Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

Prof. Surendra Kumar Makineni
Materials Science
IISc Bangalore

Week-12
Lecture-43

Welcome to this class. In the last class, we discussed about the scanning electron microscope, the column and also the scanning electron image and the backscattered electron image. Now, we will just discuss about the detection of these secondary electrons and the backscattered electrons. You can see in the right side, this is your pole piece this is your detector okay and exactly at the bottom there will be a sample your sample surface

okay so this is your inside chamber of the sem sem inside chamber okay so in this for detection this particular detector is the cross section is shown in this in in the left side figure where you can see that this is your final pole piece your electron beam interacting with the sample and the specimen is tilted at a certain angle and when the electron interacts with the specimen so it will generate the secondary electrons and these electrons when it gets ejected out usually these secondary electrons will strike a scintillator okay example it's a phosphor screen this is your scintillator And this phosphorous cream, when these secondary electrons strike, it emits light.

So here there is a cross section of a scintillator. You can see that the incoming secondary electrons will go and hit to the scintillator and this scintillator emits light or photons. And this light is passed through a light pipe which is our light guide, we can tell, and into a photomultiplier. So, this section is called a photomultiplier, where the photons are again converted to the pulse of electrons, pulse of electrons.

And these electrons, these pulse of electrons is nothing but the amplified version of that particular light or the photon, okay? And Secondary electrons, usually they have a very low energy. It is around 50 electron volt, less than 50 electron volt from 10 electron volt to 50 electron volt. And they have a very low to excite the scintillator.

Okay. So that's why what we do is first is they are accelerated bias voltage. So this bias voltage is around plus 50. 10 electron volt or it can be a 10 kilo electron volt or 12 kV. So, this is applied

remember the size sign is the plus and these are applied to a aluminum which is a thin film which is covering the scintillator so this is a aluminum coated on the scintillator and which has a positive voltage which is around plus 10 kilo electron volt or up to 12 kilo electron volt okay so now along with this aluminum scintillator these are actually connected to a Faraday cage and this Faraday cage is nothing but a it's a metal grid it's a metal grid and which also called as a collector and this is kept at a few volts 200 volts plus 200 volts okay and there is a very important role for this Faraday cage

the first thing is it prevents the high voltage of scintillator affecting the electron beam the electrons which are coming towards the scintillator, okay? And also it improves the collection efficiency by guiding these secondary electrons towards the scintillator, okay? So, there are the two functions of the metal grid or the Faraday gauge, okay? So, this is a detection of secondary electrons, okay?

Fine. If in the case of backscattered electrons, if in the case of backscattered electrons, they also hit the ET detector. Okay. And these detectors, these detectors are called Everhart-Thornley detectors. And along with the secondary electrons, it might be possible that the backscattered electrons will also hit this scintillator.

So, the image which is formed from the secondary electrons with this detector is not 100% secondary electrons. It might also include contributions from the backscattered electrons. So, now if this particular scintillator has been switched off, This plus delta kV has been switched off, or you can apply a small positive voltage. Then what will happen is these secondary electrons are excluded from the detector, and only the backscattered electrons will be detected.

But in this method, what will happen is the backscattered electrons traveling towards the scintillator can be detected because backscattered electrons can travel it's a high-energy it can travel in any direction. So, the electrons traveling towards the scintillator can be detected by ET detectors for the backscattered electrons. So that's why for backscattered

electrons, the efficiency—the geometrical efficiency—is very low when using the ET detector. For backscattered electron detection, we usually use a scintillator called a Robinson detector. This Robinson detector can be seen in the schematic.

It is kept just above the specimen, just below the pole piece. This is similar to the ET detector, but it is placed above the specimen so you can collect or detect the maximum number of backscattered electrons. This Robinson detector has very high efficiency. Nowadays, there are also solid-state detectors available. These solid-state detectors are also placed just below the pole piece, above the specimen, but they work on the principle of a p-n junction in silicon. It's a semiconductor, so when backscattered electrons hit the p-n junction or the silicon, it generates electron-hole pairs.

When a voltage is applied across the p-n junction, it produces a current. This current can be amplified, detected, and converted into contrast or an image. So, this is how backscattered electrons are detected using solid-state detectors. Now we will cover the optics of SEM. So this is the basic thing where there is a filament which produces a beam of electrons, or we can call it a monochromatic beam of electrons with a current of I0.

