

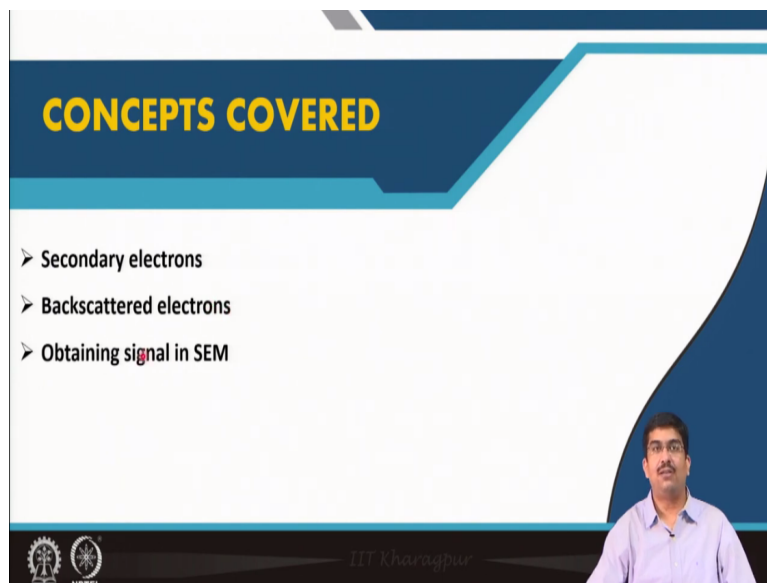
Techniques of Material Characterization
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Lecture – 29
Signal generation in SEM Continued

Welcome everyone to this NPTEL online course on techniques of materials characterization and we are in module 6 now. So, we were discussing about scanning electron microscopy and in that we are still discussing about various type of signal generations in SEM and in the last class we discussed about a very important method of a very important effect or very important event of which produces various secondary effects and that secondary effects are very useful for detection signal in scanning electron microscopy.

So, we are talking about inner shell excitation and how the relaxation of this inner shell excitation events when the relaxation happened various types of signals are generated cathodoluminescence, characteristics x-ray, Auger electrons all of this different signals was generated and how the detection or how they are related to the chemical identity of the specimens and how we can detect if we detect them how we can get information about the chemical analysis, how can we carry chemical analysis in scanning electron microscopy.

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So, from there today we will be discussing about various other signals which are mostly used for imaging purpose plus we can also use them at least couple of them we can use for other

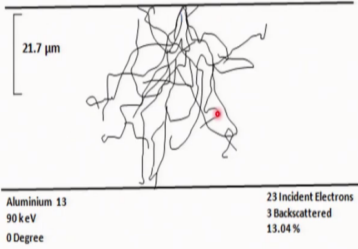
purpose we will discuss that. So, first we will discuss about a signal secondary electron signal and this is possibly the most commonly used signal for in scanning electron microscope and then we will discuss about the backscattered electron signals.

And finally we will discuss about obtaining a signal how we can utilize this different type of signals in scanning electron microscope.


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Inelastic scattering and absorption

- Even for the thinnest specimen, more than one of the inelastic scattering processes can take place.
- In a 'solid' specimen many such events will occur until the electron is stopped or leaves through the surface it entered.
- Majority of electrons are brought to a halt within the solid but a few are backscattered and leave the specimen.
- The volume within which ~95% of the primary electrons are brought to rest is referred to as the interaction volume.



Aluminum 13	23 Incident Electrons
90 keV	3 Backscattered
0 Degree	13.04%



But let us just discuss about the inelastic scattering for one last thing about inelastic scattering that is the absorption of this inelastic scattering. So, if you again check out this Monte Carlo simulation for an aluminum sample and this is again showing the trajectory of various electrons which is now contained within the specimen itself, but since this is a very thin specimen may be some of the electrons are coming out of it.

So, as we can understand that these primary electrons which are entering into the specimen they at least they will undergo inelastic scattering for one or more than one time that has to be. So, pure elastic scattering is very much rare event and even in a very thin specimen like the one electron transparent specimen that we use in case of a transmission electron microscope even with that inelastic scattering happens.

