Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-01
Lecture-04

So, welcome to the fourth class of this particular course. Just to revise, in the last class we went through some basic physics related to the field ionization of gas atoms. and the tunneling process—what are the critical things needed for the electron to tunnel through the gas atom toward the metal. And those two critical things are the applied electric field, which increases and makes the minimum potential energy near the Fermi energy of the metal surface or the metal tip. Then, the X critical, which, if it goes below the X critical, the repulsive forces will take place. So, the tunneling process cannot happen.

So, it should be at least near the Xc or more than that so that the tunneling process can take place. We also saw some examples of the FIM images where the gas atoms—which are imaging gases—get ionized under very high positive voltage when applied to the tip. And these gas ions accelerate toward the phosphor screen and hit the positions, which are nothing but the atomic terraces found on the atom probe tip. So, the next question is: what is the magnification we get through this field ion imaging? Fine. So, as I told you, the bright spots on the FIM are nothing but the places where the atoms are located on the tip surface.

Now, you can clearly visualize these atoms on the FIM images. Okay. So, there is a certain magnification associated with that. Fine. So, in the atom probe tip, you cannot resolve those atoms with the naked eye. But by using this field ionization microscopy, you can actually visualize these atoms. So, there is a certain magnification involved. Fine.

To understand this magnification, first we need to understand the effect of shape on the electric field. Okay? So, ideally what we see is that if you have a sphere, this is a particular sphere. And ideally, this has the same radius along all three directions. So, it is

R. Okay, and from the basics, as we know, if it is a positive charge, if we apply a positive charge, then we can express the field along this sphere as the voltage applied divided by the radius.

And this radius is constant. Correct? Along the three directions. So, in the ideal case, we have a field along this particular sphere as the voltage divided by the radius of that particular sphere. So, this is the ideal case.

Fine? But our atom probe tip is not in the ideal shape. It is a truncated cone shape, and only the surface has a certain radius of curvature which is equal to R. But as you move inside the tip surface, you can see that the tip surface is not an ideal sphere. It is a truncated cone.

Fine? So, if you apply a positive potential, if you apply a positive voltage, then what will happen? The electric field lines are not the same along all the directions perpendicular to the tip surface. Okay? So, you can see these electric field lines.

In this sphere condition, these electric field lines are the same along the radius, along the sphere, along the surface of the sphere perpendicular to the surface of the sphere. But this is not the case for the truncated cone. So, due to the shape effect of this truncated cone, you will have electric field lines which are not the same as in the ideal case, fine. So, now here the field which we are getting all along the tip near to the tip we can give as V by KFR, okay. So, V by R is the ideal case.

And we are adding a field reduction factor which is called Kf. Okay? And this Kf value directly depends upon the shank angle. What is the shank angle? If you have an atom probe tip, the half angle is called the shank angle.

This is called shank angle. Okay, and this Kf value directly depends upon the shank angle. The smaller the shank angle, the smaller the shank angle or the theta. The larger will be the electric field or the concentration of electric field, electric field concentration. The larger will be the electric field concentration, resulting in a decrease in the Kf value. Okay, so, the larger the shank angle, the lower will be the electric field concentration, the larger will be the Kf value, the larger will be the factor.

Which is needed to take care of modeling the electric field around the tip surface. Okay? So, this is one field reduction factor which we need to introduce to mimic the exact condition of the field lines across the tip. So, there are other parameters which govern the field reduction factor Kf. It is not only the tip shape itself but also the presence of any flat surface, the presence of any flat surface. So, usually, this atom probe tip has been kept. In a certain coupon, or we can call it a substrate, or we can refer to it as the coupon where the newer sample tip surface is.

So, the flatness of this surface is also a very important contributor to the Kf value, which increases the Kf value. Okay, and also, what we now have in the present leap, in the present atom probes, as I told you during the history of evolution, in the latest leap. What we do is, near the atom probe tip, we place a counter electrode, a hollow counter electrode. This counter electrode is very important to increase the electric field concentration around the tip, which leads to the reduction of the Kf value. Fine. So, because the Kf value is directly related to the electric field concentration around the tip surface.

Okay, so the distance between the tip and the counter electrode can also be changed, which can affect the Kf value of the field reduction factor value. Fine. So, the second important thing is the temperature. The second thing is temperature. The sample tip surface is kept at cryo temperature. So, the specimen is kept at cryo temperature, cryogenic temperature, okay?

