Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-09

Lecture-27

Welcome again. In the last classes, we covered the sample preparation by focused ion beam. We have done the basics, instrumentation, and we learned about the ion beam column, gas injection system, manipulator, and how to draw patterns by in and how this FIB functions to sputter the material from the sample, right? The sputtering process. So, we have gone through a little bit of the basics related to the sputtering process also and some methods which are used to either prepare the TEM samples or the APT samples or any of the sputtering-related things. Fine, now

In the next few sessions, maybe three or four sessions, what we will do is show you how to load the sample in the focused ion beam and how to prepare the atom probe samples. Step by step, we will approach this, and it will be demonstrated by my students, Saurav and also Shashi. Both of them, they will They will teach you how to prepare the sample before loading it into the chamber and what protocols to be used during the preparation and also the parameters which are very important during the preparation of the samples.

Fine, so I hope that you will go through all these videos, and it will be helpful for in the future for how to prepare samples. So, in this video, I am going to explain the sample loading procedure in dual-beam secondary electron ion SEM microscopy, scanning electron microscopy. So, this is the holder. This is called a quick loader holder. So, I will explain to you later what the quick loader is.

So, in this holder we can mounted our sample. So, we can use two Different set of sample. This is the cold mounted sample, which is wrapped with the aluminum foil. And another is the without mounted sample.

So this is the without mounted sample. It is an electropolis sample. So both the sample we can use for the preparation for APT sample. So now first what we will do. So we use the carbon tape because carbon tape is a conductive in nature.

So this is a carbon tape. from where we can cut a small piece so yeah we can cut a small piece of carbon tape and we pasted on the holder fine pasted and we cut another piece and after pasting the carbon tape we can mount our sample Now I stick the sample with the carbon tape. So first we have to check the sample should not fall.

Okay, and the next step is to check that the sample height is proper. Then it can go inside the quick load loader. So we have some demo holders where we can check whether our sample is going or not. So our sample height should not be higher than the circumference of this quick loader holder. Now I will remove my sample.

Now my sample is fine, and I am going to load it into the main microscopic quick loader. This is a Helios dual-beam FIB machine, which consists of two different types of beams. One is an electron beam, and another is an ion beam. So first, I will explain the electron beam. So it is a

The electron beam column: the top part is the source, the electron beam source, which is a thermionic source or a field emission source. Here we have a field emission source. After the source, we have a condenser lens system, which consists of a condenser lens and an aperture. We have an objective lens system and a scanning coil. So this is basically the part in the E-beam, a normal SEM column.

Next, you can see this is the cable which is connected to our electron beam source. This is a high-tension cable. The white one. So, this is a BSE detector. Okay.

So, this is a backscattered electron detector for imaging purposes. As I mentioned earlier, this is an E-beam column, and now this is the I-beam column. So, this is from the I-beam. You can see there is a gallium source there. That gallium source is what we will use.

For ion beam imaging, now this is a GIS needle. For platinum deposition or welding purposes, we use the GIS needle. So, this is the GIS needle, and these are the manipulator components. This is a micro-manipulator, which is used to lift out the sample. This is the quick-loader rod.

This is a special type of rod. We don't need to vent our entire chamber. Only this portion can be vented, and we can load our sample. So once I will press the venting button. So it will vent this small chamber.

First I vent the chamber and open the lid. Now next is I place the sample at this position. position the next thing is so this holder you can see there is a two hinges is there now we can push sample little back side and the hinges should fit at the middle of this gap of the holder once you move the sample backside so that hinges should be at the middle of this holder the gap of this holder now we have to lock these things so for the locking this the sample with the holder we have to turn it to the right side now the sample is locked with the holder now you have to close this holder lid

And now my sample is ready to go inside the microscope chamber. Next step is to press the pump button. Once you press the pump button, so our chamber basically will start to vent. So vent and pump button both are similar in this machine. So now our chamber is venting.

Now my sample is already locked with our quick loader holder. Now it's ready to insert inside the microscope chamber. So before inserting, we have to vent the chamber. So venting means we have to press the pump button, as we mentioned before. So the pump button and the vent button are both similar.