Now, if we consider an electron opaque specimen very thick specimen then in that case what will happen the all the electrons will undergo various different type of inelastic scattering

whatever effects we have discussed so far phonon scattering, Plasmon scattering and then it will undergo with single valance excitation, it will undergo with the inner shell excitation and it will generate characteristics x-ray, cathodoluminescence and then Auger electron all of this.

The electron primary electron beam will undergo all of these various effects, but finally when the primary electrons they lose all their energy through this various inelastic scattering events they will finally produce phonon scattering. So, phonon scattering is the end of any primary or the end event for most of the primary electron beam which enters into the specimens and they do this phonon scattering finally they produce a lot of heat.

So, other than this other than producing the phonon scattering if the electron beam can come out primary electrons if they can come out of the specimen either in the form of secondary electron or backscattered electrons. These are the two ways the primary electron beam or the primary electrons can come out of the specimen and when they come out of the specimen then they are useful for detection and they can used for mostly imaging purpose.

So, most of the electrons finally end up in the form of inelastic scattering and produce the phonons and in the process they heat up only a very small fraction of it will finally come out as secondary or backscattered electrons. Also we have already seen we have discussed about this K-O range and there we have seen that around 95% of the primary electrons are brought into rest and that where this area through which they are back to rest this area is known as the interaction volume only 5% of them basically can come out of the specimen.

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Secondary electrons

- Secondary electrons: This is not a very accurate term, but is used to describe those electrons which escape from the specimen with energies below about 50 eV.
- They could conceivably be primary electrons which at the very end of their trajectory reach the surface with a few eV remaining.
- They are also likely to be electrons to which a small amount of energy has been transferred within a short distance of the surface.

So, with this let us start discussing about the secondary electron. So, secondary electron the name as the name suggest it is a electron and it is sometimes confusing. My students many a times they get confused with secondary electron they thought that the secondary electrons are produced by secondary effect that is why they are secondary electron actually not. Now we understand that through secondary effect the electrons that is produced by secondary effect is called Auger electrons.

And this secondary electrons are different from inner shell excitation event the electrons which are produced by inner shell excitation effect as a result of the secondary emission or this is a kind of secondary emission. This secondary electrons are not produced by any secondary effect they are not the result of any inner shell excitation event. So, what are these secondary electron?

Basically secondary electrons the term is not a very accurate term. It is used to describe those electrons which can escape the specimens A and B their energy level is less than 50 electron volt. So, these two are the major criteria of identifying or defining certain electrons as secondary electrons. So, you can think that this is a very loose definition. So, there are various type of electron that can come out of that specimen.

Out of those entire range of electrons which are coming out of the specimen or which are produced when a primary electron beams hits the specimen only those electrons which are

below this 50 electron volt range can be called secondary electron. Now why this 50 electron volt range is I will come to that in a minute or so, but this is basically more or less this is what the criteria.

This is what the cut off is that does not mean that electrons cannot have energy or secondary electrons cannot have energy more than 50 electron volt they can. Still they are not useful their number is so less that they are not very useful for detection. So, the detector the energy of those electrons the very small number of high energy secondary electrons are not useful for detection so they cannot reach to the detector and cannot be used for imaging purpose.

So, we essentially keep this 50 electron volt as the cut off that is the point with a current detector efficiency. So, this is related to the detector efficiency in future if we can come up with a better detector which can even acquire those electrons which are above 50 electron volt maybe this cut off will be shifted. So, this is still now this is a very loose term very vague term or this electrons are identified based on the energy level which is less than 50 electron volt.

And obviously for detection purpose they have to come out of the specimen these are the two conditions. So, how they are produced basically. So, these secondary electrons are most generally produced or we can imagine that they are basically primary electrons the electrons which are present in a primary beam or incident electron itself and then they are undergoing various type of inelastic scattering events.

All different kind of inelastic scattering maybe inner shell excitation, maybe Plasmon scattering, maybe phonon scattering all different type of inelastic scattering events are happening with the primary electrons and they are losing their energy in the process and remember along with the loss of energy they are also getting scattered. So, their trajectory is continuously getting changed.

So, what is happening is that in that inelastic scattering processes the primary electron beam this is number one losing its energy second thing it is getting deflected also. So by this two methods if somehow they comes out of the specimen in the deflection process it so happens

that the primary electrons which entered somehow it can come out. So, one such instance he is showing that if it is coming out of the specimen.

