Lab 5 – Thermal Evaporation

Pre-Lab Reading:

Thermal evaporation is a technique used to deposit thin films of materials onto substrates. It involves heating a source material to a temperature above its melting point, causing it to vaporize. The vaporized atoms or molecules then travel through a vacuum chamber and condense onto a cooled substrate, forming a thin film as shown in *Figure 5.1*. This process is widely used in nanotechnology due to its simplicity and versatility.

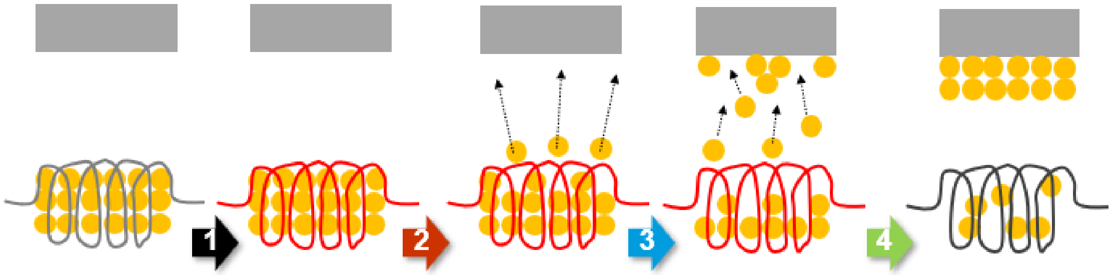

Figure 5.1: Depiction of the thermal evaporation process.

The mean free path (MFP), a concept previously introduced in labs 2 and 3, plays a pivotal role in thermal evaporation. It directly influences the quality of the deposited thin films. A larger mean free path, achieved by maintaining a higher vacuum, allows vaporized atoms to travel a greater distance before colliding with other molecules. This reduces the likelihood of collisions that can disrupt the deposition process, leading to more uniform and defect-free films. The MFP can be crudely estimated by the equation below relating the pressure in the chamber in Torr (T) to the MFP in the chamber in centimeters (cm).

$$\boldsymbol{MFP(cm) \cong \frac{5 * 10^{-3}}{P(T)}}$$

Conversely, a smaller mean free path, resulting from a lower vacuum, increases the probability of collisions between vaporized atoms and gas molecules. These collisions can cause the atoms to lose energy or change direction, hindering their deposition onto the substrate. This can result in rougher films with uneven thicknesses. Therefore, maintaining a high vacuum with a large mean free path is essential for achieving high-quality thin films in thermal evaporation. As a rule of thumb, the MFP within the chamber should be larger than the maximum chamber dimension.

Figure 5.2 shows a block diagram of a typical thermal evaporation system and the relative orientation of the different components. In the thermal evaporation system, the chamber is pumped down by a series of pumps (e.g., Roughing Pump (RP) and Turbo pump). The pressure in the chamber is monitored by a series of gauges and the deposition rate of the material is monitored in real time by a **crystal thickness monitor**

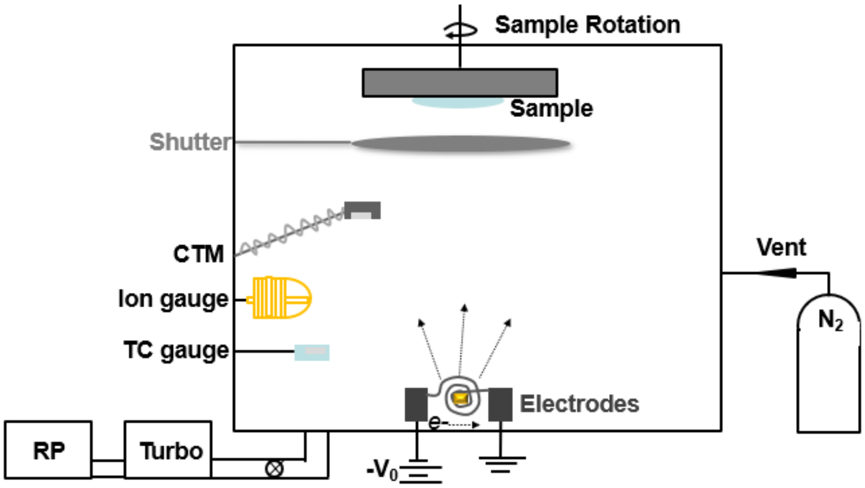

Figure 5.2: Block diagram of typical thermal evaporation system.

