Characterization of Construction Materials
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Lecture - 40
Scanning Electron Microscopes- Working Principles – Part 1

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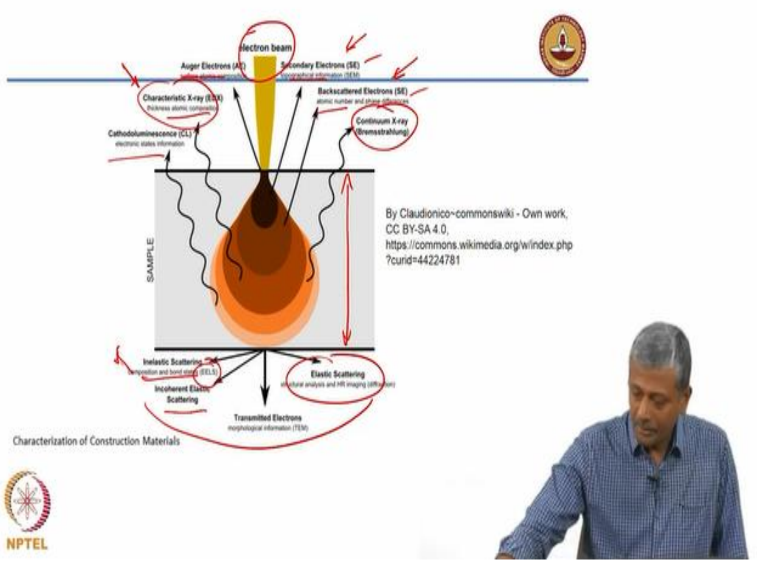

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Characterization of Construction Materials

Hello everybody, so in the last class we were talking about what sort of interactions will be generated when the higher energy, high velocity electron-beam bombards the specimen. We looked at different types of possibilities from different depths within the material. Now let us look at the primary aspects as we discussed earlier, we will primarily look at backscattered electrons, secondary electrons and characteristic X-rays that are coming out of the specimen, because that helps us especially with respect to cementations materials, the most widespread applications of scanning electron microscopy are in these three domains.

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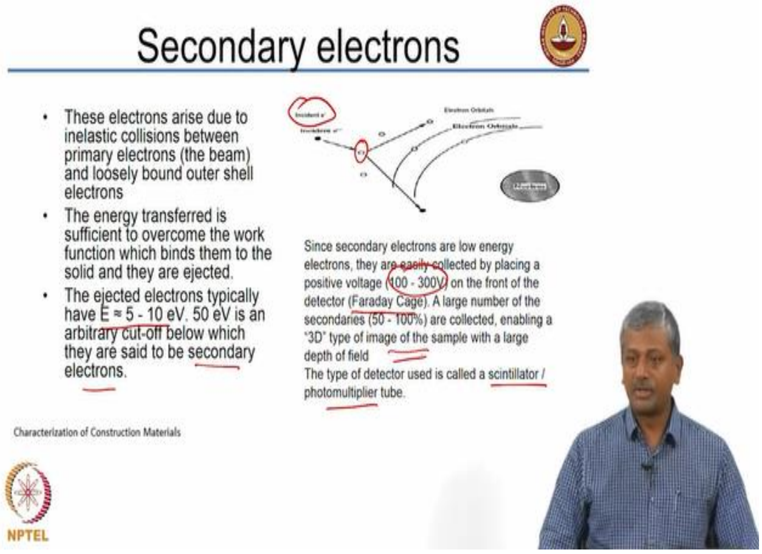

Secondary electrons

- These electrons arise due to inelastic collisions between primary electrons (the beam) and loosely bound outer shell electrons
- The energy transferred is sufficient to overcome the work function which binds them to the solid and they are ejected.
- The ejected electrons typically have E ≈ 5 - 10 eV. 50 eV is an arbitrary cut-off below which they are said to be secondary electrons.

