Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-02
Lecture-05

So, welcome to the fifth class of this course. So, briefly, I will introduce that in the last class we discussed the field reduction factor, image compression factor, and the importance of cryogenic temperature on field ionization. So, I will just briefly touch on those terms so that we can move forward. So, as you see in the slide, we have defined the field reduction factor Kf. Okay, and it directly depends upon the shape of the tip, especially the shank angle. The shank angle is nothing but the half-angle at the apex of the atom probe or the needle specimen, correct?

And this field reduction factor takes care of the fact that as the shank angle is lower, the electric field distribution is denser at the needle specimen, so your Kf will become nearer to, will be reduced. Fine. And the importance of cryogenic temperature: the cryogenic temperature is important to lower the thermal agitation of the surface atoms. Okay. What will happen if this is done? Then, what you can do is improve the thermal accommodation of the imaging gas. The gas is more efficient, okay? And this is due to the reduction in thermal energy, which is directly related to KBT of that particular imaging gas. Fine.

And this cryogenic temperature also decreases the lateral velocity. I will briefly discuss the lateral velocity and its importance for resolution because it improves the spatial resolution. Okay, so this is what we discussed in the last class. Then, we also discussed the image magnification. So, in the last class, I drew the schematic. Now, you can see here: this is your needle specimen, and this is your phosphorus screen. Okay. So, due to the image compression factor, the field lines—ideally, the field lines should be radial in this direction.

But due to the image compression factor, what will happen? These field lines will try to bend in this direction, fine. So, it will be compressed toward the main axis, the central axis of the needle specimen or the phosphorus screen, okay. So now, if you see these triangles, if you see these triangles, okay, this is your this particular triangle, and this is the distance which we call D, okay, and the length from the tip L, fine.

So now, the distance between the surface atom and the axis can be regarded as d, fine. So, and now, as these field lines are not exactly in the radial direction, they are bent toward the axis. If you project it, it will go to a position away from the center of the needle specimen or the center of the tip radius, fine. So, that can be given by a xi R, okay? Xi is the image compression factor, okay?

So now, if you see, as you can see, this particular xi R is much lower than L, okay? So, if you see these two triangles, triangle 1 and triangle 2, Okay, and if you apply analogy, then you can deal with the M projection as D by d, meaning the distance between these two D by d is maybe equivalent to, as xi R is much less than L, we can write it as L by xi R. Fine. So, this is the assumption that, usually, ideally, it should be L plus xi R. divided by xi R, but as you know that xi R is much less than L, so we can neglect this term.

So, magnification is given by L by Xi R fine, and Xi is the image compression factor. Image compression factor, and we also talked about crystallography, as I told you. In the FIM image, you have these concentric rings, and these rings correspond to a particular crystallographic direction. So, this is a tungsten BCC. BCC, tungsten BCC. This is an FIM image of a tungsten BCC needle specimen, and you can see that these concentric rings are exactly at certain crystallographic directions.

These are nothing but stereographic projections, okay? So, what we can do is, you can actually see these rings, and we know that in a crystallographic direction, the angle between the two directions remains the same. It remains the same and can be calculated, okay? It remains the same; it can be calculated. So,

what we can do is measure the observed angle, theta observed, between the two zone axes or the two crystallographic axes.

We can also calculate it by a simple equation of cos theta, which depends upon H1K1L1 and H2K2L2, that theta crystallography. And Xi can be calculated or estimated by theta crystallography divided by theta observed. Okay? So, by finishing some more classes, we will have some small mathematical questions where we can calculate the image compression factor for different types of FIM images. Fine. Next is Just briefly, projection of ions—so image formation. How is the image formed? As I told you, you are applying a positive voltage on the needle specimen and passing an imaging gas, okay?

And this imaging gas actually gets polarized near the tip surface, and due to polarization, this gas converts to plus ions. Okay, positive ions, and these positive ions will be accelerated towards the phosphor screen by the electric field. Okay, so this is a brief review of what we discussed in previous classes. So now we will also discuss a little bit about the resolution of a microscope. So, how can it be compared to the FIM?

Okay, usually the resolution of a microscope can be defined as two images being just resolvable when the center of the diffraction pattern or the center of an intensity. The maximum intensity is overlapped with the minimum intensity of another image, fine. So, that is the Rayleigh's criterion of resolution. If these two points are overlapped, then you cannot distinguish or resolve those two object features. So, this is the definition, the typical definition of resolution in a microscope.

