

Microsensors, Implantable Devices and Rodent Surgeries for Biomedical Applications

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Hello all, welcome to this lab class. Here we will look at one more physical preparation technique. In the last class we saw the E Beam operation, in this class we will see sputtering. What does sputtering do? If you recall our theory class, sputtering is a technique that is a mechanical way of depositing the material. That means you are dislodging the atoms from the source and depositing them onto the substrate. Here you are not doing the heating. So, there is no flow of current through the source.

Here we are using argon to create the plasma and the advantage of sputtering you already know that over thermal and E Beam operation, the sputtering has the advantage that it has better step coverage. So, let us see how the sputtering system works, and what it looks like, we will show you again the entire process it is which will be close to half an hour. Again we will see what the system looks like and how you can create a vacuum and you can see the system from within. In the lab I want to show, you can see the substrate holder, you will see the plate instead of having a source and this plate is on the magnets.

So, we call it a magnetron sputtering. Also in the sputtering system that you will see there are 2 kinds of ways we can sputter one is called direct sputtering or DC sputtering. DC is not called direct sputtering and the second one is RF sputtering. Now, the use of this magnet is what we call a magnetron, it is to improve the rate of deposition. If you use a magnetron along with the so, if there is a magnetic field along with the electric field the rate of deposition of the material would improve that is the reason for having magnetron sputtering we have DC magnetron sputtering and RF magnetron sputtering. You will see this in the lab component.

Welcome back to another lab session of this course. Today we will be talking about another PVD technique which is sputtering. In the previous session, we saw one of the PVD techniques which is thermal and e-beam evaporation. Now, if you already have a PVD technique why do we need another tool in the same lab? So, the difference lies in your requirements.

First thing there are certain tradeoffs between both tools. So, for example, in e-beam what essentially you do is you melt off material and after melting you deposit the vapours, naturally they flow and stick to the surface. So, what happens is you have to melt the material completely which goes well with certain material metals like aluminium and gold which have comparatively

lower melting points. Also when you are melting a material you need to contain it in some crucible and that crucible should not melt off along with it. So, when you are talking about materials like aluminium, gold and titanium you can place them in a boat of tungsten, tungsten has a very high melting point and that is the reason it is used as a filament in certain cases.

So, but what if you want to deposit tungsten, a material like tungsten which has a high melting point it is practically impossible. So, in that case, we move to sputtering. I will explain the process in a few minutes. Another reason that we might want to shift to sputtering is the stoichiometric ratio. Suppose if you are not depositing up your metal you need an alloy and let us say it has a 30-70 composition of A and B.

When you are melting it off like using an E beam or resistive heating it might happen that component A might have a different melting point than component B. So, although you have a source that has a 30-70 composition the rate of melting will vary depending on the melting point and your sample. When you have the thin film you might not have a 30-70 composition it might have something like 10-90 or 50-50, you do not know. So, in that case also we will move to sputtering. The third trade off is when you are depositing using a thermal technique the final layer the film layer that you get is quite smooth. It might sound good, but in certain cases, you might not want a smooth layer.

Maybe your device or your requirement demands that you have some roughness maybe in nanometers. In that case, also sputtering will be the go-to choice because the final layer that you have after sputtering is not smooth. So, let us talk about what exactly happens in sputtering. The main thing that we have in sputtering is plasma. So, what happens is usually we are using an inert gas like argon which will not react with your sample or your source that is why we use argon.

So, what happens is when you have a pump in argon gas between a very strong electric field the bottom will be negatively charged and the top will be positively charged. So, what happens is due to a very very strong electric field the argon splits the energy into a positive argon ion and an electron. Now, what happens is this positively charged argon ion will rush towards the negatively charged electrode and in the meanwhile while travelling towards this electrode it will encounter other argon ions and it will collide. Due to this physical collision, some more argon atoms might split into electrons and argon ions. This sort of continues like a chain reaction and ultimately you have have steady state where you have sustained a plasma.

The bunch of argon ions moving towards this electrode will create what is called as plasma and the indication is usually it will be some purplish glow that you will see near the electrode. Now that we know how to create an argon plasma, what is the use of it? So, what happens is your source the desired material that you want to code will be placed on the negative plate, negative

electrode. So, when the argon ions are rushing towards this source material they will directly hit and impinge, they will impinge on the source material and dislodge some molecules of it. When you do this multiple times for a long time, when such a thing happens these dislodged particles due to the kinetic energy move upwards and on the bottom positive electrode you will have your sample where you want the coating to happen.

