Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Lecture-42

welcome to this class in the last class we discussed about mostly about the electron microscopy transmission electron microscopy and also we have covered the diffraction and also the imaging now in this class i will introduce briefly to the scanning electron microscope and we discuss about the image formation Then we will introduce to the electron channeling contrast imaging and how these techniques can be used in combination with electron microscope which is transmission and also the atom probe tomography that I will briefly what I will do is I will present all the applications with different alloy systems. So, here the SEM is similar to TEM.

Okay. And both employees electrons beam of electrons they have an electron gun and they have a condenser system they have vacuum vacuum system so most of them are similar as in the transmission electron microscope the only difference is tem provides the internal structure while sem provides the surface or near surface structure of bulk samples Now, here in SCM, the electron gun can be similar to TM, can be thermionic, can be Fexos and the electrons are accelerated between 1 kilo electron volt to 30 kilo electron volt.

So, It has a one or two condenser lens systems. Condenser lens system. The main function of this condenser lens system is to demagnify the beam of electrons. demagnify the beam of electrons and the demagnification after that the electron beam is around 2 to 10 nanometers

Okay, and in this, the electron beam is scanned across the specimen by the coils, and while this electron beam—the primary electron beam—scans the specimen surface, then due to the interaction of the electron beam with the sample surface, there will be secondary electrons, backscattered electrons, and other radiations which are coming and

these electrons—secondary electrons or backscattered electrons—are detected by the detector Okay, and usually, they actually count the number of electrons given off from each spot. So whenever there is a specimen, if there is a beam falling on that particular spot.

From that spot, the total number of electrons coming out of the sample surface is detected by a detector. Okay. And at the same time, while the electron beam interacts—while the electron beam interacts, with the sample. Similarly, there is a spot on the cathode ray tube (CRT) monitor

that, at the same time, is also scanned across. Okay. And the brightness of the spot can be modulated by the amplified current from the detector. As this electron beam scans the surface, there is also a scanning of a spot on the CRT monitor.

So this process is called rastering. It has a condenser lens system, and this condenser lens system's role is to demagnify the electron beam to a particular spot. The electrons coming from the specimen after the interaction go to the detector. As the beam scans or rasters on the sample from that particular spot, at the same rate, in a cathode ray tube, the spot also scans along that particular screen. There is an amplification of those particular electrons which are detected, okay? So that is

directly related to your brightness. This process is called rastering, okay? So this is rastering on the CRT monitor, and this is rastering on the specimen. So, when a length L rasters—the electron beam rasters on the sample and the small L rasters on the specimen—then you can get a linear magnification that equals L by L. And this is, for example, if you have a 10 microns by 10 microns on the sample and if the same spot and a similar spot rastering on the CRT is around 100 mm by 100 mm, then you have a magnification of 10,000X. Okay, so this is the scanning or rastering of the beam along the sample surface.

Here I am showing you the electron Once incident on the sample, then it will penetrate. It will penetrate into the sample surface and it has a this particular volume is called a interaction volume. And in this interaction volume, depending upon the electron energy

E0 and depending upon the radiation which are coming out, the energy of the radiation of the coming out, various radiations are generated as a result of inelastic scattering.

And the energy of the radiation depends on the energy lost when the primary electron beam travels into the sample surface. Okay, and these radiations are not detected unless they escape from the sample surface. And that depends on the type of radiation and also on the specimen. Okay, so here, for example, the X-rays which are generated from the sample surface cannot be easily absorbed by the specimen. Mostly, these will escape because of their high energy.

So here on the left, there is a cross section where you can see that there is an incident beam. You will get an X-ray from the sample, which gives the composition information. You will get Auger electrons, which give surface-sensitive compositional information. You can get primary backscattered electrons. I will come to these backscattered electrons later.

Actually, these backscattered electrons give atomic number and topographic information. There is cathodoluminescence, which gives electrical information related to your conduction band electrons. Then there are secondary electrons, which give topographical information. And these are the electrons which are released from the specimen atoms after the interaction with the beam. Correct.

And this is your sample. So the volume inside the specimen in which the interaction occurs while interacting with the electron beam. That depends upon the following factors. First is the atomic number. The material being examined: higher atomic number materials absorb or stop more electrons.

So they have a smaller interaction volume. Accelerating voltage. Higher voltages can penetrate farther into the sample and generate a larger interaction volume. Also, one more important factor is the angle of incidence. This is: the greater the angle,

The smaller the interaction volume. Correct, and here you can see that approximately this is a schematic where it is shown that at the depth of 5 microns, you can see that as the secondary electrons have much lower energy, they usually come from the top surface. It

is within a micron, within less than a micron. Okay, backscattered electrons will be between 1 to 2 microns, and the range of x-rays, K x-rays and x-rays, these are within 5 microns.

