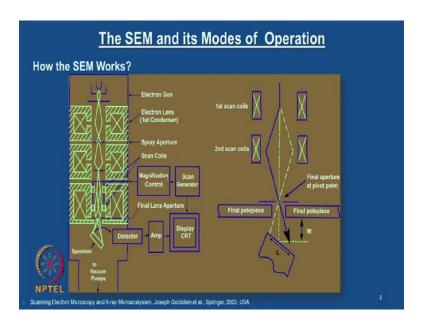
Fundamentals of optical and scanning electron microscopy Dr. S. Sankaran Department of Metallurgical and Materials Engineering Indian Institute of Technology, Madras

Module – 03
Unit-6 Introduction to scanning electron microscopy
Lecture –14
SEM and its mode of operation
Effect of aperture size
Effect of working distance
Effect of condenser lens strength

Hello everyone! Welcome to this material characterization course. In the last class, we just started with the introduction of scanning electron microscopy and we have just reviewed what are 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 micro structural information. And then, we started looking at the instrumentation details. So, we will continue in that session. So, this what I was just showing yesterday.

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The schematic shows the cross-sectional view of SME. And, I just started describing this each parts. So, you have this electron gun and then you have series of electron lenses and then something called scanning coils. Then, you have this magnification control and scan generator, and then you have final lens aperture. And, this is where your specimen is kept

in this specimen chamber, which is maintained at a vacuum of 10 to the power minus 4 Pascal. I just mentioned yesterday. 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 30 kilo electron volt. Then, the electrons are passing though this electron lenses or scan coils.

And then, the primary function of this section is to demagnify this probe diameter because typically if you take a tungsten hairpin, the probe diameter is not sharp enough to obtain the micro structural details. However, this scan coils or electron lenses demagnify that probe to a very small size in the order of 10 nanometers, when it finally reaches on the specimen surface. So, this is the primary function of this coils and electron lenses.

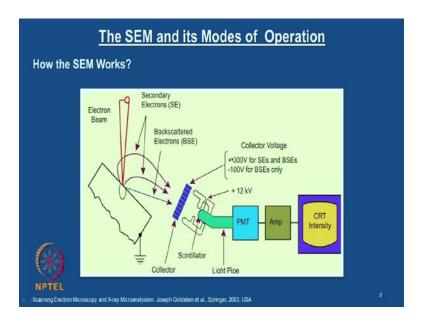
And, if you look at the right hand side schematic, which shows the specific action of this scan coils. What you see here is the high energy electron beam, which is accelerated by the electron gun, comes through this scan coils. And, you see that the electron beam is deflected off the optic axis in discrete locations in a line. You can see that. Then finally, the second coil is also deflects again on a discrete location in a second line. So, like that it will go on, depending upon the number of lens, coils, you have in the column.

And, what you have to understand is before it reaches, the beam reaches the final aperture at a pivot point, it has been deflected off 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 occurs, still it makes a rectangular raster on the specimen surface; that is a 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 the CRT screen. And, what you are seeing is also a double use working distance. We will discuss about it. It is again very important; one of the parameters to for the operator control in the scanning electron microscopy.

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So, now we will 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 Backscattered Electrons like we discussed yesterday. And, you see that these signals are collected by the detectors. 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 this detectors.

We will look at the detector's details little later. Just to give a kind of an idea to understand how SME 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 overall function of how the SME works. I hope you got some rough idea by looking at all these three schematics.

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Scanning Electron Microscopy (SEM) (Physical Basis)

- A source of electrons is focused (in vacuum) into a fine probe that is rastered over the surface of the specimen.
- As the electrons penetrate the surface, a number of interactions occur that can result in the emission of electrons or photons from (or through) the surface.
- A <u>reasonable fraction of the electrons</u> emitted can be collected by appropriate
 detectors, and the output can be used to modulate the brightness of a <u>cathode</u>
 <u>ray tube (CRT)</u> whose x- and y-inputs are driven <u>in synchronism with the</u>
 <u>real tages reatoring the electron beam</u>

x-y voltages rastering the electron beam.

