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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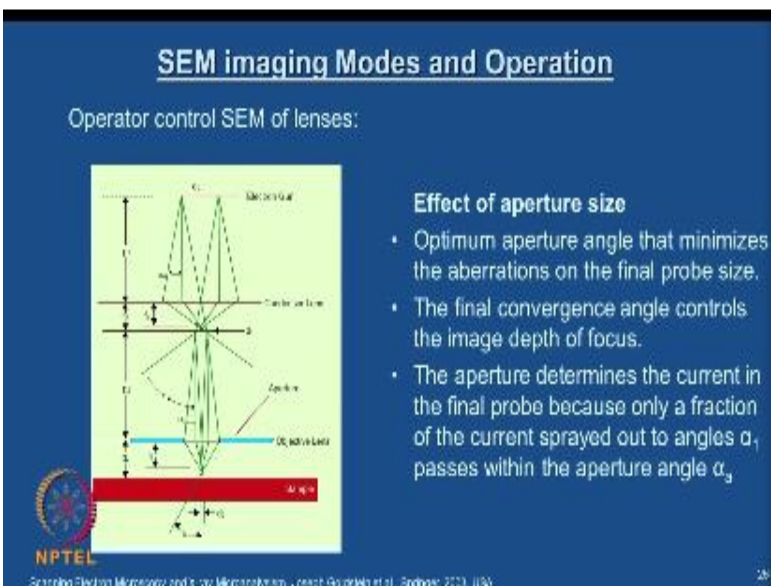

We will just quickly review this we just started looking at the operator control in ACM of lenses we have three primary parameters one of them is the aperture so this schematic clearly shows that if the final aperture which basically controls the probe diameter which finally impinge on the sample the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

The final conversion 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 alpha one passes within the aperture angle alpha a so if you look at this the initial spread of current this is what you just mentioned here the current sprayed in alpha one eventually its controls by this upper chair and then it makes alpha e 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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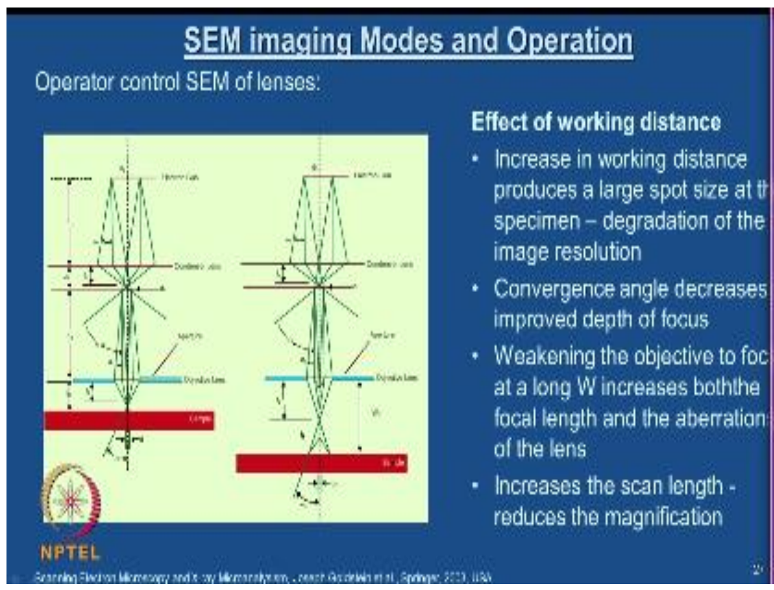

We also define this what is the working distance it is the distance between the final aperture and the specimen surface and you can clearly see this effect of working distance from these two schematics which is quite evident that if you increase the working distance you are increasing the probe sighs 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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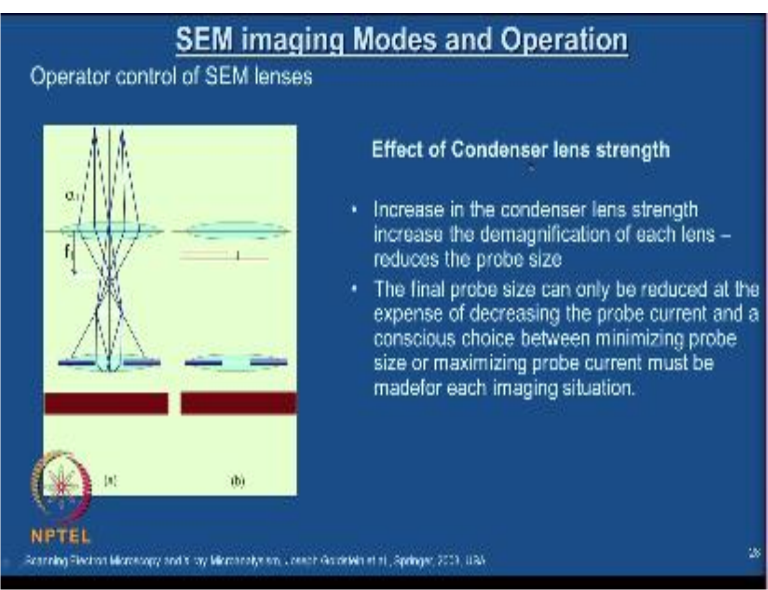

