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Lecture-14

Materials Characterization

Fundamentals of Scanning Electron Microscopy

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Hello everyone welcome to this material characterization course. In the last class he just started with the introduction of scanning electron microscopy and we have just reviewed what all the information one can get out of this scanning electron microscopic techniques and what are the salient features that you can obtain related to microstructural information and then we started looking at the instrumentation details. So we will continue in that session so this is what I was just showing yesterday.

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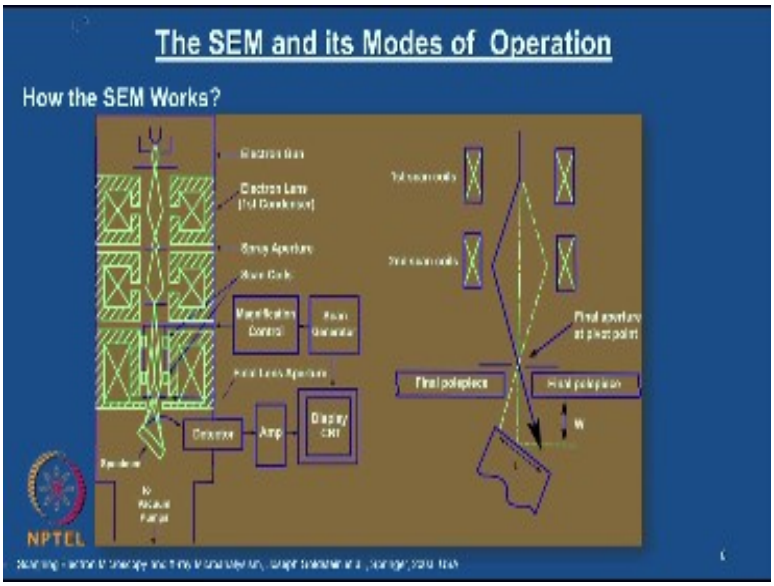

The schematic shows the cross sectional view of SEM and I just started describing this each part. So you have this electron gun and then you have series of electron lenses and then something called scanning coils and then you have this magnification control and the scan generator and then you have final lens aperture and this is where your specimen is kept in the specimen chamber which is maintained at a vacuum of 10^{-4} Pa, I just mentioned yesterday and then you have this detector system and then you have the control console.

So what it what we have to understand from this schematic, the electron gun just generates the electron and accelerates to 0.1 to 30KeV and then the electrons are passing through this electron lenses or scan coils and then the primary function of this section is to de magnify this probe diameter because, typically if you take a tungsten hairpin the probe diameter is not sharp enough to obtain the microstructural details however this scan coils or electron lenses d magnify that probe to a very small size in the order of 10 nanometers when it finally reaches on the specimen surface.

So this is a primary function of this coils and electron lenses. And if you look at the right hand side schematic which shows the specific action of this scan coils what you see here is the high-

energy electron beam which is accelerated by the electron gun comes through this scan coils and you see that the electron beam is deflected of the optic axis in a discrete locations in a line you can see that and then finally the second coil also deflects and again on a disk discreet location in a second line.

So like that it will go on depending upon the number of line coils you have in the column and what you have to understand is before it reached the beam reaches the final aperture at a pivot point, it has been deflected of the optic axis by the first coil and then the beam is brought back to this optic axis by the second coil and it crosses the final aperture and this action this deflecting off and on the optic axis of the electron beam occur still it makes the rectangular raster on the specimen surface that is scanning action here so this happens.

So finally the magnification of the image is the ratio between, the specimen region on which the electron beam is probing and the in the CRT screen where the raster is going from the one end to the other. So we will look at that ratio and understand the magnification just since we are talking about the rastering here I just want to mention the magnification related to the specimen region where the probe is scanning this area and the CRT screen what you are looking at the area of this CRT screen.

And what you are seeing is also a W is a working distance we will discuss about it is again a very important one of the parameters to for the operator control in the scanning electron microscopy.

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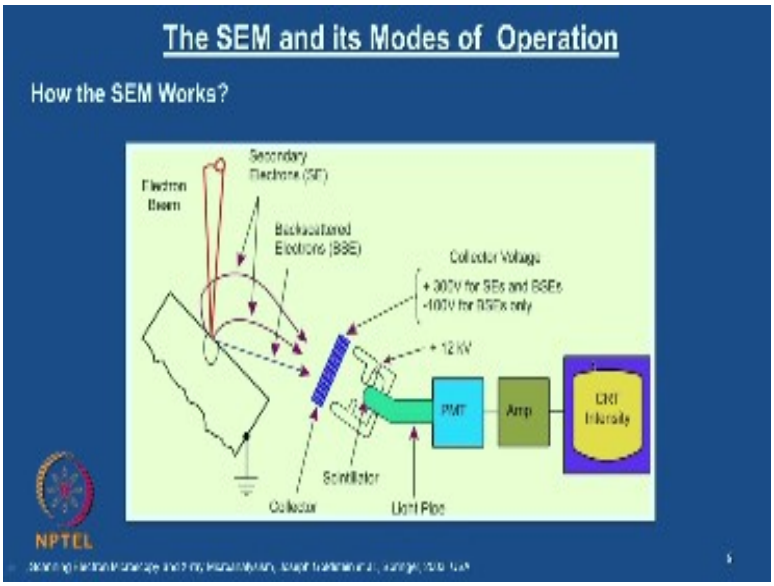

So now we look at the other schematic where it also describes the what kind of detecting system is employed in this scanning electron microscopy and you see here this electron beam strikes the specimen and you have some interaction volume shown here and from there you get typical signals secondary electrons as well as back scattered electrons like we discussed yesterday and you see that these signals are collected by the detectors okay the image is formed by I mean the electronic system converts the signal point by point and form an image.

So you see that the signals are collected by these detectors we will look at the detector details little later just to give a kind of an idea to understand how SEM works. Let us assume that this is a detector which can detect these two signals and you see the details, if you have the positive potential or positive voltage then it can accept secondary electrons as well as backscattered electrons.

But when you apply a negative voltage it can only accept backscattered electrons and not the secondary electrons this is because your secondary electrons have a lower energy which will get repelled by this field. Then the signals are collected by the scintillator and the photo multiplying

multiplier tube and which is getting further amplified with an amplifier and finally it reaches the CRT where you see the image of your specimen of interest. So this gives you a kind of an overall function of how the SEM works I hope you got some rough idea by looking at all these three schematics.

