

PART 4

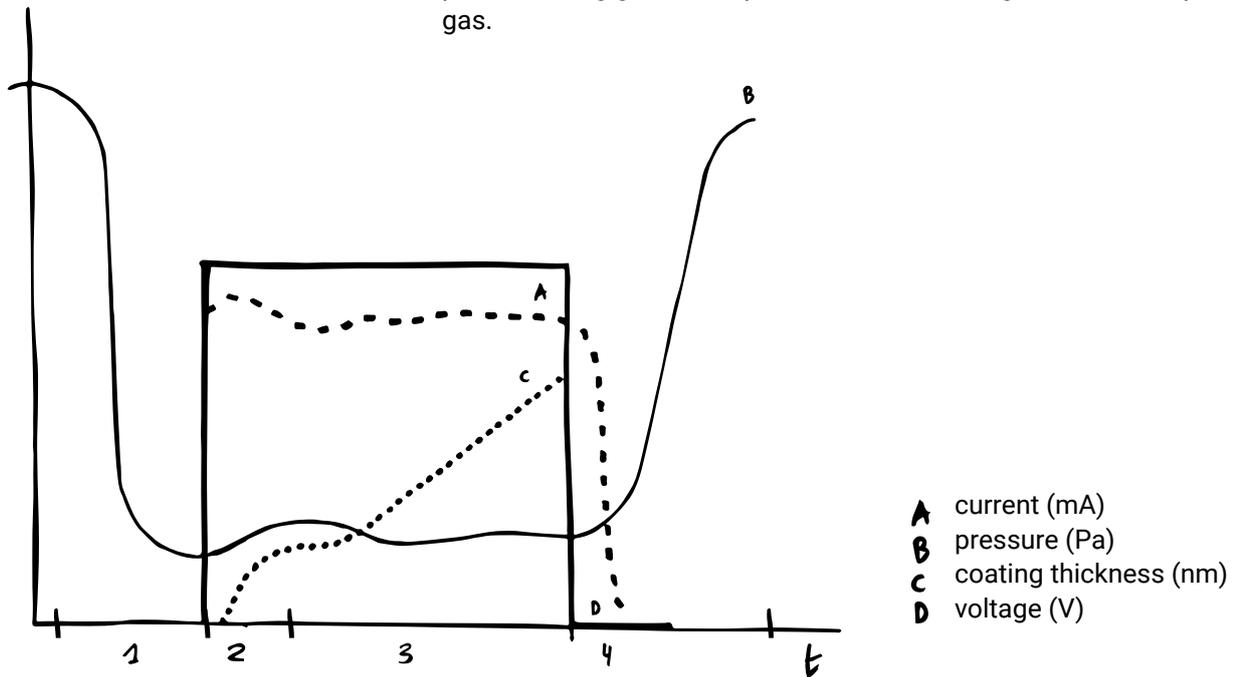
LUXOR A² TECHNOLOGY: SEM COATING MADE SMART AND EASY



INTRODUCTION

In this part we will explain how the basic principles and parameters of sputter coating have been used to develop the LUXOR A² technology, the heart of the new series of LUXOR metal sputter coaters. We will also address the question of how this new technology is further reflected in the design and automation, and what advantages this offers to the operator.

Earlier we discussed the basic flow of a manual sputter coating process using gold as a sputter material and argon as an inert process gas.



It basically consists of the following steps:

1. After evacuating the air and filling the chamber with argon, the vacuum pump brings the chamber to the pre-set target vacuum
2. When the vacuum is reached, the high voltage is switched on and the operator regulates the argon flow creating a sputtering current
3. The sputtering process starts with the set argon flow during a pre-set time
4. When the preset time is reached the sputter coating process stops and the chamber is refilled with air

From this description we can conclude that for the accurate control of a sputter coating process the control of the vacuum and the sputtering current are essential. It is also clear that the lower the sputtering current can be kept, the finer the sputtering result will be.

