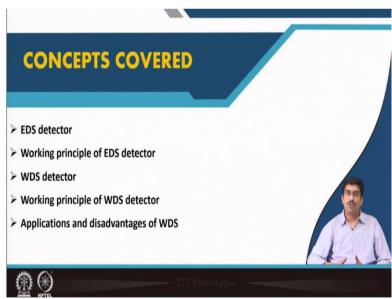
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Lecture – 36 Analytical Detectors in SEM

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. So, we just starting the module 8 we are still discussing about scanning electron microscopy and in the last module rather we had started discussing about various type of detectors, secondary electron detector, backscattered electron detectors and we also discussed about optics in SEM.

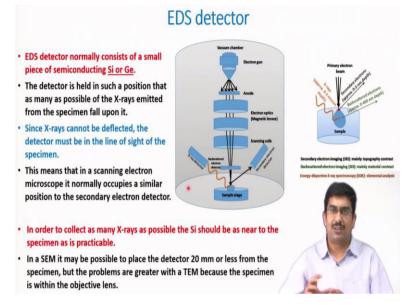
How resolution minimum probe diameter, beam current how those things are related and how to calculate ultimate resolution, how it is related to the pixel size, sampling volume so on and so forth all those things we discussed and we just started in the last lecture about the chemical analysis in scanning electron microscope and we will continue this in this lecture. We will discuss about various analytical detectors in SEM.

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And we will cover these issues we just said that there are two different type of detectors for this chemical analysis usually. One is called energy dispersive spectroscopy detectors EDS detector. So, we will discuss about EDS detector and its working principle then we will move to another type of detector for chemical analysis that is wavelength dispersive spectroscopy detector or WDS detector and we will discuss about that working principle of WDS detector and the application and disadvantage, basically we will compare these two detectors.

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So, EDS detector or energy dispersive spectroscopy detector. This normally consists of semiconducing silicon or germanium a very small piece that is what the heart of the detector basically. So, this semiconductor silicon or germanium plates that is what is basically detects the x-ray photons and how it is done is that we will come to that, but the detector is basically held at a position so that it can detect or it can capture as many x-rays that is emitted from the specimen as possible.

So, if this is the specimen let us imagine and the beam is falling over it and we already discuss that the x-ray beam or the interaction volume of the x-ray signals are coming within from very deep and sampling volume is also quite large. So, x-rays will generate through all over this place where the beam is falling and in order that the detector could capture maximum portion of this x-ray signal which is generated they should like placed in such a way that most of the x-ray detectors finally could reach them.

Now this is the same condition for any kind of detectors when we discussed about say secondary electron detectors or the backscattered electron detectors we were discussing about the same thing that detector should be placed in a way that maximum it could achieve or it could capture maximum amount of signal here. For secondary electron detector we have seen that ET detector when we were using an ET detector we were having this metal cage which

was basically kind of capturing all the secondary electrons which was generated that was not quite useful for backscattered electron detectors because of these two things.

Number one backscattered electrons of a high energy and they have a directional nature, the way they are produced. So, that is why in order to improve the capture or the amount of backscattered electrons to be captured in order to increase this backscattered electrons detectors were placed in and around the beam; same principle here for x-ray detectors. The problem with the x-ray signals is that it cannot be deflected so easily.

Like there cannot be any kind of metal cage or something or there cannot be any lens or something which can work on the x-ray signal and make all the x-ray beams to fall on the detector and of course the detector has a finite size or the detector has a capturing amount of signal it can capture over a solid cone that is finite. So, what happens is that we try to keep the detector with the line of sight of the specimen where we think that the x-ray signals have a maximum possibility to generate.

So, this means in a scanning electron microscope basically this x-ray detectors are placed at an angle with the specimen in such a way that they could capture the maximum amount of xray signal and usually they occupies a similar position to the secondary electron detector usually in the two opposite direction, but with a same kind of an angle with the specimen and that is how the detector ensures that they are in their line of sight maximum x-ray signal could be captured that is first thing.

The second thing is that in order to collect as many x-rays as possible again for the same purpose the silicon, the detector itself the one piece which will ultimately detect the number of x-ray photons or determine their character. So, this semiconductor silicon, piece of silicon should be placed near to the specimen as near to the specimen as applicable. So, two requirements the detector should be placed in a way that it could capture maximum x-ray signal that line of sight or the collection angle the semi angle or the solid angle of collection should be increased. So, detector should be placed in a way like this.

