Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

Prof. Surendra Kumar Makineni
Materials Science
IISc Bangalore

Week-08

Lecture-28

This is the propendous box where we actually keep our puck and coupon. So this that copper this holder thing this is called puck and this is a top is called silicon coupon and I will show you that how the silicon coupon looks like when I load in the holder. So first I will open this. So you can use this holder to remove the all this coupon with silicon post from these things like that only. Like that only we can remove these things.

When we load in atom probe machine we will remove like this only and we can place like this only. So in this holder we will place only this portion only this silicon post only not the puck. So puck we basically use when we load in atom probe but only we will remove this silicon post and we place it here. so we use this allen key to unscrew now i can hold this silicon coupon and place it our holder like this only now we use this screwdriver to tight to tight it with the holder this silicon coupon once you tight by this screwdriver

You use this flat tweezer to check that your silicon coupon should not move. It should stick tightly with the holder. Now the silicon coupon is already fixed with the holder. Now it's ready to load inside the microscope. Now same procedure we have to follow.

First bend the chamber. Open the lid. and now you have to carefully you have to place the sample you should not touch the black portion of the silicon coupon now i placed my holder with a silicon coupon inside this chamber now we have to lock it with our load lock rod so do always don't touch this black thing of the silicon coupon because all the silicon post is there in silicon coupon all the silicon all the 22 post is there if you touch it it will damage the 22 post so don't do not touch the black portion this is the black portion is basically a silicon wafer in that silicon wafer there is a 22 post is there and this all thing is called silicon coupon So don't touch that black portion. So you have to carefully when you load the sample. So you just slightly press it. And same process like whatever we did before.

Turn towards right and lock this. folder and then close the lid and press pump button which is basically creating venting inside the chamber we lift out our apt lamellas sample and the top right is basically same the ibm window now you can see the same thing at the napkin window is the bs image is showing now that now we will load our sample so now you can see that in top bottom right my sample is already inserted now i converted my napkin window from turning to a napkin window from electron window previously it was a basic window was there now i now i am doing actually napkin i am taking same napkin photo

for silicon coupon so you can see that silicon coupon in napkin window you can see the top view of the silicon coupon and in silicon coupon there is a 22 post is there in both the side in the right side is 11 post and left side also 11 post is there same thing we are doing we set stigmator and beam seat 0 0 and now we are actually turn on the electron beam window now we are doing auto brightness contrast little bit just to for clearly visible our silicon post now you can see that my silicon post is visible so this is so one side there is a this is a double fiducial side and the other side is single fiducial so i am basically i am now checking all the silicon post so first you have to check the how many post is available

so it depends upon your how many tips you want to prepare so now you can see this post is already used same thing i am doing focusing here and now i set my jet to 4 mm previously i mentioned the 4 mm is basically linear the 4 mm is basically eccentric for this microscope now yeah now basically we are aligning our post then you do little bit focusing again make it four Now the next step is same, the eccentric. So I tilt my sample 10 degree and same stage movement by adjusting the jet.

We are actually bringing our sample at the center by up movement. Again, I am doing same thing, 30 degree and as 52 degree as well. now my silicon post is perfectly eccentric we have to do little bit focusing at 52 degree and again come back to zero degree and you can see that it is my silicon post is almost at the same position from when i come to 52 to 0 degree so now i turn on the high end beam window and always when you use the low current so 7.7 pico amps now i am using uh so you can see in eb window you can see the top view of the silicon post but

in ibm window you can see the side view of the window so what i am doing now i am doing the scan rotation so you so a scan rotation then you can see uh straight uh in that silicon post if you without scan rotation it is like a reverse so it is for it is depend on the user convenient so i always use that scan rotation and make it straight uh that in iib mundo silicon post looks straight so same eb mundo also same little bit focusing i am doing Yeah, we are just auto brightness contest in IBM window. And using the beam set, we make it center.