So, this is the basic principle: you pump an argon gas and apply a very strong electric field to create a sustainable plasma and this plasma will dislodge the particles from your source and these dislodge molecules will go and stick to your substrate. So, now the next thing would be seeing the actual demonstration of this tool. For now, I have a source material sample source material which is called a target which we will be using in this tool. So, this is a 3-inch copper target and will be used when we need a thin film of copper. And in that, while we are operating the tool I will show you this closely and you will appreciate that the part where the actual plasma hits is a little shiny compared to the peripherals.

Now that we know the working principle of the sputtering tool let us operate it and have a small demonstration. So, firstly I will switch on the main supply before that this is the sputtering tool as you can see ATS 500 which is manufactured by an Indian manufacturer H H V. So, we will start the main supply and carry out the deposition. Now that I have turned on the main supply and also the supply to the chiller unit which you can see there is one inlet and outlet which is connected to the water jacket around this chamber. This is the chamber where the vacuum will be created and a plasma will be created later on once the vacuum has reached.

So, it is obvious that when such a reaction is happening inside when the plasma is created it is found that there will be some increase in the temperature. Also, there is a heater unit attached to this in case you want to do the sputtering at higher temperatures. So, to avoid any mishaps like a person or the user accidentally touching this chamber and getting injured because of the heat we have this water jacket which constantly cools down the chamber from the outside. So, let us switch on their tool and see what options we have. So now we have turned on the tool and this is the screen you will see when it is when it has completely booted up.

So, you see this is the main menu and you have certain options of which normally you would be using only three or four. So, the important ones are manual, system control, source control and there is a system view if you want to see. So, let us see what we have in system control. As you can see this is the system control and here we have certain values on the screen the backing pressure and the roughing pressure. Right now the pressure is a little high as the vacuum is really bad it is 10 to the power 0 and the system is on standby.

First, to start the process we will have to take the system from this pressure to 10 to the power minus 6 which is a very high vacuum. But before that, we will have to vent the chamber, take out

the substrate holder, mount a sample and then we will start the process. So, right now you see only two options are highlighted: start and vent. So, what 'start' will do is it will start both the pumps. I will come to that in a minute about what kind of pumps we have. Then we have this option vent, vent will just let the atmospheric air into the chamber so that we can open the door and take a look at the chamber.

Other non-highlighted options are stop process scene and cycle which we will see in a minute. Then we have some system view and source control which we will see in a minute. For now, let us vent the chamber and take out the substrate holder. I will click on the vent and now you might hear a hissing sound. It means that the atmospheric air is entering the chamber and it is about to reach the atmospheric pressure. So, now the chamber is completely vented what I will do and you can see that the pressure is 10 to the power 10 to the power plus 3 which is the atmospheric pressure.

Now once this is completely done I will seal this chamber and open the chamber door before that I will start the pumps. So, that they are in running condition and we will save some time and you might be able to hear the sound of the roughing pump. I will show you in a minute what all the pumps we have. So, now since the chamber is completely vented we will open the door and see what is inside. So, now you can see what we have inside this chamber are the pumps, are the electrode setups, the first one is for RF sputtering, the second one on the right is for DC sputtering, here we have the shutters that ensure that you have a sharp control over the thickness. So, when you want the deposition to happen you remove the shutter and when you do not want the deposition the shutter will cover this target. In this electrode setup there is an adjustment for the height as well as the angle and here inside this we have the target.

For example, I showed you a copper target a few minutes before that is placed inside this and there is this shutter that covers the target then we have here the substrate holder which keeps on rotating during the deposition to ensure uniformity. Above that we have two heaters in case you want the sputtering to happen at a higher temperature we will switch on the heaters. So, that is it and we can now start with the process. So, before starting the process the most important thing is to fix the sample on the substrate holder. So, I will take out the substrate holder or the chuck carefully making sure that I do not hit any other components.

So, this is a tenon chuck that rotates during the deposition and we will be fixing a glass slide on this chuck, with the help of a Kapton tape and will be depositing tungsten. So, as you can see I have fixed a glass slide on this chuck. I will be placing it inside the chamber and starting the vacuuming process which is also known as the cycling process. Again we have to be careful that while placing the substrate holder, we do not touch our samples with our hand or hit any of the components with the substrate holder. When it is done we will close the door. now that I have placed my sample attached on a sample holder inside the chamber.

So, before starting the process the most important step is to reach the desired vacuum level, a very high vacuum of the order of 10^{-6} millibars. So, after placing the samples in the chamber I will start the evacuation process or the cycling process as we call it in this tool. So, I will click on the cycle and allow the vacuum valve to close. So, what will happen initially is the roughing pump will come into action and reduce the pressure from the atmospheric pressure that is 10^3 millibars to a high vacuum of 10^{-3} millibars. Once that is done the next action would be of the turbo molecular pump working in series with the rotary power rotary or the roughing pump to reach a very high vacuum of the order of 10^{-6} millibars.

