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Lecture-16
Materials Characterization
Fundamentals of Scanning Electron Microscopy

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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.

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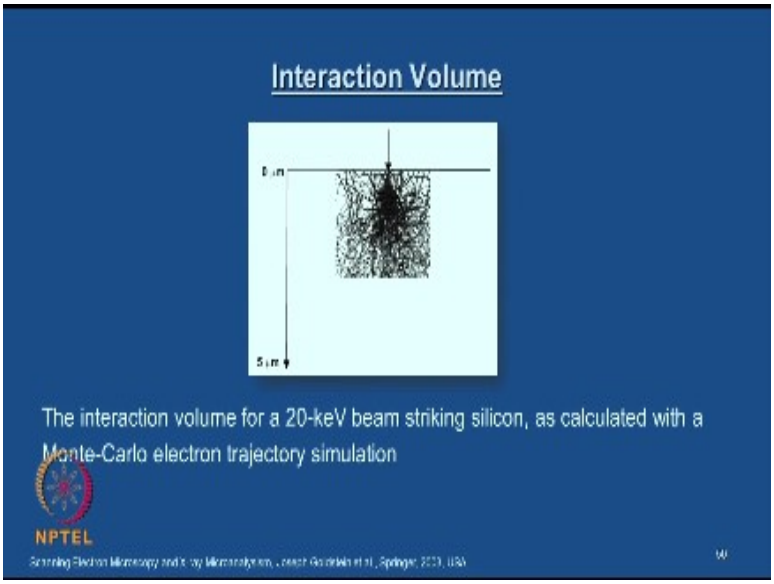

I would like to continue from that slides, if you look at this schematic again what you have seen is the interaction volume for 20KV beam striking silicon as calculated numerically with a Monte Carlo electron trajectory simulations. So it is very interesting to look at this the kind of electron trajectories as you can see that with the dark line and as well as the very light line and you it is going through a quite a bit of volume. You may be wondering that, even though the electron probe sizes in the order of few micrometer 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.

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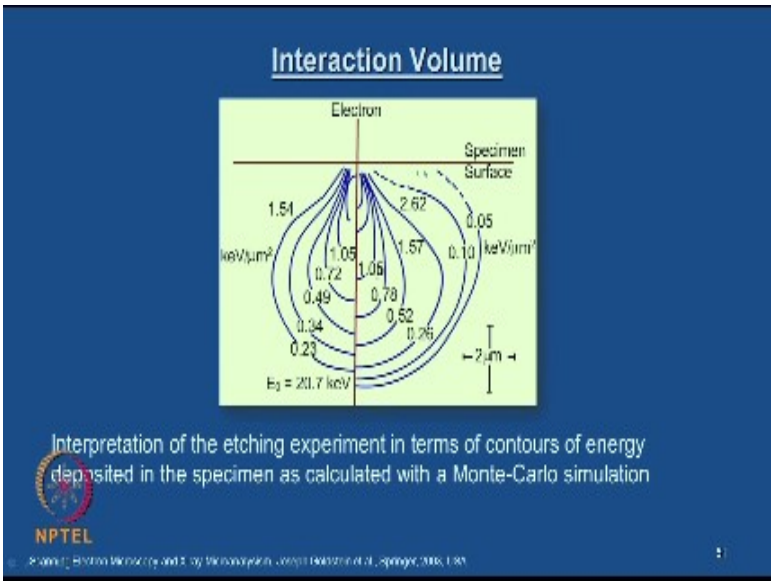

So we will just look at this, we have discussed about this the kind of voltage, I mean the energy variation from the specimen surface to the interior of the material and I just mentioned that this contours, the energy contours are generated based upon the etching experiment in terms of contours of energy dispositive on the specimen as calculated with the Monte Carlo simulation based on the etching using electron beam on the lower atomic number material like poly methyl methacrylate 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 being calculated and you may wonder that it if you look at the shape it is 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 shape. It is suggested that though the electron beam to start within the specimen surface, it is penetrating the material with a small region, but eventually it just spread out into quite a bit of an area. So this is explained in terms of as the beam enters the specimen it has got a very high energy and then and it as it travel inside the material your inelastic scattering spreads and n number 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 the this kind of 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 this electron trajectories in all over this place in three dimension you get this kind of an interaction volume shape. So as I mentioned yesterday, the left hand side contours are based on this experiments and the right hand side or the contours based on the Monte Carlo 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, backscattered electron and the characteristic x-rays.