Yeah, we are just this thing. Yeah, now I insert our micromanipulator. Yeah, you can see in I-beam window as well as E-beam window that micromanipulator actually already inserted. And now this XY movement, I actually in E-beam, we actually move the XY alignment. We'll do XY alignment at E-beam window.

Now I have to, I am actually aligning my APT lamella with silicon post. And in I-beam window, you can see that I'm bringing down my sample. Initially, step size is high. Now, I decrease the step size. Again, I align in EB window, XY alignment.

Now, my sample is perfectly aligned almost with silicon post. Now, I bring down my sample in the IB window and you can see my sample and the silicon post is almost at the center. at high magnification you have to see that it perfectly align and perfectly it at it that sample should touch at the center of the post then that bond will be a good so always try to paste sample as try to paste sample at the center of the post yeah so it almost touch so next i pause ib window and draw a rectangle pattern and i keep the pattern at the center of

my silicon post and sample and i change the current because uh now previously there is a very low current in no kind it will take a lot of time so i change current little bit so 80 pico ampere is fine so now the platinum is now depositing so it's now the bonding now it will make a bond between sample and silicon post Now, initially I deposited deposit little bit. Now I increase the pattern side and whole entire area actually recover with platinum. It takes around two to three minutes to cover entire area with this platinum.

Now you can see that platinum is covered in the area of an IBM window. and always you went to check in the window also once it fill it will come out from this outside so at that time you have to stop it sometimes it didn't come but by seeing that IV Mundo also you can confirm that it perfectly bond bonded happen or not that platinum deposition now I change current little bit because it taking too much time at 80 pico ampere that's why I change current little bit to 0.15 nano ampere

Now it's going on, the platinum deposition is going on. Now next platinum actually deposited nicely. That sample is perfectly welded with our silicon post. Now the next steps to cut, now I same rectangle pattern now. and I change current from 0.15 nano ampere to 2.5 nano ampere.

Now I start the cutting. So the dimension of the pattern is, Z is important. So whatever Z we actually give in that during cutting, during trenching time, you can put same Z like four and five. Now it cut almost. You have to check at low current.

So there is small, it not cut perfectly. You can see that there is a, it attached at the end. So we'll change the current because 2.5 nano ampere is very low current. So that's why it's not cutting. So I change current from 2.5 nano ampere to 9.3 nano.

Now it cuts perfectly. so it's clearly you can see clearly it cut so at we now we pasted uh one part of our sample one piece of our sample at one post now the next piece will actually paste the another silicon post now i removing my manipulator we little we remove from we use the x moment and the z moment now next step is we'll go to a different post yeah so another post is there you can see in ibm window the side view and the top view in ebm window So one thing you can you can notice that in IVM window actually we turn on the scan rotation.

That's why the whatever X moment we are doing, it is basically reverse. The right side arrow actually, if you press the right side arrow, it going the manipulator will go towards left. So due to this scan rotation, this thing is this thing's happening basically. So now my sample again, I touch almost the silicon post. Do change the current from 1.15 nanoampere.

Whatever we did last time for last sample testing. And now my platinum is depositing. Yeah. same it will take actually around two to three minutes for complete testing that aluminum sorry platinum so this part is very important you have to make a good bond with your sample and silicon post otherwise your deep will fracture when you run in the atom probe when you run in atom probe machine

atom professor instrument so that bonding is very very important so you it is it takes time so you have to wait till so perfect bonding is important that samples should perfectly weld or perfectly bond with the silicon post yeah it's taking time it's around one minute 10 seconds is left and you can see that platinum is depositing you can see that it's the platinum is deposit nicely now next step is we will cut sample uh we'll remove actually basically we'll remove our manipulator from sample so we'll keep jet around pipe and scan direction is top to bottom or bottom to top doesn't matter now and we'll cut