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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 electrons is focused in a vacuum into a fine probe that is rastered over the surface of the specimen. As the electron penetrates the surface, a number of interactions occurs 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 and y inputs are driven in synchronism with the x-y voltages rastering the electron beam.

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SEM-Instrumentation

- The beam is defocused by a series of magnetic lenses. Each lens has an
 associated defining aperture that limits the divergence of the electron beam.
- By increasing the current through the condenser lens, the focal length is
 decreased and the divergence increases. The lens therefore passes less
 beam current on to the next lens in the chain.
- Smaller spot sizes, often given higher dial numbers to correspond with the nigher lens currents required for better resolution, are attained with a smaller signal-to-noise ratio.

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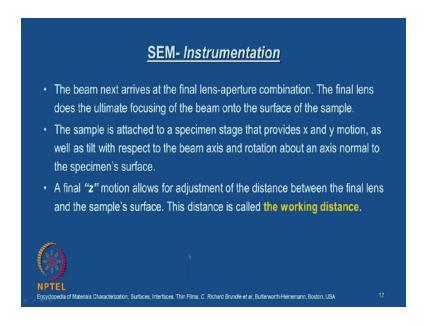
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So, if you look at the description of the magnetic lenses, the beam is defocused by a series of magnetic lenses. Or, each lens has an associated defining aperture that limits the divergence of the electron beam. So, what I just want to go back and 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 the 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 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 SME as well as TME. Probably, I will show you when you go to that appropriate lab and look at the actual equipment and their controls. You can see that all smallest spot size, often given a 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 and y motion, as well as the tilt with respect to the beam axis and rotation about an axis normal to the specimen's surface. A final "z" motion, that is, vertical motion allows for adjustment of the distance between the final lens and the sample's 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 the samples surface.

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SEM-Instrumentation

- The working distance and the limiting 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 convergence 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 popular depth of focus contributes to the ease of observation of

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Now, let us look at some of the description of this parameters. The working distance and the limiting 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 topographical effects. You see the; we also discussed this phenomenon yesterday in the introduction of SME. One of the important feature of this equipment is this can achieve very large depth of focus. And also, I said that you get feel of three dimension like image. And, this is; convergent angle is one of the parameters, which contributes to this large depth of focus.

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Scanning Electron Microscopy (SEM) An image is produced on the CRT Every point that the beam strikes on the sample is mapped directly onto a 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. NPTEL Encyclopedia of Materias Characterization, Surfaces, Interfaces, Thin Films. C. Richard Brundle et al. Bufferworth-Heiremann, Boston, USA 15

So, an image is produced on CRT. Every point that the beam strikes on the sample is mapped directly onto a corresponding point on the screen. If the amplitude of voltage applied to the deflection amplifiers in the SME 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 these CRT screen on the magnification of the appropriate specimen region.

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Scanning Electron Microscopy (SEM) What kinds of samples can be examined? Sample requirements are more stringent. They must be vacuum compatible Must be either conducting or coated with a thin conducting layer. A variety of contrast mechanisms exist, in addition to the topological, enabling the production of maps distinguishing high- and low-Zelements, defects, magnetic domains, and even electrically charged regions in sen iconductors. NPTEL Encyclopeda of Materias Characterization, Surfaces, Interfaces, Thin Films, C. Richard Brundle et al. Butterworth-Heinemann, Boston, USA

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 coat 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. See, this also we discussed yesterday.

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Scanning Electron Microscopy (SEM) They are produced by different mechanisms. 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. If the energy transfer is very small, the emitted electron will probably not have enough energy to exit the surface. PPTEL Encyclopeda of Materials Characterization, Surfaces, Interfaces, Thirn Films, C. Richard Brunde et al, Butterworth-Heiremann, Boston, USA 15

Different kinds of mechanisms are possible. 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. If 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. We will see.