So, we will wait for now till the pressure reaches 10^{-6} millibars and then we can start with our process. While we are waiting for the vacuum to reach the desired level let us have a look at the various controls this machine offers. So, at the bottom, you can see that the DC power source is the source mains for the DC power source and various controls for setting and reading voltage current and power. The next thing we have is the RF power source. This display shows various parameters that we have while using the RF power source. Next is the DTM, the digital thickness monitor which is coupled to the quartz crystal oscillator which we have seen inside the chamber and it works on the principle of frequency shift when more and more mass is loaded on the crystal.

Here we can see the temperature monitor. So, it is right now set to 300, but since the heater is not on as we are not doing the process the heater is right now off and the chamber is right now at 27 degrees Celsius. Next, we can see there is the power and reset button and the emergency off button. The main control that we have is through this HMI or the display panel. So, right now you can see it is showing the process which is going now is fine pumping and we have certain options and the pressure is indicated.

Let us have a closer look at what other options we have. So, this is the display panel as we said and you can see the pressures here. Right now the chamber pressure is 8×10^{-6} milliwatts and while the fine pumping is going on the options that you have right now are seal, process and vent. The vent will evacuate, the vent will let the atmospheric pressure rush in and the chamber will come back to atmospheric pressure. So, next is the seal which will seal the pumps from the chamber when you are opening it and this is the process that will be going into this option once we reach the desired pressure.

So, meanwhile, let us have a look at the main menu. The main menu has various options of which what is important to us is the alarms, the system view and the system and source control. So, let us go to the system view and see a schematic of this tool. As you can see here some pressure sensors are displaying the values. this is the chamber which has 2 magnetrons,

SH1 and SH2 are the shutters.

This is for RF's RF and this is for DC. We have various pressure sensors and valves connected to it. The most important thing that I wanted to show you is these 2 pumps. You can see this is the roughing pump or the rotary pump and this is the turbo molecular pump. So, what happens is when we start the cycling process or the evacuation process these 2 valves will be closed and the entire pressure will be reduced from atmospheric pressure that is 10 to the power 3 millibars to 10 to the power minus 3 millibars through this line using the roughing pump. Once you reach a high vacuum such as 10 to the power minus 3 millibars these 2 valves will open and the sequence would be that this chamber will be connected to the turbo molecular pump and then it goes to the roughing pump.

So, what happens is this turbo molecular pump will take the pressure from 10 to the power minus 3 to a very high vacuum that is 10 to the power minus 6 and then. So, these 2 pumps will act and work in a cascading method. Now, let us go back to the system control. Now, we will go to the source control and see what options we have. So, here you can see this is the menu for RF power.

If you want to use RF sputtering you will enable the RF power and then set whatever parameters you want the power and you can see the reflected and forward power here, we have options for letting 2 gases one is argon and another is some other auxiliary gas you want. You will enable it to enable which mass flow controller or MFC we want and monitor set the flow rate and monitor them here. Next, we have the DC power that will enable it and here we have the shutters for 2 magnetrons. On the left we have RF, on the right we have magnetron 2 which is the DC and here is the option for DTM. If we enable it you can see it starts displaying values.

Next, you can see there is an accessory control where we have this rotary that is the rotating chuck or the sample holder and we have an option to enable the heater. Like I said in the DTM it was showing that the current temperature is 27 and we can set it to 300. We can choose any value from between the room temperature to a maximum of 300 and enable the heater. So, it will take some time and finally, you will reach the desired processing temperature. Now let us go back to this source control and here we have the system control.

Now you can see the pressure has reached somewhat around 7.7 into 10 to the power minus 6 millibars. So, we will wait for some more time and then we can start the process. So, the desired pressure has been reached and we can now start with the deposition process. So, of all right now we are in this system control and we will be choosing this process option.

Now it goes to throttle pumping and the process sequence has been enabled. Now we can go to the source control and now like we said we have certain options like RF power, DC power and

the mass flow controller gas valves. So, since we are depositing tungsten today we will be using DC power. So, I will just enable the DC power.

You can see these panels have lit up. Next, I will also be enabling the gas valve. I will next open the valve for argon gas. I will now enable the mass flow controller for argon and right now the set values for voltage and current are 0. So, the parameters that we have optimized are 600 volts and around 0.

0.5 amperes of current which will roughly give us a power of around 280 to 300. So, I will slowly increase the voltage. You might notice that the power is not increasing and we are setting the voltage reading for the power and setting the current. So, unless and until as long as the voltage I give some current there will not be any power. So, we are now at 600 volts and slowly I will be increasing the current as well.