Now that electron will carry very less amount of energy because most of the energy that is a primary electron, but most of the energy is lost by many different other types of inelastic scattering process. Remember inelastic scattering processes only possibility are those 5 or 6 which we already discussed, but each of them and each of them has different amount of energy most amount of energy is lost by inner shell excitation process.

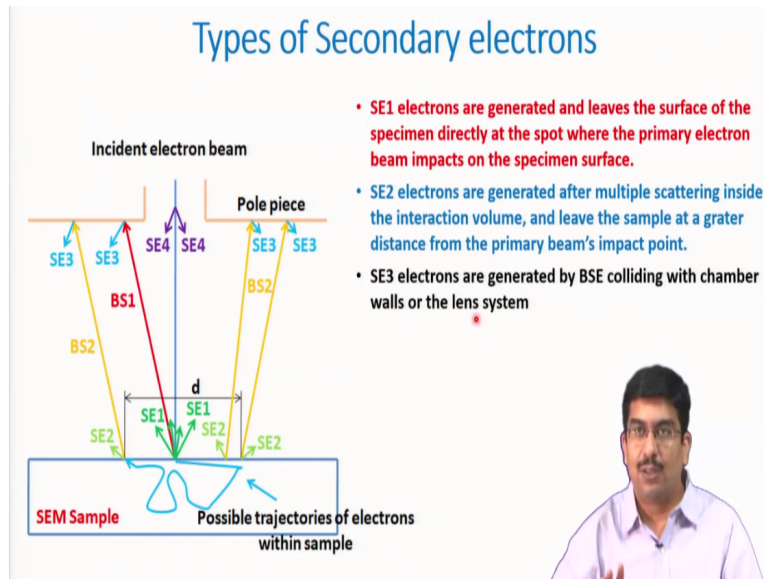
So, if the inner shell excitation happens primary electrons are losing their lots of their energy and somehow by deflection process they are able to come out of the specimen and they are within that range so they can now called secondary electrons. So, secondary electron or primary electron that primary electron which loses all part or most of this energy by inelastic scattering events and it comes out of the specimen through this deflection process.

So, that is what is the main definition of secondary electron, they are also likely to be the electrons to which a small amount of energy is transferred within a short distance of the surface. So, this can be also those knocked out electrons. So, if you remember that we discussed in the last class inner shell excitation in the inner shell excitation process those electrons some of the electrons are basically knocked out or even in cathodoluminescence process some of the electrons are knocked out.

So, those electrons are also can come out with that specific energy their energy in that specific range if they comes out of the surface then they also can be called as a secondary electron. So, the secondary electrons are term basically defines the energy rather than their formation mechanism. The formation mechanism is not so important for secondary electrons only their energy level is important.

If they are within that energy level and they can come out of the specimen that is it they are secondary electrons.

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So, according to their formation we can divide the secondary electrons into three different types. Number one, secondary SE1 that is what it is called SE1 electron. So, SE1 electrons are basically generated and they leave the specimen or they leave the surface of the specimen right from the spot where the primary beam hits. So, most often these are the electrons which are created by inner shell excitation process as a secondary effect.

So, I mean this is primary electron beam hits and an electron is removed from the specimen atom and that is having a very less amount of energy and comes out of the specimen. So, this is most likely this is the SE1 electron, but still we are not as I said the formation mechanism is not very important for secondary electrons the energy is important. So, these we are characterizing SE1 electrons as those which are mostly coming out within that energy range which are mostly coming out from the locations where the primary electron beam hits the specimen that is it that is the secondary SE1 electron that is the definition for SE1 electrons.

Now SE2 electrons are generated usually far away from this primary electron beam where the primary electron beam hits far away from there the electrons which are generated and still within that energy range less than 50 electron volt these are SE2 electrons secondary SE2 secondary electron 2. So, these are the electrons far away from the primary electron beam. How they are generated?

The process just now we discussed that this primary electron entering into the specimen undergoing different type of inelastic scattering and different type of deflection and different amount of scattering is happening and somehow finally they are able to come out some other place. So, primary electron is hitting right here and this SE2 electrons are coming out from completely far away from the primary electrons only the SE1 electrons are coming out from exactly that place where the primary electron beam hits.