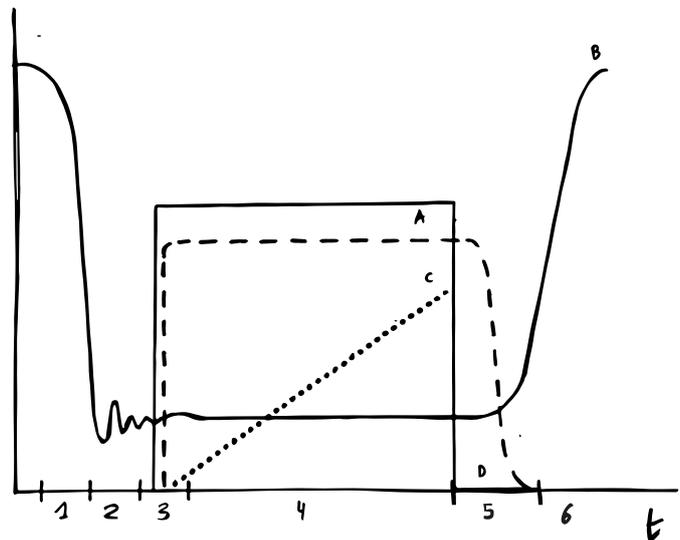
But, the lower the currents are, the more unstable the plasma behaves. A constant low sputtering current and a stable vacuum result in a high quality of the produced coating in terms of homogeneity, coverage, density and grain size.

Other parameters such as the dimensions and layout of the coating chamber and the target material are fixed but also need to be taken into account.

The basic flow of a manual sputter coating process as described above has the important disadvantage that there is no automatic system in place that controls and adjusts the balance between the vacuum applied and the amount of process gas injected. As a result, the sputtering current (which is controlled via the injected amount of process gas), cannot be controlled during the coating process either. A consequence of this is that manual and semi-automatic sputtering coaters must operate at a fairly high sputtering current, to prevent the plasma from becoming unstable or failing during the coating process. And a high sputter current also means less homogeneous coatings with a larger grain size.

WHAT DOES AN AUTOMATIC SPUTTER COATING PROCESS LOOK LIKE?

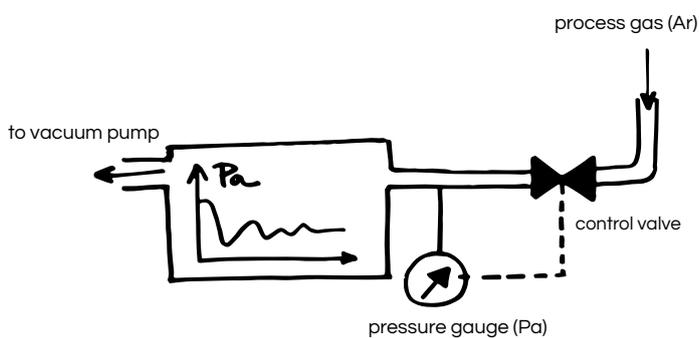
1. The air present is evacuated from the sputtering chamber by the vacuum pump and the chamber is filled/flushed with argon
2. The vacuum pump and vacuum valve that injects the argon bring the chamber to the required target vacuum
3. When the target vacuum is reached, the high voltage is switched on. The combination of vacuum and voltage ignite the plasma, causing current to flow
4. The sputtering process starts and a flow of argon is added to the sputtering chamber to create a constant coating current
5. When the end criterion is reached the sputter coating process stops.
6. The chamber is refilled with air and can be opened.