But what is the definition of FIM? FIM, the definition of FIM relies on the size of the smallest image spot on the screen. Okay. And there are three major factors which govern or control the image spot on the screen. These three factors are the size of the ionization zone, which is formed just above the tip surface, the lateral velocity of the imaging gas, and also the position uncertainty of the imaging gas. Okay, so I just briefly went through it.

So, now we will discuss these three factors which influence the image spot in a little bit of an elaborate way, okay? So, first we will discuss the size of the ionization zone. Fine. Size of the ionization zone. What is the size of the ionization? It is the So, this size is of the same order.

First factor: same order as the size of the imaging gas atom. Imaging gas atom, okay, and it critically depends upon the amplitude and distribution of the electric field. Above the atom on the tip. Okay. So, what are the two important factors which are critical? One is the amplitude and distribution of the electric field.

So, if the field is relatively low, as we see, as we discussed in the third class, if the field is relatively low, the critical distance Xc. This is the optimum distance where you can get the tunneling effect. So, the critical distance Xc will be very large. So, if Xc is very large because of the low electric field, then whatever the imaging gas is there, whatever. So, these are the atoms on the tip surface of this atom probe tip.

So, the imaging gas which is on the surface near the tip surface. If the XC is very large, then what will happen? It will not replicate exactly the position of the atoms. Okay. It means that it will not reflect the structure of the surface of the atom on the phosphor screen. So, it means it cannot mimic the structure of the atoms which are on the surface of the tip.

Fine? So, Xc should not be as large, so a low electric field. You cannot image the exact structure of the tip. If Xc is very small, meaning not, so if the electric field is very high, then what will happen? The gas atoms are very close, the gas atoms are very close to the atom probe needle.

Okay. What will happen if the gas atoms are very close to the atom probe needle? If you magnify this part, if there are two atoms, the ionization zone, the ionization zone which is created by the gas atom, it will overlap between the two atoms. Then you will get a blurred image. You will get a blurred image. So, there is a term which we need to define: the best image field, which is called BIF.

So, this is a very important term, as this is the field. What is the definition of this field? This is the field for which the volume of ionization the volume of the ionization zone is minimal and produces the highest resolution. So, this diff is directly depends upon the imaging gas especially the evaporation field of the imaging gas.

or what we can tell ionization energy of the imaging gas, ionization energy of the imaging gas, okay? So, different gases can be used for the imaging, correct? So, different gases have a different BIF value. For example, for helium, which has an ionization of 24.6 electron volt, it has a BIF value of 44.6. volt per nanometer.

Similarly, for argon, the ionization is around 15.8 electron volt. So, there the BIF value is 22 volt per nanometer. So, depending upon the imaging gas, you have a best image field where you are getting the volume of the ionization zone is minimum and also with the highest resolution. So, it is a balance between the low electric field and high electric field.

So, this is the importance of the size of the ionization zone. Now, coming to the lateral velocity, the second term—this is the first term, the second term. So, ions are formed when the gas atoms jump through the ionization zone near the surface. Okay. So, you have an atom probe needle, you have an ionization zone. The ionization zone on the atom probe tip—so these gas atoms are jumping across this ionization zone due to the electric field, fine, and their velocity. Jumping means they have a certain velocity.

And their velocity has a component parallel to the tip surface that totally depends upon the kinetic energy, and this kinetic energy depends upon the tip temperature. Okay. So, this is how it is related to your cryogenic temperature. That is why we keep. So, the kinetic energy depends upon the tip temperature—the kinetic energy of the imaging gas. And this kinetic energy directly contributes to the lateral velocity, which is along the tip surface.

Okay. So, after the field ionization, the gas ions are projected from the vicinity of the tip onto the screen. Hence, if the ions have components, which are generated

above the single atom, have a statistical distribution—statistical distribution of statistical distribution of velocity, initial velocity in lateral or tangential, then it will not follow the same trajectory. It will have some disturbance at the point when it strikes the phosphor screen.

So, since these trajectory aberrations induce, so this will induce a spread This will induce the spread on the ion impact on the phosphor screen. So, that is why we see, we do not see a point, we see a spot which is literally a spread on a spread which is contributed by the size of the ionization zone and also the lateral velocity. So, this is the importance of the lateral velocity which directly depends upon the kinetic energy and this kinetic energy is directly related to your tip temperature. The third point is position uncertainty.

