Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-08
Lecture-24

So welcome to this class. In the last session, we just briefly described about the sputtering process. So we talked about the sputtering process is nothing but a removal of atoms from the surface of the sample and the conditions for that these surface atoms has to get enough energy And the atoms has to be displaced outside the sample. And we describe this as four conditions.

One is the ion beam should have enough energy, the initial ion beam energy. So, and it also depends upon the surface, the binding energy, the binding energy of the surface atoms and the third is the incident angle, which is a very important parameter, which is an important setting where you can actually increase the sputtering process. You can increase the rate of sputtering process or we can call it as a sputtering yield. So, the sputtering yield is described by how many sample atoms are sputtered per incident ion, per incident ion.

Okay. FIBS gallium ions the FIBS where you use the gallium ion source this particular yield is between 1 to 10 for an ion beam where when the beam interacts with the sample surface perpendicular perpendicular or 0 degree tilt for the sample correct so this is particularly about 1 to 10 so yield can be defined as the number of sputtered atoms divided by the number of incident ions, number of incident ions, okay?

So, here we talked about this particular incident angle, okay? So, this is a very important parameter, a very important parameter which can control your sputtering yield, correct? So, if you have a sample surface, If your beam falls exactly perpendicular to the sample surface, your ion beam, then it is usually called an incident angle of 0 degrees. Correct?

Now, if in case you tilt either your sample surface or your ion beam, so you have a sample surface, either you can tilt your ion beam or you can tilt the sample so that the ion beam is not perpendicular to the sample surface, then your sputtering yield increases significantly, okay? So, based on the experiment, usually what we tell is if you have this sputtering yield and this is your incident angle, Okay, so sputtering yield is nothing but, as I told you, the number of ions removed per ion. Okay, so this is a scale of 30, 20, 10.

Okay, so as your angle increases from 0 to 90 degrees, your sputtering yield also increases and it reaches a maximum. at a certain angle, which is between 70 to 80 degrees, where your yield is much higher. Correct? This is your dependence on the incident angle. Dependence of the incident angle of the ion beam with respect to your sample surface.

Okay? And this sputtering yield also depends upon your accelerating voltage. So, your yield—your accelerating voltage—then your yield increases with your accelerating voltage from 5 kV to 30 kV or 5 or 2 kV, okay? So, your yield increases with the accelerating voltage. So, this is typically...

This is your typical curve of the variation of the yield with respect to your accelerating voltage, fine? So, with this, what we have briefly gone through is your ion column, okay? Your ion column—then we just talked about the source, which is usually the gallium plus ion source. Then you will have a condenser system or condenser lens. You will have a condenser aperture where you can actually control the beam current.

Correct? If you want a very high sputtering yield, then your beam current should also be increased. And if you want to increase it further, then you can actually adjust the incident angle of the sample surface with respect to the With respect to the ion beam current. Okay? Here you have an objective lens, which usually focuses, and you have a set of

Set of lenses—octopole or quadrupole—which actually controls the scanning of the beam and also the aberrations of the beam. Correct? And we have a blanking plate. So, whenever you need, you do not want sputtering to happen. Actually, then we can put the blanking plates which block the ion beam by preventing interaction with the sample surface.

Correct? So, this is a typical interaction of what we discussed about: the ion beam versus interaction with your sample surface. And we basically only talked about the sputtering process. Now, the next topic, which we discussed in the two classes before, is that you have an electron beam column, okay? This is your chamber.

This is your ion column, and there is one more column which we call the gas injection system. Okay, gas injection system. This is a very important part of the FIB for the operation of FIB. Okay, so what is a gas injection system? So, we will just briefly go through the gas injectors.

What are these gas injectors? Okay, now this gas—so if you have a sample surface here, okay, and your ion beam falls on the sample surface, this is your gallium plus ions. The gas injection system is nothing but a precursor. It has a precursor. Okay, so why these precursors?

So, these gas injectors or the gas injection system, we call it a GIS. It usually introduces reactive neutral gases onto the sample surface. Okay, so You have a sample surface. This is your gas injection system, and it is just kept above the sample surface.

It is usually kept around 200 micrometers distance from the sample surface. Okay. And you have an ion beam coming from the ion source onto the sample surface. Correct? And this gas injection system—it is nothing but the one that induces these reactive neutral gases onto the sample surface.

And these neutral gases can do etching, preferential etching, or can also be used for metal deposition. Okay? So, GIS System, it is nothing but a controlled flow of gas from the crucible. This is your crucible, which contains the GIS precursor.

