Techniques of Material Characterization
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Lecture – 30
Signal generation in SEM Continued

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are in 6th week now and we are discussing about scanning electron microscopy and in that this is the 5th lecture and we are still discussing about various type of signals that is generated in SEM and we started this signal generation discussion with brief history of SEM.

A little introduction about SEM, how it is similar to transmission electron microscope and what are the differences between them and then we stared discussing about various type of inelastic scattering that happens so we said that most of the signals that we use in scanning electron microscopy are basically generated by inelastic scattering. So, we discussed about various type of inelastic scattering, phonon scattering, Plasmon scattering so on.

Single valance electron scattering and then we saw that secondary effects like inner shell excitation phenomena and how secondary signals are generated, characteristics x-ray and Auger electron signals and how they are useful for identifying the chemical nature of the specimen and we then also discussed about the secondary electrons and backscattered electron.

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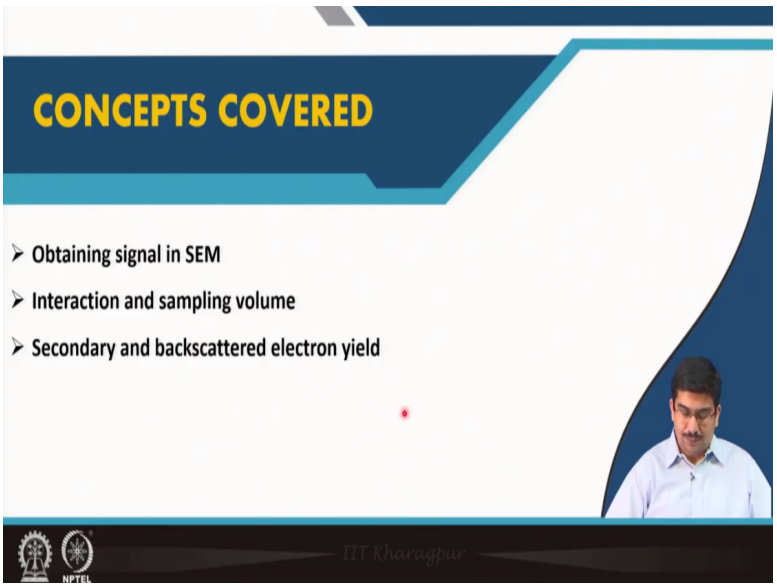

So, today we will be discussing about mostly obtaining an signal in SEM how that signal is obtained and what are the different type of signals that is obtained that we will discuss and then we will discuss about two very important things interaction and sampling volume because these are the things which ultimately controls the resolution and finally we will discuss about the secondary and backscattered electron yield.

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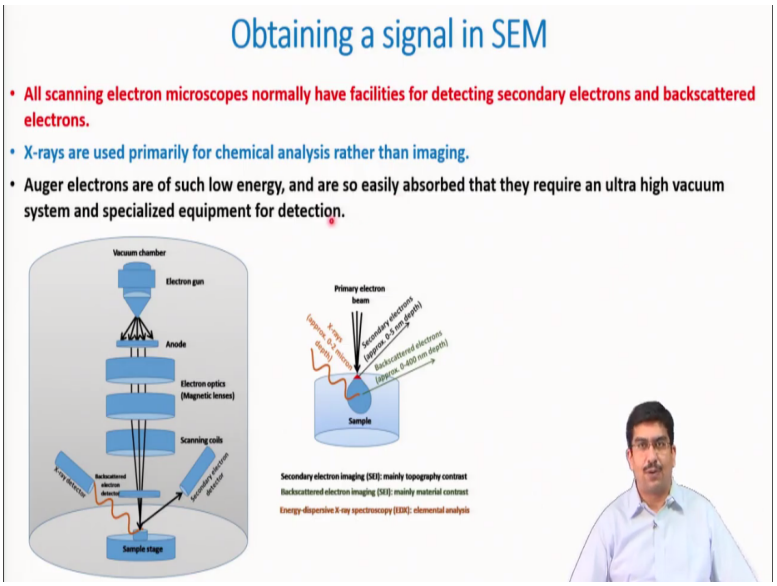

So, to begin with obtaining any signal in SEM. So, all scanning electron microscopes normally have facilities for detecting secondary electrons, backscattered electrons and sometimes characteristic x-ray as well, but out of all of these you need a detector first of all to detect some signal, you need one single detector at least by default and usually that by default detector is the secondary electron detector which is called Everhart–Thornley detector.

We will discuss about that detector possibly from next week that how it works and so on, but that is the detector by default that is present and that is possibly the oldest detector in scanning electron microscope which is there. Though it is written that is for secondary electrons, but you will realize from next week in next classes onwards that this can be also used for detecting backscattered electrons.

