

**Advanced Material Characterization by Atom Probe Tomography and
Electron Microscopy
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Week-07
Lecture-20**

So, welcome to this class. Last class, we saw how the artifacts come up during the field evaporation due to the deflection of field lines. Okay, and it is directly related to the tip surface and the distribution of the electric field. Especially, these are crystallographically dependent. In this class, we will briefly discuss the resolution.

Spatial resolution, okay? So, for the reconstruction or in the atom probe or the FDM, the spatial resolution is usually highly anisotropic. It's highly anisotropic. Okay. So, in the Z direction, why is it highly anisotropic? So, we will discuss this. If you have a needle specimen, okay, and as I told you, the field evaporation takes place when these atoms get polarized and ionized and they move along the field lines.

So, the atoms are field-evaporated layer by layer. So, it is a sequential process. A sequential layer-by-layer process. Okay? So, this sequential process directly gives us the depth resolution in the Z direction.

Okay? In the Z direction, depth resolution. Okay, so because of the sequential layer-by-layer process, and this is due to the electric field penetration, which is much less than the spacing between the two layers. Okay? So this creates the depth resolution, and it is much higher than the spatial resolution. Why is it higher than the spatial resolution?

Because the spatial resolution is affected by trajectory aberrations that we discussed in the last class—trajectory aberrations and local magnification effects due to the curvature. Due to the curvature. Okay. So, spatial resolution is mostly affected by this, and the depth resolution is much higher than the spatial resolution. So now we will discuss the depth resolution.

Okay. The first thing which the depth resolution depends on is the interplanar spacing. Interplanar spacing. Okay. Of the atomic planes, okay? Meaning the interplanar spacing of atomic planes is directly related to the size of

atomic terraces okay so you have a middle specimen you have atoms you have these atomic terraces And if you look in this particular direction, on the FDM or the field desorption map, these atomic terraces will be represented by concentric circles. And the distance between these rings is actually directly related to the interplanar spacing or the D-spacing. So, we have a low-index crystallographic plane. We have a high-index crystallographic plane.

Crystallographic plane. Low-index crystallographic planes have a distance farther apart, meaning they have a large distance between the terraces. Large distance. But in high-index planes, the distance between the terraces, the distance between the planes, is very small. Okay?

So, That is why the interplanar spacing will be a factor in determining the depth resolution. So, for low-index planes, as these are large terraces, you can actually map these planes very easily compared to a high-index zone where the atomic spacing is very small. Okay? So, your depth resolution directly depends upon the interplanar spacing of the atomic planes.

The second factor is temperature. Okay? So, as you know, field evaporation is a thermally activated process. Thermally activated process. So, the probability of field evaporation at that particular instant depends upon the

temperature, okay? This means that the accuracy—the accuracy of detection of sequence—depends upon the temperature, okay? Now, Due to this, the temperature effect also affects the size of the atomic terrace around the pole. That is why this has a direct influence on the depth resolution. Now, we talk about the lateral resolution. Lateral resolution means in the x and y directions.

So, you have a neural specimen. So, this is the z. So, if you have a plug-in, we will have x and y directions. Okay. So, usually, if you see in film, this lateral resolution is There are

two main—in film, we have discussed—there are two main components of film where we talk about the lateral resolution.

It is related to the thermal agitation of gas atoms. It means it induces lateral velocity. So, you will have a blurred image. Another is the position uncertainty of the gas atom or the gas atom of an atom. Okay, so these are the two important things which affect the lateral resolution.

In FDM, field desorption microscopy image, this is actually limited by trajectory aberrations. This is related to the distribution of the electric field. So, at very low temperatures, your surface diffusion can be minimized. But actually, the lateral resolution is limited by thermal agitation thermal agitation of the surface atoms.

Okay? So, it means that the amplitude of thermal agitation should be much less than the distance between two atoms in a plane. Okay? So, This is related to trajectory aberrations in the field desorption microscopy image.

So, therefore, the lateral resolution strongly depends on the packing of atoms, which corresponds to the crystallographic direction. Okay, but still, if you lower the temperature, there will still be some surface migration and rolling process which affects the lateral resolution. So, it cannot be avoided fully. So, there will be some roll-up process or surface migration even at much lower temperatures. Okay, so in the case of laser pulsing, the lateral resolution

decreases as your laser energy increases. Why? Because as you increase the laser energy, you are actually increasing the thermal agitation of the surface atoms. Okay? So, there should be an optimum spatial resolution.

