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Lecture-17
Materials Characterization
Fundamentals of Scanning Electron Microscopy

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Hello everyone. Welcome to this material characterization course. In the last class, we have just looked at some of the instrumentation details of the scanning electron microscopy and we also looked at the details of the detector parts and its capabilities and how one can execute this imaging experiments in the laboratory conditions and so on. And before that if you recall we were also discussing the interaction volume, that is electron beam specimen interaction volume. And then we looked at the various aspects where the interaction volume is significantly influenced by the atomic number of the elements and also the acceleration voltage under which the SEM is operated. So, in this today's class, we would like to discuss little bit about the image formation and its interpretation. You see the image formation in SEM is very different from what we normally go through an optical or light optical microscope or a transmission electron microscope.

So, in that respect it is better to get into the details of how the image formed and then how do we interpret this images and what kind of contrast mechanisms operate. All these details one should have some basic idea. Unlike the other microscopic techniques, in scanning electron microscopy you have to know the signals coming out of the specimen because of the electron beam interaction such as, secondary electrons and backscattered electrons and these signals are collected by the detectors.

So, lot of geometrical parameters involved. For example, as we witnessed in the laboratory demonstration your specimen is just kept below the pole piece and then your detectors are kept at different angles, especially the second electron detectors are kept at particular angle. So, the amount of signals a detector can collect, it depends upon the angle at which the detectors are kept as well as the specimen surface are kept with respect to the beam direction.

And also so many other parameters also involved about the characterizing the electron beam and so on. So, it is it is not that straightforward to understand the kind of contrast you one can get from these any specimen through scanning electron microscopy. It is very interesting to look at the details and how we are able to obtain the information about the surface of the specimen. So, in this class we will now review the instrumentation details once again.

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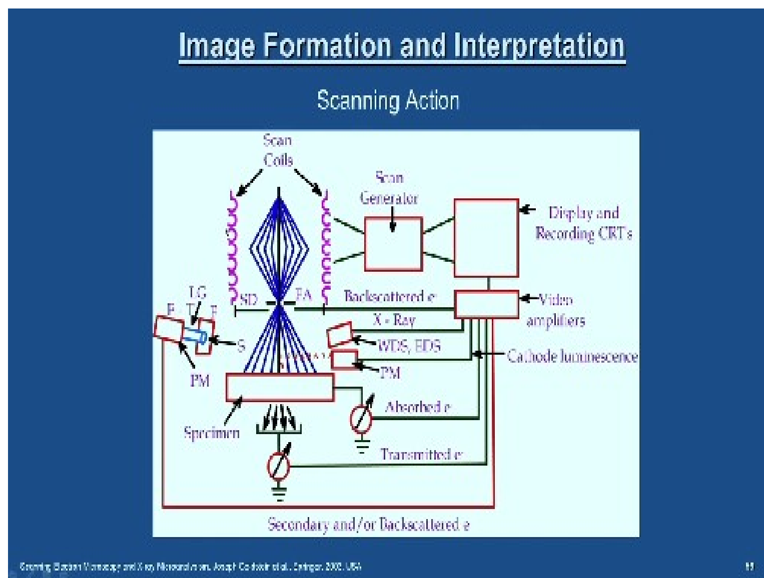

You look at the schematic. And most of the parts we have already discussed, but today we will just see the functions of each of this instrumentation parts and its action during the SEM operation. With respect to the scanning action, we will discuss the response of the each parts of this equipment. So, what you now look at here is the scan coils and then you have this is an electron beam which is falling on the sample.

And then you have this detector. It is ET detectors. We will see what is that. And then you have this Faraday cage, and then you have the scintillator, and then you have photo multiplier, and you have the stand generator here, and of course you have the display and recording CRTs, and you have a set of detectors x-ray, characteristic x-ray detectors and you have set of spectrometers wavelength dispersive spectrometer and energy dispersive spectrometer, and you have photomultiplier, and you have other way electronic device which are connected to this instrumentation details.

So, what we are now interested is, what exactly this coil do as the scanning action. As we have discussed earlier, you see that the upper portion of the coil does the job of deflecting the beam half the move away from the optical axis and then the other set of coils again bring it back to these optic axis in a such a way that a rectangular raster scan is established on the sample. And of course, we know that this is limited by the final aperture here and then it is being made down the made to fall on the sample in this fashion. So, what we have to understand here is, when we say that electron beam is falling on a specimen and it gives out some signal, we always talk with respect to a particular point. That means the signal which is coming from the specimen surface either it could be a characteristic x-ray or a secondary electron or a backscattered electron.