The third one is the condenser lens strength which operator can control which is also is nicely shown in the schematic if you increase the condenser lens strength which increases the D magnification of each lens which will cause again the reduction in the probe size so you can see that effect very clearly 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 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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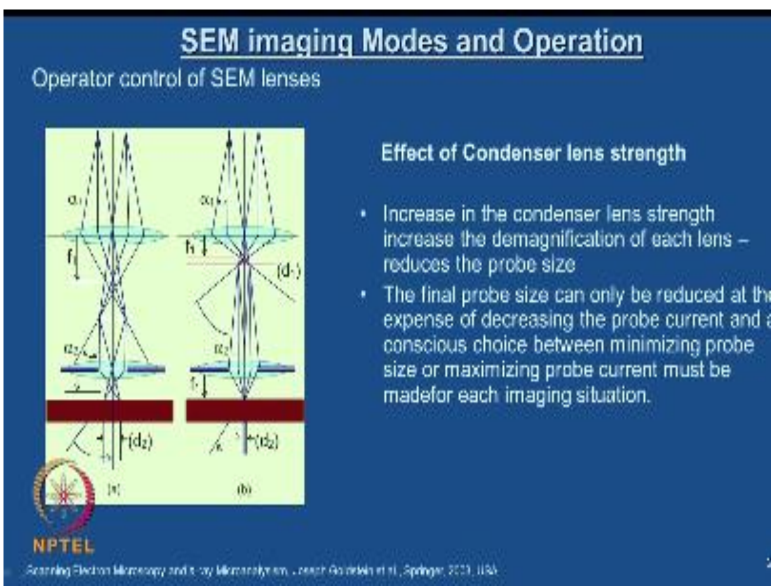

So the final probe size can only be reduced at the expense of decreasing the probe current and a conscious choice between minimizing the probe size or maximizing the probe current must be made for each imaging situation so this is exactly I was just mentioning that all these parameter controls has to 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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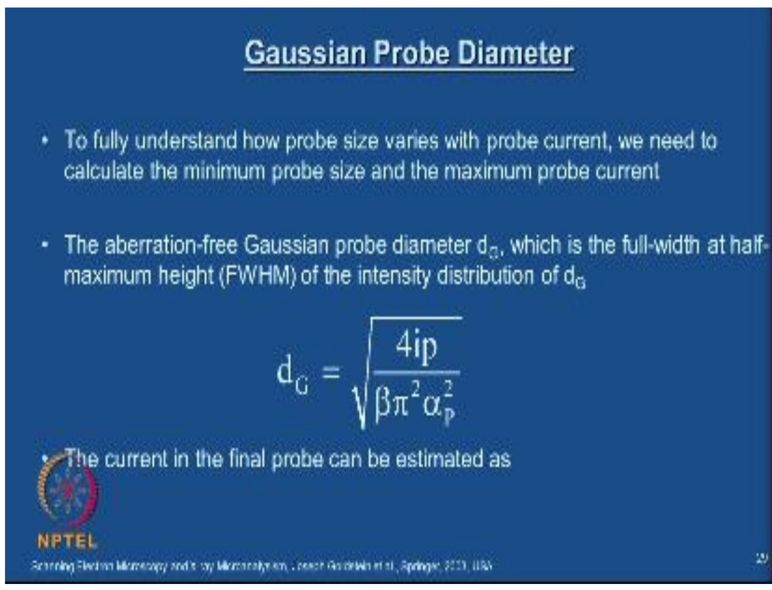

So now we will move on to the probe diameter which we yesterday we quickly reviewed I just want to give an emphasis on the probe diameter again because whatever we have just seen before ultimately the parameters controls the probe diameter which results in the complete resolution as well as and its effects 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 DG is given by DG is equal to $\sqrt{IP} / \beta \pi \alpha p^2$.

The current in the final probe can be estimated as I p = $\sqrt{}$ β π^2 α_p^2 and DG ^{2} / 4 if there were no abrasions 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 aberrations that is what we are looking at finally is a real probe diameter.

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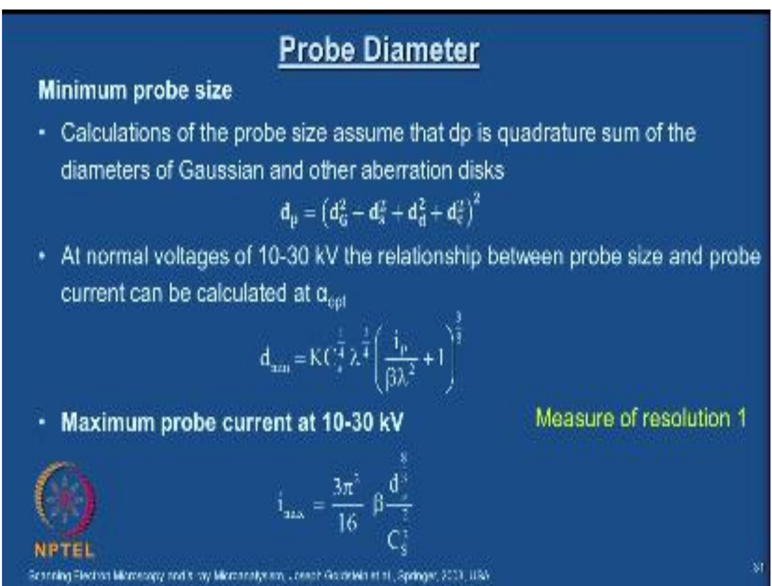

So if you look at the minimum probe size involving all this abrasions calculations of the probe size assume that DP is quadrature some of the diameters of Gaussian and other aberration you look at this expressions there was a little bit of typos which was there in the histories presentation I have made the corrections you see that DP is equal to DG where DG is Gaussian probe diameter and DA^2 spherical aberration diameter + DD^2 this is a diffraction disk + DC which is chromatic aberration whole to the power half at normal voltages sorry I just did a mistake this is not hold the power of it is square.

