

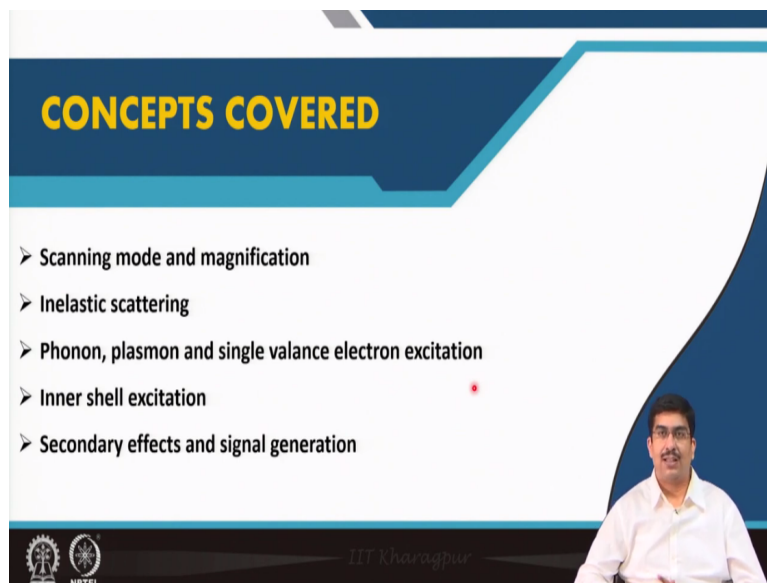
**Techniques of Material Characterization**  
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**Lecture – 27**  
**Signal generation in SEM Continued**

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are now in module 6 that is week 6 and we are discussing about scanning electron microscopy and the topic for this lecture is again signal generation in SEM which we were discussing in the last lecture. We will continue with that and in the last class we basically discussed about the image formation in scanning electron microscope how it happens.

And majorly like what is the difference between we tried to understand what is the difference between the transmission electron microscope and the scanning electron microscope and from there were seeing the basic configuration of scanning electron microscope how it differs from TEM and how actually the history of scanning electron microscope and how the working principle, how it basically works in this scanning electron microscope. So, we will continue that discussion.

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And today we will be discussing about scanning mode and magnification how these two things are happened in case of scanning electron microscope and then we will discuss about

the signal generation in SEM and most of the signals in SEM generates by inelastic scattering so we will generate that and discuss that and we will discuss some of the signals that is generated.

Some signals which we normally do not use in detection, but still better to know how they generates like phonon scattering, Plasmon scattering, single valencevalence electron excitation and also and then we will discuss about another type of signal generation mechanism that is called inner shell excitation and some secondary effects and signal generation we will discuss.

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**How scanning mode works?**

- In older instruments, the fine beam of electrons is scanned across the specimen by the scan coils, while a detector counts the number of low energy secondary electrons, or other radiation, given off from each point on the surface.
- At the same time, the spot of a cathode ray tube (CRT) is scanned across the screen while the brightness of the spot is modulated by the amplified current from the detector.
- The electron beam and the CRT spot are both scanned in a similar way, that is, in a rectangular set of straight lines known as a raster.
- In modern instruments, the same effect is achieved by digitally controlling the beam position on the sample and the resultant image is displayed on a digital display.

The slide contains two diagrams. The top diagram, 'Raster on CRT', shows a grid with two points labeled 'A' and 'B'. Below it, a horizontal double-headed arrow labeled 'L' indicates the scan direction. The bottom diagram, 'Raster on specimen', shows a similar grid with points 'A' and 'B', and a horizontal double-headed arrow labeled 'L' below it. A small video inset of a man is in the bottom right corner.

So, first of all how the scanning mode occurs as the name suggest basically scanning electron microscope is all about scanning the beam is produced. So, in the last class we have seen how the beam is produced from a thermionic gun or field emission gun whatever you have and then you have this condenser lenses and those condenser lenses through that and using some aperture.

You make the beam, you de-magnify the beam basically de-magnify the beam means whatever you get in the cross over you even make it smaller. So, the beam diameter finally you get it focused to a very fine beam around 2 to 10 nanometer and then what you do is that you make we are continuously saying you make the beam to raster, you make the beam to scan.

