Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy
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Lecture-15

Welcome to today's class. Briefly, if you recall from the last class, we discussed the parameters that must be considered during the analysis of any specimen via atom probe. So, now after going through all the parameters during the experiment, today we will talk about the reconstructions, tomographic reconstructions. Okay?

Tomographic reconstructions mean that you have to—usually, it is related to the mapping of atoms in three dimensions, okay? So, this is to reveal the structural arrangement, composition, and chemistry of different atoms inside a material. Okay, so tomographic reconstruction means the mapping of atoms in three dimensions, and it is related to revealing the structural arrangement of atoms in a material. Okay, so the first topic we will discuss is the projection of ions. Okay, so

The atoms that get ionized are dissolved from the surface, and these ions accelerate due to the surrounding electric field toward the detector. If you have a neutral specimen, it will move toward the detector. These ions will travel along the field lines. So, for each ion, the calculation of the flight path is very complex. This is due to the electric field calculation.

Distribution, electric field distribution around your specimen, okay, around the specimen. So, usually this electric field distribution is in three dimensions, okay, and it is very complex in the vicinity of the specimen surface. So, this electric field distribution can be understood by two aspects. One is near field. And one is far field.

Near field relates to the sub-micron distance, sub-micron distance near the tip surface. And this far field is related to up to the meter scale. Far away from the tip surface, okay?

The distribution of electric field in near field and far field, okay? Both are different, okay?

Near field, as I told you, it is a sub-micron level. So, it is usually when an ion, when an atom gets ionized, okay? And it gets accelerated, then the local neighborhood, local atomic neighborhood, will affect the early stage of the flight path. Will affect the early stage of the flight path.

Okay. This will lead to trajectory aberrations. Okay. We will come to these aberrations. We will discuss these aberrations during the reconstruction.

Fine. So, this will lead to trajectory aberrations in the near field. Now, the ion path is directly related to the electrostatic environment. Okay, so the presence of a counter electrode, the vacuum chamber, and the detector. So, you have a counter electrode and a detector.

So, the ion path is directly related to the presence of these three important features: the vacuum chamber, the counter electrode, and the detector. Okay, and to compute the path of this particular ion, which is accelerating along the field lines correctly, that can be done by analytical solution or we can also use finite element models. Finite element simulations. Okay, so we will not—this course is not to detail how to simulate these field lines, but just to introduce what the results are from these analytical and finite element simulations. Okay, so the second aspect is the estimation of

Estimation of electric field. Okay, the first aspect was projection of ions; the second aspect is the estimation of electric field. Okay, so usually the assumption which is made is the shape—the shape of the needle, the tip, or the apex (you can call it an apex). is spherical, and the surface of this particular needle specimen is smooth. Okay. This leads to the basic equation, which is F equals the field, written as W divided by KF and R. Okay.

This is the voltage applied. This is your radius of curvature, and this is your field factor. Okay. Field reduction factor. As explained before, this field factor accounts for the shank

of the specimen, the shank angle of the specimen, and accounts for the shank of the specimen.

Okay. And relative to a sphere, this value actually reduces the field with respect to the sphere. Okay. This is called a field reduction factor, and this particular equation can be used to estimate or approximate the amplitude of the electric field on the surface of the specimen. Okay, so here, the KF depends upon the specimen geometry.

Depends upon the electrostatic environment, and its value usually ranges from 3 to 8. Okay? So, as the electric field—as we introduce the electric field—we call it a near field or far field. Okay? So, here at the near field, the F

the electric field actually critically depends upon the local curvature—the local curvature of the specimen, okay? And due to this local curvature or the change in local curvature during field evaporation, the locations of high electric field—high electric field locations of the high electric field relate to the development of smaller curvature. Okay? Development of smaller curvatures.

Okay? This is due to, first, protruding atoms. The second is kink sites. The third is atomic terraces, and the fourth is precipitates. Okay?