So this is your V, and assume that the size of this particular one is D0. Fine? Or we can call it a filament diameter. Now, if you put a condenser lens of focal length fc, what will happen? Depending on the focus strength, it will demagnify the image of the filament at position v1.

Okay? From the condenser lens, assume that this particular diameter is d1. Okay? Then the d1 can be written as d0 multiplied by v1 divided by u1. Here, v1 can be given by the thin lens equation. It can be given by the thin lens equation. Now, if you keep an objective aperture of

focal point F0, F objective. The particular beam which is traveling can be further demagnified to a value related to D. And this D is given by D1 multiplied by V2 divided by U2. Here, V2 is nothing but the working distance from the sample surface. So, you can write D1 multiplied by the working distance divided by U2. This is your final probe diameter, which will scan across the sample surface.

Now, if you need a smaller D size, what should we do? The thing is, here the main—the condenser lens strength—controls the beam size. Meaning the focal—if you increase the condenser lens strength, your FC will reduce. If your FC reduces, then your V1 reduces. That is the distance of that particular spot.

From the condenser lens. If the V1 reduces, your D1 reduces. And if you see this particular formula, as the D1 reduces, you can see that your D also reduces. So, for a smaller spot size, what we actually do is use a higher condenser lens strength by changing the—that changes the focal point of the condenser lens. So, one more additional thing to note is if there is spherical aberration in the objective lens, you can actually use the aperture to reduce the spherical aberration.

So, this is the basic principle in SCM to control the beam size, especially with the condenser lens strength. Now, coming to the pixels, which we describe as a picture element—or we can refer to it as a pixel—what is a picture element? The picture element is, for example, if the minimum size of the spot is, say, 0.1 mm Minimum size of the spot or 100 microns, then a 100 mm square CRT can produce around 1000 by 1000 pixels. Okay.

So, the size of the pixel is given by P equals 100 divided by m micrometers. m is the magnification. This is your pixel size. Now, This pixel size is directly or importantly related to the resolution also.

So remember in an earlier class, we described that the resolution is the minimum distance at which two objects can be separated to resolve A and B. So now, for example, if in your sample there is a sample feature A and sample feature B, and if you want to resolve A and B, the resolution should be such that these two should be differentiated. Okay. And to resolve this A and B, the most important thing is that A and B should be in two different pixels.

Okay. So the working resolution of any microscope is no better than the pixel size, which is P. Okay. So if your probe diameter D, which is the final probe diameter on the sample, is greater than P, For example, here this is a D and each pixel size is P. Here the D is

greater than P. What will happen? The signal from the adjacent pixels will also contribute to it, due to which your resolution will degrade.

Similarly, if your D is smaller than one pixel, then what will happen? You will get a very weak signal per pixel. and so you will generate a large amount of noise right so the d should be optimum so d should be equivalent to the pixel size then you will get then you can differentiate the two different objects from the sample surface okay so this is the basics related to your optics of sem Now, I did not went details into the scanning electron microscopy.

So, it itself is a complete course, but what I want to bring about this is just to understand that how imaging takes place in the scanning electron microscope. So, in the next slide, what I will describe is the controlled electron channeling contrast imaging. Now, For example, if you have a sample with a lattice structure, correct? So, this is your sample with a lattice structure.

Then when parallel electron beam hits to the sample, it will penetrate into the sample surface, correct? So, it goes into the unit cell or the crystal structure. Now, In TEM, what we learnt is these unit cell has a certain crystallographic planes and depending upon the orientation, they get diffracted beams, correct? So, as in TEM, we call that if they can get the Bragg diffracted depending upon the orientation.

So, your electron beam gets diffracted. Once they diffract, then it might possible that they also heat to the crystal heat to the atoms and they can get the backscattered electrons okay so in tm this diffracted beam and the incident beam are collected so you can get the bright field or the bright contrast fine so this is the principle of tm now if you have a thicker samples what will happen these parallel electrons get backscattered So, they get backscattered and then these electrons, backscattered electrons will be detected by the BSE detector and you will see a dark contrast.