How do you do that basically and how it is related to magnification because in the scanning electron microscope you really do not have the objective lens and then you have the projector lenses and so on, so magnify the image of the first objective lens whatever you get like whatever you do in regular microscope, optical microscope or the same theory you apply for transmission electron microscope where you have this objective lens and then objective lens basically forms the image.

Then you have the projector lens the projector lens further magnifies. So, you have this magnification in various stages and so on so that is a real kind of magnification. In scanning mode it is not like that in scanning mode the magnification is achieved in a different way. So, what do you do is that basically you make a beam here, you make a beam to move along a line and that is called a raster.

So, in this rastering what we mean is that raster is that you have this rectangle set of straight lines in this scanning pattern the way the scan happen basically the beam is moved to go along one line first and then it comes down to the next line it moves like this and then if you ever seen an SEM and you try to reduce the frame rate (04:13) and then you can find out that how exactly the scanning happens.

And to draw an analogy exactly the same way the scanning happens in a TV tube. In television if you see the television also is like a scanning mode or any kind of digital display for that matter not only television any kind of digital display which works in a pixel by pixel which works in a scanning mode and this is how the scanning happens over a line where it moves from this end to this end.

From next line it moves from this end to this end again in the next line it moves from this one to this one. So, this is how the entire scanning and rastering happens and this in case of scanning electron microscope this happens physically. So, the electron beam is made to the scan coils, made to raster the beam or made to travel the beam like this. So, every such line contains lots of pixels we will come to this specific example also.

So, you have pixels many number of pixels within one line then again in the next line you have many such number of pixel you come to the next line and so on and so forth and usually this is done in kind of rectangular set of straight lines or you can have a square also it depends. So, you can have like 1085 to 1086 pixels that means that is a square grid and similarly you can like have 860 pixels into 860 into 650 or something if that is the pixel number of pixel that means it is a rectangular grid.

So it depends on what number of pixels you have finally, but ultimately but the movement is of the beam is like this only. Now what happens is that when a beam is falling on any certain point in the specimen this is happening in a real specimen. So, when you have a beam falling on this spot on a specimen it generates a signal whatever is the signal it can be secondary electron, it can be backscattered electron, it can be x-ray whatever it generates the signal.

That signal you collect using a detector basically most of the time the detector counts the number of that electron if it is an electron the number of such electron if it is an x-ray signal the x-ray quanta, number of x-ray photons and so on and so forth and then it generates it transforms that to an equivalent current signal and all. So, finally what you get is a signal with a strength with an intensity.

So, two, three things you know from the signal. Number one the x and y coordinate of the signal that exactly from which position it is generated that signal is generated and what is the strength of the signal? Strength can be in terms of number of electrons, number of x-ray photon, but whatever but you know the strength of the signal. You use that information to modulate a similar raster on your digital display.

So, earlier days this used to be done in a CRT cathode ray tube, these days it is done on some kind of digital display if you have an LCD screen or whatever you have some kind of digital display whatever basically principle is the same. So, you have a raster now in the specimen where the beam travels and you have a raster on your CRT display or on digital display you have a raster equivalent raster.

And this pixel the size of the raster is also not exactly same physically not same, but number of pixels is exactly the same. So, whatever number of pixels is here exactly the same number of pixels are there in this. So these two are related basically through the number of pixels and correspondingly the size of the pixel also varies, but that is okay. Now corresponding to the most important point is corresponding to any pixel here with an x and y.

You have a corresponding pixel here with an x and y this is related. For every pixel here you have an equivalent pixel in this digital display. What you do is that you use the signal strength from this specimen raster you use that to modulate this CRT display or digital display. Basically if it is grayscale image you decide on a grayscale value. You calibrate this if this is the signal strength this many number of electrons is there or this is the current that is generated finally in a detector.

Then that corresponds to this grayscale value if it is a 8 bit information then it will be 0 to 255 grayscale values and so on, but you will assign a number grayscale number to this point here display point here. So, finally then that means what you get an x and y coordinate plus a signal strength from the specimen raster and you use that information to modulate or equivalent pixel with an equivalent x and y.

