

Techniques of Material Characterization
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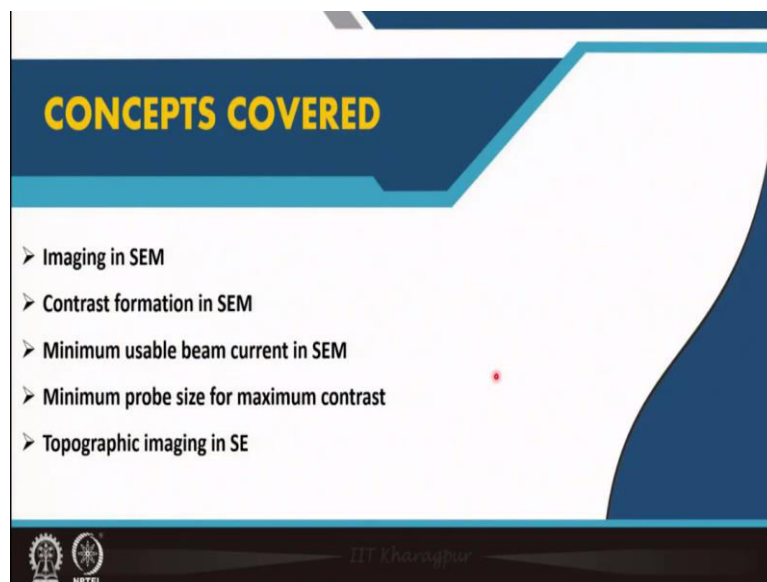
Lecture – 38
Imaging in SEM

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are in 8th week module 8 and we are still discussing about scanning electron microscopy and in this module till now we have discussed about analytical detectors for chemical analysis mostly used for chemical analysis using characteristic x-ray. So, in the first lecture we discussed about EDS energy dispersive spectroscopy detector, how it function working principle, advantages and disadvantages etc.

And in the last class we discussed again about another method of working with characteristic x-ray for elemental analysis that is WDS wavelength dispersive spectroscopy and again we discussed about the working principle of WDS, advantages and applications and all and how it is different from EDS detector and so on. So, with that we close our discussion about various detectors and optics about SEM.

And now we will be starting with the imaging in SEM how the images are forming in SEM, what their contrast, how the contrast generates and so on.

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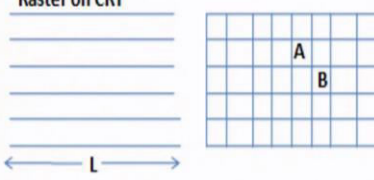
So, we will be discussing here the imaging in SEM, contrast formation in SEM, minimum usable beam current in SEM, minimum probe size for maximum contrast and if possible we will discuss about the topographic imaging in SE mode secondary electron mode.

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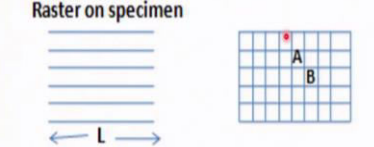
Imaging in SEM

- When an image is acquired, the beam scans the sample surface pixel by pixel.
- In each pixel, the signal is collected by the detector and translated into a (greyscale) value.
- If images are acquired in 8 bits, the range of pixel greyscale values varies from 0 to 255.
- If images are acquired in 16 bits, the greyscale values of each pixel can vary from 0 to 65535.


Raster on CRT



Raster on specimen



- In order to obtain an image in the microscope, we must have some variation in the signal obtained from different parts of the specimen.



So, imaging in SEM we have discussed it before that when an image is acquired the beams scans the sample occurs pixel by pixel. So, we discuss that there is a raster there is this rastering is there and the beam scans from one pixel to another pixel so this is one pixel this is another pixel, this is another pixel and so on and so forth. The beam scans over these different pixels and then there is a corresponding pixel or corresponding raster in the display or image capturing device.

