Indian Institute of technology Madras

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NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING

Lecture - 15

Materials Characterization

Fundamentals of Scanning Electron Microscopy

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Hello everyone welcome to this material characterization course. In the last class we just looked at the concept of scanning electron microscopy functions and this basic instrumentation and its controls and operator controls and so on. We will continue this discussion and then we will look at much more details about the electron beam specimen interactions and what is that going to affect your ultimate resolution and its effect on main in general imaging. So if you look at the controls which I talked about yesterday.

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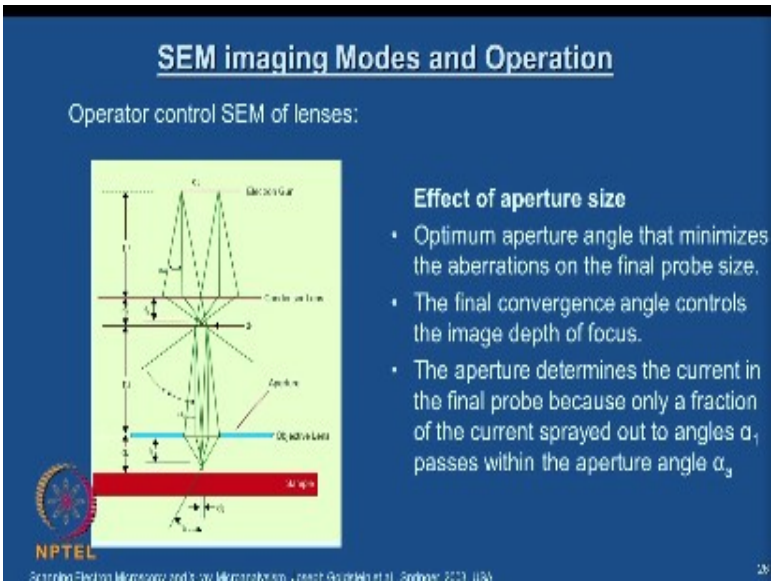

We will just quickly review this. We just started looking at the operator control in SEM of lenses. We have three primary parameters. One of them is the aperture. So, this schematic clearly shows that if the final aperture which basically controls the probe diameter which finally impinge on the sample the bite controlling this objective lens and this is what we just summarized here the optimum aperture angle that minimizes the aberration on the final probe size.

The final convergence angle controls the image depth of focus. The aperture determines the current in the final probe because only a fraction of the current spread out to the angles α_1 passes within the aperture angle α_a. So, if you look at this the initial spread of current this is what it is mentioned here the current sprayed in α_1 eventually its controls by this aperture chair and then it makes α_a this aperture angle and eventually it controls the probe size. This is one of the primary parameters which is in control of the operator and then we can see the next one the working distance.

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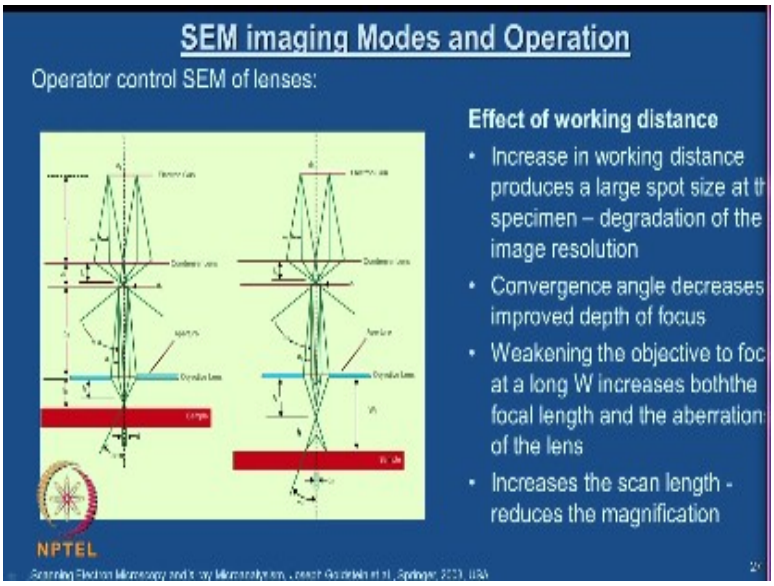

We also define this what is the working distance. It is the distance between the final aperture and the specimen surface and you can clearly see this effect of working distance from these two schematics which is quite evident that, if you increase the working distance you are increasing the probe size. You carefully look at it. You can see that the probe size is increase now and obviously it will have some significant effect on the resolution.

So we summarize this increase in working distance produces a large spot size at the specimen and which will cause degradation of the major solution and also you see that convergent angle decreases which will result in improved depth of focus and increasing working distance will also cause weakening the objective to focus at a long working distance ‘w’ which eventually increases both the focal length and the aberration of the lenses.

So, which is very clearly shown in this schematic and which also increases the scan length and which will cause reduction in the magnification as well. So, this is again a very important parameter which an operator can have a control on this and then take a appropriate decision depending upon what we are looking at what information we are looking at on this specimen surface.

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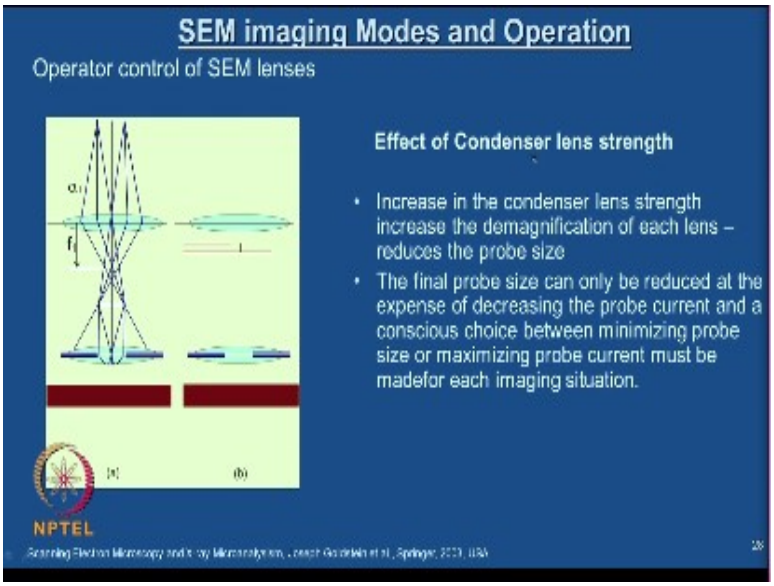

The third one is the condenser lens strength, which operator can control which is also is nicely shown in the schematic. If you increase the condenser lens strength which increases the demagnification of each lens which will cause again the reduction in the probe size. So, you can see that effect very clearly from the schematic. So, this is the first schematic is for a given field strength. If you increase it further you can see that, the final probe size is completely reduced. You can see this. This is the initial probe size with for a given field strength. But if you increase this from that and you see that there is a control of the probe diameter.

