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Lecture-18
Materials Characterization
Fundamentals of Scanning Electron Microscopy

Dr. S. Sankaran
Associate Professor
Department of Metallurgical and Materials Engineering
IIT Madras
Email: ssankaran@litm.ac.in

Hello everyone welcome to this material characterization course. In the last class we started discussing the image formation and its interpretation in scanning electron microscopy and then you see that certain things have come out very clear. The image formation in SEM is quite different from what you see in optical or light optical microscopy or even transmission electron microscopy, where the ray paths connecting the location in the specimen in the corresponding the image points is not there in SEM.

Seen in that sense there is no true image exist in SEM, where we collect only the signals which is coming out of the specimen and the signals are collected in the form of second electrons or backscattered electrons and the detector process this electronically and then the spatial features on the specimen is converted into domain of time. Since the signals recorded in SEM as a function of time, the even the very fine scale details under the SEM will have a very high frequency signals. So that is with respect to a detector frequency band which you have seen and also the kind of detectors involved how effectively it can collect these signals and process them to produce an image.

So all these features we have just a detailed manner we have discussed in the last class and we also introduced the origin of the contrast from the specimen, so today we will just continue from that point.

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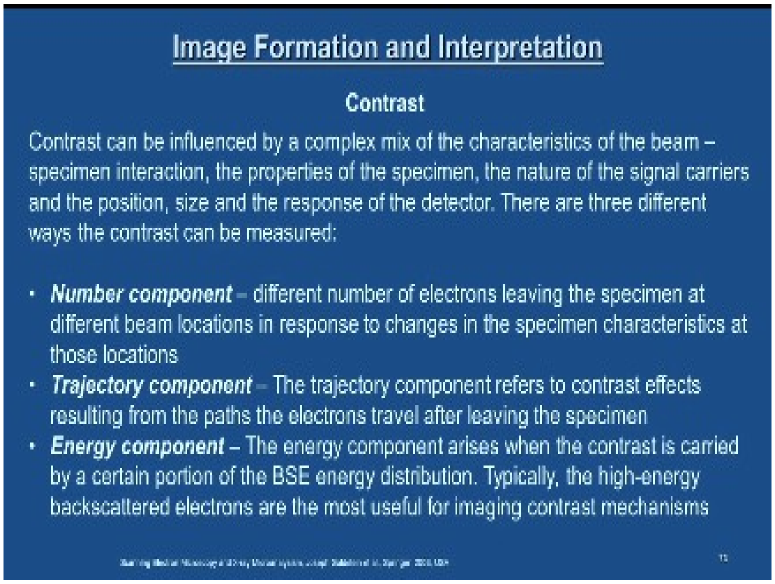

Image Formation and Interpretation

Contrast

Contrast can be influenced by a complex mix of the characteristics of the beam – specimen interaction, the properties of the specimen, the nature of the signal carriers and the position, size and the response of the detector. There are three different ways the contrast can be measured:

- ***Number component*** – different number of electrons leaving the specimen at different beam locations in response to changes in the specimen characteristics at those locations
- ***Trajectory component*** – The trajectory component refers to contrast effects resulting from the paths the electrons travel after leaving the specimen
- ***Energy component*** – The energy component arises when the contrast is carried by a certain portion of the BSE energy distribution. Typically, the high-energy backscattered electrons are the most useful for imaging contrast mechanisms

I just mentioned that the kind of parameters which is involved in the contrast we will go through that point again. Contrast can be influenced by a complex mix of the characteristic of the beam specimen interaction, the properties of the specimen, nature of the signal carriers and the position, size and the response of the detector. There are three different ways the contrast can be measured. One is number component, that is a different number of electrons leaving the specimen at the different beam locations in response to the changes in the specimen characteristics at those locations and trajectory component. The trajectory component refers to the contrast effects resulting from the paths the electrons travel after leaving the specimen and thirdly the energy component which arises when the contrast is carried by a certain portion of the BSE energy distribution, typically the high energy backscattered electrons are the most useful for imaging contrast mechanisms.

So it is very clear whether you obtain a secondary electron signal or a backscattered electron signal these three parameters are going to determine the property or the quality of the contrast which you can obtain from the a given specimen. So now let us get into the details.

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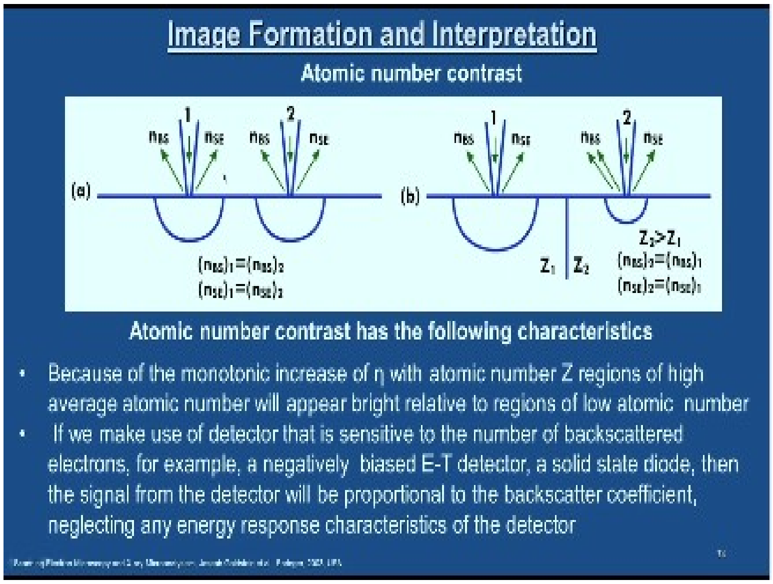

Image Formation and Interpretation

Atomic number contrast

Atomic number contrast has the following characteristics

- Because of the monotonic increase of η with atomic number Z regions of high average atomic number will appear bright relative to regions of low atomic number
- If we make use of detector that is sensitive to the number of backscattered electrons, for example, a negatively biased E-T detector, a solid state diode, then the signal from the detector will be proportional to the backscatter coefficient, neglecting any energy response characteristics of the detector

First we will look at the atomic number contrast, this also sometimes referred as a compositional contrast or a Z contrast. The schematic clearly shows that the electron beam specimen interaction and then where you have the signals coming out, n number of vaccinated electrons and n number of secondary electrons, where schematic a shows $(n_{BS})_1 = (n_{BS})_2$ and $(n_{SE})_1 = (n_{SE})_2$. So according to the contrast definition the very basic definition these two objects will not exhibit a contrast, that is what the basic definition itself says you are you should have a very significant different signal collecting from the surroundings and the object, right.

So you will not produce a contrast but when you have the situations like depicted in the schematic (b) where you see that the atomic number is different from these two objects that means there is a sharp interface between the object 1 and object 2 then you started showing the contrast or the specimen will exhibit a significant image contrast. So let us look at the general remarks. Atomic number contrast has the following characteristics because of the monotonic increase of η that is a backscatter coefficient with atomic number Z, regions of high average atomic number will appear bright relative to regions of low atomic number. If we make use of detector that is sensitive to the number of backscattered electrons for example a negatively biased E-T detector a solid-state diode then, the signal from the detector will be proportional to the backscatter coefficient neglecting any energy response characteristic of the detector. So this is very particular to the backscattered electron signal and again the with respect to E-T detector.

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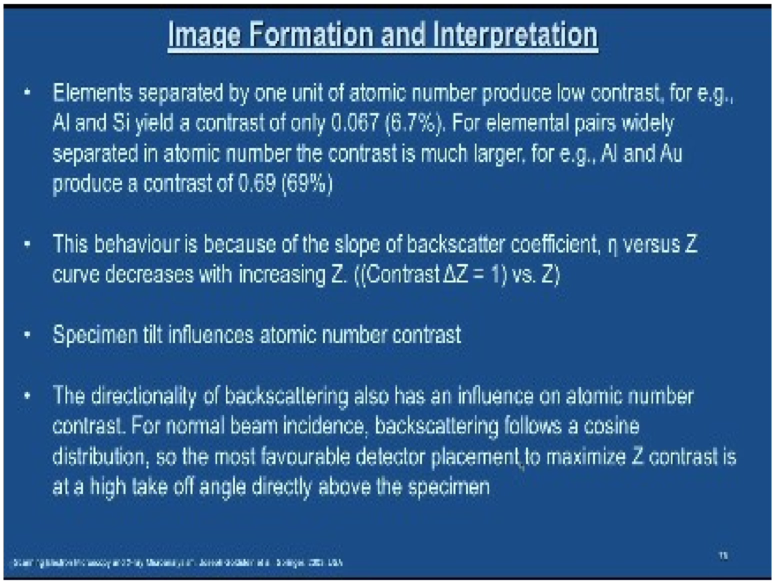

Image Formation and Interpretation

- Elements separated by one unit of atomic number produce low contrast, for e.g., Al and Si yield a contrast of only 0.067 (6.7%). For elemental pairs widely separated in atomic number the contrast is much larger, for e.g., Al and Au produce a contrast of 0.69 (69%)
- This behaviour is because of the slope of backscatter coefficient, η versus Z curve decreases with increasing Z. ((Contrast ΔZ = 1) vs. Z)
- Specimen tilt influences atomic number contrast
- The directionality of backscattering also has an influence on atomic number contrast. For normal beam incidence, backscattering follows a cosine distribution, so the most favourable detector placement to maximize Z contrast is at a high take off angle directly above the specimen

And another important point to note is elements separated by one unit of atomic number produce low contrast for example, aluminum and silicon yield a contrast of only 0.067 that is 6.7% the element pairs widely separated in atomic number the contrast is much larger for example aluminum and good produce a contrast of 0.69 that is 69% this is very important point to remember this behavior is because the slope of backscatter coefficient η versus Z curve decreases with increasing atomic number.