So, this is SE2. Now there can be SE3 electrons also mainly SE3 electrons are generated by BSE backscattered electrons. So, backscattered electrons when they are coming out of the specimen they are also can undergo. So, backscattered electron mostly generates from the place where this primary electron beam hits and then that backscattered electron and usually backscattered electron has a very high energy, much higher compared to secondary electron.

This is almost comparable to the energy of the primary electron beam. So, backscattered electrons can again so they are generated by elastic scattering firstly that is true. So, primary electron beam which is getting backscattered is like they are undergoing an elastic scattering and only thing is that scattering angle is close to 180 degree that is the backscattered electron. So, since they are generated by elastic scattering they are carrying almost the same amount of energy as the primary electron beam.

But if they are not able to come out of the specimen they are now going through or going within the specimen various paths they are following and then slowly when they are coming out inelastic scattering they are coming out from the specimen and still carries very high energy. They are colliding with the specimen chamber or the lens system or various other parts and producing a secondary electron from there.

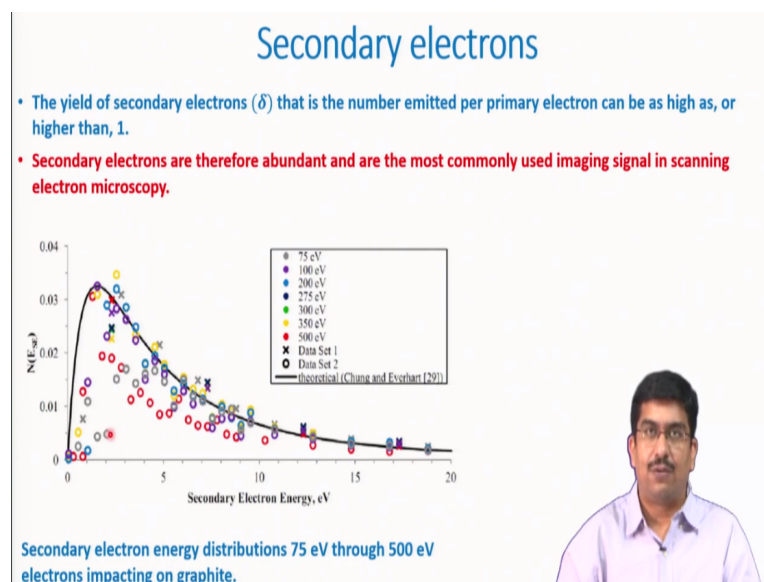
So for those parts this backscattered electrons are now working as a primary beam and producing the secondary electron or SE1 electrons for them, but here with respect to the specimen we are calling them SE3 so that is the point. So, there are many such electrons secondary electrons can be caused and these are called SE3, SE4 and so on and so forth and different, different parts, different formation mechanism.

But ultimately this is the formation mechanism is more or less these two. Primary electron beams directly hitting and these inner shell excitations happens and the secondary electron is emitted that is one process. Second process is primary electron undergoing lots of inelastic scattering using most of their energy and finally coming out as a very small energy electron. So, these are the two process with which it can generate.

Now which beam is producing that whether it is a primary electron beam or the backscattered electron beam depending on that you have SE3, SE4 so on and so forth, but mostly for most of the purposes we use SE1 electrons we will discuss about that. The SE1 electrons is the most important one for imaging purpose. We do not really bother about SE2 or SE3 electron because those cannot be used for imaging.

These are mostly artifacts which sort of I would say causes noise in the final in the detection or causes noise in the detector and finally appears as an artifact in the image the SE3, SE2, SE4 even SE2 also SE4 all of these, but we primarily mostly deals with SE1 electrons and capture that for imaging purpose.

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The yield of secondary electrons of course the delta that is we will discuss about this yield of secondary electron that is called secondary electron coefficient. This is basically defined as the number of SE produced per primary electron. So, if 100 number of primary electrons are heating the specimen how many number of SE electrons are produced. As I said the SE

electrons can be produced not only from primary beam, but from other sources like the BSE electron SE3, SE4 and so on.

So, often time what happens is that the number of SE electrons that is produced will exceed the number of electrons present in the primary beam that means the yield of secondary electrons maybe higher than one at times so many if the backscattered electrons are very high in number they can go and produce many other number of SE electrons, but ultimately they are all falling within the same energy level.