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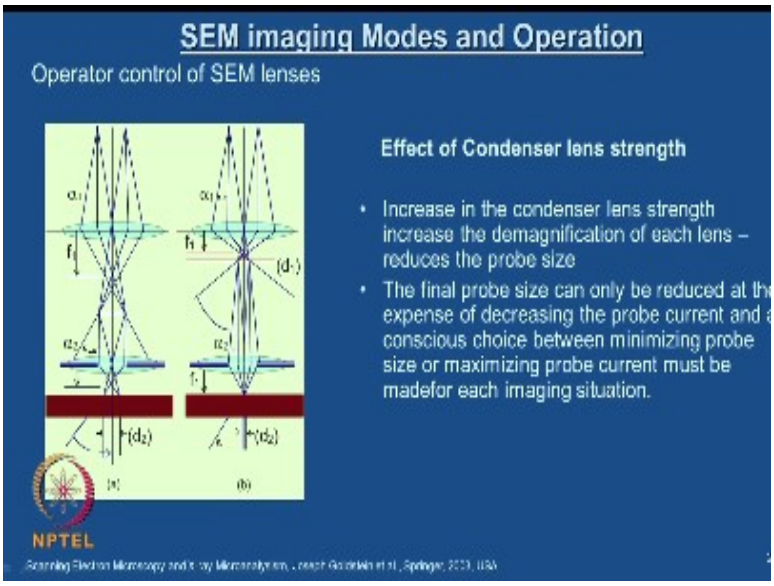

So, the final probe size can only be reduced at the expense of decreasing the probe current and a conscious choice between minimizing the probe size or maximizing the probe current must be made for each imaging situation. So, this is exactly I was just mentioning that, all these parameter controls has to be done as per the requirement for the appropriate information we are looking at from the specimen and it is completely in the user control.

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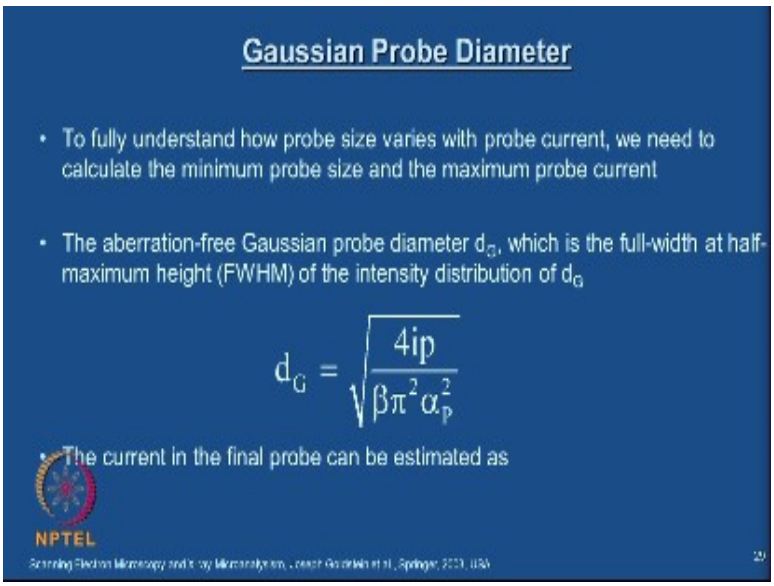

So, now we will move on to the probe diameter which we yesterday we quickly reviewed I just want to give an emphasize on the probe diameter again because whatever we have just seen before ultimately the parameters controls the probe diameter, which results in the complete resolution as well as and its effects on the imaging process. So, to fully understand how the probe size varies with the probe current, we need to calculate the minimum probe size and the maximum probe current. Say in the idealized situation the aberration free Gaussian probe diameter d_G which is the full width at half maximum height of the intensity distribution of d_G is given by $d_G = \sqrt{(4\, i_P / \beta\, \pi^2\, \alpha_P{}^2)}$.

The current in the final probe can be estimated as $i_P = \sqrt{(\beta\, \pi^2\, \alpha_p^2\, d_G^2 / 4)}$. If there were no aberration in the system, it would only be necessary to increase the convergent angle to increase the probe current at constant probe diameter. See, why we talked about this Gaussian probe diameter because this is the one which we will start with to mathematically quantify assuming there is no aberrational all but eventually that is not going to be the case. You are going to have the effect of each operations which we talked about in an electron optical system and then we can see how this Gaussian probe diameter is modified because of this aberration that is what we are looking at finally is a real probe diameter.

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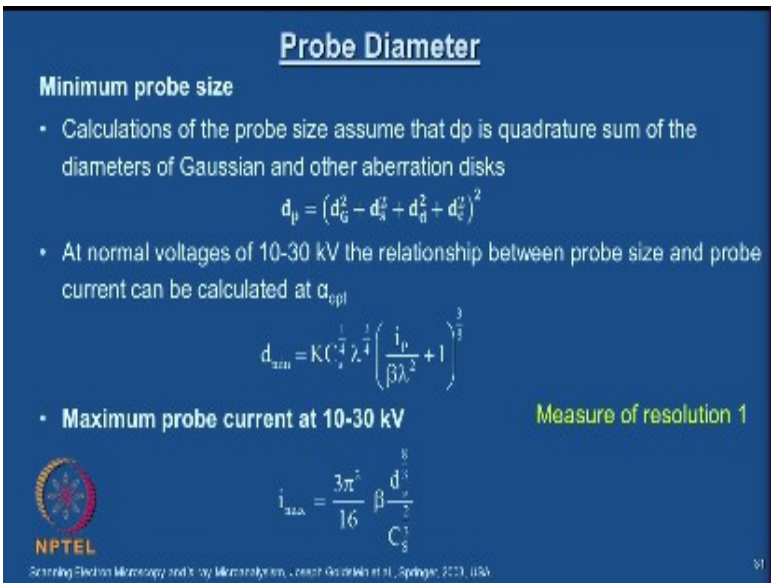

So, if you look at the minimum probe size involving all this aberrations, calculations of the probe size assume that d_P is quadrature sum of the diameters of Gaussian and other aberration disk. You look at this expression. There was a little bit of typos which was there in the yesteraday's presentation. I have made the corrections. You see that is equal to d_P where d_G is Gaussian probe diameter and d_S^2 spherical aberration diameter plus d_d^2 this is a diffraction disk plus d_C^2 which is chromatic aberration whole to the power half at normal voltages. Sorry I just did a mistake, This is whole to the power of it is square.

So, $d_P = (d_G^2 + d_s^2 + d_d^2 + d_C^2)^2$. At normal voltage of 10 to 30 kilo volt, the relationship between the probe size and the probe current can be calculated at α optimum (α_{opt}) which is

$$d_{min} = K C_s^{1/4} \lambda^{3/4} \{(i_P / \beta\lambda^2) + 1\}^{3/8}$$

where C_s is the spherical aberration coefficient. Here only considering this aberration, this expression is valid the it is assumed that other aberration do not have a significant influence. On that circumstances this expression is valid. Maximum probe current at 10 to 30 kilovolt, you have the

$$I_{max} = (3\pi^2 / 16) \beta (d_p^{8/3} / C_s^{2/3})$$

So, it is a kind of a maximum resolution one can obtain in the presence of other aberration effects.