Typically the plot is against ΔZ=1 verses Z where the slope of the backscatter η changes significantly. Another important point is the specimen tilt influences the atomic number contrast which we have already seen in the sum of the previous slides we talked about the specimen how it is being loaded and so on and also the directionality of the backscattering also has an influence on atomic number contrast. For a normal beam incidence back scattering follows a cosine distribution so the most favorable detector placement to the maximizes Z contrast is at a high take off angle directly above the specimen. You see if you recall the when we looked at the instrumentation detail in a laboratory, I will just clearly showed that how exactly this BSE detector is placed. It is placed just above the specimen, fixed on the just below the pole piece though so that that is the position we talked about that is the BSE signals are very well collected and you know that BSE electrons are very high energy electrons, their trajectories are straight lines as compared to the trajectories of low energy signals namely secondary electrons. So this I think is very clear.

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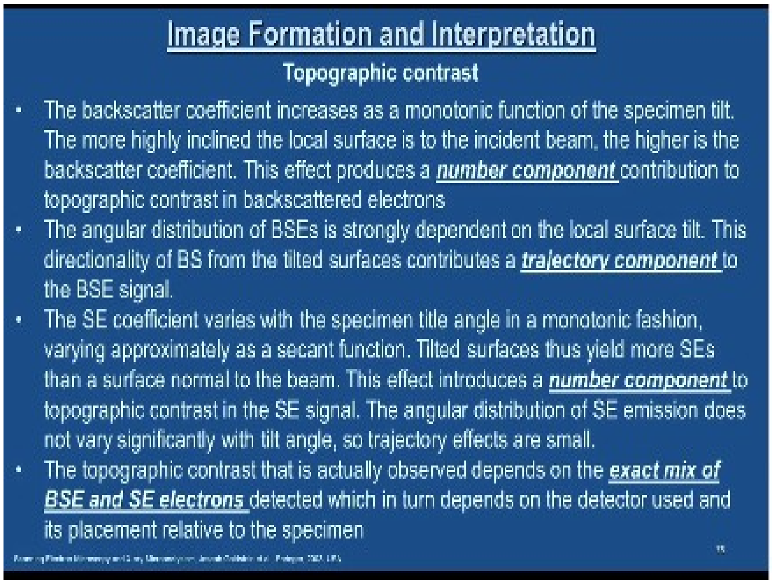

Image Formation and Interpretation

Topographic contrast

- The backscatter coefficient increases as a monotonic function of the specimen tilt. The more highly inclined the local surface is to the incident beam, the higher is the backscatter coefficient. This effect produces a ***number component*** contribution to topographic contrast in backscattered electrons
- The angular distribution of BSEs is strongly dependent on the local surface tilt. This directionality of BS from the tilted surfaces contributes a ***trajectory component*** to the BSE signal.
- The SE coefficient varies with the specimen title angle in a monotonic fashion, varying approximately as a secant function. Tilted surfaces thus yield more SEs than a surface normal to the beam. This effect introduces a ***number component*** to topographic contrast in the SE signal. The angular distribution of SE emission does not vary significantly with tilt angle, so trajectory effects are small.
- The topographic contrast that is actually observed depends on the ***exact mix of BSE and SE electrons*** detected which in turn depends on the detector used and its placement relative to the specimen

Now we move on to topographic contrast. You see the origin of the topic of topographic contrast is a very complex and we will see some of the salient features of that. The backscatter coefficient increases as a monotonic function of the specimen tilt. The more highly inclined the local surface is to the incident beam, the higher is the backscatter coefficient. This effect produces a number component contribution to the topographic contrast in backscattered electrons.

You have to understand one point here before I read through the other salient features. Topographic contrast comes from the contribution of both secondary electron as well as BS electrons. If you can you can see that when you talk about secondary electrons, the number component is more important but when you talk about BSE signals the number as well as that trajectory components are more important in producing the topographic contrast. So the origin of the topographic contrast is generally a complex thing but, it is always a mixture of these secondary electron as well as the backscattered electron number as well as trajectory components finally decides the topographic contrast okay.

Now we will move on to the next point. The angular distribution of backscattered electrons is strongly dependent on the local surface tilt. This directionality of the backscattered electron from the tilted surface contributes to a trajectory component to the BSE signal. The secondary electron coefficient varies with the specimen tilt angle in a monotonic fashion varying approximately as a secant function. See in opposition to the cosine function here it is a secant function, tilted surface thus yielding electrons than the surface normal to the beam. This effect introduces a number component to the topographic contrasting SE signal.

The angular distribution of SE emission does not vary significantly with the tilt angle so that trajectory effects are small. So this is the reason why the number component of SE only contributes to the topographic contrast as against the BSE where, the number as well as the trajectory components both contribute to the topographic contrast. Finally, the topographic contrast that is actually observed depends upon the exact mix of BSE and SE electrons detected which in turn depends on the detector used and its placement related to the specimen. So you have the detector final configuration where it is placed with respect to the specimen electron beam that also very crucial in detecting the signal and it contrasts.

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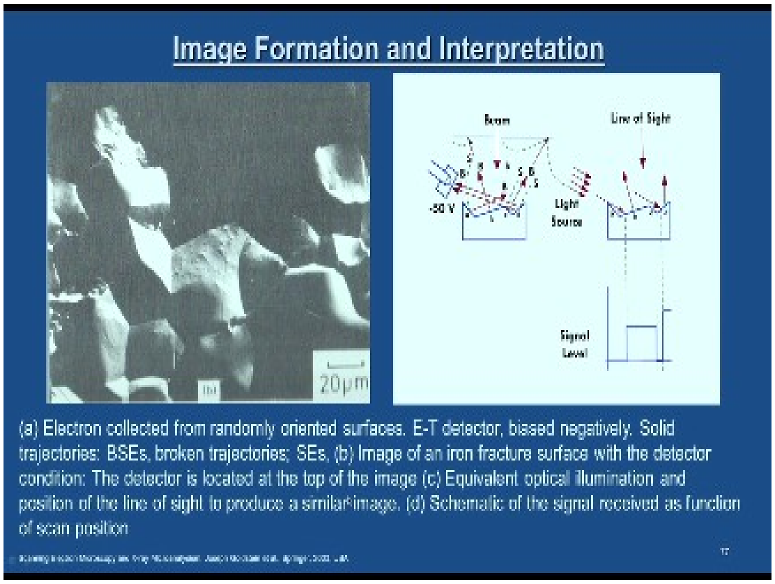

Image Formation and Interpretation

(a) Electron collected from randomly oriented surfaces. E-T detector, biased negatively. Solid trajectories: BSEs, broken trajectories; SEs, (b) Image of an iron fracture surface with the detector condition: The detector is located at the top of the image (c) Equivalent optical illumination and position of the line of sight to produce a similar image. (d) Schematic of the signal received as function of scan position

Now what I am going to show here is, look at this micrograph and the corresponding a schematic where we are going to just demonstrate how the E-T detector behaves for two operating at two different potentials. One is negatively biased potential another is positively biased potential. The image which you are seeing is a fracture surface, that is the electrons collected from the randomly oriented surface that is why you have this all the facets in the fracture surface and we will see first look at the details here what is that this is the E-T detector which is negatively biased and this is a pole piece, electron beam coming onto this fracture surface and then you get signals like BSE as well as SE are coming out of this and something is getting collected.

Right now we will just stick to this schematic and then we will look at this second portion of this after we finish the discussion here. So what you see here is the signals which are directly getting into the E-T detector will appear bright so, similar situation is shown here. This face are the signals are directly going from this to the E-T detector that means, the E-T detector is facing exactly opposite to this face. So that is what it is shown here. The other faces which are randomly oriented to this detector angle, they are not getting collected, you can see that they are going here and there they are not getting into the detector.

