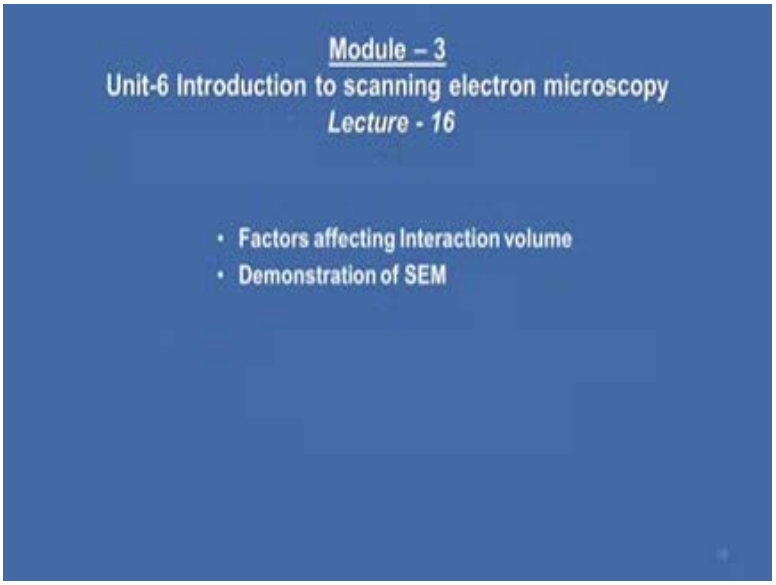
Fundamentals of optical and scanning electron microscopy

Dr. S. Sankaran
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

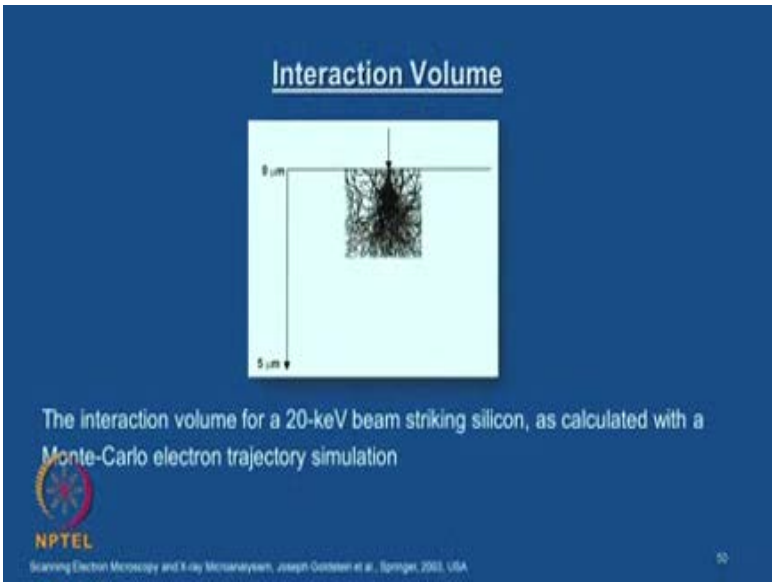
Module – 03
Unit-6 Introduction to scanning electron microscopy
Lecture – 16
Factors affecting interaction volume
Demonstration of SEM

(Refer Slide Time: 00:11)