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Scanning Electron Microscopy (SEM)

- When the energy of the emitted electron is <u>less than about</u> 50 eV, by convention it is referred to as a secondary electron (SE).
- Most of the emitted secondaries are produced within the first few nm of the surface.



When the energy of the emitted electron is less than about 50 electron volt, 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 volt, it is referred as secondary electron. Most of the emitted secondaries are produced within the first few nanometers of the surface.

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Scanning Electron Microscopy (SEM)

- · Backscattered electrons (BSEs) are considered to be the electrons that exit the specimen with an energy greater then 50 eV, including Auger electrons.
- The higher the atomic number of a material, the more likely it is that backscattering
- Thus as a beam passes from a low-Z (atomic number) to a high-Z area, the signal due to backscattering, and consequently the image brightness, will increase.
- · There is a built in contrast caused by elemental differences.



Backscattered Electrons are considered to be the electrons that exit the specimen with an energy greater than 50 electron volt, 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 due to backscattering and consequently the image brightness will increase. There is a built in contrast caused by the elemental differences.

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

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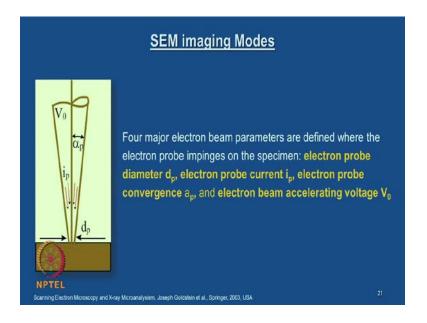
Scanning Electron Microscopy (SEM) One further breaks down the secondary electron contributions into three groups: SEI, SEII and SEIII. SEIs result from the interaction of the incident beam with the sample at the point of entry. SEIIs are produced by BSE s on exiting the sample.

One further breaks down the secondary electron contributions into three groups; secondary electron one, secondary electron two and secondary electron three. Secondary electron one's result from the interaction of the incident beam with the sample at the point of entry. Secondary electron twos are produced by Backscattered Electrons on exiting the sample.

SME's are produced by Backscattered Electrons which have exited the surface of the sample and further interact with the components of the interior of this SME, usually not related to the sample. An SME's and secondary electrons three's 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 variation happens at particular location at the event. And, that is how it has been described in the last two slides. This is just for a clarity. We will just show the effect of these 1, 2 and 3 secondary electrons, when we discuss the contrast mechanisms in detail.

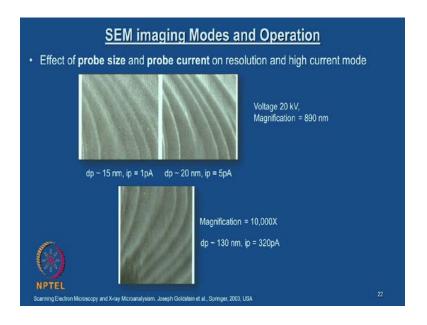
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Now, we will just go to the scanning electron microscopy imaging modes; what are the kinds of imaging mode we employ while we carry out the micro structure investigation using scanning electron microscopy. You see this schematic. This is the electron beam. You have some notation called d p, i p, alpha p and V naught. 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 alpha p, there is a typo here, alpha p and electron beam accelerating voltage V naught.

Please, remember all these 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 talk about these parameters. We will look at the effect of each one of these beam parameters on the micro structural details, which you obtained as well as on the resolution. We will see one by one.