So this phase at (d) corresponding to the very bright region what you are seeing in the actual micrograph and another important thing is you can see also the ridge which creates the shadow in front of this is also seen here. So what you can appreciate from this schematic is, when you have the negatively biased E-T detector and the electron beam falling on the fracture surface normal to this then you see as this kind of contrast only that is because the backscattered electron which is exactly getting into this E-T detector facing this face it is normal to it only will contribute to the contrast. The rest of the other signal will not contribute to the contrast, that is why you see the rest of the region either grayish or completely dark.

Now you look at this right hand side schematic where you imagine that suppose if you want to look at this same surface using the light. Suppose if the light falling in this direction the light falls on this fracture surface in this direction, then you will also get the similar micrograph in a light in the line of light or in a light microscope you will see that and the schematic here which is depicting the signal level. So what you are now appreciating here is the signal which is coming from this (d) is the highest that is why it is appearing bight and the next region which is appearing gray is showing the signal level here and rest all the region where you have the zero signal appearing a dark.

So now if you with this description if you go through this figure caption certain things will be very clear. So you have the electron collected from randomly oriented surface E-T detector bias negatively, solid trajectories, BSEs broken projector SEs, image of an iron fracture surface with the detector condition the detector is located at the top of the image and (c) is the equivalent optical illumination and the position of the line of sight to produce a similar image and (d) is a schematic of the signal received as a function of scanned position.

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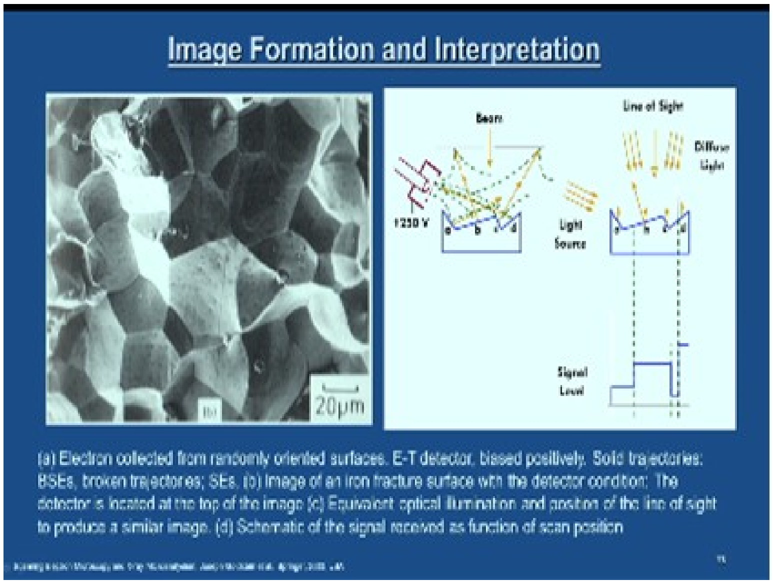

Image Formation and Interpretation

(a) Electron collected from randomly oriented surfaces. E-T detector, biased positively. Solid trajectories: BSEs, broken trajectories; SEs. (b) Image of an iron fracture surface with the detector condition: The detector is located at the top of the image (c) Equivalent optical illumination and position of the line of sight to produce a similar image. (d) Schematic of the signal received as function of scan position

So we will now look at another image where the E-T detector is positively biased. Remember when you when your detector is positively biased it is going to collect both SEs as well as BSEs. So you can see that clearly from that specimen most of your BSEs and SEs are most of the mean a SE and BSE is are all collected together in this positively biased E-T detector and the position is not changed from the previous one but you can see that since both the contributions are there you see the fracture surface appearance much more clearly whatever was appearing quite dark there you were started appearing bright here.

But still this region which appeared with the highest brightness remains the same because the detector position has not changed and you see the complete details here. And what you are seeing the right hand side is again the light source when you I mean light optical analogy basically. So you see that light source which comes here then also you will see this kind of signal profile when you use the diffuse light. So I think with these two schematics you got a very basic idea of the contrast which is obtained on these specimens either negatively biased director or a positively biased detector.

And also you have now got some basic understanding or about the contrast mechanisms either it is a Z contrast or the topographic contrast. So with that I would like to bring the imaging and interpretation part of an SEM I would like to just move on to the next topic in an SEM where people look at the chemical details of this specimen. You see most of these SEMs are interfaced with a spectrometers like energy dispersive spectrometer, a company called EDAX produces it and then most of the chemical analysis is done using this EDS or WDS that is wavelength dispersive spectrometer. Though the spectrometer as such is a very specialized topic and it requires quite a bit of time to get into the details.

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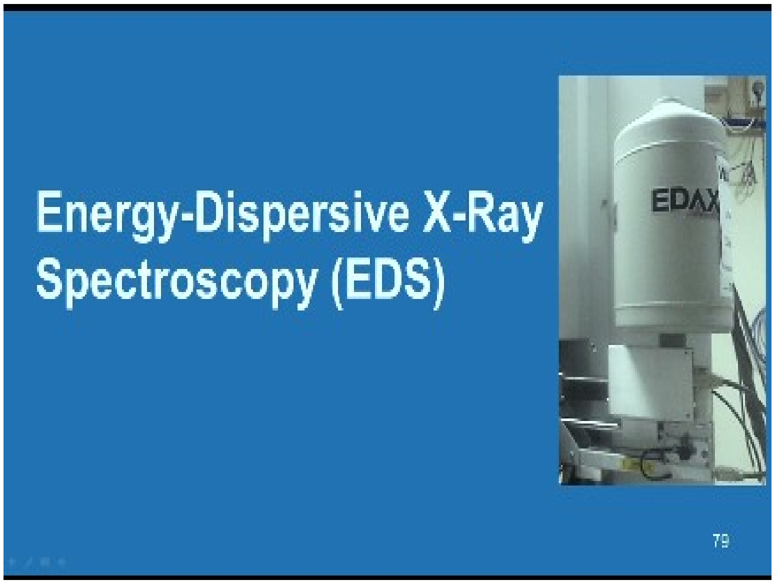

Energy-Dispersive X-Ray Spectroscopy (EDS)

Of what is the merit of each of the spectroscopic techniques and it what kind of quantitative information one can obtain from this is requires a special it will come in a special lecture series but for the completion I would like to discuss these spectrometers which is commonly used in a very brief manner.

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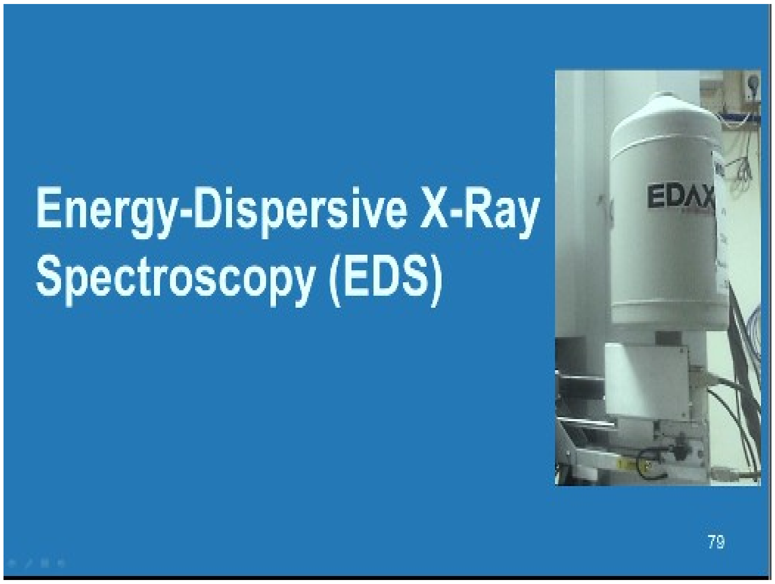

Energy-Dispersive X-Ray Spectroscopy (EDS)

So the first one I would like to take is the energy-dispersive x-rays spectroscopy called EDS and the schema I mean the photo which is shown, is from our lab which is an interface with electron microscope so just look at the introductory remarks.