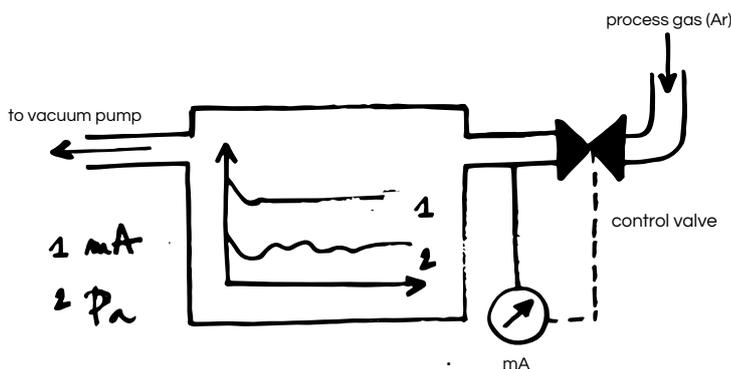
- A** current (mA)
- B** pressure (Pa)
- C** coating thickness (nm)
- D** voltage (V)

A² TECHNOLOGY: WHAT IS IT ALL ABOUT?

In a LUXOR sputter coater equipped with A² technology, after reaching the initial target vacuum, a tuning process is initiated that keeps the target vacuum constant by extremely fast dosing of micro quantities of the process gas (typically argon or air). The A² algorithm controls the vacuum control valve so that it balances the discharge through the vacuum pump and the supply through the vacuum valve (step 2 in the process graph). The initial target vacuum is selected in such a way that, as soon as the voltage will be applied, the dosage of argon will generate the requested coating current.



When the target vacuum remains within the set tolerance for a certain time, voltage is applied causing current to flow, plasma to be created and the sputtering process to start. During the initial stage of the sputtering process, variations in voltage and current occur. Again, the A² algorithm intervenes and controls the vacuum valve to stabilise the target current within a very short time span. During this part of the process the sputter current becomes the crucial parameter, and the vacuum level will possibly start to vary to keep this value constant (step 3 in the process graph)



Keeping the vacuum level constant during this stage would have a negative impact on the sputter current, resulting in a heterogeneous build-up of the metal coating. A² Technology allows the current to be very low and stable, without facing an unstable plasma.

OTHER CONSIDERATIONS

1. Optimal coating region

Due to the design of the plasma chamber and the placement and orientation of the permanent magnetic field, metal target and samples, the plasma created is focused in a specific location directly opposite to the target with a diameter of approximately 60 mm. The thickness, deposition rate and homogeneity of the coating will be constant and perfectly repeatable in this location. Outside this region, the coating becomes thinner, and consistent thickness is not guaranteed. In many cases, samples that are larger can also be coated outside this zone. The goal remains to establish a coating that avoids charging so that samples can be imaged in SEM. If necessary, a slightly thicker coating can be applied in the central area so that the edges are also sufficiently coated.

“LOW AND CONSTANT SPUTTERING CURRENTS PRODUCE SMALLER GRAIN SIZES RESULTING IN THIN COATINGS WITH HIGH DENSITY AND LOW SURFACE ROUGHNESS. THE LUXOR A² TECHNOLOGY ALLOWS THE CURRENT TO BE VERY LOW AND STABLE, WITHOUT FACING AN UNSTABLE PLASMA.”

2. Sample height

The height of the sample has a direct impact on the distance between sample and target. However, due to the orientation of the magnetic field, height variations of the sample of a few millimetres do not affect the thickness of the applied coating.

3. Other variables

Other variables such as the quality and age of the target and the ambient temperature are automatically taken into account at the start of the coating process by the A² algorithm tuning function. This leaves only the sputtering current to be controlled and adjusted through the vacuum level during the plasma coating.

If all the above conditions are met, the deposition rate will be constant, and the use of an accessory such as a thickness gauge will also become unnecessary.