We do not know whether they are SE1, SE2, SE3 and the yield when we calculate the yield in the detector we have seen that their number is much higher than the primary electron beam this can happen. Second thing we must remember is that the SE electrons and therefore the SE electrons are very, very abundant they are used for imaging signals and so on and this is how the secondary electron energy is distributed basically.

And this is done in a computational work this is done by different, different accelerating voltage, different level of secondary electron energies, electrons impacting the primary beam energy the electron which are present in the primary beam that energy is varied and depending on that if you look at here the secondary electrons that is emitted their energy shows a very long distribution and this is their number.

So, the low energy secondary electrons are most abundant of course because that process it is a most probable process the low energy secondary electron emission and as the energy of the secondary electrons is increasing their number is decreasing. So, basically it is difficult to say basically which one is SE1 which one is SE2, which one is SE3 from this (()) (19:03) it is all are mixed all of them are mixed.

But more or less what we can understand is that this SE3, SE4 and so on their occurrence is much lower, their energy is higher and their occurrence probability is much lower compared to something like SE2 or SE1 even. So, their SE1, SE2 these are the electrons which are much more abundant and their energies are also much in the lesser size. So, that we need to remember for this.

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Backscattered electrons

- Backscattered electrons are electrons resulting from the elastic interaction of the primary beam electrons with the atomic nuclei of the materials.
- Some primary electrons may leave the surface before giving up all their energy.
- They are most likely to do this while they still have a large fraction of their incident energy.

The diagram shows two atomic models. The left model, labeled 'Inelastic scattering', shows a 'Primary electron' entering from the top left and interacting with an inner shell electron, resulting in a 'SE' (secondary electron) being emitted. The right model, labeled 'Elastic scattering', shows a 'Primary electron' entering from the top left and interacting with the nucleus, resulting in a 'BSE' (backscattered electron) being emitted. A small inset photo of a man is visible in the bottom right corner of the slide.

Now from secondary electrons we are moving to another type of electrons electron signal or electron which is produced in case of in SE. So, backscattered electrons already we had a enough discussion about backscattered electrons when we were discussing about transmission electron microscope you said that backscattered electrons are produced by elastic interaction. So, inelastic interaction produces this secondary electrons.

And elastic interaction or elastic scattering produce backscattered electron. Now remember backscattered electron is the fraction of elastically scatted electrons. So, we have seen HAADF mode, for example, in HAADF mode there was this high angle annular dark filed. We also can have annular dark field where we are using not so high so particular scattering. High angle annular scattering means even for the scattering angle.

And out of this entire elastic scattering event the backscattered electrons are special in the sense their scattering angle is almost close to 180 so that is why it is called backscattered electron. Now, backscattered electrons as I said resulting from elastic interaction and they are scattering angle is very high and if they are able to come out of the specimen so that is important.

In case of an SEM signal generation which we will discuss, they must come out of the specimen. So, backscattered electrons when they comes out of the specimen they can be

detected and at that condition since backscattered electrons are generated by this elastic interaction process where the energy of the primary electron is transferred to the backscattered electron.

So, even when they are coming out of the specimen their energy is much higher than the secondary electrons and also second thing is that the backscattering electrons basically this elastic interaction or the production of backscattering electron increases with atomic number. So, as the atomic number increases this nucleus becomes heavier and heavier and that can increase the yield of backscattered electrons we have already discussed this also in previous.

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Backscattered electrons

- Backscattered electrons are not usually as numerous as secondary electrons but most of them carry high energies.
- They are used for imaging, diffraction and compositional analysis in the SEM.

The slide contains a diagram illustrating Bragg diffraction with the equation $n\lambda = 2d\sin\theta$. It shows incident and reflection angles θ and a lattice spacing d . Below the diagram is a graph showing detector response with peaks labeled SE, Auger, BSE, and Low loss. To the right of the graph is a circular SEM image showing a grid pattern. In the bottom right corner, there is a small inset video of a man speaking.

So, backscattered electrons again if we look at this secondary electron versus backscattered electron it depends on various other factors like it depends on the energy, it depends on the atomic number if the atomic number is very high than the backscattered electron yield can be higher than the secondary electron and if the acceleration voltage is such that it is very high energy electron than also the backscattering electrons will yield will be lower and secondary electron yield will be higher.