So, this one single detector it works for both secondary electron and backscattered electron then you can go for a separate detector for backscattered electron a special type of detector for backscattered electrons and finally you can add as an attachment you can add this x-ray detector. It comes as an attachment in the real SEM. So, normally these three different type of signals you work with and these are the most popular one.

Most of the SEMs possibly you will see whenever you see an SEM you will most likely going to see a secondary electron detector and a characteristic x-ray detectors EDS detectors that is what it is called. So, you are going to see the backscattered electron is a slightly special one, but sometimes these days that also it becomes almost like a norm and out of that obviously secondary electrons and backscattered electrons as we were discussing is used for imaging purpose.

And maybe another week or in a couple of lecture two or three lectures after that we will be possibly discussing about how the secondary and backscattered electrons are used for imaging purpose. X-rays as we said that characteristics x-ray and they carries the signature they are related their generation is related to the atomic number and in that sense they carries information about the chemical nature of the specimen of the elemental composition of the specimen.

So, characteristics x-ray signals are primarily used for chemical analysis. Auger electrons that also we realized that it also another important signal, another important type of emission that comes out of inner shell excitation, but as we discussed that Auger electrons normally is not numerous for most of the materials except for very low atomic number materials where Auger electron surpass that of the characteristics x-ray.

So, for detection for this low atomic number element and they also this low atomic number elements are not abundant if they are in a multiphase material if the width fraction of this low atomic number elements if that is low then possibly Auger electron is the only source otherwise even with characteristics x-ray we can go down not with the regular characteristics x-ray determination not with something called EDS energy dispersive spectroscopy not with that.

There is another type of detector we will discuss about that WDS wavelength dispersive spectroscopy. So, with that possibly we can even use the characteristics x-ray signal all the way down to low atomic number elements. So, Auger electron is a very, very specialized technique. Number one its low energy, low abundance in the signal and altogether and it can be absorbed very easily within the specimen chamber itself.

So, in order to have the detection of Auger electron is subjected to very much specialized equipment, specialized detector, ultra high vacuum and so on much of the things. So, we will not discuss about Auger electron detection we will restrict ourselves within secondary electron, backscattered electrons and characteristics x-ray.

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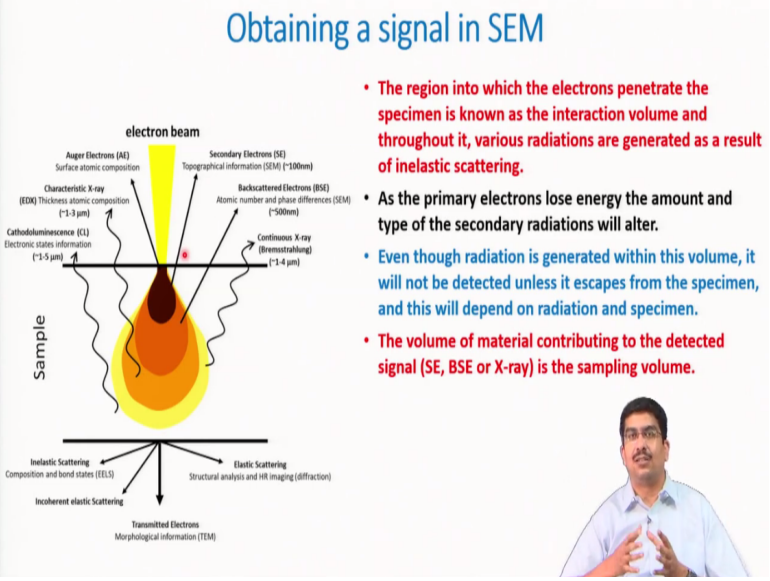

Now obtaining a signal in SEM how does it happens? So, this we have already discussed that region if we are considering an electron opaque specimen the one which is usually used in an

SEM. So, if we have that kind of a specimen then the region into which this electrons penetrate in the specimen and produce different type of signals by all means by all different type of inelastic scattering.

You have inner shell excitation, phonon scattering Plasmon whatever secondary electron, backscattered electron altogether. So, this entire volume within the specimen is called or is known as the interaction volume this we already have discussed and we have seen this nice shape of the typical shape of interaction volume that peer shape and various type of emissions that comes out of this interaction volume from this interaction volume from different depth all of this we discuss.

But this is basically the interaction volume and time and again I told you the significance of the interaction volume that is even if your beam is of this size and you are expecting very good resolution, but the resolution finally will be determined by the size of the interaction volume, but there is something else something more to it which I will discuss today. So, anyway but this interaction volume is one way of finding out the resolution.

And it is also one way to finding out the ease of detection of any signal. So, the signal the less of the energy needed for generating any signal the higher depth it is to be generated from a higher depth, but now if you just talk about in terms of a signal detection signal then not only the energy of the signal is important also important that whether that signal is able to come out of the specimen or not.