These four things we described induce roughness at the atomic scale on the tip surface. Okay? And due to which due to which the local shape, the local shape of the apex is a function or is an intersection of the equilibrium shape and the crystalline structure. Okay?

So, this is the local shape of the apex, critically depends upon equilibrium shape and the crystalline nature and these protruding atoms, king sites, atomic terraces and precipitates can induce change in the local curvature due to which there will be change in the field. That is why the roughness, it involves the creation of roughness at the atomic scale in the near field, near to the tip surface. Okay?

So, during experiment, during the field evaporation, the local curvature of end forms changes so that the local electric field enables field evaporation. Okay? So, this is the reason where the specimen surface develops facets Okay? Usually these facets are present around the poles.

Okay? To accommodate the equilibrium shape. Okay? Okay? And because of this, the shape becomes complex due to this reason.

Complex, especially for the alloys, thin films, or multilayers. This is due to the presence of atoms with different evaporation fields. Okay. So, the shape becomes complex when you field-evaporate an alloy, thin film, or multilayer due to the presence of atoms with different evaporation fields. Okay.

The third thing we can describe is the field distribution. Field distribution. So, ions, as I told you, ion trajectories are directly linked to the local distribution of the field. Okay. So, ion trajectories are directly linked to the local distribution of the field.

So, it also creates artifacts or what we call local magnification effects. This will be discussed in detail during the reconstruction. Okay. So, this arises from the local curvatures. Correct.

So, up to now, we have discussed the near-field region. In the far-field region, which is away from the tip surface, analytical solutions have been proposed. Okay. Okay. And these analytical solutions use the concept of a parabola or hyperbola.

Okay. It is in the near. So, this particular analytical solution uses the parabola or hyperbola to describe the spatial distribution of the potentials. Distribution of potential. Okay?

That is used to calculate the local distribution of the field. Distribution of field. So, here it is assumed that the potential, here the potential of the tip surface is at each location of the tip surface is equal. Okay?

But as I told you, the experimental shape, the experimental shape of a specimen, so with this technique, the analytical solution with the parabola, usually with this technique, actually the experimental shape of the precipitate, of the needle specimen is poorly fitted. So, it means that this particular model, analytical model which is used for far field, it cannot independently set the specimen radius, shank angle and also the position of the detector. This usually reduces the accuracy of of modeling the field lines.

So, this is in relation to the far field. We will come back to the near field again. So, in the near field, the near field electric distribution is more complex and it is mostly affected by the as I told you the protruding single atoms protruding single atoms lying on the specimen and other protrusions other protrusions due to the difference between the evaporation field between the phases or species and also as I told you presence of

Ledges, terraces in the near field. So, in the near field, the electric field distribution is more affected by these important features of the middle specimen. Okay, for the near field, various analytical methods failed to failed to understand the distribution of field lines. Correct?

So, this is in the near field. So, for the near field, the most appropriate methods recently developed are finite element models, elemental simulations. Okay, so, a lot of development has happened in finite element simulations where they can predict the electric field distribution in the near field. Okay, so, they have also taken care of the influence of ion trajectories and the specimen shape. Okay? This leads to the accurate calculation or estimation of the potential.

Potential and the field surrounding the specimen at atomic-scale resolution. Okay, so, here I am showing a figure which displays an FEM simulation where it shows the distribution of the field around the neural specimen surrounding the specimen. Okay, so, FEM and other alternative boundary condition methods enable the multi-scale modeling approach where they have actually modeled the electric field distribution in the near field, near the tip surface. Okay? So, complete the simulation.

So, it leads to the estimation of the complete distribution of the field from the tip surface to the detector. Okay, this leads to the full calculation of ion trajectories. From the tip surface to the detector, to the detector, okay? This leads to the ion trajectory. So, by using the estimation of these field lines, this has also contributed to

also contributed to understanding the ion trajectories from the detector to the position of that particular ion on the tip surface. Okay, so here there is a figure where you can see that at each position of the atoms, so these black colors are the atoms on the tip surface, and you can see that the electric field distribution, these regions just near to those

protruding atoms. Okay, this is how these FEM methods and alternate boundary conditions can lead to the estimation of the field lines near the tip surface and also the far field from the tip surface.