Now our microscope chamber has actually started venting. So it will take around one minute to vent the chamber. After venting, we will load the sample inside our microscope chamber. Now my chamber has vented. Now we use this button and this lock to open the gate, and we apply

some force, some pressure, to allow the rod to go inside. It will go till the end, then unlock it, and it will come back again to the same position. Now you can see the microscope software interface, which is divided into four segments. The top left segment, which is gray in color, is basically an electron beam window. The top right, which is black in color, is called the I-beam window. The bottom left is the nav cam, and thus you can see the bottom right is basically a side view of the microscope. Now, basically, I am doing the nav cam, so the nav cam is basically taking the picture of our sample. So now it's taking this picture.

taking a photo of our sample and it will take at least 10 to 15 second to take picture now you can see that it already taken and next step is to double click on my sample so you can see my sample actually wrapped with aluminum foil now i double clicking it and once i double click now my sample is perfectly aligned with respect to electron beam now the next step is to turn on the electron beam and turn on is both the beam and next step is to set the stigmator a stigmator and beam set to zero zero the main idea behind this to whatever the previous user used in that stigmator and beam shift so you make it zero zero to make it perfect condition now the next step is

unpause the electron beam window now we once we unpause the electron beam window you can see the our sample so now you have to magnify it and next step is to focusing now i am doing focusing on my sample and this is the some dust particle so it easy to focus on a dust particle initially so you have to do focusing at very high magnification around 10,000 12,000 X magnification and you have to correct astigmatism as well now next step is to link z how much distance of my sample is from the pole piece so now i am same thing i am doing astigmatism correction stick astigmatism correction And once I link Z and make it set to 4.

So, 4 is basically the eccentric height for this helios microscope. So, it may be different for the different microscope. Now, I make the nap cam window is turn into an electron beam window. Now, in that nap cam window, I will actually insert BSE detector, ABS detector, annular backscattered electron detector. Now, you can see the features.

So, in AC images, my feature is not clearly visible, but in backscatter, it is a jet contrast imaging. So, you can see nicely that two different features, two alternate lamellar plates. The sample is basically a titanium aluminate sample. The titanium aluminate consists of two alternate plates of Ti_3Al and TiAl. and uh this is uh that um now in that window we are i am basically doing uh focusing at back scattered mode now focus saying going on i am doing focusing

yeah now i am correcting astigmatism as well so it actually basically takes little bit time to perfectly focusing and perfectly to correct astigmatism so once it done this thing once actually our region of interest if uh now uh yeah so basically uh we first we have to do select our region of interest now our region of interest is fixed the next step is to draw a pattern so okay basically actually i choose a wrong yeah so now you can see I have this is actually my region of interest that green color rectangle box From this, that rectangular rectangle, that box, this is our region of interest.

From this region, basically, we will lift out sample for our APT needle. So, in this APT lamella, we actually cover both the phases. So, we will try to do to cover that both the phase should come in a tip. that we can identify what solute element is going which phases. Yeah, now I turn on the EV window in secondary electron mode and I go to view and do center crossing to easily locate my feature.

Yeah, that same positionI am, I am the same pattern. I am actually doing it in AC mode. Yeah, now the next step is to insert the platinum needle. So first, we have to pause this electron beam window. Next, I am going to the pattern option, and actually, I insert the GIS needle—that gas injection system needle. So next, in the application, I choose— Uh, platinum e-deposition surface—always choose surface. There are so many options there: surface, structure. And I make a dimension: X around 8 microns, Y 0.5, Z around 0.5. And now you can see—there are so many options there in the application. Now I set it up as surface and extend the introduction.

You can choose anything. Here, I choose top to bottom. Now I started patterning. It will take around one to two minutes. So it's showing—it will take around 1.5 minutes, one minute 50 seconds it will take. Uh, so meantime, I can tell how this thing is happening. Uh, basically, this is—that gas injection system—this platinum, that platinum precursor is there. So with that precursor, this consists of trimethyl platinum molecules. So when those trimethyl platinum molecules come to our sample surface, when a high-energy electron beam strikes, It basically—that precursor gas, that trimethyl platinum—is basically decomposed, and the platinum atoms actually deposit on our sample surface. So. This is actually the basic mechanism by which, in this way, that platinum actually deposits on our sample. The platinum is basically used to prevent our sample from gallium damage. The main purpose of the platinum deposition is to prevent.

sample from the gallium damage so the sample so my professor already mentioned that aluminium magnesium those samples are very sensitive in gallium so in this this kind of sample we need to deposit platinum more to prevent the damage from gallium now you can see our sample platinum is nicely deposited on the surface and you can see in e-beam window in ac electron mic uh by secondary electron microgram that rectangle same type of whatever pattern we put same type of platinum actually deposited on the surface and the next step is to do eccentric so first what is new sending now you can see that once i tilted my sample My features actually move from center to bottom.