So, these backscattered electrons coming from the deep inside the material carrying the information. So, now if there is a presence of defect in the crystal lattice, correct? Now, In the left side, the beams which are falling on the crystal lattice and these are producing

the backscattered electrons and detected by backscatter detector. This is called channeling of electron beam.

So now assume that there is a presence of defect and this parallel electron beam travels across this crystal. Then due to the presence of defect in TEM, what will happen? It gets diffracted. So in bright field image, wherever the defect is present, then those appears as a dark contrast or those appears as a dark contrast lines because it gets diffracted from those defect regions. Now in SCM when it is in the bulk sample the backscattered electrons due to the presence of defect the backscattering will increase.

If there is an increase in the backscattered increase from those defect locations, then relatively you will get a bright contrast from the defect region as compared to the surrounding dark contrast region, which is due to the backscattered electrons. So, in the presence of defects, the backscattering tendency will increase. So, locally there will be an increase in the intensity of the electrons as compared to the surrounding structure. So, you will get bright contrast lines or defects in the presence of the dark contrast surrounding region.

So, this is called electron channeling. So, you are channeling the electrons. And you are getting contrast imaging from the defect regions. Here, I am showing you two micrographs. These are SCM micrographs taken by the backscatter detector.

And here, you can see that we are representing a certain direction. g zero two bar zero—what is this zero two bar zero? It means that the crystal is oriented in a two-beam condition of zero two bar zero. So, it means that there is only a transmitted spot and only a zero two bar zero diffracted spot, okay? And which is in this direction. Now, it means that you are only allowing 0 2 bar 0 diffracted beam through the lattice, correct? It means you are channeling that 0 2 bar 0 diffracted beam only, and you are actually avoiding all other beams. So, this is in the two-beam condition. That's why you can see that the surrounding—the whole grain—is almost dark, correct? Now, if there is a presence of defects,

correct. So, what will happen? These backscatter electrons—what will happen? They will be diffracted at these locations very easily, and you will get a higher intensity. Due to this,

you will get a higher intensity at those lines or the dislocations. So, you can see these are the dislocation lines—these are the dislocations, correct? So, you are only channeling the 0 2 bar 0 through the lattice, so they get absorbed. But due to the presence of defects, there will be backscattered electrons generated from those regions at a higher intensity relative to the surroundings that is why your surroundings appear dark. However, at the defect locations, these regions are bright.

So, here I am just briefly going through the EBSD. What is EBSD? This is electron backscatter diffraction. So, here the principle is the sample is tilted at an angle, which is usually 70 degrees. relative to the sample stage, but it is 110 degrees to the BSE detector, fine.

So, once the sample is tilted, so this sample tilting is only to elongate the sample volume. As I told you, the signals from the BSE signals will be much higher if your angle is higher, correct? And the electron beam falls on a very small volume on the sample surface. Typically, the spatial resolution is around 20 nanometers. And as these electrons interact with the sample, they get backscattered and leaves the sample.

Once these Bragg scattered electrons leaves the sample, usually the primary beam loses some of the energy. And as I told you that if there is a, these are called inelastic scattering. And when inelastic scattering occurs, it might possible that the electron beam are in the Bragg's condition from the crystallographic planes, specific crystallographic planes. Now, if these are in the crystallographic planes and they are in the Bragg's condition, they can produce the Kikuchi lines which we described in transmission electron microscopy and these Kikuchi lines or the Kikuchi bands can be detected by using a EBSD detector near to the sample surface.

So, these are very similar or very same as the Kikuchi lines which are detected from the inner transmission electron microscope. So, here you can see in the right side, you can see these Kikuchi bands. This is a particular zone and remember as I told you the thickness of these Kikuchi bands are directly corresponds to the D spacing, corresponds to the D spacing. or the g of that particular plane correct so these are the so these you can

do on the bulk samples to get the crystallographic orientation of that particular grain so in this there is an example of two grains in a single phase

One grain is at different orientation and another grain is at a different orientation. And if you scan your probe or if you put your SCM probe At this location, you will get this particular Kikuchi pattern, but at this location, you will get a different orientation. So, you can see that with this, you can measure the orientation between these two grains or the orientation of these grains. Okay.

So, this is a typical unit cell which are oriented as per the Kikuchi line and this is your for the second grain. Okay. So, based on this Kikuchi patterns, actually you can generate the color maps. And each color, each color corresponds to a particular crystallographic direction. Okay.

