Biomedical Ultrasound Fundamentals of Imaging and Micromachined Transducers

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Lecture - 22

Hi, welcome to this class. Today, we will be discussing electron beam evaporation. Previously, we have covered the basics of physical vapor deposition techniques. These include thermal evaporation, electron beam evaporation, and sputtering. In the last lab class, we saw how thermal evaporation works, what the equipment looks like, how to create a vacuum, what a base vacuum is, what a high vacuum is, how to apply voltage to the boat or source holder, how to load the source material onto the source holder, and how to load the substrate onto the substrate holder. When you heat the boat, the material loaded onto it melts, vaporizes, and then deposits onto the substrate.

The limitation of thermal evaporation arises when the melting point of the source material is higher than that of the source holder itself. In such cases, thermal evaporation is not suitable. The alternative technique we can use is called electron beam evaporation.

In the theory class, we learned how an electron beam is generated from a filament, how it is accelerated to the accelerating electrode, how it is bent through a magnetic field, and how it is directed onto the source material. In this case, we use a crucible. You can either have a point source or a raster scanning system. With this technique, we are not heating the crucible; instead, we are melting the material using the electron beam. The electron beam heats the material loaded onto the crucible, causing it to vaporize and deposit onto the substrate.

Now, let us move to the lab demonstration and see how the electron beam evaporation system works, how to create a base vacuum with the primary pump, how to achieve a high vacuum with a secondary pump, what a Pirani gauge is, and the roles of the substrate holder and the crucible. We will also see how to generate an electron beam, although it is not visible due to safety covers. By the end of this demonstration, you will understand how electron beam evaporation overcomes the limitations of thermal evaporation. We have also recorded a session on sputtering. But first, let’s see how electron beam evaporation works.

Hello and welcome to this demonstration of E-beam deposition. In your coursework, the professor might have already discussed physical vapor deposition, and before that, the basic steps of micro and nano fabrication, where deposition is one of the crucial steps. Deposition can be of various types, and the most common ones are PVD (Physical Vapor Deposition) and CVD (Chemical Vapor Deposition).

The tool we are using today is for PVD and is capable of two types of deposition. The first is thermal evaporation, where the source material is heated using resistive or Joule heating. In this process, a current is passed through a wire, heating it, melting the material, and causing it to evaporate. The evaporated source material then deposits onto the substrate.

The second option in this tool is E-beam evaporation. Here, the source material is placed in a graphite crucible, and an electron beam is directed at it. The high kinetic energy of the electrons is converted into heat, melting the source material. Once it starts melting, and if the vacuum and temperature conditions are sufficient, it begins to evaporate. The evaporated material then deposits onto the substrate, completing the deposition process.

Now, let's see how this tool works. I'll switch it on first. Once the display comes on, I'll click on the hardware reset. You can see the safety interlocks are on, indicating that all the doors of the tool are properly closed, and the system is under vacuum.

You can also see the water-cooling option, which means that the water cooling is functioning correctly. The source enable is off right now. The source here refers to the electron beam, which is currently inactive. Once the deposition starts, the source will activate and begin to glow. This panel serves as both a display and an input device. The system status shows “standby,” which means the pumps are not running, and the tool is idle.

There are two options available: “Start” and “Vent.” When I click on “Start,” the pumps will begin running. We will discuss the types of pumps shortly. The “Vent” option allows atmospheric gases to enter the chamber, bringing it to atmospheric pressure, enabling the door to be opened. Currently, since the chamber is in vacuum, there is too much pressure to open the door.

Now, I will click on “Start” to initiate the pumps. Let’s take a closer look at the panel options. We have the main power switch, safety interlocks, water cooling indicator, and the source indicator.

The system status now shows that the turbo pump is ready, and the chamber pressure is in vacuum, but it is only at 10^{-2} millibar, which is not sufficient for deposition. Initially, we had only the “Start” and “Vent” options. Now we also have “Stop,” “Cycle,” and “Process.” Let's go through them one by one.

“Start” initiates both pumps: the roughing pump and the turbo molecular pump. The roughing pump reduces the pressure from atmospheric pressure to around 10^{-3} millibar, after which the turbo molecular pump takes over, reducing the pressure further to 10^{-6} millibar, which is ideal for deposition.

“Stop” halts both pumps. “Cycle” connects the pumps to the chamber, initiating the evacuation process. “Vent” introduces atmospheric gases into the chamber, allowing the door to be opened.

Finally, “Process” is used once the vacuum condition is achieved and we are ready to turn on the electron beam.

Now, let's look at the other controls on the panel. First, we have source control. This on/off switch powers the source, which is the electron beam, not the source material. There are indicators for power, vacuum, and water cooling.