(CTM). Once an appreciable vapor pressure of the metal is detected by the CTM the **shutter** isolating the sample from the metal vapor is opened. Once the desired amount of metal has been deposited onto the sample, as measured by the CTM, the shutter is closed. Once the process is complete the chamber is allowed to cool and then vented with ambient atmospheric pressure or an inert gas like N_2.

The vapor pressure of the source material is another important consideration. The vapor pressure is the pressure exerted by a vapor in equilibrium with its condensed phase at a given temperature. Materials with higher vapor pressures are easier to evaporate, as they require lower temperatures to vaporize. However, materials with high vapor pressures may also have a tendency to re-evaporate from the substrate after deposition, leading to film degradation. Therefore, selecting a source material with an appropriate vapor pressure is crucial for achieving high-quality and uniform thin films.

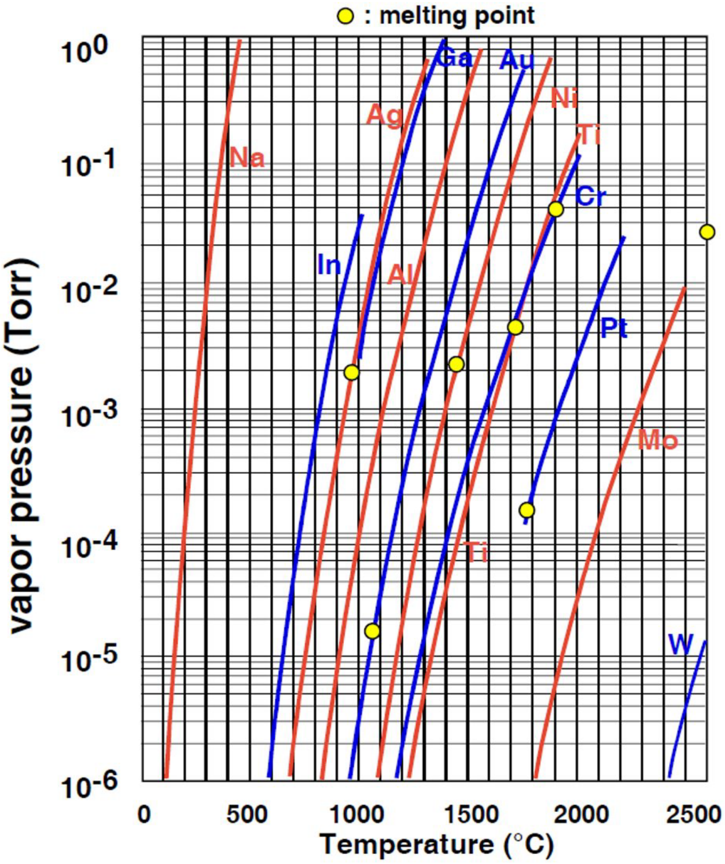

Figure 5.3: Example of a vapor-pressure graph

Material limitations can also affect the suitability of thermal evaporation for certain applications. Some materials, such as refractory metals with high melting points, are difficult to evaporate using conventional thermal evaporation techniques. In such cases, alternative methods like **electron beam evaporation** or **pulsed laser deposition** may be more suitable. Additionally, the deposition rate can be limited by the vapor pressure of the source material. For materials with low vapor pressures, achieving high deposition rates may require higher temperatures or larger source materials, as can been seen in *Figure 5.3*.