So I using the Z height stage movement by stage movement and I make I actually increase the Z actually. So bring my sample at the center. So this process is called Eucentric. So I will explain what is Eucentric is. So Eucentric is basically so at a particular objective lens current value at particular Z position where our region of interest will not move during tilting.

So basically this is a Eucentric. So at the Eucentric point or Eucentric height our electron beam and the I beam meet at the same point. so now i again i tilt at 30 degree to check now it's little bit move so again i use this z height and bring to the center and next i go to up to 52 degree because in 52 degree my ion beam column is perpendicular to my sample now again i align adjust the z height by stage movement and now my are my eccentric is done now i go to the zero degree and now you can see that my features is almost at the center

it's little bit changing but it's almost center so this position you can so 3.88 mm is basically a eccentric height now Initially, we actually set at 4, but 4 is not a eccentric position. So, 3.8 is now eccentric now. So, now again, I have to tilt 22 degree. Now, for APT sample preparation, we generally tilt our sample 22 degree.

Now, you can see the features is nicely visible in eWIMP. and now i set uh uh now i go to uh ibm window and always you can see that blue mark is there in the blue mark actually indicates that you are in that window that window is basically in live mode so that blue color represent below this so now i i go to ion beam window and now i set the current at 9.3 so 9.3 basically high current we basically generally use for trenching purpose so initially and now i set my we will do trenching from around 1.5 micron above from our region of interest now i can make i act i put a rectangle box which is 3 micron

And we will do trenching 1.5 micron above and 1.5 micron below. So initially, I will do trenching at the top part. So dimension X is around 12, Y is around 5, and Z is 6. And always that scan direction towards the sample from top to bottom. so now i set pattern perfect position and i can start this patterning so basically gallium ion actually

remove material from this pattern region so it will take at least two minutes now you can see in eb window so my material is basically are removing so the high energy gallium ion basically spat sputtering atom so trenching is now going on and our region of interest at the middle so now focus and the microscopy has some inert gas sources like xenon that will basically help for this aluminium sample preparation those who are those material which is more sensitive to gallium so we have this uh fib source is called plasma fib so here we don't have plasma fib but it's the plasma fib is very good for sample which are sensitive to gallium ion so this high energy actually gallium basically sometimes it damage sample too much but normally that nickel based super alloy titanium alloy it's fine

now almost it's strange uh i think 20 second uh is left yeah 10 20 second almost it trenched the top part Now we take that snapshot. One thing I want to mention in I-beam window, don't unpause it at a high beam current. Because if you unpause it, that high energy gallium ion actually damage your region of interest. So this is the main thing.

So don't unpause it at a high current. You can unpause it at a low current, but don't unpause high current. Now I go to zero degree. I come back to zero degree. and i am doing a little bit of focusing next step is to rotate my stage to 180 degree after rotate rotation you will see that top part will come to the bottom the top trench part will come to the bottom now my features actually move so i will use stage movement now we can see it is below and now you can see that top range part actually basically comes to the bottom now i'll do a little bit of focusing again i have to tilt my sample to 22 degree yeah you it is eucentric is disturbed little bit then again we make eucentric little bit and bring our region of interest at the center yeah so once it's center we turn on we go to the high beam window and you can see now it is in blue condition below it means it is in light mode and same rectangle pattern we'll put at the top yeah same dimension application scan direction is top to bottom and we'll start the we'll start the trenching process

Now you can see in EV window that is now trenching at the top and already you can see the bottom is already trenched because now this is 180D rotated. Now the top part is now trenching. Same the high energy gallium ion removing material. and it takes the same time around one to two minutes so the time is actually basically depends on the current value so if you use some basically high current then it will take less time if you use low current then it will take more time so always if you want to do trenching you can use the high current

but one thing you have to check that in ibm column you don't unpause it at high current value now it's trenching It's 50, 40 seconds has slipped. It's removing my... another thing so you in always you in during trenching you isp is basically you keep you can see at below there is a isp time interval so after five second it is basically taking images one so always keep isp as low because you have to see the trenching procedure

So always you have to monitor that it should not cut our region of interest. This is the main thing. Now I am doing tilting 45 degree to check that it cuts perfectly from both the side. Yeah. So now I am using the beam shift.