So, it is usually kept between 25 Kelvin and up to 120 or 150 Kelvin, correct? So, at sub-zero temperature. Why cryogenic temperature? Because at room temperature, as I told you before, the atomic vibrations. The atomic vibrations will be much higher at room temperature.

So, those have to be frozen out. These atomic vibrations should be minimal. So, why should atomic vibrations be minimal? If your atomic vibrations are minimal at the tip surface, your gas atoms can actually mimic the atomic terraces or the atomic structure very efficiently. Okay? So, your gas atoms, the placement of the gas atoms, can be fixed at those terraces.

Okay, so this directly is related to the thermal accommodation of the imaging gas, thermal accommodation of the imaging gas, and due to the cryogenic temperature, this thermal accommodation or the thermal energy of the gas KBT correct is reduced drastically, okay, and so this will directly affect the lateral velocity of the gas atoms, okay, the lateral velocity of the gas atoms thereby which in this is directly related to your resolution. Resolution means the ability to differentiate the two atoms which are present on the tip surface by the imaging gas atoms.

So, this is the reason for the cryogenic temperature, necessary for the cryogenic temperature. Okay, so now we have introduced the field reduction factor, okay, which is usually between 3 to 8, fine, and it is directly related to the shank angle of the tip surface, tip surface, tip, and higher is the, smaller is the shank angle, higher will be the electric field concentration and the smaller will be the Kf value, okay. Now, the next thing is the projection.

Now, I have talked about the image projection, correct? So, it is usually associated with the magnification, fine? So, now you have a tip surface. So, as I told you, this is the tip surface, and the tip surface initially it has a certain, assume that at the surface it is usually like a sphere, fine? And this is the central point, fine?

And this is the radius of curvature R. Correct? Now, you are applying a field, and this is your phosphor screen. Fine? And the distance between the tip surface and the phosphor screen, we assume it as L. Fine?

So, now you are applying a positive potential. A very high positive potential. So, what will happen? The gaseous atoms present at the tip surface get ionized, and your field lines, which are assumed for a sphere, are perpendicular to the surface. Correct?

For a sphere, but ideally, it is a truncated cone. So, your field lines will not be exactly perpendicular to the surface. So, the field lines will go in this direction, like this. Okay? And this change in the field lines—the path of the field lines—is directly related to the shape of the atom probe tip.

Okay, the shape of the atom probe tip. Now further, so now these electric fields, whatever the electric field that is induced, accelerates these gas ions. Okay, this accelerates the gas ions, and they hit the fluorescent screen. Based on the tip geometry, the terraces, or the crystallographic orientation, you will get an FIM image. Correct? You will get an FIM image.

Now, as I told you, this is a truncated shape, and your field lines are not always perpendicular to the surface. Near the surface, these are perpendicular. But if you go away from the surface, your field lines are compressed. So, these are called compressed field lines, compressed field lines, okay. And this will have a direct consequence on the

consequence on the magnification. How? This we will show now. So, now assume that this has a radius of curvature, this has a radius of curvature, fine. So, now assume that there is, this is the central point of the fluorescent screen, okay.

You can mark it as a 0, O. Then the atom from this position, So, I will just show the atoms. If you have an atom probe tip, assume that okay, so you have an atom probe tip. So you have an atom probe tip, okay, and you have some radius of curvature R, okay, and you have a fluorescent screen. This is the center O, and this is your axis, fine. And as I told you, the field lines are compressed towards this particular axis. This is the central axis.

This is the central axis. Your field lines are compressed towards the central axis. Now, if ideally the gas atom gets ionized at this location, it should go and travel in this direction exactly at the perpendicular. But ideally, this is not the case. Fine. It goes along the field lines in this direction. Okay.

Assuming the distance between these two is D. Okay, so this is the ideal case perpendicular to the tip, but it goes along this field line. Your gas atom will go along this field line, which is compressed towards this central axis, okay, and this is mostly related to the tip surface, the field distribution along the tip surface. Now, if you project this one at the back side, okay, so you will have a point which is P. Which is not exactly at the center of this sphere, okay, and if I magnify this, how it looks, so you will have this particular case. You will have an R.

Okay, and from this particular field line, it is like this, so you will have a P position. This is, you assume this as a Q position, the center position, okay? So, now assume that this is your fluorescent screen, this is your center, fine? And this distance is D, and the distance between the tip and end is L, okay? So, you know that this R value is very small.

It is less than 50 nanometers. It is very small compared to this L. Fine? We can assume that the distance between this particular, the distance between the tip and L is L, and R is much, much less than L. Okay? This is the assumption.