Since secondary electrons are low energy electrons, they are easily collected by placing a positive voltage (100 - 300V) on the front of the detector (Faraday Cage). A large number of the secondaries (50 - 100%) are collected, enabling a "3D" type of image of the sample with a large depth of field

The type of detector used is called a scintillator / photomultiplier tube.

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So let us first talk about what these secondary electrons are. Now you know very well the structure of an atom, how the electrons are held in these shells that define the approximate electronic positions around the nucleus. So you have an incident electron which is from your electron beam, which may come in and knock off a loosely held outer shell electron. So this electron is coming in and knocking off an outer shell electron. Now these electrons, which are getting knocked off from the outer shells are going at very low energies, typically about 5 to 10 electron volts (eV). So up to about 50 eV, the low energy electrons that are dislodged from the electronic shells by the incident electron beam, these are called secondary electrons.

So these electrons are getting dislodged from the outer shell and now these need to be collected, since they are low energy, all you need to do is give a sort of arrangement where you have a positive potential, like 100 to 300 V potential, and these electrons are simply attracted towards that potential, because it is a positive plate and these electrons simply come directly to that plate or it is also called a Faraday Cage detector. A secondary electron detector is nothing but a Faraday Cage. It is a positively charged plate, which attracts the electrons which are generated from the specimen, because of these secondary interactions, that means, incident electrons knocking off loosely held outer shell electrons.

So, the question is in X-ray diffraction the incident electrons which are generated in the X-ray tube knock off the initial electrons of the target metal, because of which you generate characteristic X-rays. But here, this beam is only knocking off the external electrons, there will be internal electrons also, we'll come to that later, but external electrons getting ejected are loosely defining the morphology of the object. So if you collect the secondary electrons that emanate from the specimen, you can now make out the external structure of the material, because we are collecting these from a very shallow depth. I talked about this earlier, that we are actually collecting secondary electrons only from about 100 Å - that is about 10 nm, extremely small depth within the specimen. So we are essentially looking at the structural features of the surface when we collect the secondary electrons.

So these secondary electrons are collected by the Faraday Cage. And basically, there is a photomultiplier, which ensures that the signal that you get can be amplified significantly to generate the right kind of image. Now of course, we'll about how morphological information can be obtained based upon the characteristic shape of the crystalline materials that you are actually trying to observe.

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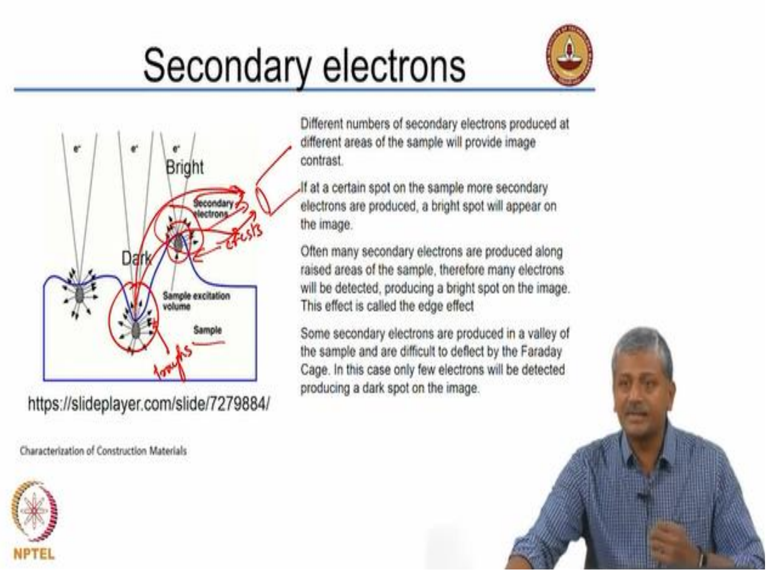

Secondary electrons

Different numbers of secondary electrons produced at different areas of the sample will provide image contrast.

If at a certain spot on the sample more secondary electrons are produced, a bright spot will appear on the image.