So, this is very much relative the number of backscattered electrons or number of secondary electron is a very relative term we will see that in a few minutes, but backscattered electrons the way they are different from secondary electrons is that their energy is always very, very

high and what lies in between secondary and backscattered electron is the Auger electrons about which we have already discussed.

And Auger electron this energy also is very much characteristics of the specimen itself, the chemical identity of the specimen. Similarly, the energy of the backscattered electrons is also very much related to the chemical or it reveals the chemical identity or it is related to the chemical identity of the specimen involved, but one difference is there of course the Auger electrons are produced by secondary effects.

Whereas backscattered electron secondary effects in inelastic scattering backscattered electrons are produced by an elastic scattering so that is the difference between Auger and backscattered electron. Now, backscattered electrons are very important and compared to the secondary electrons backscattered electrons can be used for multiple purpose, they can be used for imaging, they can be used for diffraction.

They can be used for compositional analysis. We will see all of this we will see and example of backscattered electrons used for imaging purpose, backscattered electron used for diffraction purpose and compositional analysis. Compositional analysis as I already said that the production of backscattered electron is related to the atomic number of the element. So, that is how if we can properly calibrate it we can basically get an idea about the chemical nature of this elements or rather the elements present within the specimen.

And imaging also if you capture the backscattered electron it can generate a contrast in the material depending on element. So, just now we talked about elements the compositional contrast it can generate, it can also generate other type of contrast topographical contrast and so on which we will discuss. What is important to understand here and we will discuss more about this the diffraction.

Why the backscattered electrons can be used for diffraction purpose this is sometimes my students gets confused and ask me this question. Point is and this we said during our discussion on the electron diffraction in transmission electron microscope there also I said

that the part of the electrons which are elastically scattered that only can be used for diffraction purpose.

Now why is so? If you look at this Bragg's law $n\lambda = 2d \sin \theta$. So, if you know for any known material let us say you are doing it for a known material or even for an unknown material whatever. So, your d is pretty much fix the inter atomic or inter planar spacing is very much fixed you cannot change that so often. Again your λ depends on the source signal and depending on that the $\sin \theta$ basically changes.

Now what happens is that if you have an elastic interaction if signal is produced by elastic interaction then you start with a coherent beam where you know the energy, you know the wavelength they are all in phase and so on. You start with them and you know the λ value as well your source signal you know the λ value most importantly. After elastic scattering what happens is this signal which is produced.

As we discussed in the elastic scattering there is no further change except for change in direction or sometimes the change in scattering when the scattering event or diffraction effect just there is a change and just there is a deflection. There is no change in energy that means that there is no change in the wavelength as well the beam still remains coherent even after an elastic scattering process.

So, that is why anything that undergoes an elastic scattering it still remains as a coherent beam or coherent electron and for a diffraction purpose you need to use a coherent beam. So, your incident beam must have a single λ if it does not have a single λ then you will have a considerable variation in the $\sin \theta$ as well. So, your diffraction phenomena may not be able to capture this diffraction.

I mean the event may happen over a long $\sin \theta$ range you will have a broadening if you consider about x-ray you will have a big broadening or even we have seen because of the finite thickness because of this that we have a finite broadening in the diffraction spots as well and to some extent that is related to this variation in λ as well and this is more so

in case of x-ray when we discuss about x-ray we will discuss about this broadening due to the problem with the lambda more.

But this is what why you tend to have a signal which is elastically scattered you tend to use it for diffraction experiments and the backscattered electrons in scanning electron microscopy the only signal which is generated by this elastic interaction is the back scattered electron. So, that is why the backscattered electrons are the only one that can use for diffraction purpose and one such method is called electron backscattered diffraction which we will possibly discuss in this course.

And there you can generate this kind of diffraction pattern. So, this is not really a diffraction pattern which we have seen in electron diffraction spot pattern and all those, but there if you remember I have shown one example of something called Kikuchi pattern. So, there the Kikuchi pattern was generated by transmission electron microscope, here this Kikuchi pattern is generated in scanning electron microscope by the backscattered electron beam basically not the primary beam.