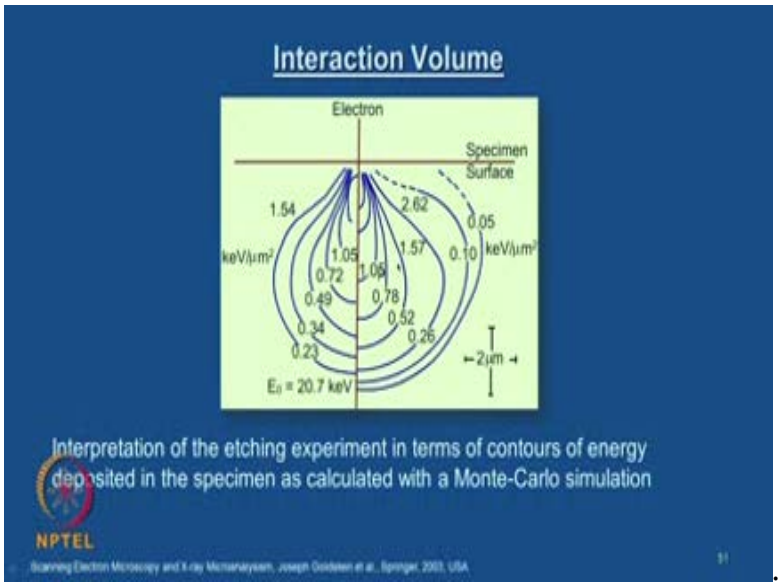
Hello everyone, welcome to this Material Characterization Course. In the last lecture, we have gone through some of the important operation controls of scanning electron microscopy and its effect on beam size and resolution and so on. And then in the last, we discussed about very important aspect of the electron beam material interaction. And then we discussed about the concept about interaction volume and then we have gone through some of the Monte-Carlo simulations based upon the etching experiments using low atomic number materials. I would like to continue from that slides.

(Refer Slide Time: 00:56)



If you look at this schematic again what you have seen is, the interaction volume for a 20-kilovolt beam striking silicon as calculated numerically with a Monte-Carlo electron trajectory simulations. It is very interesting to look at these kind of electron trajectory is you can see that with a dark line and as well as the very light line, and it is going through a quite a bit of volume. You may be wondering that even though the electron probe size is in the order of few micro meter, and if you look at this the interaction volume is quite a bit in three dimension few orders of magnitude more than what you have the dimension in the probe size.

(Refer Slide Time: 02:01)



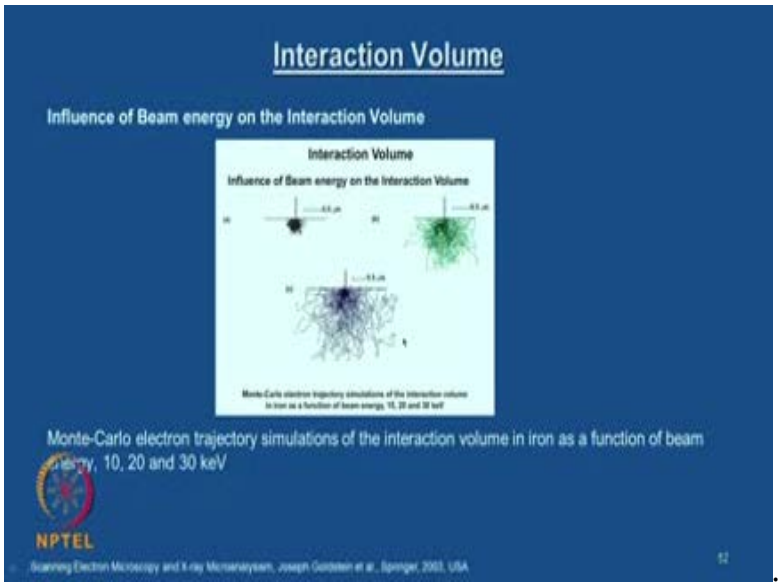
So, we have discussed about this the kind of voltage I mean the energy variation from the specimen surface to the interior of the material. And I just mentioned that this energy contours are generated based upon the etching experiment in terms of contours of energy deposited in the specimen as calculated with the Monte-Carlo simulation. Based on the etching of using electron beam on the low atomic number material like poly methyl menthol relate kind of material, when the electron beam interacts with such material the molecular structure get damaged. And how the damage occurs as a function of depth based on this the energy is been calculated.

And you may wonder that if you look at the shape it is very interesting shapes like a pear fruit kind of a shape. And there is some accountability you can give for this kind of a shape. It is suggested that though the electron beam to start within the specimen surface is penetrating the material with our small region, but eventually it just spread out into quite a bit of an area. This is explained in terms of as the beam enters the specimen, it has got a very high energy, and then as it travels inside the material your inelastic scattering spreads and many events of inelastic scattering and then in combination with elastic scattering it makes the electron trajectories to go around all over this volume.

So, that is how this kind of a volume is generated. So, you may wonder how even though you start with a very small probe, you may wonder that it should be a very straight volume. And because of this scattering phenomenon and which makes these electron trajectories in all over this place in three dimensions to get this kind of an interaction volume shape.