So DP is equal to dt square plus d square plus d d square plus DC square voter power square 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 which is d minimum is equal to k CS to the power half δ to the power 3 by 4 times IP by $\beta\delta^2$ plus 1 whole to the power 3 by 8 where CS is the spherical aberration coefficient here only considering this abrasion this expression is valid the it is assumed that other abrasions do not have a significant influence on that circumstances this expression is valid maximum probe current at 10 to 30 cloveult you have the I max equal to 3 PI square x 16 x beta into DP to the power8 by 3 divided by c s to the power 2 by3 so it is a kind of a maximum resolution one can obtain in the presence of other operation effects.

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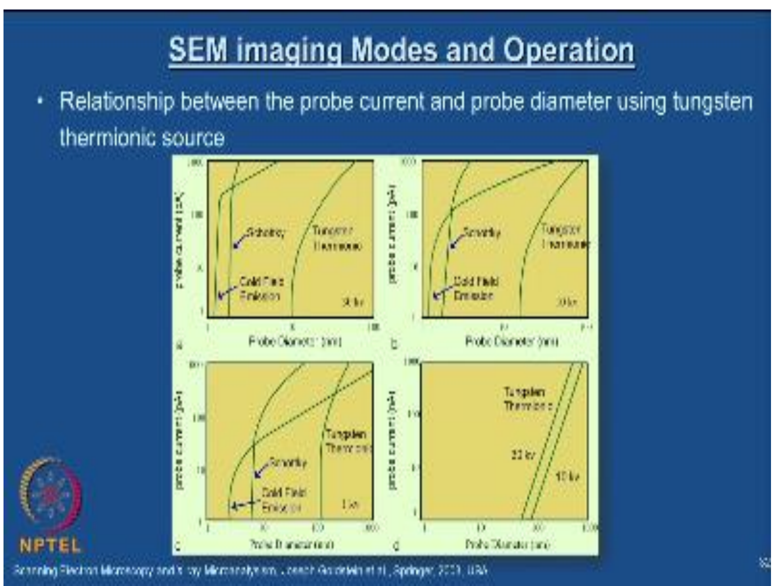

Now we will look at the plots where the relationship between the probe current and the probe diameter using a tungsten thermionic source you see in the beginning we just looked at all the electron gun sources, I just mentioned there are the two types one is thru bionic source another is field emission 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 it is a normal imaging condition and you can see that you have these harmonic 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 nazi shows 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 it under its resolution and so on in the due course.

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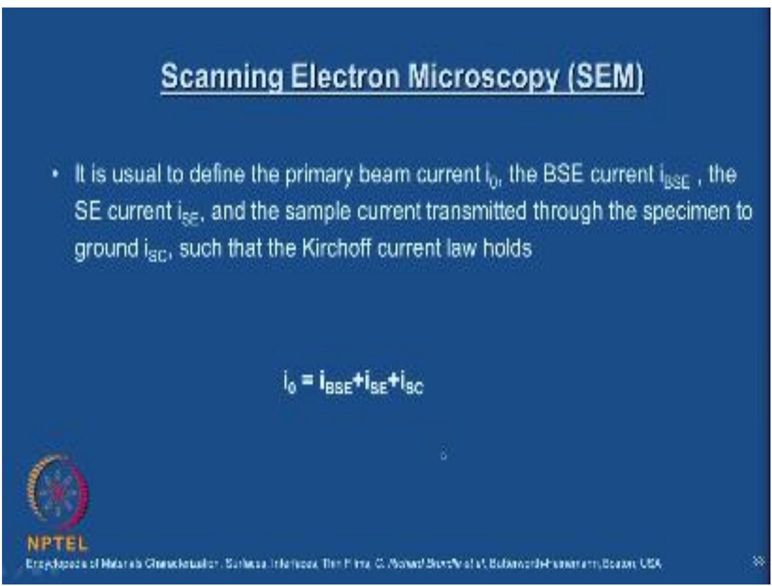

So now we will look at the much more detail about this the probing current and so on it is useful to define the primary beam current I not the backscattered electron current I BSE the AC current is C and the sample current transmitted through the specimen to the ground is C such that the tech of current law holds so the primary bream current can be s can be represented as a summation of IBS c plus I AC plus I s SC.

