

**Advanced Material Characterization by Atom Probe Tomography and
Electron Microscopy
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Week-06
Lecture-18**

So, welcome to this class. So, in the last class, we briefly reviewed the reconstruction and how to proceed with it from the obtained data. Now, in today's class, I will show you some calibration methods, which are used to calibrate your reconstruction based on the APT data. Okay, so the accuracy of the final reconstruction is determined by the precise measurement of ξ , the image compression factor, the field factor, your efficiency, and also the radius of curvature.

These are the four factors that are very important and should be precisely measured to accurately determine your final reconstruction. Okay. So, calibration can be performed using FIM. So, in atom probe tomography, you can use the counter electrode and the detector. detector.

You can actually use the imaging gas in the same instrument and perform field ionization microscopy. Fine? So, at the start of the experiment, you can determine these values. Okay?

So, FIM can be used to determine the reconstruction parameters, which are these reconstruction parameters. As I told you, these depend upon the spacing and shape, electrostatic environment, then radius of curvature, shank angle, field factor, and image compression factor. So, all these parameters can be determined by using FIM. So, we will go step by step with this, just briefly. It might be a revision of what we discussed in the last couple of classes.

Okay, so in FIM, you can see that a typical FIM image has concentric rings. Okay? And these rings correspond to certain crystallographic poles. And these concentric rings are nothing but atomic terraces. Okay?

If you identify these crystallographic poles—pole 1, pole 2—so crystallographically, they have a certain angle between the two planes, the crystallographic angle, which is a constant that can be measured by the cosine theta, correct? Now, actually, in the experiment, you can also measure this theta crystallographic or theta observed. Then you will get the image compression factor as an estimate: theta crystallographic divided by theta observed. Okay? So, this way, we can identify the ξ_i , or the image compression factor.

Okay. So, how the—so this is the first image compression factor. Now, the second is the radius of curvature. Okay. So, in the FIM, you can see these are all the rings, which have a certain diameter.

Okay. So, the ring pattern is used to derive the radius of curvature, which is determined by the size of the terrace. Okay. This is actually inversely proportional to the radius, local curvature, and radius of curvature. Okay.

So, as in one of the previous classes, we have derived this equation: $R_0(1 - \cos\theta)$, which can be equal to $n \cdot d_{hkl}$. If you identify that particular pole (hkl), you can actually calculate R_0 with this equation. Okay, so this is the radius of curvature. Another thing is the shank angle—the third is the shank angle. How to determine the shank angle? If the radius has been determined, then, as you know, due to the geometry, So, this is the particular R_1 , and if this is R_2 , the radius has evolved from R_1 to R_2 . If you—we can—this is time 1, this is time 2, okay? So, if the number of atomic layers between these two are measured, which is equivalent to a

The spacing of that particular pole, then your shank angle can be estimated by the formula: $\sin\alpha = \frac{R_2 - R_1}{R_2 + R_1 + nd_{hkl}}$. Okay? So, this is how we can measure alpha, which is the shank angle, by the evolution of the radius at two different instances. The fourth one is the field factor. So, if the radius is measured—this I am talking about in the FIM, correct? Remember, this I am talking about in FIM.

So, if the radius is measured, if you know the radius and assume that the electric field at the apex is F , then your F equals V divided by KfR . So, the field is an assumption. What is the assumption? It is assumed that in FIM—remember, I am talking about FIM—where the field required to evaporate, the field required to evaporate atoms from the surface is known to vary due to the presence of imaging gas in that particular field.

Okay, so this is the assumption we made. So, whatever field is required to field-evaporate the atoms from the tip surface is known to vary, and this is due to the presence of the imaging gas on the tip surface. Okay, so that is why, due to the presence of this imaging gas, the precise determination of the field is not straightforward. So, the determination of Kf by FIM is a little bit difficult. Okay, but in atom probe, this Kf value can be determined because you are field-ionizing, you are field-evaporating the actual ions, which are the actual atoms present on the tip surface.

Okay, so the FIM can be used for these four parameters. Now, by using FDM, this is called field desorption microscopy. This is nothing but atom-probe tomography. An estimation can be done for these parameters also. Okay? So, the desorption image, which is formed by the arrival positions, also reveals the crystallographic information used in the atom-probe, which is similar to the FIM.