And if it comes out from which area or which volume within the specimen or which area over the surface of the specimen that this signal is coming out that is also very, very important in determining this interaction volume. So, even though the radiation is generated within this volume within this interaction volume it will not be detected unless it is escaped from the specimen.

And that escaping depends both on the type of the radiation as well as the specimen itself. We have seen the size, the effect of atomic number, accelerating voltage and so on and the size of this interaction volume through Monte Carlo simulations we have discussed all of these

things and it shows for lower atomic number specimen the size of the interaction volume is larger,

And for higher atomic number specimen the size of the interaction volume is smaller. So, all of these things is still valid. On top of that now because it has to be signal has to be detected. So, the volume of the material which contributing to the detected signal to the detection of the signal is called the sampling volume that means what let us say if you have this x-ray signals generated from deep within.

And x-ray signals typically takes low amount this is a rare occurrence very high energy and is absorbed from the primary electron that is why the x-ray photon that is emitted that also carries a lot of energy. So, altogether it can emit from a deeper depth and since it is higher energy it can come out all the way from this depth to the surface and it can come out. Problem is or the point is now this x-ray signal can come out from any region within the surface.

So, although it is generated within this region, but when it comes out of the surface that is important from which area, which volume it is coming out that will basically detect or that will decide the resolution finally and that is effectively the spots or the size or the area from which this signal is coming the detector is seeing how much area within the specimen that detector is basically seeing or collecting that signal that will determine the interaction volume or the spot size and ultimately the resolution.

So, there are two terms now we understand. One is the interaction volume; interaction volume still holds the same definition that it is though that area within the specimen from which the signal is generated that actually means that backscattered electron basically can be generated even from deeper depth or secondary electrons can be generated from deeper depth.

Their interaction volume we call that now that area from which the secondary electrons can actually come out and that is very small. So, interaction volume overall we can say interaction volume of secondary electron is very less and sampling volume corresponding

also is very less same thing for backscattered electrons. So, these two terms are used interchangeably.

They are related sampling volume and interaction volume, but there are some minor differences of that for certain signals the interaction volume and this sampling volume is different we will discuss about that, but basically these are the two things which decides how you can obtain a signal and what will decide the size of this area from which you are obtaining getting this signal.

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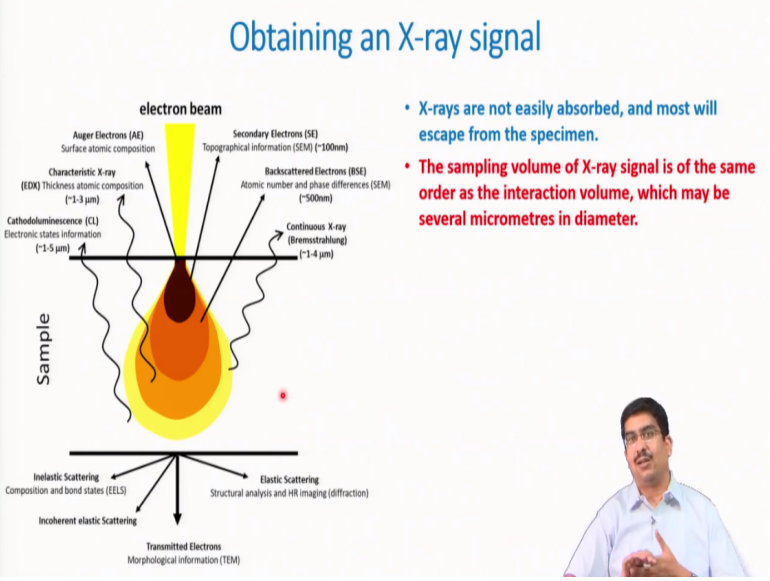

Now for x-ray signals as I already said x-ray signals are generated from deeper depth they are of high energy and that is why they are able to come out of the specimen with a larger sampling volume. They can very easily escape the specimen they are not easily absorbed within that. So, the sampling volume of x-ray signal is usually of the same order of magnitude as the interaction volume.

The point is this interaction volume and the sampling volume is both is in the micron range. So, even if you are using a electron beam usually in x-ray production the electron beam is much bigger it is a bigger spot size is bigger than your generally the BSE signal generally BSE or SE signal the spot size you tend to have a very fine spot size 2 to 10 nanometer, but for getting an x-ray signal usually the spot size is bigger.

That means the beam current increases and so on and so forth we will come on that, but what happens is that even if the beam is in the nanometer range ultimately the interaction and sample volume is in micron range. So, that is important and that means the special resolution of this x-ray signals are lower because you have to put the second beam at a distance in micron range because this interaction volume or sample volume for x-ray signal here is in the micron range it is very big.