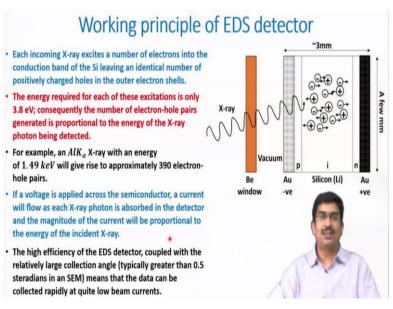
And also the silicon detector that should be placed as closed to the specimen as possible, but of course there are some practical limitations to this because you cannot come too close to the primary beam that the primary beam will start hitting the detector producing other kinds of SE signals and all sorts of problems, damage and all will start happening. So, usually in a SEM this detectors are placed at a distance of around 20 millimeter or slightly less than that.

But in TEM specimen the problem is even bigger because in SEM the beam is not such strong and the beam is already in focus. The TEM the beam is much stronger because the accelerating voltage is much higher so for that also the detector should be placed at a slightly far off distance and second thing is that the specimen in TEM is most often within the objective lens, within the magnetic or it is just like kept over the objective lens.

So, if x-ray detector is placed there so it will come very close to the objective lenses as well and it will be affected. So, the objective lens the way the detectors work the silicon their entire working principle of the detector will be altered because of the magnetic lenses, electromagnetic lenses which are placed just beneath the specimen. So, that is why in a transmission electron microscope the detection of this EDS signals are very, very difficult.

Placing the detector itself in the detector within the microscope column is very, very difficult there.

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So, let us now see the working principle of EDS detector. So, the EDS detector is basically as I said this is a semiconductor silicon or germanium material and this is usually an NIP type of semiconductors it is a circuit. So, these kind of semiconductor is used here. Now each incoming x-ray signal what it does is that basically it creates holes in the conduction band of the silicons because it is a semiconductor in nature.

So, the energy required for this kind of excitation of holes and all is very very small only 3.8 electron volt and that is fixed that is the decided by the Fermi level of semiconductor silicon. So, this energy required for creating one pair of hole electron hole pair is very much fixed for silicon and x-ray photons does exactly like that each of these x-ray is coming incoming x-rays photons basically hits the silicon detector here semiconductor silicon detector here.

And this produce an identical number positively charged holes in the outer electron shells in the conduction band basically. Now, since energy is fixed what we can understand from this the number of electron hole pairs that is generated by this process x-ray photons coming and hitting this electron and creating the hole in the conduction band. So, this electron hole pair and in the process of creation of a hole in electron also is generated.

So, this is an electron hole pair. So, the number of such electron hole pairs since the creation energy is fixed and if we know the energy of the x-ray photon or in other way round if we can capture or if we can somehow measure this electron hole pair the number of electron hole pairs that is created by this x-ray photon if we can measure it somehow what we can understand is that what was the energy of the incoming x-ray signal because that is what is providing this phenomena that is what that energy is transferred to that electron hole pair in creation of the electron hole pairs.

So, that is why in the entire detector we can find out the energy of this incoming x-ray signal and from that what we can get is that we can identify from which element this x-ray signal is coming because every element the way this characteristic x-ray are generated we have discussed about this the secondary effects and inner shell excitation and so on. So, this is related to the atomic structure of the element.

The characteristics x-ray generation and the characteristics x-ray has its particular wavelength and energy, it is fixed for every such elements. So, this way from the number of electron hole pairs that is generated we are basically finding out the x-ray signals, the energy of the x-ray signals from there we find out that from which element this characteristic x-ray is coming that is simple as simple as this. So, if we take an example like for example like aluminum K alpha signals so aluminum K alpha signal x-ray signal that has an particular energy of 1.49 kilo electron volt. This will give rise to approximately 319 electron hole pairs if you multiple this 390 with this 3.8 electron volt that is the energy required to generate one electron hole pair in case of this silicon detector.

