# Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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**Lecture-19**

So welcome to this class. Briefly, in the last class, we discussed the importance of calibration and how to evaluate the image compression factor, field factors, and how to calibrate. We have discussed several methods. In this class, we will usually go through the common artifacts. Common artifacts are induced during reconstruction. Okay, so the first common artifact usually present is what we call trajectory aberrations, trajectory aberrations.

And the local magnification effects. Okay. So, it means that because of these two—trajectory aberrations and the local magnification effects—there will be a discrepancy between the reconstructed data data and the original structure of the needle specimen, the original structure of the specimen. Okay, so this is induced by two factors: one is the imperfections, the imperfections in the reconstruction procedure, and

The other factor is the experimental effects during the experiment. Okay. These experimental effects are due to the induction of deflections in the flight of ions. Flight of ions. Okay. So, in these experimental effects, the most commonly observed is the roll-up effect, and Waugh et al. has described that these artifacts due to the roll-up effect are usually observed.

Near the edge of any terrace in FDR, okay? So if you have a middle specimen which has a series of atoms, okay? And these are the terraces: first terrace, second terrace, third terrace, fourth terrace. And usually, the roll-up happens at the edge of these terraces. What does it mean? It means the movement of an atom on the surface prior to its field evaporation and departure.

Okay, so if you zoom out, it might be possible that the atom present in this location can roll up. So, this is called roll-up, okay? And the atom will roll along the edge of the terrace, just above the position of that particular atom. Okay, so this is called the roll-up effect, and it is usually seen at the edge of the terraces. Now, another kind—we call it, because of the effect of experimental effects—this is called trajectory aberrations.

And these aberrations are usually intrinsically related to the distribution of the electric field in the vicinity of the needle specimen or the tip specimen. Okay, so this is intrinsically related to the distribution of the electric field. Okay, what happens is that it induces undesirable lateral displacements of ions during any instant of their flight. Okay, so this trajectory aberration is mostly internally connected to or related to the electric field distribution around the needle specimen and it induces a lateral displacement of the atoms or the ions during the instant of the flight.

And this is directly affected by the localized—it means directly affected by the localized variation in the electric field. Okay? And this results in, so this usually happens near the zones of high, it usually results in the zones of high and low density of ions that hit the detectors. So, it means that the low or high atomic densities are on the detector.

So, if you have a detector, you may get these zones of high and low regions as zone lines or poles. So, these are all low atomic density regions. So this is related to your distribution of the electric field in the vicinity of the mirror specimen. Usually these are located at poles or zone lines. These are the examples where you can see that there are depleted zones.

These are called depleted zones in any FDM image. This is due to the electric field in the vicinity of these zones being discontinuous. This discontinuity is due to the local geometry of the surface. So near the poles, if there is a terrace, if there is a terrace. This is a particular pole, correct?

If you look in this direction, you will induce concentric circles, and these terraces will act as a change in the local geometry. So, there will be a local change in the electric field. There will be a discontinuity in the electric field. So, the distribution of the electrostatic field around these zones tends to divert the ion trajectories.

Okay, as these zones, these terraces or the zones actually have a high density of electric field distribution. Okay, so if you have a high density of electric field distribution, then locally the atoms which are located in these regions they get ionized, and due to the curvature effect, these ionized atoms repel each other, due to which they divert the ion trajectories. Okay, so in the last class, I have given you an example in the few two or three classes before, where if there is a change in curvature on the tip surface, usually in FDM, these atoms, when ionized, their flight path will not be straight.

It will not be along the field lines. This flight path will be diverted. The ion trajectories are diverted. So you will see on the detector low-density regions just ahead of that particular high radius of curvature region. So you will have a low atomic density here and a high atomic density there.

Okay? So these are called trajectory aberrations. And these diversions of these ion trajectories Okay, these artifacts are directly related to the crystal structure. Okay, and also the condition of the experiment, or we can say that temperature and the electric field.

Okay, so the diversion of ion trajectories is related to the crystal structure and also the experimental conditions, which are related to temperature and the distribution of the electric field. Okay, it means that the shape, the shape is controlled by these factors during field evaporation. Okay, so usually these depleted zones, these depleted zones region, these depleted regions are called zone lines or poles, low-density regions. Okay, so this is related to your trajectory aberrations. This is, as I told you, these trajectory aberrations are of two kinds. So one is intrinsically related to the

atom probe tip, okay, and the electric field distribution in the vicinity of that. There is another way of trajectory aberrations, which is the from precipitates, okay. So if you are in your matrix, if you have a needle specimen, if in your matrix there is a precipitate at the apex of the needle specimen, the field, if the field evaporation or the evaporation field of this precipitate is different from the matrix, then this can also give rise to trajectory aberrations. This depends upon the difference. This depends on the relative difference between the evaporation field of the precipitate and the evaporation field of the matrix. So, there will be two conditions, correct?

So, if the field required to field-evaporate the precipitate, the matrix, So, when the electric field required to field-evaporate the precipitate is lower or higher than that of the matrix. So, the relative difference will arise with the difference in the evaporation field required to field-evaporate the precipitate. Either it is lower than the matrix or higher than the matrix. If it is lower than the matrix, this is referred to as a low-field precipitate.