So, as I mentioned yesterday the left hand side contours are based on this experiments and in the right hand side the contours based on the Monte-Carlos simulations which is estimated numerically. And it is very interesting to note that the kind of energy variations from the surface to the bottom. And we will have some kind of idea about how this inelastic scattering signals are useful in obtaining information about the materials in SEM like your secondary electron, back scattered electron, and the characteristic x-rays.

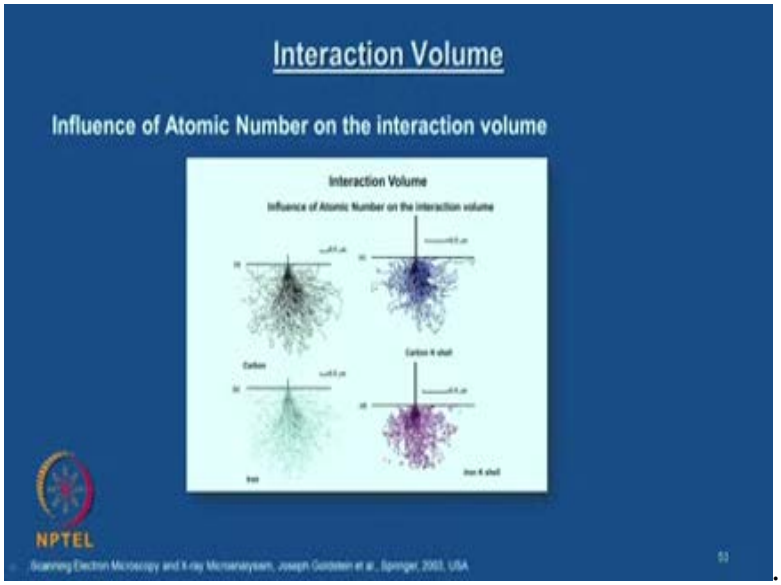
(Refer Slide Time: 05:24)



So, we also talked about this influence of beam energy on the interaction volume. This is another Monte-Ccarlo electron trajectories simulation for the iron as a function of beam energy 10, 20 and 30 kilo electron volt. You can see that with the load kv the interaction volume is small, and as the beam energy increases you can also see that the electron trajectories is spreading wider and wider inside the material. So, you see that from this

slide you can understand that the beam energy also controls the interaction volume of specimen and the electron beam.

(Refer Slide Time: 06:21)



Another important aspect is the influence of atomic number on the interaction volume. What you are seeing is, a carbon and iron, you can see that the kind of interaction volume one can achieve which is predicted by these numerical simulation. And as the atomic number increases, you can clearly see that the volume increases. And one can appreciate that the difference between nonmetal and a metal, you can see that lateral width is spreading compare to the linear width, that we can understand that this is because of the scattering cross section increases as you go with the higher atomic number which is quite obvious.

And then some more examples of this atomic number volume. And you can look at this silver metal, and silver L shell, and then you have uranium metal and then uranium M shell and so on. So, you get some idea about this interaction volume even though you start with your probe diameter which could be very a small. And it is not just atomic number you have some influence of the specimen surface tilt on the interaction volume. You see when you look at the SEM operation which I am going to show you in few

minutes, the specimen is tilted to the required angle in order to collect the appropriate signals in large quantity.

So, the specimen tilt also will have some influence on the interaction volume and hence the outcome the output I would say, for example, it is a secondary electron or back scattered electron volume which is eventually going to decide the image quality and the resolution and so on. So, this is the as the simulation which is shown here as a schematic for 0 degree tilt and 45 degree tilt and then 60 degree tilt, obviously higher the tilting angle also slightly reduces the interaction volume and so on.

So, before I go into the little more detail on the image formation and then interpretation, I would like you to look at the equipment now. I will now show you one of the scanning electron microscopes we have in our lab, and then I will take you to the lab and I will like you to see all the components in detail and then functions so that what were we have discussed so far from the beginning you will be able to appreciate much more clear manner.

