Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-12

Lecture-40

So, welcome to this class. In the last class, we have just gone through the indexing patterns and also about the Kikuchi patterns, which are used to go from one zone to another zone by keeping the g vector the same. And also, we talked about different zone axis. In this class, what we will do is we will talk about the image contrast—how we getting the image contrast in TEM. And there are three different types of image contrast what we get it. The first one is mass-thickness contrast.

The second one is diffraction contrast. And the third one is you can get the phase contrast. Okay, so here, the mass-thickness contrast is related to your angular range and the energy spread that is affected by elastic and inelastic scattering. Okay. So here, we can also use also the aperture to stop all the electrons to stop all the electrons which are scattered by which are which are scattered. by an angle alpha. Okay.

So, this is mass-thickness contrast. So, if you have a higher mass so if you have a sample and if your area this particular area contains a higher atomic mass element, a higher atomic weight, or a lighter , then you will see that the scattering from this higher atomic mass or higher atomic weight will be much higher. So, you will get a dark contrast in this region and you will get a bright contrast in this region. Okay. Similarly, the thicker samples the thicker regions of the sample will have a dark contrast,

And the thinner samples will have a bright contrast. This is a very simple mass-thickness contrast. So, here, usually the regions of the specimen which are thicker and have high density will scatter more strongly. And this will appear as dark in the image, okay? This is very important for biological samples, okay. So, what we will do is, in biological samples, what they do is usually these are amorphous.

So, the sample regions which are important will be stained by heavy metals. For example, they will mostly use osmium, and this is used to decorate the specific features that lead to higher contrast during imaging. Okay, so this is an example of the mass-thickness contrast. So, all specimens—whether this is an amorphous region, a crystalline region, biological, or inorganic samples— Okay, so all these contrasts you are getting can also be superimposed.

So here, both, as I told you, elastic and inelastic scattering occur, which actually results in a change in direction by a small amount. Okay. So, this is related to the mass-thickness contrast. What is the diffraction contrast? So, diffraction contrast corresponds to the principle related to the diffracting intensities.

Okay. And this is the most important contrast which we use most regularly in the transmission electron microscope. So it comes from the diffracting electrons leaving the lower surface of a specimen, which you can also refer to as a crystalline specimen, and these diffracted beams are actually intercepted by the objective aperture in the lens system, okay.

So, some of the diffracted rays are actually intercepted by the objective aperture. So, they are prevented from contributing to the image. So, this diffraction contrast is dominant when it is greater than 15 Angstroms. The object details or the feature details you want are more than 15 degrees or 15 Angstroms.

Now, as I told you, if you remember, there is an object to a lens, okay? Then, at the back focal plane, you get a diffraction spot with a transmitted spot, and these spots at the back focal plane allow you to place an aperture on any of them, so you can remove their contribution from the imaging, correct? So, those diffracted spots will be absorbed by the objective aperture. So, whatever image forms on the screen will not include that particular diffracting plane. Correct?

This way, you can actually do the bright field and the dark field imaging, or you can also do the center dark field imaging. I think in the last class, I briefly talked about how to take the bright field and dark field images. Diffraction contrast is also important for dislocations if there is a presence of stacking faults and other defects. Okay, so here, if any compounds are there which have a different composition, they can actually alter the structure factor F theta. So, it will directly affect the intensity, correct? This is the type of

Then, one more important parameter is the tilt angle, okay? So, it will produce a tilt angle of the specimen. Then, if your sample is not flat, if it is a bent sample, what will happen? Those bent regions might be drag-diffracted. Due to which, you will get dark regions, okay.

So, these are called bend contours, okay. Now, the third contrast is called the phase contrast, okay? Phase contrast. Here, the electrons which leave the specimen after interaction—these electrons which leave the specimen after diffraction—they actually recombine to form a high-resolution image. Okay, so it directly resembles the phase contrast present at the exit surface of the specimen, okay? And this phase contrast—the difference in phase contrast—is converted to the intensity differences in the image, okay.