Often many secondary electrons are produced along raised areas of the sample, therefore many electrons will be detected, producing a bright spot on the image. This effect is called the edge effect

Some secondary electrons are produced in a valley of the sample and are difficult to deflect by the Faraday Cage. In this case only few electrons will be detected producing a dark spot on the image.

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So, if you look at a typical sample for secondary electron microscopy, we talked earlier that sample preparation is not really anything for second electron microscopy you just need to fracture your sample and stick it under the microscope. But, of course, for ceramic specimens and for cementitious specimens you need to coat the samples.

So because of the fracture, you have these relief features on the surface, you do not have a flat surface, you are not cutting the specimen, you are simply fracturing it and putting it. So there are these relief features like you have crests and troughs on the surface. Now what will happen is the incoming electron beam, if it is striking the location of the crest, what is going to happen is because the secondary electrons that are generated here are generated from the very top of the sample, there is a good chance of collecting everything with the detector.

Since the electrons are getting ejected from the top or crest of the sample, it is very easy to collect a lot of these electrons. So, in other words, how will this crest appear in the microscopic image? It will appear bright.

On the other hand, when you have a trough, a lot of these secondary electrons that are getting out, may not get a chance to completely go towards the detector, because they need to go across all the valleys and hills that are available on the surface to really make it to the secondary electron detector. So many of these electrons may not be able to get detected by the secondary electron detector, because of which the troughs will appear darker in colour.

So here you are able to now resolve minor changes in the heights, or levels of different aspects on the surface of the specimen. Now of course this is not just minor, once we look at the kind of depth of field that you can get, you can actually look at even millimetre-sized defects, as far as scanning electron microscopy is concerned. You will be able to resolve as much as low as a few nanometres, and as high as a few millimetres, that means not only do you have a good resolution. We also have a good depth of field. At the same time, keeping more than 1 millimetre of the specimen in focus is something of a wonder, as far as microscopy is concerned. You can never do that in optical microscopy. In optical microscopy, you have to work with flat images. When you are looking at reflectivity, and you cannot simply get any way that you can extend the depth of field, beyond a few microns, you will not be able to do that.

So, because of this possibility of collecting these electrons even from deeper and higher regions, within the surface of the specimen, you can then distinguish features that are separated by a significant amount. So that is the reason why the scanning electron microscope or secondary mode imaging can have a very high depth of field.

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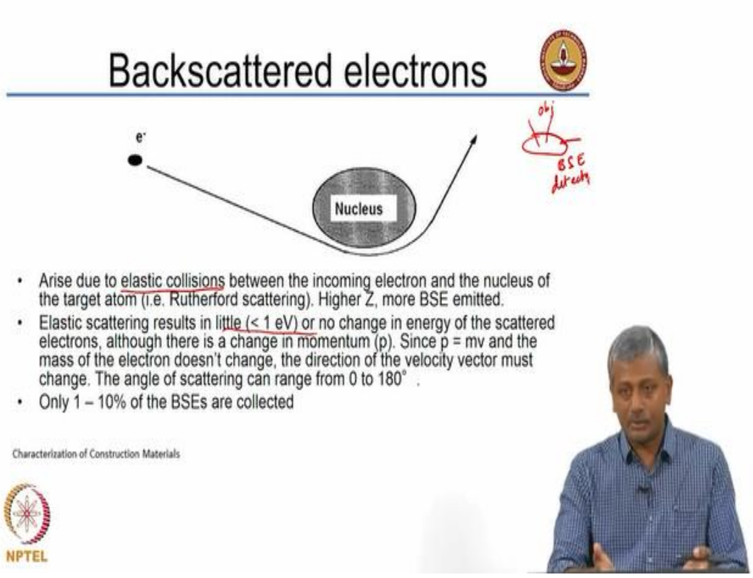

Backscattered electrons

- Arise due to elastic collisions between the incoming electron and the nucleus of the target atom (i.e. Rutherford scattering). Higher Z, more BSE emitted.
- Elastic scattering results in little (< 1 eV) or no change in energy of the scattered electrons, although there is a change in momentum (p). Since $p = mv$ and the mass of the electron doesn't change, the direction of the velocity vector must change. The angle of scattering can range from 0 to 180°.
- Only 1 – 10% of the BSEs are collected

