How to control the UNMSM magnetron sputtering

Authors: M. G. Pinedo-Cuba*

Disclaimer

This manual is a basic guide for using the magnetron sputtering system in the laboratory of Universidad Nacional Mayor de San Marcos (UNMSM). It provides a step-by-step explanation of the process for preparing thin films. Additionally, it includes descriptions of various components and procedures involved in the operation. **This manual still needs improvement, so I would really appreciate it if you could send me any additional information and let me know if you notice some mistakes.**

*Email: mgpinedocuba@outlook.com

Introduction

Sputter deposition is a widely used technique for coating various materials in a low-vacuum environment. It involves the ejection of atoms, atomic clusters, or molecules from the surface of a solid material (referred to as the "target") through momentum transfer from high-energy particles accelerated from a plasma or ion gun. When a surface atom receives enough energy, it can displace nearby atoms, initiating a "collision cascade." These cascades can result in some momentum being redirected toward the surface. If a surface atom is struck from below and gains sufficient energy, it may be ejected from the target surface, a process known as sputtering [1]. The ejected material, in vapor form, subsequently condenses onto a substrate, forming a thin coating. These coatings, referred to as thin films, typically range from a few angstroms to several hundred angstroms in thickness. The properties of the resulting thin film depend significantly on the deposition parameters.

The process described above provides a general overview of sputtering. However, several improvements have been developed over the years. One significant advancement is the incorporation of magnetic fields to confine the plasma near the target surface. This enhancement increases the deposition rate and reduces the thermal load on the substrate [2]. The sputtering process supported by magnetic field is knowing as magnetron sputtering. The advantages of magnetron sputtering have made it the leading deposition technique for both research and industrial applications. Although the configuration of magnetron sputtering systems may vary between different pieces of equipment, in my opinion, they share several fundamental characteristics. These notes describe the use of one such system.

General description of the equipment

The equipment used is a magnetron sputtering system, model ATC Polaris from AJA International, located in the Faculty of Physical Sciences at the Centre for Technological, Biomedical, and Environmental Research. This system supports the integration of various accessories and modules. However, the components described below are those that were present in the system at the time these notes were written. These components include:

- Vacuum control
  - High-vacuum chamber.
  - Mechanical and Turbo pumps
  - High/Low vacuum detector
  - Break-vacuum system
- Plasma control
  - Gas-flow control system
  - Magnetrons with its targets
  - Direct-Current (DC) and Radiofrequency (RF) sources
  - Connection chamber-pumps
- Deposition control
  - Quartz balance
  - Substrate holder
  - Shutters

The high-vacuum chamber is monitored using a high/low vacuum detector [further information on this component should be consulted], which provides real-time pressure readings inside the chamber. Accurate interpretation of these readings requires reference to standardized pressure scales, as the system operates within both low- and high-vacuum ranges. Vacuum generation is achieved through the combined use of a mechanical pump and a turbomolecular pump. The mechanical pump reduces the chamber pressure to the low-vacuum range, after which the turbomolecular pump further lowers it to reach high-vacuum conditions. To enhance the degassing process and improve vacuum quality, thermal tape has been installed around the main chamber. Additionally, the system includes a break-vacuum mechanism consisting of a nitrogen gas line connected to the main chamber. This allows the vacuum to be safely broken using nitrogen instead of ambient air, preventing the introduction of moisture and enabling faster pump-down cycles.

The equipment is equipped with a gas flow control system that allows the introduction of up to four different gases. At the time of writing these notes, only the argon gas line is operational. These gases are used to generate the plasma required for the sputtering process. The following key components are the magnetrons, where the targets are mounted. Plasma is generated and confined by the magnetic fields produced within the magnetrons. The system is designed to accommodate up to five magnetrons; however, only four are currently in use. The equipment includes both RF and DC power sources, which are responsible for supplying the electrical energy necessary to excite the plasma and accelerate ions toward the target. Since the deposition rate is dependent on chamber pressure, it is desirable to lower the pressure to a level that supports stable plasma formation. This pressure reduction can be achieved either by decreasing the argon flow or by reducing the pumping speed. In the current system configuration, the latter approach is used; argon flow is kept constant while the connection between the chamber and the vacuum pumps is partially closed until the desired pressure is reached.

In addition, a quartz crystal microbalance is used to calibrate the deposition rate for each material, based on the material's density and its corresponding Z-factor. The operation of the microbalance relies on the piezoelectric effect: the quartz crystal vibrates at a specific resonance frequency, which decreases proportionally as mass is deposited onto its surface. By measuring changes in frequency, the amount of deposited mass can be determined. Knowing the mass accumulated over a given period allows for the calculation of the deposition rate onto the substrate. The system also includes shutters, which are controlled via the gas-flow control panel. These shutters are actuated by compressed air supplied through the rear of the equipment. Substrates are mounted on a holder that can be rotated at variable speeds, and the distance between the substrate holder and the magnetrons can be adjusted to optimize deposition conditions.