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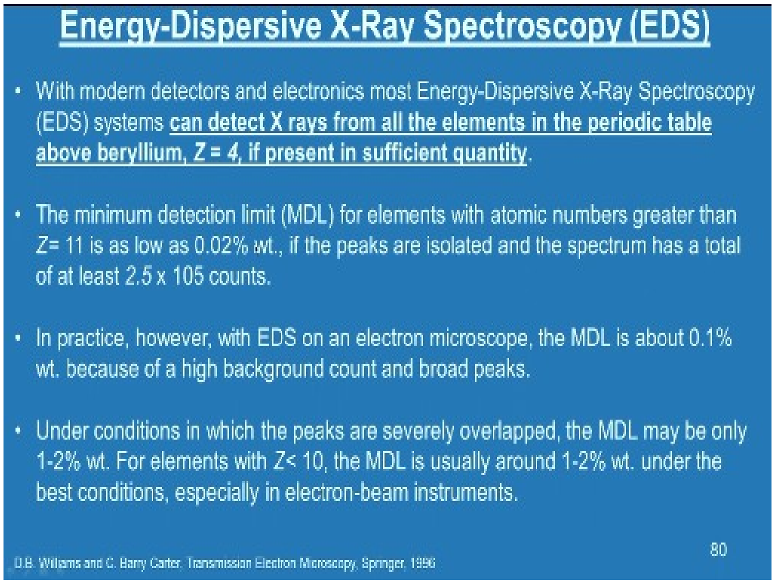

Energy-Dispersive X-Ray Spectroscopy (EDS)

- With modern detectors and electronics most Energy-Dispersive X-Ray Spectroscopy (EDS) systems **can detect X rays from all the elements in the periodic table above beryllium, $Z = 4$, if present in sufficient quantity**.
- The minimum detection limit (MDL) for elements with atomic numbers greater than $Z = 11$ is as low as 0.02% wt., if the peaks are isolated and the spectrum has a total of at least 2.5 x 105 counts.
- In practice, however, with EDS on an electron microscope, the MDL is about 0.1% wt. because of a high background count and broad peaks.
- Under conditions in which the peaks are severely overlapped, the MDL may be only 1-2% wt. For elements with $Z < 10$, the MDL is usually around 1-2% wt. under the best conditions, especially in electron-beam instruments.

D.B. Williams and C. Barry Carter, Transmission Electron Microscopy, Springer, 1996

With the modern detectors and electronics most energy dispersive x-ray spectroscopy systems can detect x-rays from all the elements in the periodic table, above the beryllium that is Z= 4 if present in a sufficient quantity. I also mentioned in the beginning of this, the fundamentals I just mentioned something like this about 10% by weight percent or so and depending upon the detector capability that varies of course and the and this is particular area where the companies keep on making an effort to produce an efficient detectors and so on and we will talk about those developments later.

First we look at the basic ideas behind this spectroscopy the minimum detection limit, MDL for elements with atomic numbers greater than eleven is as low as 0.02% weight percent. If the peaks are isolated and the spectrum has a total of at least 2.5 times 1058 counts, so this is one rough calculation. In practice however with the EDS on an electron microscope, the MDL is about 0.1% weight percent because of a high background count and abroad peeks. Under conditions in which the peaks are severely overlap, the MDL may be only 1 to 2 weight percent for elements with less than 10 atomic number 10 the MDL is usually around 1 to 2 weight % under the best conditions especially in electron beam instruments.

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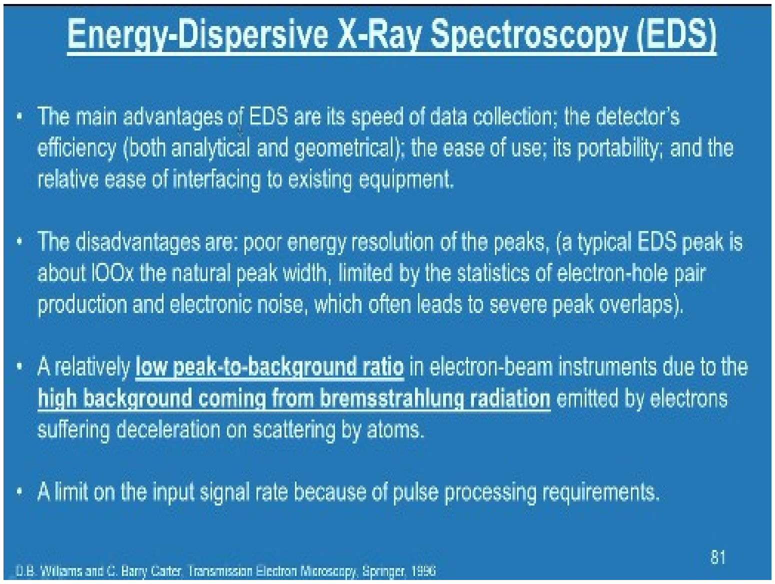

Energy-Dispersive X-Ray Spectroscopy (EDS)

- The main advantages of EDS are its speed of data collection; the detector's efficiency (both analytical and geometrical); the ease of use; its portability; and the relative ease of interfacing to existing equipment.
- The disadvantages are: poor energy resolution of the peaks, (a typical EDS peak is about IOOx the natural peak width, limited by the statistics of electron-hole pair production and electronic noise, which often leads to severe peak overlaps).
- A relatively **low peak-to-background ratio** in electron-beam instruments due to the **high background coming from bremsstrahlung radiation** emitted by electrons suffering deceleration on scattering by atoms.
- A limit on the input signal rate because of pulse processing requirements.

D.B. Williams and C. Barry Carter, Transmission Electron Microscopy, Springer, 1996

The main advantage, advantages of EDS are its speed of data collection, the detectors efficiency both analytical and geometrical, the ease of use, its portability and the relative ease of interfacing to existing equipment. The disadvantages are poor energy resolution of the peaks, a typical EDS peak is about100x the natural peak width limited by the statistics of electron hole pair production and electronic noise which often leads to severe peak overlaps. The relatively low peak to background ratio in electron beam instruments due to the high background coming from the bremsstrahlung radiation emitted by electrons suffering deceleration or the scattering by atoms.

So these are some of the details we will look at it when we get into the diffraction discussion later. A limit on the input signal rate because of the pulse processing requirements. This is again another disadvantage.

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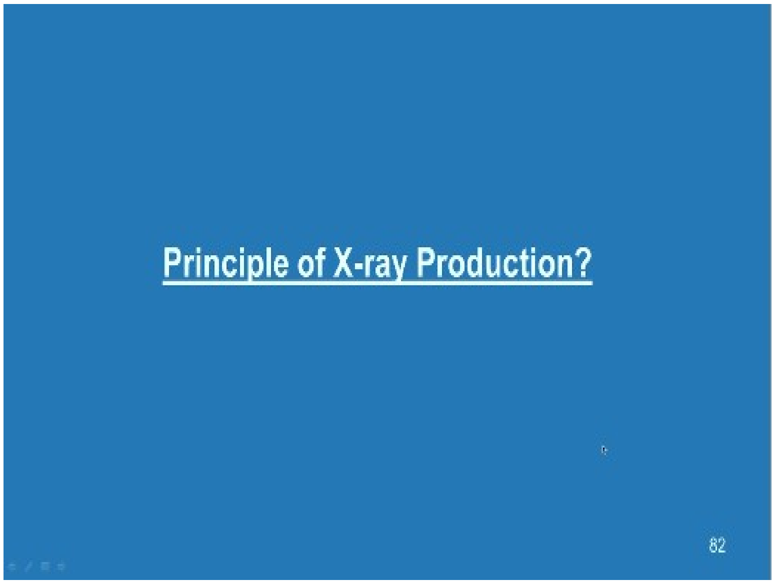

Principle of X-ray Production?

And let us look at how the X-rays are produced in this equipment.

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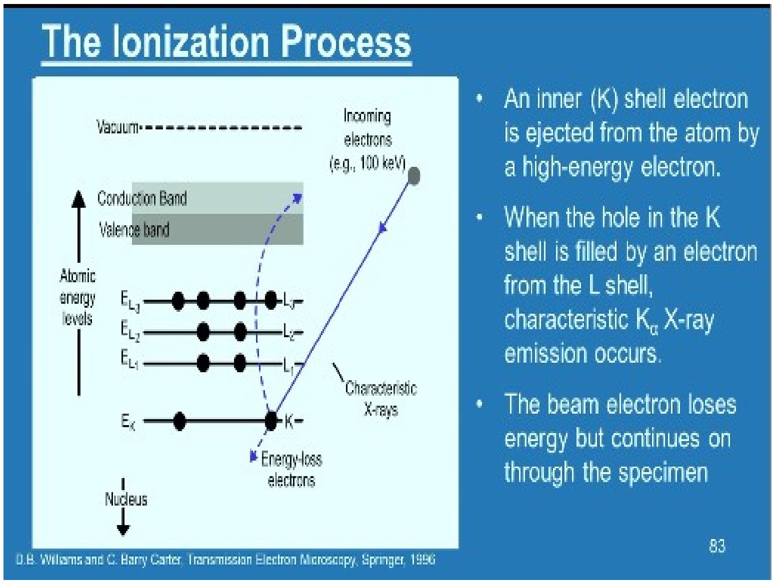

The Ionization Process

- An inner (K) shell electron is ejected from the atom by a high-energy electron.
- When the hole in the K shell is filled by an electron from the L shell, characteristic K_α X-ray emission occurs.
- The beam electron loses energy but continues on through the specimen

D.B. Williams and C. Barry Carter, Transmission Electron Microscopy, Springer, 1996

Look at this schematic where you are seeing that atomic energy level in the one axis and here you see that all the electron levels energy levels like K, L, M and so on and then you have this valence band and conduction band and let us assume this incoming electrons having the energy of 100 KeV. So let us look at this schematic what is happening you have the incoming electrons and which is coming and knocking out an electron, so in that process the energy is getting lost and then there is a transition from the upper shell to the lower shell. In this case it is L_3 to K and during that process, you have the characteristic rays X -rays coming out.