We call it a precursor on the sample surface through a nozzle. So, this is called a nozzle. Okay, so these precursors are usually solid, correct? And we need gas. So, usually these GIS needles—these precursors—are heated prior to the injection. Okay, so due to the heating, this converts to a gas, and this gas is injected onto the sample surface.

Okay, and the gallium ions... which are accelerated toward the sample surface, they can interact with this gas and decompose. Decompose the precursor. How? These gallium ions can interact with the precursor.

Gas. These gallium ions and they can decompose these precursors. And during decompose or cracking, you can call it as a decomposition or cracking, then these gas, these precursors which is having a certain metal, that metal gets deposited onto the sample surface. Okay. So, based on the metal which is contained in the precursor, it can be titanium rich or

it can be tungsten rich it can be carbon based carbon rich based on the metal which is which you want to deposit on the sample surface okay so you can use different types of precursors which we call as a gis needles where you can actually use a precursor which is a platinum rich tungsten rich or the carbon rich and when the gallium ions gets interact with these platinum rich tungsten rich or carbon rich precursors they usually these precursors usually crack or decompose and the platinum or the tungsten or the carbon gets deposited on the sample region

wherever the gallium plus ions are are going to interact with this with the sample correct and these gaseous precursors are nothing but uh They have a chemical compound as 3, either it is platinum, Cp, Ch3. Okay. So, this particular precursor is get cracked by the ion beam, by the interaction of the ion beam and these metals, whatever the metals you want to deposit on the sample surface gets deposited. So, this is the role of gas injection system.

Okay. And then, the gas injection system—what is the use of the gas injection system? One is, as I told you, it is used for etching, for deposition, and also for welding. Okay. Welding.

So, We will come to welding at a later stage. First, we will describe another part of the FIB system, which is called the manipulator. This is also a very important function. So again, I can draw for you This is the electron beam column. You have a chamber, fine. Then you have an ion source, a gas injection source, and a gas injection needle. Correct?

So, and you have a sample surface. Now, there is another system, which we call the manipulator. Okay? The manipulator is nothing but a sharp needle, which is used for lift-out or removal of Removal of the sample surface.

Removal of any region. Removal of any region from the sample surface. This is used for the lift-out procedure. Okay. So, the lift-out procedure means you can lift out either the TEM lamellar or APT lamellar.

Or any region which is important for your investigation; this manipulator can be used for the lift-out procedures. Correct? So, there are three important things in the FIB system. One is the ion source. The second one is the gas injection system.

And the third one is the manipulator. By combined usage of three important components, actually you can prepare the sample surface and you can remove the region of interest from one place of the sample surface and you can paste into the grids. And these grids can go further inside the TM or inside the atom-probe tomography. Correct?

So, these are the manipulators, sharp needles. Fine, so basically you have an electron beam, an ion beam column, an electron beam column, a gas injection system, and also you will have a manipulator. First, second, and third. Correct? By using these three on the sample surface, you can actually remove the region of interest and paste it onto the grids.

Fine. So, I will briefly show you a video. where you can understand how a sample is prepared, especially for the atom-probe needles. Okay, so this is courtesy of Max Planck. So here you can see the sequence. So here, there is a grain boundary. You are actually sputtering out this particular region, sputtering out this region. So this is your grain boundary, correct? And we are removing these two regions by using the gallium ion source. Fine, now

once you can see that one end of it got cut from the gallium ion source, which is independent of the sample. Then, you use this manipulator and we use the gas injection system to paste or weld the sample to the manipulator. And this particular sample is then pasted onto a silicon coupon. Okay, and this particular sample with the grain boundary is

again used with the gallium plus ions for making an atom probe needle specimen. Fine. So here you can see the importance of the three important parts of this particular

So one is sputtering by the gallium ion source, correct? Another one is the gas injection system and the manipulator, okay? So, by using the manipulator, you can actually lift out this particular lamellar, correct? So, this is... This is your manipulator.

You use the gas injection system to deposit the platinum, and it gets deposited and welded. Then you lift the lamellar and paste it on the silicon post. The silicon post is subsequently milled, keeping the area of interest—which is a grain boundary—for field evaporation in the atom probe. Correct? So,

I hope you now understand the role of the focused ion beam. Now, by using these ion beams, as I told you, you can deposit platinum or carbon on any material. Okay? On the sample surface. So, if you have a certain region of interest, you can mark that particular region with platinum or tungsten deposition on the region of interest.

Okay? Then you can do the cross section. Then also you can mill the sample in such a way that you can extract the different shapes of the specimens. Either Either a cylindrical rod or you have a lamellar, okay? Based on the requirement, okay? So this particular usage we call it a patterning mechanism.

