Techniques of Material Characterization

Prof. Shibayan Roy
Department of Materials Science Center
Indian Institute of Technology – Kharagpur

Lecture – 39
Imaging in SEM Continued

Welcome everyone to this NPTEL online course on techniques of materials characterization. We are continuing with scanning electron microscopy. We are in now week 8 module 8 and this is lecture 4. So, still now we have covered in this week about detectors for analytical chemical composition detection, elemental concentration detection. So, two types of detectors we covered under working principle advantage, disadvantage etcetera.

One is energy dispersive x-ray spectroscope and another one is wavelength dispersive x-ray spectroscope and then in the last lecture we just started about the image formation in SEM and the first thing we discussed is how the contrast generates pixel by pixel when a scanning goes on how the contrast generates from each point and what we need to do in order to get a very good contrast that is close to the natural contrast which our human eye can detect.

And from there we have derived certain relationship in terms of the critical current which is needed, the frame scan rate or the time for each point what is the scan time or how long the beam should stay in each pixel, the time we discuss we derived. And from all of these we finally discussed about the relationship between resolution that is in terms of special resolution and contrast.

So, earlier we have seen the resolution for SEM at least is detected by or is determined by the minimum probe size that we can have and that is related to some of the pixel size as well and then again in order to get that minimum probe size what should be the minimum beam current and now we have also discussed in terms of contrast. So, how the contrast is dictating the minimum current that we need.

And in turn how that probe size is determined from that requirement of contrast. All of these things we discussed previous lecture and now we will be starting about imaging in SEM and some specific examples of doing it in scanning electron microscopy mode and backscattered mode.

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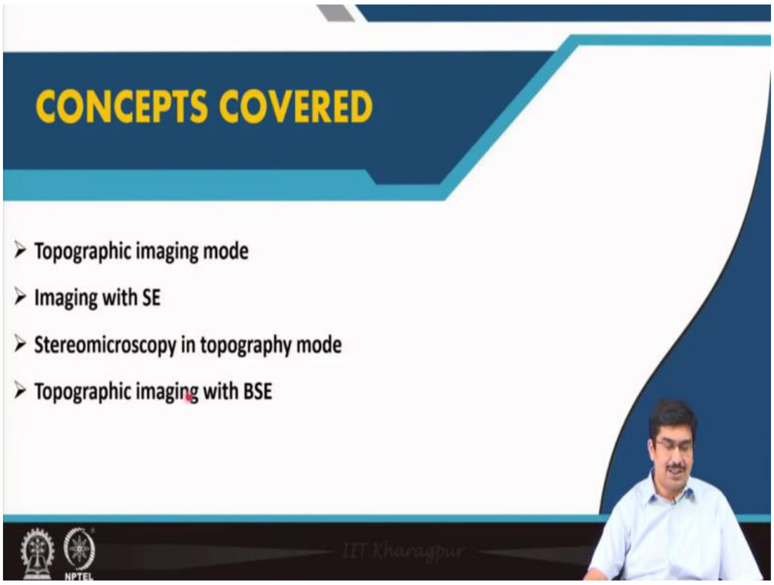

So, first we will discuss about the topographic imaging mode what exactly it means then we will see that how imaging with SE works. One particular application of SE microscopy or scanning electron microscopy is in the stereo to develop 3D images that we will discuss very briefly and if time permits we will discuss about the topographic imaging with BSE signal.

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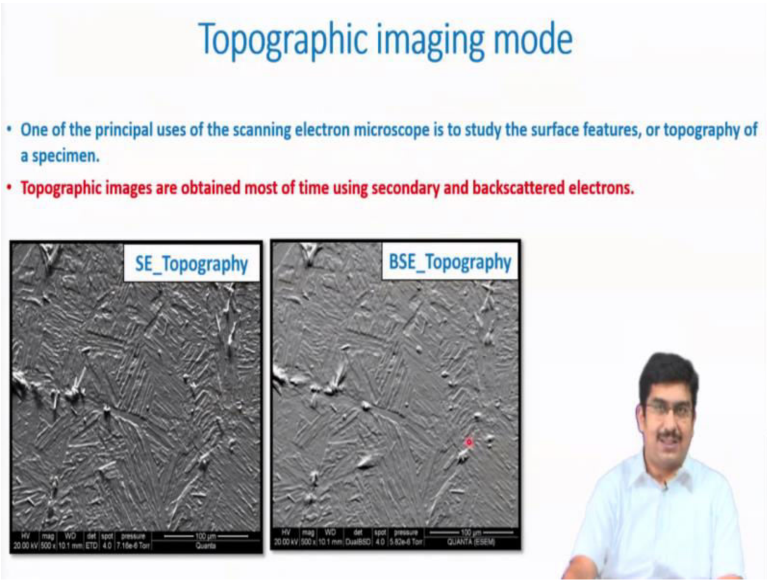

So, topographic imaging mode is something seeing it in this way and this is one of the principal use of scanning electron microscopy basically to check the surface features or topography. Topography basically means surface feature so just like what we say we can see that our Earth topography the Google map what it shows, the image of the Earth is basically a topographical image. So, it shows exactly where you have the forest, you have river or where exactly you have certain like human habitation and so on.