Okay, so now in the next thing, we will just discuss the ion trajectories. Okay, so for ion trajectory, for reconstruction, the thing is you have to identify the ion trajectory from the tip surface towards the detector so that you can mimic backward so that from which location of the tip this ion or the atom is accelerated. Fine, so you need to So, for this first condition, you need to determine the electric field distribution. Okay, to model the ion trajectories, to model ion trajectories.

So, several authors have done this modeling, okay. So, the trajectory of the ion created, the trajectory of the ion created at the specimen surface—wherever these atoms get ionized—several works have demonstrated that the the trajectory of the ion is not dependent on its mass and voltage. They depend upon the distribution of electric field lines. Okay, assuming that the initial velocity of these polarized ions or polarized atoms is zero.

Okay, so once these ions get emitted from the surface, okay, these ions strictly follow these field lines, okay, and this is valid also for the far field. However, in near-field distribution, it is more complex. Okay, near field means a few tens of nanometers from the tip surface, okay, as an example, for example, ions very close to the surface leave the surface almost normal. So if you have a needle specimen, if you zoom out this region, assume that this is a hemisphere at the tip surface, and this is at the center, okay. So the ions which are very close to the surface, very close to the surface,

the atoms which are very close to the surface, they actually leave the surface almost normal because the initial trajectories are very close to the radial. Correct? These are the radial. So, at the very near to the surface, these ions close to the surface leave the surface almost normal to the local surface. And as these trajectories, these atoms...

These are, and there is a detector. These ions, or these field ions, are actually compressed as they go towards the detector. Like this. Okay. Okay.

So, as these trajectories are progressively compressed after a distance which is a few times the radius. A few times the radius. Okay. It means that if you see, I will draw this diagram again. Okay, if you assume this is a sphere, okay, and this is your center, correct?

If there is an atom here which gets ionized and field evaporated, if it gets field evaporated, you have a detector here. Okay? So, if it gets field evaporated, then at the initial point, it goes radially perpendicular to the field surface. As it goes, it gets compressed towards the axis.

Okay? And it gets detected here. Okay? So, this distance is nearly a few nanometers. Or a few times the radius, fine?

So, this is the axis. It means that we can consider the atom which is ionized here. If you see that trajectories are straight, they originate from a point that is not the center of the hemispherical cap, okay? So, if you If this is the point where it got detected, then if you make a projection of this particular needle,

you have the projection point which is not at the center of the hemisphere, but it is way behind that particular point along the axis, along the apex axis. Okay? So, this is like the specimen axis, along the specimen axis. Okay? So, this is your original trajectory point.

Correct. So, this has led to the proposal of a simple projection law that was employed. Okay. It means that the ions follow a straight path. But the trajectory of that particular ion, if you extrapolate, does not match with the actual radius of the hemisphere. It goes well, well, well away from that particular point along the specimen axis. Okay.

So, this is the basis for the proposition of the point projection model. Point projection model. Okay. So, I hope in this diagram it will be explained. Okay. So, here you can see that there is a schematic where this is your needle specimen. Assuming this is a hemisphere, okay, and the radius of curvature of this particular radius of curvature is R. Fine. It is at the center.

Now, the atom which is present at the surface, at the initial near field, goes along the perpendicular, along the field lines, perpendicular to the surface. As it moves towards the detector, it gets compressed towards the specimen axis. Okay? So, if you draw a straight

line, the position it will hit on the detector, at position D, okay, X. Okay, so assume that this is hit at a position XD and YD, and if you draw a straight line,

Connecting to that particular ion which was located at the tip surface, usually if you project it, you will see that it is not exactly at the position of the center of that hemisphere. Okay? It goes beyond that certain distance along the specimen axis. Okay? So, in HV pulsing, if you see in HV pulsing,

The ion's flight, the ions' flight, experiences the electric field that varies with time in HV pulse. Correct? So, this will induce an impact of the pulsed field on the trajectory. Okay, so Gault et al. has shown that, as modeled or computed, the difference between the position of arrival of ions with the DC voltage and the pulsed field evaporation. Okay, so on application of DC voltage, the ions hitting the detector

and the ions which are hitting the detector with the pulsed field evaporation, he has computed the difference in the position uncertainty. Okay, so this position uncertainty changes as the pulse field is applied. Okay. So, the position accuracy he calculated is around 1 to 3 percent during HV pulsing. Okay.