In conclusion, thermal evaporation is a versatile technique used in nanotechnology for the deposition of thin films. By understanding concepts such as the mean free path, vapor pressure, chamber geometry, and material limitations, researchers can optimize the thermal evaporation process to achieve high-quality thin films with the desired properties.

Objective:

The objective of this lab is for students to learn the basic concepts behind Thermal Evaporation and understand why it is important. Students will perform a mock thermal evaporation utilizing the Kurt J. Lesker vacuum training system and some electronic heating devices that will outline some basic concepts of thermal evaporation and how the process occurs in a general sense. The educator will provide a detailed experimental appendix for students to follow to perform the process.

Background:

Thermal evaporation is one of the most basic methods of deposition in nanotechnology. Utilizing concepts that have been mentioned in previous labs, as well as some trivial ideas, it is possible to deposit very thin (in the order of nanometers) films, of high material integrity, onto the surface of substrates such as silicon wafers or glass slides.

This process functions by first placing everything needed into a vacuum chamber. This chamber consists of a few especially important parts, such as the aperture, shadow mask, shutter, heating element/basket, and a crystal thickness monitor as was shown in *Figure 5.2*. Each of these components is key in the operation of this system, and it is exceedingly rare to find a system that does not possess all these components.

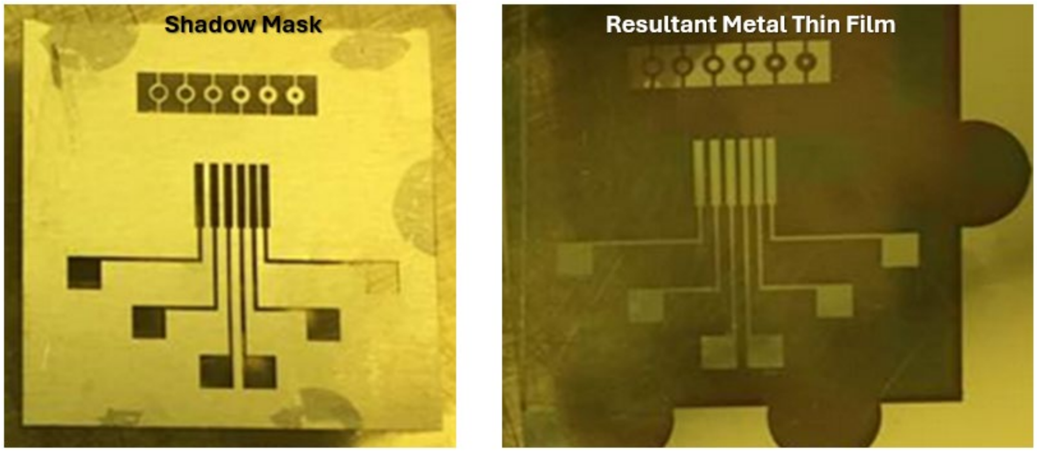

Figure 5.4: Shadow mask (left) metal thin film deposited through this mask on glass substrate (right)

The most basic elements in the thermal evaporator are the shutter, shadow mask and aperture, which operate nearly in tandem. The simplest of these three is the shadow mask. This is effectively just a metal plate of a particular size, that has a pattern defined on it with hollow areas. This pattern is a cutout, very similar to a stencil. A typical shadow mask and the resultant film deposited through the shadow mask is shown in *Figure 5.4*.

Continuing, the aperture is simply just a metal table, with a hole in the middle. The aperture is very similar to a table, which holds the substrate above the source material, or the material that will be deposited onto the substrate. The shadow mask works with the aperture too, but this can be done in two different ways. Some shadow masks are large enough to not fall through the hole of the aperture. If this is the case, usually the substrate is placed on the top of the shadow mask while the process is run. In other cases, this larger shadow mask can be used to hold the substrate above the source material directly, simply by taping the substrate to the shadow mask. Simply put, the substrate is secured to the large shadow mask by using a special type of tape, known as Kaptain tape. The entire plate is then flipped upside down and placed in the aperture, with the substrate facing down. Additionally, if a smaller shadow mask is desired, it can be taped to the top of the substrate before it is hung upside down, therefore allowing the mask to be used.