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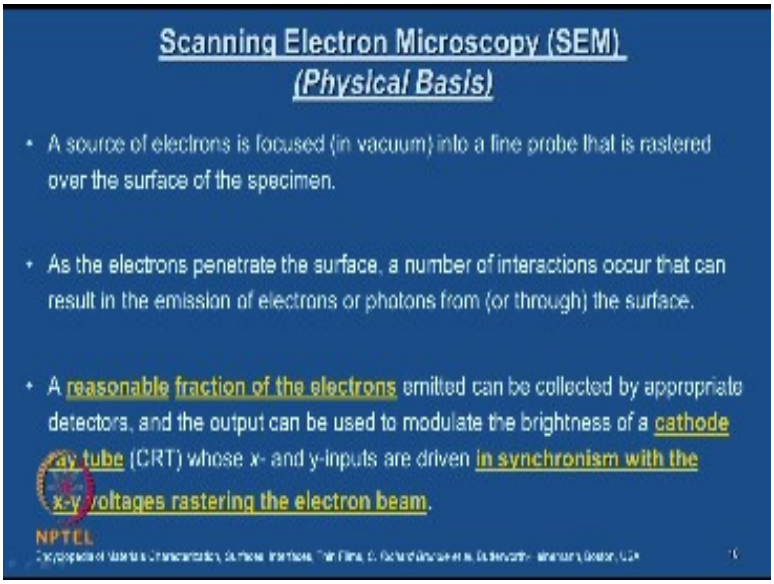

What now we will do is we will summarize whatever we have just discussed in the form of few sentences, so that you can just verify this again and again. A source of electron is focused (in a vacuum) into a fine probe that is rastered over surface of the specimen. As the electron penetrates the surface a number of interactions occur that can result in the emission of electrons or photons from the surface. A reasonable fraction of the electrons emitted can be collected by the appropriate detectors and the output can be used to modulate the brightness of a cathode ray tube whose X&Y inputs are driven in synchronism with the XY voltage rastering the electron beam.

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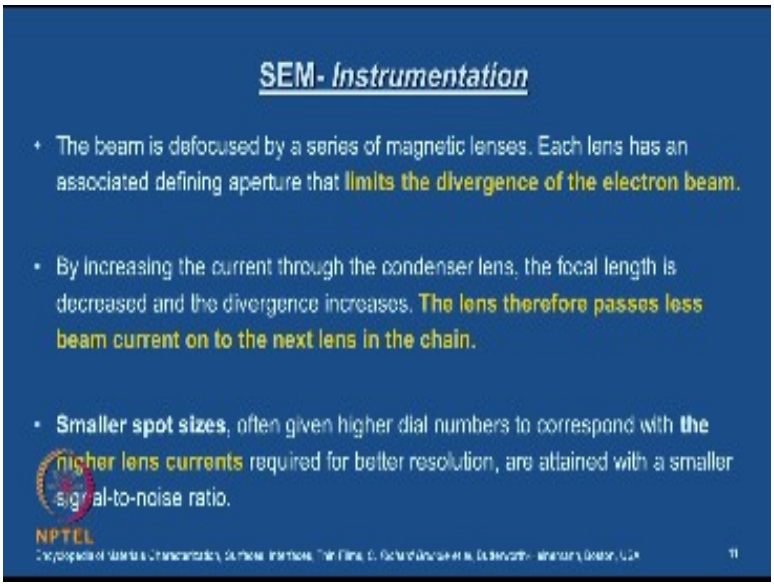

So if you look at the description of the magnetic lenses the beam is d focused by series of magnetic lenses are the each lens has an associated defining aperture that limits the divergence of the electron beam. So what I just want to go back and show these are the apertures we talk about each lens has got some kind of aperture which decides the divergence of the electron beam by increasing that current through the condenser lenses, the focal length is decreased and the divergence increases the lens therefore passes less beam current on to the next lens in the chain.

Remember the smaller the spot sizes, often given higher dial numbers to the numbers to correspond with the higher lens currents required for the better resolution are attained with a smaller signal-to-noise ratio. This is very common practice in an SEM as well as TEM. Probably I will show you when we go to that appropriate lab and look at the actual equipment and the controls, you can see that all small smaller spot size often given higher dial numbers.

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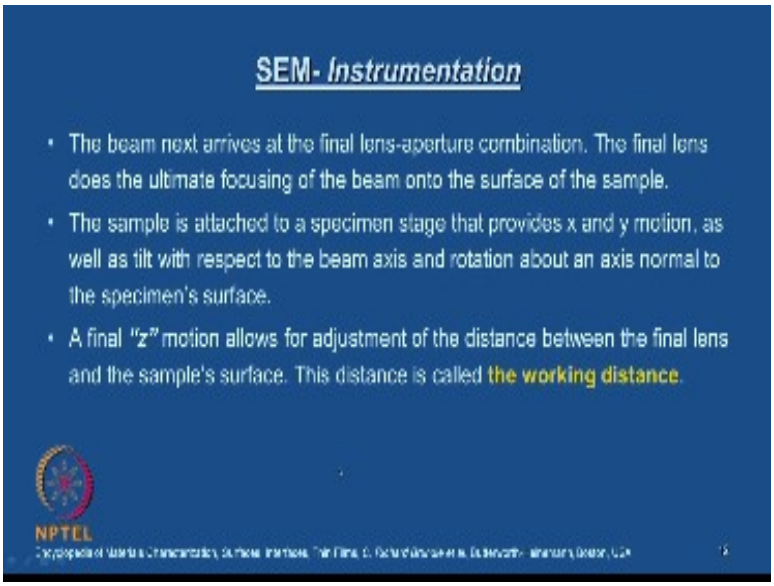

The beam next arrives at the final lens aperture combination; the final lens does the ultimate focusing of the beam onto the surface of the sample. The sample is attached to a specimen stage that provides X&Y motion as well as the tilt with respect to the beam axis and rotation about an axis normal to this specimen surface. Your final 'Z' motion that is vertical motion allows for the adjustment of the distance between the final lens and the sample surface this distance is called the working distance I just mentioned in the schematic. So the working distance is the distance between the final lens and this sample surface.