So, the next beam that has to fall somewhere over here that distance will be in the micron range then only the two interaction volume and sample volume will be separate. If you put it too close again within nanometer then their interaction volume will overlap and you will be actually getting a signal not entirely from this region, but some part from this region. So, there will be a signal overlap and this will introduce error in the final chemical composition determination that you do.

So, that is why you have two interaction volume size of the interaction volume and size of the sampling volume is so important here. So, this is for x-ray signal.

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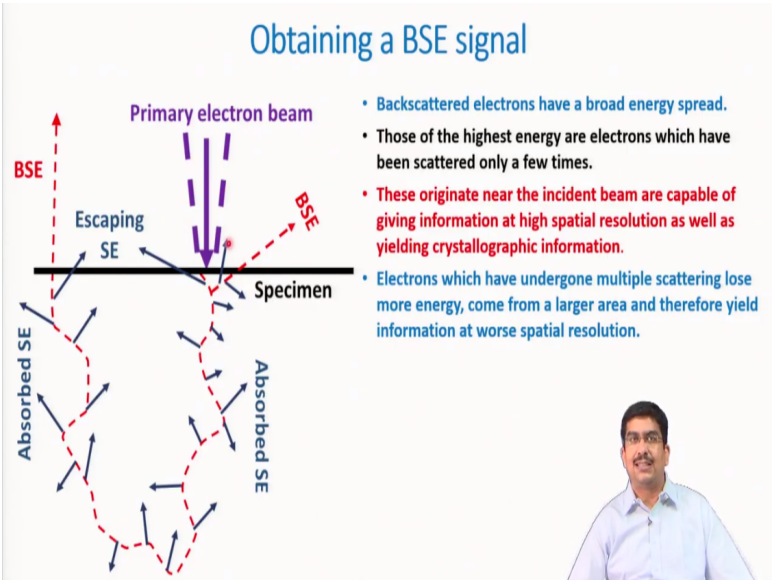

Now let us discuss about the backscattered signal. So, backscattered signal usually have a very broad energy spread because what I understand the backscattered electrons can be generated where exactly the beam electron beam is hitted. Backscattered electrons are special in scanning electron microscope in the sense that they are mostly generated by elastic

interaction and because of that the energy from the primary beam, the electrons which are present in the primary beam that energy is transferred to the backscattered electrons.

And that means that is very high energy the backscattered electrons. So, the backscattered electron which are just coming out from this place just next to or beneath the spot where the primary electron beam is hitting. If the backscattered electron comes out from that place then two things will happen. Number one that backscattered electron will be of very high energy because they are undergoing a pure elastic interaction with the primary electron beam.

So, they are of high energy and that energy is almost similar to the primary electron because they have undergone only pure elastic interaction. Now there is also a possibility that backscattered electron which is generated here undergoes or it goes deep within or the backscattered electron is generated somewhere from deep within and in the process of coming up to the surface.

These backscattered electron undergoes many, many inelastic scattering and finally when it comes out from this surface it is still a backscattered electron just because it is in the energy range. So, remember when I showed you this energy spectrum I told that everything is electron, the only difference that we have we classify something as backscatter, something as SE, something as Auger electron just depending on the energy of those.

And that energy has a spread. Secondary electrons has a spread of energy that also we showed. Similarly, backscattered electrons has a spread of energy. So, this backscattered electron which comes out from somewhere else other than the region over which this primary electron beam is falling those backscattered electrons will have much lower energy A and they undergoes a lot of inelastic scattering.

So, those energy of the backscattered electrons is not it will be a quite random quite spread whereas this backscattered electrons which are produced here they will be generated by pure elastic scattering and they will be having a very definitive energy. So, this backscattered electrons which are generated here these will be suitable for diffraction experiments we

discussed about that why the backscattered electrons are suitable for diffraction experiments because their energy is very much well defined.

Meaning their wavelength also is well defined or those backscattered electrons are only effective for producing the diffraction effect from this regions. So, that is what the backscattered electrons which are or which are generated or produced somewhere else they are not very suitable at least for diffraction experiment. For imaging purpose it is fine because imaging I do not really care much about this energy of the backscattered electron.

Whether they are of high energy only elastically scatter or they have undergone some inelastic scattering does not matter much, but still one thing is important that when the backscattered electrons are generated from this regions then what is happening is that their sampling volume is quite similar to their interaction volume. but when I am considering all the backscattered electrons which are generated from this region.

Now what happens is the interaction volume that means the depth from which they are generated and the sampling volume is not the same.