The final basic component of the evaporator is the shutter. The shutter is one of the most important elements of the evaporator, as it dictates when the deposition begins, and when it ends. This is very important, as the initial stages of evaporation can possess various contaminates and other unwanted materials, such as oxides, which would ruin the thin-film quality. The shutter protects the substrate from the deposition of these unwanted materials and allows the tech to begin the deposition once the unwanted material burns off. *Figure 5.5* shows what a typical shutter looks like. In this case the shutter is in the "open" or "on" position as it is not isolating the metal source material from the substrate above. To close the shutter, it would be positioned directly above the metal source so as to block the line of sight between the metal source and the sample.

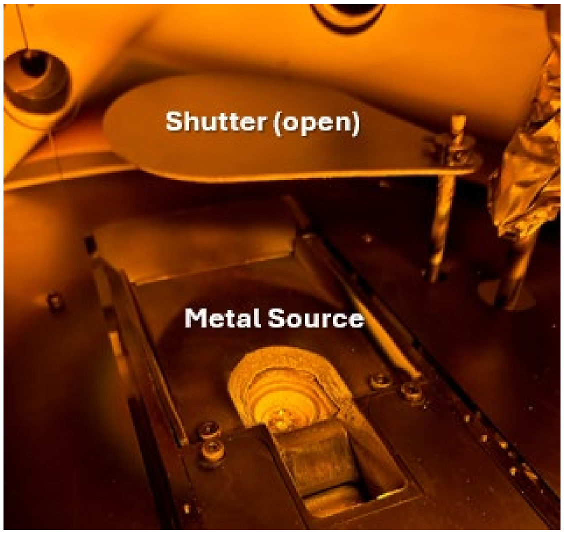

Figure 5.5: Shutter used in the evaporation process.

The more complicated elements in the thermal evaporator are the heating element and the crystal thickness monitor. The heating element usually consists of a tungsten wire in the shape of a basket or a thin plate with a bowl depression in the middle of it. This basket's overall purpose is simple: to hold the material to be evaporated. There are various basket types depending on the thermal evaporation system configuration, as shown in *Figure 5.6* (left). The source material to be deposited is placed in the basket, then the entire system is pumped down to under a high vacuum. The source material is typically an ultra-high purity (>99.99%) metal which comes in the pellet form as shown in *Figure 5.6* (right). Once the basket is loaded between the electrodes and the source material is placed within the basket the system is pumped down to a high enough vacuum to allow the MFP within the chamber to be much larger than the chamber dimensions. At this stage, a high current flows through the wire or plate, causing it to heat up. Once the proper temperature is achieved, the source material will begin to vaporize, beginning the deposition process.

https://www.lesker.com/newweb/evaporation_sources/basket-heaters.cfm

Figure 5.6: Typical basket designs (left) and pellet source materials (right)

The second complex component involved in the thermal evaporator is the crystal thickness monitor or the CTM. In theory, the CTM is a complicated system, but in practice, it is simple to explain and understand. The CTM, as its name implies, is responsible for monitoring the thickness of the deposited film. A CTM is known as an indirect measurement device because it does not directly read the film's thickness on the substrate but uses external information to make an educated guess of the thickness. The CTM consists of the quartz crystal as seen in *Figure 5.7*, held above the source material but not in front of the substrate. This crystal has a natural resonate frequency of ~6 MHz, rather, it naturally shakes with a set rhythm of 6 million times per second. As the deposition progresses, material is deposited onto both the substrate and the CTM. As material builds up on the crystal, its frequency slows down. Using the amount that the frequency slows down, a computer controller can not only dictate the rate at which the film is being deposited but also the total amount of material deposited onto the substrate. However, as previously said, this is an indirect reading measurement device, and it is not uncommon for the CTM to be inaccurate when measuring the film thickness. This is why it is important to measure the film thickness with another measurement tool after the deposition, to ensure that the thickness is within the desired range.