So, there also these pixels are there and exactly and equivalent pixel just a difference in the size which is defined by the magnification itself basically. So, in each of these pixel the signal is basically collected, the detector collect so when the beam scans, when the beam stays over this pixel, certain signal generates be it is a SE signal, be it is a BSE signal, but certain signal is generated from this region, from this pixel.

Now, the detector detects it that is also we discussed how the SE detector, Everhart–Thornley detector works, how the annular BSE detector work and so on and so forth, how the in-lens detector work. So, let us assume that it is any kind of detector ET detector or in-lens detectors or a BSE detector whatever it is that collects the signal, the detector basically collects the signal that is generated from this pixel.

And then this signals strength depending on the strength of the signal and basically depending on the number of electrons and that signal strength can be the number of electrons or their energy and so on. Let us assume that for now it is the number of electrons that is collected certain time and the detector then translates this signal or this number of electron certain time it is translated to a value and usually that value is a greyscale value.

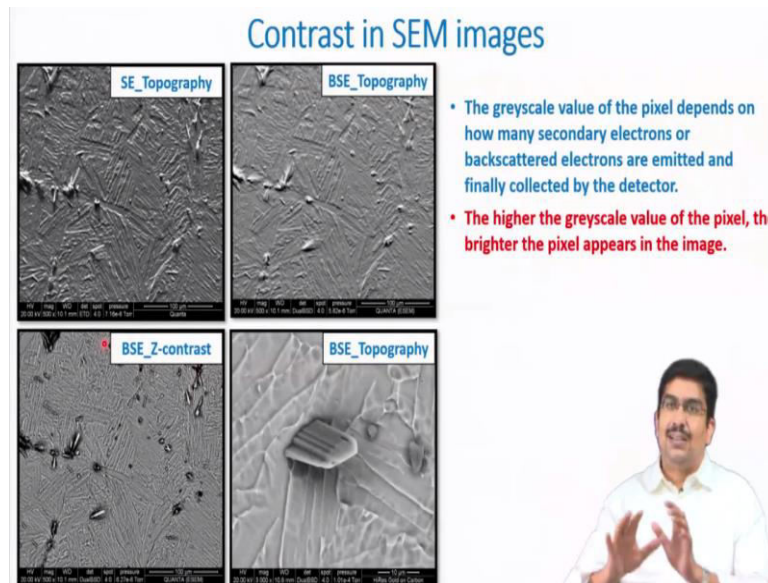
So, if the image is acquired in 8 bits signal the range of pixel in the greyscale the range of greyscale values basically pixels greyscale values that ranges between 0 to 255 and if image gets acquired in 16 bit signal so then the greyscale value will be 0 to this value 65,535. So, this is basically this varies from 8 bits to 16 bits and so on it varies with a multiple of 8 in order of 2 basically.

So, **2 raise to power 8** and so on so that is how it varies and that is how the greyscale values are varies that means in fact if you are capturing this images in 16 bit that means the greyscale value is increasing and you can get much more contrast in your specimen, but whatever it is ultimately this is how it happens the beam stays in a pixel the secondary or backscattered electron is generated.

Then that is detected by the detector and detector translates it to a corresponding greyscale value in the final display whatever. Now, in order to obtain an image in the microscope finally in order to able to see the image you must have some variation in signal obtained from different parts of the specimen. So, if you consider two different pixel you need to have certain difference in the signal which will finally generate the contrast.

Otherwise you will not be able to see anything on the specimen it will be just absolutely if every pixel is showing is giving you the same amount of signal then the entire specimen will have the one single greyscale value and there will no feature nothing will be visible from that image.

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So, if you look at this various different types of images that is produced in a scanning electron microscope. This is secondary electron in a topography mode we will come to all those modes what is topography mode and all. So, this is a secondary electron topography that means this is generated by capturing the secondary electrons here. Then this is a BSE topography mode.

So, this is again a mode this topography is a mode and this is captured using the BSE signal. This also is a again the BSE signal with a topography mode which can give very good basically this before that let us discuss this. So, this is another mode that is called contrast atomic number contrast Z contrast mode and this is typically done with the compositional contrast. This is called the compositional contrast mode.