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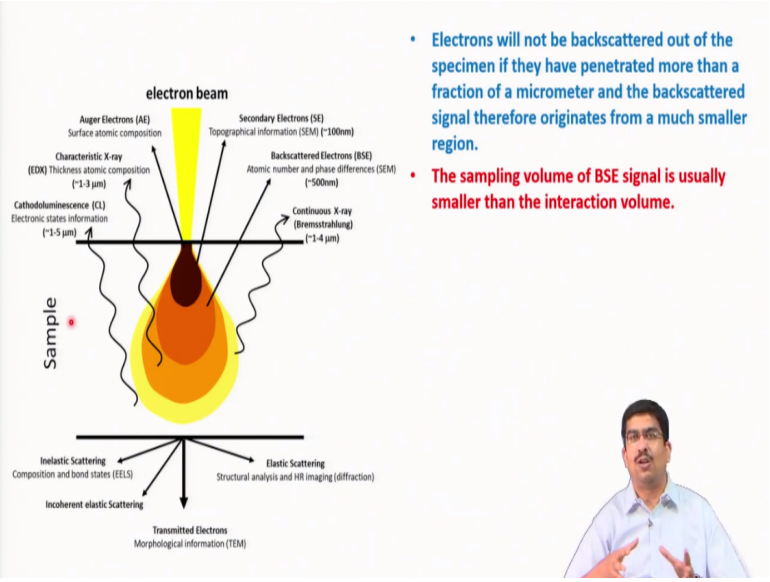

Now this what is happening that this backscattered electrons is sampling volume of the backscattered signal is usually smaller than the interaction volume and why is so? Because this interaction volume of the backscattered electrons is pretty small, much smaller than the

EDS signal energy x-ray signal even though the backscattered electrons are of high energy, but if they are produced from deep within the specimen then they will not be able to come out of this.

So, even if backscattered specimens are produced from a higher depth they are not of that high energy as compared to the x-ray signal. So, those backscattered electrons will not be able to come out of the specimen. So, there sampling volume means the area from which they are coming out actually though they are produced from a larger depth. The area finally from which they are coming out will be much smaller than their interaction volume that which is deep within which may be bigger.

So, this is an important point to remember that for at least for backscattered electrons the sampling volume is usually smaller than the interaction volume it can be same also depending on the acceleration voltage basically. If you suitably control the acceleration voltage then possibly you can generate this backscattered electrons of the same sampling volume and interaction volume to be almost of the same order.

But whatever you do ideally the backscattered electrons will still have some energy spread and usually their interaction volume or that sampling volume will be little bigger than the electron beam, the size of the spot. So, for backscattered electron also the spatial resolution is determined by this size of the interaction volume or size of the sampling volume. It is not as big as the EDS signal definitely not in the micron range, but still considerably bigger than the spots the electron beam size.

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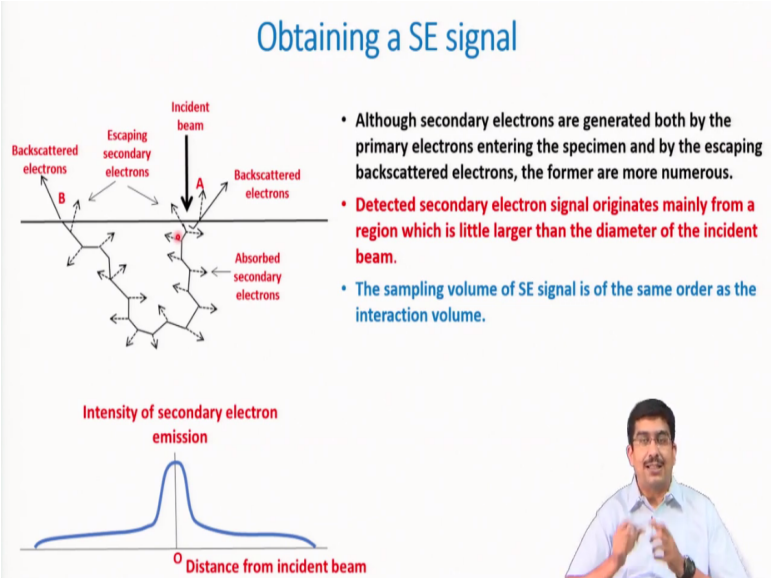

Finally, when you discuss about the SE signal already we discussed that SE signals are different type there is SE1 there is SE. In fact the BSE signals the way they are produced we can imagine that this is BSE 1 and this is BSE 2 and so on, but that kind of a nomenclature is not so popular basically. So, all are called BSE whether if they are produced here or they are produced there they are called BSE signals.

And they are mostly captured both of them are captured in the imaging purpose at least. Only for diffraction purposes these two are different these backscattered signals produced here and these backscattered signals produced somewhere else these two are different when I consider the diffraction purpose, diffraction experiment otherwise for imaging purpose both the backscattered electron are equally good and detector sees or detector captures both of them.