Figure 5.7: Crystals used in a CTM

Experiment:

SAFETY DISCLOSURE: This lab contains the use of mechanical and electrical elements that may cause injury if used improperly. Be sure to use all equipment in a dry environment and ensure all digits are clear before tightening any screws or clamps. The overall vacuum system is heavy and should be lifted by two people if needed. Be sure not to lean against the system or push it to avoid tipping.

Required Materials:

1. KJL Vacuum Trainer
2. Soda can (Empty)
3. Hammer
4. Low melting point wax beads
   a. Link
5. Mug Warmer
   a. Option 1
   OR
   b. Option 2
6. USB battery bank
   a. Option 1
   OR
   b. Option 2
7. DVD/CD Case
8. Pens
9. Cutting tool (Tin Snips preferred)
10. Glass slide
    a. Link
11. A small, heavy object (A ratchet is used in the example photos)
12. Tape

NOTE: Two options are given for the battery bank and warmer. The first option is shown in the images, while the second option is not. Both are functional; however, the first options have shown some degree of degradation after many (50+) deposition runs, as well as the fact that the battery bank can only perform around 2 depositions per charge and requires up to 12 hours to fully recharge. The second option does not require frequent charging, and the hot plate reaches a higher temperature, allowing for a somewhat faster deposition.

Step 1: Set up evaporation system and aperture

The first step of this experiment will outline the set-up process for the vacuum trainer as well as some other components of the lab. The first step will be to ensure that the vacuum trainer is level via a quick rocking test, or with a level if one is available. Once the system is confirmed to be as level as possible, you will need the DVD/CD case and cutting tool. From the case, cut an approximately 4.5x4.5-inch square. In the middle of this square, cut a square hole approximately 1x2 inches. Do not dispose of the removed square, as it will be used later. Around this square hole, around a centimeter from the top, and bottom left and right corners, utilize the cutting tool to create a small hole. Push the tips of the pens through, continuing to push the pen through to small pen clip, creating a small table about 1-2 inches tall, with the rest of the pen sticking through the top. Depending on the type of plastic used, it may be necessary to cut into the sides of the hole to allow it to widen. Returning to the removed square, cut smaller holes or shapes into the removed plastic of any desired shape or design, therefore creating a shadow mask. There is a chance that the shadow mask may crack or break, and this can be fixed simply with some tape, as shown in the figures.

Figure 5.8: Process of creating the aperture table.

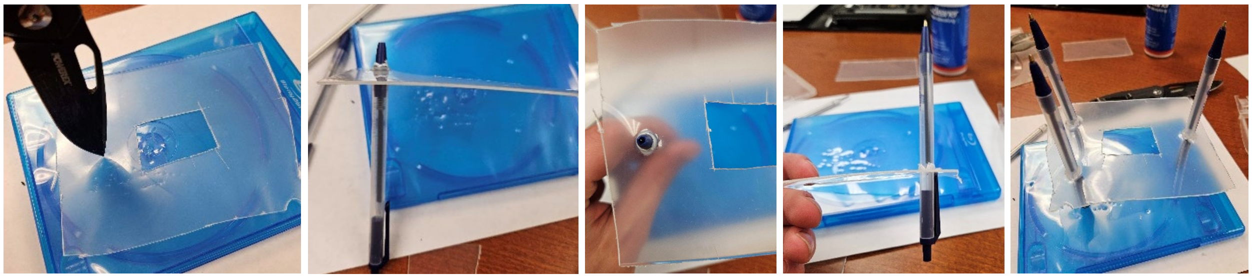

Figure 5.9: Assembling the aperture table.