So usually, this phase contrast is used if your sample features are less than 10 angstroms, okay. So these are the different types of image contrast which we can get in a transmission electron microscope. Now, first we will understand how the intensity is generated on the image, okay. So, what we can do is calculate your total intensity. It can be given by I0 plus Ig, where I0 resembles the direct beam and Ig is the diffracted beam, okay. And the Ig, the diffracted beam.

So, first we will talk about the diffracted beam. The intensity can be proportional to the phi g, which is called amplitude, and it is given by pi t by psi g square dot sine square pi t as effective. divided by pi T as effective whole square, okay? So, here there are certain terms which we need to understand. So, we will just describe them one by one, okay?

And this terminology is very important to understand the diffraction contrast, okay? And this particular equation, Ig equal to this, is the solution to calculate the intensity of the diffracted beam, okay. So, here there are several terms in the equation. The T is thickness, thickness of the sample. Psi g is the extinction distance of the selected g vector.

So, here the xi g is given by pi V cos theta B divided by lambda. So, here V is the volume of the unit cell, theta B is the Bragg's angle, lambda is the wavelength of the beam, and

Fg here is the structure factor. Okay, so for any given material, for any given sample material, in this xi g equation, your volume, lambda, and Fg are known. So, here the only thing which is unknown is theta B, which is the Bragg's angle. So, your xi g is proportional to your theta B.

So, xi g for different planes is different. For example, the xi g of 100 is not equal to the xi g of 111. So, this is the definition of extension distance. So, a more physical interpretation of extension distance will be discussed later. But to understand this equation, you have to remember that this extension distance is directly proportional to your theta B. So, your extension distance for different atomic planes is different.

Fine. Now, there is another important term in this equation: S effective. Okay, this S effective is called the effective excitation error. Effective excitation error. What is effective excitation error? Effective excitation error is given by S effective equals the square root of S squared plus 1 divided by xi g squared.

Okay, what is S here? S is the deviation parameter. It is the deviation from the perfect Bragg's condition. Remember in the last class, I described to you about the relrods. Correct?

And when your sample is present here, if you have some crystallographic planes, if your beam is interacting and it is forming, it is forming certain spots. These spots, because of the thickness, Z thickness, are few unit cells. Then what will happen? The intensity along the Z will get broadened. So, you will see the relrods.

But in the X and Y direction, as the number of unit cells is infinite, you will see a strong spot. So, in the X and Y direction, you will see a very sharp spot. In the Y direction, you will see a very sharp spot. But in the Z direction, you will see a very diffused intensity. And this diffused intensity is related to the appearance of the relrod.

So, the maximum will be at the center, and it will diminish. And the deviation from Bragg's condition is called S, which is a deviation factor. So, at the center of the relrod, S is equal to 0. If the Ewald sphere cuts at the center, it means that S is equal to 0.

If the Ewald sphere cuts at the top, you can say S is greater than 0. If it is at the lower part, you can say S is less than 0. Okay? So, this is called the deviation factor from Bragg's condition, and this is very important in the diffraction contrast imaging of defects, fine? So, that particular S value is this particular term, okay?

Here, we can see that even though S, even though the Ewald sphere cuts the relrod exactly at the 0 position, meaning S equal to 0, your S effective is not equal to 0. Okay, so, when S is equal to 0, your S effective becomes 1 by xi g. Okay, so, if your sample is very flat, meaning there is no bending. Okay. So, it means that if your sample is bent, what will happen? If this is your incident ray, this is your crystallographic plane. At this position, if S is equal to 0, because of the bending, your S will vary while going along the sample surface due to the bent nature.

Your S changing. S changing means your evolved sphere is not cutting at the Center of the rail rod. It is going away from the center of the rail rod, okay? So, imagine that if your sample is very flat, there is no bending, and S is equal to 0. Then S effective is 1 pi psi g, and your intensity Ig becomes

pi t divided by psi g whole square dot sine square pi t divided by psi g divided by pi t divided by psi g. This is just keeping the S effective value in this equation. Correct? Now, with this, what you can do is you can cut this position. You can cut these positions. Now you will get the intensity of the diffracted beam directly equal to sine square pi t by psi g. Okay? With this, what we infer is Ig is a function of sine square pi t and also a function of thickness, okay.