And then we can just interpret from these signals about this specimen surface at that particular point. But then if you we are interested in the complete surface area of the specimen so that means this beam has to scan from the one point to the other point. So, it is like you know this beam is scanning point by point in the or displacement surface, and then that is what the scanning action is all about.

So, you see that, each line shown in the schematic is scanning the specimen at particular point here 1 to 10. Actually, these numbers are displaced here. Actually, just be written here to in accordance with these lines so that it scans, it displays the scanning action of these electron beam from the left to right in one direction. So, similarly, you have the scanning action is possible in the Y axis or Y direction. So, this and then you get the information I mean you collect the signals and then you interpret the images and so on.

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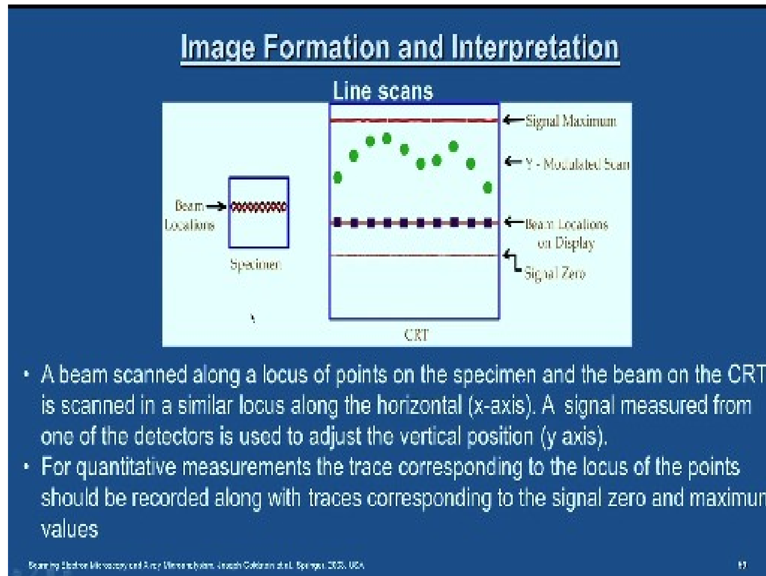

So now with that background we will get into the first line scans. The schematic shows you have the beam location on the specimen and this is on the CRT. So, what you have to understand here from this schematic is, so whatever is the beam does on the specimen surface that is the it is scanning action, this action is synchronized with the CRT screen 1:1.

So, for example, we are talking about a line scan. Though the line scan starts from this one end and it scans point by point, actually this signal is having some dwell time on each point and then it proceeds like this scanning action. And also, you have the signal in Y axis as well which sometimes we can use it for the characterizing the characteristic x-rays and other signals and so on.

So, what we can have the summary of the remarks for the line scan can be a beam scanned along a locus of points on the specimen and the beam on the CRT is scanned in a similar locus along the horizontal X-axis. So, this is the X line of action which we are talked about. A signal measured from one of the detectors is used to adjust the vertical position. See you can use this backscattered electron or characteristic x-ray which you can adjust the vertical position and this kind of signal is very useful when the, for the quantitative measurements.

The trace corresponding to the locus of the points should be recorded along with the trace is corresponding to the signal 0 and maximum values. So, that is how the line scan is performed. Especially this particular line scan operation is useful if you are interested in a local composition in the some of the fracture surface or some of the interface or some multi-component alloys or segregation and so on. We will see whether we can take up some specific examples in new course.

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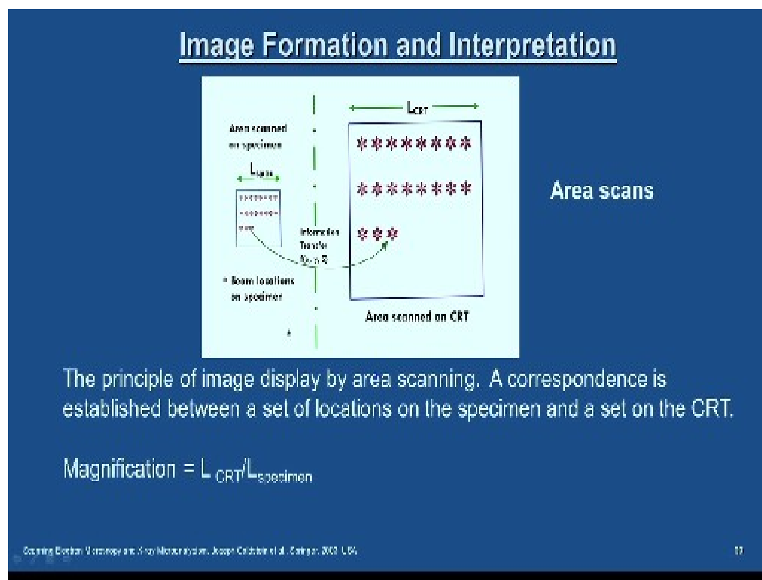