Meanwhile, I will also enable the gas flow. So, I will be setting this argon MFC to 12 SCCM, but in steps. First I will keep it at 5, let it reach 5 and slowly I will also increase the current. Now it has stabilized at 5 SCCM, I will be increasing it to 12. I will also be monitoring the plasma through the viewport. So, the power was set at 300 watts and still, the power is not showing.

That means that there is no plasma right now and that is why you cannot see any power being delivered. I will just enable the shutter for a moment to see if there is any plasma strike. Not yet, I will be increasing the argon flow. So, now the plasma is sustaining as I can see in the viewport. We have a very bright blue plasma and the parameters that we have right now are 600 volts around 280 watts at a current of around 0.

53 amperes. So, now we can start with the deposition process. I will just enable the DTM, the digital thickness monitor and reset it by stopping and starting. So, now it is reset. I will be opening the Magnetron 2 shutter which is for the DC sputtering and noting the time. We will be depositing for around 10 minutes and the thickness we will be having is to be seen.

The parameters are such that for 5 minutes it will give a thickness of around 200 nanometers. So, now I will just disable that shutter. So, what will happen is the shutter will cover my magnetron. So, whatever deposition is happening it will be on the shutter and it will not be on my sample.

I have disabled it and I will also disable the DTM now. Slowly I will reduce the current. So, the step sequence was first we set up the voltage and then we slowly increased the current. We will go in a reverse order. We will first start reducing the current and also the argon flow.

So, you will see as I am reducing the current the power also reduces. As we know power is the

function of voltage and current. So, that makes sense as we are reducing the current the power will also go down and we have an extinguished plasma. So, now it is 0. I will also reduce the gas flow first to 5 SCCM and also start reducing the voltage.

Now, I will reduce the gas flow even further making it 0. Once it is stabilized at 0, I will turn off this mass flow controller and the gas valve. I will also now disable this DC power source and now only the vacuuming is going on. So, what I will do I will also I forgot to mention we also had the rotary drive in motion at 10 rpm. I will disable the rotation and go to system control and now you can see the pumping is still going on. So, now what I will do is seal my chamber and put it on cycle for a few minutes.

So, whatever residual gases which were there, it will be taken out. Now, we will allow it to cycle for a couple of minutes and then we will come back and vent our chamber and take out the sample. So, now we have allowed the chamber to go for a cycle for a couple of minutes. Now, the next thing would be to take out the sample sample from the chamber which is now right now in a vacuum-evacuated condition.

So, the next thing would be to vent it out. So, I will just click on the vent and as the venting starts you might be able to hear a small hissing sound which indicates that the atmospheric air is entering the chamber. So, once the venting is complete we will open the chamber, take out the sample holder and we will be showing you the deposited sample. Now, the venting is complete and the chamber is at atmospheric pressure. So, it is safe to open and take out the samples. Now, you can see there is a mirror-like finish on the sample that we have the glass slides which we had initially kept and this is due to a thin 200 nanometer coating of tungsten.

So, we will just take it out and put the chamber back in vacuum conditions because the longer it is kept in atmospheric condition the more the chamber keeps on degrading. So, it is a good practice to always keep it in an evacuated condition. So, what you have seen in this particular lab is sputtering right? Now, sputtering can be DC sputtering and sputtering can be RF sputtering. We have a system that has both DC sputtering and RF sputtering which you have seen.

Additionally, you can always bring the source to the substrate distance closer and also you need to understand that the reason for having a vacuum in all this physical vapour deposition is to have a better mean-free path. Mean free path as you may know is a path before which how much the atoms will travel before colliding with another atom. So, how much if the distance is less the number of collisions the better the deposition. The mean free path can be improved by having a vacuum of high value, for example, 10^{-6} to 10^{-7} and so on the better the mean free path is. Going back to the sputtering, we can do magnetron sputtering and non magnetron sputtering.

So, DC magnetron, RF magnetron or DC sputtering, RF sputtering. The reason for using this magnetron again is to improve the rate of deposition. So, this is all about the sputtering system and using sputtering you can like I said in the theory class as well that the sputtering will overcome the limitations of thermal evaporation and even evaporation by having better step coverage. The difficulty with a sputtering system is that it is a low throughput system with only one wafer at a time you can use it. You may have already seen in the lab class that we have a plate which is having a source right at a material that you want to sputter and you have a substrate holder.

Because we have DC and RF you can change the angle of the source. So, to use each in a respective position generally, you should have the source and substrate right on each other like this. So, this is all about the sputtering system and I hope that you now have a better understanding of what a sputtering system looks like and how it can operate the system. So, now, I will stop this particular lecture and I hope that you have understood from this lab component as well as the previous lab component how the evaporation and sputtering system looks like again you need to follow the lab governing protocol while entering the clean room.