So, this is the this is the uncertainty in the position of the ion detected when a pulsing DC voltage is applied and when a DC plus pulse field evaporation is happening during the HV pulsing. This is during the HV pulsing. So, there will always be an inaccuracy of 1 to 3 percent of that particular ion detected on the detector. So, coming back to the point projection model, it was assumed that the

ions fly in a straight line from the specimen surface to the detector. This was the first assumption during the point projection model. This is the point projection model. The second is the line originates from a single point along the specimen axis. it can be projected to a point P, which is way away from the position O, which is the center of the hemisphere.

Correct? So, this is a point projection model. Now, from this figure, you can see that the P is located along the axis. This is your specimen axis of the specimen at a distance of xi r. Okay? So, this is the factor which is to be

Multiplied to the r, okay? xi r from the apex, okay? And actually, this xi, this xi actually represents the compression of a few lines, and this is called the image compression factor. Okay, and this xi R can be equal to m plus 1 R, where you can put m R as the distance between O and P. So, it will be m R plus R. Correct? So, the distance between O and P can be written as m R. So, this particular Xi is directly related to the distribution of the field, the distribution of the field. And that is why it depends upon the electrostatic, electrostatic environment, and the most critical factor is the shape.

It is the shape of the specimen. Okay? This particular xi value also affects the magnification. Assume that this distance is D. Okay? And as you know here, if you assume that this is a distance D and the

The atom which is sitting on the tip surface has a distance small d from the tip axis. From the tip axis. This is the specimen axis. From the specimen apex. Then the M projection.

The M projection. can be defined as d by t, which is if you see the geometry, you can put it as l by l by xi r. Okay, l is the distance between the tip and the detector, okay. So, if you assume that L, usually it is around—L is usually around in the instruments 90 to 500 nanometers—and R is usually less than 200 nanometers. So, your xi value usually is between 1 and 2. One is for the radial projection, and so in radial projection, r equals psi r, correct?

So, it means that it assumes the projection of that particular atom exactly matches the center of the hemisphere, and if it is stereographic projection, then the highest value is 2, which is related to the stereographic projection. So, now we will come to the reconstruction. So, we understood the distribution of electric field—near field and far field is different—and there will always be a compression of these field lines towards the specimen axis, and that can be

That can be compensated by the factor which is called xi. Correct? So, reconstruction. So, what is reconstruction? It is nothing but built atom by atom by the reverse projection.

Okay? So, the detected positions—the detected positions on the detector, that XD and YD on the surface—okay? And these detected ions, the positions are again reversely

projected, are reversely projected to reconstruct the neural specimen. Okay? So, in this reconstruction,

There are two aspects which are related to the—the first is lateral positions, and one is the important depth coordinate. Okay. So, there were several models which were proposed for this reverse projection model. Method to actually reconstruct the needle specimen. So, the first model was proposed by Boesel and Professor Plavitt, and this was again used by Baas et al.

So, they proposed These two methods, they combinedly used to describe a point reverse projection method. And this method is the basis for most of the software. Most of the software packages that are used to reconstruct the atom probe data. So,

What I will do today is end this class now, and in the next class, we will deal in detail with the point reverse point projection model. and how to reconstruct it, how reconstruction can be done by this method, and we will also discuss the important parameters during reconstruction to actually make the reconstruction as equal to that of the real needle specimen. Okay, so we will meet in the next class. Thank you.