Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Week-10
Lecture-30

Hi everyone, I am Shashi there. I am one of the TAs for this NPTEL course on atom probe tomography. So, today we will be showing a live demonstration on how to load the samples in the atom probe and how to acquire the data from the atom probe. So, here we are seeing the LEAP 5000 XR, a relatively new generation of an atom probe. This XR represents the reflectron which

which the professor was explaining to you, which can increase our efficiency of So now I will show you the individual parts of the atom probe as a whole. So here, what we see is a load lock chamber. This is the first entry into the atom probe instrument. Here we have different carousels, which we will be seeing later on.

The next part is our buffer chamber. So initially, the samples which load into the load lock chamber, after they reach a required level of vacuum, we transfer them to our buffer. So this is the buffer chamber, where we transfer the samples from the load lock chamber, and this transfer is done through this transfer rod. And this transfer rod is also used to transfer our chambers from the buffer chamber; the puck is transferred to the analysis chamber. So we see four different camera views of the analysis chamber over here.

So this will be the typical puck which we transfer, and you can see this is the local electrode, and we place the puck just before the local electrode. and bring the tip into the view of the local electrode for doing the final acquisition, which we will be seeing later on. So this, as we see, is our analysis chamber, and now we see the most important part of the APT: the laser. This is the laser source which we are using for running our samples. Now I will show the loading of the puck into the carousel, which is taken from the load lock.

Now I have taken out the carousel from the load lock. Now I am removing this puck and loading it into the carousel. Now I will take this carousel and load it back into the load lock. Thank you. What we are seeing now is the loading of the puck into the analysis chamber from the cameras fitted inside the analysis chamber.

Now we are done with the loading of the samples into the APT. What we see from now is the acquisition part of the data. Now, before acquiring the data, we have to align the samples with the local electrode. What we see in the right bottom window is the local electrode. You can see a cone-shaped object. This is the local electrode.

And what you see in the left top window is my puck. As you can see, I am aligning the center of this puck with the center of the cone-shaped object in the left top window. Now, you can see in the left bottom window that I will try to bring this puck closer and closer to this local electrode. Once I see something in the field of view of the local electrode, in the right top window, you can see some bright spots, patterned bright spots in this right top window, which are nothing but our tips.

So now, I will try to bring these sharp and bright spots before the local electrode. Now, I will try to move this puck slightly upward. And then bring these sharpened lines right in front of the cone-shaped local electrode. You can see I am doing that. So, what we are using right now are pre-sharpened silicon tips, which are usually used for alignment purposes.

So, all the acquisitions we do today are on these pre-sharpened silicon tips. You can see that I am moving the puck such that finally, I will bring these bright lines right in front of the local area. So, once this is done, you can see some tips have come into the field of view of the local electrode in the right bottom window. So now, I will align this tip right in front of the local electrode. I will center it and then try to focus these tips.

So, focusing is nothing but bringing this tip into the sharpest possible contrast using the fine adjustments. You can see that I am making some movements to center the tip. And then I will use plus and minus adjustments—nothing but focusing—to bring the tip to the sharpest contrast. So once these basic steps of aligning my tip are done—since we are done with the alignment of the APT tip with the local electrode—now we will focus on

the acquisition part. What we see—what we are seeing on our screen—is the main acquisition control window of APT, as the professor was mentioning throughout his course.

There are two basic modes of acquisition in APT: one is laser mode, and the other is voltage mode. As of today, we'll just focus on the voltage mode of acquisition, so I'll move my window. You can see that I have kept two windows on my screen. So, the left window is the AP suit window, which shows live data as it is being acquired. And the right window is the main acquisition control window. You can see I have changed the mode of acquisition to voltage.

And you can see just below that various parameters of voltage mode. Specimen temperature, specimen voltage, reduction rate, pulse fraction, pulse rate. So, initially, to get a feel of what all these windows show as we acquire, let us acquire some data. So, I will start the acquisition and increase the specimen voltage. So, as we have studied in this course,

For the atoms to get evaporated, they need a certain voltage for field evaporation, depending on the tip radius, the nature of the atom, etc. So now, I am increasing the voltage of the specimen until the point where I can achieve some field evaporation. So now, towards the left, you can see some purple dots popping up. So, this Circular maps are called, and are nothing but detector event maps.