And this is typically done with the BSE signal SE signal cannot give you compositional contrast we have seen that that SE signal the generation of SE does not depend that much with atomic number, generation of BSE on the other hand depends on the atomic number. So, depending on the atomic number different in a multiphase material depending on the atomic number difference it can generate a certain kind of difference pixel by pixel which can give ultimately some contrast.

So, that is what the chemical contrast here from backscattered electron signal and this one although it is written in the BSE topography, but actually this contrast is coming from both, both the topography and the Z contrast compositional contrast both of these together give this BSE this image. So, we will discuss about all of these in the coming classes, but for now just

remember the greyscale value of each of these pixels basically depends as I discussed basically depends on the number of secondary electrons.

So, backscattered electrons how many of them are emitted when the beam hits the specimen on a certain pixel, how many such SE and BSE electrons are emitting because of the material electron material interaction and finally how many of those secondary electrons and backscattered electrons are collected by the detector that will determine the greyscale value of any pixel.

Now the higher the greyscale value of the pixel the brighter the pixel appears in the image. So, basically the higher the number of secondary or backscattered electrons the higher will be this greyscale value in the detector and correspondingly that regions higher greyscale value means that regions will be appearing brighter. So, we have already discussed about the contrast where I said that the greyscale value of 0 is complete black.

And greyscale value of 255 is completely white so that is it. That means that if certain region in this images if you look at this images for example if you look at here this regions are appearing much brighter than any other region here that means this regions are giving more number of BSE electrons compared to this other regions which are appearing in a darker contrast.

If you go to secondary electron topography mode again certain regions or rather these regions are giving much more number of secondary electron producing more number of secondary electrons that means this greyscale value of these regions are higher and finally these regions are appearing brighter in the end image that is produced. So, this is how the contrast or imaging contrast is generated in an SEM image that is the basic of it.

And similarly you can see that these are the regions which are possibly in compositional mode if you just simply go in compositional mode possible these are the regions the same regions if you compare this BSE topography mode versus BSE compositional modes these are the regions which are now giving less number of BSE because of the compositional effect this is giving less number of BSE.

So, somehow we have reduced this contrast generation here we will come how this is done. So, we have reduced the BSE signal generation because of this topography difference and only considering BSE signal generation the difference in BSE signal generation because of atomic number difference and then we see that these are regions which are appearing bright in topography mode now they are appearing dark in compositional mode because now they are giving less number of BSE signal because of atomic number difference.

So, whatever it is ultimately this greyscale difference is giving you the contrast in the final image and that is related to the number of electrons, number of SE or BSE electrons generated from different pixels and how they are translated into greyscale value by that detectors. So, that is it that is how the contrast is generated in SEM image.

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Contrast formation

- The intrinsic signal from the specimen vary with scan position.
- In order to resolve two points on the specimen, there must be a discernible difference between the signals from these two regions.

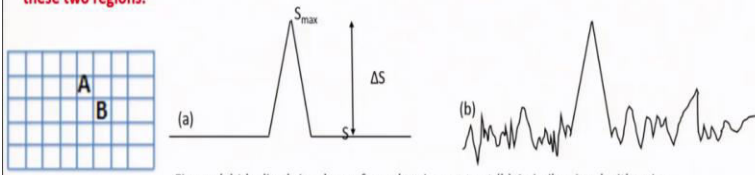


Figure: (a) Idealized signal waveform showing contrast (b) A similar signal with noise

- If we compare the signal S_{max} from one point of the specimen, with the signal S from an adjacent point, then the contrast from the specimen

$$C = \frac{S_{max} - S}{S_{max}} = \frac{\Delta S}{S_{max}}$$

- C lies between 0 and 1, and is called the natural contrast.