Typical procedures to control the equipment

Break the vacuum and open the chamber

This procedure is intended either for cleaning the interior of the main chamber or for removing a prepared sample. To open the main chamber under high-vacuum conditions, the following steps must be followed. First, it is essential to check the barometer to ensure that the chamber is indeed under high vacuum. Currently, nitrogen is used to break the vacuum; therefore, it is important to verify that the pressure indicated on the nitrogen cylinder manometer is at the correct level (typically around 5 psi).

1. First, the turbomolecular pump is turned off by pressing the lower-right button on the control panel.
2. Immediately after turning off the turbomolecular pump, slightly open the nitrogen stopcock to allow nitrogen to enter and help break the vacuum in the pump.
3. Next, wait until the turbomolecular pump's frequency reaches zero or stabilizes at a specific value (typically around 40 to 50 Hz) before switching off the mechanical pump power supply.
4. The chamber latch must be released to prevent over-pressurization of the chamber.
5. Slowly open the vacuum break valve of the main chamber to allow nitrogen to enter and monitor the pressure until it reaches approximately 754 torr.
6. Once the pressure reaches ambient levels, close the air valve and open the chamber door. Ensure that the nitrogen stopcock is also closed.

Produce a high vacuum in the main chamber

To ensure proper thin film deposition, it is necessary to achieve a high vacuum to maintain a clean environment and enable stable plasma generation. The following steps are therefore carried out:

1. It is verified that both the chamber door and the vacuum break valve are fully closed.
2. The mechanical pump power supply is turned on (assuming the vacuum pump is functioning properly). Wait until the pressure in the main chamber reaches approximately 10^{-1} Torr.
3. Next, the turbomolecular pump is activated by pressing the lower-right button on the control panel. The pump frequency should increase until it reaches approximately 1500 Hz.
4. The goal is to reach the lowest pressure limit achievable by the turbomolecular pump, typically on the order of 10^{-8} Torr. However, in most cases, the base pressure is considered acceptable when below 5×10^{-7} Torr.

Prepare a thin film sample

As an example, a multilayer film of Fe and Ti was deposited. The working conditions included a base pressure of 8.5×10^{-7} Torr and a working pressure of 4.5×10^{-3} Torr. The argon flow was maintained constant at 20 sccm throughout the deposition, except during stages when it was turned off to achieve vacuum. Radio-frequency power sources were used, set to 50 W for titanium and 40 W for iron. The substrate rotation was continuously maintained at 20 rpm. The deposition rates were 0.17 Å/s for titanium and 0.15 Å/s for iron. The following steps were performed for the growth of the magnetic multilayer:

1. The positions of the targets and their corresponding sources were recorded: titanium was placed at source 1, and iron at source 2.
2. The thickness of each layer was calculated in the following table based on the deposition time and calibrated deposition rate for each material.

Layer	Material	Time (s)	Rate (Å/s)	Thickness (Å)
1	Ti	231	0.17	39.27
2	Fe	147	0.15	22.05
3	Ti	101	0.17	17.17
4	Fe	147	0.15	22.05
5	Ti	231	0.17	39.27

3. The base pressure should be recorded each time the film deposition process is initiated.
4. The Argon flow is activated by selecting the ‘ON’ option on the control panel touchscreen.

5. The connection between the chamber and the pump is closed using the hydraulic handle until the plasma pressure setpoint is reached.
6. It is verified that the selected source corresponds to the one specified at the beginning of this section.
7. When using the RF source, it is turned on and the power is gradually increased in small increments until the required power for the target material is reached. In this case, the power settings were 50 W for titanium and 40 W for iron. Calibration of the reflected power is performed, ensuring it is zero using the tune and load controls.
8. When using the DC source, it is turned on and the power, voltage, and current settings are configured. Then, the plasma activation button is pressed to initiate plasma generation.
9. Once plasma ignition is observed, the connection between the chamber and the pump is gradually opened using the hydraulic handle until the desired working pressure is reached, which in this case is approximately 4×10^{-3} Torr.
10. The shutter of the target designated for deposition is then opened, and the timer is immediately reset. The process continues by waiting for the specified deposition time.
11. Once the specified deposition time is reached, the shutter is closed immediately.
12. For an RF source, the power is gradually reduced to zero, ensuring that the reflected power remains calibrated at zero. Once the power reaches zero, the RF source is switched off. For a DC source, the shutdown is performed by pressing the turn-off button. After that, the DC source can be switched off.
13. The argon flow is deactivated by selecting the 'OFF' option on the control panel touchscreen.
14. The connection to the vacuum pump is fully opened, and a waiting period of approximately 3 minutes is observed to allow vacuum regeneration. The same procedure is then repeated for the deposition of the next layer.
15. Once the sample deposition is complete, follow the established procedure for opening the chamber while it is under high vacuum.

References

[1] D. M. Mattox, ‘Physical vapor deposition (PVD) processes’, *Metal Finishing*, vol. 100, pp. 394–408, Jan. 2002, doi: 10.1016/S0026-0576(02)82043-8

[2] G. Bräuer, B. Szyszka, M. Vergöhl, and R. Bandorf, ‘Magnetron sputtering – Milestones of 30 years’, *Vacuum*, vol. 84, no. 12, pp. 1354–1359, Jun. 2010, doi: 10.1016/j.vacuum.2009.12.014