So what you are now seeing on the screen is, scanning electron microscope. I am going to explain to you in detail about all these features. And this is how the equipment appears from the front side and this is the specimen chamber. We are going to open this and then tell you what it can reveal. So, you can have a closer look. This microscope is having a thermionic emission, possibly tungsten filament, I will explain to you as we just move around. So, just have a look at the equipment. And this is a display screen where you are going to see the output. I will just tell you this first screen and second screen what all it will show I will tell you in a minute. And the last quadrant shows the equipment inside the chamber what is happening that you can see. And we will go through all this components of this and here is our scholar who is going to operate this and then show you in much more detail.

This is inside the chamber that is how it looks like just you first observe it, and then we will explain one by one. And this is how inside the chamber and the important components are there. First have a look at it, we will go through one by one what are the importance of each one. So, it is a long I mean low magnification, you can see that the

total machine is appearing like this which has two monitors and this is an FEI machine model Quanta 200.

And this has got set EDS attachment. And what now you are seeing is just opening of the chamber again. And this from the side view, this is from the top view, this is the electron column, this is where all your important electromagnetic lenses are there. So, basically this electron column has got a two parts; one is a electron source - this portion, the top portion; and the rest of the column you have this electromagnetic lenses and scanning coils everything is inside. You can have a close look at it. So, this scholar is explaining the total structure of this equipment.

So this is a source, and this is a column as I said. Inside the electron source as we have seen there are various types of sources are available. What you are now seeing is the specimen stage, from the top view this is where you can keep your samples. So, from the specimen stage you understand that at least the size which can fit inside this about 10 centimeter, you can kind of a sample you can analyze in the SEM. And now let us get into the details. And this is the secondary electron detector, which is just below the pole I mean a side by side of the pole base this is the back scattering BSE detector this is how it is like look like. And this is a pole piece and just behind this you can see that your characteristic x-ray detector, it is very difficult to view but we will focus that you can see that tube just behind this that is the x-ray characteristic detector. Then you also have this EBSD detector, we will look at all this usage of this detector and we will perform an actual experiment then you will appreciate much more.

So, you now have some idea about how this inside chamber will look like and this is the chamber which I talked about which is being maintained at very low vacuum. Now, what you are now seeing is the typical a tungsten filament which is this is a failed one, but you can have a look at it, how does it look like this is a tungsten filament just for a demonstration.

So, now we will go into these details of how to perform an experiment. We will take up a particular material, probably a metallic specimen we can take and then we will put inside. And I think before that we will also explain what is involved in the control

system. So this is a sample we are going to examine, and this is a metallic sample where you have two phase. I will show you once we start examining. Just the standard metallographic preparation which we have gone through during optical microscope is good enough for this kind of microscope analysis. You see this sample is placed on the stage now it is going to get, now we are fixing the back scattering detector, just below the pole piece.

And you may wonder why the secondary electron is kept in an angle and back scattered electron detector is fixed just below the pole piece; there is a reason for it. You see the back scattered electrons are high-energy electrons as compared to secondary electrons. So their trajectories are more or less straight, so this specimen is here then the back scattered electrons will directly come and then hit this detector on the other side you can see that this is the region where your back scattered electron will be collected and you are going to fix this under the pole piece like this. And then, but your since second electron is low energy electron it can just trajectories can bend and get into this collector which is a faraday cage basically. And you can also appreciate that this chamber has got lot more slots a vacant, some of the slots are vacant where we can insert any number detectors at least two, three, we can accommodate here two more detectors similar to this can be accommodated here.

So now we will try to close this chamber so that we will start our experiment. The type of specimen which you are looking at in SEM is also depending upon what kind of a vacuum it can handle. There are high vacuum mode, a low vacuum mode, and environmental mode. We will look at it and the monitoring screen, how we adjust these things. And now you are closing the chamber and then we switch on the vacuum pump. So, as I mentioned in the beginning that chamber is maintained with the pressure of 10 to the power minus 4 to minus 5 pascal. And you can see now the sample is placed here, these the fourth quadrant monitor which also shows the inside configuration and what is happening inside.

First screen will display the output of a secondary electron, and the second screen is a output of back scattered electron, the third screen of the quadrant combines these two SE and BSE and then it displays an output. And this is how you can look at your sample

whether it is close to the pole piece or not you can monitor it. So, now, you can see that it is been just closed and opened and then closed again just for the clarity.