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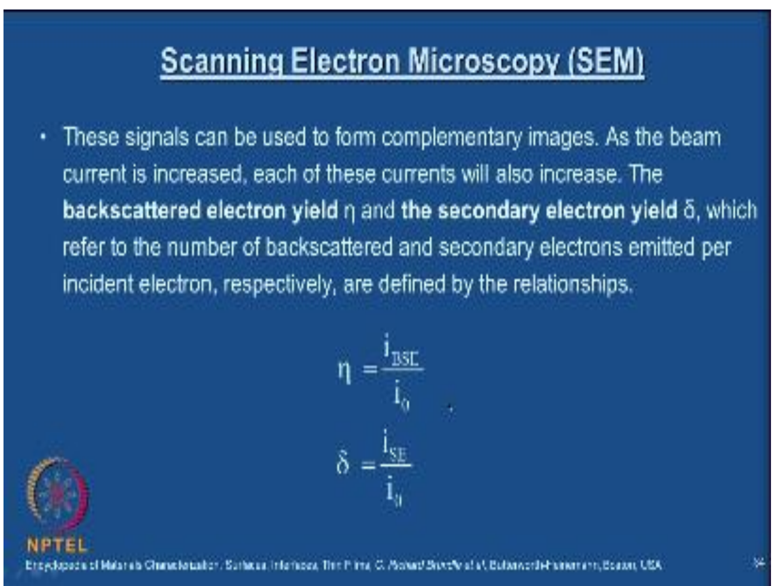

And we are interested in the signals which is coming out of the samples so basically how they are quantified we know that a secondary electron signal and the backscattered electron signals are going to come out from the sample and how they are quantified this is what is about we will see so these signals can be used to form a complementary images as the beam current is increased each of this 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 where incident electron respectively are defined by the relationship.

Where η is equal to i BAC that is the backscattered electron current / I not similarly the secondary electron e δ is ISE / I_0.

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Scanning Electron Microscopy (SEM)

- Both the secondary and backscattered **electron yields increase with decreasing glancing angle of incidence** 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 excellent **topographical contrast in the SE mode**; as the surface changes its slope, the number of secondary electrons produced changes as well.

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 ACM 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 be SE 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.

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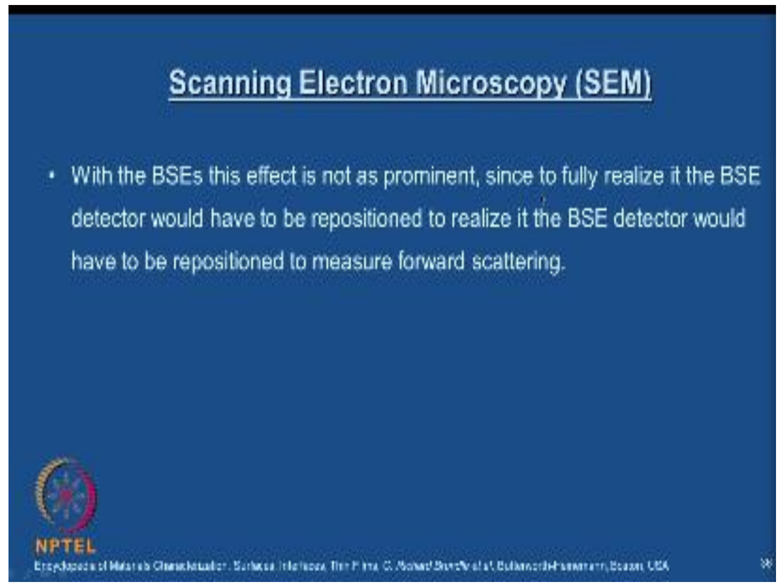

To fully realize it the backscattered electron detector would have to be repositioned to realize it the backscattered detector would have to be repositioned to measure the forward scattering this is an operation detail for detecting this signal we will see how it is being actually done in the lab.

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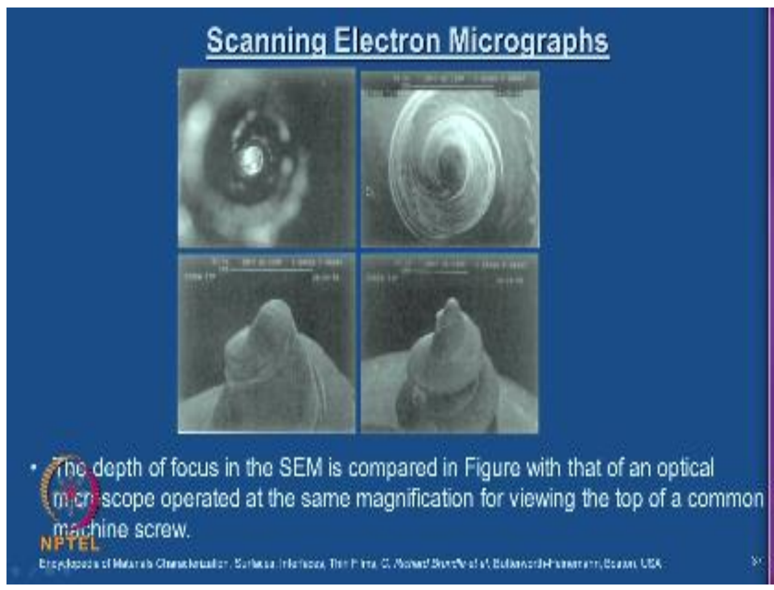