You assign a grayscale value to that pixel and that is what you do. You just basically continue this process for every such pixel. So, as the beam is moving over the specimen creating a signal you mention the signal strength you have an equivalent point on your digital display or CRT you modulate that with a grayscale value and depending on the contrast formation mechanism the signal strength will vary between point to point.

And then correspondingly you should see a contrast generation in this raster on the display and that is your image basically. Finally, when you capture this image on the display or on the CRT tube that is what exactly will replicate your specimen features here that is how the scanning mode basically works and that is what the relationship between this digital display finally the display that you are seeing and your specimen that is why it is a scanning electron mode, basically modern days everything is software controlled.

You do not have a CRT any longer and here just automatically when you see this image you know or when you try to capture you just click on the software what it does the software does is that it ask the beam, it ask the scan coil to make the beam raster over the specimen following this grid and correspondingly the digital display in the display or in the capturing finally in the image you just basically software just uses that signal and assign a grayscale value that is all. It was the scanning mode.

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### How magnification is achieved?

- The raster scanned by the electron beam on the specimen is smaller than the raster displayed on the CRT/digital display.
- The linear magnification is the side length of the CRT/digital display ( $L$ ) divided by the side length ( $l$ ) of the raster on the specimen.
- For example, if the electron beam is made to scan a raster  $10\ \mu\text{m} \times 10\ \mu\text{m}$  on the specimen, and the image is displayed on a CRT screen or digital display of  $100\ \text{mm} \times 100\ \text{mm}$ , the linear magnification will be  $10000\times$ .

Now, how the magnification is achieved by this scanning mode. Basically as I said the raster on the specimen and the raster on CRT are equivalent. So, for every pixel here you have an equivalent pixel here the difference is between the size. The size of this raster in the specimen is much smaller than the size of this raster on your display. So, the pixels also in the specimen the pixels are much smaller than the pixels corresponding pixels in the display.

So, basically what happens is that you have a relationship linear magnification happens between this specimen, grid, the length of the raster of the grid in the specimen and the length of the raster in this display. You have a direct relationship a linear relationship between them and if it is a square grid then you can extend this in the y direction also if it not a square if it is rectangular grid then you will have an equivalent something like a equivalent relationship between this width direction as well.

So, the final magnification that you get is basically this length of this grid on your display CRT or digital display the length of this grid, this raster here divided by the length of this raster on the specimen. So, basically it is  $L / \text{small } l$  and that gives you the amount of magnification. So, that is why I said that unlike the transmission electron microscope here there is no such length involved in the magnification in between.

It is not a direct physical, magnification of the electron beam. It is done here by this scanning mode and through this detector and finally on the display or whatever your image capturing device you have, if you have a image capturing device let us say instead of display you have a camera exactly same things happens in a camera you have an equivalent point. In the camera you have the same raster and you have an equivalent pixel there.

And this is how the magnification is achieved in case of a transmission electron microscope. So, now if we take a particular example it will become very clear then. So, let us say you have this raster on the specimen which is 10 microns by 10 microns so it is a square grid and here you have this kind of a grid and here you have this side it is let us say 10 microns this side it is 10 microns.

Now your image and remember one thing this image rastering is basically pretty much constant. So, this size is very, very constant. Let us say that size is 100 millimeter / 100 millimeter you have a CCD camera and that CCD camera the size of that CCD camera let us say the detector is 100 millimeter / 100 millimeter. So, what you will then achieve is that linear magnification will be this by this.

And finally you will see that this will come around 10,000 x magnification. So, that is the linear relationship. Now in a real sense if you want to change the magnification basically here this one you cannot change basically this is fixed your final camera is fixed basically what you change is that you change it here and this image. So, here you change the size of this specimen rastering.