What is position uncertainty? I think everybody must have heard about the Heisenberg principle. So, for any gas atom, the position and energy of the atom cannot be precisely determined. For any gas atom, the position and energy. cannot be determined at the same time.

Okay? So, for example, the de Broglie wavelength of a helium atom, the de Broglie wavelength of a helium atom at a thermal agitation of 20 Kelvin is around 0.2 nanometers. Okay? So, bigger than the size of the zone in which the atoms are ionized. Okay?

So, this is the position uncertainty. So, you cannot neglect the quantum quantum nature of imaging gas atom, fine. So, quantum nature of imaging gas atom. So, this effect will be much less in FIM than in the first, in the second class, I just introduced about the FEEM. FEEM

Okay, so, what is FEEM? So, I must remind you that this is the first microscope which was built by Müller, where he used electrons for imaging. He has imaged the electrons. Now, you can see that in the field ion, in FIM, we are using ions for imaging to see the image on the phosphor screen. So, ion is much heavier.

Electrons are much lighter. So, what will happen? The position uncertainty of electron is much higher. But the position uncertainty of heavier ion is much less. So, the fluctuation or the spread due to the position uncertainty of electron will be very high.

But for ions, it will be much less. So, you can resolve atoms by using imaging gas ions. However, you cannot resolve less than 2 nanometers using electrons. So, that is why Müller transitioned from FEM to FIM to image gas atoms. Fine.

So, now you can correlate how important is the position uncertainty for imaging on the phosphor screen. So, here there is a simple equation to give an approximate spot size when an ion strikes to the phosphor screen. Delta zero square is related to your size of the ionization zone. The second term is related to your lateral velocity. The third term is related to your position uncertainty.

Now, here the xi is the image compression factor, fine? Your Kb is the Boltzmann constant, Kf is the field factor, okay? F is the field, fine? Then here actually H is the Planck's constant, fine? H is the Planck's constant and M is the mass of the imaging gas atom, okay?

So, this is the resultant spot is the addition of square addition of and square and square root addition of size of the ionization zone, lateral velocity contribution and also the position uncertainty contribution. I hope now you understand the how the definition of resolution in film which is directly related to the size of the smallest image spot on the screen. And also more important is why Mueller has gone through from field emission electron microscope to field ionization microscope. This is directly related to the resolution which directly depends upon the position uncertainty due to the Heisenberg principle.

And as I told you, these electrons are much lighter and ions are much heavier. The position uncertainty will be much higher for the electrons than for the ions. So, your resolution will be much lower in FEM, but in FIM, we can actually resolve the atoms. So, now we will briefly go through the instrumentation, which is very important to understand how the typical FIM is reconstructed at an early

stage. So, the instrument, usually the low-temperature field ion microscope (FIM), is better described as a low-temperature sharp emitter field ion microscope.

And this was first made to work, as I told you before, in 1955 by Bahadur and Muller. When Bahadur suggested making the sharp needle and also reducing the temperature to cryogenic temperatures. So, this is a typical diagram, a schematic where you can see a chamber. The instrument has an ultra-high vacuum chamber. So, this is an ultra-high vacuum chamber.

With an ultra-high vacuum chamber with a base pressure of 10 raised to the power of minus 8 pascals, okay? And this ultra-high vacuum is achieved by a series or by the high compression ratio. High compression ratio turbo molecular pumps—turbo molecular pumps—these are backed up by several rotary pumps, okay? So, rotary pumps will give an initial vacuum up to 10 raised to the power of minus 3 to minus 4, then these are picked up by the turbo molecular pumps up to 10 to the power of minus 8. So, as I told you, for high resolution, The most important part is that the specimen temperature should be at cryogenic condition, cryogenic condition, okay.

So, in previous FIMs, the specimens were cooled down by using liquid nitrogen, hydrogen, or helium. But now, in the latest FIMs, they use cryostats. What are cryostats? They use a closed-cycle, closed-cycle helium gas. Okay.

And these can reach a temperature of up to 20 Kelvin. Okay. So, the specimen temperature can be brought up to 20 Kelvin. The third important part is the very high DC voltage. Which is applied to the specimen.

So, this is a specimen holder. This is your specimen tip. So, a high DC power supply is connected to the specimen in order to generate a very high electric field in front of the tip, in front of the tip, a high electric field. So first is a high vacuum chamber, an ultra-high vacuum chamber, then after that, the tip is to be cooled down to liquid nitrogen temperature by using cryostats and the application of a very high DC voltage to the needle specimen.