So, that is the surface topography pretty much that same thing we used to do in case of scanning electron microscopy. And that is one of the major and basically that is what makes scanning electron microscope so popular for at least we know it is used for opaque specimens and for opaque specimen definitely we cannot see the internal structure unlike a transmission electron microscope.

So, what we basically see is what lies on the surface how does the surface looks like. So, exactly this is done and this is called the topography mode of topographic imaging mode in a scanning electron microscope. So, of course, this topographic images can be obtained either by using SE secondary electron signal or by using backscattered electron signal both topography can be obtained by both.

So, often times my students they are confused or people who just started to do their research work they get confused, they think that BSE image means it is a compositional difference it is a atomic number contrast it is not like that. BSE also gives topography image that is compositional imaging in BSE mode is a special application. SE mode does not work in generally it does not work in compositional mode.

It is specialized imaging mode for BSE signal, but topography mode can be for both SE signal and BSE signal although topographic mode in secondary electron mode is more popular. So, you could see that this is same this is a titanium alloy basically. So, this is alpha plus beta titanium alloy titanium-6 aluminium-4 vanadium alloy. So, these are two different phases are there.

Definitely you can see that one is appearing in the grey contrast or darker contrast and one is appearing in the brighter contrast and this is exactly the same area is image in secondary mode and in backscattered electron mode and both of them are giving you certain level of contrast. This here the secondary mode the contrast is much more prominent whereas the BSE mode its contrast is little bit subdued.

We will come how to improve this and this like what is the different, why it is so and so on in that, but basically ultimately both of them are giving you the surface features and this surface features here is basically produced by creating certain undulations on the surface that means some roughness on the surface either it can be there as it is or often times what we usually know that like in order to get this kind of a contrast.

In order to see the surface topography what you have to do you have to do polishing sample surface you have to made flat first and then you have to remove, you have to do something called etching. You have to basically create selective undulations on the surface depending on different phases. For example here these two are different phases where darker contrast and brighter contrast these are two different phases.

And the difference is created here by this process called etching where the chemical etchant is creating, it is corroding one phase more than other one and that is how there is a step formation on the surface and from those steps we are actually getting two different type of signals same thing for BSE here.

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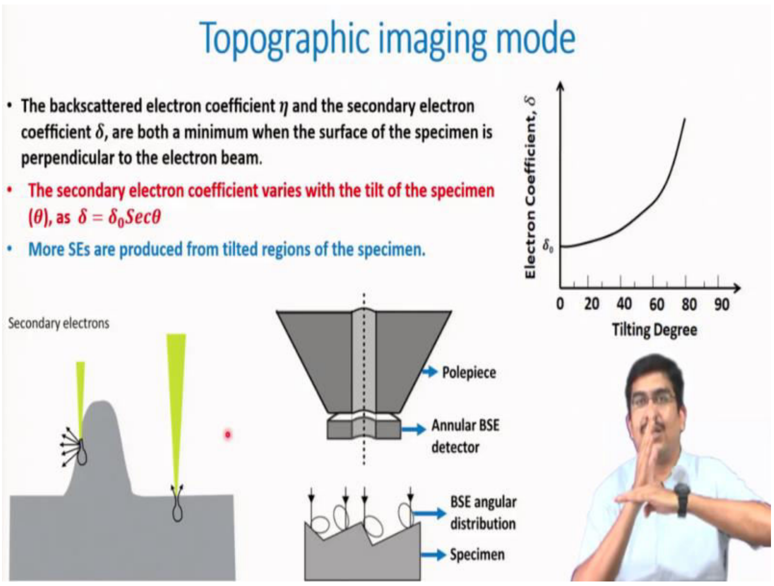

Now how this topographic imaging mode basically works? So, the backscattered electron coefficient eta and secondary electron coefficient delta both of them are minimum where the surface of the specimen is perpendicular to the electron beam. So, if you imagine this is a secondary electron mode and imagine that this is exactly representing this, but we are now seeing instead of a top view we are seeing a side view of this specimen just imagine that.

So, now as I said that certain phase let us say this is one phase and this is another phase. Certain phase is getting corroded more than the other by the chemical attack. Initially both of them were possibly of the same height because of the polishing now due to the etching one phase is getting less corroded so it is staying like a hump on the surface or a hill on the surface. The other one is possibly creating a valley.

So, both secondary electron and that same thing you can imagine that this is in backscattered this is the same specimen and here also you are getting the surface undulations because of same effect you are having it. Now what happens is when this electron beam; electron beam is always perpendicular to the specimen or surface the flat portion it is always perpendicular let us say.