This you change if you want to magnify basically the size here will go down. So, if you want to zoom it another if you reduce it another 100 times if you reduce it to 1 micron / 1 micron

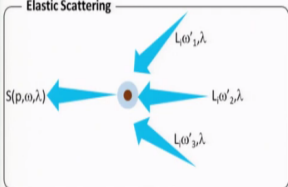
you still have this specimen raster, you reduce the specimen raster to one micron / one micron and you still have the display or the image capturing device you still have the raster as 100 millimeter / 100 millimeter you will have a linear magnification of 100,000 times.

So, that is how this magnification is achieved here and that is how this scanning this raster are related between in the real specimen and in your image capturing device. So, this is the relationship and this is how the magnification or this is how the scanning mode basically works, the scanning mode of image capture basically works and this is how the magnification is achieved finally.

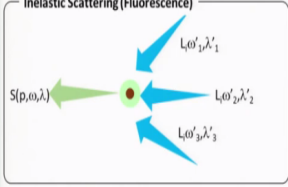
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### Signal generation in SEM

**Elastic Scattering**



**Inelastic Scattering (Fluorescence)**



- Inelastic scattering is a very general term which refers to any process which causes the primary electron to lose a detectable amount of energy.
- In electron microscopy,  $\Delta E$  would need to be substantially more than 0.1 eV before it could be detected.
- There are many interaction processes which could cause energy to be lost by the primary electron and transferred to the electrons or atoms of the specimen.
- Only four of the most probable types of scattering event should be considered in SEM.

Now we are coming to the signal generation in scanning electron microscope. As I said most of the signals unlike transmission electron microscope where the imaging most of the imaging modes there the signals are mostly generated by elastic interaction or elastic scattering plus you have the diffraction also and diffraction is again only responsible, diffraction is caused by electrons which are undergoing elastic scattering.

The entire diffraction phenomena is basically an elastic scattering there. The imaging if you consider bright field image or dark field image whatever electron you are capturing is basically the elastically scattered electrons. The in-elastically scattered electrons you do not really consider there unless of course the couple of special modes like analytical mode if you want to go for a electron energy loss spectroscopy or something that mode.



Then you can possibly use those inelastically scattered electron. Otherwise, in transmission electron microscopy all the signals that you use either imaging or for electron diffraction these are basically the elastically scattered electrons, but contrary to that in scanning electron microscope most of the electron or most of the signals that you use either for imaging or for analytical studies are generated by this inelastic scattering.

An in elastic scattering as we discuss that time also inelastic scattering basically a process in which the primary electron or primary electron beam losses some amount of energy by any process. We will see what are those process, but there should be some amount of loss of energy associated in the process.

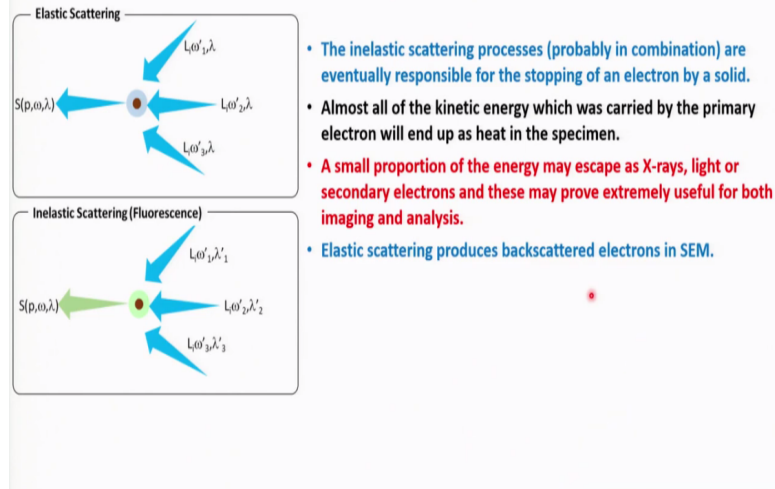
Now with a present current capabilities we cannot detect any such event, any such inelastic scattering event if the change in energy associated with that event is less than around 0.1 electron volt. If it is lower than that our current abilities will not allow for any such event to be detected that is our capability at this moment and that is also the restriction that we have in order to find out or in order to under this inelastic scattering mechanisms.