Once this tip is stabilized and thermally Once the tip is positioned and thermally stabilized, an imaging gas is introduced into the chamber with a low pressure between 10 raised to the power minus 3 to 10 raised to the power minus 5. 10 raised to the power minus 3 to 10 raised to the power minus 4 pascals. So, here the imaging gas, the most commonly used imaging gases are helium, neon, and sometimes also hydrogen is used in some cases.

And also, we can use multiple combinations of gases because, as I told you, these different gases have different ionization energies. Based on that, they have different BIF values. So, depending upon your sample, depending upon your sample's crystal structure or sample phases present, you can use a mixture of gases to resolve that particular atomic structure of the needle specimen, correct. So, these are the four important things.

Now, coming to the after-imaging gas, the one more important part is the phosphor screen. So, in previous FIMs, in older FIMs, the detector is just a single phosphor screen, okay? And these ions, as I told you, are very heavy. The imaging gas ions, which are polarized, when they strike this phosphor screen, usually this phosphor screen gets damaged very easily. Okay, so, and also the problem is, as I told you, these field ion images are extremely dim because I will come to that—these field imaging gases release very few photons per ion.

So, previously they used only the phosphor screens; now, in the new FIMs, just in front of the phosphor screen, they use another plate, which is called an MCP plate. These are called microchannel plates, microchannel plates—MCP plates. Okay, so these MCP plates, the most important function is they amplify the signal. Okay, how does it get amplified?

So, this increases the gain by 1000 times. By converting—how does it come? They convert the ions to electrons. Fine? They convert ions to electrons.

So, these electrons, which are generated by these MCP plates—I will describe their function later. Once the heavy imaging gas ions hit the MCP plates, or microchannel plates, what happens is these ions get converted into a very high

number of distributed electrons. And each electron will produce at least 100 times more photons than the ion. That is why you get very bright intensity spots on the phosphor screen.

So, this is the role of the MCP plate. Remember, the gain increases by 1000 times compared to the ion striking the phosphor screen. So, MCPs, as I told you, are located just 1 to 2 mm apart. in front of the phosphor screen. So, this is an MCP plate, and this is your phosphor screen, fine?

So, the distance between these two is 1 to 2 mm. These MCP plates are nothing but a micro channel plates, okay? And these are acts as a image intensifiers. Now, if you talk about multi-channel plates, here in the right side, in the left side, I am showing you a mesh type of thing. This is a typical MCP plate which is seen exactly in the perpendicular direction.

Okay, so, it is nothing but a thin disc. containing a two-dimensional close-packed array of glass tubes. These are composed of glass tubes, which have a diameter of 8 to 25 microns. Okay, and this is typically a honeycomb structure. Honeycomb structure.

Okay, so, on the right side, I am showing you a typical MCP plate, and you can see that these have a hexagonal array of glass tubes. and these glass tubes are typically called channels. So, this is a two-dimensional projection, okay? But in three dimensions, it is actually a glass tube. You can see a single tube here: first tube, second tube, third tube.

So, it has a certain thickness. Okay. So, this is the cross-section image. Cross-section image of a typical multi-channel plate. Okay.

So, and these are the glass tubes. Now, these glass tubes at this surface, the surface of the multi-channel plate. Both at the front and the back, both at the front and the back, these are covered by a thin conductive layer which is a nichrome layer, nichrome layer. This is a conductive layer, okay? This is a conductive layer.

And a voltage is applied across the surface from this surface to the back surface. So, this is a front surface, this is the back surface. So, a voltage is applied between these two, bias voltage is applied between the MCP, the surface of the MCP plate. Fine? What is the role of this bias voltage?

I will come to, I will describe in a few minutes. Okay? And these multi-channel plates are aligned at a certain angle, which is around 5 to 15 degrees. I will come to this at the end of the lecture—why these are aligned from 5 to 15 degrees to the normal axis. Okay.