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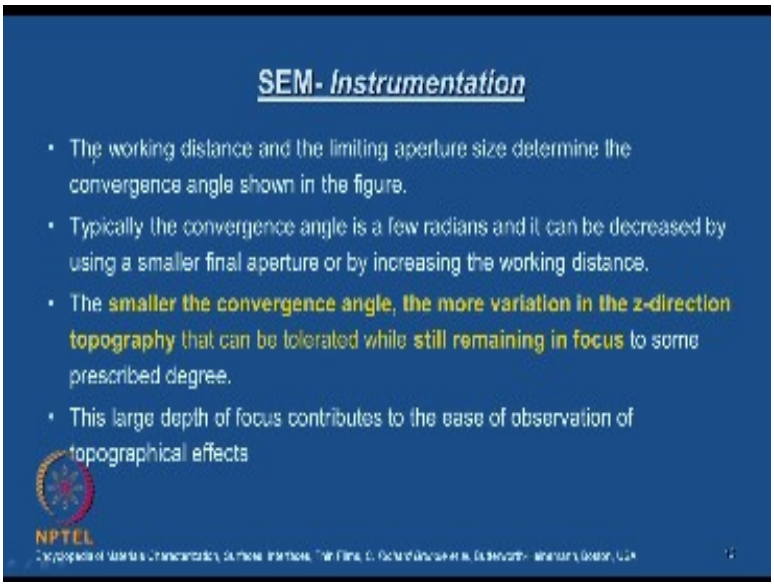

Now let us look at the some more description of these parameters. The working distance and the limit in aperture size determine the convergence angle shown in the figure. Typically the convergence angle is a few radians and it can be decreased by using a smaller final aperture or by increasing the working distance. The smaller the convergent angle, the more variation in the z-direction topography that can be tolerated while, still remaining in focus to some prescribed degree. This large depth of focus contributes to the ease of observation of topographically effect.

You see we also discussed the this phenomenon yesterday in the introduction of an SEM, one of the important feature of this equipment is this can achieve very large depth of focus and also I said that you get a feel of 3d like image and this is convergent angle is one of the parameters which contributes to this large depth of focus.

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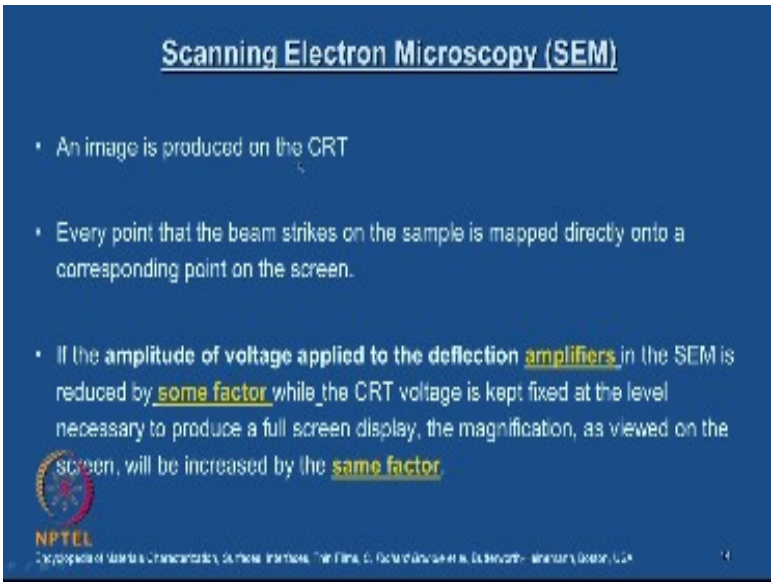

So an image is produced on CRT. Every point that the beam strikes on the sample is mapped directly onto the corresponding point on the screen. If the amplitude of voltage applied to the deflection amplifiers in the SEM is reduced by some factor while the CRT voltage is kept fixed at the level necessary to produce a full screen display, the magnification as viewed on the screen will be increased by the same factor. See this is just in terms of operator control the magnification is explained.

I also talked about the ratio between the region on the sample as well as the CRT screen area and this particular explanation is in terms of what you actually control on the specimen that is, so you have the deflection amplifiers which has been controlled by some factor whether you reduce or increase, the same effect you see it on the CRT screen on the magnification of the appropriate specimen region.

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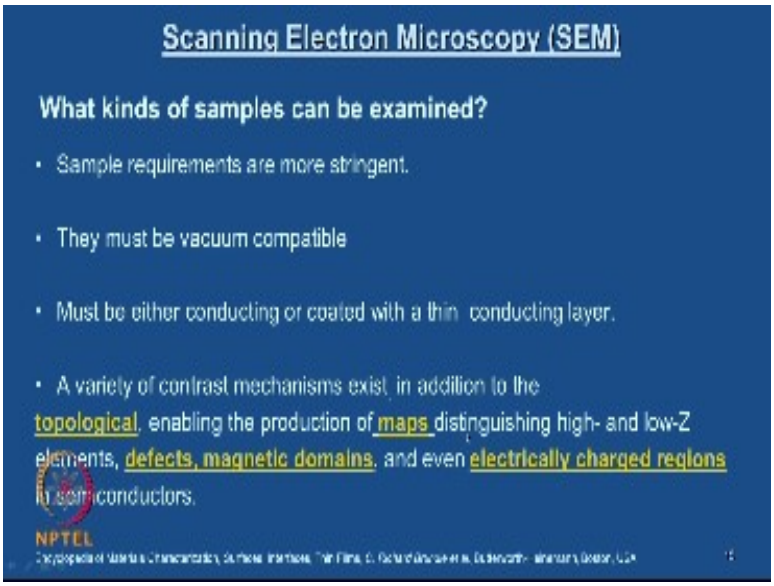

So now, what kind of samples can be examined? The sample requirements are more stringent they must be vacuum compatible, they must be either conducting or coated with a thin conducting layer, and we will look at the details of the sample preparation and its requirements little later we will see it. But just give you a kind of introductory remark, you should realize that the material should be vacuum compatible and it should be either conducting or we have to quote a thin conducting layer on the specimen.

A variety of contrast mechanisms exist in addition to the topological, enabling the production of maps, distinguishing high and low atomic number elements, defects, magnetic domains and then even electrically charged regions in semiconductors. Since this also we discussed yesterday different kinds of mechanisms are possible.