And these are called IPF maps. And if you see the pole figure, if you, then this pole figure is for a cubic structure, usually the low index 1, 1, 1, 1, 1, 0. So you can see that 0, 0, 1, 1, 1, 1, 0 corresponds to different colors. So this corresponds to green, blue, and red, uh, green, red and these are blue.

So, you can see that this particular sample most of the grain is textured along 110. So, this means most of the grains are oriented along the 110 direction. So, here you can briefly see how in an electron microscope we can obtain electron channeling contrast imaging by channeling the electrons, and with the presence of defects, you get the backscattered electron intensity higher at those locations. These backscattered electrons can also be used for electron backscatter diffraction patterns to obtain the orientation map. Now, I will show you how this orientation map can be used to determine the crystallography of defects in a bulk sample.

So, in this demonstration, I will show how to use electron backscattered diffraction to obtain a properly controlled electron channeling image. So, here there is a cross-section where there is an incident beam and a specimen chamber. There is an EBSD detector, and the specimen is tilted at 70 degrees. When the electron beam falls on the specimen, it generates backscattered electrons. Since these are inelastically scattered electrons, they

produce Kikuchi patterns, which are detected by the EBSD detector. So, you will obtain a Kikuchi pattern and the orientation map of a particular sample.

Now, if you place a backscattered electron detector, and position your sample very close to it, and if you align your crystal in such a way that it is in a two-beam condition where the diffracted beam is channeling through the sample surface inside the sample, then under this condition you can actually obtain ECP patterns on the BSE detector. So, the backscattered electrons can produce an electron channeling pattern on the BSE detector, which is positioned above the specimen.

So, if you image these electrons, if you image these electrons correctly, you will see a grain where the grain appears much darker because you are channeling only one electron beam. So, the grain becomes much darker. Based on this principle, what we usually do is first record the We record the EBSD pattern on a sample surface. So, and here you can see that we have selected a region of interest which is in the green color.

So, the green color corresponds to 110 direction. It is a cubic pattern. So, ROI is 110 direction and with this orientation, you can get your pole figure of 110. which is a initial orientation of that particular grain. Then this particular orientation can be simulated back into a crystal software, which is a single crystal software to determine the, so to determine the misorientation angles.

Correct? Once these angles are determined by the single crystal software, now it is in the exact zone of 110. Now, what we do is from this pattern, you can simulate your Kikuchi pattern. So, this is your real Kikuchi pattern, simulated Kikuchi pattern from that particular region which has a grain which is oriented along near to 110 direction. Now, based on the tilting of X or Y and the Z, Z is the rotation.

and in SEMs, mostly the tilt is either X or either Y. So, usually it is X. So, based on the X and Z actually you can reorient your Kikuchi pattern such that it is in the channeling condition or the Bragg's condition. Once these X and Z values are recorded for the two beam condition, those angles can be again used for the sample stage Z moment and sample stage X moment. So you are reorienting your grain to the two beam condition.

Now, if you reorient it in the two-beam condition, you are channeling that particular diffracted beam. Then, when defects are present in the sample surface or on the sample surface, those get highlighted by the bright regions. So, this is the standard pattern for electron channeling contrast imaging, which is called controlled electron channeling contrast imaging, because you know the orientation of that grain and, based on the orientation, you are actually seeing the Z and X getting the Z and X for the two-beam condition, and you are putting it in the sample stage,

okay? So, you can see those steps in the ECCI. First, there is an initial microstructure; then, you do the EBSD. You are getting the orientation, which is nearly pink in color but is close to red. So, it is near to the red, meaning it is near to the 001. Then, you simulate this particular orientation map in a single crystal and get the exact simulated Kikuchi pattern from that particular grain orientation. This particular Kikuchi map can be reoriented by Z and X to get a proper two-beam condition.

So, you can see that in this particular case, this particular beam is in the two-beam condition, and you can get the stereographic projection. These Z and X angles are again incorporated into the SEM stage, and you can actually record the ECC image from that region. Now, you can see a large amount of contrast related to the dots, and also at the interfaces, you can see the bright contrast, which was almost absent in the initial microstructure. So, after going to the two-beam condition, you can see that these correspond to the defect regions.

