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Micro and Nano Technology

Laboratory Report

Deposition of thin metallic layer by DC Sputtering

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Abstract:

This study looks at how to deposit thin layers of metal using a method called Direct Current (DC) sputtering, which is common in making materials and electronics. We focused on adjusting different settings like power, pressure, and temperature to create smooth and high-quality thin films of metals such as copper, aluminum, and titanium. By changing these settings, we studied how they affect the thickness, stickiness, texture, and electrical properties of the films. We used advanced tools like Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and X-ray Diffraction (XRD) to closely examine the structure and content of the layers. Our results show that by carefully controlling the sputtering process, we can significantly improve the quality and performance of the films. This demonstrates that DC sputtering is a useful and efficient method for making thin metal layers for various technological uses.

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Equipment Description:

The equipment which we are using is UNIVEX 300 from Leybold which is shown in the below figure. The Cylindrical chamber on the left is Vacuum chamber where the Substrate is placed for undergoing the process. This chamber is attached with a rubber bush which helps in creating Vacuum. The Black Rack on the right side of the figure contains the controls for managing vacuum supply (rotatory pump and turbo-molecular pump) and Power supply for the operation of glow discharge. The following two actions should be taken before manually filling the vacuum chamber with ambient air via the valve (2) in Figure 4 to prevent damage to the apparatus:

Figure 1: Carrier gas inlet (Ar)

Figure 2: UNIVEX 300

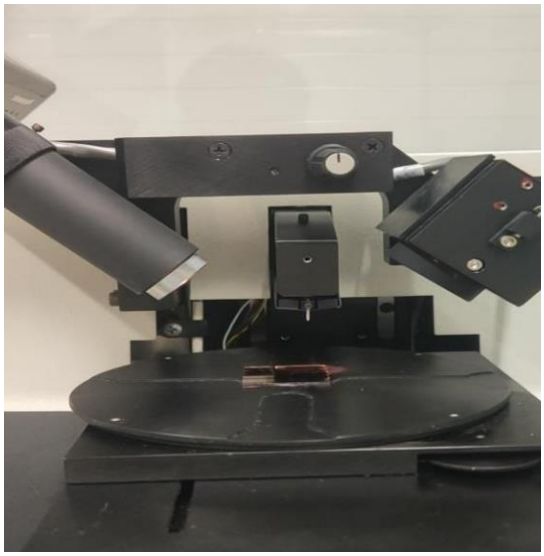

Figure 3: Measuring height using microscopy

Figure 4:Sample Holder

In the above figures,

- The vacuum measurement unit must be turned off. (Hold down the HV button until it says OFF).
- The turbomolecular pump needs to be turned off and its rotation stopped.

When the turbomolecular pump is still rotating, do not open valve. Once the power source is turned off and the black right switch is turned on, it takes several minutes for the turbomolecular pump to slow down its rotation speed. It is safer to flood the vacuum chamber by switch and not utilize the valve at all. The sample holder accepts a substrate that has been loaded and can hold a mask for the deposition of structures. The revolving shutter can be positioned to protect the substrate from deposition from the source as needed. It is operated from outside the chamber.

Procedure:

1. **Unlock the sputtering compartment:** Activate the main power supply by means of the left silver switch (7). Both the turbomolecular pump (8) and the DC power supply (5) need to be turned off. Fill the room with ambient air using the manual switch (2) or the black switch (7, right, position "0"), if needed. The metallic bell jar can then be carefully raised (it is heavy!) and set away to prevent damage or dust from getting on its gum-tightening lip.

2. **Attaching the substrate:** In order to shorten the time needed for the subsequent pumping, it is preferable to avoid touching the inside of the vacuum chamber with your bare hands. The substrate needs to be positioned over a hole in the circular sample holder located in the chamber's upper left section. Examine the shutter's rotation (black knob beneath the chamber, axial in the chamber) and note which position it opens in. The sputter source, which has a 50 mm diameter copper target, is situated at the base plate's bottom in the chamber's left section.

3. **Shutting the room**: Both the base plate where it joins and the bell jar's gum lip should be spotless and securely positioned at the rim. Place the jar onto the base plate such that the metallic pin slides into the opening in the white plastic section at the base plate's rim. To prevent the substrate from being disturbed, avoid touching the sample holder by the bell jar.

4. **Taking out of the chamber:** It is necessary to confirm that the flooding (2) and gas inflow (3) valves are closed after shutting the chamber. There is a knob in the wall to the left of the equipment that controls the cooling water supply (you can see two water lines going to the wall). Verify the presence of the water flow by looking at the green LED bar on the yellow measuring device that is visible at the rear of the black rack. If the DC voltage is turned on, the sputter source will be damaged if there is not enough cooling water! Now, select position "1" on the black switch (7 right) to activate the rotary vacuum pump. There is a booming sound as the rotary pump turns on. Turning on the vacuum meter (1) is possible. The turbo molecular pump (8) can be activated when the vacuum gauge (1) registers a pressure lower than 9.9x10-1 mbar. A pressure lower than 6x10-4 mbar may cause the sputter process to begin.