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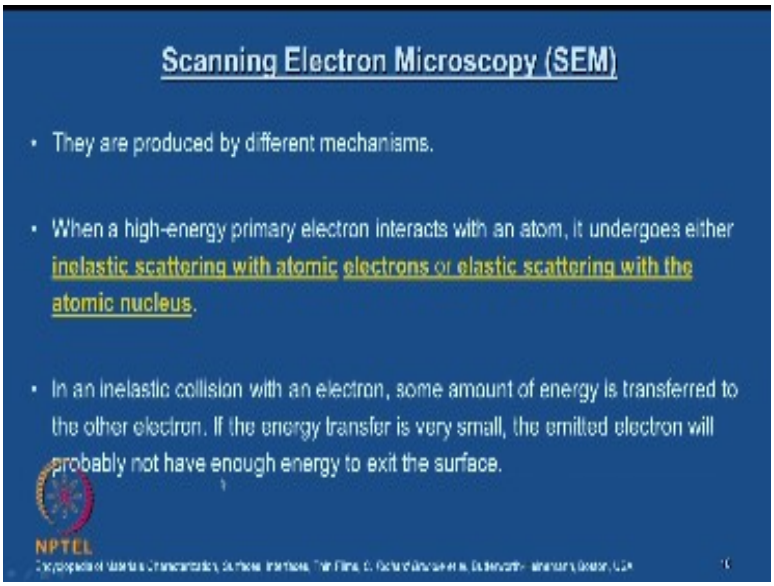

When a high energy primary electron interacts with an atom, it undergoes either inelastic scattering with atomic electrons or elastic scattering with the atomic nucleus. In an inelastic collision with an electron, some amount of energy is transferred to the other electron. The energy transfer is very small the emitted electron will probably not have enough energy to exit the surface. So we are now getting into the details of electron beam and interaction with the specimen you will see.

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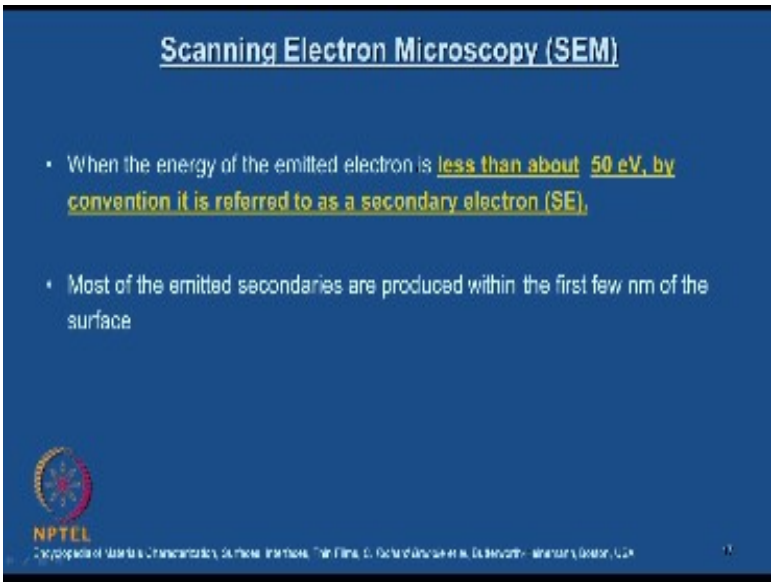

When the energy of the emitted electron is less than about 50 electron volts, by convention it is referred to as a secondary electron. Yesterday I just mentioned that the classification of this signals something like secondary electron and backscattered electrons is based upon its varying energies. So you have the effects number here when the emitted electron is less than about 50 electron volts it is referred as secondary electron. Most of the emitted secondary are produced within the first few nanometers of the surface.

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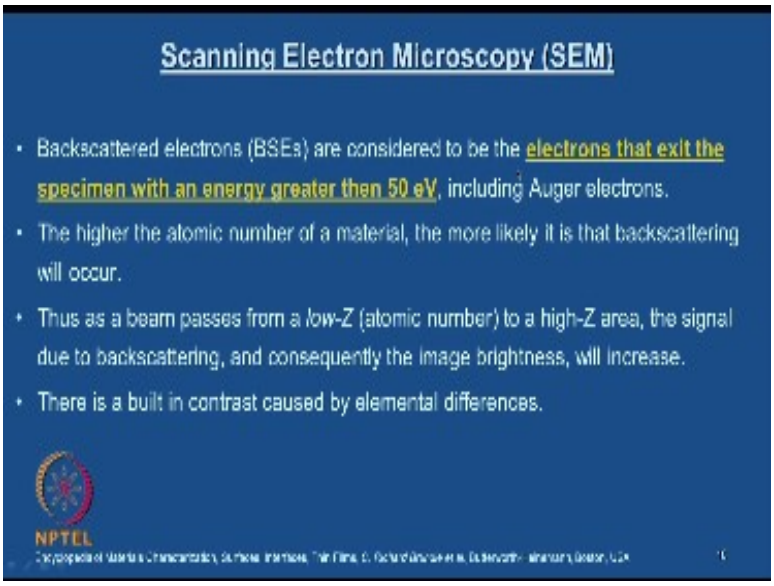

Backscattered electrons are considered to be the electrons that exit the specimen with energy greater than 50electron volts, including Auger electrons. The higher the atomic number of the material the more likely it is that backscattering will occur thus a beam as a beam passes from a low atomic number to a high atomic number area, the signal do to back scattering and that consequently the image brightness will increase. There is a built in contrast cost by the elemental differences.

So you have to understand that the atomic number of the element increases the scattering event also increases and eventually you get image brightness as well we will look at this kind of electron beam and it is interaction and its volume everything we will look at them in later and this is just and I am introducing how the signals are classified and what kind of interaction they will make with the specimen.