Sample: au 10nm

Fit Results	Optical Model
MSE = 0.855 Roughness = 0.53 ± 0.053 nm Au Thickness = 10.96 ± 0.072 nm n of Au @ 632.8 nm = 0.32415 k of Au @ 632.8 nm = 3.56704	Roughness = 0.53 nm (fit) Layer # 1 = Au, Au Thickness = 10.96 nm (fit) Substrate = Glass, substrate, Substrate Thickness = 1.1000 mm

Sample: au 15nm

Fit Results	Optical Model
MSE = 0.751 Roughness = 1.63 ± 0.046 nm Au Thickness = 17.81 ± 0.062 nm n of Au @ 632.8 nm = 0.35414 k of Au @ 632.8 nm = 3.57743	Roughness = 1.63 nm (fit) Layer # 1 = Au, Au Thickness = 17.81 nm (fit) Substrate = Glass, substrate, Substrate Thickness = 1.1000 mm

Thickness measurement by ellipsometry (the wavelength range from 245 to 1690 nm) on sputtered Au coatings (10 nm and 15 nm) with LUXOR Au on glass

The above measurements show a good consistency between the requested and measured layer thickness of the gold coatings. Moreover, sputter coating is a sample preparation technique where the absolute thickness of the coating is less important than the homogeneity and density of the applied coating.

The ultimate goal is to apply a coating to both planar and 3D structures to prevent charging. Ideally the original topography is minimally affected (due to minimal coating layer thickness) and the highest image resolution is achieved (due to a preferably invisible particle structure of the gold or platinum coating).



TMP: "interior view of a turbomolecular pump (TMP) - source: wikipedia

4. Vacuum control

A stable vacuum in the range of 5 Pa to 10 Pa is usually used for sputter coating applications with gold and platinum. Sometimes, for high resolution applications with platinum, some commercially available sputter coaters use a turbo-molecular pump is used (the so-called "secondary vacuum"), in combination with a pre-vacuum dual stage oil pump (the so-called "primary vacuum").

"BECAUSE OF THE UNIQUE A² TECHNOLOGY WITH WHICH EVERY LUXOR METAL COATER IS EQUIPPED, THE USE OF A TURBO MOLECULAR PUMP IS NOT NEEDED TO ACHIEVE HIGH RESOLUTION PLATINUM COATINGS"

A turbo-molecular pump (TMP) will move faster to or below the requested vacuum level, but the time gain compared to a dual stage oil pump is quite small. On the other hand, the purchase price and maintenance cost of a TMP are high. And although a TMP can achieve a stronger vacuum, the final coating process is done in the range of 5 Pa to 10 Pa, which is easily achievable with a dual stage oil pump. It is especially important to control the vacuum (and therefore the amount of process gas) to keep the sputtering current constant. The unique A² technology with which every LUXOR metal coater is equipped does exactly that. This means that in this case a TMP is not needed.

The use of a TMP is only necessary for high-resolution applications when working with for example chromium (Cr) or Iridium (Ir), where a high vacuum in combination with argon purging reduces the partial pressure of oxygen enough to avoid oxidation.

WHAT HAPPENS AFTER THE PLASMA COATING PROCESS?

After the sputter coating process, the voltage is switched off, a waiting time ensures discharging of the capacitors, and the venting of the plasma chamber starts. Next the vacuum is gradually removed until atmospheric pressure is reached. This process is performed in a controlled manner to prevent sudden pressure differences from blowing away parts of the sample or putting the coating under sudden mechanical stress.



the simple setup of the coating reactor



LUXOR touch screen display

LUXOR A² TECHNOLOGY AND UPSIDE-DOWN DESIGN IN ACTION: FEATURES AND USER BENEFITS

A² technology and full automation

Previously we discussed how LUXOR A² technology works, and how it guarantees reproducible and homogeneous coatings with minimal particle size. However, the A² technology also offers a number of other advantages for everyday use in the lab:

- 1 Due to the generation of very small gold and platinum particles combined with the focusing of the plasma in a specific part of the coating chamber, the use of a rotating sample holder (also for complex 3D structures) or a coating thickness monitoring system is unnecessary. This means no moving parts, a very simple setup of target and samples, and less chance of manipulation errors.