So that is how you have to visualize this whole ionization process. So an inner K shell electron is ejected from the atom by a high-energy electron, when the hole in the K shell is filled by an electron from the L shell characteristic K_α X -ray emission occurs. The beam electron loses energy but continues on through the specimen.

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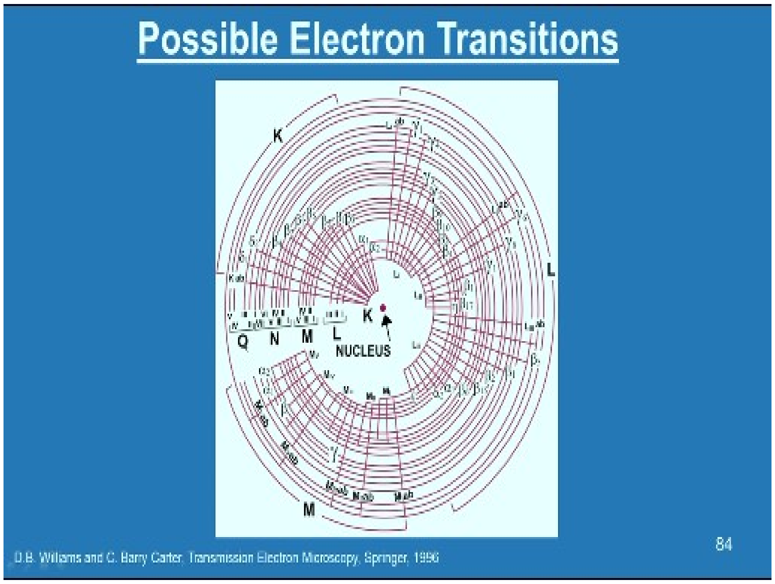

Possible Electron Transitions

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And this you all might have already have some idea all possible transitions from different electron shells like you know you have the orbits you can say L, M, N, O all this, so depending upon the transitions the energy levels also varies and this is just for a completion I just brought this schematic.

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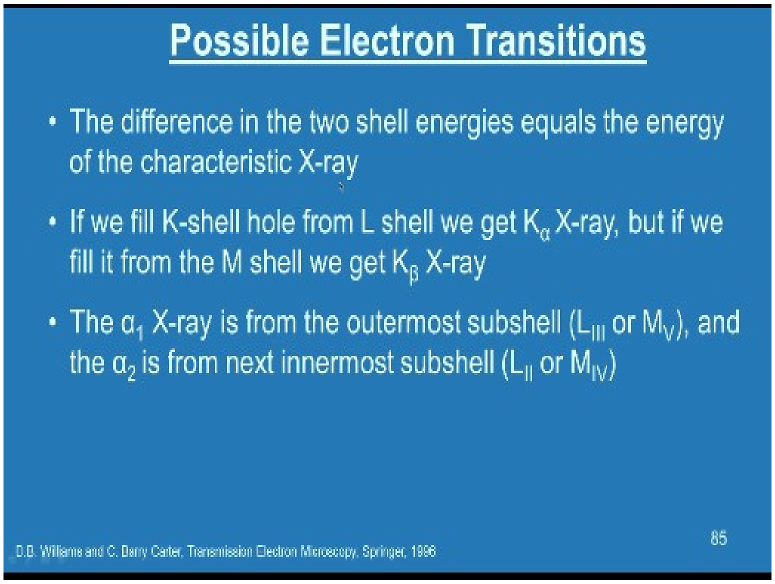

Possible Electron Transitions

- The difference in the two shell energies equals the energy of the characteristic X-ray
- If we fill K-shell hole from L shell we get K_α X-ray, but if we fill it from the M shell we get K_β X-ray
- The α_1 X-ray is from the outermost subshell (L_{III} or M_V), and the α_2 is from next innermost subshell (L_{II} or M_{IV})

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The difference in the two shell energies equals the energy of the characteristic X-ray. If you fill K shell hole from an L shell, we get K_α X- ray but if we fill it from the M shell we get K_β X- ray. The α_1 X- ray is from the outermost sub shell like L_3 or M_5 and the α_2 is from the next innermost sub shell like L_2 or M_4. so that is the convention and the instrument details are like this you have X-ray come in,

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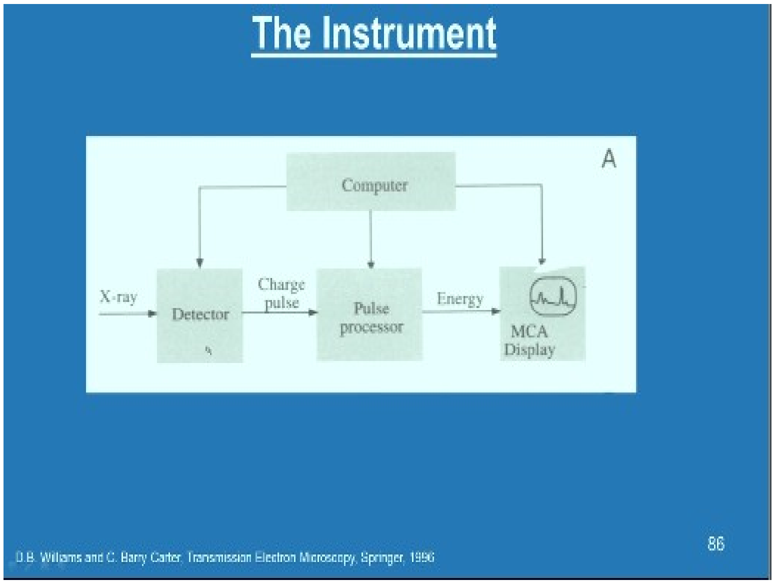

The Instrument

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and you have the detector and which is having a charge pulse and pulse processor and then you have energy and then you have MCA display everything is controlled by the computer.

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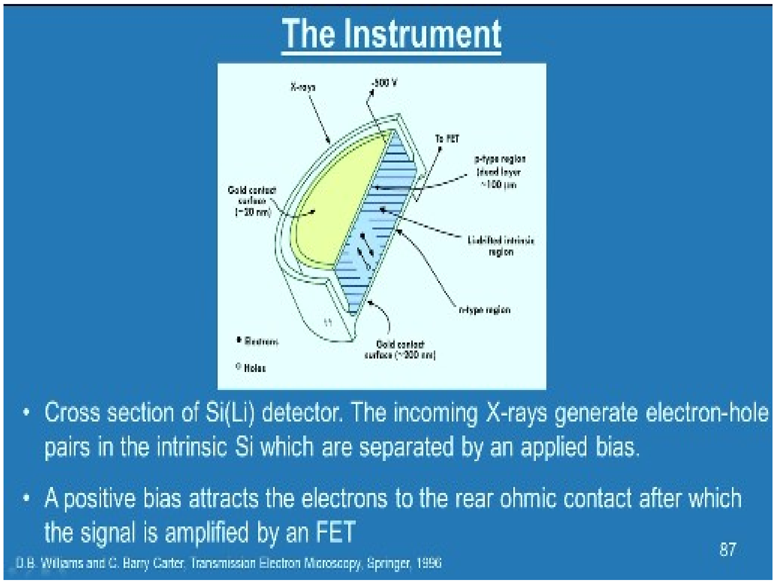

The Instrument

- Cross section of Si(Li) detector. The incoming X-rays generate electron-hole pairs in the intrinsic Si which are separated by an applied bias.
- A positive bias attracts the electrons to the rear ohmic contact after which the signal is amplified by an FET

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And little more details on the detector system so this is the detector called a Si(Li) detector, silicon lithium detector. It is a cross-section view what you have seen is you see the complete details here and the cross section of Si(Li) detector is schematically shown here. The incoming x-rays generate electron hole pairs in the intrinsic silicon which are separated by an applied bias. So here this is what is that reaction happens. This is a lithium drifted intrinsic region where you have this p-type region as well as n-type region. A positive bias attracts the electrons to the rear ohmic contact after which the signal is amplified by the FET.

When an X-ray photon enters the intrinsic region of the detector through the p-type end, there is a high probability that it will ionize a silicon atom through photoelectric effect. This results in an x-ray or an Auger electron which in turn produces a number of electron hole pairs in the Si(Li) detector. One pair per 3.8 eV of energy, that is the quantification here. For example a 6.4KeV x-ray absorbed by the silicon atoms will produce about 1684 electron-hole pairs or a charge of about 2.7 X 10^{-13}C. Both charged carriers move freely through the lattice, and are drawn to the detector contacts under the action of applied bias field to produce a signal at the gate of especially designed field effect transistor that is FED a mounted directly behind the detectors crystal.