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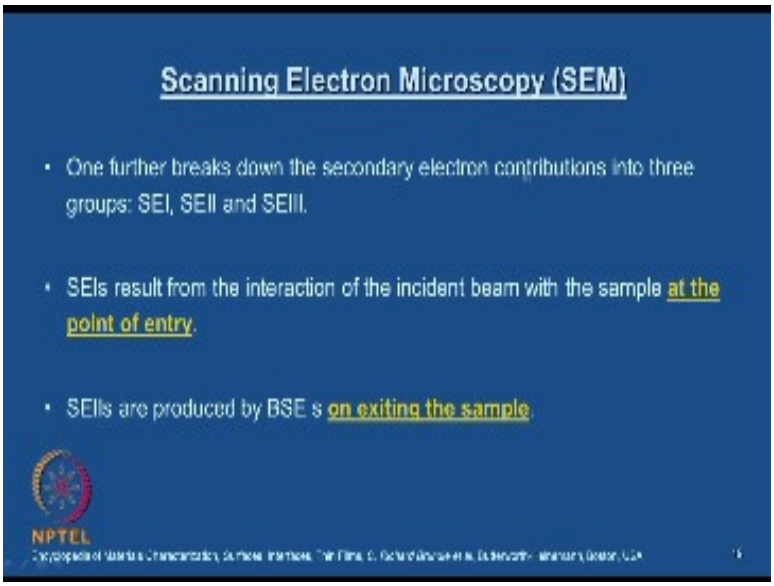

One further breaks down the secondary electron contribution into three groups' second electron I, secondary electron II and second electron III. Secondary electron I results from the interaction of the incident beam with the sample at the point of entry. Secondary electron II is produced by backscattered electrons on exiting the sample.

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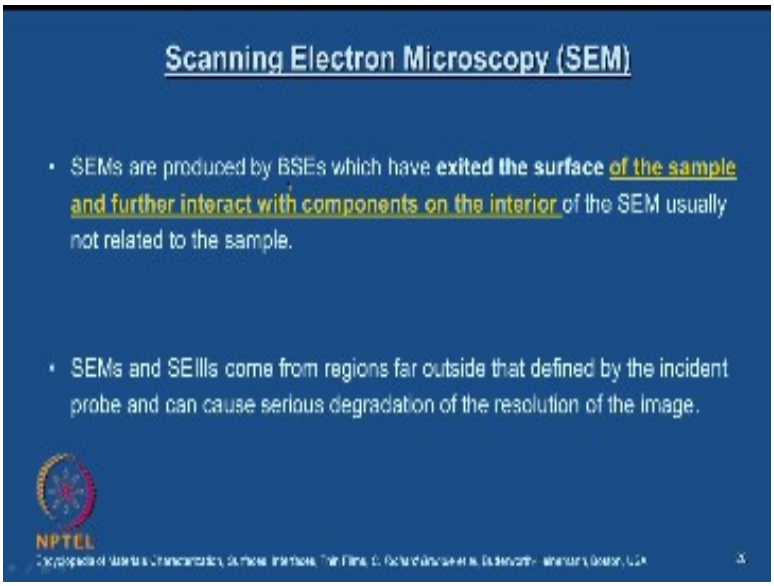

SEMs are produced by backscatter electrons which have exited the surface of the sample and further interact with the components of the interior of this SEM, usually not related to the sample and SEM secondary electron these come from the region far outside that defined by the incident probe and can cause serious degradation of the resolution of the image. You see these classification the further classification of the secondary electrons again based upon the energy variation.

However these energy variations happen at particular location at the event and that is how it's been described in the last two slides. This is just for a clarity, we will just show the effect of these one two and three secondary electrons when we discuss the contrast mechanisms in detail.

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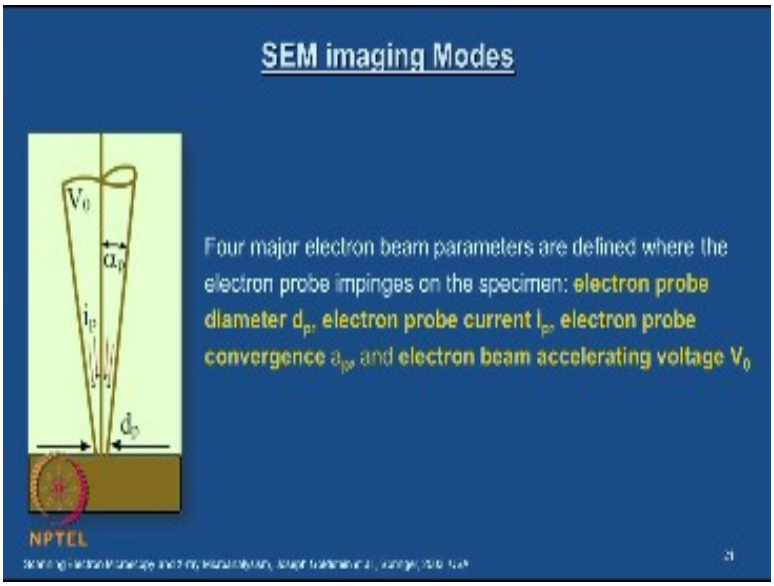

Now we will just go to the scanning electron microscopy imaging modes. What are the kinds of imaging mode we employ while we carry out the microstructure investigation using scanning electron microscopy? You see this schematic this is the electron beam, you have some notation called d_P, i_P, α_P and V_o. Four major electron beam parameters are defined where the electron probe impinges on the specimen. What are those four parameters? Electron probe diameter that is d_P, beam size electron probe current i_P, electron probe convergence α_P there is a typo here α_P and electron beam accelerating voltage V_0.

Please remember all this beam parameters will have a specific effect on the image quality and the information which you get as well as on the resolution that is why we specifically talked about these parameters. We will look at the effect of each one of these beam parameters on the micro structural details which you obtain as well as on the resolution we will see one by one.

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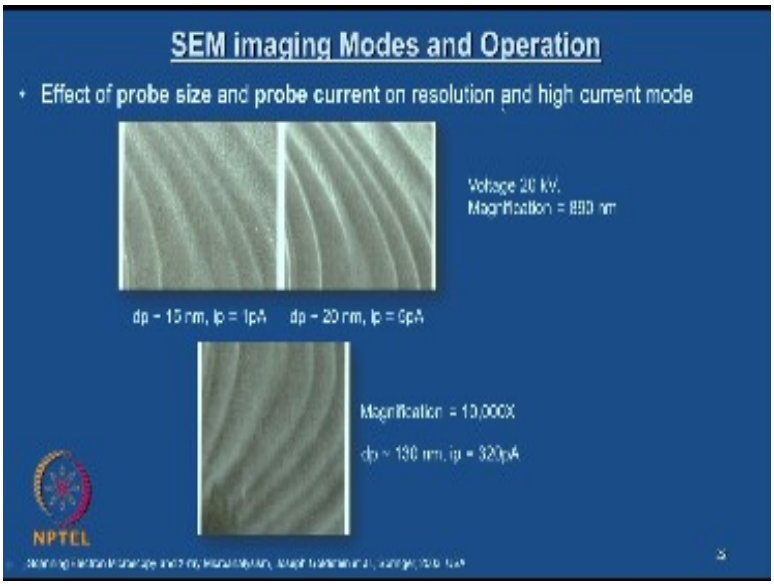

The effect of probe size and the probe current on the resolution and the and the high current mode. So you have a three micrographs of some surface, the voltage employed is 20 kilo volt the magnification for these two would sorry this is not magnification is something wrong here, the magnification is about 10000X for all these three you have d_P of 50 nanometers i_P of 1 pA and you see that schematic I mean. So this micrograph B is obtained with the d_P in order of 20 nanometers and an i_P in order of 55 pico amperes and the third micrograph is obtained with the d_P of 130 nanometers and i_P of 320 pA.