Now let us discuss a little more how the contrast is related to the signal. So, intrinsically the signal and you know the signal is basically the number of secondary or backscattered electrons, but collectively we will call them signal for now. So, this signal varies according to the scan position. So, wherever basically what does it mean that if you go move from one pixel to another pixel the signal basically varies.

The number of electron generated secondary or backscattered electron generated will vary and that will finally lead to contrast generation. So, in order to resolve two points on the specimen if we imagine that this is one pixel here A and this is another pixel B here in order to resolve these two points on the final image. These are the pixels on the specimen and there

is a corresponding pixel in the image in order to differentiate them in the image there must be certain difference between the signals generated from these two regions that is it.

So, if we just consider that one of these pixel is giving a signal S_{max} which is here. So this is the kind of signal this is happening. So, I am moving let us say the beam is somehow moving like this and one region let us say the region A is giving a signal S_{max} and the corresponding region the next region adjacent region which is the region B is giving a signal of S . So, the ΔS here is $S_{max} - S$.

Now the contrast we know the contrast is a difference in signal basically. So, the contrast from the specimen we can explain or we can express that as C equals $S_{max} - S$ which is $\Delta S / S_{max}$ so this is how this contrast lies we have discussed it before this contrast lies between 0 and 1 and this is called natural contrast for any kind of image not necessarily an SEM image.

For any kind of image this is the natural contrast for between the two pixel basically the signal difference between two pixel giving rise to this natural contrast.

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Contrast formation

- The signal that is detected in the SEM is not a continuous signal, but for each pixel is derived from the number of SE/BSE arriving at the detector in a fixed time period.
- Because these events are randomly distributed in time, if the average number of electrons detected from a particular point on the specimen is \bar{n} then \bar{n} will vary by an amount up to $\sqrt{\bar{n}}$ about the average value $\bar{n} = S_{max}$.

Figure: (a) Idealized signal waveform showing contrast (b) A similar signal with noise

- The noise N in the acquired signal is defined as $\sqrt{\bar{n}}$.
- In a real situation the variation of signal with scan position is random, with the noise tending to obscure the natural contrast of the specimen.
- Human eye can only distinguish two points on a CRT/digital display if $\Delta S = (S_{max} - S) > 5N$.

Now the signal that is detected in the SEM is not a continuous signal it is not like S_{max} at one pixel and the next pixel will be S , but this is sort of a continuous this is like an analog signal which is continuously varying. For each signal as I said for each pixel basically the signal is corresponding to the number of SE or BSE electrons that is arriving at the detector at certain fixed period.

So, first they have to be generated when the beam is falling on the pixel this electrons the secondary or backscattered electrons needs to be generated and then they have to go to the detector and then detector has a response time and they will be finally detected. So, this entire process has a time response time of its own. There is a finite time involved in the entire process and because of that this signal has this noise.

So, signal is like an analog signal and this signal is basically have a noise because these events are randomly distributed in timescale. So, the average number of electrons detected from any particular pixel if that is n let us say that means the n is basically here S_{max} the signal. So, this average value S_{max} or n that is the \bar{n} that is the number of electrons generated at any given pixel.

Then this n will have a variation by an amount of $\sqrt{\bar{n}}$ so this is the rms value basically. So, average signal is \bar{n} and then the variation of that signal at any particular given point. So, if I take this that this is coming from one single pixel this is the average value which is \bar{n} here that is S_{max} and then on either side it is varying by an amount $\sqrt{\bar{n}}$ that is a noise.

So, this noise of this accurate signal is basically this much. So, in a real situation basically the variation of signal the scan position is completely random and this noise will basically because of the noise the natural contrast will go down in this material because as we define the natural contrast here we have only taken the average value S_{max} here, but because of this spread in the signal the natural contrast will go down between two adjacent pixels because there will be a chance of signal overlapping again the signal mixing will be there because the error associated with one pixel may overlap with the error associated with the another pixel.