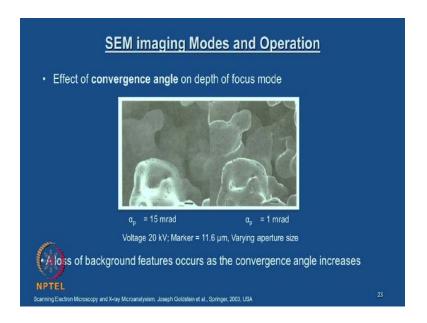
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The effect of probe size and the probe current on the resolution and the high current mode. So, you have a three micrographs of some surface. The voltage employed is 20 kilo volt. The magnification for these two, sorry, this is not magnification. There is something wrong here. The magnification is about 10000 x for all these three. You have d p of 15 nanometers, i p of 1 Pico amperes. And, you see that schematic, I mean, this micrograph b is obtained with the d p in the order of 20 nanometers and an i p in the order of 5 Pico amperes. And, the third micrograph is obtained with the d p of 130 nanometers and i p of 320 Pico amperes.

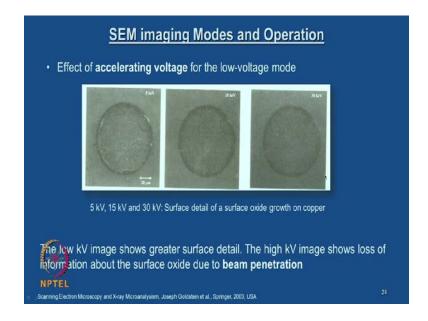
So, what do you see? It is not that you have a specific combination of all these parameters is well defined. You see that as the probe diameter increases, you are not seeing a clear resolution here. The resolution is not improved. At the same time if you increase the probe current also, the resolution is not improved. But at this particular combination of d p and i p, you have a better result compared to these 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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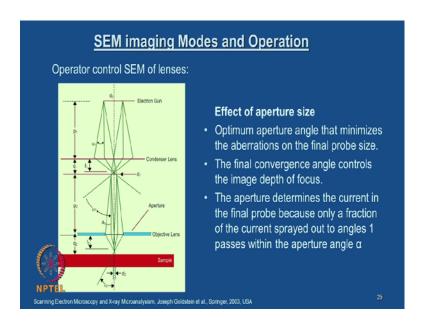
And, next we will look at the effect of convergence angle on the depth of focus. You see the image taken in the same region. Here the alpha p, that is, convergent angle is 15 milliradians and here it is 1 milliradian. The voltage is 20 kilo volts and this marker is 11.6 microns and you have the varying aperture size. You see that a loss of background features occurs as the convergence angle increases. So, if you want; if you 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 lying 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 that the surface detail of a surface oxide growth on a copper is seen with different voltage. You would clearly appreciate that the increase in the acceleration voltage will 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 k V image shows greater surface detail. The high k V image shows loss of information about the surface oxide due to beam penetration.

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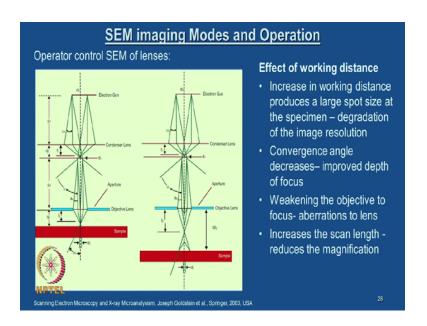
So, now we will see what are all the operator control in SME to obtain a better resolution or a control. We will see that the animation which is showing the kind of aperture size effect. We will see. And, let me first describe this schematic and then we will see what is that we try to understand from 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, where the objective lens. And then, you see finally it reaches the sample. What we are trying to say here is when you obtain optimal aperture angle that minimizes the aberrations on the final probe size. That means, we need to understand what is this optimum aperture angle by looking at the image quality, where relatively it is free from this aberrations. You judge this.

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 sprayed 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 alpha one is 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 objective lens. It controls the final angle from the large sprayed 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 these particular settings will be useful in obtaining the information with minimal aberrations.