So, we can find out that this is exactly equal to this energy of this aluminum K alpha. So, if we have from this entire circuit if we have 390 electron hole pairs when an x-ray photon comes and hits it we know it is aluminum K alpha that is it, but the point is how we are going to measure this number. For that what we can do is that we can apply a bias in this circuit this entire circuit we can apply a voltage across the semiconductor.

And because of this voltage because of this bias there will be a current flow the energy and electron and hole pair. Electrons will flow towards the positive side and holes will be flowing towards the negative side we will affectively get a current flowing through this semiconductor in the entire circuit we will be getting a current. From that current we can find out the number of electron hole pair or if you properly calibrate finally we will get the x-ray photon the energy of this x-ray photon.

And basically what we will finally get is that the current that is flowing because of this x-ray photon coming and hitting the semiconductor that will be proportional to the energy of the incident x-ray in the entire process. So, this is how the x-ray photon their energy is measured and from there the nature of this incoming x-ray signals it is assigned from which element it is coming in the specimen.

And remember one thing the beauty of this entire process that is what I always tell to my students is that this kind of chemical analysis you can do it with any kind of spectroscopy. If you take atomic absorption spectroscope if you take any other AES, any other emission spectroscopy anything you take. You will get the chemical identity you can identify the chemical nature of this specimen or the element which are present in a specimen.

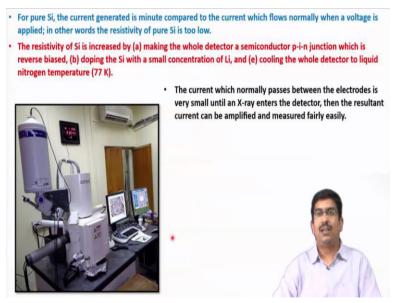
You can also measure the quantity of that very properly and in fact sometimes even better than an SEM. What you all will be missing is the special resolution that is in case of a scanning or in case of a microscope it will give you exactly where that element is in the microstructure. If you have a structure and if you have somewhere some precipitate let us imagine you will be able to pinpoint that this element is here.

This you cannot get it from any kind of spectroscope, that is why the measurement of elements or to identify the chemical nature of the elements in a scanning or in a electron microscope it is so important and it is so popular. You are getting the position as well along with the chemical identity or even the quantity you are getting their position in the microstructure so that is very very important.

So, this EDS detector usually they are of very high efficiency and their collection angle because of all these arrangements that line of sight EDS detector is placed with a maximum with a very high solid angle of collection and their silicon detector is placed very close to the specimen. So, with all of these adjustment the efficiency of EDS detectors is very very high and the collection angle is very high typically it is greater than 0.5 steradians in SEM.

The data is collected quite rapidly even with a low beam current and low beam current remember that means the lower probe size. So, with a good resolution quite a good resolution the EDS detector can obtain a signal at a much rapid rate so that is not a problem there.

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Now couple of other issues about this detector itself. So, for pure silicon the point is the current that is generated because of this process x-ray photon is creating a hole in the conduction band and so on and so forth. And then there is a bias applied and a current is

flowing. This current is quite small because if you can understand there that for one x-ray photon of aluminum K alpha there is hardly 400 electron hole pair is generated.

And in the first place the number of x-ray photons which are generated from the specimen is quite low for that you have to increase again the beam current and then the beam diameter will increase, special resolution will go down and all of these things and then the rapid collection was a problem so all sorts of problem will start in. If we use this pure silicon detector and then the number of hole pairs will generate that will also be a quite low.

Ultimately, what it means is that the resistivity of pure silicon is pretty low, that means even if you apply a voltage the current that you are getting is too lower. Higher voltage lower current is very low resistivity that is all. So, the resistivity of silicon can be increased or in other word the efficiency of this electron detectors silicon based EDS detector the efficiency of silicon based EDS detector can be increased if we somehow can increase the resistivity of this pure silicon.

For that there are few ways number one the whole detector is made as a semiconductor PIN junction. If we use the pure silicon just the pure silicon and then apply the bias then the current will be very less. If we use it like a PIN junction and then apply a reverse bias then the current will increase. I am not going into how it is done and so this is basic semiconductor physics, but this is how the detectors are made that is the first thing.