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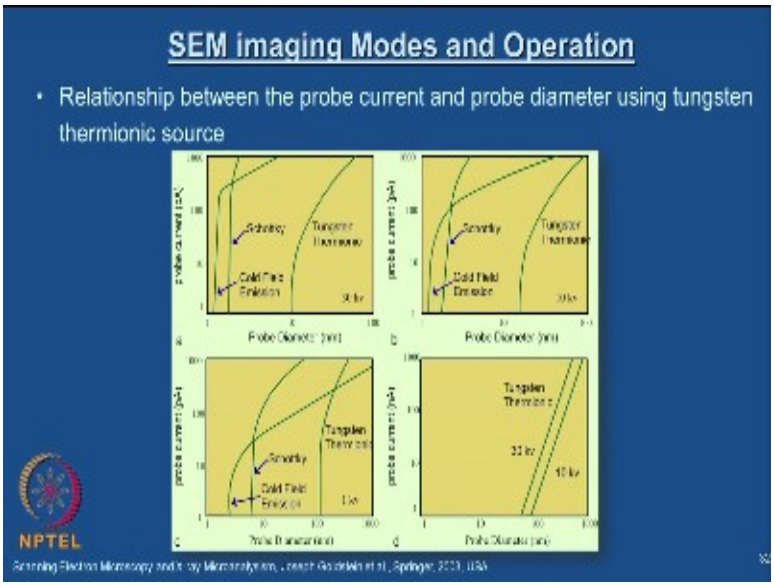

Now, we will look at the plots where the relationship between the probe current and the probe diameter using a tungsten thermionic source. You see, in the beginning we just looked at all the electron gun sources, I just mentioned there are the two types one is thermionic source another is field emission source. So, this how this probe current and probe diameter varies with the function thermionic source versus the field emission sources shown in all these four plots.

You can carefully look at it this the probe diameter which is varying from 1 to 100nanometers versus probe current which is a normal imaging condition and you can see that you have these thermionic field I mean thermionic source and as well as you have the field emission source. Obviously you can see that field emission source exhibit a superior probe diameter for at the given 30 kilo volt, which is a normal imaging and then you have another low KV imaging. You can see that similar plots are obtained and the plot C very low voltage imaging where you can see that how the probe current varies with the probe diameter and this is kind of plot, where mostly this kind of situation is used for the chemical analysis and you can see most of this plot shows that the field emission can source exhibit superior diameter compared to the thermionic source and then it also varies with the as a function of operating voltage. Just to give you an idea

how this electron sources controls the probe diameter as a function of operating voltage. We will look at this aspect in the imaging and under its resolution and so on in the due course.

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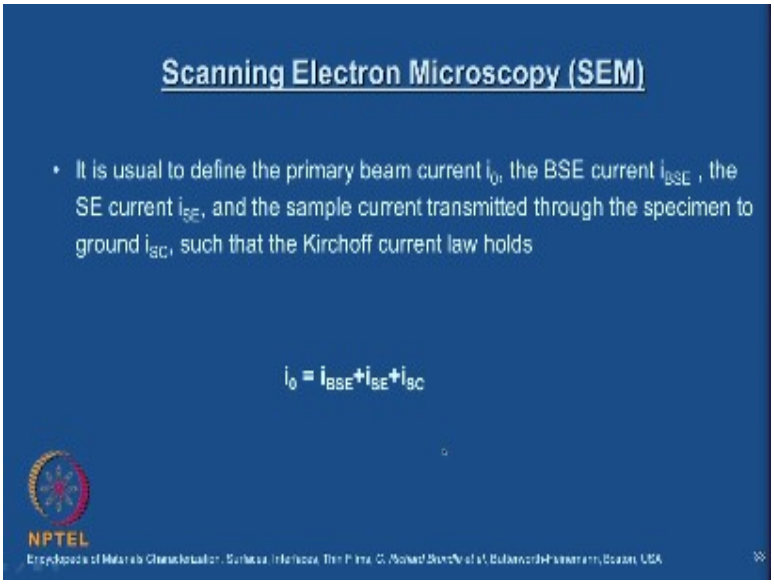

So, now we will look at the much more detail about this the probing current and so on. It is useful to define the primary beam current i_0 , the backscattered electron current i_{BSE} the SE current is i_{SE} and the sample current transmitted through the specimen to the ground is i_{SC} such that the Krichoff current law holds so the primary bream current can be s can be represented as

$$i_0 = i_{BSE} + i_{SE} + i_{SC}$$

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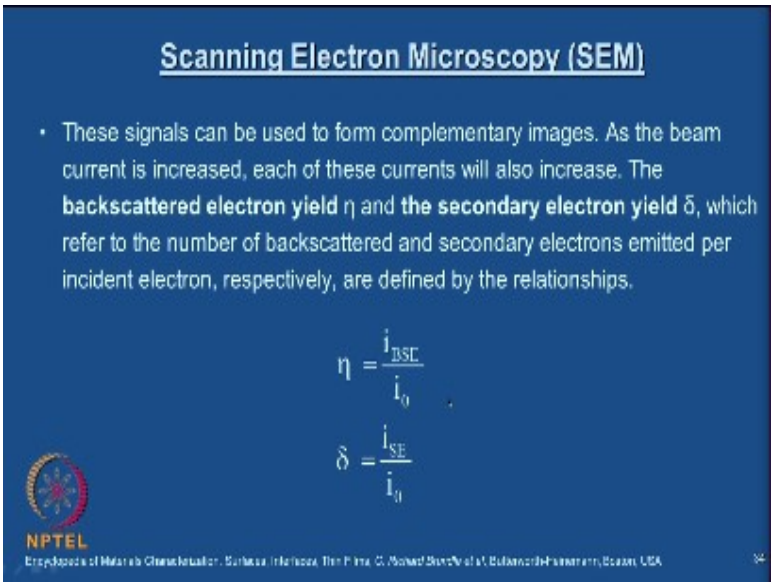

And we are interested in the signals which is coming out of the samples. So, basically how they are quantified. We know that a secondary electron signal and the backscattered electron signals are going to come out from the sample and how they are quantified. This is what is about we will see. So, these signals can be used to form a complementary image. As the beam current is increased, each of these currents will also increase. The backscattered electron yield **η** and the secondary electron lead **δ** which refer to the number of back scattered and secondary electrons emitted per incident electron respectively are defined by the relationship.

$$\eta = i_{BSE} / i_0$$

$$\delta = i_{SE} / i_0$$

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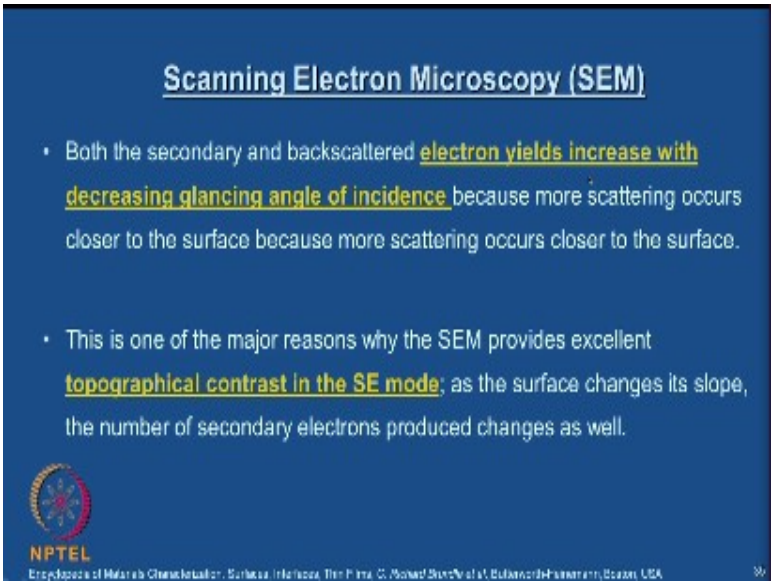