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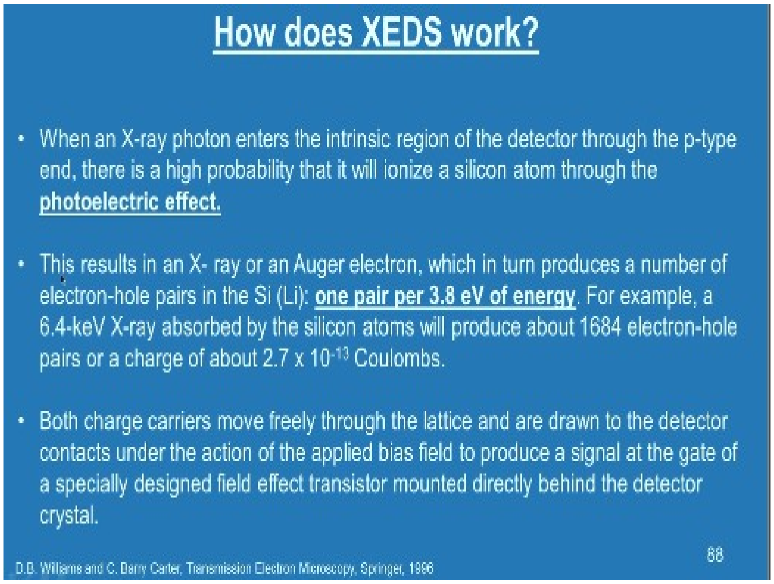

How does XEDS work?

- When an X-ray photon enters the intrinsic region of the detector through the p-type end, there is a high probability that it will ionize a silicon atom through the **photoelectric effect.**
- This results in an X- ray or an Auger electron, which in turn produces a number of electron-hole pairs in the Si (Li): **one pair per 3.8 eV of energy**. For example, a 6.4-keV X-ray absorbed by the silicon atoms will produce about 1684 electron-hole pairs or a charge of about 2.7 x 10^{-13} Coulombs.
- Both charge carriers move freely through the lattice and are drawn to the detector contacts under the action of the applied bias field to produce a signal at the gate of a specially designed field effect transistor mounted directly behind the detector crystal.

D.B. Williams and C. Barry Carter, Transmission Electron Microscopy, Springer, 1996

The transistor forms the input stage of a low noise charge sensitive preamplifier located on the detector housing. The output from the pre-amplifier is fed into the main amplifier where the signal is finally amplified to a level that can be processed by analog to digital converter of the multi-channel analyzer. The height of the amplifier output pulse is proportional to the input pre amplifier pulse and hence the intensity is proportional to the x-ray energy.

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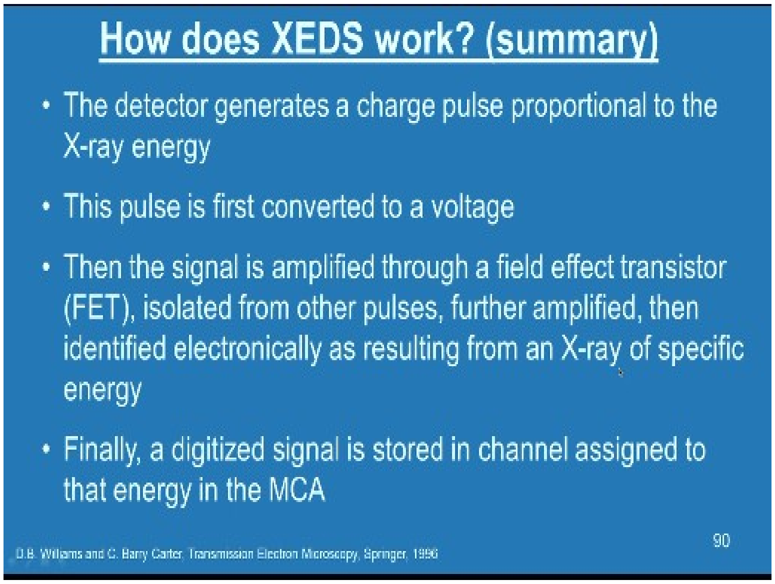

How does XEDS work? (summary)

- The detector generates a charge pulse proportional to the X-ray energy
- This pulse is first converted to a voltage
- Then the signal is amplified through a field effect transistor (FET), isolated from other pulses, further amplified, then identified electronically as resulting from an X-ray of specific energy
- Finally, a digitized signal is stored in channel assigned to that energy in the MCA

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How does X-ray EDS work? So it is a summary the detector generates a charge pulse proportional to the x-ray energy. This pulse is first converted to a voltage, then the signal is amplified through a field effect transistor isolated from the other pulses. Further amplified then identified electronically as resulting from an x-ray of specific energy. Finally a digitized signal is stored in a channel assigned to the that energy in an MCA. So very briefly and quickly I have gone through this function of EDS for a time being this is efficient enough for you to remember it EDS is one of the very popular interface with electron microscopes the other variant in this segment is WDS and before we look at it.

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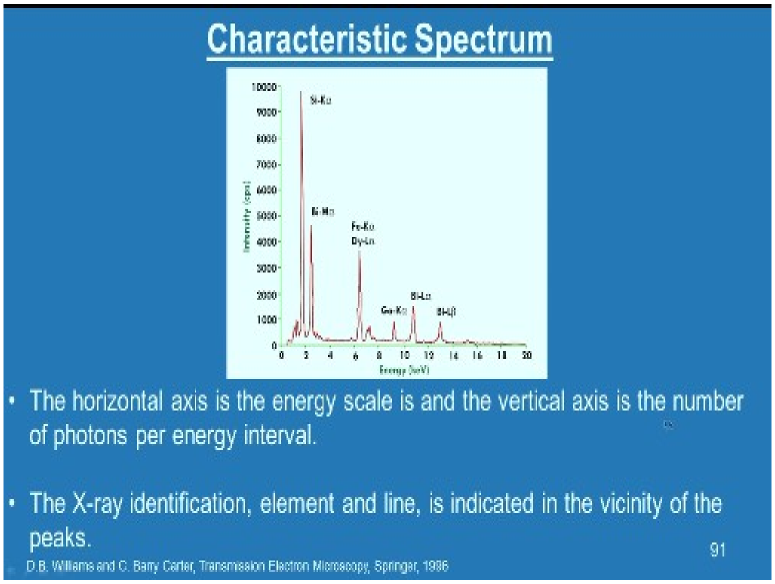

Characteristic Spectrum

- The horizontal axis is the energy scale is and the vertical axis is the number of photons per energy interval.
- The X-ray identification, element and line, is indicated in the vicinity of the peaks.

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Let us look at the characteristic EDS spectrum and you have this intensity versus energy spectrum the horizontal axis is energy scale and his and his vertical axis is the number of photons per energy interval. The x-ray identification element and the line is indicated in the vicinity of the peaks. So this is the index indexing of the peaks are shown how it is indexed.

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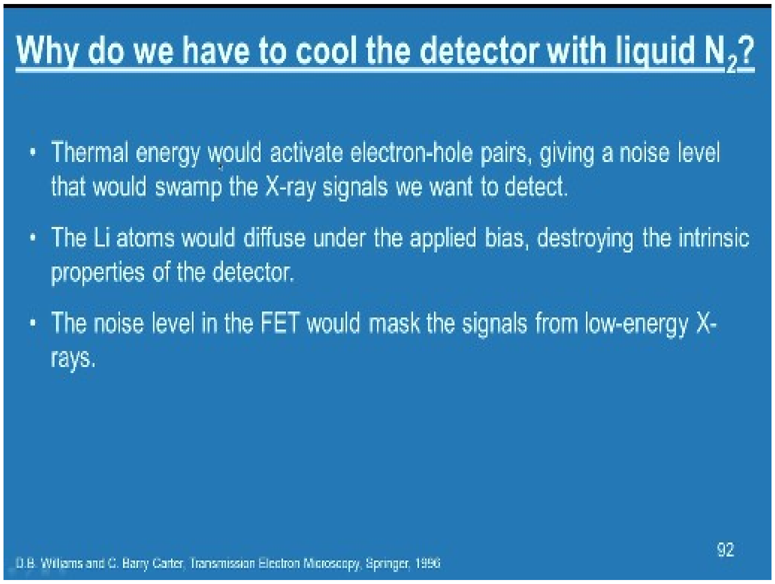

Why do we have to cool the detector with liquid N_2?