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What about backscattered electrons? Now we are talking about scattering of the incoming electrons by the atomic regions of the sample, that means the atoms of the sample are scattering the incoming radiation. Now scattering can happen in all directions, but you know that the heaviest part of the atom is the nucleus and when this incident electron comes and gets scattered by the nucleus, there is a good chance that you get a completely elastic scattering, because electron mass when compared to nucleus mass is negligible. So when it hits the nucleus, it is going to get scattered, quite significantly back in the same direction. So basically these are elastic collisions that arise between the incoming electron and the nucleus of the target atom.

That is why the backscatter detector is pleased right around the objective lens, so that is the objective lens here, and that is the backscattered electron detector (refer to drawn representation in slide). I told you earlier that it is an annular ring, just around the objective lens. So that all the electrons that go and strike the specimen can come up straight up almost with 180° turn in their path and get detected directly by the detector which is placed right around the objective lens. So, because it is elastic scattering there is very little change in the energy of the scattered electrons. But there are of course other inelastic scatterings that are also happening. I told you from, from this picture in the earlier slides, there are a lot of other things taking place, you can see inelastic scattering, you can have transmission of electrons through the specimen and so on, depending upon the energy of the electrons and the density of the phases that you are trying to observe. But we are interested in backscattered electron primarily from the point of view the elastic collisions that are happening.

So generally, because the scattering can happen in several directions, only about 1 to 10% of the electrons which are getting scattered can be collected by the backscattered electron detector, because only some of them will have a completely 180° reversal in their path many of them may stray off to the sides. We do not collect those electrons we primarily collect the ones which are coming directly back. And the denser the phase is, the greater will be the reflectivity. The nucleus is dense and so you can imagine the elastic collisions will be much more from that type of a nucleus. Also please remember there is another issue, the denser the phase, the greater will be the absorption also and the denser the phase, the lesser will be the penetration depth of your electron beam, so to ensure that we get good penetration what should you do for the electron beam is to increase the accelerating voltage. We need to increase the accelerating voltage to get good penetration. If you are looking at very dense phases, you need to work with high accelerating voltages. Nevertheless, these electrons that are going into the sample or then getting scattered by the nuclei, some of the scatter happens directly back, that is the elastic scattering which results in very low change in energy and these electrons can be detected by the annular rings placed around the objective lens. So that is the backscatter electron detector.

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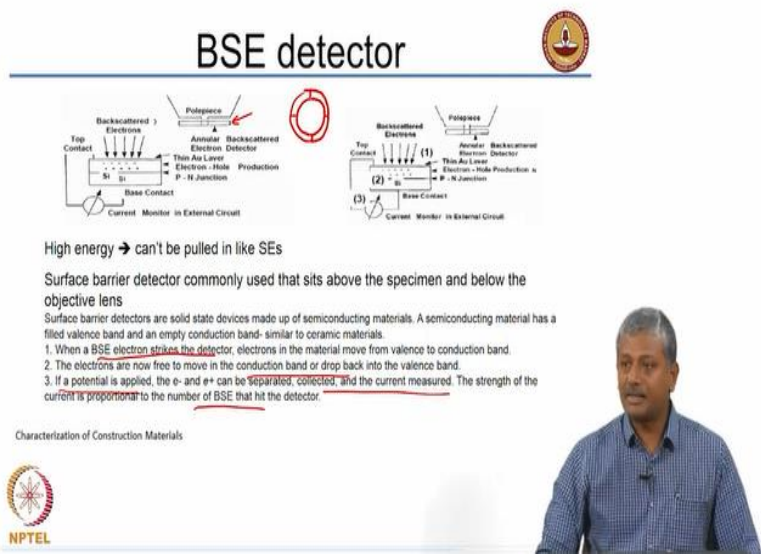

BSE detector

High energy ➔ can't be pulled in like SEs

Surface barrier detector commonly used that sits above the specimen and below the objective lens

Surface barrier detectors are solid state devices made up of semiconducting materials. A semiconducting material has a filled valence band and an empty conduction band- similar to ceramic materials.