So, on the left side, you can see this particular schematic, the cross-section schematic of a typical multi-channel plate. And you can see that if this is normal, then these multi-channel plates are aligned between 5 degrees to 15 degrees. Okay. So, okay. So, now, as I mentioned, between this black color,

The black color shaded area is a nichrome layer. Nichrome layer. Now, if you apply a voltage between the two surfaces of the multi-channel plate, the front part and the back part, the voltage applied is typically between minus 0.8 to 1.2 kilovolts. The application of voltage across the front and back part of the multi-channel plate will create a very high surface charge density, a very high surface charge density on the surface of the glass tube. Okay, so this is your channel

and a very high surface charge density will be created on the surface of the glass tube, okay? So, in the middle, I am showing you one glass tube, which is part of a multi-channel plate, where you can see that on the application of a very low bias voltage between them the front part and the back part—this is the front, this is the back part—a negative voltage of around 0.8 to 1.2 kilovolts will induce a very high amount of surface charge density on the glass tube This is the glass tube. The surface density on the glass tube, okay? So now, what will happen is if an ion travels along this direction

Okay, if a positive ion, a gas ion, travels from the tip surface towards the multi-channel plate, what will happen? These ions will strike the surface of the

glass tube. Of the glass tube. And as you know, this glass tube has a heavily charged surface density, having a very high surface charge density. Due to the striking of ions on the surface of the glass tube, which has a very high surface charge density, they will emit a very high number density of secondary electrons.

As you can see in the schematic at the middle, if there is an incident ion, which is striking the surface of the glass tube, there will be a very high number of secondary electrons generated from the glass tube. And out of the secondary electrons generated, a single electron can strike back to the surface. So, it can strike back to the surface to produce a thousand more electrons from the charged surface. So, this is how a cascade of electrons will travel along the glass tube, and due to the electric field applied between the surfaces of the MCP plates,

this cascade of electrons will travel across the channel towards the phosphor screen. So, this is the function of a typical glass tube, which is one of the many glass tubes present in an MCP plate. So, when these electrons come out of the MCP plate—assume this is an MCP plate, okay? So, when the electrons—these are MCP plates—when the electrons come out of the MCP plate, Okay, these have to be converged to a spot. For that purpose, there is a phosphor screen, okay? And this phosphor screen is coated with a transparent conductive layer.

might be indium oxide or is mostly indium oxide. So, these electrons—then what will happen? If you apply a bias voltage of a few kilovolts across the back end of the MCP plate and the phosphor screen, what will happen is these electrons, which have a spread, will convert to a sharp spot on the phosphor screen by the application of a bias voltage again. bias voltage between the back end of the MCP plate and the phosphor screen.

So, this is due to the, this is main function is to reduce the lateral spread of these electrons which are coming out of the MCP plate from the back side, okay? Then these converged electrons which are on the spot, these spots actually hit to the fluorescent screen. As I told you, the energy The typical energy of an emitted or

the photon emitted by electron is 0.5 photons per electron, which is much, much higher than 0.01 photons per ion. Okay.

So, and it produces a very bright spot on the phosphor screen and it does not damage the phosphor screen. That is why we use MCP plates, MCP. which avoids the direct contact of ions on the phosphor screen. So, as a common sense, we can see that the detection efficiency of an MCP can be directly correlated to the area of the open space or open channel across the MCP plate. Fine?

The fraction is usually around 50 to 60% open channels on the MCP plate. Okay? And one more thing to remember is that one ion or multiple ions can contribute to one particular spot on the phosphor screen. Correct. So, the detection efficiency of the MCP plate is directly related to the open channel space on the MCP, which is around 50 to 60 percent. So, remember again, I told you that these channels are actually oriented around 15 to 5 degrees with respect to the normal axis.

Imagine that the MCP, imagine that the channel plates are aligned exactly along the normal axis. So what will happen? The tendency of the ions to strike the glass surface or the glass tube will be very minimal. The ions can travel along this normal axis easily and hit the phosphor screen. But if you keep these channels at an angle,

then the efficiency of heating ions on the glass surface will significantly increase, resulting in a very high density of electrons coming out of the multi-channel plate. Fine, so So, with this, I will end this class now. I hope that you understood the importance of the instrumentation in FIM. And what are the basic things which are required to construct a basic FIM microscope. Okay? In this, the most important points are these: ultra-high vacuum, cryogenic temperature,

Then, the use of high DC voltage, a sharp tip, and imaging gas should be at a lower pressure. And the use of MCP, multi-channel plates, just ahead of the phosphor screen, so that you will have a large amount of photons released on

the phosphor screen without damaging the screen. Okay, so, we will meet in the next class, and I will proceed with how to interpret the FIM images for a solid solution, for an element, or a multi-component or multi-phase alloy. Then, we will meet in the next class. Thank you.