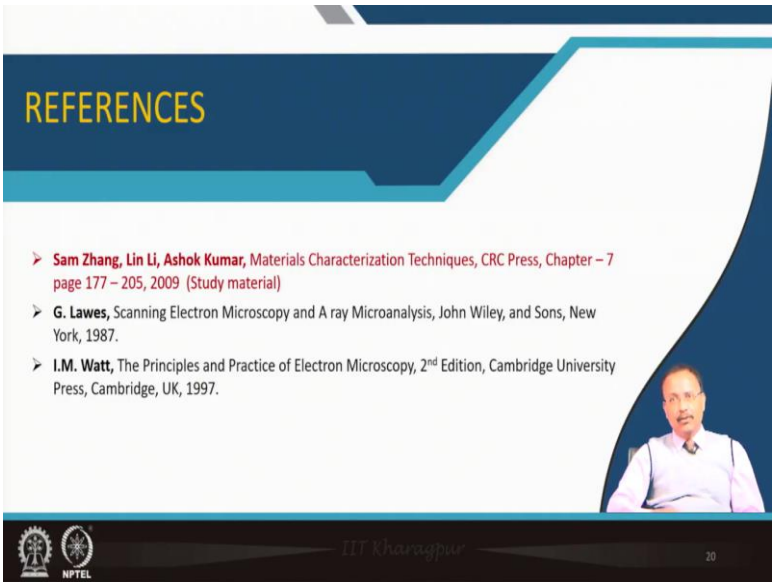

So, the study material is the book by Zhang Li and Ashok Kumar, Materials Characterization Techniques, Chapter number 7. And other than that the Scanning Electron Microscopy, other books they can also be used as a reference material.

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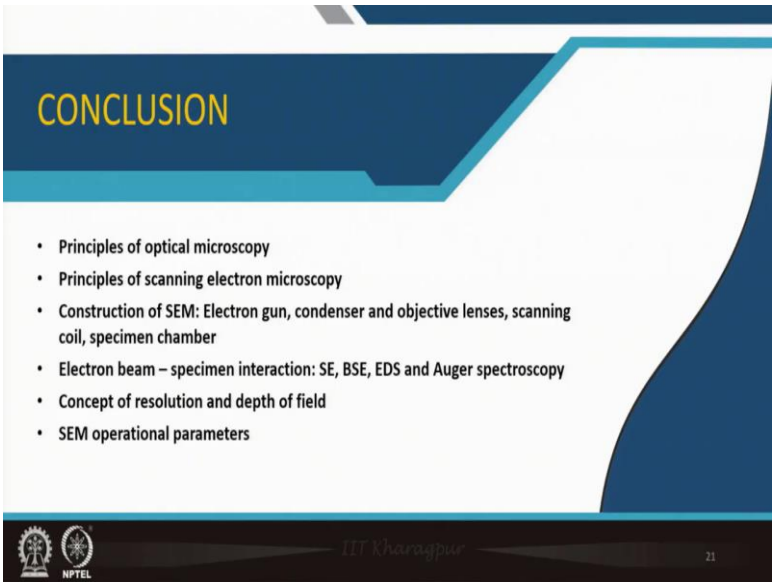

So, in this particular lecture, I have taught the principle of optical microscopy I just touch, but mostly concentrated on scanning electron microscopy, what is the construction of an SEM, electron gun, conducts condenser lens, and objective lenses they are magnetic in nature.

So, they are basically coil which possess electric current to produce the magnetic field to tune the electron beam direction. Scanning coil is important. Specimen chamber is having lot of other detectors, secondary ion detector, backscatter detector, electron dispersive spectrometry, auger spectroscopy, the concept of this analysis I have introduced. Then what is the concept of resolution and depth of field I have highlighted, and various same parameters operation parameters are introduced.

Thank you for your attention.