Okay? So, So, there should be an optimum spatial resolution that consists of a depth resolution related to the order or sequence. So, here the high resolution obtained with field evaporation occurs in a controlled manner. Okay, usually, this depth resolution can be

We will get the depth, a very good depth resolution near the poles or the zone lines. Okay? Lowering the temperature increases the influence of the electric field on the

evaporation field process. That's why this improves your overall spatial resolution. So, in this section, we have discussed how spatial resolution can be improved.

Usually, spatial resolution is divided into depth resolution and lateral resolution. Since it is a sequential process, we say that the resolution in the Z-direction is much higher than the lateral resolution. Usually, lateral resolution is affected mostly by trajectory aberrations during FDM (field desorption microscopy) and local magnification effects. Right? Now, as we finish these basics related to reconstruction—the importance of parameters—what I will do is briefly discuss

the working principle of atom probe tomography so that you will get a revision in this course. You can see that in atom probe tomography, there is a needle specimen, a DC voltage is applied, there is a local electrode, and this is an MCP plate. This is a PSD, a position-sensitive detector called a delay-line detector. Okay. This particular chamber is kept at a very high vacuum.

The temperature is sub-zero, between 25 and 100 Kelvin. Okay. You are applying a DC voltage. When you apply a DC voltage, you will see that there will be a polarization of atoms present on the surface. Okay.

Due to this polarization and the electric field on the tip surface, these atoms get ionized and are accelerated along the field lines. They travel through the local electrode and go to the MCP plates. These multi-channel plates convert the ions hitting the charged glass tubes into a number of electrons. So, it generates a number of electrons, and these electrons from the MCP plate are biased with respect to the position-sensitive detector. The electrons then hit the delay line detectors.

These are nothing but conducting wires, and as they hit the conducting wires, they create a pulse. The pulse travels along X1, Y1 and X2, Y2. The time recorded in this process can be converted to the position of the hit. Okay? So, in this process, you are getting a sequence. You are getting the X1 position, Y1 position, and the time of flight.

Fine? And the Z coordinate is measured based on the atomic volume. Atomic volume. And it is related to your sequence. Okay?

And as I told you, the position of heat the XY coordinate is around the resolution of 0.2 nanometers, which is the lateral resolution in the evaporation sequence. The Z coordinate is usually 0.04 nanometers, which has a very high depth resolution compared to the lateral resolution. The time of flight allows you to identify the chemical identity by this basic equation, where the mass-to-charge ratio equals two times the electron voltage applied, multiplied by the time of flight divided by L.

L is the distance between the tip surface and the position-sensitive detector or hole square. Fine, so This is the basic equation we use to determine the chemical identity. Okay, so we discussed the two pulsing methods. One is voltage pulsing, and the other is laser pulsing. Fine? In voltage pulsing, what you do is apply a DC voltage.

There is a base DC voltage, and in the voltage pulsing, you are actually increasing the voltage for a few nanoseconds and then reducing it. Due to this increase in voltage, you are usually polarizing the atoms on the surface, and they get ionized. So, this sequential process over time is called voltage pulsing. In laser pulsing, you are actually increasing the temperature as a pulse. So, you are creating a pulse.

Then you are increasing the temperature of the tip surface, reducing it, and increasing it again. So, these are related to a few nanoseconds of this pulse. Okay? So, laser pulsing is nothing but thermal pulsing. And voltage pulsing is nothing but HV pulsing. Okay, so in HV pulsing, as I told you, the goal is to increase the electric field at a constant temperature.

But in thermal pulsing, the temperature is increased while the electric field is kept constant. So, this is the difference between the two pulsing methods we use in the atom probe. Fine? So, we also discussed how the measurement of time is done. So, you can see that if you consider a pulse, okay?

So, once the pulse is applied, either HV or laser pulse, when and as the pulse starts, usually what will happen is, as the pulse starts, there is a detection window that opens up, and this detection window opens for some time. Okay, so, once this detection window is up, as the pulse reaches the top, there will be a detection of the particular ion on the delay

line detectors. Correct? It might be possible that during these windows, there may be a blank event, a single event, or multiple events. Okay.

And the time, the highest point of the pulse, there is a difference between the highest point of the pulse and the start of the detector window. This time difference is called t_{naught} , which has to be accounted for during the measurement of the mass-to-charge ratio. Okay. So, this is how the measurement of time of flight is done, okay? In this, the mass resolution is defined by M divided by ΔM , which can be 50% or the 10% FWHM or the FWTM, okay?