So, there is ultimately the signal the contrast between these two adjacent pixel will go down. Now the point is our human eye can distinguish two points on a display or in an image if the ΔS is greater than $5N$ that means greater than the 5 times the noise. If the ΔS the difference in average value ΔS remember it is $S_{max} - S$ that means this S_{max} is the average number of electron detected from this pixel minus the average number of electron detected from this signal.

If this difference is greater than 5 times of the noise value then we can very safely detect the contrast in the image. So, this is the resolution or other way you can think that this is basically the resolution in terms of contrast about our eye. We have discussed that our eye has a spatial resolution. So, similarly our eye has a contrast resolution and this is the kind of contrast resolution.

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Minimum usable beam current

- Human eye can only distinguish two points on a CRT/digital display if $\Delta S = (S_{max} - S) > 5N$.
- The minimum level of contrast which can be observed by human eye (i.e. the natural contrast),

$$C = \frac{(S_{max} - S)}{S_{max}} = \frac{\Delta S}{S_{max}} > \frac{5N}{S_{max}} = \frac{5\sqrt{\bar{n}}}{\bar{n}} = \frac{5}{\sqrt{\bar{n}}}$$
- The minimum level of signal necessary to observe a contrast level of C i.e. $S_{max} = \bar{n} > \left(\frac{5}{C}\right)^2$
- The mean number of electrons detected for each pixel i.e. \bar{n} can be related to the operating conditions of a microscope with a beam current I and a frame scan time F .

Now from this what we can have is that the minimum level of contrast which can be observed by human eye the natural contrast. So, we take C and we express the way we did it $\Delta S / S_{max}$ and we have already seen that this value ΔS has to be greater than $5N$ that means C and this S_{max} is \bar{n} . So, this natural contrast now has to be greater than this value $5\bar{n} / N$ bar.

And finally what we will see that C value the natural contrast has to be greater than $5 / \sqrt{\bar{n}}$. So, if natural contrast is higher than this value then human eye will be able to see that level of signal difference between two adjacent pixels. So, the minimum level of signal necessary to observe a contrast level natural contrast so that means the S_{max} or the average number of electrons that minimum average number of electrons that is needed in order to generate natural contrast C will be given by this expression \bar{n} should be greater than or at the max minimum $5 / C^2$.

So, this is the signal strength the signal strength from each pixel should be this much then only it will produce a natural contrast and we will be able to see that. Now the mean number of this electrons the average number of electrons this \bar{n} this is related to the operating

condition because as we already said the number of electrons here is basically the number of secondary or backscattered electrons generated from this specimen due to the interaction primary beam interacting with the beam.

So, the number of secondary or backscattered electron generated because of the specimen A and then the number of electrons or secondary or backscattered electron detected by the detector both will control this \bar{n} value here the total average number of electrons detected. So, that will be depending on both the number of electrons generated from the specimen due to inelastic interaction and the number of electrons detected by the detectors.

So, ultimately this \bar{n} will be depending on two different factors. One is the beam current that means how many number of electrons were present in the primary beam that will detect that how many electrons are finally generated from the specimen due to this interaction and the frame scan time. Now the framed scan time means how many were up to what time the beam was staying at each of these pixels.

So, these will generate this will dictate that how many electrons will be generated due to the electron material interaction. The beam current how many electrons was there and how much time that beam stays on certain particular point.

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- Assuming a display with frame of 1000×1000 pixel resolution (total of 10^6 pixels), the time, t that the beam dwells on any particular pixel, $t = F \times 10^{-6}$.
- The number of electrons (of charge e^-) which enter the specimen during this time

$$n_o = \frac{I \times t}{e} = \frac{I \times F \times 10^{-6}}{e}$$
- The number of electrons actually detected (\bar{n}) will depend on the beam/specimen interaction, and the efficiency of the detector.