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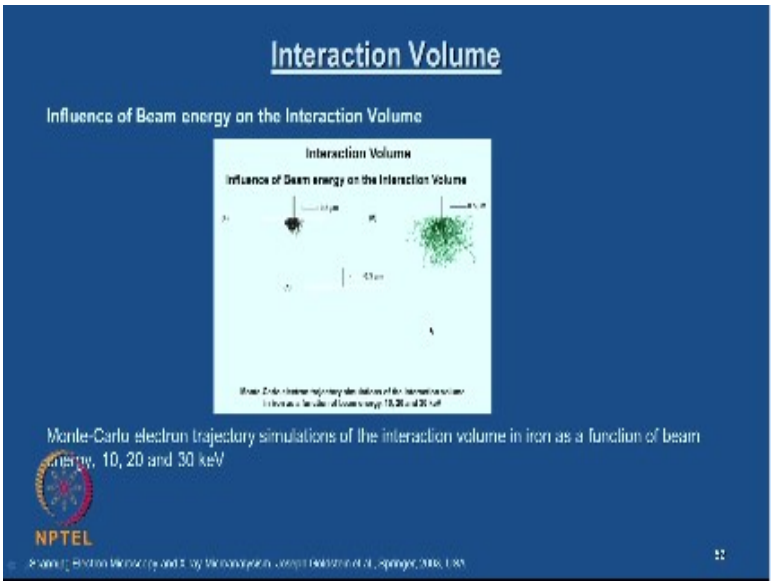

So we also talked about this influence of beam energy on the interaction volume this is one another Monte Carlo electron trajectory simulation for the iron as a function of beam energy 10 20 and 30 KeV. You can see that with the load KeV the interaction volume is small and as the beam energy increases you can also see that the electron trajectories going 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.

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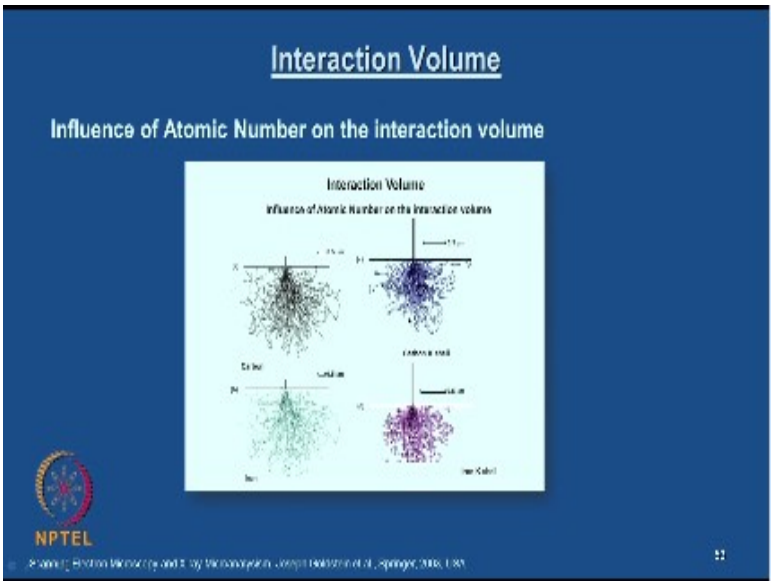

And another important aspect is the influence of atomic number on the interaction volume what you are seeing is a carbon and iron you can see that the kind of interaction volume one can achieve which is predicted by this simulation numerical simulation and as the atomic number increases you can clearly see that the volume increases and one can appreciate that the difference between a non-metal and a metal you can see that lateral width is spreading compared 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 rhenium M-shell and so on. So you get some idea about this interaction volume even though you start with your probe diameter which is it could be very 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 this 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 a backscattered electron volume which is eventually going to decide the image quality and the resolution and so on. So this is the simulation which is shown here as a schematic 0 degree tilt and 45 degree tilt and then 60 degree tilt obviously the more the higher the tilting handle 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 the one of these scanning electron microscopes we have in our lab and I will take you to the lab and I would like to see you I would like you to see all the components in detail and then functions so that whatever we have discussed so far from the beginning you will be able to appreciate much more clear manner.

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So what you are now seeing on the screen is in a scanning electron microscope. I am going to explain to you in detail put 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 at tungsten emission possibly a 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 this the last quadrant show 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 much more detail. This is inside the chamber how that is how it looks like just you first observe it and then we will explain one by one and this is how the inside the chamber and the important components are there first have a look at it we will go through one by one what the importance of each one.