You can use different patterns on the sample surface by using the gas assisted injection system or gas-assisted deposition. One more important application for this particular is the 3D reconstruction. 3D reconstruction. Now, if you have some sample surface, okay? So now if you have a certain region, you have a certain region and you have a certain grain boundary at the cross section and you want to see this grain boundary evolution in three dimensions, correct?

In three dimensions, in this direction. So you need to slice out each layer by layer. First layer, second layer, third layer, fourth layer. So you can use this ion source or the gallium ion source to remove this material sequentially and you can take a cross-section image by using the electron beam

and you can stitch it to see the 3D reconstruction of that particular grain boundary or the grain. Okay. And one more important thing: if you have an EBSD attachment, Then, actually, you can do the EBSD on this particular surface and then remove this surface. Again, you can do the EBSD on the second surface, and these EBSD patterns can be stacked together to get the 3D EBSD data.

So, this is how you can use this ion source. It is a powerful tool where you can actually do the 3D reconstruction. Or 3D tomography. We can call it 3D reconstruction—either the EBSD maps, the grain orientation maps, or also the imaging 3D reconstruction of the features which are inside the surface. So, this is your top surface.

This is your bottom. Okay? So, by using the FIB, we can actually do the 3D reconstruction in three-dimensional construction. So, the video has to say, The beam which is falling on the sample surface also directly depends upon your accelerating voltage.

Okay? As in the electron microscopes, you have an interaction volume based on the voltage. Correct? As your voltage decreases, your interaction volume also reduces. This is your sample surface.

So, this is your electron microscope. Electron interaction volume. Similarly, for the gallium FIB also, the interaction volume for 30 kV will be much higher, for 20 kV it will be less, and for 5 kV it will be much less. Okay? So, based on that, this is the interaction volume.

It is nothing but the travel path of the gallium ions until they lose all their energy and get deposited. Okay? So, based on the accelerating voltage, You can have different interaction volumes. And as I told you, ion implantation, which is a major artifact, may change the composition or structure of the sample surface.

So, the higher the accelerating voltage, the greater the interaction of these gallium ions with the sample, and the higher the ion implantation rate will be. Okay? So, based on your applications or the sample's atomic number, you can actually use the proper choice

or the optimum choice of accelerating voltage for this. Correct? So, this ion beam, as I told you, in the ion column, we have octopoles and quadrupoles.

Correct? And by using these two sets of poles, actually, you can scan the beam on the sample surface. So, if you have a sample region, you can scan the beam from left to right and right to left, or top to bottom in the X-Y direction. You can scan the beam as per your need by using the combination of these octopoles or quadrupoles.

Okay, so this is how we can scan the beam. So, actually, then you can use the required patterning on the sample surface. Correct? Now, as in the electron beam case, the electron beam should be sharp enough as a point source, correct? In SEM or TEM, okay?

And they also have certain aberrations in electron microscopes. Similar aberrations also exist in the ion source, okay? So, your ion beam alignments are very crucial, correct? If your ion beam alignments are not proper, correct? Then you may not get proper patterning.

Okay. Even though you have kept a pattern for the rectangular section, but due to the aberration in the beam, the ion beam, due to that aberration, might make it possible that you cannot get a sharp rectangular cut or sharp rectangular sputtering. You may end up with a bulge due to the aberrations. So

The first important point is the beam should be focused on the sample. If it is a defocused beam, you may induce very unsharp edges. So, if you want a rectangular cross-section or a rectangular cross-section sputtering process, these edges will not be sharp. It might be bulged due to the unfocused ion beam. If you have a stigmatized beam, astigmatism, this is also very important because it also has electrostatic lenses due to which astigmatism may form.

So, your beam is not focused, and due to astigmatism, you may get distorted sputtering. of that particular pattern or shape, okay? And both these two can also exist at the same time, which can lead to very distorted milling—we can call it milling or the sputtering process and you will not get the exact morphology of the specimen that you need. Fine.

So, with this, what we have described now is the sample preparation. By focused ion beam, correct? It can also be used as a focused ion beam microscopy field, okay?

And we have described the basic cross-section of the column. What are the important lenses? So, we call them electrostatic lenses. I explained to you why electrostatic lenses are used, not electromagnetic lenses. That is due to the...