Both the secondary and backscattered electron yields increase with decreasing glancing angle of the incidents because more scattering occurs closer to the surface because more scattering occurs closer to the surface. This is one of the major reasons why the SEM provides an excellent topographical contrast in the SE mode; as the surface changes its slope the number of secondary electrons produced changes as well. This point we just discussed in the introduction of the SEM class as well. I just mentioned why only these two signals BSE and SE for widely used in SEM that is because only these two signals vary as a surface modulation or surface slope changes very sensitive to the surface unevenness. With the backscattered electrons this effect is not as prominent since to fully realize it the backscattered electron detector would have to be repositioned to realize it the backscattered detector would have to be repositioned to measure the forward scattering. This is an operation detail for detecting this signal. We will see how it is being actually done in the lab.

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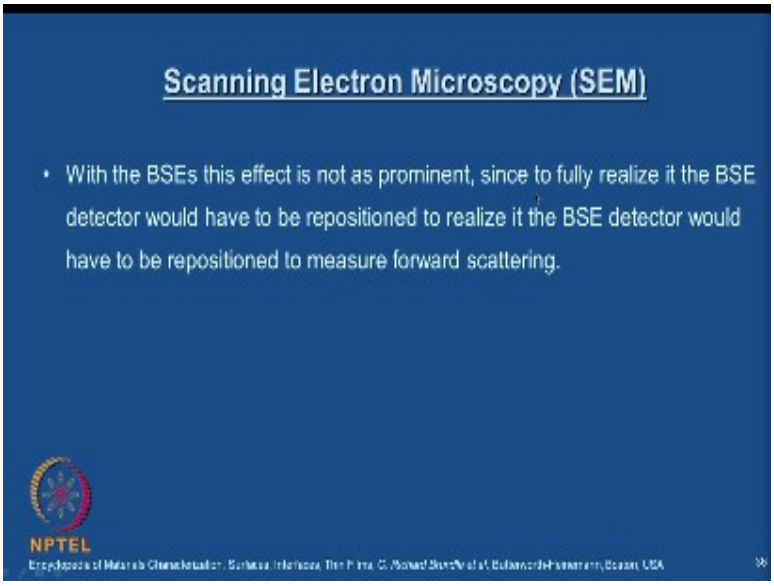

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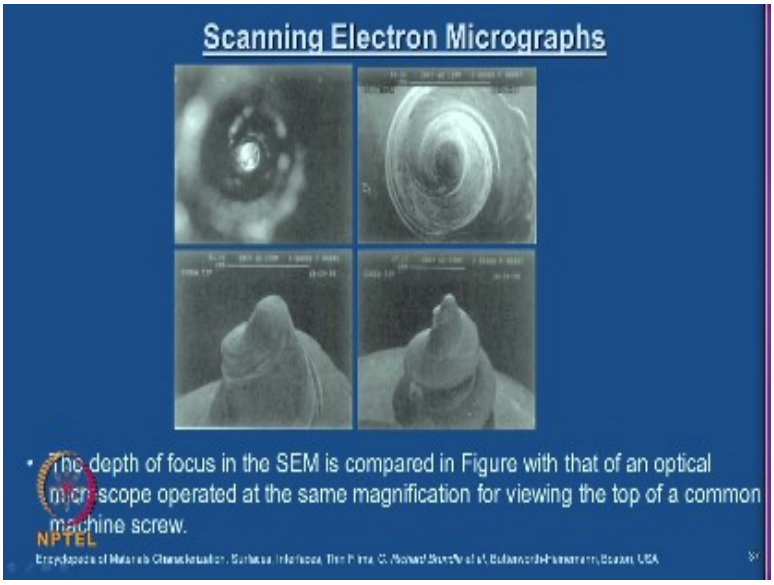

The another important aspect of this SEM be mentioned is a depth of focus and this set of micrograph clearly illustrate that aspect. So, what you see here is, a, this is a machine screw viewed at under the optical microscope and this is understanding electron microscope. You can see that in an optical microscope, you do not see any of this detail when you look at this crew from the top. You can see the all the other the circular details of the screw and C and D are taken

with the sides of the screw. You can see that the much more clear details are obtained using scanning electron microscope. This is just to illustrate that effect you have a very high depth of focus. And you know by now you know that why we get very good depth of focus.

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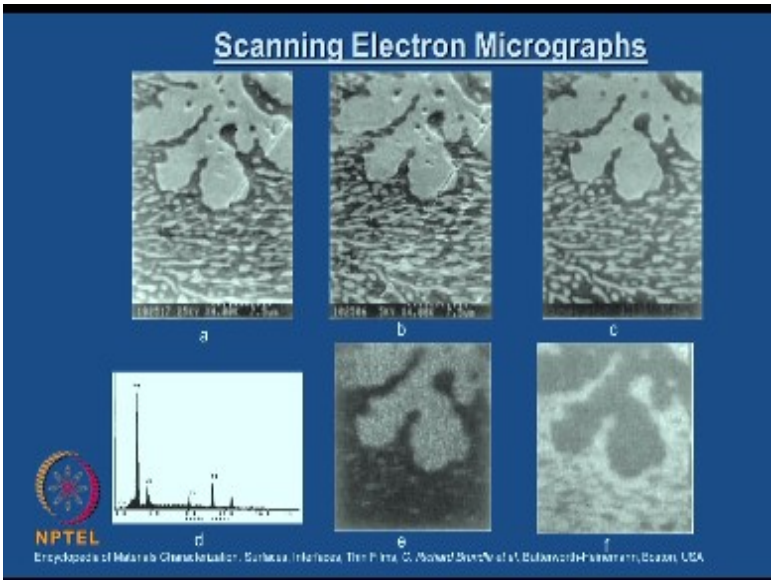

The another set of micrographs illustrates the effect of both secondary electrons as well as backscattered electrons. What you are seeing is it is a lead-tin alloy surfaces what we are seeing as bright as an eutectic, lead-tin eutectic. People who do not understand this metallurgy of this you can assume that there are two phases and you can clearly see that this particular micrograph is obtained at 25 kV and this micrograph of the same region is obtained at 5 kV and these two are obtained using secondary electrons and the same region was imaged using backscattered electron in this image C.

So, I would like you to look at this three images little more carefully. And what is the difference you are seeing and if you are able to figure out the differences then that means you have clearly understood the previous information what we have discussed. And if you are not able to catch that differences, I will help. You look at this the scratch here scratch mark here and look at this scartch mark here.

