Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

Prof. Surendra Kumar Makineni
Materials Science
IISc Bangalore

Week-12

Lecture-41

So, welcome to this class. Now, in the last class, we described some contrast image contrast, and we described the intensity variation as a function of s and t. These two are very important, as is also the importance of Psi g, which is the extension distance. Now, we will see how this contrast is used to analyze the defects in the samples. So, I will just briefly go to the examples. Okay, so, we have gone through those two equations, okay? Those two equations resemble

related to your d phi0 and dZ and the amplitude of the change in amplitude of diffraction with respect to the thickness, correct? So, here the first term, as I told you, corresponds to the scattering from the undeflected beam, which is related to your phi0 in both terms, okay? And the second term corresponds to your phi g scattering from the diffracted beam. Now, if if you assume there is a crystal which has a certain defect, okay?

If you have any crystal and if there is any defect or dislocation, a crystal defect disturbs the operating planes. It means that locally, due to the presence of a defect, the deviation parameters will change. What are the deviation parameters? Especially the S, okay?

This is due to the bending of the planes. This is related to your bending of the planes. Okay, and in this particular case, if there is a presence of a defect in the crystal, then you will have an additional term which is related to your forward intensity, which is related to 2π g dot r. Okay, and this g dot r, g corresponds to your operating diffraction vector. Or we can call it a reciprocal lattice vector.

Okay? And R represents the displacement of atoms from their lattice positions. Due to the defect, and this displacement may actually vary along the Z, okay? So, this R is the displacement vector. Now, G dot R, okay?

So, here G dot R is the condition where the defect has an effect on the operating planes. It means that if your G dot R equals 0, the displacement does not disturb the operating planes. So, what will happen is your defect will be invisible. So, we use the invisibility criterion to identify the nature of the defect. So, how is the nature of the defect identified?

It is related to the Burgers vector. Correct, it is related to the Burgers vector. Now, the Burgers vector depends upon your crystal system and the type of dislocation present in the sample, okay? So, you can use this g·R criteria to identify when the defect will be visible and when it will be invisible. This criteria is very useful to know the Burgers vector of any dislocation in the sample.

So, here there are two images where you can see reflecting planes, correct? And these are called your incident beam; these are reflecting planes, and you can see that the orientation in the first case—the orientation of the dislocation—so this is your dislocation, which is present on a certain other plane. This dislocation—the Burgers vector of the dislocation—is in this particular direction. Correct?

Now, you can see that this particular dislocation does not affect the incident beam because it is carried on those particular reflecting planes. But in the second case, where you have the lattice planes, okay? These are the lattice planes. You can see that these are not on those planes. The defect is not present on those reflecting planes.

So, these can diffract. Okay? So, this is how we can see the invisibility criteria for the defects. Now, I will give you two or three examples of dislocations and also stacking faults.

Fine. Now, in the first case, I will talk about stacking faults because stacking faults, grain boundaries, and phase boundaries are all present in most microstructures. Okay? So, on the left side, You can see the schematic of the orientation of certain planes, okay? And these planes are cut by a stacking fault, and you can see that it imposes a plastic strain. Okay? In the first half, in the first part, this is your first part, and this is your second part.

In the first part, you can see that R is equal to 0 for all positions above the fault plane. Correct? But in the second part, R is not equal to 0. R has a certain value. The deviation has a certain value below the fault plane. Okay. So, while going across the stacking fault, your R changes.

It means that the fault has a displacement, but you can see that the deviation parameter S is equal at the top and also equal at the bottom. So, you are assuming that your S does not vary. So, there is no bending in the sample. So, there is no change in S. But there is a change in the R. At the top, R equal to 0. At the bottom, R equal to R. It might possible that the sample is bent and the fault has misorientation also.

So, that S also changes. It might possible that there is a bending in along with your R changes. So, this also has a contribute towards the diffraction contrast. So, in certain circumstances, we call it as R as a displacement and your S contribution is called the misorientation.

Okay? And it is quite possible that the contrast in imaging can be resultant of both R and S can be resultant from the both R and S. So, based on the appearance you can see that the at certain locations at each locations there is a change in the while going the intensity goes along the thickness it your R changes while moving across the stacking fault and due to the interference between these beams that the beams that are diffracting

in the upper part of the crystal there is a difference between the beams the diffracting in the lower part of the fault. So there will be a phase change at the fault plane. So, depending upon the vector which is used for the imaging purpose, if your g.r is equal to 0, then the stacking fault contrast will be nullified or will be 0 means it is if g.r is equal to 0. your stacking fault will appear as a single crystal.

There is no contrast in the image. Okay? So, in this figure, the R for this particular case is 1 by 6, 1, 1, 2. And you can see that in this third condition, if the G is 2, 2 by 0, then your G dot R will become 0, and you can see that the faults are not visible as these fringes in the third case.