The “On” switch powers the transformer or unit operating the gun. After that, the actual power is applied to the gun, and you can control the current supplied to the electron beam with the knob.

We also have the EB3 sweep control. When the electron beam hits the source material, it can remain at a single point, or you can choose to sweep it, or raster it, in the x and y directions. This option helps to cover a larger area and achieve more uniform heating. The knobs control the amplitude of this movement in both directions. There is also a switch to toggle between point source (0) and sweeping (1).

We usually use a sinusoidal waveform for the x and y movements because it provides a smooth motion, reaching the extremities slowly, speeding up in the middle, and slowing down again, similar to simple harmonic motion.

On the right, we have the turret controller panel. The chamber can hold up to four materials at a time, each in a separate graphite crucible. Currently, we have titanium in the first slot, gold in the second, platinum in the third, and aluminum in the fourth. The turret controller allows us to switch between these materials using a knob, and we can also control the speed of the turret.

Lastly, we have the Digital Thickness Monitor (DTM). This tool helps us monitor the deposition process, ensuring it is happening as expected and estimating the thickness of the deposited layer. This is crucial to avoid wasting time and material if the deposition process fails.

So, how this works is that inside there is a quartz crystal, which is why this DTM (Digital Thickness Monitor) is sometimes referred to as QCM (Quartz Crystal Monitor). Basically, there is a quartz crystal suspended near the source or inside the chamber. This crystal has a particular resonant frequency, but as material is deposited onto the crystal during the deposition process, there will be an increase in the mass of the crystal due to the added thickness of the material. This increase in mass causes a slight change or shift in the resonant frequency. By calculating this shift in the resonant frequency, we can estimate the amount of mass being deposited, which is what the digital thickness monitor does.

Since we know the exact area of the crystal where the material will be deposited and the thickness is what we need to find out, if we know the density of the material being deposited, we can easily calculate the thickness.

That's about the DTM. Then we have some other options as well. On the left here, we have something for thermal deposition. We won't be focusing on that, but rather, we'll see what's on the right. These five knobs control various functions. The first one is for the DTM. When you switch it on, the digital thickness monitor starts.

Next, there is an E-beam gun shutter. If you turn it on, you will see a shutter inside the chamber, located on top of the source material. The function of the shutter is to cover the crucible while the material is being heated. The material needs to be heated at a specific temperature in a favorable vacuum, and this heating process, done using an electron beam, cannot be very sudden. It must be slow and steady. Initially, some vapor may come out, but you cannot accurately predict the deposition rate.

So, while the material is heating, the shutter remains closed to prevent any irregular deposition from affecting the sample or substrate. Once the material is sufficiently heated and the deposition is about to start, the shutter can be opened. Similarly, when you want to stop the deposition, simply reducing the current is not advisable because the material is still heated, and some vapors will continue to come out. To precisely control the deposition time, as soon as the desired time is reached, you close the shutter. This prevents further deposition on the sample even if some vapors are still coming out.

Now we have something called the rotary drive. Inside the chamber, there is a substrate holder. At some locations, the deposition might be less due to distance from the source, while at other locations, it could be higher. To ensure uniform deposition, the substrate holder rotates. Using the rotary drive, the substrate holder can be set to rotate at a specific speed, like 3 rpm. Depending on the requirements, it can be adjusted up to 5 rpm or lower, like 1 rpm.

Once the chamber reaches atmospheric pressure, we can open the door and look inside. The hissing sound stops, indicating that it is safe to open the chamber door. The first thing we see is the substrate holder, which I'll remove to show you the inside. The spindle, where the substrate holder is mounted, rotates because of the rotation drive for uniform deposition.

Now, here is the main block of the EBM deposition system, where the filament, or the electron gun, ejects electrons directed in a circular fashion from the port to the material holder or source holder. Here we have the shutter, which is used to control the deposition time. We can switch it on, and you can see the shutter moving. In the crucible, I have aluminum, which is not in pellet form because it has been used for some depositions and has melted and stuck to the sides.

I will carefully place it back and close the shutter. There are also some extra hanging structures. This is the holder for the quartz crystal; one is for electron beam deposition (E-beam deposition), and another is for thermal deposition, which we will not cover today.

Now, I will show you how to place the sample on the substrate holder and attach it back to the spindle. Here is the substrate holder, which I removed from the chamber. I have a glass slide that we will be using for depositing aluminum, which is in crucible number four. I will place the glass slide in this region and secure it with these screws and holders. This is to ensure that the sample does not fall off when we attach the substrate holder back to the spindle. I'll use these holders and an Allen key to secure it. Now, using tweezers, I'll check if it's secure. Since the sample is secured, we can load it back into the chamber and start the evacuation process.