And you usually get a peak. So, you get a y-axis as the number of ions and an x-axis as the mass-to-charge ratio, and you can see there is a width for each peak, and this width of the peak directly corresponds to your pulsing. It might be possible that during HV pulsing, Okay, during HV pulsing, it might be possible that at the top, if one ion gets detected, it might also be possible that ions may be detected at these lower voltage values, and due to this, it creates a spread in the detection of that particular mass-to-charge ratio ion, resulting in a broadening of these peaks.

and this broadening of the peaks are directly related to your mass resolution which is given by m by Δm okay in the laser you are actually heating the heating the sample so so the temperature when you when you each pulse When you do each pulse there will be increase in the temperature and as soon as the second pulse will come the temperature has to come down. If the temperature does not come down with the second pulse usually you will develop a thermal tail in the peak. So you will get a large peak and you will get a thermal tail.

And this thermal tail directly affects the mass resolution because the other mass to charge ratios which can be detected here can be suppressed with this thermal tails. This is related to your laser pulsing. That's why laser pulsing, higher is the energy, lower will be the resolution. So you have to use the optimum laser conditions for this factor. And this resolution as I told you for voltage pulsing it is 500 and for laser pulsing it is much higher as 1000.

HV pulsing, remember, for conducting samples, HV pulsing works out. For insulators and semiconductors, HV pulsing is very difficult or it is very challenging. As the conduction is very low, correct? But in laser pulsing, we are playing with the temperature, correct? So, usually, it depends on how the sample, how the sample thermally conducts, correct? So, usually for laser pulsing, we use thick samples so that the temperature which is kept on the tip surface can be removed very easily.

Okay, so these are the factors which influence these: pulse fraction, temperature, voltage, and geometry of the tip. So, you can see that we also discussed the energy deficits in the voltage. As I told you, the ions which are, when the voltage, during HV pulsing, when the voltage decreases, it might be possible that the ions at these, time frames may get field ionized. They may get field evaporated.

So, it will be detected, and you will get a broadening. Okay? These are called energy deficits. In the laser, the finite duration of the thermal pulse, so that is a thermal tail.

So, as I discussed, the temperature has to come down. Fine? So, to reduce these energy deficits, we can use energy compensation devices. These are called reflectrons. And for the laser, we can actually improve by the specimen geometry to make it a thicker specimen and also use proper laser conditions for the field evaporation.

Correct? So, for energy compensation devices, as you can see, this is typically a grid, a grid with a change in the electric field as you go into the grid. Okay? This is called a reflectron. So, if there is a species which has a mass-to-charge ratio and if, in the voltage, if some ions are field-evaporated at this location and some are at these locations, usually they have an energy difference. So, what it does is the ions which have higher energy can actually travel into these field lines at a farther distance while the ions which have lower energy will deflect much earlier. So, overall, both the ions will reach nearly the same position. So, what will happen?

Your mass spectrum, which is thick, will become much thinner. So, your mass resolution improves by using the reflectron. But remember, with the reflectron, you are increasing

the time of flight. So, you will lose most of the ions in the background. So, your efficiency will reduce.

Okay, so without reflectron, usually the efficiency can go up to 72% detection efficiency. But with using reflectron, it may go up to 52 to 57%. So, it gets reduced. Okay, so this is how an increase in the time of flight can actually improve the mass resolution for both the pulsing modes. Okay, this is due to the correction for the energy deficits and also the finite duration of the thermal pulse.

Okay, so you can see that in the mass spectrum, you can see how much the thickness of these peaks has reduced, and you can also see the number of counts reduced for these ions. Correct, so detection efficiency. Now, compositional measurements. Okay? So, here there is a composition. We can measure the composition as the number of atoms of that particular species divided by the total number of atoms present in the needle specimen, multiplied by 100.

Okay, so this is usually calculated as the compositional measurement, and this particular value is called a statistical error. Okay. Which is given by $C_j - 1 - C_j$ divided by the number of atoms. Remember that it depends upon the total number of atoms in that volume. Okay.

So, this is how compositional measurements will measure it. Now, just briefly from the instrument, you have an X detector, a Y detector, time of flight, and voltage of the specimen. Fine. And in the reconstruction, we will use reverse projection to get the X tip. Y tip, Z, the depth, and also the mass.

In this way, you can actually calculate and map the three-dimensional volume of the specimen tip. Okay, so on the left side, you can see these golden-colored atoms correspond to aluminum, and these red and green regions are the zirconium-rich and copper-rich precipitates. Okay. And on the right side, as I told you, the Z depth gives you atomic plane resolution. You can actually map the composition of the atomic plane.

So you can see that each plane corresponds to the 100 plane of copper. Okay. So this is called depth resolution. Briefly, I will go through it. The important factors that have to be considered.