So you can it is a long I mean low magnification you can see the total machine is appearing like this which has two monitors and this is an FEI machine model Quanta 200 and this has got EDS attachment and what now you are seeing is just opening of the chamber again and this from the side view this is from their top view. This is how the again the inside this is the electron column this is where all the important electromagnetic lenses are there. So basically this electron column has got two parts one is 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 the 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 you 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 is which can fit inside this for 10 centimeter you can kind of a sample you can analyze in the SEM. Now let us get into the details and this is the secondary electron detector which is just below the pole I mean side by side of the pole piece, this is the backscattering 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 a x-ray characteristic detector and 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 what is how this inside the chamber will look like and this is the chamber which I talked about which is being maintained at a very low vacuum. And now what you are now seeing is the typical tungsten filament which is this failed one but you can have a look at it what is the 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 the sample we are going to examine and this is a metallic sample where you have two face. I will show you when we start examining just the standard metallographic preparation which you have gone through during optical microscope is good enough for this kind of microscopic structure 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 second electron is kept in an angle and backscattered electron detector is fixed just below the whole piece. There is a reason for it. You see the back scattered electrons are high-energy electrons as compared to secondary electrons. So their projectors are more or less straight so the specimen is here, then the be the backscattered 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 what ever since second electron is a 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 as vacant some of the slots are vacant where we can insert any number of 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 experiments. So all this the type of specimen which you are looking at in SEM is also depending upon what kind of vacuum it can handle.

There are high vacuum modes, a low vacuum mode and environmental mode. We will look at it in the monitoring screen how we adjust these things. And now you are closing the chamber and then relay switch on the vacuum pump. So as I mentioned in the beginning that chamber is maintained at the with the pressure of 10^{-4} to 10^{-5} Pascal and you can see now the sample is placed here. This is the fourth quadrant monitor which also shows the inside configuration what is happening inside and the first screen will display the output of a secondary electron and the second screen is the output of backscattered 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 how it is whether it is close to the pole piece or not. You can monitor it. So now you can see that it has been just closed and opened and then closed again just for the clarity so that in this window is essential because you will not raise the specimen very close to the tip and then spy the pole piece and so on you can avoid this so major accidents. You can have a close look at the tungsten filament which you 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 parameters which is listed on the right hand side and which will give you kind of signal okay. Once the vacuum is done it will show the status with a green light so we are ready to go. You can see that range was in that order 10^{-5} Pascal and then you have the other controls here.

As I mentioned you have a high vacuum mode very mostly in the metallic specimens examined lower low vacuum mode. The material which requires this kind of pressure can be employed. Then environmental SEM or E-SEM 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 spot size and high voltage and monitor this also you can control the display contrast and

brightness using these two console so now we will slowly go to the imaging details and how this is going to help us.

So for this particular specimen we are maintaining this 30KV 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 this source control. Here that is the second electron output and this is a backscattered electron output. So you see that a metal matrix which is having a second phase particle of very high atomic number that is why it is you see that kind of satellite spots here.

Now let us see how this is focused all this controls are much more easy because of this software interface and everything is controlled by this the interface software here so you do not really have to do any you know unlock button control like the old equipment. 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 now what now you are seeing is like a secondary electron image and this is a backscatter electron image we will see how it you can see that all the details you have. The date, time and working distance, magnification this is once this the signals are coming from this photo multiplier 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 a Z contrast. What I will do is so now you are going with the higher magnification they are very simple we can just is a mouse-click operation in this monitor and the magnification which you can go with this kind of a tungsten filament up to 20K you can try. But if the source is field emission gun then the very high magnification & high resolution is possible.

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 how can it be interpreted and so on. And I

will now show some more details of the this, is we are now looking trying to looking at the chemical analysis of this using energy dispersive spectrometer which I will introduce you 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 a 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 great advantage with EDS as well we will discuss those things in coming classes. So these are the experiments one typical experiment one can perform using this equipment and you can also look at the typical fracture surface. This is one of the live experiments which you are conducting.

It is a live video, so you can see that we always said that in 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. You can also go to BSE mode you can see that the difference between the SEM is 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 you with this you would have got some idea about how this microscopy is used I will stop here with this experiment. We will continue this experiment for obtaining crystallographic information such as electron backscattering diffraction, electron backscatter 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 you 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.

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