So, you see that these two are up to even though they are obtained using the secondary electron signals, that is a small difference. And also you see that this scratch is not at all visible as clearly as in the micrographs obtained by secondary electron signals. So, that clearly indicates that your secondary electrons are much more sensitive to the surface unevenness. And the difference between this 'a' and 'b' is because of further complications, because of the electron specimen interaction. What is that ? You see that this micrograph is obtained at lower kv, 5kv and this is obtained at 25 kv.

So, if you recall we just discussed in the beginning of this lecture, probably yesterday or day for yesterday, I had mentioned that the higher the operating voltage the severe will be the beam specimen interaction and then you also produce a c1 c2 and a c3 and these signals will get produced more if the electron beam specimen interaction is intense and when this sc2 and AC three signals they are not going to promote the topological details in fact when they come out of the specimen. They are going to interfere and reduce the resolution. That is what is happening here.

You can see that the scratch details are not as clear as what you see in the image 'b'. So, it is not that if you keep on increasing the operating voltage you are not you are going to obtain much more a clearer image there is an optimum voltage and other parameters under which circumstances you get the much more clear picture. So, this is just to explain that phenomenon and what you see in other images I mean this figure 'd' is a EDS spectrum and 'e' and 'f' are our maps elemental analysis maps and this particular about the spectroscopic details we will discuss later in a separate lecture series.

Right now my focus is only on the SEM imaging. We will talk about this elemental analysis and how it is done and what are the limitations with existing spectrometer and so on we will discuss in a separate lecture series.

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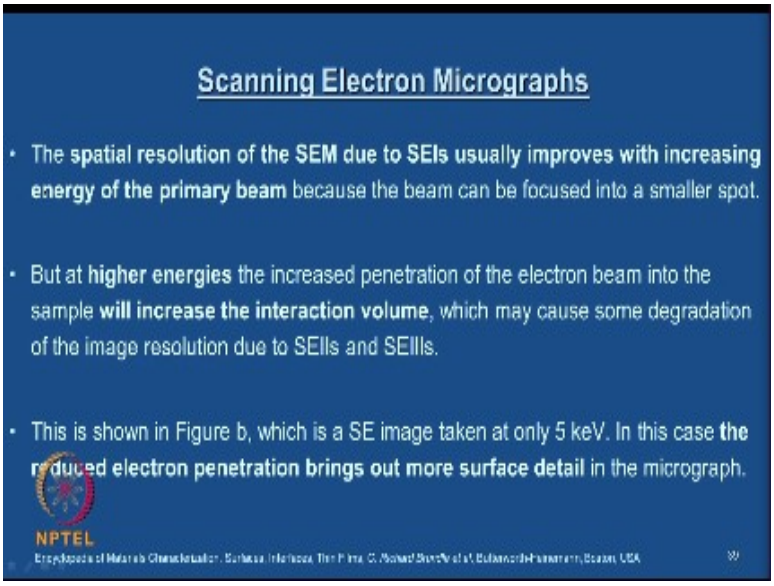

Now we will just summarize what we have just looked at in the previous slide. The spatial resolution of the SEM due to SEI usually improves with increasing energy of the primary beam because the beam can be focused into a smaller spot. But at higher energies the increased penetration of the electron beam into the sample will increase the interaction volume. We will quickly see in few minutes what is this interaction value about which may cause some degradation of the image resolution due to SEII and SEIII s. This is shown in image figure ‘b’ which is a secondary electron image taken at only 5 kilo electron volts. In this case the reduced electron penetration brings out more surface detailed in the micrograph.

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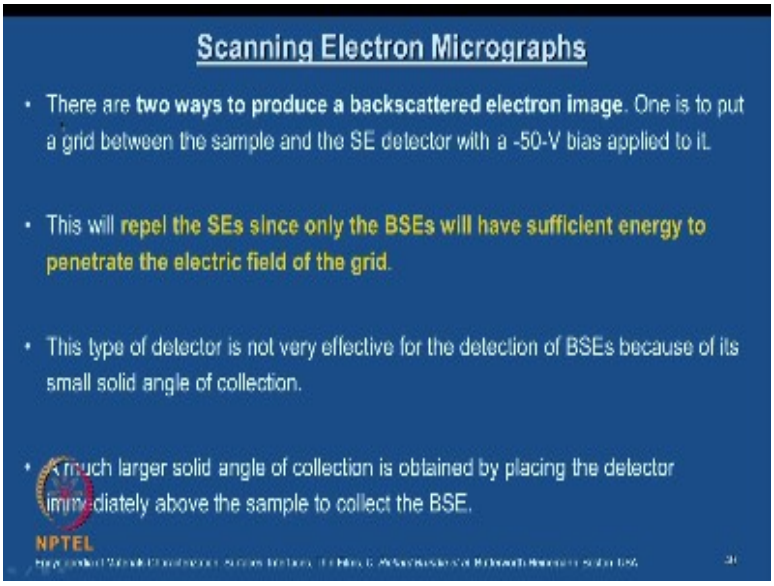

And if you look at the method of producing the backscattered electron image, there are two ways to produce BS image. One is to put a grid between the sample and the secondary electron detector with the negative voltage that is minus 50 volt bias applied to it. If you recall when I just introduced the instrumentation schematic where I said that if you put positive voltage, it will collect both BSE and SE. If you put negative voltage it will repel and then it will correct only one.

So, similar thing so that is the bias. This will repel the SEs since only the BSE will have sufficient energy to penetrate the last electric field of the grid. This type of detector is not very effective for the detection of BSEs because of its small solid angle of the collection. We will look at the detector system and its details little more as we go along and this right now we are discussing about how this signals are collected and how what are the immediate effect of these two individual signals on its image formation.

A much larger solid angle of collection is obtained by placing the detector immediately above the sample to collect the BSE. Two types of detectors are commonly used here. One type uses partially depleted n-type silicon diodes coated with a layer of gold, which convert the incident BSEs into electron hole pairs at the rate of one per 3.8 electron volt. Using a pair of silicon detectors makes it possible to separate the atomic number contrast from topographic contrast.

The other detector type, the so-called scintillator photomultiplier detector uses a material that will fluoresce under the bombardment of the high energy BSEs to produce a light signal that can further amplified.

So, these are all some of the specific operations of the type of detectors which eventually give the image in the CRT. We will look at this detectors separately and we will talk about all the functions much more detail in the new course.