Now about secondary electron signal now SE signals are generated both by primary electrons that is entering in the specimen or the primary electrons which are going out losing most of their energy and going out and also the backscattered electrons which are sort of lost their energy so that is how SE 1 and SE 2 signals are generated. SE 1 signals are generated mostly by the incident beam the primary electron beam that mostly generates the SE1 signals we discussed about that.

So, secondary electrons and that is quite numerous. So, primary electron beam generating SE1 signal is quite large. It is very large number of primary electron is there in the beam and

that can produce a large number of secondary electron signal right in the spot where it hits where the primary beam heats here compared to that the backscattered electrons which are producing secondary SE 2 electrons that is less in number and the energy also for those electrons the energy also is quite less.

And this is what you can see the intensity of secondary electron emission with the distance of the incident beam. So, the maximum number of secondary electrons basically generates very close to the incident electron beam that is SE1 signals are most numerous and very close to the primary beam itself. So, here what happens is that those are the electrons this SE1 signals are mostly coming out of the specimen.

And you remember in SE signal we said that it is those part of the electrons which are able to come out of the specimen. So, SE1 signals can come out very easily from this spot and from there. So, what we can think is that only if we capture the SE1 signal then we can that will be good enough for the detector to produce a contrast in the final image. So, SE1 signal our aim is to capture only SE1 signal and we are done.

BSE the SE2 signals are far off and there is no point capturing then there are less in number their energy is quite random so and so. Primarily, we try to capture this SE1 signal point is the SE1 signals are not since they are low energy remember this. SE signals are very low energy. So, if they are produced somewhere deep down inside they are produced there. The SE signals are also produced from deep within.

The only point is they are not able to come up to the surface and can escape the surface because of their small energy. So, ultimately what happens is the SE1 signals are generated from a very less depth meaning that their interaction volume is very, very small and at the same time the primary electron beam is producing the SE1. So, their sampling volume is also small.

So, for SE signal again just like x-ray signal SE signal also sampling volume and interaction volume is almost of the same order, but it is much less, much smaller both the size of the interaction volume and sampling volume it is much smaller than the corresponding x-ray

signal otherwise interaction volume, sampling volume these relationship is basically the same for SE and BSE signals.

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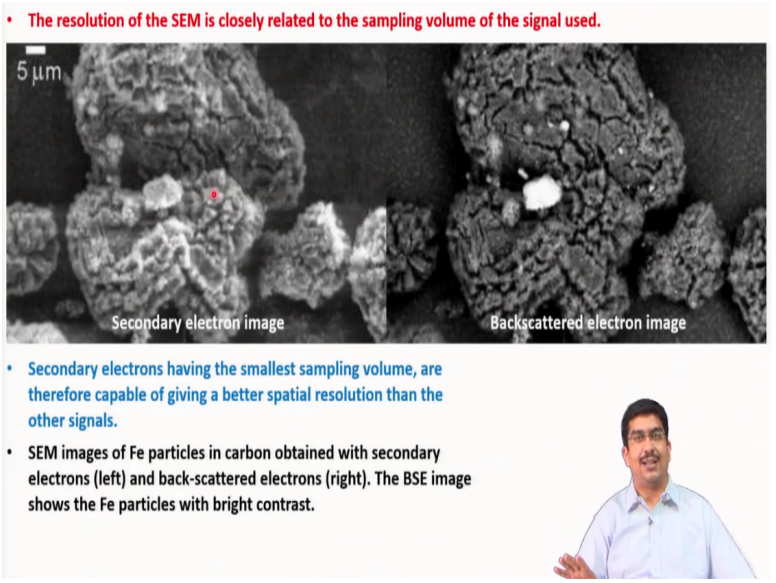

Now as I discussed the resolution of the SEM also depends on the sampling volume of the signal used whatever signal we used whatever signal we use the sampling volume. It depends on the interaction volume or sampling volume if that is small then the special resolution will correspondingly also very small other than diffraction, aberration all the effects and all these also another big important.

And that is why we were in the first place we were interested to use this SEM scanning mode so that we can use a very fine beam and then they make the beam raster so that we can increase the field of view, but in the process what is happening is even if we are having a very fine electron beam still the spot size or the area from which the signal is produced that is ultimately controlled by what type of signal it is and what type of specimen it is.

If we now consider the difference between SE and BSE signals. SE signals they are having very small sampling volume and therefore the special resolution for SE signal is much better than other signals for example from BSE signal. If you compare these two images this is captured with a secondary electron and this is captured with a backscattered electron definitely in the secondary electrons I am able to see much more features this is something called topographic contrast.

So, I am able to see more features here at a much better way than this backscattered electron image. Backscattering electron is in fact showing compositional contrast this is an iron particle which is not so obviously in SE signal. If you look at in the SE signals you do not know which one is the iron materials here. So, only if you look at in the backscattered mode then you will be able to know which one is iron here.