So, when it is falling on this surface in a perpendicular manner that time the yield of secondary electron on backscattered electron is minimum why we will come to that? But this is minimum compared to that and that varies in this way. So, when there is a tilt for example if the beam is falling over this area then we can imagine that the sample surface is now tilted with respect to this electron beam or in the backscattered electron case the beam is falling over here and this is the sample tilt here.

This is the sample tilt here and this is the sample tilt here so it varies. Different parts of the specimen has different tilting. Now this effects the secondary electron yield particularly secondary electron yield is very, very sensitive backscattered is not that sensitive we will come to that, but secondary electron yield is very sensitive to this tilting angle and this is given by this relationship.

It is a kind of secant relationship so there is a sec theta component which is the tilt which is basically the angle between this beam and the specimen surface. So, this gets modified the delta gets modified so delta 0 is basically when the electron beam is exactly perpendicular to the specimen surface then it is the electron the yield of secondary electron is delta 0 and that gets modified for a tiled specimen by this relationship delta equals delta 0 sec theta.

This is basically you can find it by geometric simply geometric, but it suggest is that because the sec theta is always greater than 1 delta will always be greater than delta 0 and that is what it is shown here this is the electron coefficient and this is the tilting angle initially the effect will not be that prominent, but if the tilting angle is increased after a certain around 20 to 30 or around 40 degree.

So, after 40 degree tilting this difference or this yield just shoots up it increases very rapidly here that means for a tilted ultimately the message is the tilted for a tilted specimen with respect to the beam if the specimen if the surface is tilted and this is the beam then the yield of backscattered electron or secondary electron will be higher than something beam is exactly perpendicular to the specimen surface that is what is the lying philosophy and that is true for secondary electron that is true for backscattered electron for both.

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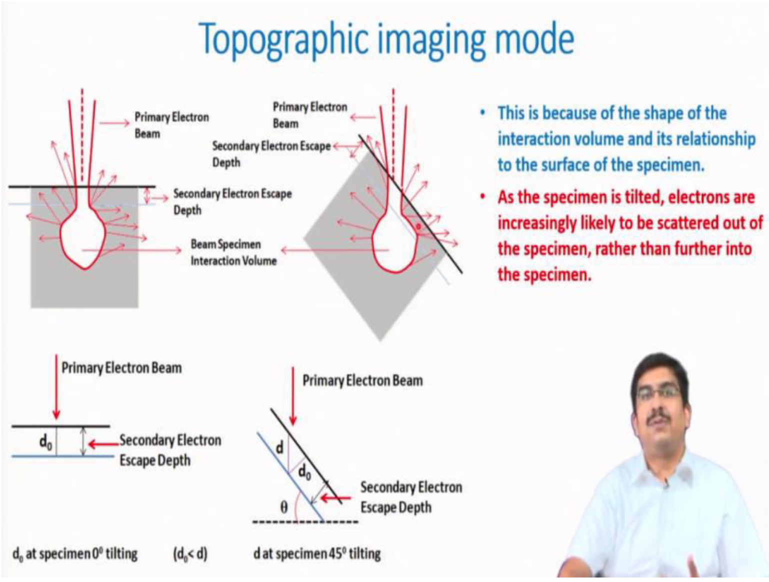

Now why is it so? This is related to the shaped of the interaction volume and its relationship with the surface of the specimen. So, if the beam is directly falling over this specimen surface or the beam is falling at an angle with the specimen surface the shape of the interaction volume does not change. It always remains of this shape and always is beneath this beam directly.

Whether it falls perpendicular to the specimen surface or it is falling the beam is falling at a tilted angle the specimen the interaction volume always remains of this shape, it is irrespective of the specimen tilt that is what is also shown here and if you imagine that this interaction volume it is always of this shape. So, interaction volume is not going to change its shape, it is not going to change its angular relationship with the primary electron beam is not going to change.

What it changes is the angle between the specimen and this interaction volume. So, let us imagine and this secondary electrons are generated let us imagine all the secondary electrons are generating within this interaction volume, we know that for secondary electrons at least most of the secondary electrons are generated from a very narrow depth of within the specimen and very close in and around this primary electron beam.

So, how this with specimen tilt how this secondary electron yield is basically changing. So, what is changing here when the beam is falling exactly perpendicular to this specimen surface then this is the secondary electron escape depth. So, if the secondary electrons are produced from this depth they have to travel minimum distance that is what I am saying. This is not exactly distance is not the same for all of the secondary electrons definitely.

The one which are produced at larger depth has to have a larger it has to escape a larger depth. What I am talking about is the minimum depth. The minimum depth or the minimum distance the secondary electrons has to travel before it could reach the surface and escape. So, that is called the secondary electron escape depth that is now this much. What happens if you tilt this again this secondary electron escape depth this is here in this case the tilting depth is 45 degree.