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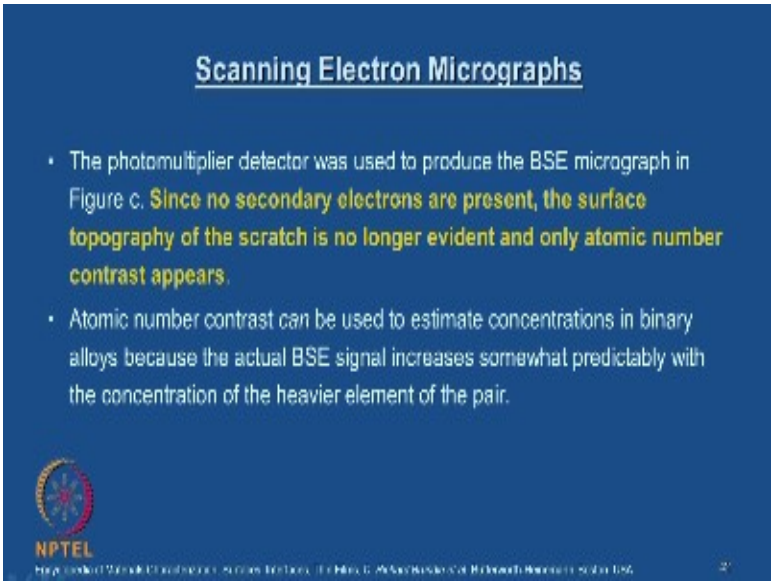

The photomultiplier detector was used to produce BSE micrograph in Figure-C. What we have just seen in two slides before, since no second electrons are present the surface topography of the scratch is no longer evident and only an atomic number contrast appears. Atomic number contrast can be used to estimate the concentrations in binary alloys because the actual BSE signal increases somewhat predictably with the concentration of the heavier element of the pair.

So, this point is about the material detail. And what you have to understand this BSE is sensitive to atomic number that we will anyway we will talk about much more detail when we discuss the image contrast and contrast mechanisms and so on. Now, we will dive at our focus to the very

important aspect of imaging, that is electron beam specimen interaction. In it involves lot of physics are scattering physics. We need to understand this clearly then only you will be able to interpret all the images which we are going to see.

So, I would like to request all of you to pay much more attention to look at this particular section is more fundamental it may be very difficult to understand in the beginning but if you look at them again and again and if you are finding it difficult to follow this, I request you to go through some of the basic physics book about the scattering phenomenon and then come back to this section, then things will be alright.

So, as the beam of electron enter the specimen, they interact as negatively charged particles with the electrical fields of the specimen atoms. The positive charge of the protons is highly concentrated on the nucleus while the negative charge of electrons is much more dispersed in a shell structure. The beam electron specimen atom interaction can deflect the beam electrons along the along a new trajectory which is considered elastic scattering causing them to spread out laterally from the incident footprint.

I am going to show you some of the schematic regarding this to understand the point 3 what we are now talking about. So, the elastic scattering after numerous events actually result in beam electrons leaving the specimen, process called backscattering. It gives a kind of a definition for the backscattering that is, the elastic scattering after numerous events actually result in a beam electrons leaving the specimen. A mathematical description of elastic scattering process at angle greater than a specified Φ_0 as the form

$$Q (> \Phi_0) = 1.62 * 10^{-20} (Z^2 / E^2) \cot^2 (\Phi_0 / 2)$$

So, this is events scattering events greater than Φ_0 divided by the electron which is atoms per centimeter square, where Q is called the cross-section which is in centimeter squared for elastic scattering that is probability of elastic scattering which is given in this form.

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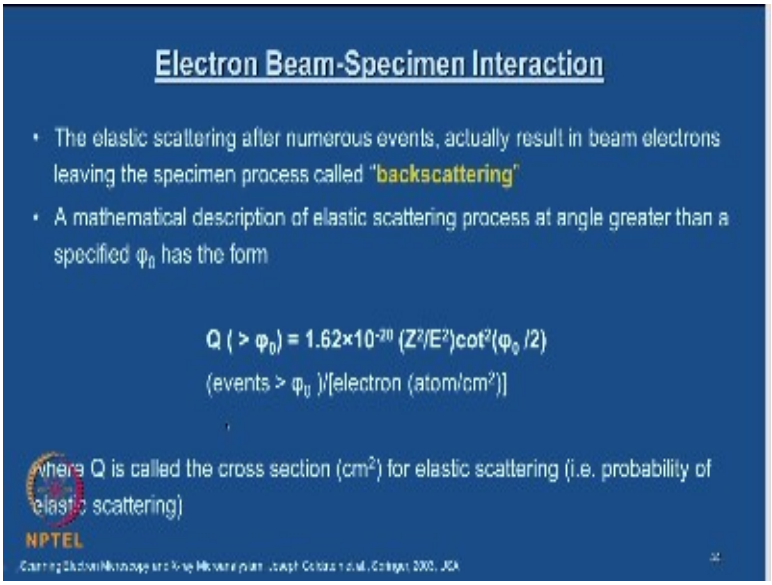

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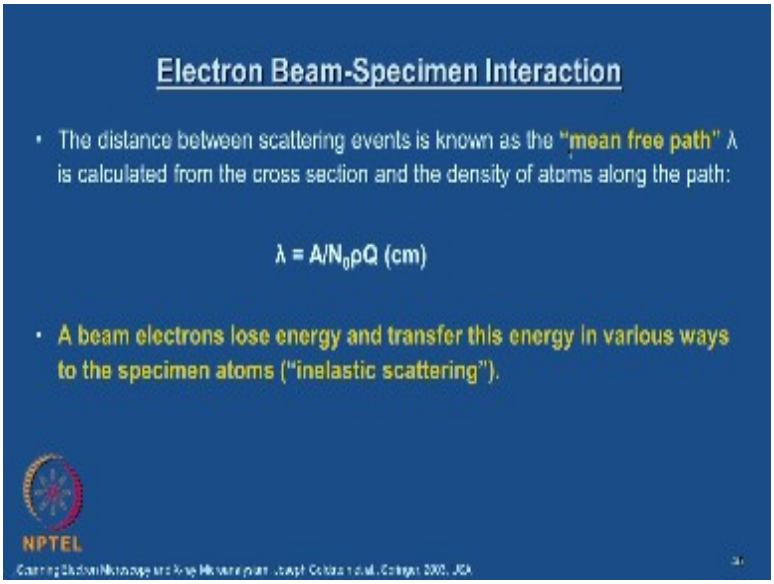

The distance between scattering events is known as the mean free path lambda (λ) is calculated from the cross-section and the density of the atoms along the path.

$$\lambda = A/N_0\ \rho\ Q\ (cm)$$

A beam electrons loose energy and transfer this energy in various ways to the specimen atoms which is nothing but inelastic scattering. See, you see in an SEM we get a characteristic x-rays for chemical analysis like we discussed in the beginning.

The basic fundamental physics of that event is what we are now discussing. This the beam of electron lose energy and transfer this energy in various ways. So, one of the ways is like know you are getting attacked rustic x-rays and you have SEs, BSEs and all the signals solve basically inelastic scattering. This transfer takes place gradually. So that the beam electrons propagate through many atom layers into the specimen before losing all their energy.

