

**Advanced Material Characterization by Atom Probe Tomography and
Electron Microscopy
Prof. Surendra Kumar Makineni
Materials Science
IISc Bangalore
Week-10
Lecture 31**

So, in this session, we will focus on the acquisition of epigree data in laser mode. So, initially, we do the same step of focusing the sharp tip in front of the local electrode. Then, an additional step in comparison to voltage motors, we turn on the laser beam and try to align the tip with the laser also, so that the laser falls on the tip of our needle. So, you can see I have turned on the laser, and I am moving the tip such that the laser gets aligned on the tip of the needle. you can see as I move, the laser is getting aligned on the tip of the needle. Now, once this alignment part is done, we can proceed with the acquisition.

Now, you can see the acquisition mode is laser, and similar to voltage, we have various acquisition parameters in laser mode as well. So, today also, as we have done for voltage, we will try to change various parameters and try to see what the effects of those parameters are on the data acquisition. In laser mode, initially before we collect the data, we have to align the tip with respect to the laser. So, for that Once we start the acquisition, you can see I have started increasing the voltage.

So, this is similar to what we have done in voltage mode. So, we just see when we can get a field of operation of atoms, and we see the detections. So now I can see a peak appearing in the mass vector as well as atoms popping up on my detector events. Okay, now I have changed the direction rate to phi. So now, in laser mode, we have to do laser scanning of the tip to identify the peak position as well as z. So initially, we'll scan for the peak position.

Maybe we can slightly increase the voltage so that we get some detection. And we can clearly see the peak as well as detector events popping up. Now I have started the laser scanning. So the laser will scan for the peak of the or tip of the needle. So you can see that it's scanning.

In the equation control window, you can see some scanning is going on to identify the peak. So, once this scanning is done, then we also scan for the Z. So, in this scanning, we have various options, Scout, Standard and Wide Standard. So, depending on how much area we choose. So, right now you can see we are scanning across a 10 micron cross 10 micron area in Scout scan. So there are various kinds of scans as well.

So depending on the appropriate alignment of your laser with respect to the tip you can choose whatever mode of scanning you want. So initially it's a good practice to initially scan with scout mode which is like very which has a higher field of view and then go to the decrease the step of scan like decrease the range of scan once the peak is identified in this mode then you can go for it so you can see that the detector events are appearing on the window So usually these scans are done at higher detection rate so that they can be done faster and significant signal also reaches. So now we can see that the peak got identified.

The laser detected the peak of the tip. And now we can scan for the depth as well, Z as well. Okay, as I have told you earlier, so after the long field of view scan, then you can see I have changed the scan from scout to standard. Then the size of the scan, field of view of scan has changed to 1.5 micron from 10 micron. So, once you find a peak in scout mode, then you can shift to standard mode.

So, it is in some sense like a high resolution scan. Here also the peak got identified. Now, I will scan for the Z depth. So, basically through this scanning process, we are defining the X, Y and Z of the needle, tip of the needle with respect to laser we are giving the feeding the information of where is the tip with exact X, Y and Z of the tip of the needle.

So, the laser is now scanning for the scanning across Z. So laser scanning is in laser mode the scanning is takes quite a bit of time. But as discussed by professor in all his courses that laser mode is in sort of universal mode which can be applied for range of materials for ceramics, intermetallics, semiconductors, insulators, any material it can be applied. But whereas

voltage mode can be applied only for that cannot be applied for a range of metals it can be applied for few metal metals as and the two metal materials also have less yield in

voltage mode yeah so the zero also got so you obtain this Gaussian kind of profile So, the laser has identified the peaks X, Y and Z now. Now, I will change the detection. So, similar to the experiments we have done in voltage mode, now we will try to change systematically parameters and try to see their effect on the data equation.

So, now to start with M, I want to change the detection rate. So, I will start with a detection rate of 0.25. So, a pulse energy of 50 picojoules is what I am using. These will all vary later on. Keeping everything else constant, I will vary the detection rate initially.

So, initially, we will start with a detection rate of 0.25. So, now you can clearly see. So, the ions are detected, atoms are popping up on the editor event map, and also getting popped up in the mass vector. And again, we will make a note of the single events, multiple events, as well as background values. For this condition.

Just for comparing how this varies as we change our parameters. So, I have zoomed in the 14 and 28 region for our convenience, since I already know that these are silicon tips. So, you can see that single events are around 88. Multiple events are around 5.8. Background is around 3 BPM per nanosecond.