So, this is called the invisibility criterion to identify the stacking faults in the sample. So, this I am talking about when you have a single grain with a stacking fault across the planes. Correct? It is possible that you will have a grain boundary where you have two grains, and your deviation parameters S1 and S2 are totally different. Your lattice spacing is different.

So there is a G1 and G2. Correct? So these correspond to your grain or phase boundary. So if there is a presence of a grain boundary, what will happen? There is a change in the orientation of the grain while going across the grain boundary when you have an incident beam.

So there will be a change in the orientation. So diffraction conditions on both sides cannot be described in terms of the same G. Your G value changes. So because the inter-laminar spacing of this grain and the inter-laminar spacing of this grain totally change. Okay.

So for one grain, if you set up a two-beam condition with G and G1 and S1, another grain might not be in the two-beam condition. They might be in some G2 S2 condition. Okay. So the grain boundary contrast is usually similar to the thickness fringe contrast.

For the first grain, it might be possible that the first grain contributes to the intensity, but the other grain will not contribute. Due to this, there will be a variation in the intensity while moving from one grain to another. So you can have a diffracted grain that is bright, and another grain will be dark. So here, there is an image showing the presence of a grain boundary. You can see these fringes, which look like thickness fringes.

This is due to the grain boundary, which is present in differentiating the two oriented grains. Now, in this case, it is a very simple example of line defects. Imagine these are the planes—crystallographic planes—and you can see there is an incident beam and a presence of a dislocation. Due to the presence of the dislocation, you can see that these planes are bending near the dislocation. So, there is a bending of planes.

So, at the core of the lattice, at the core of the dislocation, the lattice is bent severely. And the extent of the bend decreases as we move away. So, if your crystals are in a two-beam condition, if you orient your crystals in a two-beam condition—near to the two-beam condition but not in the Bragg condition—then it might be possible that the incident beam Due to the bent nature of the planes near the dislocation, they get Bragg diffracted.

Bent planes. They get Bragg diffracted. This means that these planes are in the $s = 0$ condition where there is Bragg diffraction. It might be possible that the planes... Away from the dislocation, those are not in the exact Bragg condition or S is very large.

So you will see a contrast difference in the imaging by the dark lines you can see in this image. It means that at those locations, the planes are bent even though your particular grain is not in the Bragg condition, but due to the bent nature, it turns out that your S becomes 0 and there will be diffraction. So, in the bright field, you will see dark lines. Those indicate the presence of dislocations in the image.

So, here the R, you do not have to remember this equation, but it is a function of your X, Y, and Z. For the dislocation, the displacement field of a dislocation in an infinite isotropic solid is given by this equation. Here, mu is the Poisson's ratio, B is the Burgers vector, and BE is the edge dislocation. Similarly, for screw dislocation, R is given by B phi over 2 pi. Okay, so this is how we can identify what type of dislocation is present, and usually what we do is tilt your sample.

You orient your crystal in such a way that, under different g1, g2, g3, and g4 conditions, you can take the image from the same region where the dislocation is present, correct? And based on the vector analysis, g dot b. Actually, you can get the invisibility criteria where you can identify what type of Burgers vector is present for that particular dislocation. So, this is how we can identify the nature of dislocation in any sample. Here, a very nice schematic view from Professor Hovey's book shows that there is a presence of dislocation.

This is your bent bent planes, and you can see that away from the dislocation, S is greater than 0, and down here, S is both where your S is greater than 0, meaning it is in both these locations; your planes are away from the exact Bragg's condition, which is S equal to 0. But due to the bent nature of this dislocation, you can see that S is equal to 0 at those bending regions. So, what will happen?

You will see a change in contrast in the diffracted intensity. Fine? So, this can be used to identify the conditions where these dislocations will be invisible or not. Assuming that this is your bent plane. Okay.

And this is your dislocation present, and your Burgers vector is in the right direction. Assume that the delta K points into the plane of the paper in this direction. This means your G vector is into the plane. Fine. Now, if you see the front view.

Your g vector is into the plane. The g vector is into the plane, so your g dot b equals 0. It means, in this case, your dislocation will not be visible. But in this condition, if you see in this particular condition, your g vector is into the plane. Into the plane. So, your G dot B is not equal to 0. So, you will see the contrast.

Based on this contrast, here it is shown schematically that if you have a 4 dislocations in the orientation 1 and in the orientation of 2, if you take a bright field from a transmitted spot, you will see that all the 4 dislocations are visible, correct? If you do a 2 beam bright field image or the dark field image from this particular spot which is having a G vector in this direction, correct? So, the Burgess vector for this particular in this direction, it is in this direction. So, you can see that the G dot B for this particular dislocations is 0.

So, you will see that these are invisible. In this condition. If your Burgers vector is, if your g vector is in this particular direction, you will see that these two dislocations are invisible in this condition. So, based on that, you can actually, by using the invisibility criteria, solve which Burgers vector corresponds to what type of dislocation in any sample. So, this is just briefly I have gone through your defect analysis using the diffraction analysis, which corresponds to g and B vectors.