The another important aspect of this SEM be mentioned is a depth of focus and this set of micrograph clearly illustrate that aspect so what you see here is at his is a machine screw viewed at under the optical microscope and this is understanding electron microscope you can see that in an optical microscope 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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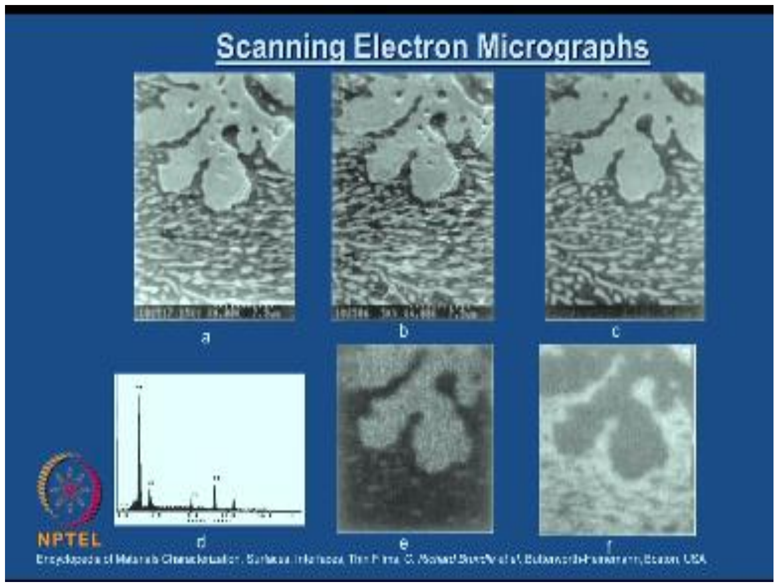

The another set of micrographs illustrates the effect of both secondary electrons as well as backscattered electrons what you are seeing is it is a legend all I surfaces what we are seeing as bright as a new tactic let in eutectic people who do not understand this metallurgy of this you can assume that 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 see.

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 notable to cash that differences I will help you look at this the scratch here scratch for here and look at this crash 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 update 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 kv5kv and this is obtained at 25 kv.

So if you recall we just discussed in the beginning of this lecture I probably yesterday our 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 layer 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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Scanning Electron Micrographs

- The spatial resolution of the SEM due to SEIs 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, which may cause some degradation of the image resolution due to SEIIs and SEIIIs.
- This is shown in Figure b, which is a SE image taken at only 5 keV. In this case the reduced electron penetration brings out more surface detail in the micrograph.

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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 a c1usually 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 AC 2 and s III s this is shown in image figure B.

Which is a secondary electron image taken at only 5 kilo electron volt in this case the reduced electron penetration brings out more surface detailed in the micrograph.

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Scanning Electron Micrographs

- There are **two ways to produce a backscattered electron image**. One is to put a grid between the sample and the SE detector with a -50-V bias applied to it.
- This will **repel the SEs since only the BSEs will have sufficient energy to penetrate the 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 collection.
- A much larger solid angle of collection is obtained by placing the detector immediately above the sample to collect the BSE.

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And if you look at the method of producing the backscattered electron image there are two ways to produce v s 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 BAC and SC if you put negative voltage it will ripple and then it will correct only one.

So similar thing so that is the bias this will ripple the ACS since only the BSS 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 BAC 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 per3.8 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 the 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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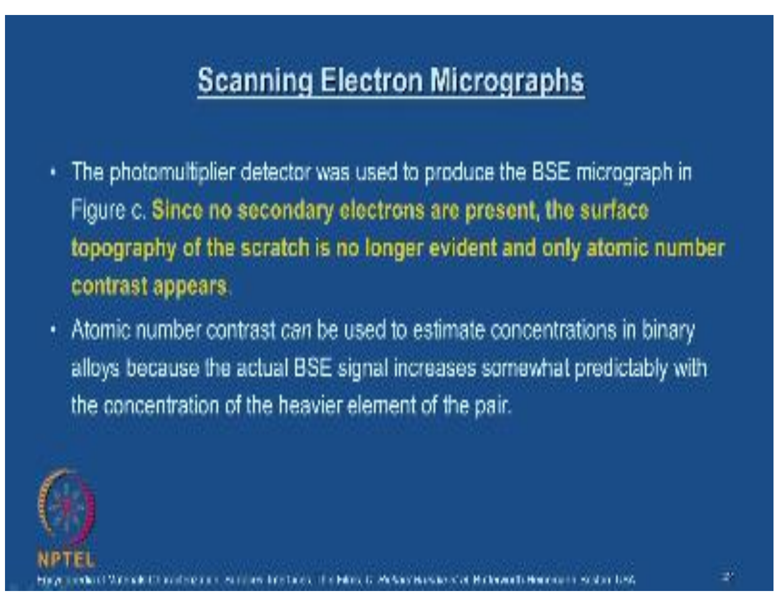

The photomultiplier detector was used to produce BAC micrograph in Figure see what we have just seen in two slides before since no second electrons are present the surface topography of the scratch is no longer evident and only anatomic number contrast appears atomic number contrast can be used to estimate the concentrations in binary alloys because the actual BAC 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 have requested 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 Adam interaction can deflect the beam electrons along the along a new trajectory which is considered elect 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 13 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 why not as the form Q which is greater than Phi naught is equal to 1point 6 2 into 10 to the power minus 20 times z square by a square cos square π0/ 2.