Fine? So, now if you see the triangles here, if I do the triangular geometry, you can see that the distance, this particular distance is D. Okay? And this is your central line. And the position of the gaseous atom at this position from the central line is D. So, you can see that in this particular case, it is nothing but a triangle. Okay?

So, if you see the, if you get the definition of projection, the position of the gaseous atom from the center and the position of the gaseous atom on the fluorescent screen from the center, that is called the magnification. That is nothing but M projection which is equals to D by this particular D. So, this is particular D and divided by this particular D, okay? So, how it is equal considering the r is much much less than the l it is directly related to your. So, this is from this distance it is r and you can see that this particular distance it is around we can what we can do is we can write it as a to this position phi r and this distance

So, if you see the triangle, if you see this particular triangle and if you see this particular, then by definition of this particular triangular geometry, what we can write is d by d m projection and assuming that r is much much less than l, l can be d by d is equals to l by phi r. This is called image compression factor. Image compression factor. So, based on this we can, so this is equivalent by assuming that your r is much, much smaller than L. So, M projection is equals to L by psi r. This is the actual image compression factor.

So, now there are two extreme cases here. Phi can be either 1. If it is 1, it can ideally mean that the tip surface is a sphere because your r is constant, or it is very near to the electric, near to the tip surface. The second extreme is when phi equals 2. It means phi equals 2, which means it is a 2r.

So, it means the total diameter is a flat surface. So, it should either be a flat surface extreme or a sphere. So, the phi value is always between 1 and 2. Usually, we will set it as 1.5 to 1.6, depending on the geometry. Correct? So, how do we calculate this phi?

How do we calculate this phi? This can also be calculated crystallographically because of your atom probe tip. If your atom probe tip is crystallographically oriented in a particular direction, then what you see on the field image is nothing but the image of the terraces of the atom probe tip. Here, on the right side, you see an example of a BCC lattice of tungsten, and you can observe that at the tip surface

there are certain atomic terraces related to the crystallography. Fine? As I mentioned, you can measure from the distance P. You can measure if these gaseous ions hit the fluorescent screen, and you get the distribution of these spots, which relates to the atomic terraces. If you view this particular FIM image in a top projection, you can see concentric circles at the center.

Fine, you have concentric circles at each location here, and these concentric circles, you can see that they have a particular symmetry. Fine? And you can index these crystallographic concentric circles. For example, I can index this as a 110 at the center, and at the right, you can have a minus 110. Now, if you project this particular, for example, you will have this particular zone. If you talk about 112, you can see that this 112 is here.

This is a 110 at the center. Now, if you project this particular angle, okay, so you know that these crystallographic directions for a BCC, the angle between 110 and 112, will not change. It is a constant. Correct?

You can measure by the cos theta crystallographically. That particular angle, say, is some x degrees. Now, if you measure this particular angle, theta observed. Correct? So this is the theta crystallography, which is x degrees.

This is called theta observed and the phi can be calculated by theta crystallography by theta observed. Fine? So theta crystallography always will be higher. Correct? Theta crystallography will be always higher and theta observed we can get.

So by this we can get the image compression factor which is in between 1 and 2. Okay? So typically for any value of L, so for example if you have a value of L is equals to around 90 mm. So usually 90 mm L and R equal to 50. Okay?

And your ICF value, if it is around 1.5, so you will get a magnification of 10 raised to the power 6 of individual atoms as compared to the atoms which are present at the tip surface. Okay? So, with this, you can see that we have briefly introduced the field reduction factor. which is very important for simulating the field lines around the truncated cone or the tip surface which is K of value and these values are between 3 to 8. Kf is a strong function of the tip shape and the shank angle.

Smaller is the shank angle, more will be the electric field concentration, lower will be the Kf value. Means you are going towards the idle way, towards the idle way for the sphere. So, Kf is smaller, higher is the electric field concentration, correct? Then we discussed about the cryogenic temperature. What is the need for cryogenic temperature?

It actually decreases the thermal energy. So, it is directly related to your thermal accommodation of the imaging gas on the tip surface. So, it reduces the lateral velocity of the gaseous atoms which directly controls the resolution. This particular lateral velocity of the gaseous atom we will talk again when I will discuss about the resolution of the during the field ion microscopy. And the second important factor is the image compression factor which is a xi.