Here, there is another nice example. So, you can see that this is in the zone of 110, and these are the dislocation networks. This has a dislocation network which is formed in a superalloy, which is FCC, correct? Now, you can see that the dislocation segments are divided into these four segments. Which have Burgers vectors in different directions, fine, and based on the g condition,

like this image is taken in 002 bar where all the dislocations are visible, the dislocations which are taken in 1 bar 1 bar, and 1 bar 1 3, you can see that in this condition, these dislocations are invisible, most of the dislocations, and in this condition, the other dislocations are invisible. Okay, now we will just go through another contrast, which is, as I told you, about the phase contrast. We have described the mass-thickness contrast, we have shown the diffraction contrast, which is used for the imaging purpose, dark-field, bright-field imaging, and also how to do the defect analysis in the sample.

Now we will move on to the phase contrast. As I mentioned in the last class, phase contrast occurs when an incident beam falls on the sample, and the transmitted and diffracted beams merge to form an image called a phase contrast image. It is the constructive and destructive interference of those beams. Okay, and this usually gives a structure at a very local scale, at the angstrom level.

Okay, so here on the right side, in the bottom image, you can see a nanoparticle. If you zoom in on this particular region, you can see bright intensity spots that are almost uniform. These intensities are nothing but the resultant phase or the resultant intensity of the total transmitted and diffracted beams. This results in a series of bright spots on the image. These are nothing but the atomic structure of the sample from the region, okay.

Now, if you go to a two-beam condition, for example, you can see this is your central spot, the 00 spot, and this is your 1-bar-1-bar-1 spot, correct. Now, if it is in a two-beam condition, it means you are allowing only one set of particular planes for diffraction. What does this mean? There will be interference only between the central spot and this particular diffracted beam, due to which you will see intensities corresponding to only the 1-bar-1-bar-1 particular plane.

So that is why you will see these lines. And these lines are nothing but the atomic planes. You can see that if your G vector is in this direction, as I told you in few classes before, your trace of that particular plane corresponds to perpendicular to this particular G vector. So, these lines are corresponds to the 1 bar 1 planes in the sample and if you are taking this phase contrast image in a zone axis where you are getting a uniform intensities of the deflected spots.

Then what will happen the intensities of these all spots will have a resultant on these two bright regions this is this is it means that this particular crystal is in the zone axis and this is nothing but the atomic structure along that particular 110 direction fine based on the two beam and based on the conditions and if for example it is possible that you have a sample there is a precipitate inside your sample which is having a different D spacing or orientation and your matrix has a different D spacing, okay. If you go for a high resolution imaging when an electron beam falls, the diffraction from your matrix and the diffraction from the precipitate,

they will generate the diffraction intensities at different G values or at different G values. And if you take a profile, if you take an image at this particular condition, G1 condition, so that you are making a diffraction spot only from the matrix, you will get these lines which are the planes which corresponds to the matrix. If you take the precipitate, spot X for the imaging then you will see that the prespirate has a different lattice spacing but these are parallel both

the spots correct similarly if you put the if you take if you put the objective aperture on both the spots the D which is a matrix, the X which is a precipitate, then what you get is you will get the overall intensity summing of these two intensities of these planes. So you will get this type of pattern. And these are nothing but your Moray fringes. And actually you can deconvolute these intensities

To resolve the actual spacing of that precipitate and the matrix. Similarly, with the different G1, G2, and G3 values and depending upon the aperture size, what type of beams you can select, you can get different types of moiré fringes, okay. You can see these particular moiré fringes. So, you will get different intensities which correspond to a summation of these intensities of these two diffraction patterns. So, this is called phase contrast imaging in the electron microscope.

In an electron microscope, as in the last few classes, I have gone through some basics to understand and just to give a feel that in transmission mode, what type of contrast you are getting, what type of analysis we can do; we have discussed the diffraction analysis. We have discussed the diffraction contrast, images, and also the stacking faults. So, these have several applications. So, you will have the nature of stacking faults, vacancies, and interstitials.

You can actually have anti-phase boundaries. They have a different They have different planes and the Burgers vectors. Low-angle, high-angle boundaries, strain boundaries, coherent interfaces, strain fields, precipitates, and orientation can be investigated by an electron microscope. Now, whatever analysis we are doing up to here is related to the structure.

As you go with my first class, where I talked about the use of electron microscopy for structural analysis, correct? All these analyses can be correlated and used with the atom probe, okay. So I will end this class now, and in the next class, I will briefly talk about the advantages of the atom probe over electron microscopes when used for chemical information, okay.

So we can get the chemical information in TEM by using energy-dispersive spectroscopy. However, the advantages of the atom probe will be covered in the next classes. Thank you.