So this is events scattering events greater than π0 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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Electron Beam-Specimen Interaction

- The elastic scattering after numerous events, actually result in beam electrons leaving the specimen process called "**backscattering**"
- A mathematical description of elastic scattering process at angle greater than a specified φ_0 has the form

$$Q(>\varphi_0) = 1.62\times10^{-20}\,(Z^2/E^2)\cot^2(\varphi_0/2)$$

$$(\text{events} > \varphi_0)/[\text{electron}\ (\text{atom/cm}^2)]$$

where Q is called the cross section (cm^2) for elastic scattering (i.e. probability of elastic scattering)

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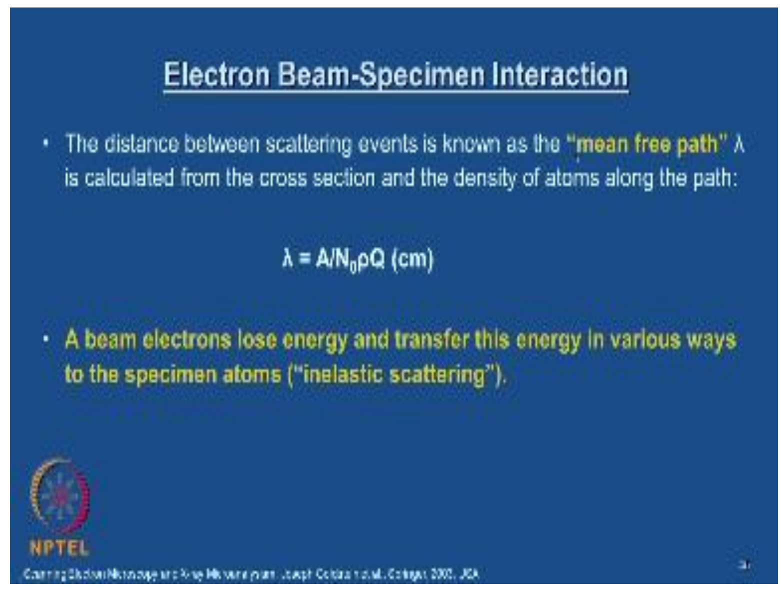

The distance between scattering events is known as the mean free path lambda is calculated from the cross-section and the density of the atoms along the path when lambda is equal to a divided by n knot ρ Q which is in centimeter a beam electrons loose energy and transfer this energy in various ways to the specimen atoms which is nothing but inelastic scattering see you see in an SEM we get the characteristic x-rays for a chemical analysis like be discussed in the beginning the basic.

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 seized BSEs and all the signals solve basically inelastic scattering this transfer takes place gradually. So that the beam electrons propagate through many Adam layers into the specimen before losing all their energy.

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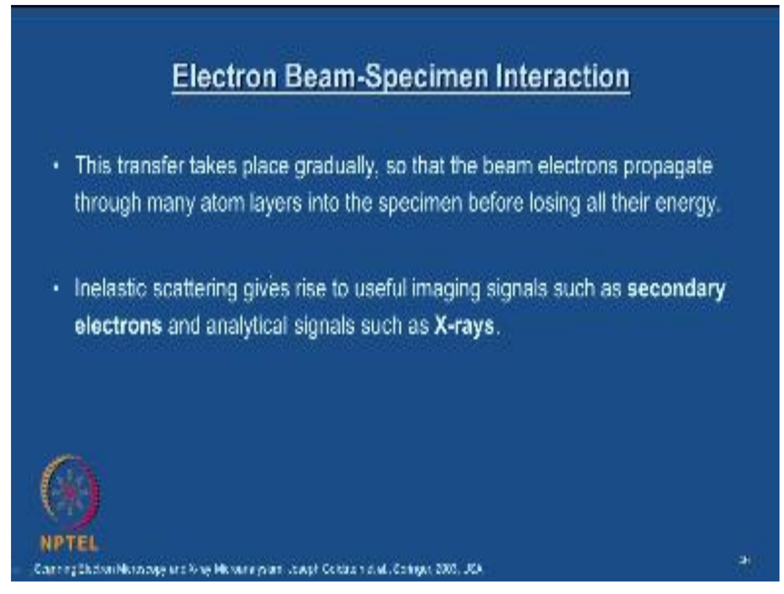