The secondary electron depth is basically reduced the secondary electrons which are produced from here because of this tilting now the surface is much closer to this interaction volume at certain level at certain points. Imagine in this areas the interaction volume or the regions from which the secondary electrons are generated becomes much closer to the surface that means the secondary electron escape depth in those regions are reduced when the sample is tilted.

So, as the tilting is increasing more and more secondary electrons will be able to escape because the escaping depth the distance from which where the secondary electrons are originated and where the surface is this distance is now becoming smaller for many of the secondary electron. So, they will easily able to escape and in that process what we will be getting a more secondary electrons for a tilted specimen.

And the same thing is true for backscattered electrons as well the escape depth will decrease as the specimen is tilted, but for them it is slightly different which we will discuss when we go for image topographic image in BSE. For now let us just concentrate on this secondary electron signal. So, one thing is clear as the tilting is increasing the yield of secondary electrons will also increase.

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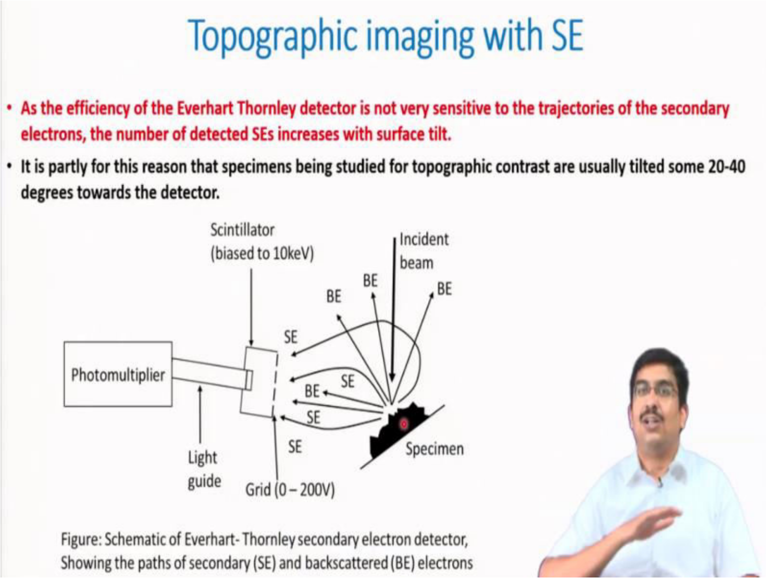

Now let us see how we can use this difference in secondary electron yield of secondary electron how we can use this for imaging. Basically this will be a source of contrast formation. Now easily you can imagine that these two regions let us already I said that these two we can imagine them to be two different phases. So, one phase is like a hill and another one is within the valley.

Definitely the yield of secondary electrons will be much higher from these regions compared to this regions which are flat. So, this area if I now collect secondary electron if the detector basically collects this here is let us imagine that I have a detector somewhere over here and that detector now or somewhere over here let us say the detector is somewhere over here that detector will collect more amount of secondary electrons from this region the tilted regions compared to this flat regions and that will be used as a contrast exactly the same thing is happening here.

The brighter phase basically does not get corroded and forms this hill whereas the darker phase is forming that valleys and that is why the brighter phase produce more secondary electron and that phase is that is actually the beta phase that is called beta phase. So, that phase is appearing brighter and the other phase which is in the valley that phase is forming the darker contrast.

Now, for this there are couple of considerations in the topographic imaging mode. Number one the detector itself the ET detector should not be sensitive to the depth in itself. If it is so if the detector is sensitive to the surface roughness of this specimen then what will happen these two affects may get cancelled or at least get altered. So, we will not get a true representation of surface topography remember this.

So, detector here the important point for the detector that the detector should not be sensitive to the specimen surface or the contrast generation another word the detector should not be sensitive to the trajectories of the secondary electrons, it just should count the number of electrons that is it. It should not count where from this electrons are coming. Whether that is why this metal cage comes very handy because this one when the primary beam is falling depending on the surface roughness.

Secondary electrons may generate from all different trajectory. Some of them will be directly targeted towards this detector some of them maybe going somewhere else, but this metal cage cause them or make them to reach finally to this detector and that is important that means the detector is basically not sensitive to the surface roughness of the specimen and only here the difference is produced by the specimen roughness itself.

The contrast only source of contrast is this surface topography. Detector is not sensitive to the surface topography, detector efficiency is not sensitive to surface roughness only the secondary electron generation, the number of electron generation is sensitive to this specimen roughness. This is an important point that is why ET detector should only be used and no other detector if the detector become sensitive to this then there will be a chance of mixing the contrast.

Detector may be sensing less number of electrons imagine for certain valley area. We know the valley area is producing lesser secondary electron now if the detector is more sensitive to valley area then these two effects will be neutralized and we may get equal number of electrons from all spots. So, the entire contrast will be gone so this should not happen this is an important point.