And there are many such inelastic scattering interaction processes which could cause energy to be lost from the primary electron to the other kind of electron signals that is generated or to the atoms of the specimens out of all of these various ways the inelastic scattering can happen and signal can be generated only four of most probable types of scattering event is considered in SEM again.

That is our limitations our capabilities at this moment that only few of this in-elastically scattered signals that is generated by inelastic scattering we can use it for either for imaging or for analytical studies chemical identification of any specimen we can still now use. So, we will understand those.

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## Signal generation in SEM



And again the inelastic scattering process mostly happens in combination. So, it is not a pure inelastic scattering. If the electron undergoes that is the primary beam we have seen that when we are discussing about the interaction volume then at that time we saw that the primary beam, primary electrons undergoes many number of inelastic scattering and those many different number of inelastic scattering basically cannot be one single type.

There can be multiple type of inelastic scattering happening over the path or over the trajectory of the electrons and finally out of this inelastic scattering basically primary electrons are stopped within the electron microscope, within the specimen because it is the thick specimen ultimately primary electron beam will lose all its energy due to various type of inelastic scattering and it will stop there.

And most of the kinetic energy which was carried by the primary electron, primary accelerated electron that will end up heating the specimen we will see that and only a very small proportion of energy from this primary electron is responsible for signal generation whatever signal, x-ray signal, light signal, electron signal only a very small fraction of the primary beam electron energy is responsible for signal generation.

Most of the energy is finally lost by heating the specimen that is why it is a severe problem that entire specimen needs to be cooled down. The entire specimen column needs a very high

amount of vacuum not only to stop the electrons from interacting with the air if there is air we already discussed if air is there electron will interact with them.

And we will never reach to the specimen but also because the specimen if it is carbonaceous material on the surface of the specimen you may have some carbonaceous material all of this will burn because of this large amount of heat generation. Of course, just like in transmission electron microscope we have one signal which is generated by inelastic scattering and that carries some useful information about the chemical identity.

We can have in the scanning electron microscopes we have one such signal which is generated by elastic scattering and which is very useful that is called the backscattered electrons and backscattered electron we already discussed how it is generated we will again come to that, but just remember that in scanning electron microscope the only signal generated by elastic scattering is the backscattered electrons.

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**Phonon scattering**

- Phonons are the quanta of elastic waves, that is caused by the atomic vibrations in a solid.
- The amount of energy lost in phonon scattering is rather small, generally  $<1$  eV, and the mean free path for high energy electrons is quite large, of the order of  $\mu\text{m}$ .
- Phonon scattering is important for two main reasons:
  - All electrons which remain in the solid are likely to excite phonons eventually, and this is how the solid is heated by the electron beam.
  - Also, when phonon scattering occurs, the scattered electron is generally deflected through quite a large angle, typically  $\sim 10^\circ$ . This is important especially for secondary electron generation.

So, the first type of inelastic scattering the signal that is generated is phonon scattering. Now the phonons are the quanta of elastic waves and caused mostly by the atomic vibration in a solid. So, we know that in a solid so atoms vibrate about their mean position and that generates basically one type of elastic wave which is known as the phonons and what happens is that when a primary electron beam basically heats this atom it can increase or decreased.

It depends on what basically the kind of nature or exact nature of the inelastic scattering, but ultimately it will change this atomic vibration of atoms and in the process it will generate some elastic waves in the form of phonons. So, usually these phonons are the possibility or the amount of energy that is lost in the phonon scattering is quite small and generally it is less than one electron volt.

And the mean free path for high energy electron is also quite large of the order of micron. So, mean free path we have already discussed that it is basically the probability or it is the distance minimum distance between any two interaction events and in this case that interaction event is phonon scattering. So, between any two such phonon scattering events their mean free path is quite large basically in micron range.

So, the probability of such phonon scattering events is also quite low and energy lost in the phonon scattering process is also quite low. So, what happens is that is why this phonons that is generated cannot be usually used as a signal of detection because already we have discussed that our current limitation is in terms of energy we cannot detect anything which is less than 0.1 electron volt.