So this the loss of energy of the electron beam is not going to be instantaneous so it will be more I mean the you can see that how some of the models are being made for this how the electron beam is losing energy which I will show you in few minutes we will come that we will get an idea how the electron 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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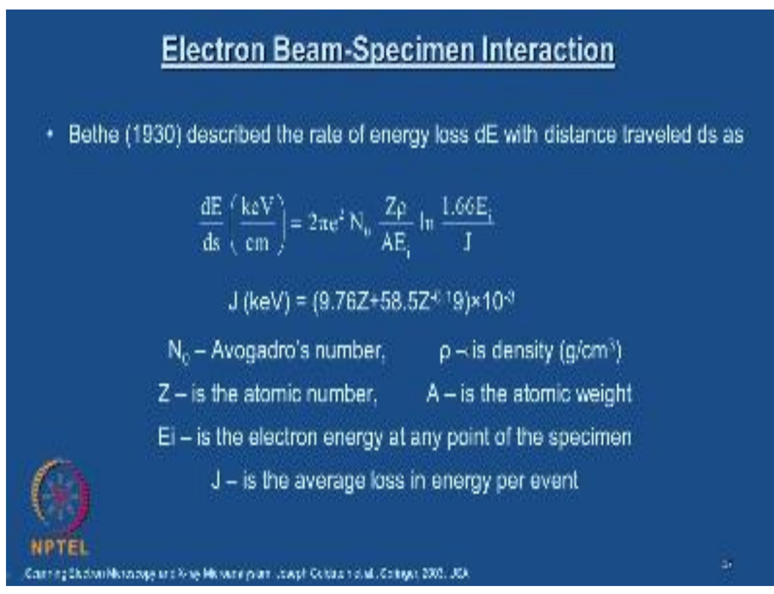

Method described1930 the rate of energy loss de with the distance travelled d s as de by D s the energy is given in kilo electron volt and the distances in centimeter which is equal to 2 pi e square n not into z Ro /AE I l on 1.66 I by j where j is equal to9.76 z+ 58.5 z to the power minus 0point 1 into 9 into 10 to power minus 3where n naught is called Avogadro's number Rho is the density Z is the atomic number is the atomic weight I is the electron energy at any point of the specimen jay 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 this variables I just want you to appreciate that point rather than getting into the details at this mode.

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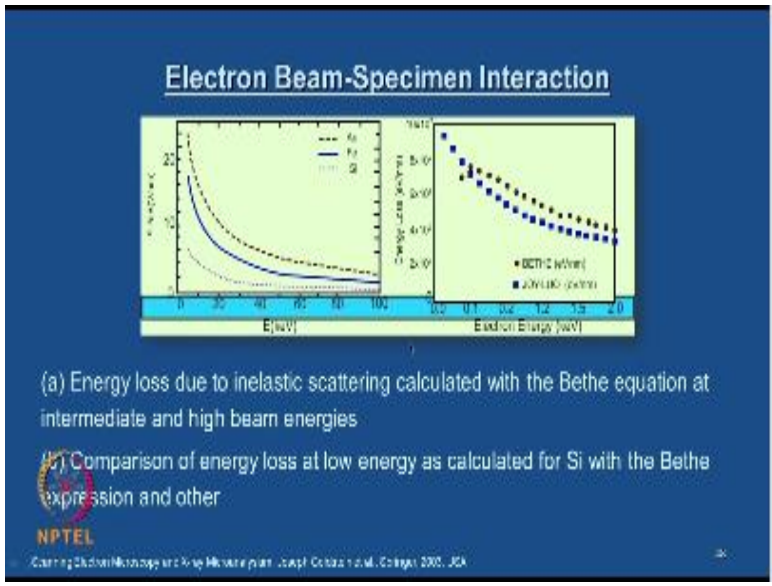

So you can see that two plots which are based on this be that equation how the energy loss due to an elastic scattering is calculated you can see that plot a is energy loss in rustic scattering calculated with the Betty equation at intermediate and high beam energies for all this elements and the plot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

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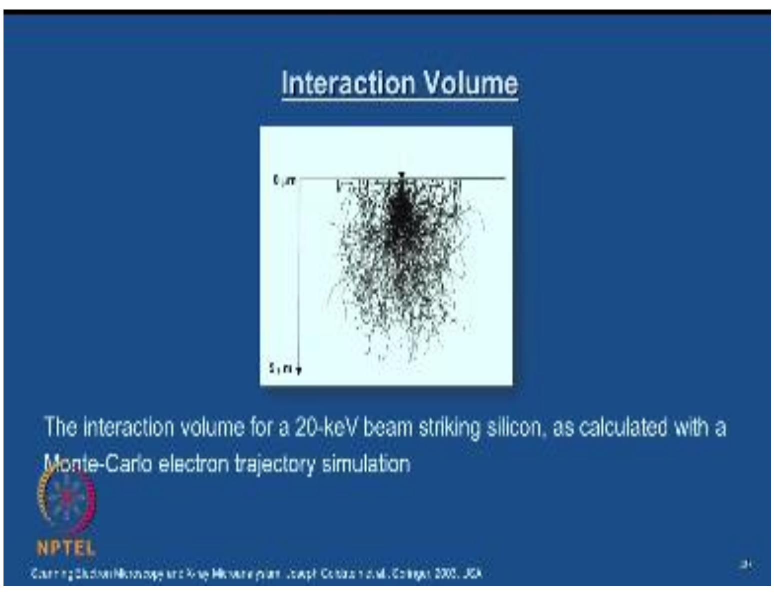