That is why backscattered is so important that it can give you some kind of compositional information as well even the special resolution is less than secondary electrons still backscattered is used because of its compositional contrast that it can produce which SE cannot produce, but more or less SE signals has a better spatial resolution.

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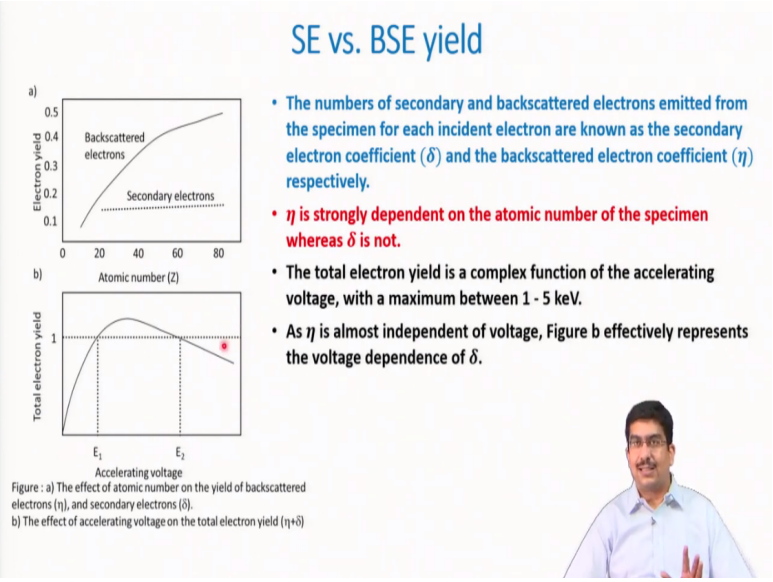

Figure : a) The effect of atomic number on the yield of backscattered electrons (η), and secondary electrons (δ).
b) The effect of accelerating voltage on the total electron yield (η+δ)

Now one last thing that SE versus BSE yield. So, this SE yield or BSE yield that is also called expressed by something called secondary electron coefficient delta and backscattered electron coefficient eta. So, this is expressed as the number of secondary or backscattered electrons emitted from the specimen per primary electron or incident electron primary beam whatever electron is there.

Whatever number of electron is there in the incident electron or the ratio between the number of secondary electrons or backscattered electrons generated to the number of electrons present in the primary beam that is giving the secondary electron coefficient and

backscattered electron coefficient. As we already discussed from the genesis secondary electron and backscattered electron

This backscattered electron coefficient is strongly dependent on the atomic number whereas the secondary electron it does not depend on the atomic number this is what is shown here. This is the atomic number and you can see that backscattered electron yield is increasing and almost become 0.5 that means 50% of primary electrons are converted to backscattered. If you go somewhere within like an atomic number of 60 to 80 which is very heavy metals tungsten, mole and then we are possibly going towards the radioactive material.

For them the backscattered electron yield is around 50% whereas the secondary electron remains almost constant whether it is very low atomic number element like aluminum, carbon and so and to all the way to very heavy elements like tungsten mole and all. This secondary election yield is not going to change, but backscattering electron yield on the other hand it depends strongly on the accelerating voltage.

So, this dependence of backscattered and SE electron the total electron yield that is the total should be 1, but it is more than that we will come to that because you know that we discussed about this SE yield can be more than one because SE has many other sources. BSE signals can produce SE2, SE3 and all SE4 signals so those altogether if you count then sometimes it can surpass one the ratio.

But one or more this entire variation is because of the SE signal with the accelerating voltage. The BSE signal does not show any dependence more or less we will see that it is not true, but BSE signal more or less does not show much of an effect from accelerating voltage it is mostly the SE signals that is depending on the accelerating voltage.

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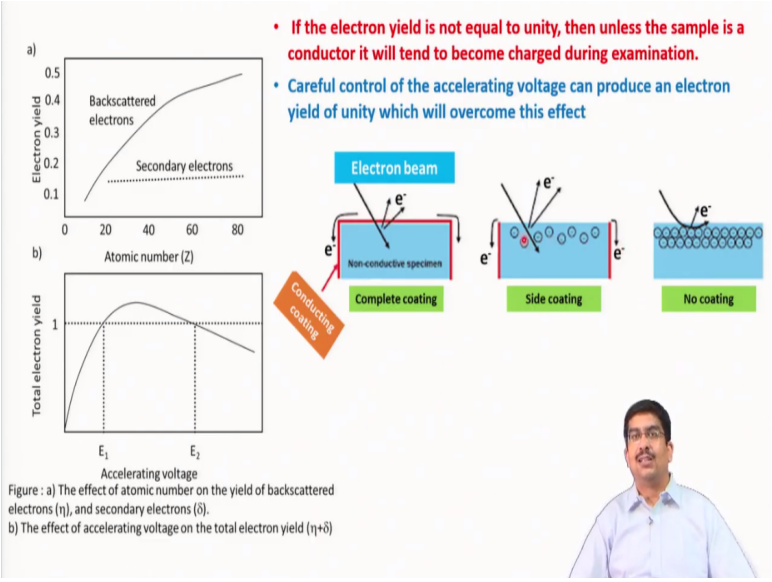