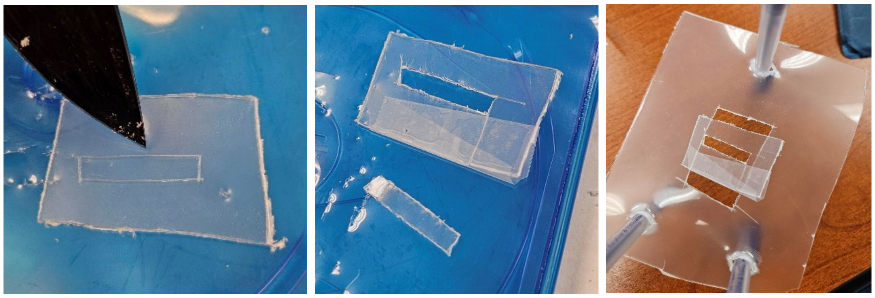

Figure 5.10: Creating the shadow mask and placement demonstration.

Step 2: Set up electronics and source material

Once the vacuum trainer has been set up and the aperture made, it is now time to set up the heating element. First, gather the mug warmer, battery bank, and weight and place them onto the baseplate of the vacuum system. Plug in the mug warmer and place the weight on the non-cup side. A small click should be heard, and a light should appear (if the same brand of warmer is being used as the example). This indicates that the warmer is now on, as the safety system has been triggered. Remove the weight for now, to avoid using more battery than necessary. ***NOTE: make sure all wires and electronics are within the diameter of the baseplate, in order to maintain the seal.***

Figure 5.11: Creating the crucible.

Then, take the soda can or other beverage can and remove the bottom as shown in the photos. The bottom part of the bowl shape (when inverted) should come into contact with whichever surface it sits on, rather, the bottom of the bowl should be the lowest point. Taking the hammer, flatten the bottom of the bowl slightly, so that more of it is in contact with the surface it is sitting on. This will ensure that the heating element heats as much of the bowl as possible, while still containing the wax. Once finished, put the bowl, or rather the crucible, with the mug warmer and move to the next step.

Step 3: Perform mock film evaporation

Turn on the wax warmer and place the bowl onto the warming area. Allow the bowl to warm for at least 10 minutes before beginning the deposition process. Once the bowl has warmed, place 26 total wax beads into the bowl, and place a glass slide on top of the aperture. Ensure the aperture, battery pack, warmer, and all other devices are within the diameter of the base plate. One secured, the weight can be placed to turn on the warmer, the bell jar replaced, and the vacuum turned on. ***NOTE: It may be necessary to tape the warmer to the baseplate, and the dish to the warmer, as they may try to move due to the vibration.*** Allow this to run for 40 minutes, then vent the system and remove the bell jar. Use caution, as the slide and warmer will be hot. Be sure not to run the deposition for more than this time, as any additional time will lead to adverse effects on the deposition.

Figure 5.12: Assembling the thermal evaporation demonstration.

Step 4: Preform a mock pattern evaporation

Similar to the previous step, set up the glass and aperture as previously done, except this time, place the shadow mask in the aperture and tape it to the bottom of the glass. Replace everything as done in the previous step, with the addition of the shadow mask. Observe the deposition process as done previously and remove the substrate after 40 minutes.

Questions:

1. What are some known limitations of thermal evaporation? Use at least one source to support your answer.
2. Why is the mean free path important in thermal evaporation?
3. What is nucleation? How was it observed during the experiment?
4. Why is it important to perform a thermal evaporation in a vacuum environment?
5. Research one real-life thermal evaporator, and report on its cost, capabilities, and uses in either industry or research.
6. What is a vapor-pressure curve, and how are they related to thermal evaporators?
7. What are some common metals used in thermal evaporators? Why can't Tungsten be deposited in a thermal evaporator?
8. Thermal evaporation is one example of Physical Vapor Deposition (PVD). Research one other method of PVD and summarize it.
9. Besides PVD, there are a host of other deposition methods used in nanotechnology. Research a method and quickly report on it, citing at least one source.
10. What is a CTM, and how does it work? Explain in your own words.