and see in ibm you can you can see and always take a snapshot yeah now it cut perfectly and we'll check at low current seven p coms yeah you can see that there is no attachment between sample and manipulator now it's moving also so now you can see that i am actually placing the left side button but it's going the right side that manipulator so because uh due to this that scan direction i uh turn on the scan direction scan rotation sorry scan direction of the scan rotation that's why it's the moment is this uh reverse now the next step is we want to rotate our state 180 degree now we will do pasting the opposite side

now one side we have to platinum pasting now we'll do the pasting opposite side this is the important step so to make a perfect bond with sample and the silicon post so to make a strong bond that's why we have to paste another side as well so you can see in ib window there is a lot of gap is there so that entire gap or will fill with this platinum same rectangle pattern will draw and cover this entire region with this with platinum so now i'm taking snapshot so jd is around two now it started now platinum is depositing around one minute is left still is going on so this part is i previously i mentioned that this part is important don't stop it because you need to make a good bond between your sample and the platinum so yeah in any moment you can see that platinum will come out from a sample at that time you can stop it you can top you can see that platinum is actually coming out in eb window you can see now the one tip is done one tip is perfectly welded in both the side with platinum now i'm move to next step and same thing i will do here also

I already started platinum deposition. Yeah. now increase the pattern size to cover entire area with platinum um you have to take snapshot in between uh after 10 to 15 seconds to check that how it depositing it is important step to taking all during trenching depositing you always try to take a snapshot in the ibm window and now i rotate my stage to 90 degree and now testing part is over

now my sample now is the next part is to thin down my sample so why now I want to make a sharp needle so I tilt my samples to 52 degree now my sample is actually basically tilting to 52 degree now you can see that Now the situation is reversed. Whatever you have seen in the e-beam window, now we can see same similar features in i-beam window. And whatever you have seen in i-beam window before, now it is similar in e-beam window.

At 52 degree, your sample is basically perpendicular to your i-beam. now I align my sample at the center basically I am aligning it now I draw some now I will do a circular milling so I put a circular pattern so next now there is a some small but protrusion is there so first we have to remove this so that's why I put rectangle pattern now so just removing this part so you can see that that's this is removing okay yeah now it's fine now can we can use same circular pattern and that application silicon and diamond outer diameter should be around 5 micron

inner diameter 1.5 micron z is 4 and scan direction is important it always from outer to inner and now i started okay start it and you can see in e beam window that material is basically removing yeah initially it will take around three to five minutes to removing a material now you can see that now my needle type shape is coming from top you can see in eb window that sap needle sip is coming once it coming then you can insert some measurement box now i already i inserted that measurement box and you can check the dimension now you can see that around height around 1.36 micron my tip diameter is around 854 now i can change

my dimension now you you always see you have to stop where you you can see there is no there is no thinning up to a certain height you can observe there is no thinning is happening so at that time you can stop it and change your uh dimension the basically inner dia now i change this inner dia now it is 0.8 now i change to 0.6 micron and i change the current as well now you can see that it's it's thinned down in eb mundo you can see initially it's 854 now it is around 700 that's around at uh 850 nanometer height is around the 500 around diameter now again we change current point point four three two point two three nanometers and same we always want to change because our aperture actually not aligned and

it will land for different current values so you have to always you have to adjust by beam shift so now i change from 0.6 to 0.4 in a day and it was you can see in EV window yeah you can see it's thinned down it's thinning radius is basically decreasing if diameter is decreasing yeah so it's at 600 nanometer is around less than 200 nanometer that now tip radius is so i'm focusing the focusing important to measure correctly now it's around 182 nanometer again i decrease my inner dia to one point 0.15 micron 150 nanometer now it says around 160 nanometer now it's 150 to 1 over 200 nanometer is good enough for laser mode to run laser mode in voltage mode you need more