Figure : a) The effect of atomic number on the yield of backscattered electrons (η), and secondary electrons (δ).
b) The effect of accelerating voltage on the total electron yield (η+δ)

Now if this electron yield is not equal to unity that means if the SE and BSE together is not one for the primary electron then what will happen is that some electrons from the primary beam may get absorbed on the surface of the specimen if the specimen is not conducting then what will happen is this electrons will stay there and it will repel the incoming electrons and that is a typical phenomena called electron charging.

And that happens because there is no conducting path because the backscattered and secondary electron yield is not equal to unity means the primary beam is not able to produce enough number of secondary or backscattered electron then this charging problem can happen particularly it will happen if the sample is non-conducting that is why non-conducting samples like ceramic materials like polymeric material we tend to give them a coating usually a gold coating that we give.

And then we make, we provide a conducting path and through that path whatever the extra electrons are present from the primary beam these are pass through. So, there is no such electron deposition or excess electrons at the surface of the specimen and there is no charging problem happens.

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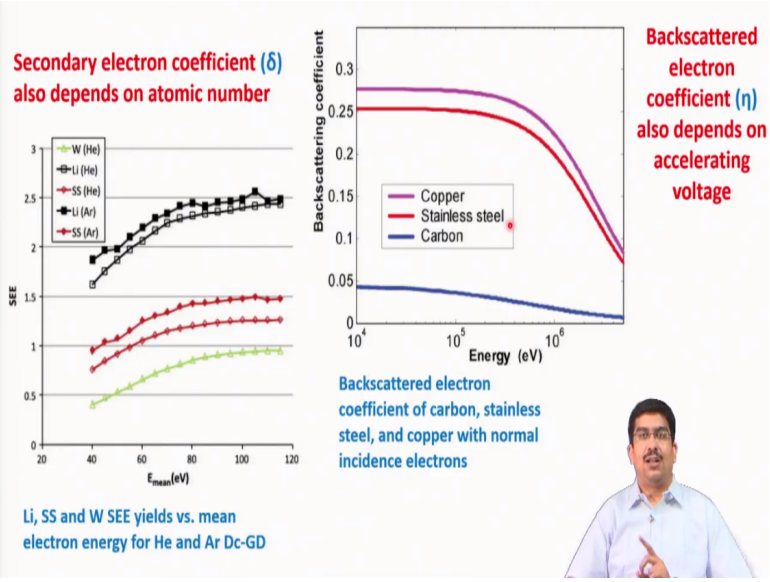

And finally I said that the secondary electron coefficient does not depend on the atomic number. Backscattered electron coefficient does not depend on the accelerating voltage this is not exactly true. Secondary electron coefficient also depends on atomic number, for example, if you see something like between tungsten and lithium secondary electron yield of tungsten is much smaller than lithium.

Lithium produces much more secondary electrons because it is atomic number is less, binding (30:45) energy is less the secondary it can easily knock out some of the electrons from the atoms in case of tungsten it is not possible. In tungsten, obviously backscattering yield will be much higher that is invariably there, but secondary electron remember secondary electrons SE1 at least is generated because from the atom of the specimen and electron is knocked off that is something like one of the source of secondary electron generation inelastic scattering afterwards can happen obviously and that also can generate secondary electron.

So, overall this lithium that atomic number lower atomic number usually produces much higher yield for secondary electrons. Similarly, backscattering electrons again will depend on accelerating voltage that dependence will not be as strong as secondary electron, but after a certain critical voltage after a certain critical voltage and this is particularly true for high atomic number elements that backscattering yield will drastically drop down.

What will happen is that accelerating voltage are now the electrons are of so high energy that they deflecting them by an scattering angle close to 180 degree becomes very, very difficult. So, they just pass through the atom without having much of a scattering or even if the scattering happens by the positively charged nuclear that is not enough for producing a backscattered electron.

So that is the backscattered after a critical voltage the backscattered electron yield drastically drops for this high even for the high atomic number elements. So, with all of this now we close this discussion about signal generation in SEM and we will be discussing in the next class onwards we will be discussing how to detect these signals and how to use it for producing image and various other kind of chemical analysis and so on and maybe diffraction experiments how to do in SEM. So, with that good bye.