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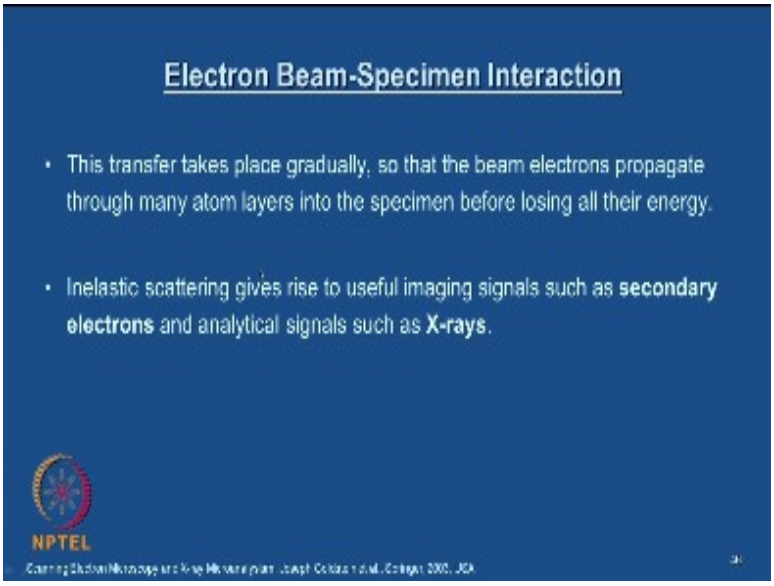

So, this the loss of energy of the electron beam is not going to be instantaneous. So, it will be more I mean the you can see that how some of the models are being made for this how the electron beam is losing energy which I will show you in few minutes. We will form that we will get an idea how the electron beam after impinging on the specimen surface loses energy

gradually as a function of interaction volume. Inelastic scattering gives rise to useful imaging signals such as second electrons and analytical signals are just such as x-rays.

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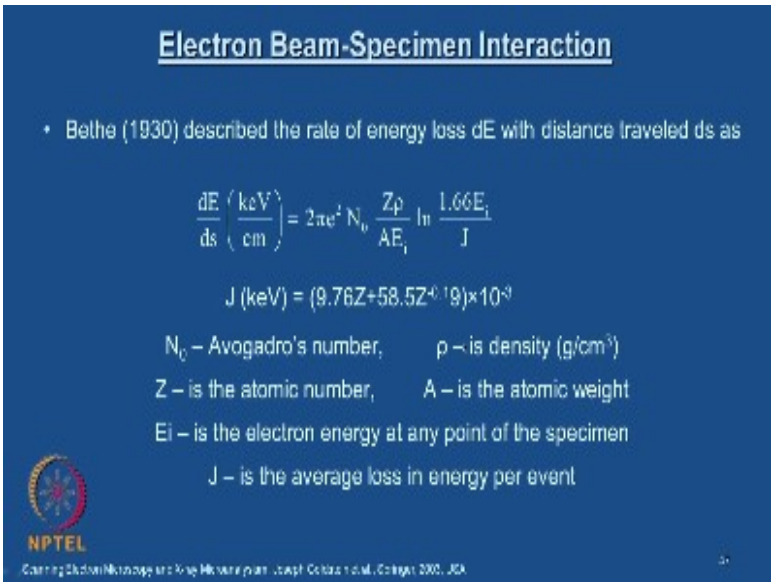

bathe described in 1930, the rate of energy loss dE with the distance travelled ds as dE by ds the energy is given in kilo electron volt and the distances in centimeter,

$$\mathbf{dE/ds\ (KeV/cm) = 2\pi e^2\ N_0\ (Z\ \rho\ /\ A\ E_i)\ ln\ (1.66E_i\ /\ J\ )}$$

where $$\mathbf{J\ (keV) = (\ 9.76Z + 58.5\ Z^{-0.1} * 9)\ *\ 10^{-3}}$$

where $\mathbf{N_0}$ is called Avogadro's number, **ρ** is the density, **Z** is the atomic number, **A** is the atomic weight, $\mathbf{E_i}$ is the electron energy at any point of the specimen, **J** is the average loss in energy per event.

It is just this expression simply tells you how this energy loss takes place and how we can visualize quantitatively with all these variables. I just want you to appreciate that point rather than getting into the details at this mode.

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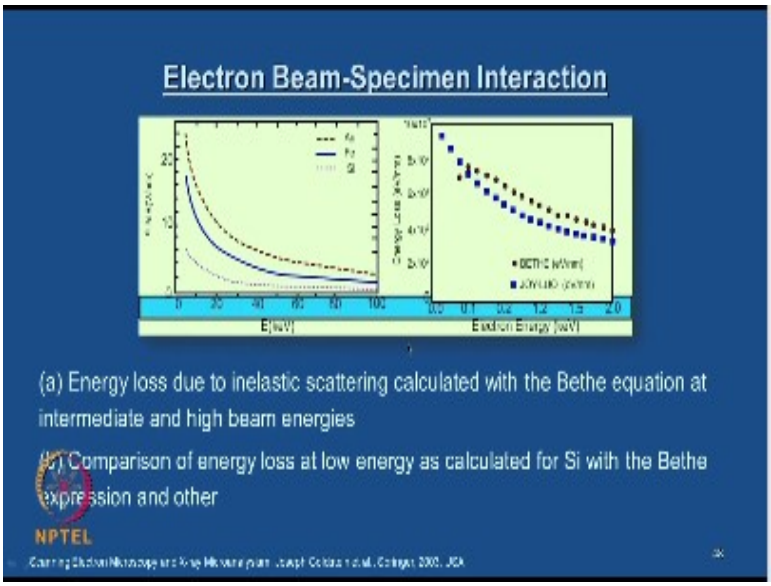

So, you can see that two plots which are based on this Bathe equation how the energy loss due to an elastic scattering is calculated. You can see that plot a is energy loss due to inelastic scattering calculated with the Bethe equation at intermediate and high beam energies for all this elements. And the plot B is the comparison of energy loss at low energy as calculated for silicon with Bathe expression and others. So how this energy loss occurs as the function of the electron volt.

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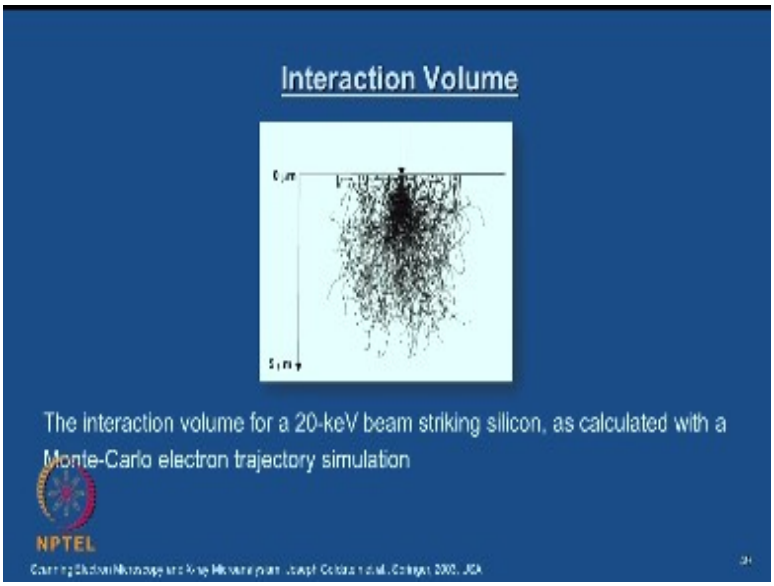

Now, what you are going to see is, we will look at what is this interaction volume and the electron beam comes and interacts with the specimen surface. And what you are now seeing is the similation is the interaction volume for a 20 kilo electron volt beam striking the silicon as calculated with a Monte Carlo electronic trajectory simulations, a numerical simulation, and what you see is you see that to know there is there are thick black line and then very light black lines with just getting inside this specimen to the order of what few microns. So, this is happening in a three dimensional.