$$\bar{n} = q n_o = q \times \frac{I \times F \times 10^{-6}}{e}$$
 where q is the product of the detector efficiency and the electron yield.
- For secondary electrons, detector efficiency is approximately unity, and electron yield is $\sim 0.1 - 0.2$, so that $q = 0.1 - 0.2$.
- If critical current I_c is required to discern a contrast level C in the specimen (assuming $e^- = 1.6 \times 10^{-6} C$),

$$\bar{n} = \left(\frac{5}{C}\right)^2 = q \times \frac{I_c \times F \times 10^{-6}}{e}$$

$$I_c = \frac{4.2 \times 10^{-12}}{qFC^2}$$



Now, let us assume that display has like one million pixel $1000 / 1000$ pixel resolution that is the frame we imagine that in the specimen both in the both in the specimen raster or specimen number of pixels on the specimen as well as the number of pixel on the display or

image capturing device. In both the case there are one million pixels. Then the time t that the electron beam will stay at any particular pixel will be given by this value t equals F into 10 raise to the power -6 .

So, total scan time if F and t is individual time up to which in each pixel the beam is staying. So, that is given by dividing this F by the total number of pixels. Now, the number of electrons which enter the specimen during this time that means the primary beam. From the primary beam how many number of electrons is actually interacting with the specimen that is this number.

So, that is given by the number of electron present in the beam in the first place multiplied by the time it stays at any given pixel divided by the charge of electron and finally we are getting an expression of this. This is the number of electrons that is entering in the specimen or that is taking part in the interaction. We can also think it in that way number of electrons taking part in the interaction basically that is this many number of electrons which are going inside this interaction volume.

Then the number of electrons actually detected now we are coming to the detector part. As I said there are two parts electron generation and electron detection. So, we have seen that this is the number of electron that is basically generated. If we consider that the efficiency is 100% that means the number of electrons which are entering in the specimen 100% it is generating secondary electron or the backscattered electron which is pretty much a good assumption at least for secondary electron that electrons primarily whichever electron was present in the primary electron beam.

Most of them finally generate the secondary electron then we can imagine that this is basically the number of SE electrons which are generated from the specimen. Then the question come that how many of those electrons that is generated from the specimen are finally detected by the detector. So, this will be again depending on the beam or specimen interaction and the efficiency of the detector.

The number of electrons finally detected by the detector that will be depending on the beam specimen interaction basically what fraction of n_0 is generated or what fraction of n_0 is converted to secondary or BSE electron I already said that at least for SE electrons this

efficiency is unity that means almost 100% of primary electron will be converted to SE electron for BSE it will be obviously much less than this.

So, we are just considering SE signal here and we are expressing this plus the efficiency of the detector how many of those electrons which are generated SE signal, SE electrons are generated will finally reach to the detector. So, both of these together we are expressing with a quantity called q . So, n that is a average value of electrons detected by the detector finally will be a fraction of this n_0 that we mean the number of electrons actually entering in the specimen from the primary beam.

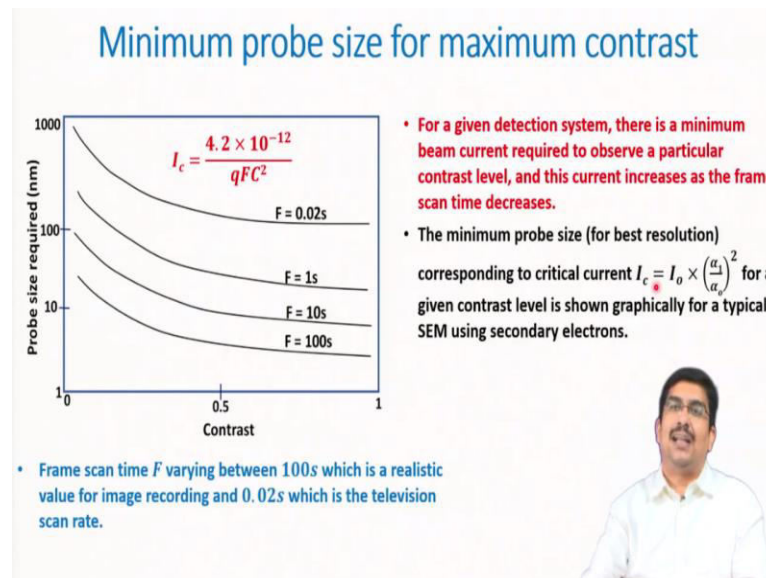
So, this is again given by this expression. So, as I already said q is the product of the detector efficiency and electron yield. Electron yield for secondary electrons actually at least it is pretty much 100%, but here we are taking a value of let us say 0.1 to 0.2 if you take that then q is 0.1 to 0.2 detector efficiency you can imagine that detector is 100% true which is pretty again a good assumption for at least for ET detector that 100% of this secondary electrons are captured.