So using the beam shift and bring my region of interest at the center and you can see that the sharp line is there so it cut from the both the side you can check at 52 degree as well so it cut perfectly now i will come back to a geo degree now you can see in even window that in both the side it cut perfectly so trenching part is done now next step is to cut at the left side so we are we basically in cut in left side to bring our manipulator for lift out sample so next i will do will draw some rectangle pattern at the left side and the dimension is around X is around 3 or 4 and Y should be a entire height of this thing

around 19 8 to 10 and JD's around 6 and it's that patterns would be from left to right scan direction towards the sample always so now it's started now you can see that that left part of our sample is basically removing so the main idea behind this so you will can insert the manipulator easily otherwise our manipulator actually touch so the main idea behind to this trenching this process to insert our manipulator now it's almost cut yeah you can see in IB moving as well by snapshot so you can take snapshot to check Always, always you have to check.

I have to take a snapshot after a time interval, three to four seconds, during cutting. And now I cut a little bit at the top part for safety purposes, so that it should not be attached below or anywhere. It can move easily, freely move. Now I change the current from 9.3 amps to 7 picoamperes. Now this is a very low current, and this current is fine. For unpausing my E-beam window, now insert the manipulator. Now you can see in the E-beam window our manipulator is there. That white color is basically our micro manipulator.

Now, in the E-beam window, we are basically aligning the XY. So, in the E-beam window, you can see the XY movement. You can align XY. So, I am using this XY movement to align my manipulator with our sample, and my manipulator and my sample should align perfectly. You can see in the E-beam window, it is aligned perfectly.

Now, I am going downwards using this jet. So basically, I am going down, and now once it's reached almost near to the sample. So it's best to insert the platinum needle. Now I am inserting the platinum needle to weld my sample with the manipulator again. Now once you come very near to the sample, you can decrease the step size. Now this step size is around 500 nanometers, you can see. And slowly, slowly you have to move, and once you see that once it touches the sample, at that time you have to stop it. So you can see in the E-beam window, it's almost touching.

And I-beam also, you can see it almost touches. Yeah, now I change the current value. I, yeah, sorry, first I'll draw some pattern, the same rectangle pattern. Now we will make a bond of platinum with our sample. Now I change the current from 7 picoamperes to 80 picoamperes. So basically, at 7 picoamperes, it's very difficult and will take a long time to weld, so that's why we increase the current a little bit. Now the Z we put around 2 microns, and XY is basically 0.8 and 2. So now the platinum deposition is going on. Now it's making a bond with this micro manipulator and our sample. So you have to take a snapshot in between to check that.

It's how bonding is happening, whether it's bonding or not, the welding. Yeah, bonding or welding, welding or not. Yeah, yeah, it's bonding, actually welding. The deposition is going. Yeah, it's nicely deposited, you can see. So once this the position done then we will cut from the left side then then we can easily remove our lamella from we will use high current for cutting purpose so same 9.3 and nano amps current i use for cut sample yeah same start patterning and it's cutting and see it will take few second to cut once you get cut you can see

yeah now i d uh change the current now you can see it cut nicely and i unpause the window ibm window yeah there is some it is attached and that's why I cut little bit yeah yeah now it's cut perfectly so sometimes you feel during when you try to lift out you feel it attached so then you have to cut this so now it's perfectly free and it will come out yeah it it's coming yes coming initially you have to make small step like 400 nanometer and after removing a three four times you have to do small step then you can increase the step size now now 10 micron after that you can retract the manipulator you can see it almost

Coming from sample, you can see that now we can retract our platinum GIS needle and also you can retract the micromanipulator. Now the one part is over, that lift out part. So same removing, removing time of the sample. So you have to press the same pump button, pump button. that pump button actually vent my chamber so it will take at least 30 second to one minute to vent the chamber once the chamber vented so we use this this rod

quick loader rod to go towards the at this stage and turn to the slip and go and move forward and again you have to turn right to lock the sample and it will come back again so now my sample actually Come back from the microscope chamber to this small venting box chamber, quick loader chamber. Now I press the venting button for vent this chamber. Open the lid. And here that this rod will turn towards the left to unlock sample.

Now I take sample from the microscope. So after removing sample from the microscope now we have to remove the sample from the holder. You just remove it by hand. You just apply some pressure, and it will come out from the holder. We have to remove the carbon tape.