What we are interested in, is not just a line scan but we are interested in an area of the imaging. So, this schematic illustrates the principle of area scans. You have this beam locations on the specimen and the area is scanned on the specimen and this is a linear dimension L on the specimen which is in synchronization with the area displayed in the CRT which can be understood by this. Each area is scanned and then it is also simultaneously shown on the CRT screen.

The principle of image display by area scanning is shown here. Basically, a correspondence is established between a set of locations on the specimen and set on the CRT. As we all recollect, the magnification in SEM is

Magnification = L_{CRT} / $L_{specimen}$

- This is the linear dimension where the magnification is realized.

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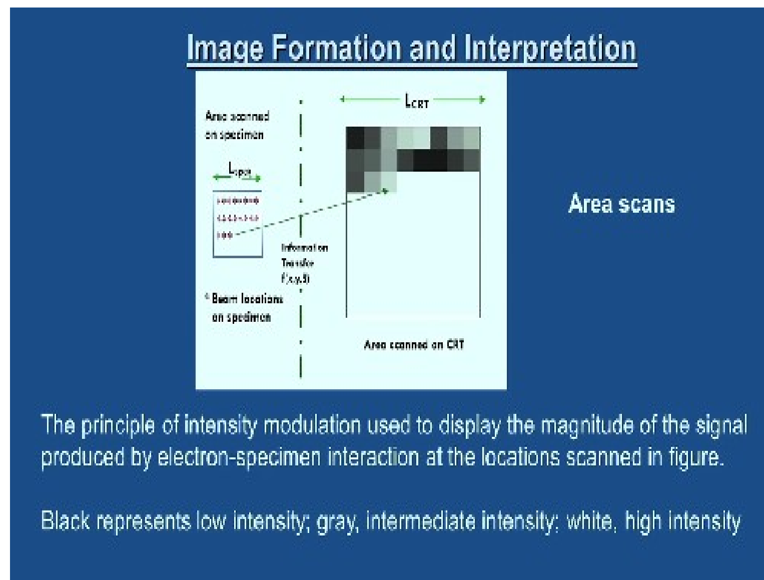

So now, what we actually do here is, you can see that the specimen area where you have the beam locations. What actually you see it on the CRT is the intensity modulation. The principle of intensity modulation used to display the magnitude of the signal produced by the electron specimen interaction at the location scanned in the figure. Most of the time when the SEM is operated and if you in order to adjust the bright signal from the specimen, this operation is performed, called intensity modulation.

You see that, you optimize the electron or a signal collection from the each detectors and then proceed with the scanning action, where you have the black square represents low intensity, gray is intermediate intensity and white is the high intensity signal. And that is how the intensity modulation is adjusted. You see, another important point to remember in SEM is, in SEM the image is generated or I would say the image generation operation consists of the signal mapping.

Rather I would say that whatever we obtain in SEM is not a true image, something like what you get in light optical or transmission electron microscope, where you have the light ray path corresponding to this specimen points connecting to the image points. 1:1 ray path connection is correspondence is there and such where you put a film or a recording medium to record the image in those devices is not possible in this SEM.

In a way, it is a signal collection and it is mapped. Fortunately, it gives very interesting and useful information in terms of topological details of the specimen. So, here the signal is converted into a digital domain or you can say that the digital mapping or a signal mapping. We can understand it like that as compared to light optical and transmission electron microscope.

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In SEM, you should have to understand another important parameter, where the magnification is talked about. Numerical value of magnification in this instrumentation as we have just seen before it is L_{CRT} by $L_{specimen}$, because this maximum CRT scan length is fixed to the full dimension 'L' of the tube. For example, 10 cm and an increase in the magnification is obtained by reducing the length one of the scans on the specimen. Since the CRT screen length or dimension is fixed, the any adjustment you do in terms of increasing or reducing the specimen area will reflect on

the magnification of the imaging. So, the table summarizes the kind of magnification one can obtain and the corresponding area which is scanned on the specimen. For example, and 10X is equal to 100 mm^2, 100X - 1mm^2, 1000X - 0.01 mm^2 and so on. Assumes that magnification is related to the CRT screen that measures 10cm x 10 cm.