And second point is as I said the tilting is increasing for around 20 to 40 this range is here. So, tilting causes more and more secondary electron generation. So, if I just tilt the entire detector with respect to the specimen then what will happen I will possibly able to capture most number of electrons. Most number of secondary electron that means the secondary electron the signal strength will be highest if this detector is tiled with respect to the specimen.

That is why that is why the secondary electron detector is kept at an angle with respect to the specimen in order to increase the tilting and increase the number of secondary electrons captured in that.

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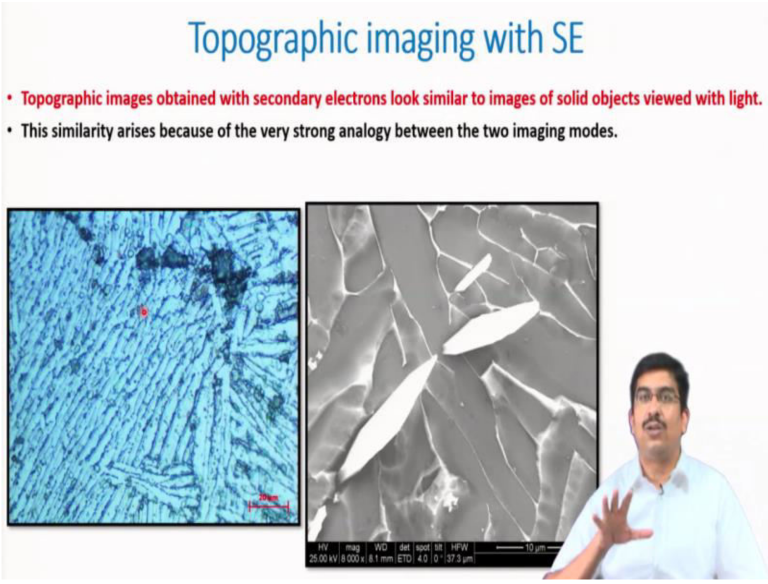

So, topographic imaging of SE also one more point very important is that it looks pretty similar to the image viewed with light. So, if I compare this is a light microscopy image from the same titanium alloy, alpha beta titanium allow this is the light edge so filter is used forget about that, but it is basically giving almost the same kind of contrast. Here also the two phases the very distinctly appearing.

And here also the two phases are distinctly appearing and both of these appearance is similar and produced by the surface roughness. So, light also is sensitive we have already studied the light is sensitive or the light signal which is generated in a reflection mode is basically sensitive to the surface roughness. Similar way the secondary electron topographic imaging mode here also it is sensitive to the surface roughness.

So, that is why finally the appearance of these two surfaces in either if I check it with optical microscope of if I check with topographic imaging mode they will appear pretty much similar.

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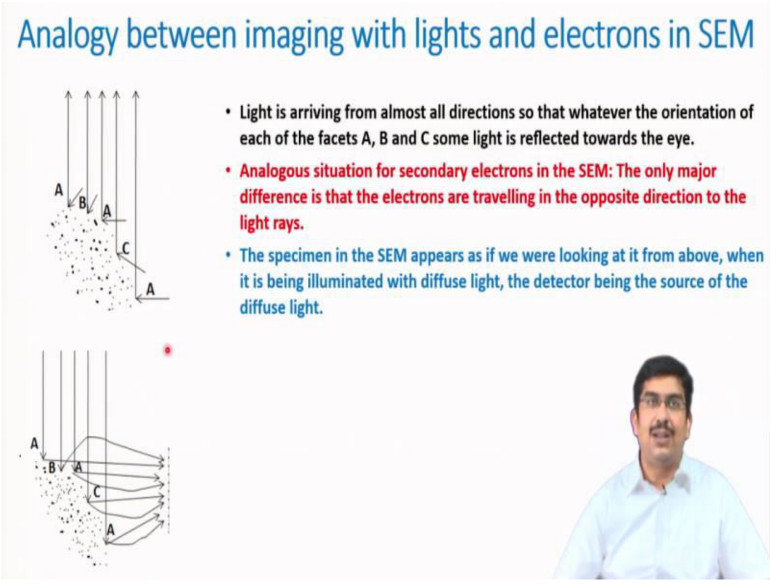

Now why is it so that is because we can imagine this let us say our eye is somewhere over here and this is the light source here and this is the sample with different, different facets on the sample. What happens is now the light is coming at all possible angle various different angles the light is coming, but after reflection some light or certain amount of light will always be there which will travel parallely and will reach to our eye.

So, whether it is this facet or this facet or this facet which are all having different, different angle facet A, B, C all have different, different angles with respect to each other. So, this is the surface roughness. So, for all of them the lights are coming from different, different angles and finally certain amount of light will always be like this they will travel parallel and they will reach to our eye.