If you have an ideal charge of a sphere, you have a field along the charge as voltage applied by the radius of curvature. But your tip shape is a truncated shape. Okay? So it is a truncated shape, and due to this, your field lines are not homogeneous at each place. Okay, so this relates to their field enhancement factor, which has to be added to get the correct path of the field around the truncated needle specimen. Okay? And these Due to this shape of the specimen, the field lines actually have to go perpendicular to the surface exactly at the local point, but they get compressed towards the axis, towards the axis.

Fine. This is called this is related to your near-field and far-field electric field distribution. And this directly relates to your specimen's local curvature effects. Fine. So there will always be ion trajectories that are compressed as per the field lines. So from the basic point projection model, actually, you can assume that the atoms, which are assumed to be radially accelerated, can be but usually, it is compressed and hits the detector at this position at a distance D from the center axis. You can draw a straight line along with this particular atom

which is on the surface and projected onto the axis to a point P , which is far from O . Okay? And the distance between the tip surface to P is related to ξR , and ξ corresponds to the image compression factor. Okay? And if you see the distance D from the tip axis to the detector hit and the small d , which is the distance from the tip surface to the projected two-dimensional plane of that particular atom on that plane, that D distance, you can, by the triangular symmetry, assume that M projection is related to D by d , And it can be equal to L by ξR . And ξ is called the image compression factor, which is between 1 and 2.

1 and 2: 1 is due to the radial, and 2 is when we consider it as a stereographic projection. Correct? So, these are the two important factors: the field factor and the image compression factor, which have to be taken care of during the reconstruction. That takes

care of the field lines, the actual field lines which are distributed near the tip surface. Okay.

So, this is an instrument which is at IIC Bangalore. So, you can see this is an atom probe instrument. And if you see the cross-section from the top view, okay, if you see the cross-section from the top view, you have a specimen transfer rod. So, this is called the specimen transfer rod. This is called the specimen transfer rod.

Okay. These are called carousels. This is the part which I am indicating. This is in this direction. If you see from the top, you will see these are the carousels.

Okay. And this is called the analysis chamber. Analysis chamber. And the chamber between the analysis chamber and the loading chamber is called a buffer chamber. Okay.

So, the sample is loaded into this particular loading chamber. Okay, and it will be passed on to the buffer chamber by using the specimen rod or the specimen transfer rod, and it gets fixed to the stage in the analysis chamber. Okay, so this is your atom probe specimen, this is your laser, this is your electrode, and on the backside, there is an MCP plate and these are your delay line detectors. Okay, so you are applying a voltage, a DC voltage, between the tip and the electrode. So, if you zoom out here, you have a needle, you have a electrode, so the ions get field-evaporated along these field lines, and on the backside, these are MCP plates and the delay for the detection.

Okay, so this is kept at a very high vacuum. And to maintain the temperature of the tip, you use the cryo-head. Okay? Which is supplied by helium gas to maintain the temperature of the tip surface. Okay?

So, this is the top cross-section of the typical atom probe instrument. Fine? So, here, I will briefly give you an overview of how the samples are kept. So, these are called So, I will discuss more when your sample preparation is ongoing.

So, these are called silicon silicon coupons. These are called silicon coupons. And on these silicon coupons, you can see that there are some protrusions. These are called silicon protrusions, which are called silicon posts, and if this particular length of the silicon post is around 100 micrometers,

and if you see from the top surface, this particular diameter of that post is around 2 microns. Okay, so usually, we will paste the samples on this particular silicon coupon and make a sharp needle by using a focused ion beam. I will cover these topics later, and this particular silicon coupon is held on a copper clip, which will be inserted into the chamber in front of the electrode, fine. So, this is how we usually put the sample inside the chamber, okay.

So, I hope you got a good understanding of the basics related to field evaporation, field ionization, and the difference between the atom probe, the difference between the atom probe and field ionization microscopy, and also what field desorption microscopy is, how it evolved into atom probe tomography using time-of-flight spectroscopy, and we also discussed several parameters which are responsible for proper reconstruction.

Okay, so, we have discussed the image compression factor, field factor, temperature, the types of pulsing, and the artifacts which usually occur during the reconstruction of the sample. These artifacts might be induced experimentally. This is related to local curvature effects that can be due to trajectory aberrations, surface migrations, or roll-over atoms. Okay, so, these things can be controlled by temperature, the type of pulsing conditions, and the shape of the specimen.

So, with this, I will end this class now, and in the next class, we will discuss more about sample preparation methods. Okay, so, I will discuss the types of sample preparation methods related to electropolishing, and also focused ion beam instruments. Thank you.