The presence of defects in the microstructure, fine. So, these are the steps in the ECCI. Now, ECCI is very useful. You can actually map or you can quantify the defects in a bulk sample. And as compared to the TEM, TEM is a very localized information, correct.

So, ECCI, usually the lateral resolution is around 8 to 10 nanometres. TEM it is 1 to 2 nanometers. Depth of observation is 50 to 100 nanometers it is 100 to 200 but the observation area is much much higher than the observation area in the TEM samples which is 4 order of magnitude higher. Then samples can be bulk samples here you can you have to use the thin films okay. So this is the difference between TEM and ECCI.

Now similarly for the thin foils you can also do the transmission kikuchi in sem which is called transmission kikuchi diffraction okay so this is for the thin foils so this was first proposed by taylor and guys in 2012 usually what you do is you prepare you prepare a tm sample you load on a special holder correct and that particular holder is tilted around 38 degrees as as compared to the um ebsd angle then here then your beam your beam falls onto the sample and whatever the transmitted

beam which is inelastically scattered this beam inelastically scattered produces a kikuchi map which can be detected by the EBSD detector. So, that is why it is called a transmission EBSD. Here special resolution is very high down to 2 nanometers. Conventional EBSD is 20 nanometers.

Sample is electron transparent here and these are mounted horizontally and back tilted away from the EBSD detector and very short working distance of 4 mm. So, here the diffraction patterns originate from the bottom surface of the sample and a smaller diffraction source volume. So, this is shown schematically, and this is for an APT needle, which is an aluminum alloy where this aluminum alloy has different grains, and these grains can be mapped by transmission Kikuchi diffraction. So, this is related to transmission Kikuchi.

So, first we introduce the SEM. Then we briefly go to the electron channeling contrast imaging. Then the importance of EBSD orientation maps and how these orientation maps and electron channeling contrast imaging can be used together to get the defect structures on the bulk samples. So, these are bulk samples. For the thin foils, we are doing this in the TEM, but even in SEM, we can do the transmission in BSD, where the electrons transmitted across the sample

are inelastically scattered, producing the Kikuchi pattern on the EBSD detector, which can be used to get the orientation of a particular grain or the crystal structure. So, here I am giving you a very nice example of a superalloy. So, you can see in this that usually these superalloys have gamma prime and gamma precipitates. So, these are the two-phase structures: gamma prime and gamma matrix.

So, gamma prime is ordered, and gamma is a solid solution. Now, these are the creep-induced samples, these are CREP samples. So, you can see that this is an SCM image, a backscattered image taken in a GIS microscope, where you can see that there are certain bright features located inside the gamma prime phase. And these bright features are exactly similar to what we see in TEM of stacking faults.

So, this particular grain is oriented in two beam Bragg's condition and you can see there are four variants of these defects, these fault planes A, B, C and D. And you know this orientation of the grain now because it is in the channeling contrast of 0, 0, 2 bar. So you get the stereographic projection and based on the stereographic projection actually you can measure the angle of these stacking faults which they are making from the sample surface. So stacking faults A and D belongs to 1, 1 bar and 1, 1, 1 dislocation planes and One, one bud.

and triple 1 planes having an angle of 36 and 38 degrees with the surface plane. So, you can measure the angle of the stacking fault from the sample surface. Stacking faults B and C appear as a sharp line which appears that is almost at 80 to 90 degrees from the sample surface, okay. So, and these straight lines corresponds to the stacking faults which usually we will see in the microscope. Now, you have a orientation.

of that particular grain and stereophilic projection is there. So, you can actually do the Z and X tilt from the software and you can put these values in your stage so that you can get the different two beam conditions. So, here you can see that that particular region. So, usually in these alloys, the displacement vector is 1 by 3, 1, 1, 2. And we can do similar G.B or G.R invisibility criteria as it can be done in the TEM.

Fine. So, you can see that here in the 1 by 1 condition, your stacking fault B is invisible. Your stacking fault B is invisible. Okay. So, these faults.

These faults are invisible. But in the 1 bar 1 1 bar condition, your stacking fault C is invisible, and in 2 bar 2 0, both A and D become invisible. Correct. So, based on this, you can do the G dot B analysis condition and actually determine the nature of stacking faults. Or the nature of stacking faults present in the sample.