Whenever some atoms hit the detectors, these purple dots pop up. And towards the bottom, what you see is counts versus mass-to-charge state ratio. This is a mass spectrum. So, I am increasing my voltage and waiting for the atoms to get detected. You can see now that a peak has appeared in the mass spectrum, as well as a bunch of purple dots.

This means some atoms are getting field-evaporated and detected on the detector. So now, since we have a field of acquisition, what we will try to do is increase the speed of our acquisition. I will try to change the Detection rate to 0.5. Okay. Before that, since I know these tips are silicon, I have zoomed in the mass spectrum to around 14 and 28, as this is where the silicon peaks are expected.

So, you can see that near 14, you can see some peaks appearing, which indicates that silicon atoms are getting field-evaporated and detected. So now, to slightly make our process faster, let me increase the detection rate to 0.5. Okay. So, right now we are running the samples in voltage mode. Now, let us try to understand the effect of pulse fraction on the voltage data acquired in voltage mode.

So, right now we are using a pulse fraction of 20 percent. Okay. Let some data get acquired, and we will see. We can see the peak again appearing at 14. I have reset the detector event map as well as the mass spectrum.

So, this data was collected already at 20% pulse fraction. Now, you can see just beside the equation parameters, there is a blue-colored window in which you can see single events, multiple events, and background. So, the typical background we are getting at 20% pulse fraction is around 7 ppm per nanosecond, which is quite high. Single events acquired are 90 to 91 percent, and multiple events are 8.2 percent. Okay.

Just remember these values for the purpose of comparison. As we increase the pulse fraction, we will see what happens. So, now, I am stopping the equation. Now, I will try to slightly increase the pulse fraction to 40 percent, and we will see what happens.

Okay, and I am keeping all the other parameters constant. I have just increased the pulse fraction from 20% to 40%. Again, you can see the atoms popping up on the detector. You can see these detector events increasing, as well as the peak also popping up in the mass spectrum. Now, if you observe, there is a significant reduction in this background.

Earlier, in the case of a 20% pulse fraction, we could see around 6 to 7 ppm per nanosecond of background. Now, you can see 2 ppm per nanosecond of background. So, there is a significant decrease in background, which means our signal improves with an increase in pulse fraction. Now, you can see the single events as well, have slightly increased.

Earlier, it was around 90 to 91 percent. Now it has increased slightly by 1 or 2 percent. And multiple events, which were around 8 percent, have slightly decreased. So, this means our data has slightly improved. So, as we change the

pulse fraction, we could see that the background has decreased, and slightly our single events have increased while multiple events have decreased. And you can see. Okay, let us stop the acquisition here, and now let us try to change the pulse fraction to 60 and see what happens. Okay, I am starting the acquisition again at a 60 pulse fraction with all the other parameters constant. Okay, you can see the atoms getting popped up on the detector, and the peak also appears at 14 in the mass spectrum. Okay, now you can see

As I increase the pulse fraction further to 60%, you can see the background has further reduced to 2 ppm per nanosecond. And the single events and multiple events have also slightly improved compared to those at 20%. You can see the peaks are also getting clearly resolved in this case. So, more or less, we have understood the effect of pulse fraction. So, as we increase the pulse fraction, we are seeing a better signal.

We are seeing the background is getting lesser, which means our data is improving. Multiple events are getting fewer. What are multiple events? Okay, let us stop this acquisition. And further increase the pulse fraction to 80 percent. Okay, now I have increased the pulse fraction to 80 percent.

So the effect is more or less similar to what we have seen earlier. The background has reduced and The multiple events have reduced, and single events have increased, which means as we increase the pulse fraction in the voltage mode, our signal gets better and better. So, we have briefly seen the effect of pulse fraction on the data acquired in the voltage mode. So, as I am today, We can end the acquisition, and in the next video, we will try to focus on the acquisition in the laser beam.