And any event any inelastic scattering event if it is less than 0.1 electron volt we cannot detect and the phonon scattering is a typically very low energy process. The energy involved in phonon scattering is very, very low, mean free path that means the occurrence of such changes is also very low, but still phonon scattering is pretty important in case of scanning electron microscopes to consider for electron microscopy in general.

And in particular in scanning electron microscopy is because all that electrons ultimately all the electrons remains in the primary electron beam which are entering in the specimen in the electron opaque specimen. There finally all of them lose their energy through phonon scattering. So, what happens is basically when all other processes when the primary electron loses most of their energy through all other inelastic scattering processes.

Then the final amount of whatever little amount of energy the primary electron beam is having that is used for phonon scattering and that number is very high. So, all the primary electron if they end up giving most of their final amount of energy in phonon scattering so you can imagine that number is really, really large and that will cause a lot of heating in the specimen. So, that is the first importance of phonon scattering to consider.

Second importance in phonon scattering is that the scattered electrons. So, after the phonon scattering the primary electron beam the amount of scattering that happens or amount of deviation from its path that happens is almost of the order of 10 degrees. So, that is really huge amount of deflection through this phonon scattering. So, that is why the phonon scattering is also important for secondary electron generation.

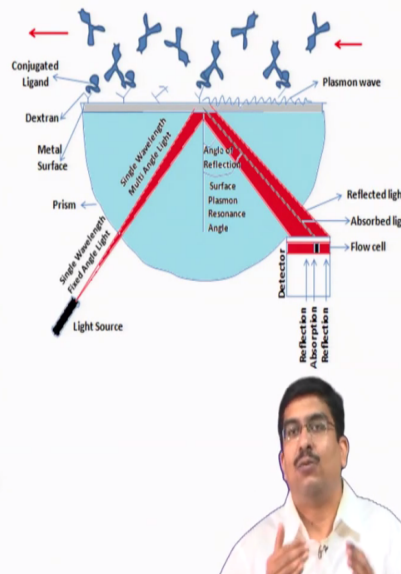
So, when we discussed about the secondary electron generation we will realize that what is the secondary electrons are basically primary electrons which are almost at the end of their life or almost end of their energy. So, they have a very minimal amount of energy left the primary electrons and this phonon scattering can cause them to come out of the specimen because the amount of deflection that this phonon scattering does is very high.

So, the secondary electrons may come out of the specimen and that is how they can be detected. So, that is why phonon scattering is important to consider in case of scanning electron microscope.

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## Plasmon scattering

- A plasmon is a wave in the 'sea' of electrons in the conduction band of a metal.
- There are similar effects among the bonding electrons of non-metals.
- In exciting a plasmon the primary beam loses 5-30 eV of energy.
- The mean free path for this event is around few hundred nm in most materials.
- Consequently, plasmon scattering is a frequent occurrence in all electron-solid interactions.
- Energy losses by plasmon scattering dominate the 'energy loss spectrum' but are not very useful for analysis because the energy loss is not particularly characteristic of the scattering element.



Now the next type of interaction possible is called the Plasmon scattering. So, Plasmon are also elastic waves so Plasmon are just like phonons, but now this Plasmon basically we can imagine the conduction band if it is a metal we already understood that metal we can imagine a band structure there are valence band and there are conduction bands and then the difference between the valence band and conduction band is very less for a metal, for a non metal for a semi conductors and all so our insulators that is quite large.

Insulator is very large semi conductor is not so large and so on, but for a metal it is conduction band and valence band are almost very close together. So, if we consider a metal of that conduction having lots of free energy or free electrons in the conduction band then if a primary electron beam hits that conduction band it generates kind of a elastic wave in the sea of electron.

A very similar to something like if you throw a stone on a lake and the kind of waves that is generated from the impact (26:08). So, similar kind of things if in primary electron beam heats this sea of electrons free electrons in a conduction band and it will generate some kind of a wave in the form and that is called the Plasmon scattering. So, this is similar kind of effect also can generate for non-metals where this effect will be generated for bonding electrons where there is no free electrons or no metallic bonding and all.