The second thing that can be done is the doping the silicon with a very small concentration of lithium and making an extrinsic semiconductor. So, this inherently the number of holes will increase then and resistivity will increase current will increase that is the second thing. So, silicon that is used is usually doped with lithium in the EDS detector. The third thing can be done is that cooling of this hole detector to a liquid nitrogen temperature because generally the electron hole pairs have a tendency to recombine.

And if the thermal energy is reduced if the atomic vibrations are reduced then these recombination process becomes quite difficult electrons and holes try to stays as it is and then that will increase that means that will increase the current. So, this EDS detectors are typically, they are used or they are employed with a liquid nitrogen condition. So, usually this

if you look back this image the detector is basically only this much that is the EDS detector the big tank many a times my students think that this big tank is the EDS detector.

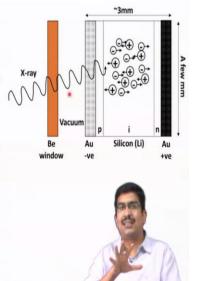
No, this big tank is just a liquid nitrogen for storing liquid nitrogen so that ultimately this EDS detector can operate at a very, very low temperature so that is do not get confused with this entire bigger size of this EDS detector. The detector finally is very small and kept very close to the electron beam which is possibly you do not see also. And then another important point is the current which normally passes between these electrodes when there is no excess signal is there.

There is still I mean as I said that you have lithium silicon and lithium in that and you have this PIN junction which is reversed biased and so on. So, even if there is no x-ray signal still because of this entire arrangement the current that passes through this entire circuit is very, very small. It is only accompanied to or rather when I use this word that is a very small what I mean is that the current that flows without when the detector is not in operation that amount will be very minute or very less compared to when it is exposed to x-ray signal.

When x-ray photons comes and hits the detector that time the current will be maybe an order of magnitude higher than the current when the detector is not working so that after words that current which is generated can be amplified, measured fairly easily and so on we will come into those things now.

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- In Si(Li) semiconductor junction, the *l* region occupies most of the 3 mm thickness.
- Thin layers of gold are necessary on both surfaces of the detector so that the bias potential can be applied.
- The film of gold on the outer face of the detector must be as thin as possible so that very few X-rays are absorbed in it; a layer only 20 nm thick provides adequate conductivity.
- The gold-coated outer surface is usually further protected by a thin window of beryllium or a polymer.
- This window is necessary to prevent contaminants from the specimen chamber of the microscope from condensing on the very cold surface of the detector
- It also forms a further barrier to the entry of X-rays.
- The window, despite being made of beryllium (Z = 4) or carbon (Z = 6) and only being a few micrometers thick, absorbs a significant proportion of the low energy X-rays falling on the detector and therefore makes light elements particularly difficult to detect.



So, a little more about this detector and little more about the construction of the detector, working principle of the detector. So, this silicon lithium conductor junction this i region inside this region is usually around 3 millimeter of thickness then what we do is that we use a very small, a very thin layer of gold are put on the either side of this PIN junction and this thin layer of gold usually around 20 nanometer thick these gold layers works as an electrodes through which the reverse biased or through which the voltage is applied and this completes the electronic circuit.

So, this gold layers are the electrodes here. They are usually kept very thin around as I said 20 nanometer or so, so that they does not interfere with the incoming x-ray signal they should not absorb the x-ray signals which are coming and they should not even take up energy from this x-ray signal. So then there will be mistakes, there will be problems or confusions in the measurement ultimately.

So, that is why this gold electrodes are kept very thin, then there is another usually another component in the detector and that is a beryllium window or usually either it is a beryllium window or some kind of a carbon or polymer basically polymer window which is kept here just right after this gold where the x-ray signals basically enters where this if you think about this detector where basically the detector is exposed to the specimen.

At that place itself there is a beryllium window or a carbon window here. Now this window the purpose of this window is basically this is not about electrodes or anything. The purpose of the window is to prevent contamination. So, as you understand as I said that within the specimen itself there are many more carbon, many other organic materials are there and they just burnt off and those carbons and those burnt off products is just basically flying around all over the specimen chamber and so on.

So, this contamination this contaminants they can from the specimen chamber they can come to the detector, they can sit over this gold electrode, they can basically contaminate the entire detector can affect the detection efficiencies as such. In order to prevent that these window is kept. Now the window is basically transparent, it is supposed to be transparent towards this x-ray signal.