This is related to the tendency of these electric field lines which gets bent towards the axis of the atom probe tip. So, this will have a direct consequence on the magnification of the atom probe tip on the fluorescent screen. And this can also be calculated by the crystallography with the help of crystallography. So, you can measure actual the theta crystallography between the two poles which can be identified based on the symmetry on the field line image. And you can also

Measure the theta observed from the field ion image, the actual theta observed. And the ratio between the theta crystallography and theta observed is nothing but the psi, which is called the image compression factor. Okay, and which directly relates to the magnification, okay. So, typically your values of L are around 90 mm to 100 mm;

usually, we will keep 90 mm for L, and if you have a radius of 50 and ICP of 1.5. You can reach a magnification up to 10 raised to the power of 6; that is the power of the field ion microscopy.

So, we discussed the projection of ions and also the image formation. Okay, so now I will just briefly introduce the spatial resolution. Okay. So, I hope that most of the students or most of the listeners have an idea about the resolution of a microscope. Okay. And this is a very simple thing which is related to the Rayleigh's criteria. Correct? So, what we can tell is if any two images are just resolvable,

when the center of the diffraction pattern of one is directly over the first minimum of the diffraction pattern of the other. Okay. So, this is the basic criteria by which any two objects can be resolved. This is called Rayleigh's criteria. So, here I am showing you a nice schematic which is taken from this reference, Lumen Learnings, where if you have two objects, object 1 and object 2, and if you have a small aperture here, and if these two objects illuminate and form an intensity profile on this particular screen, okay.

be resolved at different positions only when the maximum intensity of one object overlaps with the minimum intensity of the other. So, this is the minimum criterion which is related to a theta minimum, which is related to the resolution limit or the Rayleigh's criterion, okay. And this is a nice schematic from this particular reference micromagnetic.fac.edu where you can see that Usually, we see it is in a three-dimension. So, these are termed as airy disks.

So, any object, if it illuminates, if there are two objects in three-dimension, So, you will have an airy disc where the intensity will be maximum or diffused at the center and you will have a loss in intensity again it increases again it loses. So, you will have a certain pattern. of this intensity profile, fine? And if there are two objects which has to be resolved, as I told you, the maximum intensity of one object should overlap with the minimum intensity of the another object, fine?

So, this is the Rayleigh's criteria. So, what is the resolution, spatial resolution in terms of film, field and microscopy? So, that this also is very important. So, in field line microscopy the definition or in FIM is the size of the smallest image spot on the screen on the fluorescent screen means size of the smallest image spot on the fluorescent screen which is related to the gaseous atoms or gaseous ions which are projected or which are hitting to the ah ah fluorescent screen ok.

So, there are three major factors. There are three major factors which directly influence the resolution in FIM. Okay. So, what are those three factors? One is the size of the ionization zone, the second one is the lateral velocity, and the third is position uncertainty. So, what is the size of the ionization zone?

The size of the ionization zone is related to each spot, the spot which is formed by the successive impact successive impact of a continuous flow of ions projected on the fluorescent screen or detector. Okay. So, this particular thing is directly related to the ionization zone, which is exactly above the sample tip surface. which is exactly above the sample tip surface. So, there is a certain ionization zone exactly above the sample tip surface, and the size of the ionization zone directly influences the resolution in FIM, which is directly proportional to the distance between the gaseous atom and the tip surface, okay?

How is that? So, we will discuss this. So, the second term is called the lateral velocity, okay? So, how about this lateral velocity? So, lateral velocity is directly related to your trajectory aberrations.

Trajectory aberrations. Okay, we will discuss these trajectory aberrations in the next class. So, I will just introduce these terms so that you will be familiar with these three important terms. The other one is the position uncertainty. Remember that these gaseous atoms we are talking about are at the atomic level, okay? And it creates an image of the atomic surface of the surface, fine?

And this position uncertainty is directly related to the confined volume, the confined volume on the tip surface. On the tip surface, okay? So, this is related to your position uncertainty, okay? So, we will come to all these three major factors in the next class, where I will briefly explain these three important factors which directly influence the resolution limit of the FIM. Okay, so with this, I will end this class, and I hope you understand how the imaging gas atoms in the FIM and the application of the electric field we can actually magnify the structure of the atom probe tip on a fluorescent screen. And this magnification is directly estimated by taking care of the image compression factor and also the distribution of electric field lines can also be directly mimicked by putting kf, which is a field enhancement factor, the kf value, okay, which directly depends upon the the shank angle of the atom probe tip. So, with this, I will end this class, and we will meet in the next class to discuss more details related to the spatial resolution of FIM.

Thank you.