So, they also have a desorption image. They also have a desorption image where there will be a change in the density or appearance of rings, concentric rings. And these rings correspond to the ions which are field-evaporated from the tip surface. Okay? So, This is field desorption microscopy.

Okay. So, remember, in FIM, you are actually creating an image from an imaging gas. But in FDM, your atoms are ionized and get desorbed directly from the surface of the specimen. Okay. So, these can also have concentric rings, an appearance of ring structure around the main poles.

So, similar to film you can actually also measure the χ value can this is related to the theta crystallography by theta observed. You can get the R_0 can be determined by the ring structure depending upon the terraces correct. So, here the R_0 so in that in that equation you have a θ_n which is the angle between the center and the edge of the end terrace.

around the pole. So, remember the equation in the previous section, we wrote $r_0 = \frac{1}{1 - \cos \theta_n}$ equal to $n d h k$.

This θ_n corresponds to the angle between the center and the edge of the n th terrace around the pole, okay. So, before we determine the r_0 , it is necessary to determine the ξ and also the k_f , which is the fuel factor, okay? So, k_f , as we explained, is v equal to f_r , and we can actually during the FDM estimate the k_f or the f by the kingdom diagrams. Kingdom diagrams or plots. From these plots, actually, we can accurately determine the electric field which is present near the tip surface based on

the species or the specimen. Correct? With this, we can determine the actual value of the k_f , okay? Now So, in FDM also, or we can say atom probe tomography also, you can actually use the crystallographic features within the specimen to determine the k_f . For example, if you have an alloy, and if you know that you have two kinds of precipitates which are crystallographically on certain planes, and you know that the angle between these two precipitates is θ , which doesn't change due to the crystallographic nature of those precipitates.

So, you know that the θ is constant between precipitate 1 and precipitate 2. You can actually use this feature, and the θ to actually calibrate the KF value for that optimum value. In the last class, I showed you an example of how the spherical morphology of the precipitates can be used to estimate the K value, field of value. Similarly, we can use the angle between any of the crystallographic plates or the crystallographic precipitates or phases, which is a constant and does not change, to

calibrate the KF value. Okay, so once these planes are identified, okay, and this spacing between these planes can also be calibrated in the depth dimension, okay. So, this is one way of doing the calibration. Another way is if you have a pole—in the last class, I showed you atomic planes, which, in the reconstruction, okay? And these atomic planes have a certain pole, either it is a 100 or 110 crystallographic axis. And if you know the material—for example, if it is aluminium—we know that the d -spacing of 100 of aluminium is known, okay?

So, it might be around 4.03 Ångstroms, correct? And this particular value, as you know, 4 Ångstroms, this value can be used as a constant value, and you can calibrate your KF. You can calibrate your KF so that your reconstruction is done in such a way that at different values of KF, you can get a value which shows that your planes, your d-spacing, is near to the 4 Ångstroms or equal to the 4 Ångstroms.

So, this is related to, as you see in the formula, the $d_z k_f^2$ iron square divided by η^2 square. Okay. So, you can see that the depth of penetration is actually proportional to the k_f^2 field E_f and the E_f . Here, the E_f is constant, okay, and η is known from FIM or FDM, and it means that here, if this d_z is able to resolve that this particular d_{hkl} , you can see that the d_{hkl} measured is proportional to the k_f^2 .

So, with this, we can determine the K_f value accurately based on the reconstruction. So, we can fine-tune the value of K_f such that your atomic plane distance comes to a value equal to the d-spacing of the 100 plane of aluminium. With this, we can also use multiple poles for calibration. So that you can accurately determine the K_f value. Okay.

So, usually for high solute content, so if there is an alloy, this is usually very easy for pure elements or very dilute alloys. But if you have a very high solute content alloy or multi-component alloys, Okay.

In that, for getting poles on the FDM field desorption images, it is very difficult. So, in those kinds of alloys, we use a technique where you can see, where we can use additional microscopic techniques such as TEM. To identify first the precipitate nature or to identify the crystallographic nature of those precipitates. And those features can be reused in a reconstruction where we can actually calibrate the K_f value based on the measured value in the reconstruction. Okay. So, several microstructural features which are crystallographically oriented in the alloy or the interfaces, those and the size.