So what do you see? It is not that you have a specific combination of all these parameters is well defined you see that as the probe diameter increases you are not seeing a clear resolution here. The resolution is not improved and at the same time if you increase the probe current also the solution is not improved but at this particular combination of DP and IP you have a better result compared to this first one and third one. So you see that you have a combination of a probe diameter and the probe current gives a better resolution.

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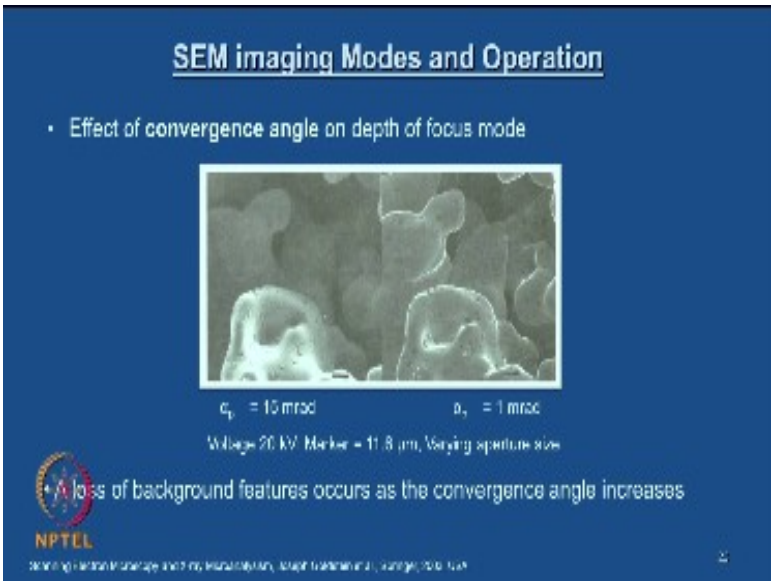

And next we look at the effect of convergent angle on the depth of focus. You see the image taken in the same region here the α_P that is convergent angle is 15 mil.radian and here it is one mil.radian, the voltage is 20 kilo volt and this marker is 11.6microns and you have the varying aperture size. You see that a loss of background features occurs as the converging angle increases. So if you want if you look at this image the background is not details are not clear however you can see that much more details are seen which is right in behind this region. So you have the convergent angle effect as well on the resolution of the micrograph.

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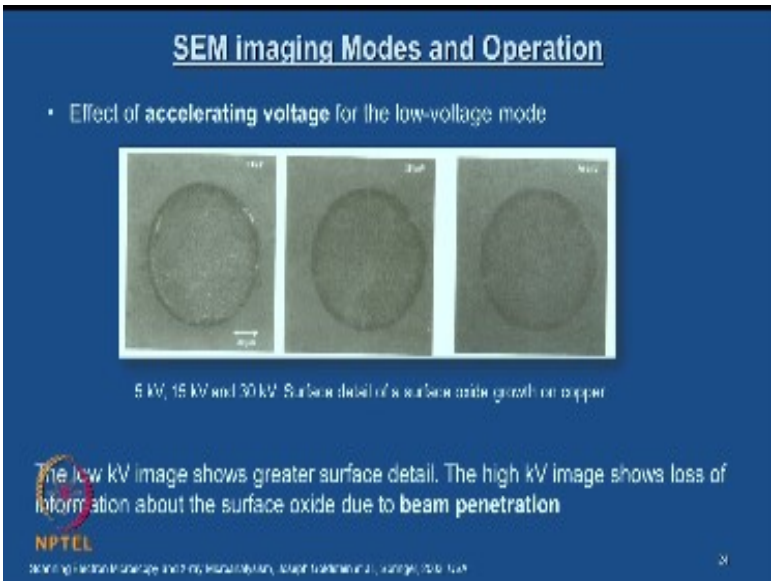

Now if you look at the effect of accelerating voltage in a low voltage mode, the micrographs taken here with 5 KV and 15 KV are 30 KV and you see that the surface detail of a surface oxide growth on a copper is same with the different voltage. You would clearly appreciate that the increasing the acceleration voltage, not necessarily help the resolution you see only at the lower accelerating voltage you are able to look at the details of you are able to see the details of the oxide layer on the specimen. The low-KV image shows greater surface detail, the high KV image shows loss of information about the surface oxide due to beam penetration.

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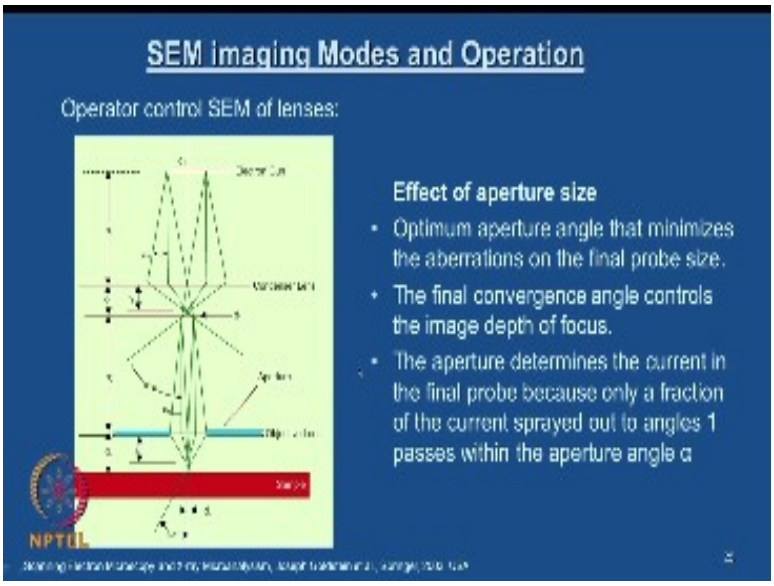

So now we will see what are all the operator control in SEM to obtain a better resolution or a control. We will see that the animation which is showing, the kind of aperture size effect. We will see and let me first describe this schematic and then we will see what is that we try to understand from the schematic. So this is an electron gun then again the beam comes through the condenser lenses and then final aperture and then again goes to the next stage and the objective lens and then you see finally it reaches the sample.