And each volume from which these radiations are generated, each of these volumes is called a sampling volume. Okay, so you can see that there is an interaction volume. And there is a sampling volume. The sampling volume corresponds to each type of radiation which is coming up. For example, the sampling volume for x-rays is much larger than the sampling volume for BSE, and the sampling volume for the secondary electrons. So, the sampling volume definition—what is the definition? The volume of the material which contributes to the signal. Okay, for x-rays,

you can see that the sampling volume is very large, and it is almost equal to the interaction volume because of the higher energy. Okay. In backscattered electrons, the electrons will not backscatter if they penetrate more than a fraction of a micrometer. Okay. And BSE usually originates from a very small region.

Okay. Now, here I am showing you an intensity versus the electron energy. E0 is the incident primary energy. Now, you can see that backscattered electrons are the electrons. Once there is an incident beam, this primary beam actually loses energy while going into the sample.

Interior, and it actually comes back as backscattered electrons. Okay, so it might be possible that the backscattering can occur at a a very low sampling volume, meaning near the surface. So these are backscattered electrons, and these are type A. So there are two types type A and type B. Type A backscattered electrons have very high energy because they lose very little energy from the E0, okay? And also, they have a very good high spatial resolution They have a high spatial resolution because they are coming from only the top surface. However, the electrons which travel

into the sample and then come out lose a lot of energy, so these are the type B backscattered electrons Okay, so you can see in this figure that this particular B corresponds to the backscattered electrons, and it has a large spread. Okay, so they have

been scattered only, so the higher energy It means that the scattering has taken place only a few times. Okay. So, A has a high spatial resolution.

So, you will also get the crystallographic information. B has lost a lot of energy. So, the spatial resolution will be very low. Correct. And one more important part is the secondary electrons.

What are secondary electrons? When an incident beam interacts with the sample, what will happen? The electrons from the atoms on the sample get knocked out, and those electrons are called secondary electrons. And when the backscattered electron travels, And when it comes out of the surface, it might be possible that it has sufficient energy to knock out these secondary electrons again from the sample surface.

So these are SE2, and the initial electrons are called SE1. So, SE2 are the electrons which come out of the sample surface or some sample atoms by the backscattered electrons that are coming out. Okay. So, secondary electrons are generated by both the primary electron and also the escaping backscattered electrons. However, the primary electrons from the generator will be numerous, very high.

Okay, and these secondary electrons usually originate from a very small area or small area near to the beam size. Okay, and secondary electrons have the smallest sampling size. You can quantify them with a coefficient. These are called secondary secondary electron coefficient, which is written by delta, and backscattered electron coefficient, which is by eta.

eta. The terminology is that secondary electrons refer to the number of electrons that are secondary or backscattered from the specimen for each incident electron. or the number of electrons that are secondary or backscattered from the specimen for each incident electron. These are known as the secondary electron coefficient or the backscattered electron coefficient. And here, the N strongly depends on the atomic number. So, electron yield versus atomic number—you can see that for eta,

which is a backscattered electrons, you can see that as your atomic number increases, your electron yield also increases for the backscattered electrons. So, eta is dependent.

But however, the secondary electron delta does not depend upon the mostly on the atomic number. So, here you can see that the backscattered coefficient with the tilt angle. So, as

I just introduced about the interaction volume. So, as I told you, as you increase the incident electron voltage, you can see that the interaction volume also increases. So, interaction volume also increases. So, these are the Monte Carlo electron trajectory simulations. And with depending upon the atomic number,

Also. So, for iron, we can see that the interaction volume is very small, but for the low atomic number, the interaction volume is very large. And also, as I told you, specimen tilt, if you see that, and this depends upon your angle phi, the angle between the incident beam with respect to the sample surface. Now, if it is at 0 degrees, you can see that the interaction volume, and while at 60 degrees, you can see that the

escaping tendency of these electrons from the sample surface increases significantly. So, the electron yield will be much higher as we increase the tilt angle. Fine, and this is the thing which is shown here, the backscattered coefficient increases as your tilt angle increases. Fine. Now, backscattered coefficient versus the energy, you can see that the backscattered one, which, as I told you, when the beam interacts from the top surface itself, these are backscattered.

But there is a backscattered tube which travels along the surface and it comes out. Okay, so the intensity, the efficiency coefficient of backscattered is much higher for the A type. These are A type, these are B type, second type. Okay, and with the tilt angle, as I told you, with the tilt angle, delta theta, you can see that as the tilt angle increases, your secondary electron coefficient also increases.

Okay, so that also depends upon your theta. Now, if there is an interaction volume at the center of the specimen and the interaction volume near the edge, you can see that near the edge, the yield of these electrons will be much higher. But at the center, the yield will be much lesser due to this issue. What will happen? You will get the edge effects in the sample. These are called edge effects. You can see that here, this region is brighter, while the central region is relatively darker.

So, these are called edge effects. Now, if your sample surface is something related to the fracture surface—okay, so you can see that the topographic contrast—if your sample surface is not uniform, then you can see that wherever there is a dip, There, the yield of these electrons will be much less, but wherever there is a tip, the yield of electrons will be much higher. So, you can see that these secondary electrons are easily used for topographic contrast.

So, with this, I will now end this class, and we have briefly gone over the SEM. We also discussed how there is a difference between the backscattered and the secondary electrons. Now, in the next class, we will briefly go through the detection of these secondary and backscattered electrons.