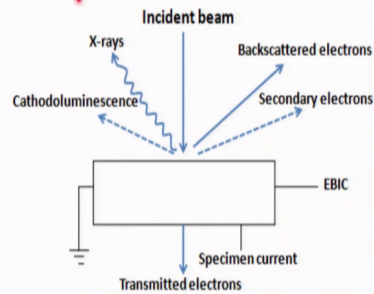
The backscattered electron which is generated after the primary beam hits the specimen those backscattered electrons basically they are able to come out when they are within the specimen itself they causes again a diffraction and through that they generate this Kikuchi pattern so we will discuss more about this. Just remember the backscattered electrons are much more they have they can play multiple role and that is how they are different from secondary electrons as well.

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Obtaining a signal in SEM

• Due to the interaction of electrons with a thick specimen, the energy of the incident electrons is dissipated resulting in

- Various secondary emissions from the specimen (cathodoluminescence, characteristics X-ray, Auger electrons etc.)
- Emission of electron as a result of inelastic scattering (secondary electrons)
- Some of the elastically scattered electrons is backscattered out of the specimen



And now obtaining a signal in SEM so what are the different kind of signals that we can obtain in an SEM from this discussion that we have and due to the interaction of specimen in the thick specimen. So, we are now discussing about a thick specimen no longer in a thin specimen like what we used in electron transparent specimen in TEM. We have seen that in TEM and that is something that we have discussed in TEM you only have the direct beam or diffracted beam.

Whatever change happens in the direct beam or the diffracted beam we can use it for imaging or electron diffraction or whatever. In case of SEM it is much more versatile because you have multiple sources of signals in SEM. So, in this case if you are having an electron opaque specimen the energy of your incident electron the primary electron now can be dissipated and it can produce various types of signals which are very useful for either imaging or doing diffraction experiments or even for identifying the chemical nature of your specimen.

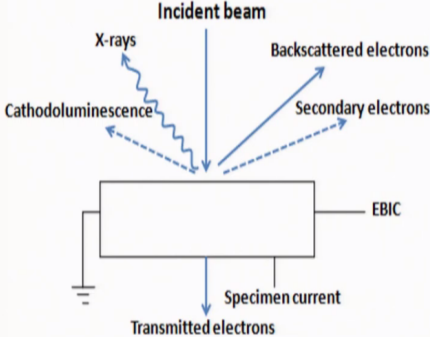
So, all of these you can do because you have so many different type of signals produced in case of an SEM. So, for example, you have various secondary emissions from the specimen like cathodoluminescence, characteristics x-ray, Auger electrons etcetera which are mostly used for identifying the chemical nature of your specimen then you have emission of electron as the result of inelastic scattering.

That means the secondary electrons which we mostly use for imaging purpose and finally you have the elastically scattered electrons backscattered electrons which are used for diffraction, imaging chemical identity all three. So, this is why SEM is so versatile.

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Obtaining a signal in SEM

- One of the main features of the SEM is that any radiation from the specimen or any measurable change in the specimen may be used to provide the signal to modulate the CRT/digital display and thus can provide contrast in the image.
- Each signal is the result of some particular interaction between the incident electrons and the specimen and may provide different information about the specimen.



The diagram illustrates the interaction of an incident electron beam with a specimen. The incident beam enters from the top and interacts with the specimen, which is connected to ground. This interaction produces several signals: X-rays, Cathodoluminescence, Backscattered electrons, Secondary electrons, and Transmitted electrons. The specimen current is also shown. An EBIC (Electron Beam Induced Current) detector is connected to the specimen to measure the specimen current.

And one of the main feature in SEM that the radiation as I already said the radiation needs to come out of the specimen then only they can be detected and once they comes out from the specimen then any change in the specimen that happens that signal any change on the signal in the scanning mode. We have already discussed how the scanning mode happens. So, in the scanning mode whatever the signal comes out any change in the signal from point-to-point different pixels according to that we can modulate the CRT or digital display or image capturing device whatever it is.

We can modulate an equivalent pixel there and that is how we can provide the contrast we can generate the contrast in the final image. So in each signal here is a result of some particular interaction between the primary electron and the specimen and it can provide different, different information about the specimen what we already discussed about backscattered electron.

Various different type of interaction is possible and various different type of information can be obtained by using one single signal itself. So, with that we end it here.