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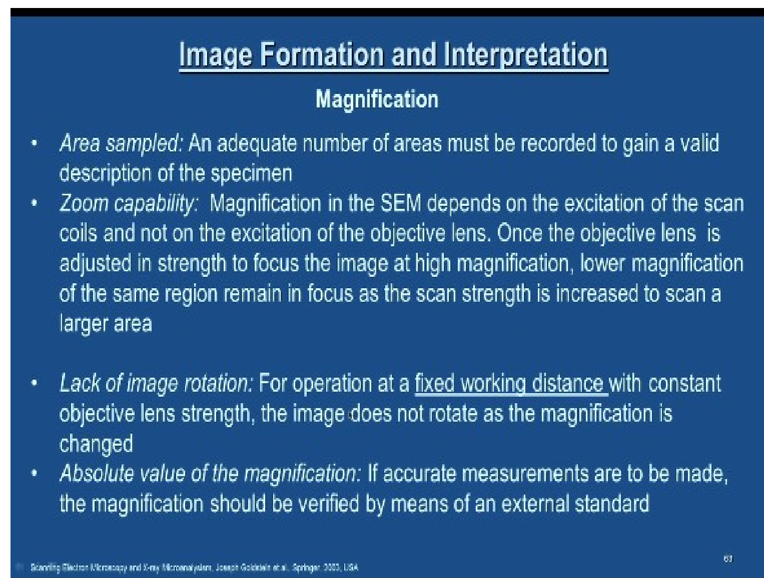

We have to be very careful about this magnification high magnification images because the area sample has to be kept in mind. An adequate number of areas must be recorded to gain a valid description of the specimen. So, then only you can say that you have imaged the specimen with a reasonable representation of the bulk information. If you collect only very high magnification images in one or two locations it may not represent the bulk information of the specimen. So, we have to make sure that the number of area which we examine on the specimen is adequate enough to represent the bulk nature of the specimen.

The second thing is, zoom capability. Magnification in the SEM depends on the excitation of the scan coils and not on the excitation of the objective lens. Once the objective lens is adjusted in strength to focus the image at high magnification, low magnification of the same region remain in focus as the scans strength is increased to a scan larger area. And the another important point

is a lack of image rotation. For operation at a fixed working distance with the constant objective lens strength, the image does not rotate as the magnification is changed.

So, at a fixed working distance your image is not going to rotate. Absolute value of the magnification of course, if you somebody is interested in this aspect, if accurate measurements are to be made the magnification should be verified by means of an external standard. So, for example, if you want to do very sensitive measurements very accurate measurements like then let us use some kind of standard a scale or something or a grating under which you calibrate the distance as you do it with the different magnification, you generate a calibration chart and then you use that values rather than simply reading a marker on the display which comes a long with the images in the SEM.

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Image Formation and Interpretation

Picture Element (Pixel) Size

The picture element is the size of the area on the specimen from which information is collected and transferred to the CRT image or computer memory.

Usually the PE is considered as a circle ot square, described by a linear measure of diameter or edge length. Considering the rectilinear scan to be divided into equal-sized square boxes filling all of the scan frame,

The linear dimension D_{PE} of the picture element is given by the expression

$$D_{PE} = L_{spec}/N_{PE}$$

Where L_{spec} is the length of the scan on the specimen and N_{PE} is the number of discrete locations along the scan line

Table 4.2. Size of the Picture Element as a Function of Magnification. ∝

Magnification	Edge dimension of picture element
10X	10 μm
100X	1 μm
1000X	0.1 μm (100 nm)
10,000X	0.01 μm (10nm)
100,000X	0.001 μm (1nm)

∝ Assumptions: 1000 pixel x 1000 pixel scan matrix, 10 cm X 10 cm display CRT

Scanning Electron Microscopy and X-ray Microanalysis, Joseph Goldstein et al., Springer, 2003, USA

So, the next important aspect is picture element size, a pixel size. The picture element is the size of the area on the specimen from which the information is collected and transferred to see art image or computer memory. Usually, the picture element is considered as the circle or square described by a linear measure of diameter or edge length. Considering the rectilinear scan to be

divided into equal sized square boxes filling all of this can frame, the linear dimension D (pixel) of the picture element is given by the expression

$$D_{PE} = L_{spec} / N_{PE}$$

Where, L_{spec} is the length of the scan on the specimen and N_{PE} is the number of discrete locations along the scan line.

So, you can see that the size of the picture element as a function of magnification. So, you have this magnification 10X, 100X, 1000X and so on. You have the corresponding edge dimension of the picture element given as 10 micron, 1micron, 0.1 micron and so on.