less steep radius because you if the radius is very high then you have to apply very high pulse voltage so is always better to in laser mode it's 160 to 100 nanometer is fine it can run nicely in laser mode now I came same thing I move to the next tip same I draw a rectangle pattern now i started thinning same same whatever i did before outer dia around 5 micron in a dia 1.5 micron initially z is 4 micron and initially i use high current around 0.7 or 0.8 nano ampere now you can see the material same material is removing yeah now i changed my outer diode to 4.5 and I might change I change my current from 0.79 to 0.43 and inner diode 1 so stepwise you have to do like that initially 1.5 inner diode then 1 then 0.8 like that and as well as you have to change the current Now again, same thing. I draw a measurement that box. Now you can see my tip radius is 1.7 micron height.

It's around 1 micron around. So now I change again from 1 micron 2.8 and inner outer from 4.5 to 4 and I start. I started thinning. You can see. My tip radius is decreasing from both the sides.

You can see it's decreasing. So now I did a beam shift and bring my features to the center. And you can see at one micron height, it's around 782. Again, I changed from 0.8 to 0.6. And you can see.

both the side it's thinning so at 800 nanometer in e-beam you can see at 800 nanometer height uh it radius is around five both yeah around 550 you can say now i adjust i changed now i change current from 0.23 so once when he wants to change the current you have to adjust by you have to take a snapshot because your aperture is not aligned so your features actually will move and now again i started my sample is thinning so it's around the 450 nanometer now I change to 0.2 nanometer so you have to do stepwise from starting from 1.5 micron to 1.8 0.6 0.4 0.2 like that and

you have to change current from higher to low low current now you can see this it almost around 2 to 20 500 nanometer height now i change to 1.15 and scan direction is outer to inner now you can see my sample now it's decreasing is around the 250 to 15 it's around 200 at 500 nanometer So it depends upon the sample to sample. Some sample thin down very nicely, some sample it takes time to thin down. So it depends on the sample.

But in voltage mode, you have to prepare very subnatal. Otherwise, you have to apply very large voltage. Already my professor mentioned before. But in laser mode, you apply the thermal energy. laser pulsing so little bit high higher radius sample is okay so around 150 to 200 nanometer sample is fine now the next last step is to

this cleaning the whatever our sample basically we are using the gallium ion to thinning down so gallium actually deposit at the top surface so we want to gallium atom will come when we run in the atom probe tomography so we it basically it a good practice to remove at least one up to 50 to 100 nanometer height sample to remove this layer so basically now at low kv 2 kv and very low current to 27 pico ampere current we basically removing the material from the top so you can see the top 50 to 100 nanometer top layer actually basically removing the main purpose of this is to remove the gallium damage area now you can see we i did on the first step next i will do same thing i will do in the second tip also so you can see my tip

and same i did same i will do here also so i'll take auto brightness contrast in ib window take a snapshot and same i put a circular pattern at top of the whole of our sample now i'll actually basically removing this top layer So it will take around three to four minutes. So this is the this the total, the three state is there, one is called. the lift out you have to lift out stage where you have to from the site is basically you can prepare grain boundary when any interface

so first you have to locate your region of interest then put a platinum uh put a platinum deposit at that at the region of interest and then you trench it In both the side and lift out your APT lamella. This is the first part to lift out sample and next part is first is up to paste it at the silicon coupon the pasting is important and Next you have to pin down so I now the Entire process is done will prepare to atom probe needle and in this uh exp in this uh tutorial session in this uh in this tutorial so the you have to take care the few things uh when you will prepare the atom probe needle

first is to you have your sample should not be dripped because your region of interest should you you it should not removed during trenching or when you deposit platinum it should be there so that this part is important so so in atom probe now my sample is prepared both the tip we thin down to around 100 150 to 200 nanometer and uh So you have to now I will tell you some steps that are basically important what we have done in this session. So first we find out our region of interest from where actually we want to lift out and we want to prepare our atom probe lamellar.