1. When a BSE electron strikes the detector, electrons in the material move from valence to conduction band.
2. The electrons are now free to move in the conduction band or drop back into the valence band.
3. If a potential is applied, the e- and e+ can be separated, collected, and the current measured. The strength of the current is proportional to the number of BSE that hit the detector.

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Now of course, the type of detector, and the composition of the materials that make up a detector is, of course, probably beyond the scope of this course but then again. What we want to understand is, it is basically made up of the same kind of materials that we call as semiconductors, solid-state devices.

So if you remember some part of your physics, about semiconductors, there is a valence band. And when you supply energy to the semiconductor, there is an electron which is basically transmitted from the valence band to the conduction band. So when a backscattered electron strikes the detector, electrons of the material move from the valence band of the semiconductor. What type of materials are semiconductors? What is the most common element that is used? Silicon, germanium are elements that are used to make semiconductor devices. So here you have what is known as a valence band and a conduction band. So when you have a backscattered electron, which is striking the detector. It is moving the electrons in the material from the valence band to the conduction band. Now the electrons in the conduction band can either continue to move in the same band or drop back into the valence band. Of course, for stable configuration they need to drop back into the valence band.

But what happens is if a potentially is applied, you can now separate the electrons and collect them, and measure the actual current in the detector, because now these electrons in the conduction band, you can actually then start separating them, and measure the current in the detector and the strength of the current is proportional to the number of backscattered electrons that hit the detector. So, more number of backscattered electrons hitting the detector will cause more number of events of electrons in the valence band moving to conduction band, so there will be higher current and that current can then measure the intensity of the backscattered electron collection.

So, what you need to remember first is electron beam striking the sample, more backscatter will happen for denser specimens, because there will be greater mass of the nuclei, so there will be elastic scattering and these electrons are collected by this detector. Within the detector the electrons go and strike the solid-state semiconductor devices like silicon or germanium, typically silicon devices, and transfer the electrons in the valence band of silicon to the conduction band and when the electrons are moving in the conduction band you can measure the current in the conduction band. And that current is proportional to the number of times this detector has been hit by the backscattered electrons.

So that is the simple schematic principle of how a backscatter detector works. Of course, you have to be a material scientist to fully appreciate how solid-state devices actually work. So it is beyond the scope of our understanding here, but again, as I said, your backscatter detectors are typically placed around the objective lens. And generally, if I draw the annular ring, it is usually divided into 4 quadrants, and each one of these is your solid state device. So again there is some averaging that takes place to determine, what exactly is the intensity of the backscattered electrons that you are collecting in the solid-state device.

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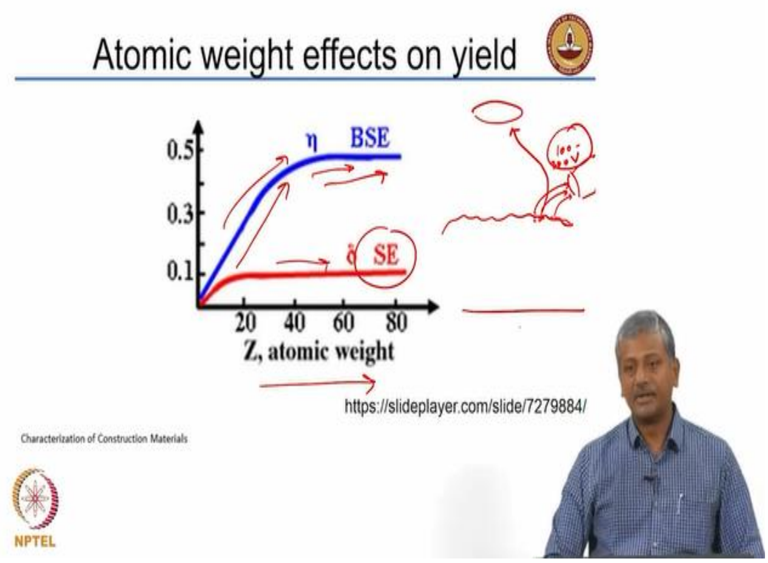