So, this window is essential because you will not rise the specimen very close to the tip and then spoil the pole piece and so on you can avoid this some of the major accidents. You can have a close look at the tungsten filament which we have seen. Anyway we will now get into the analysis. So, now we will start collecting the information about the specimen. And we will also go through some of the basic parameters, we will go through some of the important parameter which is listed on the right hand side, and which will give you kind of signal. Once the vacuum is done, it will show the status with the green light so we are ready to go. You can see that that range was in that order 10 to the power minus 5 pascal. And then you have the other controls here. As I mentioned you have a high vacuum mode, where you mostly in the metallic specimens are examined, low vacuum mode the material which requires this kind of a pressure can be employed then environmental, SEM, ESEM where all the biological and biotechnology specimens can be used.

And then you have the pressure monitor here, and then you have the electron voltage control, and then you have also the spot size. So, I hope now you all know what is the meaning of this spot size. And high voltage can monitor this and also you can control the display contrast and brightness using these two consoles. So now, we will slowly go to the imaging details, and how this is going to help us. For this particular specimen we are maintaining this 30 kilovolt. And let us go and you can also see that source of tilt from the position here, and you also will be able to monitor the filament current and filament voltage and so on from the source control. Here that is the second electron output and this is a backscatter electron output.

So, you see that a metal metrics which is having a second phase particle of very high atomic number that is why it is you see that the kind of satellites spots here. Now let us see how this is focused. All this controls are much more easy because of these software interface and everything is controlled by this interface software here, so you do not really have to do any unlock button control like the old equipments. Today everything is computer controlled. And you can see that the kind of information also appears in the

display screen which will also come along with the image. The details like working distance magnification and the region and so on.

So, what now you are seeing is like secondary electron image and this is a back scattering electron image. You can see that all the details you have the date, time, and working distance, magnification. Once this signals are coming from this photomultiplier like I showed in some of the schematic, and you have everything get recorded in the digital format. And you also will have another monitor to control the chemical analysis and you can see that the bright spots are coming because of the higher atomic number contrast or z contrast.

What I will do is, so now you are going with the higher magnification, they are very simple you can just is a mouse click operation in this monitor. And the magnification which we can go with this kind of tungsten filament up to 20 k you can try, but if the source is fulfilled emission gun then the very high magnification with high resolution is possible. To the image formation in this SEM is entirely different mechanisms as compared to your optical or transmission electron microscopy. So, we will discuss about it much more detail in the coming classes, how this image contrast appears and how can it be interpreted and so on.

And I will now show some more details of the, this is we are now trying to looking at the chemical analysis of this using energy dispersive spectrometer which I will introduce. We have not talked about it much more detail. As I said one of the uses of SEM is to look at the chemical composition of the constituent in the microstructure. So, this is achieved by that EDS spectrum which is attached to this. And you also get this information here on particle of your interest and you have the variants like EDS and WDS. WDS is much more powerful in terms of achieving the resolution. And of course, you have a great advantage with the EDS as well we will discuss those things in coming classes. So these are experiments one typically experiments one can perform using this equipment.

And you can also look at the typical fracture surface. This is one of the live experiments which we are conducting, it is a live video. So you can see that we always said that in a SEM the depth of focus is very high. You are able to see the details inside this fracture

surface much more clearly, which is simply not possible with the optical microscopy. And you can also go to BSE mode; you can see that the difference between the SE mode and BSE mode and which is the same region is looked at very high magnification. So, what you are now seeing is a difference between BSE and SE, you can see that the contrast is very bright in BSE mode that means, you have the constituents which is having higher atomic number which is readily revealed by this BSE mode of operation.

So, I think with this you would have got some idea about how this microscope is used. I will stop here with this experiment. We will continue this experiment for obtaining crystallographic information such as, electron back scattering diffraction technique, which I will introduce very briefly in the coming classes. And then we will also perform one of the experiments in the lab, and then we will show the live video how those things are interpreted and then how do you get this information from this tool. So, we will continue that in the coming classes.

Thank you.