But it will just stop all the carbons that is flying around in the specimen chamber and trying to sit on the detector itself. And that is more so because remember this detector is kept at liquid nitrogen temperature. So, the carbon contaminants, organic particles or even the dust and particles which are there in the specimen chamber they also try to sit at this very cold side of the detector where the x-ray signal basically enters.

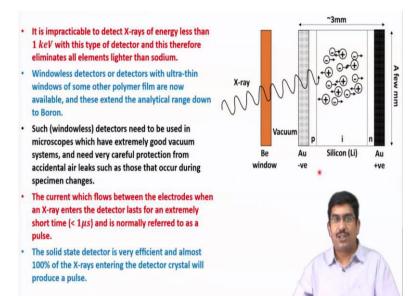
At that side if there is no protection they will come and they will sit on this detector plate here and that will possibly damage the detector and will affect its collection efficiency as well that is why this beryllium window it provides. The problem of this one is providing a beryllium or a carbon. So, purposefully the beryllium or carbon is used because their atomic number is very very less.

So, this characteristic x-ray signal which is passing through which is coming here usually are from high atomic number materials compared to beryllium or carbon at least. So, those signals are much stronger or their energy is much higher and they does not interfere with the beryllium that means the beryllium atoms does not really absorb anything I am not suppose to absorb anything from this x-ray signal so long as you use a high atomic energy elements x-ray are coming from higher atomic energy elements so long as that is happening the x-ray signals this beryllium window or carbon window is not supposed to interfere with the x-ray signals.

Also this windows are purposefully made a very few microns thick very small so that they does not really absorb any kind of electrons and this is true so long as you are using very high atomic number ion, aluminum, copper, metallic samples, transition metals and so on. So long as you are using them no problem with the beryllium window or carbon window. Problems happens when you are using a very light elements.

So, light elements means those x-ray signals are also of low energy or at a comparable energy than that can excite on the beryllium window or the beryllium atom that can interact with the beryllium atoms can generate secondary signals from them. So, that means the incoming x-ray signal will be absorbed here or they will be mixed with the beryllium window some signals generated from beryllium window they will be mixed with that or their energy will be changed in coming x-ray signal that detection will be a problem.

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So, that is why so long as this beryllium signal, beryllium windows are used or carbon windows are used EDS detectors are not so good for detecting light elements that was basically the problems with earlier generation or the first generation or previous generations of EDS detectors and the detector limit also there is a limit to the detector the amount of x-ray energy that it can generate.

That means basically the current that can be generated by the x-ray signal if that is if the incoming signal is less than an energy x-ray photon energy is less than 1 kilo electron volt than the amount of electron hole pairs that is generated here that will be very, very small and that cannot be detected. So, that is the limitation of this EDS detector. So, that is why the EDS detector if we want to measure or if you want to use this EDS detectors with beryllium window or carbon window to certain light element most of the x-ray energy will be absorbed.

And in the process what will happen there will not be enough signal or there will not be measurable signal in the detector itself. So, this entire detector will not be able to perform properly the way it is expected and usually the limit is up to sodium. So, anything less than sodium or in fact sometimes even with sodium anything less than sodium is a big difficultly for measuring with this EDS detectors.

Modern innovation of course then people have come up with smart design of this EDS detector and nowadays there are detectors which does not have any window or these are called window less EDS detectors or detectors with a very ultra thin polymer film very ultra

thin windows of some polymer films are now available. And that is basically extend the analytical range of this EDS detectors all the way down to Boron level.

Qualitative not quantitative I am not talking about quantitative detection qualitative detection of EDS detectors nowadays with a good kind of EDS detector we can go down all the way up to Boron. The problem or rather the requirement for such windowless detector is that these detectors should have very high vacuum system. So, inside the specimen chamber there should be very high vacuum system so that the contamination or the contaminant flying around all over the specimen chamber that problem should not happen.

So, vacuum system if that can be maintained within the specimen chamber then this kind of windowless EDS detectors can be used which is these days very easily can be achieved using some vacuum pumps. Another point to remember that there should not be any accidental air leaks most often that happens during changes of specimen and so on. So, there is any smart design of the specimen chamber if it happens so that there is no air leaks then this sort of windowless detectors can be achieved.