So, if your total intensity is 1, which is equal to I total, then your incident intensity plus then your I0, the incident undeflected intensity, is equal to 1 minus I, okay. So, you can see that here by this equation, if your thickness is near to Psi g, then what will happen? Your intensity becomes zero.

Understand? That is why xi g is called the extension distance. What is extension distance? It is the distance from the sample surface. It is the distance from the sample surface where your intensity goes to 0, rises again, and then goes to 0.

So, this particular value is called the xi g value. By the definition of xi g, this xi g value is different for different atomic planes. Okay? So, the intensity of the diffracted beam, I g, changes along the thickness while passing through it, okay? And when your thickness is xi g, then your intensity becomes zero, okay?

So, if there is a two-beam condition—what is the two-beam condition? Only the transmitted spot and the diffracted spot. This means your Ewald sphere is cutting only the transmitted and the diffracted spot. So, you are tilting your sample in such a way that your Ewald sphere is only cutting the transmitted and diffracted spot. So, even if one set of planes is diffracting, if your sample thickness is xi g, then there will be no diffraction spot. You will not see any spot, any diffraction spot.

You will always see only the transmitted spot. Why? Because your thickness is near to the xi g, which is the extinction distance, okay? Now, I will rewrite this equation equal to sine squared Pi t by xi g. Now, as we know, any wave equation—the total wave equation—is given by Here, phi 0 is the amplitude, and omega is the phase difference.

Omega is the phase. Fine? Based on this equation, your total wave function can be written as phi 0, which is for the incident beam, e raised to the power 2 pi i x0 dot r, plus phi g1 2 pi i x g1 dot r. This is for the spot g1. Like this, you can write an equation for each diffraction spot.

If you are assuming a two-beam condition, we should consider only these two equations, assuming that it is in a two-beam condition. So, in your two-beam condition, your equation will include only the incident beam and the diffracted beam. Okay. Now, how much to quantify—how much magnitude of the electron beam travels across the sample thickness? So, to calculate that, what you have to do is—there are two important terms, okay, which are called and d50 by g.

And these equations are called Darwin-Howey-Wellen equations, okay? And this equation is represented by d phi g by del z. So, it is the intensity of the diffracted beam changing with respect to your Z, with respect to your, if your sample thickness with respect to your Z changing is given by, if you differentiate this particular equation, you will have value of phi by psi g, phi naught 2 pi i e to the power 2 pi i x0 minus xd dot r plus phi i by psi g. Similarly, for the transmitted d phi 0 by del z is equal to phi i by psi g.

psi g phi 0 plus pi i divided by psi g phi g 2 e raised to the power 2 pi i xt minus x naught dot r. So, here the x naught minus xt is the change in vector, change in vector of phi g when the beam when the beam scatters into phi 0 okay. So for example if you have a sample thickness you have intensity I0 and this is your transmitted this is your diffracted IG where it is possible that the IG can diffract again. and again it can contribute to the incident beam, okay? So, it is the change in vector of 5G beam scatters again back to the 5, 0.

Similarly, XD minus X naught is the change in vector 5, 0. which scatters back to the 5G beam again. Okay? And these equations are the solution, the I, the intensity which we have given this equation is the solution to this equation. So, based on the definitions, what we tell is

The xi g is a constant, which is defined as the critical distance in a perfect crystal at which the transmitted intensity falls to 0 again before increasing, again before increasing. So, this is called the extension distance, okay? And in this term, the e raised to the power 2 pi i s xd minus dot r is the, this particular term is called the phase factor from the scattering process. So, the first term in each equation is the scattering from the undeflected beam, and the second term is the scattering, the second term.

First term, this is the second term; this is called the first term and the second term. is represented by the scattering from the diffracted beam. So, here the amplitude of each wave, the amplitude of each wave changes as the wave propagates, propagates through the crystal. due to the contribution from the others, okay. So, based on the equation, what we see is okay.