- Thermal energy would activate electron-hole pairs, giving a noise level that would swamp the X-ray signals we want to detect.
- The Li atoms would diffuse under the applied bias, destroying the intrinsic properties of the detector.
- The noise level in the FET would mask the signals from low-energy X-rays.

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And most important activity of keeping this EDS spectrum interfaced with the machine is, we have to keep on cooling this detector with liquid nitrogen for the some of the following reasons. The thermal energy would activate the electron hole pairs giving a noise level that would swap the x-ray signals we want to detect. The silicon atoms would diffuse under the applied bias destroying the intrinsic properties of the detector. The noise level in the effect would mask the signal from the low energy x-rays.

So that is why I do not know how much you would have seen where the electron microscopes are interfaced with the EDS spectrum, whether you use it or not this devar has to be filled with liquid nitrogen all the time in order to prevent some of this reactions happening in the detector. Now we will just look at again quickly the wavelength,

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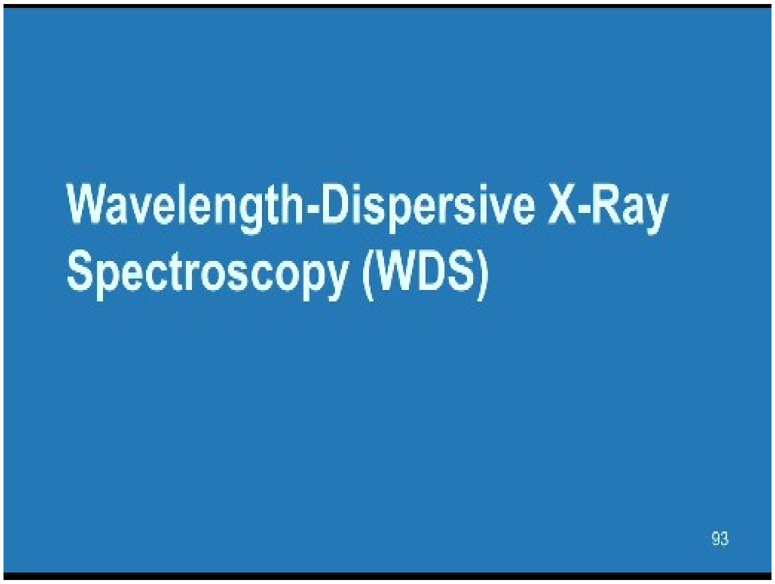

Wavelength-Dispersive X-Ray Spectroscopy (WDS)

dispersive x-ray spectrometer and look at the schematic.

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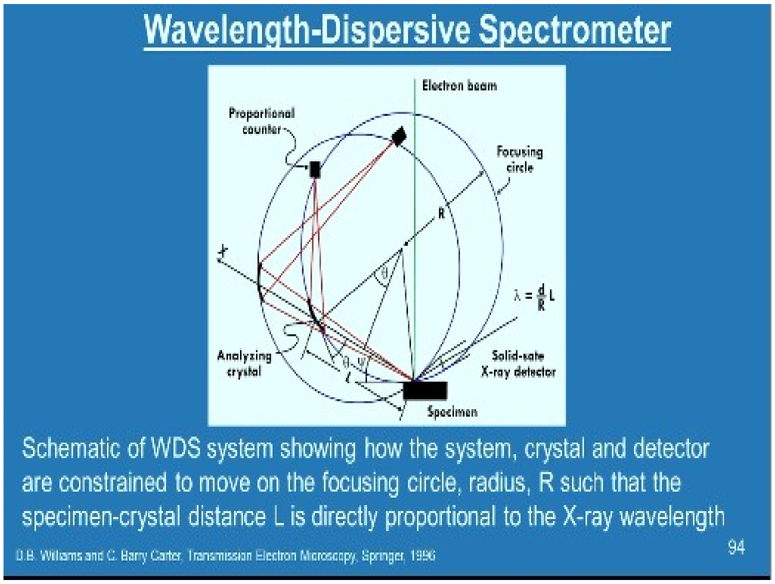

Wavelength-Dispersive Spectrometer

Schematic of WDS system showing how the system, crystal and detector are constrained to move on the focusing circle, radius, R such that the specimen-crystal distance L is directly proportional to the X-ray wavelength

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This is how the system configuration is displayed. So you have the specimen and you have the crystal and you have the proportional counter and everything is kept in a fixed geometry. So you have this schematic of WDS system showing how the system crystal a detector are constrained to move on a focusing circle radius R such that the specimen crystal distance L is directly proportional to the x-ray wavelengths. So this is the specimen crystal distance L which is proportional to the wavelength **$\lambda = (d/R)* L$**.

This is the relation and you see that these configurations are shown in a focusing circle, at two types of configurations are shown where you have the specimen crystal detector are shown in two configuration you follow this one circle and for other configuration, I mean the change in the θ-2θ then you follow this the next circle they are kept in this manner why do we keep this in this position that we will see now.

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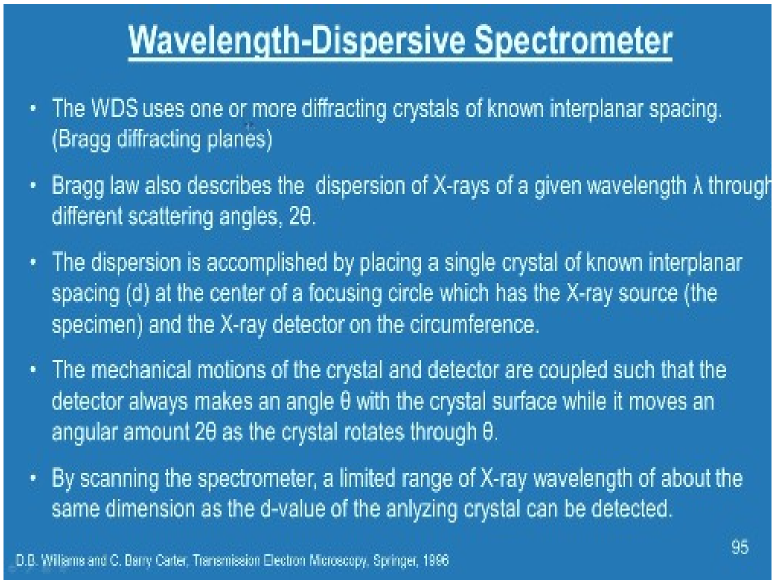

Wavelength-Dispersive Spectrometer

- The WDS uses one or more diffracting crystals of known interplanar spacing. (Bragg diffracting planes)
- Bragg law also describes the dispersion of X-rays of a given wavelength λ throug different scattering angles, 2θ.
- The dispersion is accomplished by placing a single crystal of known interplanar spacing (d) at the center of a focusing circle which has the X-ray source (the specimen) and the X-ray detector on the circumference.
- The mechanical motions of the crystal and detector are coupled such that the detector always makes an angle θ with the crystal surface while it moves an angular amount 2θ as the crystal rotates through θ.
- By scanning the spectrometer, a limited range of X-ray wavelength of about the same dimension as the d-value of the anlyzing crystal can be detected.

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The WDS uses one or more diffracting crystals of known inter planar spacing, Bragg diffracting planes that means when the diffraction actually happens those planes are responsible for Bragg diffraction. Bragg law also describes the dispersion of x-rays of a given wavelength λ through different scattering angles 2θ in a normal x-ray spectrum. The dispersion is accomplished by placing a single crystal of known inter planar spacing d at the center of a focusing circle which has the x-ray source the specimen and the x-ray detector on the circumference.

So this is what it is, you have a specimen, a crystal detector everything is placed on the circumference where the crystal is placed in between the specimen that is x-ray source and the detector this is what written here. The mechanical motions of the crystal and the detector are coupled such that the detector always makes an angle θ with the crystal surface while it moves an angular amount 2θ as the crystal rotates through θ. By scanning the spectrometer a limited range of x-ray wavelengths of about the same dimension as the d-value of the analyzing crystal can be detected.

So by moving this keeping this, same θ between detector and the crystal and the specimen by moving this you will be able to detect some of the characteristic x-rays which has the dimension within the range of the a known inter planar spacing so that is the idea.