So, in that sense this loading of the specimen is very, very important when you are trying to use it for some kind of compositional detection measured qualitative as well as quantitative detection of this x-ray signal then the specimen loading is very important. Many of these dedicated chemical analysis systems they have specimen loading system where the specimen chamber is never exposed to the outside conditions.

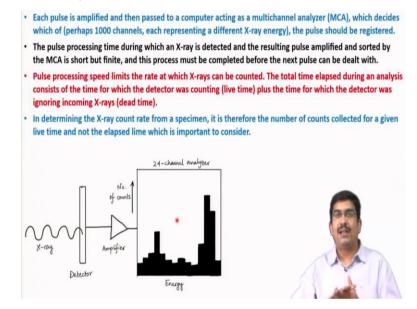
So, specimen chambers are always kept at a very high vacuum and that specimen loading is done in such a way that they are always this specimen chamber whether detector is there, specimen chamber is there, sample holding is there that setup is kept at a very high vacuum and the entire setup is never exposed to outside conditions. So, detectors now the way they detects is that the current which flows between the electrodes in here generally that is generated like a pulse.

So, when a x-ray signal is entered here that generates this currents and that current is used as a pulse. So, sometimes x-ray is entering here generating one stage of current and then the detector stops there itself. So, one pulse is generated that means it is not accepting any more x-ray signal after this one pulse is generated. And that pulse is generated the generation time for each pulse is very short lived that is usually less than 1 microsecond.

So, this solid state detector that is used here is very efficient and almost 100% of the x-ray signal enters in the detector signal can produce this pulse here. I am talking about the efficiency of generating a current signal here. Once capturing the x-ray signal is a separate thing, but x-ray signals which are capturing in the detector to make them enable to generate this current that is another one.

That part is very, very efficient this electron this PIN junction based silicon lithium detectors they are very efficient in at least in generating these pulse signals.

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Then once each pulse is amplified, each pulse is generated they are then amplified and they are then passed to a computer so this is an x-ray signal, this is a detector. It is then pass through an amplifier there that amplifier then is transferred to a computer which acts like a multichannel analyzer. And that multichannel analyzer what it happens is that it has a library and in that library it works like a different channels normally 1000 channels or even.

So, this each of these channels represent a different, different x-ray energy. So, in the detector the x-ray energy is detected by the electron hole pair generation and the current that is generated. So, this from the amount of current we generate we detect what is the energy of the x-ray signal. Then it is matched with these channels each of these in library and we find out that this pulse is basically from this specimen or from this element.

Now the point is the pulse processing time during that x-ray signal during which an x-ray signal is detected then the pulse is amplified and sorted by MCA. This is short very fast this entire process, but this is also a finite time and this process must be completed before next pulse is generated. So, that means the detector has a lifetime when the detector capture x-ray signal and generate a pulse.

And then it has a dead time when it does not capture any x-ray and that dead time during that dead time this amplification and this detection of the energy, detection of the elements is happening. So, it has two parts. One time detector is live another time this one is active this entire MCA is active the amplifier part is active. So, detector has a lifetime and a dead time. In lifetime the detector detects the x-ray signal it captures the x-ray signal, generates a pulse and then it stops and those that time that pulse is identified here.

So, this pulse processing time so it is not only the detector what is the time the detector develops a pulse that is important that in a like how much time the detector is live and capture the x-ray signal and generates a pulse that is one thing. And then the dead time also is pretty it is also very much important here because the total time elapse the total time for analysis of one spot.

So, one pixel the x-ray signal is falling on one pixel generating x-ray which is going to the detector. So, the total time that is needed for doing an analysis for each pixel that will be a summation of the time for which the detector is live capturing x-ray and then detector is dead when the signal is processed both. So, in determining the number of x-ray count from a specimen for each pixel, how many x-ray signals are generated.

It depends on the number of counts collected for a given lifetime and it is not related on the total elapse time. So, the count x-ray count when we calculate the x-ray count it is only from the life period when the detector is live not the entire part because the entire part also consists this when the pulse or the x-ray signal is identified there. So, with that we will end it here and then in the next class we will discuss about the wavelength dispersive spectroscopy and its working principle and so on. Thank you.