Like 4 to 5 BPM per nanosecond. So now let us see what happens if I change the detection rate. So I would like to change the detection rate from 0.25 to 0.5 and see what will its effect be on the data applied. One clear difference you can see is, so when we ran the tip in voltage mode, we could not see the predominant 28 peaks at nearby 28 of mass to charge state ratio. But whereas in laser mode, we are seeing predominant peaks at 28 of mass to charge state ratio.

So, the charge to charge state ratio also depends on the kind of field of operation we are achieving, we are employing. So, now we will change the detection rate from 0.25 to 0.5 and see what will its effect be on the data coin. And we can make a note of these values of single events, multiple events and background before we do that. As you can see that it's the time of equation but same number of ions changes with the reduction rate. So, we have

So, now I am sending the detection rate to 0.5 and we will see what will be its effect. So, you can see. So, I am resetting the mass pattern and also the defector event map. You can see that the single events, multiple events has decreased which means our data got improved. Single events slightly increased and background slightly decreased.

Similarly, we will increase the detection rate further to 0.75. We can see that similar observation that at 0.5 we have slightly improved our background and also multiple events. So we are just waiting for correcting significant amount of ion atoms at each parameter so that we can the values can stabilize and we can compare better. So, once this detection rate comparison is done, then we will try to change the pulse energy also and see the effect of pulse energy on the data as well. While the data gets acquired, we can see other options which we have owned while acquiring.

So, we have kept auto laser scanning on which means as the tip radius changes and the field atoms get field evaporated, so the position of the tip also changes. So, this laser will be scanning in regular intervals to identify the to re-identify the tip position and align itself on the tip position so that's the function of auto laser scanning As we have mentioned already earlier that an interesting observation compared to voltage mode in laser mode we see the different like in voltage mode we have seen the prominent protein mass to charge state ratio peak while in laser mode we have seen prominent mass to charge state ratio peak at 28 which means the

28 represents the plus 1 state as while charge state while putting represents plus 2 charge state. So the kind of field wave operation also depends on the kind of stimulus we are giving for field wave operation. So now I would like to change the detection rate from 0.5 to 0.75 and acquire the data again. So I'll change the detection rate and reset the mass spectrum as well as as well as the treatment map.

You can see that a slight improvement in single events, multiple events and the decrease in background. So more or less we can conclude that detection rate has a positive effect on background. So background gets improved slightly as we increase detection rate. So, once we complete the acquisition of this at 0.75, then we will put the detection rate constant and change the pulse energy and see the effect of pulse energy on the acquired

data. You can clearly see that the background has decreased and multiple events have also decreased with the increase in detection rate.

So, after this let us change back the detection rate to 0.5 and at 0.5 detection rate let us change the pulse energy and see what will be the effect of pulse energy on the data acquired. Usually pulse energy represents the amount of in some sense it represents amount of hitting the tip receives. So, it will be very interesting to see how the Thermal tail which professor was teaching in few last few lectures will be affected with the pulse energy. Now as I said I am changing back the reduction rate to 0.5.

and i'll try to increase the pulse energy from 50 picojoules to 75 picojoules and let's see what effect does it show just remember the nature of this peaks at 50 picojoule And let me reset the mass spectrum as well as detector event map. So this feature which you are seeing, which is fourfold, which you are seeing on the detector event map is nothing but called as pole, which we were seeing throughout this course, that is pole. So now we can see that the width of the peak has slightly increased. We can see clearly a thermal tail of the peak.

So, as we increase the pulse energy, the thermal tail increased. We can see that the peak has become wider. And we can see that multiple events has increased. So, as we increase the pulse energy, the thermal tail of the peaks increase as well as the multiple events has increased. So, as a next step, we will try to increase the pulse energy further to 100 picajoule and see whether we see the same effect or not.

So, I am changing the pulse energy from 75 picosecond to 100 picosecond. Now, I will reset the digital event map as well as mass spectrum. Once we acquire a certain amount of data, let's compare the parameters. So we can see that when we put the auto laser scanning on, simultaneously as the position of the tip changes, the laser auto scans for the tip. So, now we can clearly see that the thermal tail of the peak has increased further.

So, it is the increase in pulse energy where width of the peak increases because of the thermal tail. This conclusion we can draw. So once if you try to conclude the make a note of all the conclusions we have made through this acquisitions. So initially we started with

the demonstration of APT, its basic parts, loading, sample loading and then we started with the acquisition part. In the voltage mode we have

seen the variation of pulse fraction and its effects on the effect on the acquired data and then we have seen how to scan the how to align the tip in the laser mode and then how to acquire the laser mode data in laser mode and then what's the effect of detection rate and pulse energy individually on the data So, more or less, with this, we just wanted to give a brief feel of how the sample loading, as well as the acquisition of APT, is done.