“IN A LUXOR COATER THE USE OF A ROTATING SAMPLE HOLDER OR A COATING THICKNESS MONITORING SYSTEM IS UNNECESSARY . THIS MEANS NO MOVING PARTS, A VERY SIMPLE SETUP OF TARGET AND SAMPLES, AND LESS CHANCE OF MANIPULATION ERRORS.”

- 2 Because of the simple setup of samples and target the operator only needs to enter the desired coating thickness and push “start”. This minimises the need of extensive training for new operators and the risk of manipulation errors that is often related to complex sample preparation methods.
- 3 This level of automation also avoids other common manual interventions such as switching the vacuum pump or voltage, or manually controlling the inlet valve for process gas, which means greater user comfort and less risk of errors.
- 4 The investment, cost of ownership and lab space for a turbo-molecular pump are avoided by using A² technology for coating with gold and (high-resolution) coating with platinum. This means a serious saving in budget and lab space.

THE UNIQUE "UPSIDE DOWN" DESIGN

In LUXOR metal coaters, the samples are mounted upside down in the lid of the plasma chamber, while the target is located at the bottom. Although this may seem a little controversial at first, it is actually a consequence of our "form follows function" approach, and this concept has many advantages



First of all, all voltage and current wires are safely hidden inside the housing of the sputtering device. This, of course, greatly reduces the danger of exposed wires due to frequent opening and closing of the lid, and the risk of electric shock associated with it.

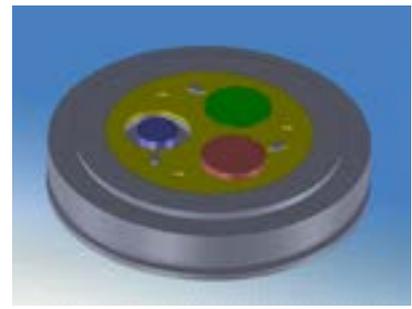
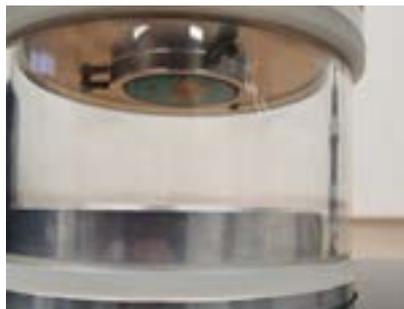
"IN LUXOR METAL COATERS, THE SAMPLES ARE MOUNTED UPSIDE DOWN IN THE LID OF THE PLASMA CHAMBER, WHILE THE TARGET IS LOCATED AT THE BOTTOM. ALTHOUGH THIS MAY SEEM A LITTLE CONTROVERSIAL AT FIRST, THIS CONCEPT ACTUALLY HAS MANY ADVANTAGES."

Next, the sample loading station is actually the lid of the coater, and is easily accessible which means samples can be inserted or removed without the need for special pliers or tweezers. This not only simplifies daily use, but also speeds up productivity.



The loading station (or lid) of the LUXOR coater is equipped to work with standard pin stubs with a 3.15mm diameter and either 8 or 9.5mm pin length.

However, LUXOR coaters can also work with mounted/embedded samples or cylinder stubs of different diameters as used by a number of SEM suppliers.



technical drawing of the sample holder that accommodates up to 3 cylindrical samples of different diameters

The top-loading design ensures that loose particles are removed at the start or during the coating process, and not during the loading or application of vacuum in your microscope. In this way, contamination of the electron optics is avoided, and the column of your SEM is optimally protected.

LUXOR SEM COATING MADE SMART AND EASY

LUXOR metal coaters are used extensively in industrial and academic electron microscopy labs worldwide where image quality and high resolution imaging are of the utmost importance. The revolutionary A² coating technology combined with full automation and the unique upside down design have turned the LUXOR SEM coaters into an indispensable sample preparation tool in today's SEM lab.



LUXOR

APTCO TECHNOLOGIES NV
NIEUWE STEENWEG 20 BUS A
9810 NAZARETH
BELGIUM

INFO@LUXOR-TECH.COM