So there it is similar kind of effect can be generated in case of a non-metal and this process in the Plasmon scattering process the energy lost is quite high almost in the order 5 to 30 electron volt from the primary electron volt. So,  $\Delta$  is very, very high in case of a Plasmon scattering also the mean free path of Plasmon scattering is very small as compared to the phonon scattering it is around nanometer range in most of the material.

So, Plasmon scattering actually can be used as a signal inelastic scattering. So, whatever electron is losing energy primary electrons are losing energy through Plasmon scattering and all this inelastic wave this waves that is generated if we can somehow detect them we can possibly use it in SEM. Difficulty is that the energy losses by Plasmon scattering is not useful we can use it for chemical identity.

Basically Plasmon scattering is very, very related we can relate it possibly to the chemical identity because we can differentiate between a metal, non-metal, it is in the conduction band. So, we can possibly if we detect the Plasmon waves we can possibly use it to understand the chemical nature of certain element present in the specimen. The problem is the Plasmon scattering the kind of when it generates from the specimen it is not or the energy loss which is there what we can measure is basically the energy loss in the primary electron beam.

So, that energy loss in the primary electron beam is not a typical characteristics of the scattering elements. So, the element which is causing this Plasmon or the primary electron beam whatever element it hits the amount of energy that lost is not a very characteristics of that element. So, the same amount of energy can be lost if you have say aluminum versus same amount of energy if you have let us say cooper.

So, Plasmon if you just check up to the Plasmon scattering event if you take this primary beam and if you measure its energy before the Plasmon scattering and after the Plasmon scattering that amount of energy lost you cannot directly correlate with the element which is present so that is the problem with the Plasmon scattering. It is not a very true characteristics of the element which is present.


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## Single valence electron excitation

- It is possible (but less likely), that a primary electron will transfer energy to a single valence electron rather than to the 'sea' (in the conduction band) collectively.
- The mean free path for this process is quite large (in micron range), the energy loss is small (~ 1 eV) and the typical scattering angle is also small.
- The process is not useful in electron microscopy.

Single-photon absorption  
(High-energy photon)

Multi-photon absorption  
(High-density photon)



And one more effect we will discuss here that is called the single valence electron excitation and here what happens in this case it is not in the conduction band, but in the valence band the primary electron beam may also transfer its energy to a single valence electron and then can make it move to the higher energy state maybe in the conduction band it can do that. It is very less likely to happen it is not frequent as frequent as Plasmon scattering the single valence electron excitation.

Obviously, the mean free path for this process is also quite large since its occurrence is not that frequent like Plasmon scattering. So, mean free path is very large for this. The energy that is lost in this process the single valence electron excitation. So, basically primary electron beam comes and it hits instead of a Plasmon scattering in the conduction band. It comes and excites an electron in the valence band.

And that valence band electron now moves to the higher energy state maybe in the conduction band also. So, similar to what it is shown here the different virtual states are there this is a quantum mechanical process and this also can happen, but and this is typically happens or typically happens for non-metals where the conduction band there is a considerable difference between valence band and conduction band for those kind of material single valence electron excitation is also possibility.



But the problem again inelastic scattering possibility is one side, but detection ultimately whether we can detect that effect or not and whether we can get some information out of that effect or not that is one question and that those kind of questions whether we can detect certain events, whether we can detect certain signals that is generated through that event and we can utilize it for imaging or for understanding the nature of the chemical identify of the element that is causing that event.

Whether we can detect that or not that is what decides whether we can able to use that event for a characterization technique. So, on that aspect none of these events like phonon scattering, Plasmon scattering or even single valence electron scattering are at this moment are useful for different, different reasons as I described phonon scattering, single valence electron scattering very low amount of energy change is involved and they have usually very high mean free path.

So, the number of such events is also very, very less, Plasmon scattering high energy change is involved and mean free path also is small that is fine, but it does not carry a true characteristics or does not reveal the true characteristics of the element from which this inelastic scattering event is happening. So, these are the problems with these three kinds of inelastic scattering.

And then now we will discuss about other kind of inelastic scattering which possibly can be used for a signal generation in case of a scanning electron microscope, but that we will discuss in the next class. So for now good bye.