5. **Sputtering:** First, 1 gas entry valve (3). Take care not to put too much pressure on the turbomolecular pump; it should stay below 2x10-1 mbar. The following adjustments will now be made:

6. **Look for three red lights to see if any of them are on:** Here, a red light indicates "OK." Switch on the DC-sputtering supply (5) Set the voltage setpoint potentiometer back to zero (5). Choose the voltage "U" mode. Turn on the DC Raise the voltage by gradually increasing the potentiometer to the plasma's ignition point. Gradually decrease the gas inlet (3) and raise the voltage even more.

7. The goal is to progressively increase the discharge current to 0.907 A, which is constrained by the DC power source. The issue is that the target's surface is initially polluted and oxidized, so rapidly raising the voltage causes audible unpleasant noticeable sparking. In order to prevent sparking, lower the voltage. To start a continuous discharge, try varying the pressure between 1x10-2 and 2x10-1 bar. Then, try raising the voltage gradually. Following a few trials at different pressures, a consistent glow discharge at low current should occur in a small voltage band that is located just below the sparking.

8. After a few seconds, let the target's surface clean up, and then gradually reduce the pressure. Prior to sparking, a lower pressure should permit a higher voltage and current. The glow discharge is extinguished by an inadequate pressure. As a result, it is best to gradually raise voltage and decrease pressure at the same time. When a stable discharge and a working pressure between $3 \cdot 10\text{-}3$ mbar and $9 \cdot 10\text{-}3$ mbar are achieved, the current should approach 0.907 A, indicating that the deposition process can begin.

9. The substrate will be covered if the shutter beneath it is opened. Take note of the deposition time. In the event that the discharge goes out and doesn't rekindle on its own, gradually raise the pressure. Lower the pressure once again after ignition. Following the predetermined deposition duration (to be monitored using a stopwatch), the gas inlet valve (3) is closed and the sputter power supply (5) is turned off. The turbomolecular pump (8) and the vacuum gauge (1) are then both turned off.

10. The rotary pump can be turned off (black right switch (7)) once the turbomolecular pump begins to humbly slow down. The chamber will then automatically flood. Once the sound of the turbomolecular pump's operation stops, the chamber can be manually flooded using the valve (2).
    The metal bell jar can then be taken out, just like it was mentioned in paragraph 1. One can perform a fresh sputter procedure after reloading the substrates. Deposit a total of five substrates with the integrated copper target during the course of one minute, two minutes, three minutes, four minutes, and five minutes, all under similar (as much as feasible) discharge circumstances. Using a multimeter, determine the deposited films' electrical conductivity.

Results:

We deposit a thin film of copper (Cu) on a glass substrate using the sputtering process following the given procedure. The experiment is conducted with substrates for 1,2,3,5 and 10 minutes by carefully controlling parameters such as vacuum, water level and pressure for successful deposition. When a high-energy ion beam bombards the copper target, it causes the ejection of copper atoms or ions from the surface. These atoms/ions then travel through the vacuum chamber and eventually condense on the glass substrate, forming a thin film. Deposition process starts around pressure $4.8 \times 10{-}3$ mbar.