So, we can use the orientation. We can use the size of those precipitates. If you know the size of the precipitates from TEM, then we can actually calibrate the reconstruction by changing the K_f value based on the known size of the precipitates. Thickness. Thickness of a thin film.

Okay. So, these features can be used to calibrate the KF value. Okay. So, So, we just go through the use of FIM and FDM for better calibration to get an optimum reconstruction which mimics the actual tip surface.

So, I will also introduce the detector efficiency, okay, which is a very important factor. Detector efficiency is nothing but related to the number of ions detected by the number of ions which get ionized or field-evaporated. So, not all the atoms get detected at the detector because some of them will be lost to the environment. They will strike the solid areas of the MCP plates and bounce back. Okay, so they will not enter those glass tubes.

So, there will be a certain detector efficiency, and this is based on—this can also be estimated by the crystallographic direction, okay. This is similar to how to fine-tune the KF values. So, you know that if you have an interplanar spacing for any particular material, okay. So, we have to estimate the detector efficiency, given that if the interplanar spacing can be fixed.

What we have discussed presently, if we know that $dhkl$ can be fixed, then the ratio KF^2 , FE^2 divided by efficiency and ξ^2 , okay, is fixed by the ratio between these two. numerator and the denominator. So, it is possible to tune the efficiency and adjust the KF value such that your $dhkl$ remains the same. Okay? So, this is where we can actually get the efficiency.

So, you know that $dhkl$, measured $dhkl$ is proportional to Kf^2 . So, this is based on the formula of dz . Okay. Kf^2 field squared divided by ξ^2 . Okay.

Based on this, you can estimate the detector's detection efficiency. Okay. So, another thing is if you have any crystallographic feature, if you have atomic planes, and if you observe the field desorption map of that particular pole, you will see these low-mass-density regions or the concentric rings, correct?

If you change the Kf value much lower or higher, then what will happen? There will be a stretching of these planes. in the reconstruction. So, it can induce stretching and also shrinkage in the lateral dimension of the volume, whatever you are analyzing. Okay? So, if you have a pole here,

it might be possible that it will be stretched or shrunk based on the Kf norm accurate value of the Kf value. So, this variation in the lateral dimension of the area accompanies the variation in the measured angle between the crystallographic axes. So, this is your x and z. If you see perpendicular to the reconstruction, you will have a you will have certain poles at different locations of the detector, correct? Now, if you apply Kf values, different values from low to high, then it is possible that at certain Kf values, the particular plane in this area can be stretched.

So, the distance between these two poles can change. And this distance between the two poles is nothing but the crystallographic angle between the poles. Correct? So, that crystallographic angle cannot change. So, it is measured by the cos theta.

So, based on this cos theta value, you can calibrate your Kf value. Once the Kf value is calibrated, you can use it to make the DHKL the same. You can actually calculate the detector efficiency. So, this is the way we can estimate the detection efficiency of an atom probe. So, here for example, in old atom probes where the MCP plates are not so advanced, they have a detection efficiency of about 30 to 50%.

Correct? And now, in recent times, it has increased to 57% with the reflectron, and for a straight path, it can go up to 72% detection efficiency. Okay? So, with this, what we get is the importance of calibration. What are the important aspects? What is the importance of calibration?

You can see that, through experiments, the xi value, ICF, and KF values are all variables. These all change with the experimental values for form. Okay. So, that is why calibration is very important to get a reconstruction that exactly mimics the tip surface or the tip volume you are analyzing. This way, you will minimize distortions during the reconstruction so that your analysis will be optimal.

Okay. So, with this, I will end this class, and in the next class, I will discuss the artifacts. Artifacts that occur during reconstruction. And these artifacts in the reconstruction are due to changes in electric field conditions near the tip surface. This is related to your tip geometry, local variations in the radius of curvature, and the presence of different kinds of precipitates or phases.

which has different evaporation field values. All these things can be related to your artifacts. So, the most common artifacts are rollover and trajectory aberrations. We will cover this in the next class. Thank you.