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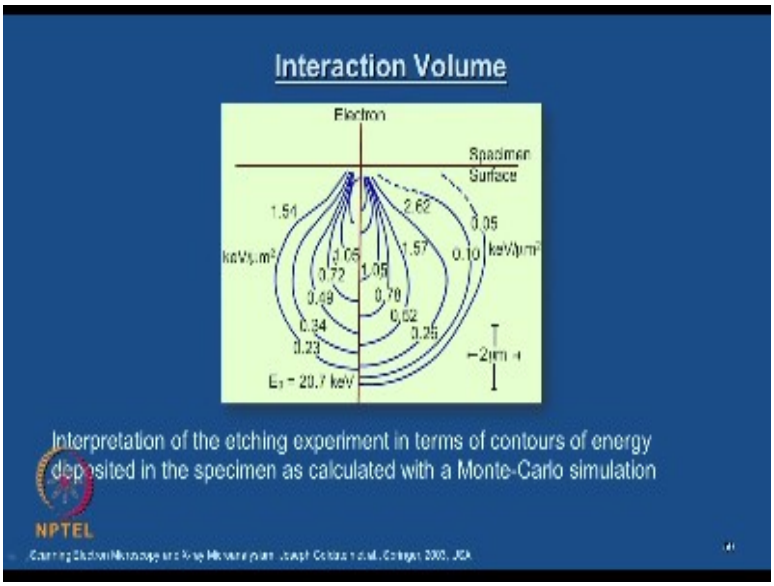

So, let us try to understand how this happens. This is, you can see that the another schematic showing that, this kind of interaction volume is interpreted through an etching experiment in terms of contours of the energy deposited in the specimen as calculated with the Monte Carlo simulation. So. the left-hand side is how the energy varies as a function of depth using an etching experiment. What is this is etching experiment? People have taken some of the low atomic number of materials like poly methyl meth cry late kind of a specimen and then they just do an etching experiment within a bombardment of electron how it just I mean damage this molecular polymeric molecules and then how it that the intensity of the damage decreases from the surface to the core. And that is done with that model that is called etching experiment. And then the left hand side is the experimental measurement how the energy varies from the surface to the core in the three dimension. And the right-hand side is the same thing is done numerically through Monte Carlo simulation and then you get some kind of very close agreement with this.

So, the important point to appreciate here is you get a kind of an idea what is an interaction volume is and how it occurs three-dimensionally and what are its dimensions. So, it gives you a kind of a basic outline about an interaction volume. And please remember whatever we are now

just showing is only a static image and actually it is happening dynamically between the interaction between electron beam and the surface.

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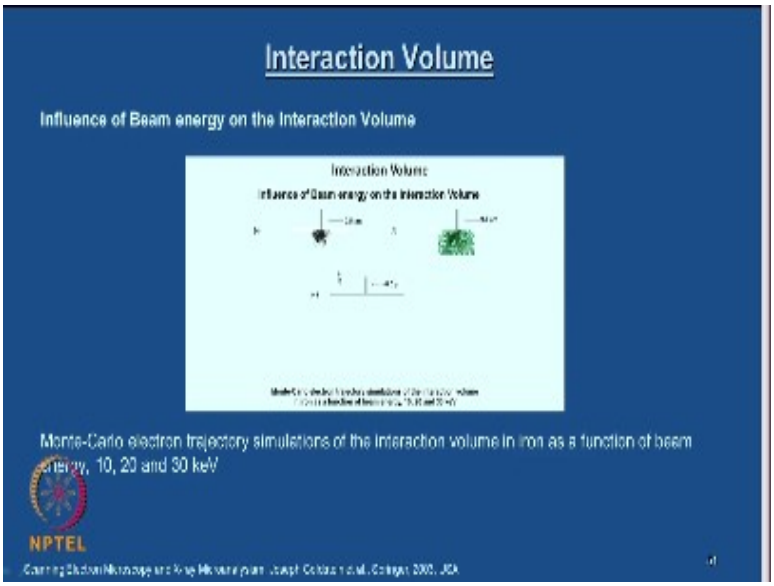

And, I will just show you few more schematic which you have the just excuse me it means. So, I would like to show this as a function of electron beam energy versus interaction volume. You actually what you will see that the as the electron energy increases the interaction volume also will increase and somehow this simulation is not working right now.

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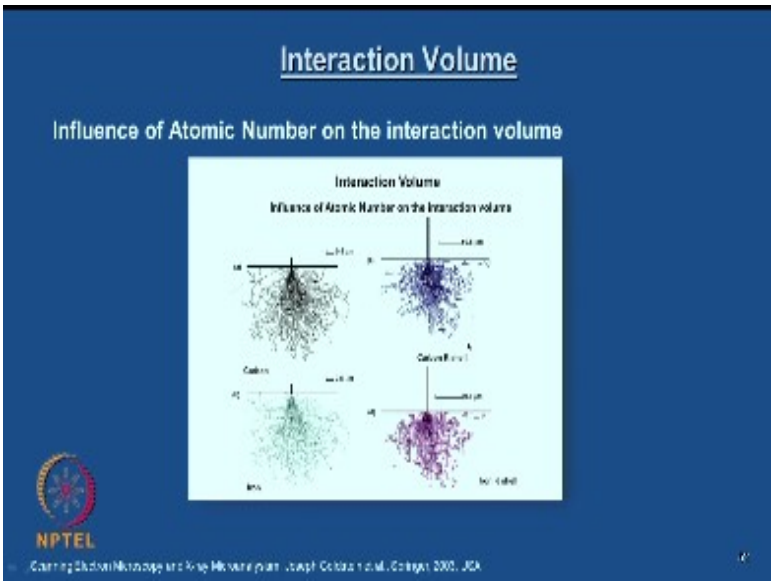

So, you can see that the same effect of atomic number also you can see. Influence of atomic number on the interaction volume. You can see it for different material. Here it is a carbon and this is for a carbon K shell and then you have the iron and then you have the iron and K shell. You can also see that as the atomic number increases the linear dimension decreases. That is a very much understandable because the that that is because your a scattering cross section varies as the atomic number increases.

So, you can see that the linear dimension also decreases in accordance with that number. And you can see that a similar another systems same effect for a silver L shell and then you have uranium and uranium M shell and so on. So, what I try to tell here is, depending upon the atomic number as well as the energy of the electron beam which is impinging on this sample, your interaction volume is going to change. And the scattering physics involved is little more complicated. And this has got a significant influence on your image resolution and the kind of details one can get from the specimen surface. That is all I just want to emphasize here and then we will look at the scanning action, how this the electron beam is scanning the surface and how exactly the image is formed all those details we will see it in the next class. Thank you.

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