So here, these are the two equations, and I have shown the The solution of these two equations is related to phi g, i g intensity is a function of sine square and also the thickness, and you can see that if there is your sample thickness and if an incident beam is falling, then if you divide these into several volumes of dz then each volume will have a diffracted intensity and the transmitted intensity. And while traveling, this beam, it might be possible that the diffracted intensity diffracts again and contributes to the phi 0, and again it can diffract to contribute to the phi g. Okay?

And there is a—this is why the intensity of I0 and Ig changes along the thickness. Okay? And the distance at which the intensity of I0 changes to 0 means that it is—that is, I0 changes to 0 or the diffracted intensity changes to 0—is called xi g. And at half of xi g, you can see that the intensity of the transmitted beam becomes 0. So, if your thickness is equivalent to this particular value xi g,

then you will not see any diffraction spot on the back focal plane. So, based on this particular equation of the intensity, your sample cannot be ideally a flat sample. Okay? Your sample might possibly have a thickness that changes as you go away from the edge. Correct? So, consider this schematic where it is shown that the sample edge thickness varies as you go into the sample.

Okay? So, this is your Z. Now, as I told you, at xi g value, you will see Ig is almost equal to 0. So, if the thickness of the sample—so, your thickness is changing—and the locations where the thickness is equal to xi g, what you see is in the diffracted intensity, you will see the intensity of Ig becomes 0, and suddenly it will increase again and become bright.

So, you will see continuous fringes of dark and bright as your thickness changes. So, on the right side, I am showing an image of a TEM image where you can see thickness fringes. These are called thickness fringes, okay? And these thickness fringes—these thickness fringes— The distance between the two bright intensities or the difference between the two dark intensities is directly related to which size you are considering. during your diffraction.

So, if you orient your crystal in such a way that your g vector changes, it is possible that the thickness—the distance between the two bright fringes—will also change because your Psi g value will change. So, it might be possible that you can get a higher number of thickness fringes. You can see that the Psi g value for aluminum for different crystals is written here.

These are in the Armstrong's. You can see for the 111 the size is 556. So, if you are in the orientation of 111 the G condition, then you can see that the large number of thickness fringes due to the change in the thickness. Similarly, the reverse will happen when you are doing the bright field imaging. So, wherever the psi g becomes 0, your I0 becomes maximum.

Correct? This is the reverse of the, so you will see the inverse contrast in the dark field and the bright field. Now, in the term Ig, in the term of In Ig, we see another term which is called Ig, which equals $\sin^2$. There is another term which has a $\sin^2(\pi t\ S\_effective)$ divided by $(\pi t\ S\_effective)^2$. into πT^2 by ψg, S_effective.

So, this S_effective is also given by $S = \sqrt{(S + 1/\xi g)}$. whole square. Now, as your sample, we assume that the sample is flat with no bending or local bending. So, if your sample is bent, imagine the sample—this is your sample—and your thickness is constant. Your thickness is constant, but as your incident beam falls, What will happen is that even though your Bragg planes are not in the Bragg condition at a certain value,

it can be Bragg-diffracted, or it might be possible that the S will vary as your bending angle increases. Correct? If it is Bragg-diffracted at S = 0, it might be possible that those same planes at a certain location again, you will see that the intensity, the S changes. As S changes, it will have a direct effect on the intensity Ig.

So, you will see the bend contours in your samples. So, if there is an appearance of bend contours in your sample during imaging, whether it is in the bright field or the dark field, You will see that the bend contours look like this, okay? And the sample is bent, due to which these bend contours appear. This is due to the same thickness but S varies. So, these two types of contrast you will always get in the TEM foils, either it is bend contours or it is thickness fringes.

So, with this, I will end this class now. We have gone through briefly about the bend contours, about the thickness fringes, and the intensity. In the next class, I will go through how these conditions can be used for defect analysis. So, I will just briefly touch on those topics. So, you will see that actually you can use this weak beam or S, the S positive or S negative, you can use this to get the diffraction contrast from the dislocations which are present in the crystal lattice. Thank you.