Now what you are going to see is we will look at what is this interaction volume and the electron beam comes and interacts with the specimen surface and what you are now seeing is the assimilation is the interaction volume for a 20 kilo electron volt beam striking the silicon as calculated with a Monte Carlo electronic eject trajectory simulations and 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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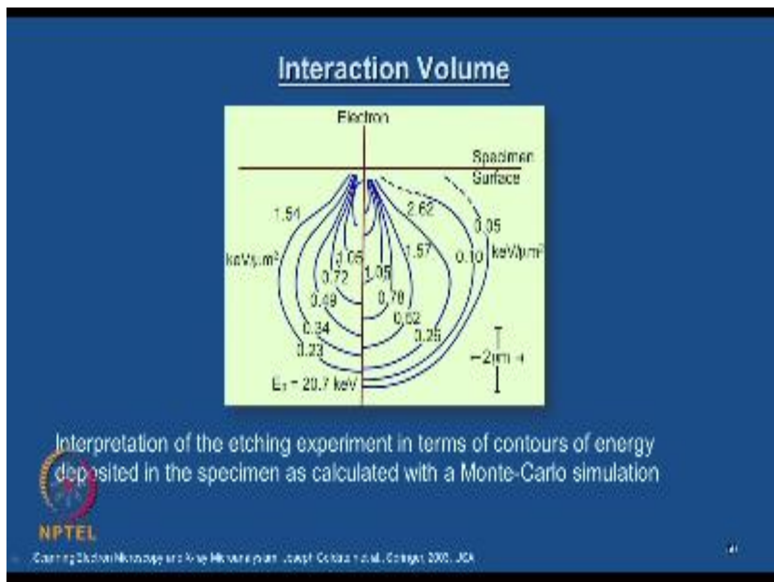

So let us try to understand how this happens this is you can see that the another schematic showing that this kind of interaction volume is interpreted through an etching experiment in terms of contours of the energy deposited in the specimen as calculated with the Monte Carlo simulation. So the left 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-dimensional you know 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 nights only a static images and actually it is happening dynamically between the interaction between Ron beam and the surface.

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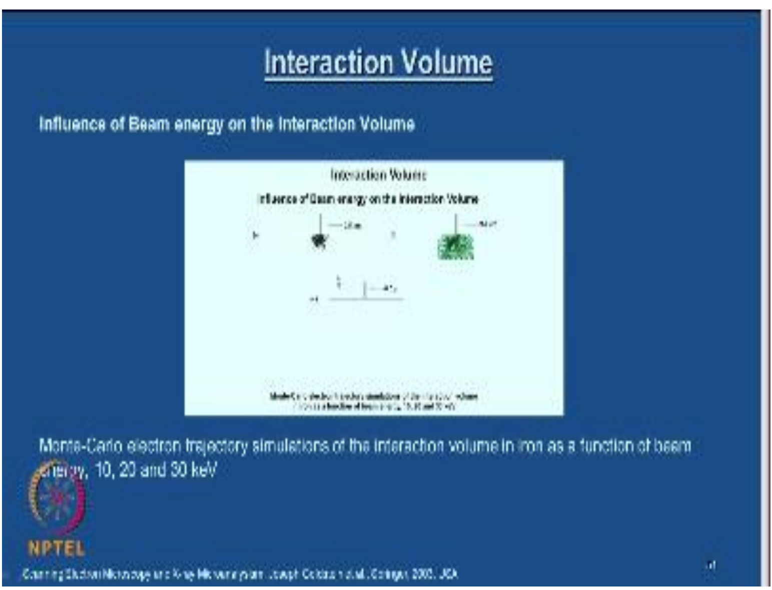

And I will just show you few more schematic which you have the just excuse me it mean so I would like to show this as a function of electron beam energy versus interaction volume you actually what you will see that the as the electron energy increases the interaction volume also we increase and somehow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

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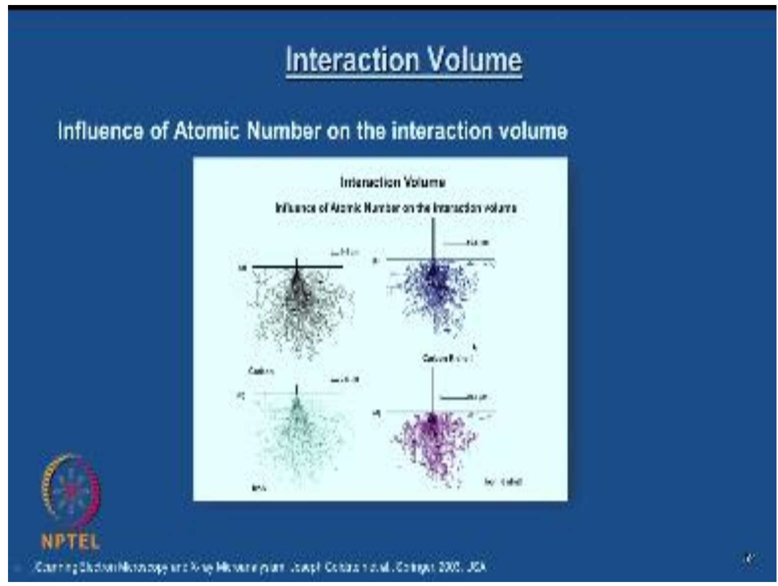

Now so you can see that the same effect of atomic number also you can see influence of atomic number on the interaction volume you can see it for different material here it is a carbon and this is for a carbon case shell. And then you have the iron and then you have the iron and case shell you can also see that as the atomic number increases the linear dimension decreases that is a very much understandable because the that that is because you are 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 systems same effect for a silver else 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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