So first you have to locate the region. Next step is you have to that region you have to deposit the platinum. after depositing the platinum you have to go to uh you have to do you centric to bring that your regional printers at the center at you centric position your both iron beam and e beam meet at the same point and your features will not move during tilting so once the eucentric is done you can go to a 22 degree tilting so because the trenching in atom probe trenching will do in at 22 degree in normal tm lamella we'll use we'll do trenching at 52 degree but in atom probe we'll do it 22 degree and once we tilt my sample to 22 degree then we'll go to

I beam window and turn on the beam once I turn on the beam so always use the low current initially so you use low current and then you increase the current value but once you increase the current I beam current don't unpause the window already I mentioned uh so in at high current we will actually trench the both the top part and the bottom part after trenching you have to tilt sample little bit to check that it cut perfectly from the both the side once it you can see in eb window that it cut perfectly from the both the side then you have you come back to again zero degree position and then you draw a rectangle pattern at the left side so the main purpose is to draw a rectangle pattern to insert our manipulator we can easily insert in that location

otherwise my manipulator will touch our sample and it will bend and maybe sometime it damage so the main purpose of this left side trenching is to we can easily insert our manipulator so once the manipulator inserted so in that manipulator uh option uh you can see that right side there is a slow many small uh option is there there so in manipulator option you can click and you adjust the manipulator by x y and exit in you can do x y movement in eb mundo you can do exit movement in ib mundo So, first you have to align your manipulator with your APT lamellar. so in eb window once you align the x x y alignment in eb then you turn on the ibm window and always you change current very low value so around 7 pico amps 10 becomes is nice and you can

unpause it in that low current so you unpause it and now you do x jet movement in ibm window so you use that same down j arrow so you basically you are bringing down the manipulator is going down and near to the sample when once it comes near to the sample insert platinum a needle or gas injection needle and then you decrease the step side and slowly you can move and once the our micro manipulator touch the sample at that time you draw a rectangle pattern jet size you put around 2 micron and you start patterning half before that you have to change the current value once you change the current value you have to take a snapshot and

so once you take a snapshot you see that your features will move little bit once when you change the current value because of this aperture all this it is uh this alignment is uh not uh because due to this alignment so you have to by beam shifting you have to bring your sample at the center once you bring and then you start patterning uh so it will take around the one one minute to make a good bond between your sample and the manipulator once you can see by taking snap by taking snapshot you once you see that yeah my manipulator is perfectly bonded with my sample then you cut the portion which is attached that tm lamella which is attached to the sample main sample the bulk sample

so then for the cutting purpose we will use very high current so around 9 nano amps current so at 9 nano ampere current you cut it from the right side and now you can see sometimes it attached so you can cut perfectly and you at use always initially when you will take lift lift out your sample or so you use the small step and check that sample is coming out or not if it is coming out then you can increase the step size so initially always that first you decrease the steps like first moment so once it is coming out then you you can perfectly lift out your sample now now the this part is over the lip lift out part is over the next step is to pasting my sample on the silicon coupon

So you have to carefully load the silicon coupon. I already mentioned before that you should not touch that black portion. Otherwise, it will damage all the 22 posts. So so during loading that holder, that quick loader holder inside microscope, that time you have to you have to be careful. uh and once you inserted the silicon coupon inside the microscope you take same nap cam photo

and once you take the nap cam photo you can see in nap cam window uh bottom left that you see your silicon post uh silicon coupon and in silicon coupon there is there are 22 post is there so you click double click it once you double click that your stage will move and your silicon coupon is basically aligned perfectly with electron beam now you can turn on the your electron beam and set this stigmator and beam shift zero zero this once you've done this and again you have to unpause the eb eb window and you have to do little bit of focusing so once the focusing is done then you have to jet link so jet link in a link is important so now basically in jet link