Ions, not electrons, as in SEM or TEM. Correct? It will have better control of the ions because ions are heavy; their velocity is very low compared to the electrons, which are fast-moving. In electromagnetic lenses, the force on those electrons is proportional to the velocity of the electron, but here the velocity is very low, so you need very large magnetic lenses. Okay, so due to this, you use electrostatic lenses. Electrostatic lenses don't depend on the velocity of the charge, okay? That's why we use electrostatic lenses. Okay, so now these ions get accelerated. They can actually be used for patterning, for the sputtering process. They have a certain projection depth, projection depth. Okay, which is the interaction of the gallium FIB inside the sample until it loses all the energy. We also talked about this: your ion source is only for the sputtering process.

The second important thing is the gas injection system, which we call GIS. These are nothing but the precursors, chemical precursors. Okay, which has a certain element like platinum, tungsten, or carbon, and these precursors are decomposed or cracked when these precursors interact with the ion beam, due to which it can deposit on the sample surface. Correct. Now, if you want to remove your material or the region of interest from the sample surface, you have to use the manipulator, and this gas injection system can also be used to weld the lamellar or the region of interest to the manipulator, and this manipulator again can be used

to transfer your region of interest onto the TEM grids or APT sample holders, which is nothing but a silicon post. Or silicon coupons, we will have a silicon post, correct? So this we will briefly—I will go through it in the later stage when we talk about correlative microscopy, fine? So I hope you got the clear picture of this most-used sample preparation method nowadays, which is FIB, okay? So in FIB, you use a gallium source.

So nowadays, this gallium, as I told you, with certain elements, with certain elements, the gallium can actually interact with those elements and can form compounds which are detrimental or which act as artifacts. Sometimes gallium implantation also leads to the fracture of samples, brittle fracture of samples during field evaporation. Okay, so this is due to the interaction of gallium with those elements. The most highly interactive element is aluminum or the light aluminum.

It forms, it contaminates the aluminum, especially if it is in a nanocrystalline form. So, gallium diffusion along the grain boundaries is much higher in the aluminum case. In some cases, with magnesium alloys, it can form certain compounds—magnesium gallium-based compounds—which are actually artifacts in the TEM sample preparation. Correct? So, nowadays, depending upon the source, we also have xenon plasma FIBs. These are almost inert compared to gallium, and these xenon plasma FIBs—these xenon sources—usually do not react with the elements because of their inert nature.

So, these plasma FIBs are now emerging, and instead of gallium plus ions, we can use xenon plus ions, okay? So, another method is the low-temperature milling—cryo-temperature, we can call it cryo-temperature. Usually, the milling in FIB is done in a very high vacuum atmosphere at near room temperature, and the diffusion of gallium into the sample at room temperature will be much higher. Because these are liquid gallium plus ions, and diffusion at room temperature is much higher into the sample surface.

If you perform cryo milling—cryo-temperature milling—then the diffusion of gallium plus ions can be limited. So, you can reduce gallium damage, especially on aluminum samples or magnesium samples. If you do the milling at cryo-temperature—meaning liquid nitrogen temperature. So, there are certain cryo holders or cryo setups available now where we can install these cryo setups and perform low-temperature milling to reduce or prevent gallium damage on the sample surface.

Okay. And this cryo setup also—this cryo setup also— provides an opportunity for these ion mills to be used for biological samples, sectioning of biological samples, or polymer

samples. So, these are the different accessories that can be added on. to the existing gallium source FIBs, where we can reduce the damage caused by the ion beams, and it

can be used for biological samples or polymeric samples by low-temperature milling. So, I will end this class now. So, now we have completed the sample preparation methods. In two classes before, I briefly described electropolishing, which is still being used, but the only problem is that site-specific samples are very difficult to prepare with electropolishing.

So, if your sample is homogeneous, and if your sample has precipitates or phases that are distributed uniformly along the microstructure, then electropolishing can be used effectively. But if you need site-specific samples, for example, a particular phase, particular interface, grain boundaries, twin boundaries, or faults, then you actually need to prepare the sample site-specifically. Okay.

So more on sample prep—sample preparation methods. In FIB, we will just briefly go through in the next class where we have different lift-out methods. Okay? So, as I told you, your ion beam falls on the sample surface. You are actually milling the sample just...

The cross-section samples you are making—what we can do is we can also do the in-plane lift-out method. So now, if you have a sample surface, you know this is a particular boundary. But if you see the cross-section, you don't know if this boundary can go inside the sample or it just comes out. We don't know whether the boundary exists or not. So what we can do is we can prepare a sample which is in-plane. From the sample surface, we can make sure that in the needle specimen, whatever you are making the needle specimen, you have that particular boundary perpendicular to the axis—the tip axis, correct?

So, these are different in-plane lift-out methods which we will go through briefly in the next class. So, we will meet in the next class with these further descriptions. Thank you.