And if it is higher than the matrix—if the evaporation field of the precipitate is higher than the matrix—these are called high-field precipitates. Okay. So, this low evaporation, low field of precipitate, usually what will happen if you have a needle specimen, okay, and if you have a detector here, if you have a precipitate, if this precipitate has a low field or low evaporation field than the matrix, then what will happen is usually this curvature. Which gets flattened.

This gets flattened. Due to this flattening, the atoms from the matrix, from the matrix, get deflected inwards. Okay? So, this causes a flattening of the surface of the needle specimen. Okay?

So, if you zoom out, you will have a precipitate which is flattened on the needle specimen, and the atoms near the surface will get deflected inwards in the case of low field precipitates. So, this results in lower field regions that deflect the ion trajectories inwards. So, it results in lower field regions that deflect the ion trajectories inwards.

It means that at this location in front of this precipitate, there will be a high density of atoms or ions, and here there will be a low density. This is due to the flattening of the surface. Because of the low field precipitate present in the matrix. It means that if the precipitate evaporation field, the evaporation field required for the precipitate is lower than the matrix, usually the precipitates will field evaporate very easily. So, the surface, the evaporation surface, the surface gets flattened due to which the atoms in the matrix will get deflected inwards.

and you will get a very high density of ions just above that field precipitates. In the case of high field precipitates, you have a needle specimen, you have a precipitate, this is having a high field, means evaporation field required for the precipitate is more than the matrix, then what will happen if there is a detector here, what will happen is this particular precipitate it gets bulged out. Okay?

It means that the radius of curvature increases. The radius of curvature increases. It means that the electrostatic field distribution in the needle specimen, if you magnify this, then you will have a tip. You have a precipitate like this. Okay?

And if you have a detector, this particular region will have a high electric field distribution, okay? And due to this high electric field distribution, what will happen is the atoms which get ionized will reflect themselves. The ions will reflect themselves, and these ions will be deflected away from the region. So, in the detector, you will see a low density of atoms or ions on the detector, and you will see a high density away from that precipitate location okay? So, this is the difference between the low-field precipitate and high-field precipitate, and this condition can also be regarded as a trajectory aberration.

Okay, so locally it generates a very high field because the precipitate needs higher field evaporation. So, by the time the atoms get ionized from the matrix, it gets sharper and sharper. And due to this local change in the curvature, the electric field distribution becomes intense or denser, and due to which the atoms which get ionized in the matrix will be reflected. And due to that reflection, usually the trajectories will get deflected away from the surface or the sharp surface or the precipitate. Okay?

And this results in a decrease in the density of atoms which are hitting just ahead of the precipitate. Okay? So, this is a second type of aberration which is called as a trajectory aberration. So, first was the roll-up aberration. The second one is the trajectory aberrations.

And the third type of aberration is related to a, so if you need a specimen, there are certain atoms which needs high evaporation field for field evaporation. if these atoms or the species needs high evaporation field for the field evaporation, then what will happen is, then the matrix, then the matrix, then even though you are applying an electric field, as this requires higher field, these atoms are retained on the surface, on the surface, until it finally gets, gets a enough, enough electric field or the field evaporation such that these atoms can field evaporate. So, the atom stays longer time at the surface.

Okay? And this longer time stay at the surface, this is called preferential retention. Retention. And this preferential retention causes errors in the atom location in the reconstruction, due to which there will be an error in the composition, errors in the composition profiles, especially in the locations where the grain boundaries are present.

Correct? So, this is a preferential retention of atoms. If the atoms present in the needle specimen have a low evaporation field, have a low evaporation field, then it will have an opposite effect. Okay?

So, the field evaporation before the most, so if in the needle specimen, if there are protruding atoms, if there are protruding atoms which need to be field evaporated, but there is a, there are also atoms which have a low evaporation, low evaporation field, what will happen is that if there is a low evaporation field, even though these protruding atoms did not feel evaporated, these low evaporation field atoms or the species get field evaporated before these protruding atoms. Okay? And this is directly related to your chemical nature.

It means that it is directly related to the strength of bonds. between those atomic species, between the nearest neighbors. Okay? And this particular aberration, which we also call as a chromatic aberration. Okay?

This is particularly the chromatic aberration. Okay? So, we discussed mainly Three types of aberrations, which are rollover or roll-up trajectories, trajectory aberrations, which can be due to the specimen intrinsically, the type of specimen, and the electric field distribution. The other is if there is a precipitate which has a different evaporation field value than the matrix.

Another is the species, or atoms, which have different evaporation field values than the matrix. It is usually called preferential retention. Preferential retention. This occurs when the field evaporation of those solutes or species is higher than that of the matrix. Correct?

This particular effect usually affects the depth resolution of ions. Okay? Remember, as I told you, the Z-coordinate is a sequential process. Sequential process. Okay, and this sequence of process is layer by layer.

And this sequential process depends upon the penetration of the electric field on the surface. This penetration of the electric field on the surface is less than the interplanar spacing. of that particular atomic layer. Okay? So, this is a sequential process.