What we are trying to see here is, when you obtain optimal aperture angle that minimizes the abrasions on the final probe size that means we need to understand what this optimum aperture angle is. By looking at the image quality we are relatively it is free from this abrasions you judge this. The final convergent 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 angle alpha one passes in the aperture angle alpha finally.

So what we are trying to say here is you see that the beam is spreading to the angle of α one quite large and only fraction of this is going to enter the final aperture and which has the aperture angle controlled by this objective lenses or aperture the aperture belongs to the lens controls the

final angle from the large spread out angle in the previous lenses, so that is what we are trying to understand here.

So that aperture an operator can control and then decide whether this particular settings will be useful in obtaining the information with minimal aberrations.

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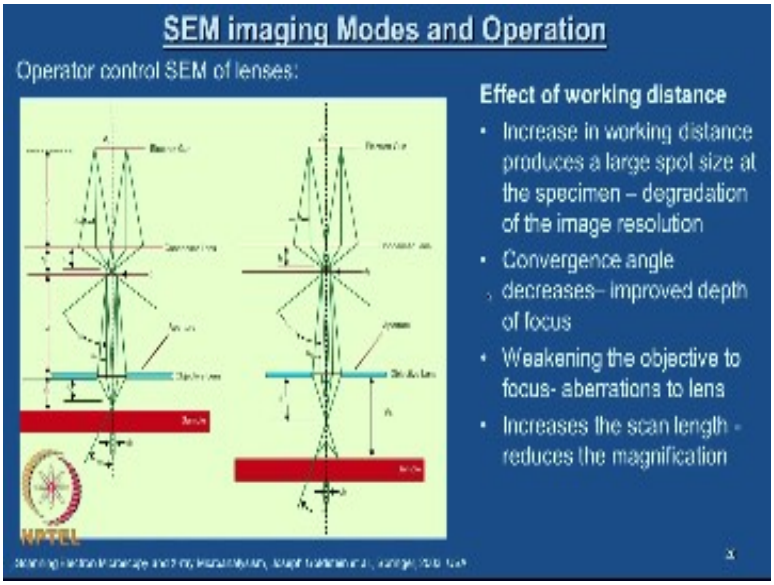

Now you will look at the effect of working distance. Again you see these two animations of the ray diagram and you see that the distance between the final aperture and the sample surface is working distance like me to define in the previous one. So you have this to schematic displaying the ray diagram with two different working distance this is W_1 and this W_2 and then the schematic nicely displays increasing the working distance how you are converging the ray converging positions are, I mean displayed here or how they are different with the adjustment of the working distance.

The increase in working distance produces a large spot size at the specimen, so you can see that here it is small spot and here it is a large spot and the degradation of the image resolution obviously that is going to cause some resolution decrease in the resolution. Convergence angle

decreases and improve depth of focus and the convergent angle which we have already discussed and smaller the convergent angle the improved the depth of focus that we have seen already. Weakening the objective to focus aberration to lens, so you increase the working distance this also will happen and which also increases this current length and reduces the magnification so working distance if you play around these are all the points which have to keep in mind and the operator should again judge by looking at the working distance and the image quality he obtains and then decide what gives him the best.

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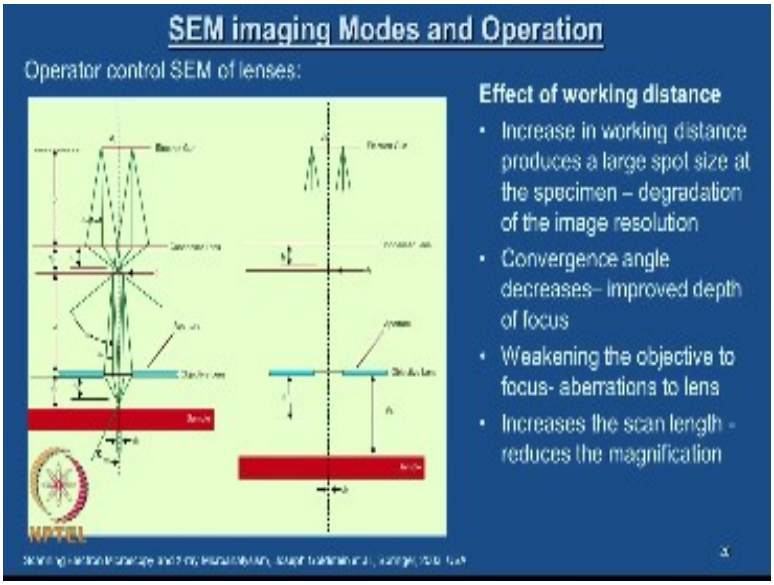

Now we will look at the effect of condenser lens strength here is the schematic again you can see that the effect of condenser lens strength on the final probe diameter. The increase in the condenser lens strength increase the demagnification of the each lens and reduces the probe size. The final probe 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.

So either you if you want to reduce the final probe size either you play with the minimum probe size or maximum probe current that you have to take a call by looking at the again the kind of

information you are interested in and also kind of resolution you want obtained at particular magnification.

So you can clearly see that from the schematic depending upon the condenser lens current so you see that how the final probe diameter which is falling on the sample is reduced to a very small probe here. So having talked about this probe diameter we will go through some of the important aspects to be noted we are always interested in minimum probe size if he in order to resolve very smaller details and if you recall in the beginning of this second part of the course where we talked about fundamentals of electron optics we also discussed about a quite a bit of quite a bit of information on the lens aberrations in the case of electromagnetic lenses and its optical systems where we discussed that all the aberrations are going to contribute little bit to the final probe diameter or the electron beam. So this is what we are now worried summarizing here.