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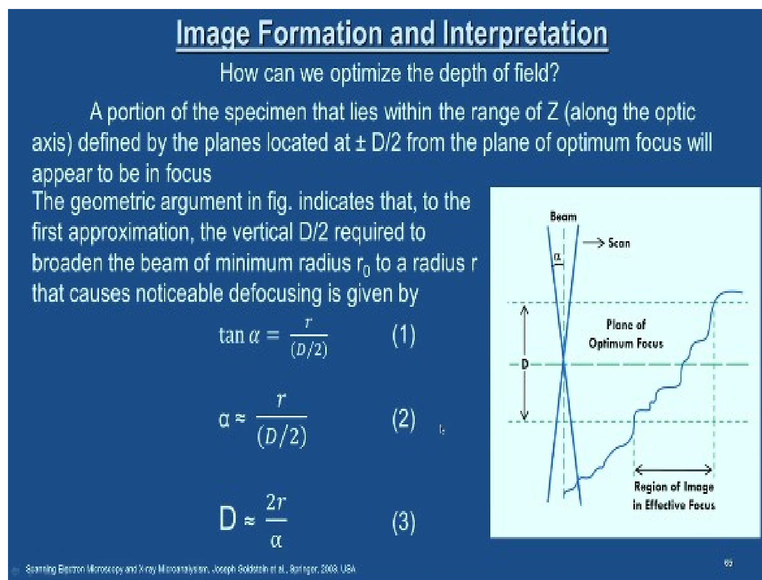

As you all know that in SEM, the very important feature is the depth of field. We discussed in the beginning. As well as some of the theory involved. The depth of field in SEM is an important feature. So, how can we optimize the depth of field. So, let us look at this schematic. So, this is the specimen surface where the beam is falling on this and suppose if you assume this the plane of optimum focus, where the beam converges and this is your α and let us assume that this is a kind of a fracture surface which is having lot of uneven ups and downs, are a lot of inclination.

With this figure in mind, let us look at the remarks. A portion of the specimen that lies within the range of Z (along the optic axis) defined by the planes located that plus or minus **D / 2** from the plane of optimum focus will appear to be in focus. So, let us assume this is the plane of optimum focus. So, this distance is **D / 2**. So, from the geometry, argument in the figure indicates that to the first approximation the vertical **D / 2** required to broaden the beam of minimum radius **'r_0'** to your radius **'r'** that causes noticeable be focusing is given by,

$$\tan \alpha = r / (D/2) \qquad (1)$$

So, with this geometry it valid, where the minimum radius **'r_0'** from there it opens to **'r'**. Where **α** is the glancing angle

$$\alpha \approx r / (D/2) \qquad (2)$$

$$D \approx 2r / \alpha. \qquad (3)$$

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Image Formation and Interpretation

How can we optimize the depth of field?

On a high resolution CRT display where the spot size = 0.1 mm, most observers will find that defocusing becomes detectable when two pixels are fully overlapped, that is, when the condition in figure and eqn. (3) has been reached with r =1 pixel.

The pixel size refred to the specimen space is given by 0.1 mm/M, where M is the magnification. Substituting r = 0.1/M into eqn. (3) gives a practical expression for the depth of field

$$D \approx 2(0.1\text{mm}/M)/\alpha = 0.2\ \text{mm}/\alpha M \qquad (4)$$

Eqn. (4) indicates that to increase the depth of focus D, the microscopist can choose to reduce either M or α. The divergence angle is adjusted by the section of the final beam defining aperture. For aperture with, located at the scan crossover in the exit of the objective lens and working distancradius R_{ap} e, D_W we find

$$\alpha = R_{Ap}/D_W \qquad (5)$$

Scanning Electron Microscopy and X-ray Microanalysis, Joseph Goldstein et al., Springer, 2003, USA

So, on a high-resolution CRT display where the spot size is point 1mm, most observers will find that defocusing becomes detectable when the two pixels are fully overlapped. That is, when the condition in the figure and the equation 3 has been reached with the **r = 1** pixel.

The pixel size referred to the specimen space is given by **0.1mm/M,** where 'M' is the magnification. Substituting **$r = 0.1/M$** into the equation 3, gives a practical expression for the depth of field.

$$D \approx 2\,(0.1\text{mm} / M) / \alpha = 0.2\ \text{mm} / \alpha M \qquad (4)$$

That is, this equation 4 indicates that, to increase the depth of focus D, the microscope is can choose to reduce either M or α. Normally, we are interested in higher magnification. So, the divergence angle is adjusted by the section of final beam defining aperture. For aperture with, located at this scan crossover in the exit of the objective lens and working distance radius R_{AP} and D_W we find that

$$\alpha = R_{AP} / D_W$$

So, from this expression you can see that how one can optimize the depth of field in a SEM.