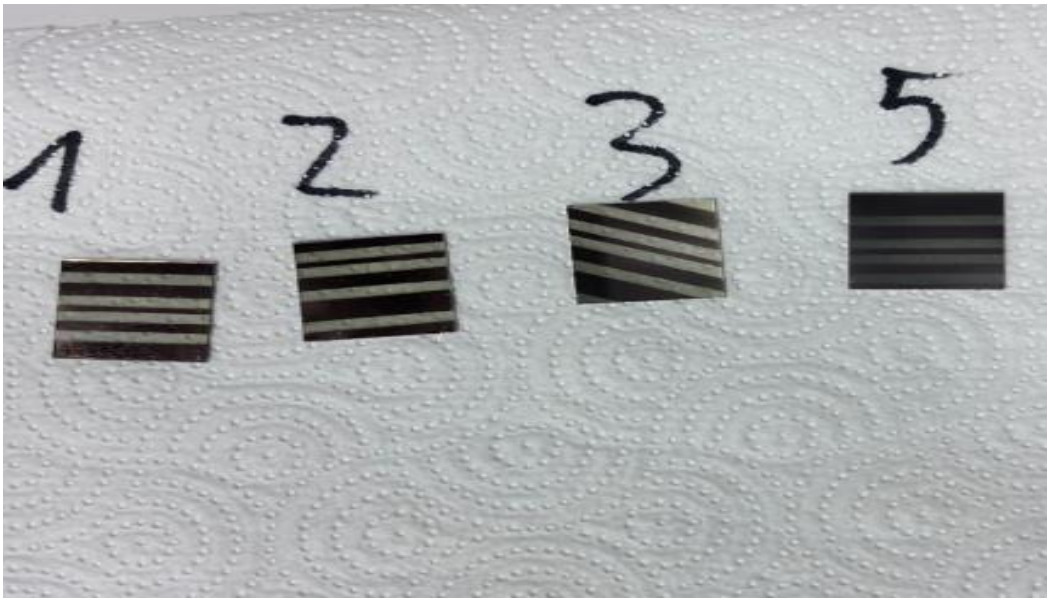

Figure 5:Sputtered samples at 1,2,3,5 minutes

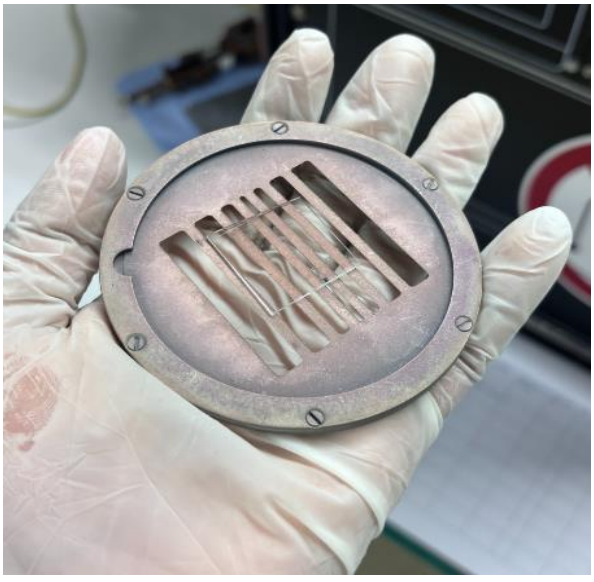

Figure 6:Sputtered samples at 10 minutes of exposure

Time (Minutes)	Plasma	Current(mA)	Power(W)
1min	403	907 mA	350 wt.
2min	403	907 mA	
3 min	383	908 mA	
5 min	387	903 mA	
10 min	Machine Failure		

Table 1:Deposition at various time interval

Electrical Conductance:

In electrical engineering, conductance (denoted by the symbol **G**) represents the ease with which electric current can flow through a material. It's the reciprocal of resistance (R). The formula for electrical conductance is:

G = 1 / R

Here,

- **G** is the conductance measured in Siemens (S).
- **R** is the resistance measured in Ohms (Ω).

The below table explain conductance at different time intervals,

Sample	Resistance	Conductance
A & B	4.5 ohm	0.22 Siemens (S)
C	1.8 ohm	0.55 Siemens (S)
D	4.6 ohm	0.21 Siemens (S)
E	5.3 ohm	0.18 Siemens (S)

Table 2: Conductance of various Sputtered Samples

Using microscopy, the sputtered sample is studied and the deposition height is determined. The samples' deposition heights are displayed in the table below.

Time(Minutes)	Deposition Height(A°)
1 min	1431.741 A°
2 min	
3 min	1930.07 A°
5 min	2071.611 A°
10 min	851.178 A°

Sample @ 1 & 2 Minute sputtering:

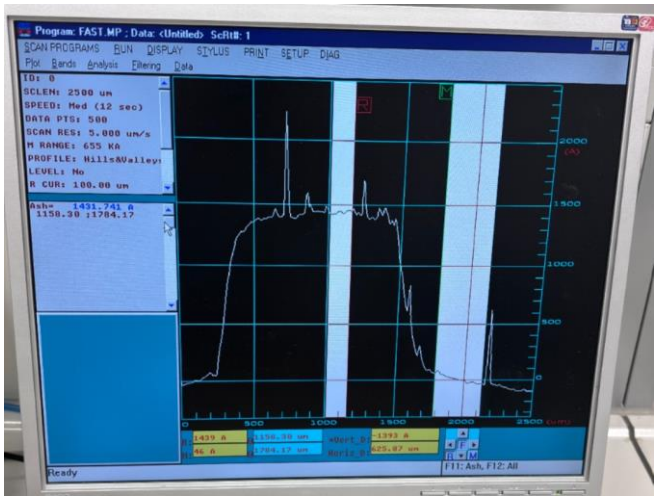

Figure 7:Height Determination at 1 and 2 minutes

Sample @ 3 Minute sputtering:

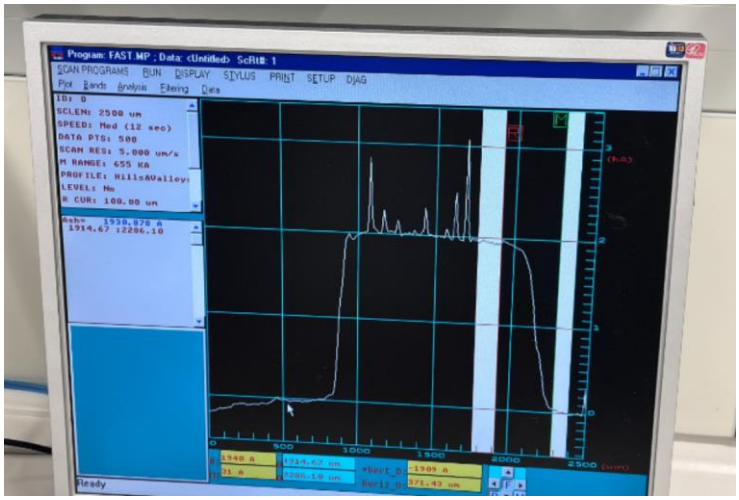

Figure 8:Height Determination at 3 minutes

Sample @ 5 Minute sputtering:

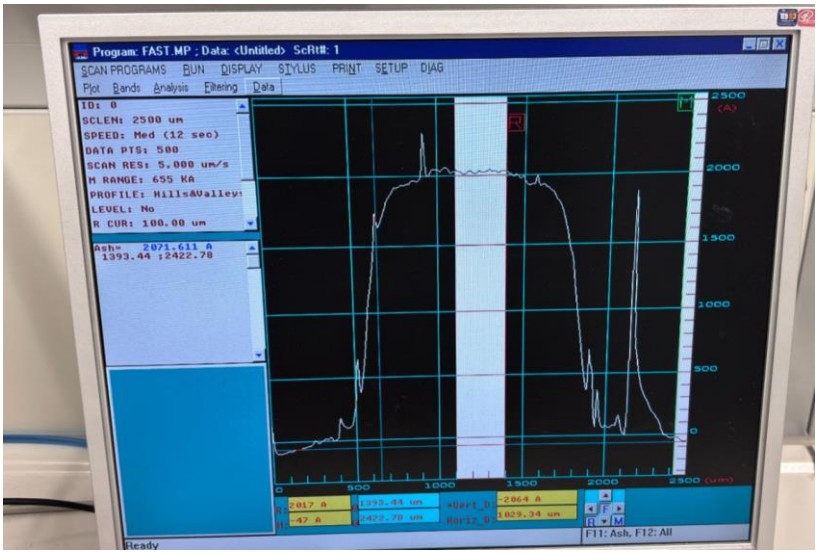

Figure 9::Height Determination at 5 minutes

Sample @ 10 Minute sputtering:

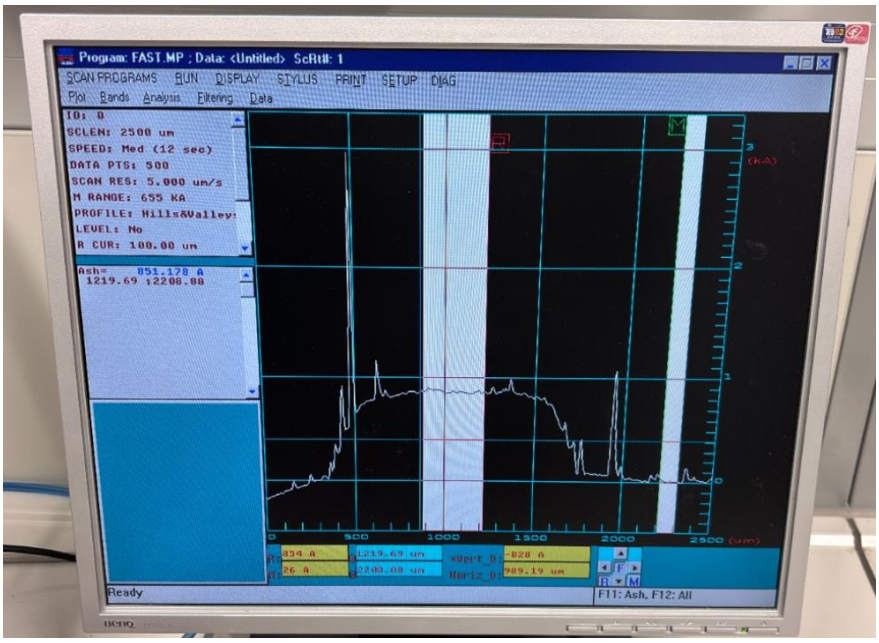

Figure 10:Height Determination at 10 minutes

Conclusion:

Overall, sputtering remains a cornerstone technique for thin film deposition due to its versatility, controllability, and wide range of applications. By understanding the principles and advancements in sputtering technology, researchers can continue to push the boundaries of material science and develop innovative solutions for the future.