And it is pretty you can imagine or it is a good assumption for at least the BSE detectors, in-lens detectors and BSE detector that 100% of the BSE electrons are captured by the detector. Now, if I now imagine that there is a critical current of this beam this eye this is a critical current is required to generate a contrast natural contrast in the specimen then we know that this average value this one in order to get a natural contrast I need to have a signal strength of this value.

So, this is the minimum signal strength I need to have in order to generate the natural contrast. If I bring it back and put it here then n bar that is the signal strength minimum signal strength now I imagine that in order to get the natural contrast I will be getting a minimum signal by using a critical current I_c that is the minimum current I need to use in order to get the natural contrast and that is now given by this relationship.

And from here what I can determine is that amount of that critical current which we will depend on this q value and F value that is the frame scan time and the natural contrast all of this what natural contrast I want. So, this critical current and here I have put the values for all other quantities that is electron charge is put here.

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So, from this again I can derive certain condition and I can try to understand the relationship between the minimum probe size and for maximum contrast because again the minimum this critical beam size is related or critical beam current is related to the probe size we have already seen that. The probe size and the beam current these two are related and final resolution is determined by both.

So, for a given detection system the detector and the specimen material interaction there is a minimum beam current that is required, but that much we understand that this is the minimum beam current that I need to have in order to observe a particular contrast level which is the natural contrast. And this current that I_c or rather here this I_c number one it increases with frames as the frame scan time decreases.

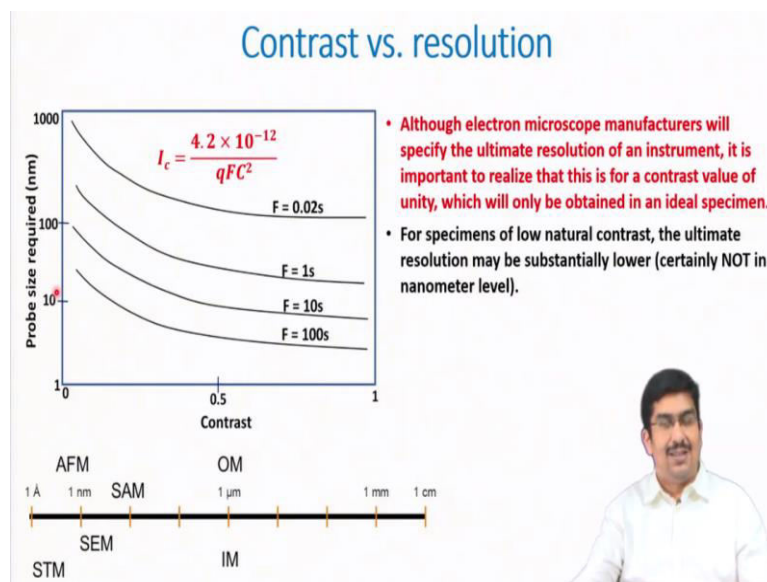
So, if I decrease the frame scan time here this I_c I mean the critical current will increase and also I know that minimum probe size for best resolution the probe size corresponds to this critical current by this equation. So, I_c equals I_0 into α^2 / α_0^2 from here I can calculate the d minimum there which we were discussing in the last week. So, how to calculate d minimum from this I_c the relationship between them.