So, ultimately we will be able to see this surface roughness the amount of light will always be different of course this amount of light let us say from A facet will be different from B facet and will be different from C facet, but certain light will always be reaching here. Now the same thing happens if we imagine that the electrons are coming parallely and after depending on this surface tilting with this electron beam there will be different number of SE electrons generated and that will be detected in the detector.

So, what we can imagine is that in case of SE our eye if we still have our eye here the detector basically works like a source just in order to compare these two. Here the detector works like an electron source and we are seeing our viewing we are doing the viewing here then we can imagine that these two situations are exactly the same and that is why both of them the surface roughness is responsible for contrast generation in both the light microscope and the secondary electron microscope.

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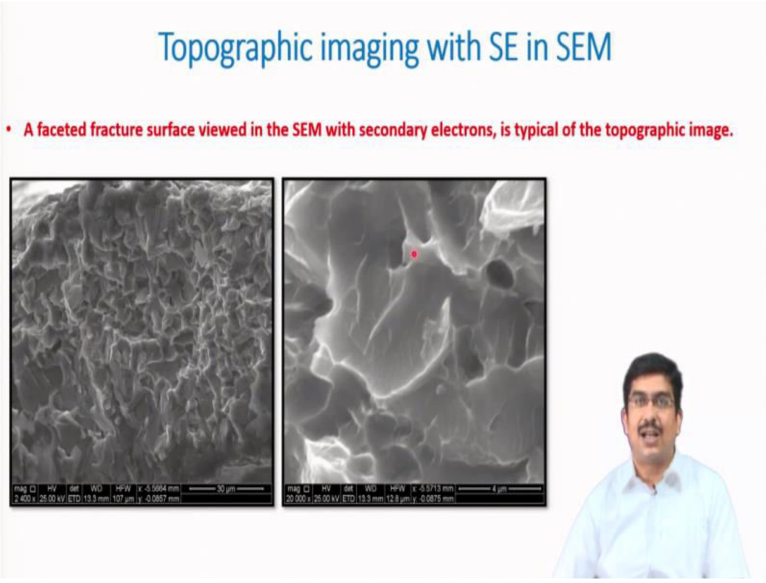

Topographic imaging again very interesting example already I said that the SE signals or SEM is very much used for studying the fracture surface and for fracture surface of course it is completely in topographic mode. So, you see it this is a single phase material and the topographic imaging here is giving the contrast because of the difference in surface roughness.

This surface is very, very rough in this fracture surfaces is very, very rough and these are very nicely viewed in the topographic mode here.

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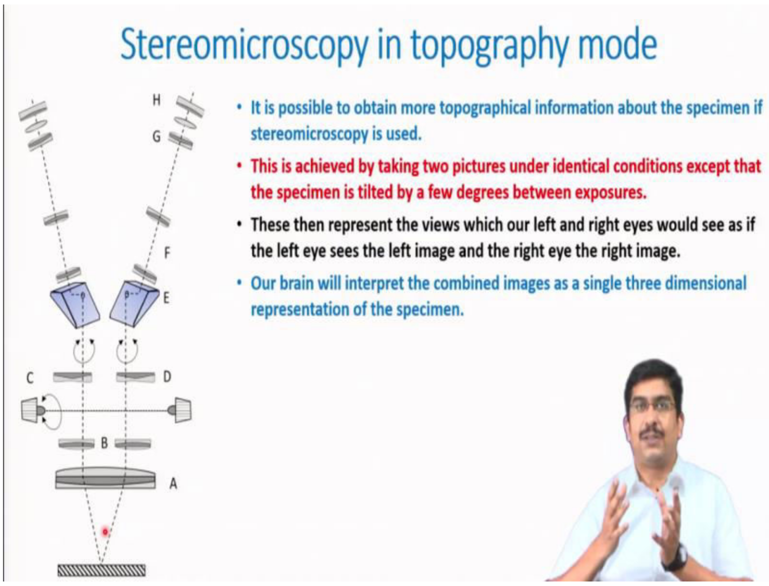

One more very important application of this topographic mode or secondary electron image usually it is done in secondary electron image is called the stereo microscopy. Now what is stereomicroscopy? Here very briefly we discussed about stereomicroscopy. So, what stereomicroscopy does is pretty much the same way we see images with our own eye the way we see images like for example our human eye we have two eyes.

And then we see two different images and somehow they are superimposed and that is how we could see that backside of any object that means we could see the depth perception. So, the same way we can make our SEM or make our microscopes work. Basically this is done with optical microscopes as well as stereomicroscope and same thing we can do with a scanning electron microscope.

What we can do here that we capture two different images under identical condition everything else remains the same like the beam current, like probe size, like scanning raster everything remains the same except that the specimen is tilted by a few degrees between the two exposures. So, what do we do? We have the specimen the beam is falling in the same position exactly the same position the beam is falling and with the same current with the same size and all the beam is falling and it falls two times basically.