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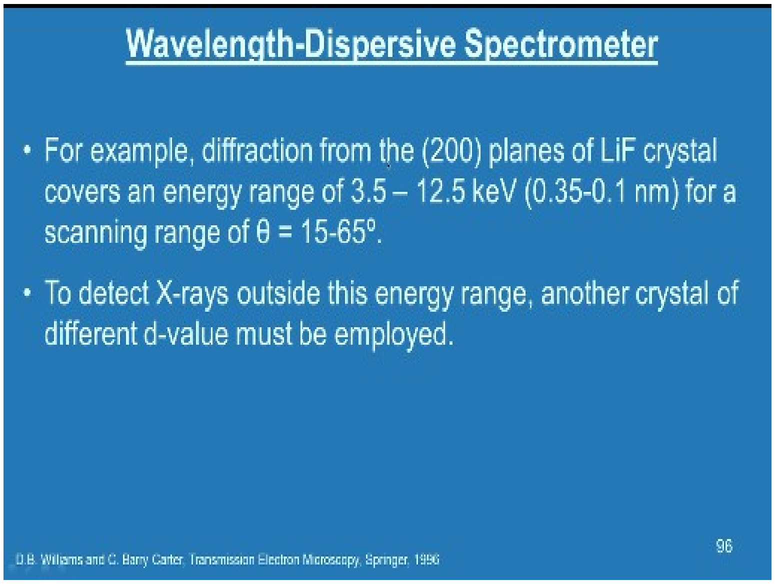

Wavelength-Dispersive Spectrometer

- For example, diffraction from the (200) planes of LiF crystal covers an energy range of 3.5 – 12.5 keV (0.35-0.1 nm) for a scanning range of θ = 15-65°.
- To detect X-rays outside this energy range, another crystal of different d-value must be employed.

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For example the diffraction from (200) planes of lithium fluoride crystal cover some energy range of 3.5 to12.5 KeV for a scanning range of θ is equal to 15° to 65°. To detect the x-rays outside this energy range another crystal of different d-value must be employed.

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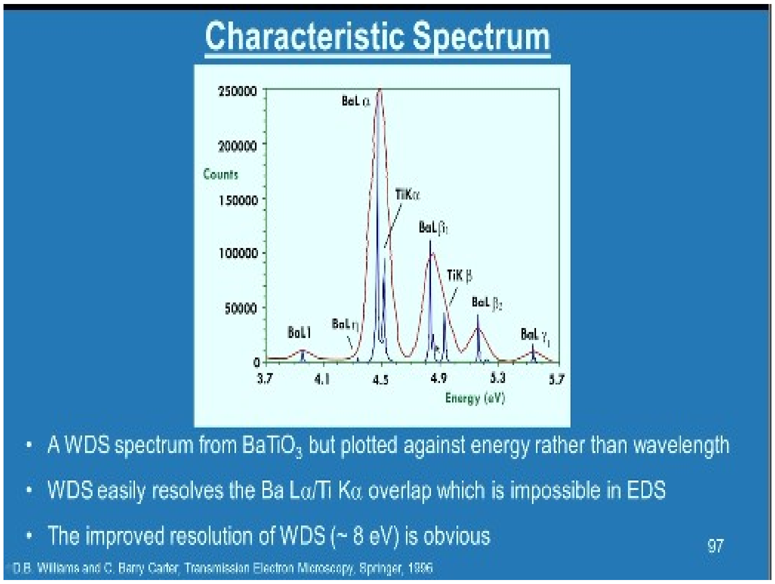

Characteristic Spectrum

- A WDS spectrum from $BaTiO_3$ but plotted against energy rather than wavelength
- WDS easily resolves the Ba Lα/Ti Kα overlap which is impossible in EDS
- The improved resolution of WDS (~ 8 eV) is obvious

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And you look at this typical characteristic spectrum of a WDS. For the purpose of comparison the EDS spectrum is superimposed into this. So you clearly appreciate the resolution capability of WDS superior as compared to EDS. This is the WDS spectrum of barium titanate but plotted against energy rather than the wavelength. WDS easily resolves barium L_α by Ti K_α overlap which is impossible in EDS. This is what is shown here be a very Ba L_α and titanium K_α. The improved resolution of WDS is quite obvious just about the order of 8 eV.

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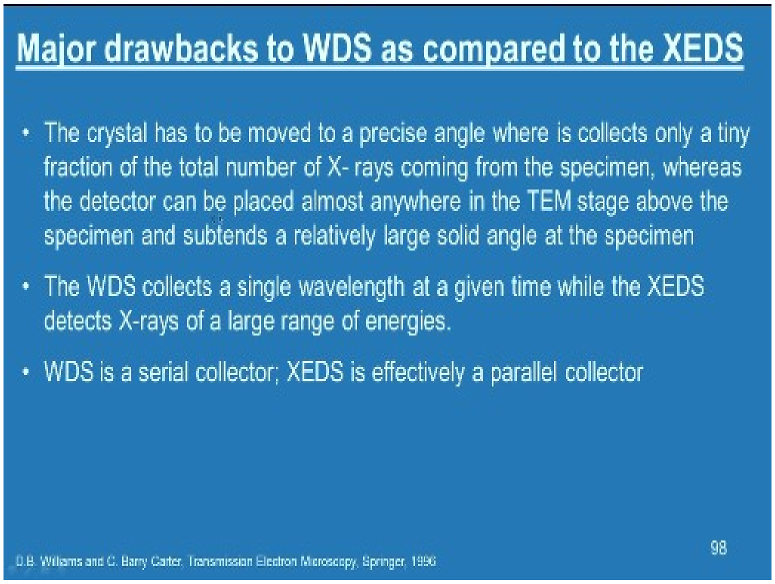

Major drawbacks to WDS as compared to the XEDS

- The crystal has to be moved to a precise angle where is collects only a tiny fraction of the total number of X- rays coming from the specimen, whereas the detector can be placed almost anywhere in the TEM stage above the specimen and subtends a relatively large solid angle at the specimen
- The WDS collects a single wavelength at a given time while the XEDS detects X-rays of a large range of energies.
- WDS is a serial collector; XEDS is effectively a parallel collector

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And we look at the major drawbacks of WDS has compared to EDS, the crystal has to be moved to a precise angle where is where is collects only a tiny fraction of total number of x rays coming from the specimen, whereas the detector can be placed almost anywhere in the TEM stage or any electron microscope stage above the specimen and subtends a relatively large solid angle at this specimen. The WDS collects a single wavelength at a given time while the EDS detects x-rays of a large range of energies. WDS is a serial collector, X-EDS is effectively a parallel collector.

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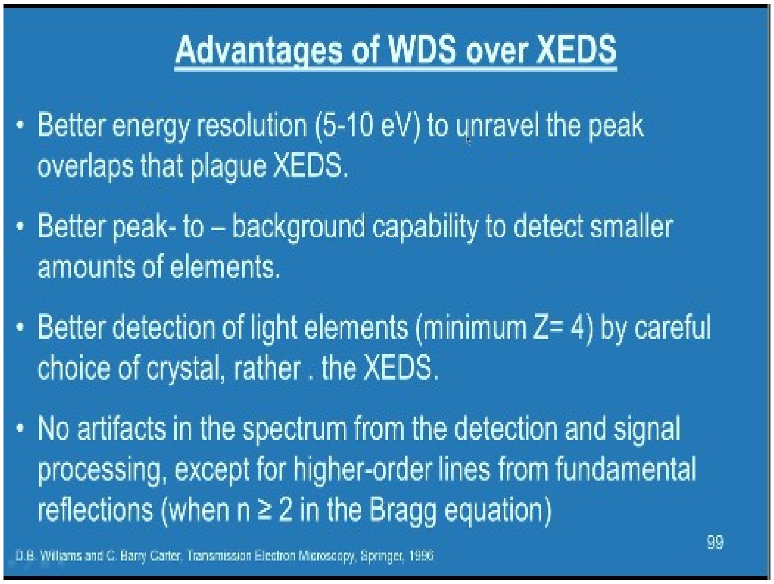

Advantages of WDS over XEDS

- Better energy resolution (5-10 eV) to unravel the peak overlaps that plague XEDS.
- Better peak- to – background capability to detect smaller amounts of elements.
- Better detection of light elements (minimum Z= 4) by careful choice of crystal, rather . the XEDS.
- No artifacts in the spectrum from the detection and signal processing, except for higher-order lines from fundamental reflections (when n ≥ 2 in the Bragg equation)

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And what are the advantages better energy resolution 5 to 10 electron volts to unravel the peak overlaps that like X-EDS, better peek to background capability to detect smaller amounts of elements better detection of light elements which is minimum atomic number four by careful choice of a crystal rather than the EDS, no artifacts in the spectrum from the detection and the signal processing except the higher order lines from the fundamental reflections, when the order of reflection is greater than or equal to 2 in the Bragg equation.

So these are all the very brief introduction to this spectrometers, which is commonly used or interface with the scanning electron microscope. As I just mentioned we have not gone into the details of how the quantitative information is taken from this spectrometer or how they exactly the mapping of the elements, how sensitive these detectors are, we have not discussed in detail but however I just for a sake of completion I have just introduced this very briefly and fastly about this two spectrometers, which is very common.

The other details we will look at in the different lecture series. So in the next class we will look at the some of the special topics enough SEMs. Again I will just only highlights those mechanisms for example where special mechanisms because of the magnetic contrast as well as electric contrast and also we will look at some of the how the backscattering diffraction will enable to find crystallographic orientation mapping called EBSD and so on very briefly in the next class thank you.

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