Now, these aberrations—chromatic aberrations or trajectory aberrations—directly affect the depth resolution of the ion. Okay? So, usually, as I told you, the reconstruction is built layer by layer during reconstruction. Okay? And if there is a difference in the evaporation field of solutes,

of solutes and the matrix or the precipitate and the matrix, what will happen is the low evaporation field species will depart the needle specimen very early. So it means that the reconstruction, if it departs early, will be detected much earlier than the other atoms or the ions from the matrix. So in the reconstruction, this will be reconstructed early. So it will directly impact the accuracy of the atomic position. Along the Z direction.

The depth. The Z direction means the depth direction. Fine? So, this is how it affects the depth resolution of this particular aberration. Chromatic aberration or trajectory aberrations will affect the Z coordinate. Okay?

There is another issue called surface migration. Okay? We will discuss surface migration. This is another type of artifact we usually get in tomographic reconstruction. So, in the image, we know that the zones or the poles display a high concentration of solutes in the vicinity of

poles and zone lines. This surface migration causes the regions or zones that display a high concentration of solutes in the poles and the zone regions. Okay, this is because these poles and zones are regions of high field gradients. Okay, so along with trajectory aberrations, surface migration can also take place. For example, in an aluminum-copper-magnesium alloy, it can happen that there will preferentially be a loss of aluminum atoms at the region of poles, which

leads to the higher concentration of copper and magnesium at the poles. Okay? And this related, the loss of aluminum atoms is related to your trajectory aberrations. Okay? So, this might lead to the composition which can be seen as a higher concentration of at the poles. Okay? And the copper and magnesium atoms which are present in that layer, these are actually less affected by the trajectory aberrations. Okay? So, this is related to a surface migration.

So, usually the high... Concentration of solutes, high concentration of solutes, usually they are seen in the poles and zones. It means that these are related to the crystallography, crystallographic features. And in steels, we see that some of the solutes get surface migrated to these poles. Okay?

And this is related to your surface migration. Okay? Similarly, in film, we see this surface migration. This is due to the very strong field gradients at the surface of the specimen. This leads to the atoms

relocating from one position to an adjacent one, usually as a thermally assisted process, okay? Due to the field gradients near these poles, it induces a difference in relative energies between the atomic sites, which facilitates the diffusion process. This is related to the surface migration. So, if the solutes have a high evaporation field—higher than the matrix—they are preferentially retained, as I told you before. These are preferentially retained.

So, they spend more time on the surface. This means they are more prone to surface migration. Remember, this is different from trajectory operations, which are related to your field distribution—the intensity of field distribution near the poles or the ions. And this surface migration is a thermally assisted process. Thermally assisted process.

And this thermally assisted process, these surface migrations actually you can minimize by lowering the temperature. By lowering the temperature much below the 50 Kelvin or near to the 20 Kelvin. But even though if it goes to the lower temperature, we cannot due to the difference in the evaporation fields, your trajectory abrasions you cannot avoid. So, these are the two important things, surface migration and trajectory aberrations which are little bit different. Surface migration is a thermally activated process and it usually the solutes, if the solutes gets field evaporated much earlier near to the poles or zones, then you will get a higher concentration of copper. Example, if you have the matrix atom, if the matrix atoms field operates at these zones much earlier then you will get higher amount of solutes at these zones ok and also due to this field gradients and you can actually this assist the diffusion process so usually the solutes which is having a higher electric field which are retained and which are stays on the surface for longer time can actually diffuse along these zone lines or the poles ok

And usually it is a thermally assisted process. And a thermally assisted process, it can be minimized by lowering the temperature. However, even though if lower temperatures due to the trajectory aberrations, there will be some inconsistency in the reconstructions. Okay. So, we discussed about in this class that there are different types of artifacts which are related to your, the first artifact which we discussed is a rollover process.

which usually happens near to the atomic terraces. The second is trajectory aberrations and this can be intrinsically related to the needle specimen and the distribution of field in the vicinity of the atom proptech and also if there is a difference in the evaporation field of precipitates and the matrix. So you will get different regions of atomic density. On the detectors, depending upon whether the precipitate evaporation field is low or higher than the matrix, the third

one we discussed is if there is a difference in the field evaporation field of atoms or species from the matrix, it might create a retention. Retention of that atomic species or it might be an early departure of those species. Okay, and the fourth thing we called it is surface migration, which also occurs near the poles or the zone lines, okay, due to the—and this is a thermally activated process. Which can be minimized or removed when the temperature goes to low temperatures. However, at low temperatures, you cannot avoid these trajectory aberrations.

So, with this, I will end this class now, and I hope you must have got an idea of how the trajectory, how these artifacts can come up, which are—some of them are experimentally produced, and some of them are from the reconstruction. So during reconstruction, what will be the artifact that we will discuss in later classes, but experimentally, these are the aberrations which we usually see due to the local electric field distribution that creates the deflection of field lines, due to which the solutes or the matrix atoms get deflected and create low-density regions and high-density regions.

So, with this, I will end this class, and we will meet in the next class. Thank you.