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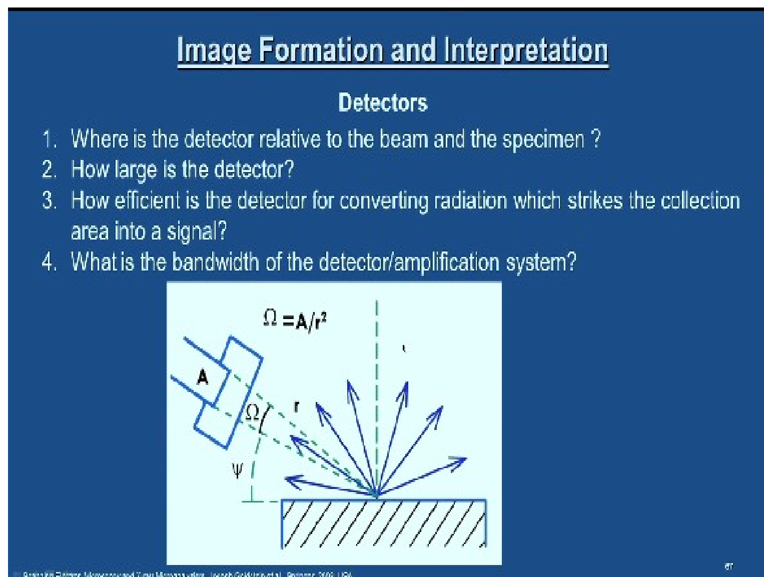

Now, we will move on to some of the characteristics of detectors. As I mentioned that the detectors play a very important role in the quality in obtaining the quality images. And we just look at the characteristic of the detectors and then we move on to the actual details. What is the

detector relative to the sorry where is the detector relative to the beam and the specimen ? That is under the pole piece where exactly your detector is kept and where exactly where specimen is placed. So, if you recall in the laboratory demonstration, the specimen is placed just below the pole piece and if detectives are kept in the particular angle in the side one of the sides.

How large is the detector and how efficient is the detector for converting radiation which strikes the collection area into signal? And what is the bandwidth of the detector or amplification system ? So, here is a schematic which clearly shows. So, the detector is kept at an angle, I would say the take-off angle ψ. The angle between the specimen surface and the line connecting the center of the detector is ψ. And the how large is the detector which is defined by the solid angle Ω,

$$\Omega = A/r^2$$

So, which is defined by this solid angle. So, you can understand this by a sphere has got the solid angle of 4π and how efficient is the detector for converting radiation which strike the collection area into signal. You see, you the signal which is coming out of the specimen has got a wide range of signals with varying energies. So, how efficient the detector can convert this into the useful signal is also an important aspect. We will we will just see that some of the examples how we can understand this.

And what is the bandwidth of the detector or amplification system? So, each detector will have a set of bandwidths. For example, if you are scanning a very fine details and then your signal frequency will be very very high. In fact, your signal is converted into time domain, that is high frequency. So, that means each detector will have a frequency cutoff, beyond which the detector will not collect that signal and then you will lose that information. So, this the bandwidth of the detector is also an important aspect of the detector.

So, the signal which is being collected from the specimen, it is primarily depend upon the type of detectors and its efficiency which depends upon these four parameters what we discussed.

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Image Formation and Interpretation

Detectors

1. Back scattered electrons are beam electrons which escape the specimen as a result of multiple elastic scattering and have an energy distribution $0 \leq E_{BSE} \leq E_0$, with the energy distribution peaked in the range (0.7-0.9) E_0 for intermediate- and high- atomic number targets
2. Secondary electron are specimen electrons given a small amount of kinetic energy by inelastic collisions with beam electrons and are emitted with energies in the range of $0 \leq E_{SE} \leq 50$ eV, with a most probable energy of 3-5 eV and 90% of the total between 0 and 10 eV.

So, when we talk about detectors, we have already classified these signals. Let us recollect those. Backscattered electrons are beam electrons which escaped the specimen as a result of multiple elastic scattering and they have an energy distribution between $0 \leq E_{BSE} \leq E_0$, with the energy distribution peaked in the range $(0.72 - 0.9)E_0$ for intermediate and high atomic number targets, This is the kind of characteristic of BSEs. And secondary electron or specimen electrons given a small amount of kinetic energy by inelastic collisions with the beam electrons and are emitted with the energies in the range of that is there between 0 and 50 electron volts. $0 \leq E_{SE} \leq 50$ eV

With the most probable energy of 3 to 5 electron volts and ninety percent of the total signals between 0 and 10 electron volts. So, this is about the primary signals.