Atomic weight effects on yield

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So, again this is just showing you the atomic weight effects on the number of backscattered electrons that comes out. So, with the atomic weight, the amount of secondary electrons that come, sort of, saturate out. But the amount of backscattered electrons can have a significant increase with the increase in atomic weight of the sample. Now of course beyond a certain point, this also starts getting levelled.

Why do you think this happens? Why does the intensity of the backscattered electrons that you can collect sort of levels off after a certain atomic weight is reached? Why is that happening? More scatter and more absorption, there is lot more absorption. So beyond a certain atomic weight the effects of the absorption will be dominant because of which the backscatter intensity starts tapering off. Otherwise, it should keep on increasing with the atomic weight of the phase that you are having in the specimen. So, with more and more atomic weight, a greater proportion of the incident energy is getting absorbed by the sample, because of which you do not get a major increase in the yield of backscattered electrons beyond a certain point.

The yield is related to the weight by a polynomial function and the atomic weight in the polynomial function, you have a third order polynomial you have a Z^3, sort of behaviour.

So this is not really a simple function to track, so that polynomial function gives approximately, the dependence of the backscattered electron yield on the weight.

If this is your sample, the backscattered electron detector is right on top, secondary electron detectors are here. So what is happening is the secondary electron yield is only from the surface and because the electrons are of low energy, they are getting captured by the positive potential, that is 100 to 300 V that you apply in the Faraday cage. So, it is very rare that some secondary electrons will deflect all the way and go to the backscatter detector. It may happen, because a lot of electrons are getting generated at the surface. Some electrons may have sufficient energy to actually go right up. But the charge here with a Faraday cage, will be able to pull many of these low energy electrons towards the cage, and you may not have any specific instances of these electrons going to the backscatter detector.

So for example, if you have a very flat specimen and you are trying to look at it with a secondary electron detector. You will not really see much of an effect, because in a flat specimen, you have almost an equal number of electrons from the surface that will go towards secondary electron detectors there will be no difference in the surface morphology. You will not be able to appreciate that if the specimen is flat. Also if you are trying to observe the fractured specimen with the backscatter, then again you have a problem, because you do not really get a proper scattering, all your electrons are going towards the side because you do not have a level surface. So, for each of them you need to have sample preparation done in a different way.

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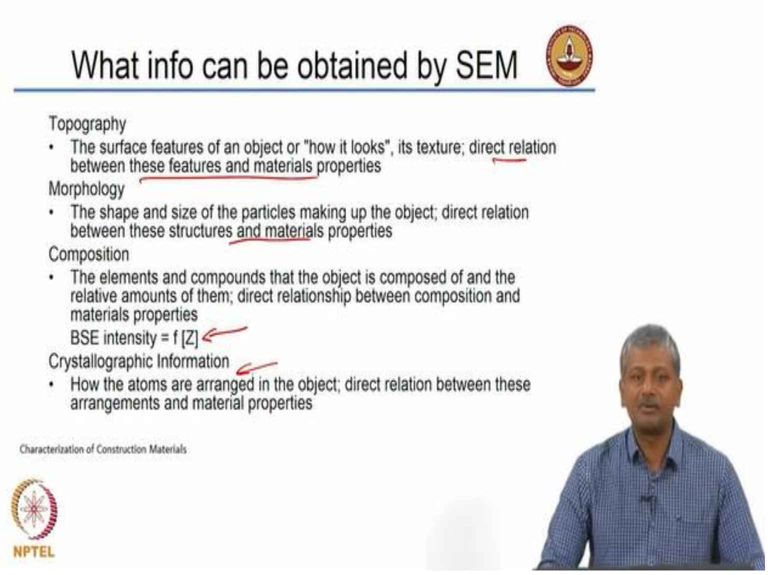