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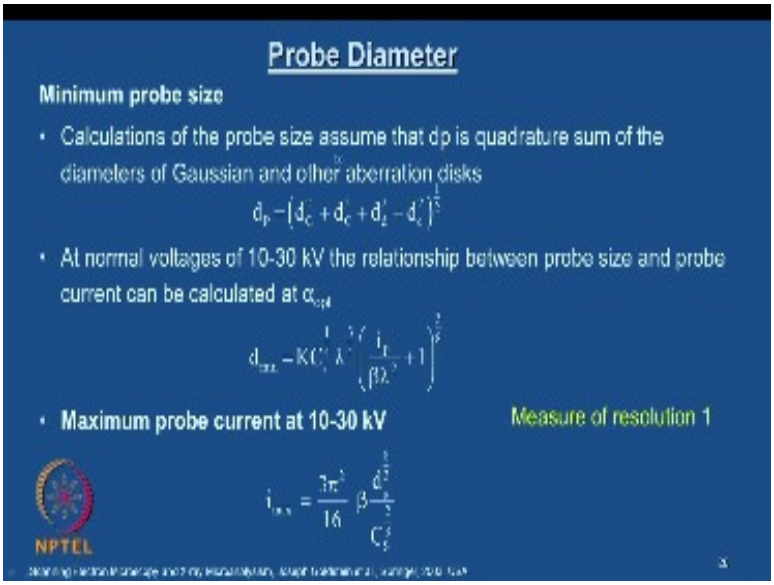

The calculations of the probe size assume that d_P is quadrature sum of the diameters of Gaussian and other aberration disks. So the final diameter is $\mathbf{d_P = (d_G^2 + d_C^2 + d_d^2 + d_c^2)^{1/2}}$. At normal voltages of 10 to 30 KV the relationship between probe size and probe current can be calculated

at α optimum, $d_{min} = K. C_s^{1/4}.\lambda^{3/4}[(i_P/\beta\lambda^2) + 1]^{3/8}$ and the maximum probe current at 10 to 30 KV has got an expression, similar to this $i_{max} = [(3\pi^2/16).\beta.(d_P^{8/3}/C_s^{2/3})]$.

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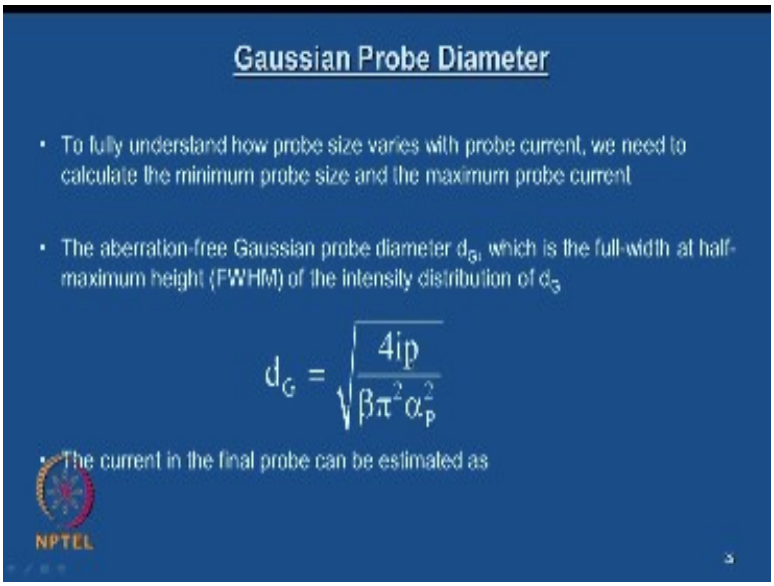

And we also look at what is this Gaussian probe diameter to fully understand how probe size varies with the probe current we need to calculate the minimum probe size on the maximum probe current. The aberration free Gaussian probe diameter d_G which is the full width at half maximum height of the intensity distribution of d_G where $d_G = \sqrt{[(4.i_P)/(\beta.\pi^2.\alpha_p^2)]}$. The current in the final probe can be estimated as $i_P = \sqrt{[(\beta.\pi^2.\alpha_p^2.d_G^2)/4]}$.

So all these expressions will give you a kind of an idea the important four parameters which we talked about how they are related basically with respect to the probe diameter. Please understand that is you should not confuse this probe diameter with the electron beam size so electron beam along the column is not called probe diameter the probe diameter is a final probe electron beam which exit from the final aperture and next to immediately to the specimen surface.
So that is called probe diameters do not confuse this parameter with the electron beam size along the rest of the column and then you see that that probe diameter has got the dependence on all the

other four parameters and that is what this mathematical expressions relate that is all I want you to appreciate.

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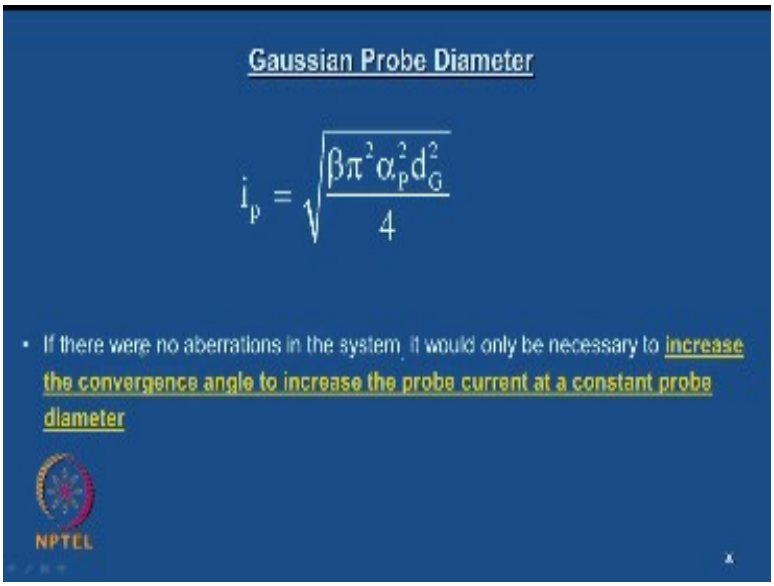

If there were no abrasions in the system it would only be necessary to increase the convergent angle to increase the probe current at the constant probe diameter. So I would like to stop this lecture here and then we will continue on the various aspects of the SEM operations and little bit of theory of contrast mechanisms and how this equipment can be exploited can be exploited in order to obtain more micro structural details we will continue in the next class. Thank you.

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