As you can see, we have attached the glass slide and secured it using holders and Allen screws. Now, we will load it into the chamber and start the evacuation process. This is why we had to secure it—so that when we keep it upside down, the sample will not fall into the chamber. Now, I'll show you that when we turn on the rotary drive, it starts rotating, which is more evident when looking at the spindle.

I'll now close the chamber. Whenever you are depositing, it's a good idea to observe what's happening inside. For that, we have a viewport—a glass window. On the opposite side, we have a metal shutter. The idea is that whenever you want to see inside the chamber, you pull this lever to open the viewport. Once you are done, you close it back to prevent material from depositing on the glass. If it remains open, the material being deposited will also be deposited on the viewport glass, making it very difficult to clean or replace.

I will lock the door, and you can see we have three options: Process, Cycle, and Seal. The Process option is used to start the deposition, but it is currently not possible because our chamber is not in a vacuum condition—it is at atmospheric pressure. We won't be using the Seal option here. Instead, we need to start the evacuation process, which is controlled by the Cycling option.

Once I click on Cycling, the pump will start running. The roughing pump, or the main pump, will take the pressure from atmospheric (10^{3} millibar) down to around 10^{-2} or 10^{-3} millibar. Once that happens, the turbo molecular pump will take over and reduce the pressure further to around 10^{-6} millibar, at which point we can start the deposition process.

So, let us quickly go to this mimic diagram and see the system view. Now, we will have a closer look here and see exactly how the schematic of this tool looks. In this display, you can see the mimic diagram. The first rectangle at the top represents your chamber, which is directly connected to a line leading to your roughing pump. Right now, this is your turbo molecular pump, and this is your roughing pump. As you saw, this valve was initially on, and this valve was off. What happened was the roughing pump was directly connected to my chamber as the pressure was decreasing from 10^{+3} to approaching 10^{-2}. As soon as it reached 10^{-2}, this valve turned off, disconnecting the chamber from the roughing pump. Now, it is running through this high vacuum valve, which is connected to the turbo molecular pump and further connected to the backing valve, which in turn is connected to the roughing pump. So, right now, the pumps are working in series,

with my chamber connected to the turbo molecular pump, which is connected to the roughing pump. The pressure decreases like this: we have atmospheric pressure at 10^{+3}, and it goes down to somewhere around 10^{-2} or 10^{-3}, and then through this turbo molecular pump, the chamber reaches a pressure of approximately 10^{-3} millibar.

We also have various accessories, such as a pressure indicator, interlock status, and some other valves. That is the entire gist of this representation diagram of our tool. Now, let us get back to the system control to see what is happening here. We have the cycling sequence; fine pumping is going on. Earlier, we saw that it was rough pumping, where the pressure was approaching 10^{-2} or 10^{-3} Now that stage has passed, and we are in the fine pumping region where the turbo molecular pump has kicked into action. Right now, the options we have left are seal and vent.

Once the fine pumping is complete—that is, when the pressure has reached somewhere near 10^{-3} to 10^{-6} millibar—we will be able to start our deposition. We will wait until the desired pressure is reached and then continue with the process. The vacuum has now reached where we want it to be, which is in the order of 10^{-5} to 10^{-6} millibars. As we discussed earlier, when a sufficient vacuum is reached, the mean free path of the particles increases. This means that the particles coming out from your source will reach your substrate with minimal collisions, ensuring they have enough kinetic energy when they are deposited on your sample.

Now that the vacuum has been achieved, I will turn on the E-beam. I have switched on the main supply for the E-beam. Next, I will turn on the transformer and finally turn on the power. I will also put this system in process mode. First, I need to seal the chamber, and once it is sealed, I will click on process. The system status shows that the process sequence is pumping down. The pump-down sequence is on, and throttle pumping is complete. Now, I will turn on the power for the electron beam gun. You can see the first LED display shows the kilovolts (kV) supplied to the E-beam gun, and the second one shows the current being drawn.

Now that the power is on and the vacuum has been reached, I will turn on the gun. If you can see here, we have a voltage of 5 kV. The next step is to slowly increase the current for the gun, and that is how the power will be delivered. Before that, I need to ensure some prechecks. First, I need to confirm which crucible I am using. For example, today we are going to be depositing aluminum on a glass slide. I need to make sure that, in the turret, which has four slots, we can load four different materials at a time. The materials available are titanium in crucible number 1, gold in crucible number 2, platinum in crucible number 3, and finally, aluminum in crucible number 4. I have set crucible number 4, which is what we will be depositing.