now you are telling your microscope so where my sample is now microscope now after know that your sample is around something like 9 mm from the pole piece so jet leak is important if you don't jet link you directly go to 4 degree then sometimes it touch the pole piece so that is step is important once you focus it first focusing it and do jettling so once you jettling once you tell that microscope now know that your initial your sample is 9 mm then you press uh 4 mm and go and go to the 4 mm distance because the around the 4 mm our microscope is in at your center so i have already mentioned

before what is what is 20 keys so you have to do at 4 mm you have to do perfectly focusing you have to correct the astigmatism after correct after focusing and correcting correcting astigmatism you tilt your sample to do eccentric so tilt sample and once you tilt you can attend degree you can see that your features or your silicon post basically move from the center so using the stage movement by jet up or down so you bring to the center and stepwise you to do 30 degree and the 52 degree as well so once the your eccentric is done you will come back to again 0 degree and once you come back to the zero degree now the you now you turn on the ivy window so always when you turn on the ivy window your current should be minimum so

at 7.7 pico ampere you turn on ivy window and unpause it so i use the scan rotation because the my silicon post looks reverse from uh so make it straight that's why i use the scan rotation uh so in this up scan rotation you use the shift F12 button in this FEI Helios microscope and make it 180 degree. So once I annotation on that, yeah, this thing done. once we align this silicon post uh when once we align our apt laminar with silicon post and we can go to the ibm window in the ibm window also same thing

we use the jet moment initially you can increase the step size should high the once it comes near to the at the post then you can decrease the step size and slowly you bring down your atom probe lamellae and now once it touch then you insert the platinum ladle the gas injection ladle and you draw a rectangle pattern and make a good bond between your sample and the silicon post once the one side bond is done then you go to the next post same thing you have to do and then once the both the thing is done you have to cut you have to remove manipulator from the sample and retract it and then you have to rotate the stage 180 degree

after 180 degree rotation you have to fill the platinum the back side also to make a strong bond between your sample and and silicon post once this this bonding is done in the both the sample then the my sample is ready for the thinning so for thinning we have to rotate the stage 90 degree once i rotate the 90 degree state rotation then we will go back to a 52 degree so in 52 degree we will draw a circular pattern if any non if the sample is not perfectly rectangle some something protrusion is there then you can use the some rectangle thing to remove this portion otherwise your tip some uh non-uniform your tip should not be uniform some protrusion something is there

that will actually uh that when you uh run the atom probe tip from this region also atom will coming so it's not so sample should uniform and then you put a circular uh pattern and initially you uh put outer dia around four to five micron and in a day around one one point two to one point five micron and current is initially you started from point five nano ampere to one nano ampere so once that sample your sample sample actually thin down and to one micron you can decrease the current from 0.5 to 0.3 nano ampere current and accordingly you have to decrease the current and you decrease the inner diameter from 1.5 to 1, then 0.8, 0.6, 0.2, like that only.

And always the monitoring that your tip radius is reducing. So once it comes down to around 150 nanometer at height around 500 to 600 nanometer, then your tip is fine. It can run nicely inside the atom probe tip. And the last stage is to remove 50 to 100 nanometer layer from the top. So we use very low kV, around 2 kV and 27 pA.

In IBIM window, we put a circular pattern and put inner diameter is 0, 0. And it's basically removing the top layer, this thing. So this is the main process. This is the process for the atom probe sample preparation. So it will take around three to four hours depends on the user expertise.

So, the more you prepare samples, the less time it will take. Initially, it took me around four to five hours, but now I can prepare a sample within two and a half hours. If you have any questions, feel free to comment below. I am happy to answer them. When we want to remove the sample, So, first of all, after venting is done, you use this folder to bring the sample from the microscope chamber to this small vent chamber. Now you can see the sample has already come into the vent chamber, and you slightly press the vent button to vent this small chamber. Open the lid.

This chamber, and now you have to hold it by hand so that it does not move too fast. Then, slightly turn the rod to the left to unlock it. Now it's already unlocked, and you can carefully remove the sample.