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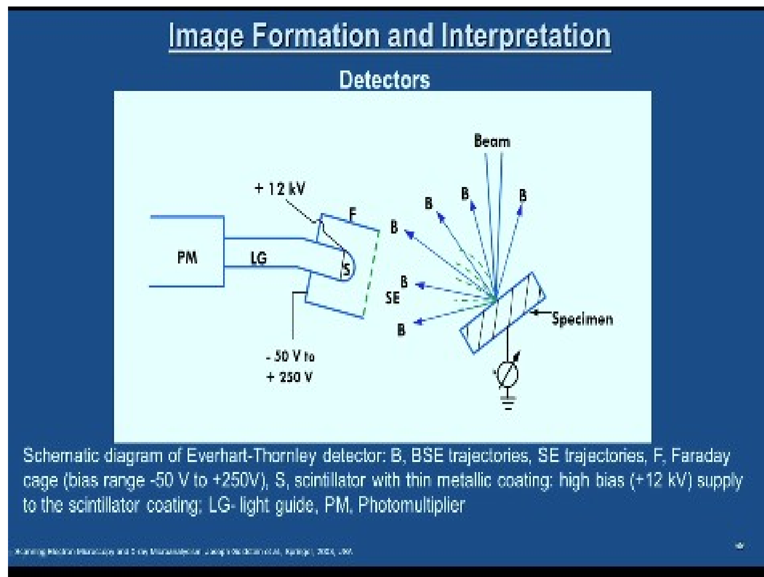

And what I am just showing the detector in the schematic, is a very popular detector called Everhart-Thornley detector. In 1960s, these detectors were invented from a Cambridge group and almost all the SEMs will have this ET detector, so-called ETD, ET detectors and this detector is capable of capturing both secondary electrons as well as back scattered electrons and as we have already seen we can this is the Faraday cage and this is a scintillator and this can be operated with the biased voltages either - 50 volt to + 250 volts depending upon what kind of signal we want to collect. And what you are now see here is signals are coming from the specimen. The solid lines are backscattered electrons. Brokered lines are secondary electrons which is coming from this specimen and that is how it is given in this description image description. So, you have back scattered electron trajectories, second electron trajectories, F is a Faraday cage which is this and you can ask you can give the bias voltage in the range of - 52 volt to +250 volts. S is scintillator with the thin metallic coating.

High bias +12 kilovolts applied to the scintillator coating and LG is light guide and PM is a fault of photomultiplier. So, this is a typical detector. Shematic ETD which is primarily used in most of the SEMs.

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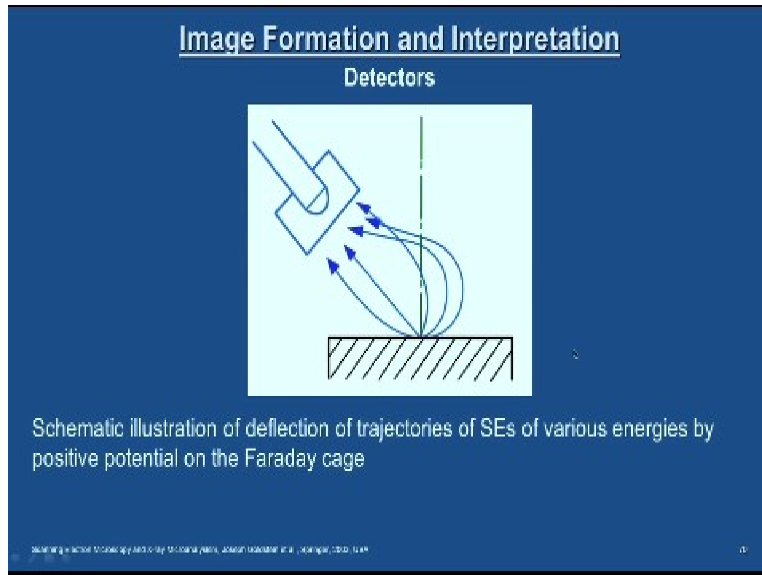

And this is the schematic illustration of a deflection trajectories of second electrons of various energies by positive potential on the Faraday cage. You see, in the ETD also which is capable of collecting both secondary electrons as well as the backscattered electrons. So, depending upon how we energize the detector or how it is biased, for example, if you the detector has the positive potential on the Faraday cage it will deflect the trajectories of secondary electrons like this and if it is a negative potential it will collect only the backscattered electrons. So, you can go back and then see one more important point. What you can understand from this, you see the back stated electrons the trajectories are quite straight line. Just to indicate that they are all high energy electrons as compared to secondary electrons. We will just look at the collection details of this with respect to the specimen geometry in due course.