What info can be obtained by SEM

Topography
- The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties

Morphology
- The shape and size of the particles making up the object; direct relation between these structures and materials properties

Composition
- The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties

  BSE intensity = f [Z]

Crystallographic Information
- How the atoms are arranged in the object; direct relation between these arrangements and material properties

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So, what information can be obtained from SEM? One is obviously topography. That means the surface features of the object and how it looks generally, the texture and all these obviously have a direct relationship with the material properties. For example, in metals for instance if you actually have a tension test performed on steel, and you look at the fractured surface, the shape of the fracture surface is very much defined by the ductile behaviour of the steel.

Morphology is obviously the shape and size of particles making up the object, so if you are looking at specific crystalline features in your material. For example, in cement hydration we know that we form calcium hydroxide and ettringite and these are very well defined crystalline species, and you will see later that we can actually make out the size and shape of these crystalline objects that are forming as a result of cement hydration.

The composition is because your backscattered intensity is a function of the atomic weight. So when you are looking at a flat polished sample, the phases that appear bright, obviously have a higher atomic number or atomic density, as opposed to the phases that appear dark. If you are looking at a void or a pore, you will get almost zero reflectivity or the surface will look black if it is pore.

You can also extract crystallographic information in terms of how the atoms are arranged in the object. However, to get to this level, you may have to actually go to much finer resolutions. You will not be able to get this from a simple secondary electron image, you will have to go at very high magnifications to really make out crystallographic properties of different types of crystalline species that are forming in your object. That is something which is little difficult with typical SEM, but then you can actually get there if you have materials that are capable of getting resolved in that scale.

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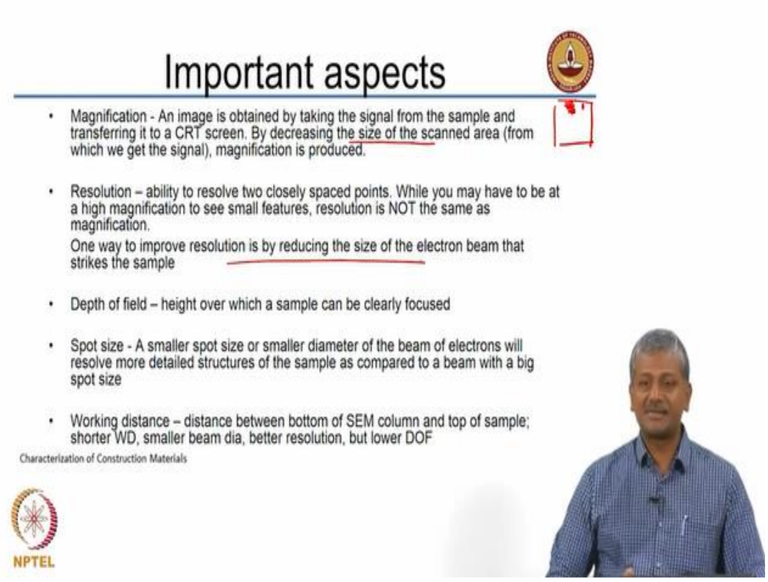

Important aspects

- Magnification - An image is obtained by taking the signal from the sample and transferring it to a CRT screen. By decreasing the size of the scanned area (from which we get the signal), magnification is produced.
- Resolution – ability to resolve two closely spaced points. While you may have to be at a high magnification to see small features, resolution is NOT the same as magnification.
  One way to improve resolution is by reducing the size of the electron beam that strikes the sample
- Depth of field – height over which a sample can be clearly focused
- Spot size - A smaller spot size or smaller diameter of the beam of electrons will resolve more detailed structures of the sample as compared to a beam with a big spot size
- Working distance – distance between bottom of SEM column and top of sample; shorter WD, smaller beam dia, better resolution, but lower DOF

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So, again, similar to optical microscopy, we also have the same sort of terminology as far as SEM is concerned. Of course magnification, you are just making a small location appear larger, but what is magnification in terms of SEM? What you are simply doing is you are decreasing the scanning area. So please remember in SEM if this is your sample, your scanning coil is scanning a certain area here. If in the same area you take a smaller area and scan it, that is basically magnification, because the ultimate size of the screen is the same on which you are seeing the image. So you scan a small area and transmit that to a large screen as opposed to scanning a large area and transmitting that to the same screen. So, magnification simply means that your scanned area is getting smaller.