Now, I will start increasing the current slowly. Once I increase the current, the electron beam will travel from the source gun in a circular path and fall on my source material—in this case, aluminum in a crucible. When this electron beam falls on my crucible, it will start melting, and that is how

the vapors will travel. Since we have a very good vacuum now, the mean free path is sufficient, allowing the aluminum vapors to reach the substrate without collisions.

The SOP states we cannot heat any material with a large current due to the thermal shock risk. Therefore, we will perform the deposition at around 35 milliamps at a voltage of 5 kV. We cannot apply 35 milliamps all at once, so we will increase it in steps: 10 milliamps, then 20 milliamps, then 30 milliamps, and finally 35 milliamps, with intervals of 30 seconds and 1 minute.

So, let's get started. I will increase the current to 10 milliamps and start a stopwatch. Once it reaches 30 seconds, I will increase it to 20 milliamps to ensure that the material is gradually heated up. We are almost there, and I can also see a yellowish glow because of the electron beam. Now that 30 seconds have passed, I will increase it to 20 milliamps. After another 30 seconds, I will increase it to 30 milliamps.

Now, let's increase the current further to 38 milliamps. Once the metal is adequately heated, if you look through the viewport, it will have a red-hot appearance, indicating that evaporation has started, which is a good sign. Once the preheating is done, we will open the shutter, and the deposition will start on the actual sample.

As we discussed, there is a shutter on the source material. When the electron beam is on and the shutter is closed, all deposition occurs on the shutter, allowing for precise control over the deposition rate and time. Once we open the shutter, deposition on the sample will begin, and to stop it, we will close the shutter.

Now that we have maintained 30 milliamps for 1 minute, I will increase it to 35 milliamps. We will keep it at 35 milliamps for one more minute before opening the shutter to start deposition. Meanwhile, I will turn on the digital thickness monitor (DTM) and ensure that the sample rotates during deposition for uniformity.

Since the DTM is malfunctioning, we will leave it as is. My source material has now been subjected to 35 milliamps for about a minute, and I can see that my source is red-hot, meaning the aluminum vapors are being produced. However, because my shutter is closed, no deposition is occurring at the moment.

We will deposit for approximately 10 minutes, so I will now open the shutter and start the stopwatch. I will constantly check to see if deposition is happening, as the electron beam and the glowing source material provide enough illumination to assess whether the sample is coated. We will continue this for 10 minutes.

Now that it has been almost 10 minutes, as soon as I hit the 10-minute mark, I will close the shutter to stop the deposition. It is now 10 minutes, and I have closed the shutter. The deposition has

stopped, but the rotation is still ongoing, and the electron beam is still falling on my source material, causing further evaporation.

I will slowly reduce the current, and once it reaches zero, I will turn off the gun. You will see the voltage drop from 5 kV to zero. After that, I will also turn off the power to this panel and to the turret controller, which I use to switch between my four crucibles. Finally, I will turn off the rotary drive.

Before taking out my sample, I need to be cautious because the electron beam is still affecting the source material, and the chamber temperature is high. If I remove the sample immediately, it might still be hot, and exposure to ambient air could cause a reaction. So, before taking out my sample, I will start the cycling process to cool the chamber for about a minute or two.

After cycling for around 5 minutes, we can sense that the chamber has cooled down. The next step is to vent out the chamber. You should hear a slight hissing sound indicating that atmospheric air is entering the chamber. Once the venting stops and the chamber reaches atmospheric pressure, it will be safe to open it and remove the sample.

Now the hissing sound has stopped, and we can see that the chamber pressure is equal to 10^{+3} millibars, which is atmospheric pressure. I will now open it carefully. You can see the chamber now. I will carefully unload the substrate holder and then close it. Here is the substrate holder with the glass slide, which was transparent when we started but is now coated with aluminum, showing a shiny, mirror-like finish.

Once I have closed the chamber, the next step is to ensure it remains in an evacuated condition. If my chamber is exposed to the atmosphere for an extended period, it might absorb gases and particles. This could lead to longer pump-down times when creating a vacuum in the future. Therefore, it is good practice to keep the chamber at a moderate vacuum, around 10^{-3} millibars, after deposition.

Next, I will keep it on cycle. You can see the pump starting to run, and you will hear noise due to the evacuation, which will eventually die down. A fun fact is that this noise decreases because sound waves cannot travel well in a vacuum.

That’s it for today. Thank you! I hope you enjoyed the lab lecture. If you have any questions, feel free to ask us through the NPTEL forum. Thank you!