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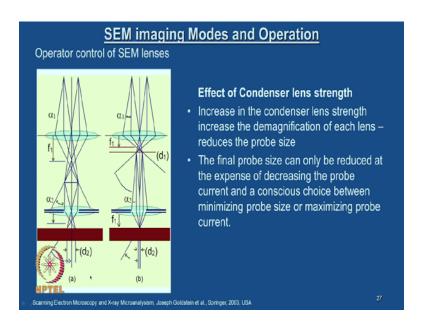


Now, we will look at the effect of working distance. Again, you see these two animations of the ray diagram. And, you see that the distance between the final aperture and the sample surface is working distance, like we defined in the previous one. So, you have this two schematic displaying the ray diagram with two different working distance. This is w one and this w two. And then, the schematic nicely displays increasing the working distance, how your 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 and decrease in that resolution.

Convergence angle decreases and improved depth of focus. And the convergence angle, which we have already discussed. Smaller the convergence angle, we improved the depth of focus. That we have seen already. Weakening the objective to focus aberration to the

lens. So, if you increase the working distance, this also will happen, and which also increases the scan length and reduces the magnification. So, working distance if you play around, these are all the points which we 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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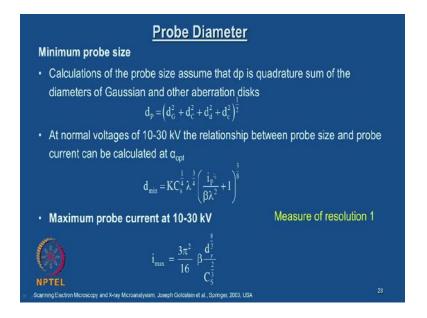


Now, we will look at the effect of condenser lens strength. Here is the schematic again. You can see that the effect of condenser lens strengths the final probe diameter. The increase in the condenser lens strength increase the demagnification of each lens and reduces the probe size.

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 again the kind of information you are interested in and also kind of resolution you want to obtain at particular magnification. So, you can clearly see that for 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.

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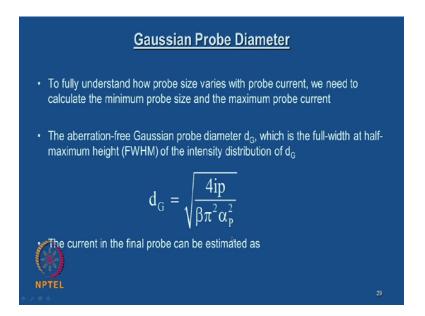


So having talked about these probe diameter, we will go through some of the important aspects to be noted. We are always interested in minimum probe size. If we in order to resolve very smaller details and if you recall in the beginning of this second part of the course, where we talked about fundamentals of electron optics, we also discussed about a quite a bit of 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 what we are now summarizing here.

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 d p is equal to d G square plus d c square plus d c square plus d c square to the power of half. At normal voltages of 10 to 30 kilo volts, the relationship between probe size and probe current can be calculated at alpha optimum d minimum is equal to K C s to the power one by four times lambda to the power three by four into i p by beta lambda square plus one whole to the

power three by eight. And, the maximum probe current at ten to thirty kilo volt has got an expression similar to this; i max equal to three pi square by sixteen into beta into d p to the power eight by three divided by C s to the power two by three.

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And, you 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 and 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, which is the full width at half maximum height of the intensity distribution of d G; where d G equal to square root of 4 i p divided by beta pi square alpha p square.

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$i_p = \sqrt{\frac{\beta \pi^2 \alpha_p^2 d_G^2}{4}}$ • If there were no aberrations in the system, it would only be necessary to increase the convergence angle to increase the probe current at a constant probe diameter NPTEL

The current in the final probe can be estimated as i p is equal to square root of beta pi square alpha p square d G square by four. So, all these expressions will give you a kind of an idea, the important 4 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 the, immediately to the specimen surface. So, that is called probe diameter. So, 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.

If there were no aberrations in the system, it would only be necessary to increase the convergence 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 SME operations and little bit of theory of contrast mechanisms. And, how this equipment can be exploited in order to obtain more micro structural details. We will continue in the next class.

Thank you.