So, this is how we use ECCI to determine the Burgers vector of the dislocations and the displacement vectors for the stacking faults. So, as I discussed about SEM, you are rastering the beam on the sample, correct? And you are getting the information as each sample At each spot interacts with the sample surface, and it rasters on the sample surface. Similar to that, we can control the beam in a transmission electron microscope which is we called as a scanning transmission electron microscope.

So, the beam scans parallel to the optic axis at all times so that it mimics the parallel beam in PM even though it is a scanning. So, this is how you can control the the beam with the deflection scan coils. Okay. So, the stem image quality depends on the probe size.

So, there as I told you the probe size is between 2 to 10 nanometers. So, the probe size here in TM is sub nanometer. Okay. So, the probe size is very fine and you can there might be aberrations which can be corrected so that your probe size become very sharp so that you can get the atomic resolution imaging also. Okay, so we can use in transmission electron microscope also.

Now here, so as you are scanning beam, scanning the beam on a sample surface, okay, and the sample has a different features. okay and you can see that this is a carbon film and this is a gold sample on the carbon film so both have a different atomic number so what will happen when your beam scans on the sample surface depending upon the atomic atomic number actually the it can scatter at an angle theta higher is the atomic number higher will be the scattering angle so it can generate it it can it can scatter by theta 1, theta 2 or theta 3 depending upon the atomic number.

So, theta 1 is the highest greater than 50 milli radians of axis theta 2 and theta 3. So, based on this, what we call is a HADAAF, which is a high-angle annular dark-field detector. So, here, what you can see is that if your beam transmitted beam—if your electron beam transmits and if you are putting a bright-field detector here that so which collects the transmitted beam—then that is called a bright-field image, a bright-field STEM image.

But if the beam is scattered by an angle theta and if there is an annular dark-field detector Because dark-field means the scattered beam—the intensity of the scattered or diffracted beam—is taken for the imaging. These are called annular dark-field images. Now, if it scatters more than 50 radians, okay, it is near to or greater than 50 radians, and this depends upon the atomic number of that particular element.

Then these are called high-angle annular dark-field detectors, which is the HAADF image. Here, I am showing an example where this is an aluminum alloy. An aluminum alloy, which has copper precipitates. Both have different atomic numbers. Copper has a higher atomic number.

Now, you can see that this is a dark-field image. So, these are the copper-rich plates. Copper-rich plates. Aluminium is the matrix. So, as the beam strikes these copper-rich plates, because of the higher atomic number, it will scatter at a higher angle.

So, what you will get is more signal in the high-angle dark-field detector. So, you will get a higher intensity. So, the intensity here scales up with your atomic number. So, that's why you can see the contrast between the two regions: aluminum and copper-rich regions. Similarly, you can see that there are three regions here: P1, P2, and P3.

p2 is related to col, P1 is related to ammonium-rich CO3, and P3 is the matrix. You can see that aluminum has a much lower atomic number than the moly and niobium. So, that's why the P1 and P2 phases become much brighter but the aluminum contrast, you can see, is relatively darker. Okay? So, these all scale up with your atomic number. Okay? So, as I told you, if the probe size reduces, your resolution also improves.

If the probe size is in the order of the lattice, near to the lattice spacing, then you can actually get atomic-resolution imaging. However, there are certain aberrations which are very important. Chromatic aberrations, spherical aberrations. Okay. So, chromatic aberrations can be avoided by using a monochromator, where you are actually playing with the source.

But these astigmatism and spherical aberration can be corrected by using several special types of CS correctors. Okay. So, these CS correctors are used for correcting the spherical

aberration so that your probe size becomes almost near to the lattice spacing of any material, allowing you to achieve atomic resolution. Here, I am showing you an aluminum alloy.

These are your copper-rich precipitates. You can see that the bright intensities correspond to copper-rich atomic columns. So, this you are seeing this sample in a particular zone axis, and each spot corresponds to an atomic column. These bright spots correspond to a copper-rich atomic columns as compared to your aluminum spots. Okay, so with this, I will end this class now

So, Briefly I have gone through some basic methods which can be used with the scanning electron microscope in correlation with the transmission electron microscope and how these can be additionally used with atom probe tomography that will give you a brief example with certain applications related to some structural materials in the next class.