So, if I do that and then what I can calculate is in order to get certain probe size required to get certain amount of contrast for different frame rate. If I look at here and what we have chosen here a frame scan time of around 100 seconds which is realistic value the image

generally for capturing an image the frame in SEM the frame time is kept around 100 seconds.

Whereas for seeing the images and for moving specimen almost for a live mode we used to use something as scan frame time of around 0.2 second which is called the television scan rate. So, if you use an SEM in the software sometimes you will see that there is a TV mode or live mode and so on and that just simply means that the frame scan time is of this much value.

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So, now what we can see it here from this, that in order to get certain amount of contrast and the most preferred contrast is let us say 1, natural contrast where the difference between two adjacent pixel is the 100% is maximum. So, if I want to achieve that kind of a contrast level the probe size that I will be requiring will be increasing with decreasing frame scan that means if I want to get this contrast again with a minimum probe size.

Minimum probe size means this minimum probe size is required for getting minimum beam current and that is related to the resolution of the material. So, in order to get that kind of probe size very fine probe and at the same time to get a very high contrast I need to increase the frame scan time. Other way round if my frame scan time is constant let us say in order to get if I use a larger beam then I will get a very poor contrast.

In order to increase the contrast I have to decrease the probe current. So, that is the relationship between the probe size and probe size means resolution here so if I want to have

very high level of resolution then I have to increase the frame scan time in order to get very good contrast that is the relationship. So, this way the contrast and resolution is also again related through the beam current.

The special resolution of SEM is related to the contrast that you can obtain through the minimum attainable beam current. And this is sometimes very critical because many a times the SEM manufacturer they quote a spatial resolution of around nearly nanometer. So, for those specimen it is very much optimized for contrast. So, if you want to have a natural contrast very low.

The specimens with very low natural contrast the ultimate resolution is substantially lower because in order to enhance the contrast with a reasonable frame scan time in order to increase the contrast they have to basically make the probe bigger which will reduce the spatial resolution. So, these goes in opposite direction because ultimately what it means is that the contrast and resolution goes in opposite direction for reasonable frame time.

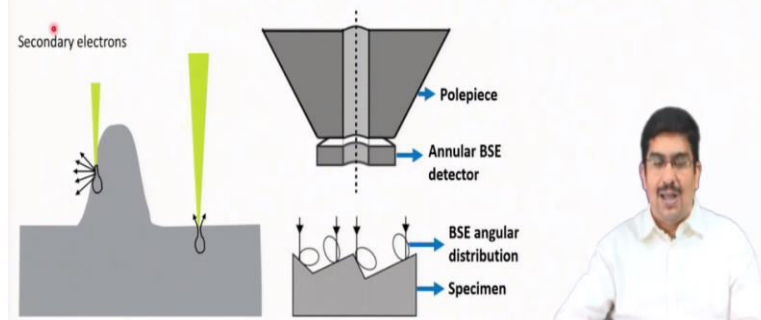
And remember the frame time also you cannot increase arbitrarily because that is related to your charging and lot of others problem, your material may get damaged and so on. So, frames scan time can also be increased up to a level and for that if you have a low natural contrast material you need to have a bigger probe size. So, this one if some SEM manufacture is coating that our machine can give a resolution of let us say nanometer scale.

Then that must be for a material which it shows very high natural contrast which normally that do not sort of they do not reveal that what kind of material they use, but this is the relationship from this relationship you can basically find out that you cannot achieve that kind of a resolution for a low natural contrast material.

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Topographic imaging mode

- One of the principal uses of the scanning electron microscope is to study the surface features, or topography of a specimen.
- Topographic images are obtained most of time using secondary and backscattered electrons.
- The backscattered electron coefficient η and the secondary electron coefficient δ , are both a minimum when the surface of the specimen is perpendicular to the electron beam.



So, with this we will stop here and this part we will be covering in the next class that is the topographic imaging mode and we will be also discussing about the BSE mode. Thank you.