Resolution, it is the ability to resolve two closely spaced points. Resolution is not the same as magnification. I keep repeating that because very often, when people want to look at features more closely, they tend to increase magnification. But we learnt earlier, that when you increase magnification the confusion also increases, and because of the confusion increasing the resolution becomes lower. So, magnification and resolution are not the same, although in electron microscopy, you can get good resolution, and high magnification.

Now, one important factor, which governs the resolution, is obviously the size of the electron beam. Let us just go back to the secondary electron image that I showed you earlier. Now here, if this electron beam was wide, that is, if the diameter of electron beam was even bigger than the relief features that I see on the object, I will not be able to make out these crests and troughs easily. To ensure that I pick up each and every individual point on the surface properly, I need now to have an electron beam that has a small enough diameter. So that small enough diameter will ensure that I am able to resolve, very closely spaced points.

The depth of field obviously is height over which a sample can be clearly focused, same thing as you see in photography or in optical microscopy.

Spot size is the size of electron beam - the diameter of the beam of electrons. So again, smaller the diameter is, the greater will be the resolution. So how do you control the diameter of the electron beam? By the aperture which is provided in the objective lens. In the objective lens system, you have the aperture, you can control the size of the aperture that allows the electron beam width to be defined.

So working distance is the distance between the bottom of the SEM column and top of the sample. As I said, when you have short working distances, you can get better resolution, but lower depth of field.

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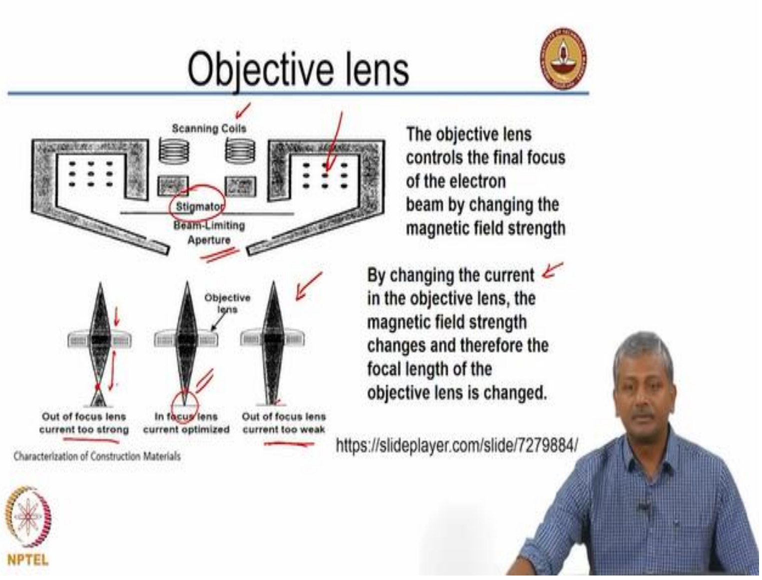

Objective lens

The objective lens controls the final focus of the electron beam by changing the magnetic field strength

By changing the current in the objective lens, the magnetic field strength changes and therefore the focal length of the objective lens is changed.

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Again, coming back to this slide here, if you have a shorter working distance, you have a stronger current that is hitting the sample, and if you have longer working distance your current becomes weak. So as a result of stronger current, you ultimately get a better resolution. But please remember always you have this compromise between resolution and depth of field. If you want greater resolution you will be having less depth of field.