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Image Formation and Interpretation

Detectors

Beam
+ 12 kV
F
B
B
B
B
PM
LG
S
B
SE
B
Specimen
- 50 V to
+ 250 V

Schematic diagram of Everhart-Thornley detector: B, BSE trajectories, SE trajectories, F, Faraday cage (bias range -50 V to +250V), S, scintillator with thin metallic coating; high bias (+12 kV) supply to the scintillator coating; LG- light guide, PM, Photomultiplier

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Image Formation and Interpretation

Detectors

Beam
Polepiece
Indirect BSE
E-T Detector
+ 250 V
Direct BSE
Chamber Walls
Specimen

Schematic illustration of the indirect collection of backscattered electrons by a positively biased E-T detector. The BSE strike the chamber walls, where they create secondary electrons. These SEs are collected by the E-T detector with high efficiency. Although nominally a contribution to the SE signal, these SE actually represent the BSE signal component because their number must rise and fall according to the behaviour of the BSE

Scanning Electron Microscopy and X-ray Microanalysis, Joseph Goldstein et al., Springer 2003, USA

So, another important information about the detector is shown in this schematic. What you are now seeing in this schematic illustration of the indirect collection of backscattered electrons by a positively biased ET detector. What you see here is, this is a pole piece and this is the electron beam which is coming and falling on the specimen surface and then you get signals coming out of the specimen surface like BSEs and SEs.

And what you are now seeing here is, you have this ET detector, a positively biased. So, that means it can collect both BSE as well as SE. The important information which you have to observe in this schematic is, you see all this backscattered electron which are shown the solid line which hits the bottom surface of the pole piece as well as the chamber walls, in that interaction they lose lot of energy.

In that process it produces SEII and SEIII of varying energies and then they are being collected by the ET detector. So, we have been telling that SE secondary electrons contribute to the image contrast. We will look we will see what this SEII and SEIII will represent in terms of image contrast and so on in the coming slides. So, what you have to appreciate from this schematic is, the BSE strike the chamber walls where they create secondary electrons. That is conversion of BSE to SE. These second electrons are collected by the ET detector with high efficiency. Although nominally a contribution to the secondary electron signal, these secondary electrons actually represent the BSE signal component because their number must raise and fall according to the behavior of the BSE. We will see why we talk about the number of these electrons is important.

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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

So, now we will come to the important aspect of this lecture is on contrast. Everybody is interested in good contrast images and we have already seen the basic definition of a contrast in the when we looked at the fundamentals. So, you know the definition of the contrast. So, in SEM, contrast can be influenced by a complex mix of characteristics of beam-specimen interaction, the properties of the specimen, the nature of the signal carriers and the position size and the response of the detector.

You see, n number of variables now come in to contribute or influence the image contrast. There are three different ways the contrast can be measured. One is number component and second is trajectory component and third is energy component. So, the number component what is that ? Different number of electrons leaving the specimen are the different beam location in response to changes in the specimen characteristics at those locations. So, it is that, how many electrons are coming out of this specimen surface is also an important aspect. And then you have trajectory component. That trajectory component refers to a contrast effect resulting from the paths the electrons travel after leaving the specimen. You see, the electrons are coming out of the specimen surface as I shown in the previous slide you can see that the electron travels in the very arbitrary path phase depending upon the energy it has.

If it is having a high energy, if it is a BSE, the trajectories are almost straight. And if it is a secondary electron, that is low energy electrons the trajectories are quite arbitrary. And even when the BSE goes and hits on the pole piece or a chamber walls, then it becomes again loses energy and becomes SEII and SEIII and then their trajectories are also quite arbitrary. So, the trajectory is very important.

The 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. So, what you have to appreciate here is, when it comes to the contrast of the imaging, these three components have an important role to play. Whether you get a SEM sorry secondary electron image or aback stated electron image.

So, we will see one by one, how this these three components play a crucial role in the case of contrast types. What are the contrast types we talked about? We talked about a composition contrast or atomic number contrast or Z contrast. They are all same. And then second type is a topographic contrast. There are two primary contrast types. We will look at those contrast types and it details and then how these three components contributes to each type under what conditions and then what is their contribution from the detectors and its location and geometry and so on in the next class. Thank you.

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