First time it falls it captures an image and after that most likely the first one is captured in flat mode and then a second time an image is captured, but this time the surface or specimen is slightly tilted by only a few degrees. So, virtually what happens is these two images are representing or it is seeing the sample the same sample, same spot almost, but at a two different angles.

And this is exactly the same way we used to use with our human eye the same thing we used to see because of the angle in our eye we used to see any object at two different angles and we capture at the same time lights are coming and it is coming to our human eyes, but at a two different angles. And so we are finally getting two images and these two images are taken at two different angles.

Then our brain basically interpret the combined image as a single three dimensional representation of the specimen that means we are able to see the depth in the third dimension of the specimen depth sensitivity. So, try this if you do not believe try this close one of your eye and try to see any object most likely if you see it for too long it takes time for brain to forget and undo things.

And then again to see it you will see that you will lose the depth perception of any object. You will not be able to see the depth of the second object. So, the people who lost one of their eyes this is the problem with them they do not able to see that depth of the two images. This is happened because we have these two eyes and exactly the same way the stereomicroscopy works.

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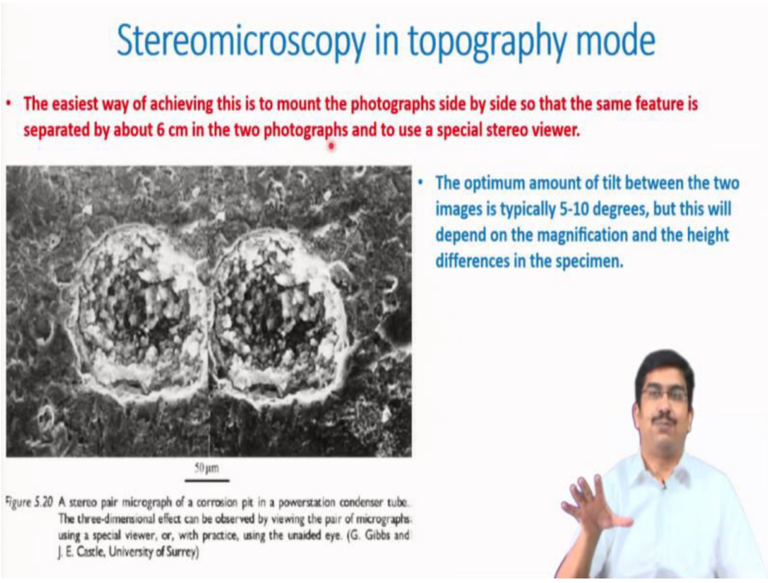

So, stereomicroscopy of course in topography mode now the easiest way of doing it is basically these two photography we take it side by side. And then the same features we separate them at a slight different angle this is exactly same what we do with our human eye it is a slightly different images same object, but we see them at a slightly different or specially they are slightly away from each other exactly the same thing we do it along with tilting we see it and the tilting causes that the same feature we see at two different angle.

And in between we have a very slight spatial difference between these two angles and that difference is around 6 centimeter for this two photograph and after that we basically see them next to each other in a stereo viewer which then recombines basically this is the stereo viewer is there and we recombine these two images that two images that we capture we see them somewhere here these two images are again recombined.

And finally we are seeing them as one and that time we are able to see the back side of it. So, there is a slight depth perception which is created. So, the amount of tilting that we do here is very less it is around 5 to 10 degree to match the kind of angle that we see with our human eye. Of course, this depends with angle of course depends on the magnification at which we see it and height difference in the feature.

So, basically these two features that we are seeing what is the roughness, what is the sample roughness size. So, depending on that the tilting is adjusted so that we can see this we can keep these features in focus when we capture. The maximum amount of these features are in focus so that we can able to see finally after recombination.

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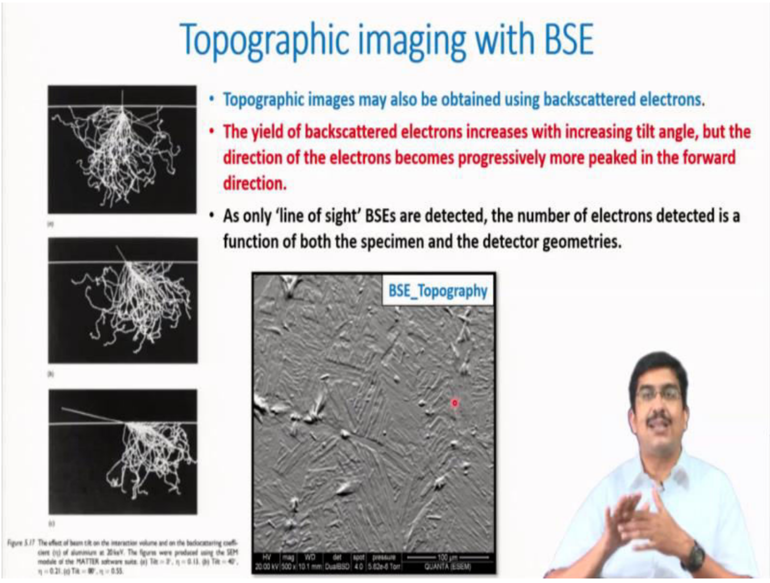

So, with this we will be moving to topographic imaging with BSE and topographic as we said topographic images can also be captured in BSE mode just like in SE mode. Here also the same thing when we tilt the specimen the shape of the interaction volume does not change. So, in this case that is the Monte Carlo simulation again shown here instead of specimen tilting what we can imagine that the beam itself is tilted basically by the same thing.

So, in this case it is 90 degree the beam is falling 90 degree with the specimen surface in this case the beam is falling at a slight angle that means the specimen tilted. So, this is tilted with respect to the beam and this tilting is increasing and what you can see that the shape or size of the interaction volume does not change, but the escape distance for the backscattered electrons are changing.

Now so the backscattered electrons are more easily able to come out if this tilting angle is increases, but for the backscattered electron there is one more thing to consider that is the backscattered electrons have a directionality. Most number of backscattered electrons are generated in and around the beam. So, their directions so they are very much forward peak so their direction is very much forward biased.

So, this backscattered electrons will always generated in and around so their direction will be around this primary electron beam that is why the line of sight of backscattered electrons is like how many backscattered electrons will be captured is not only depending on the tilt of the specimen. It will always depend on the detector geometry so that not only specimen is tilted, but that tilt is sensitive.

So, basically what does it mean is that the yield of backscattered electrons is not only depending on the specimen tilt alone. It will also depend on the specimen tilt with respect to the detector. So, this is unlike the SE generation so in case of ET detector the ET detector was itself not sensitive to the surface roughness, but the backscattered detectors are sensitive to the surface roughness because the BSE electrons that finally generates is basically very, very sensitive to which direction they are generated.

So, the backscattered electron finally are the detector that is used in backscattered detection of backscattered electron there what angle it makes with the specimen that is also important for the backscattered electron coefficient or finally the number of backscattered electron that the detector captures. So, this is some one extra additional effect again as I said this will cause some other kind of contrast on itself.

The angle between the specimen and the detector that will cause another type of contrast which can neutralize the natural contrast which is coming purely because of sample roughness, the yield of backscattered electron how the yield is changing with respect to the sample roughness or sample surface. On top of that the detector will also have its own relationship with the specimen tilting and these two can mix or these two can be matched.

And we can very well suitably we can adjust this also and we can play around with the topographic image that generated in a backscattered electron mode. This we cannot do in case of an SE detector we can only do it in case of a backscattered electron detector because there the detector is sensitive to the surface roughness as well.

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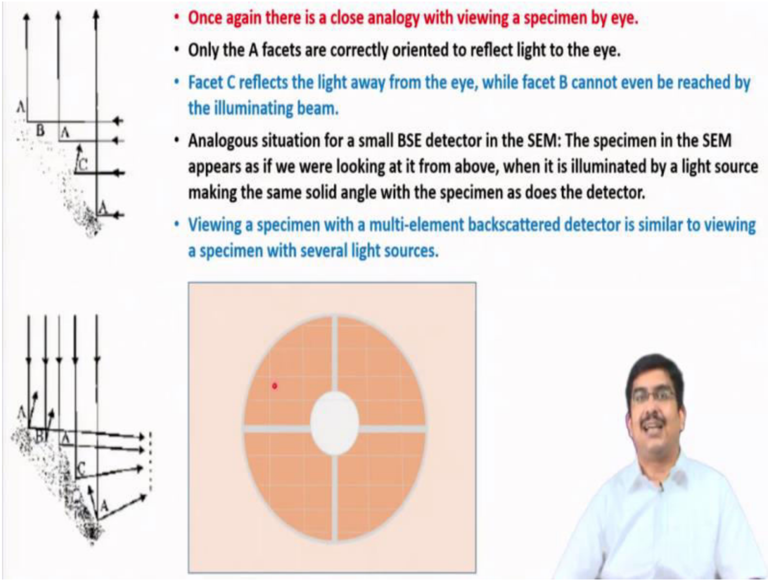

And this we can do it with this multi component detector so you know this multi element detector backscattered detector when I was discussing I said that there are four different detector and you may be wonder why we are creating four different detectors we could have only single detector here. This is because the backscattered detectors are such that their topographic contrast they are sensitive.

They have a directionality the way they generate this has a relationship with the primary electron beam and the specimen itself and that is why we have multiple elements in the backscattered electron detector and just by adjusting them we can play around with the topography mode as well as with the compositional mode we can play around. We will discuss this in the next class.

And that time we will discuss about these relationship between the light signal that is generated or light images and the backscattered electrons. So, this we will discuss in the next class for now good bye.