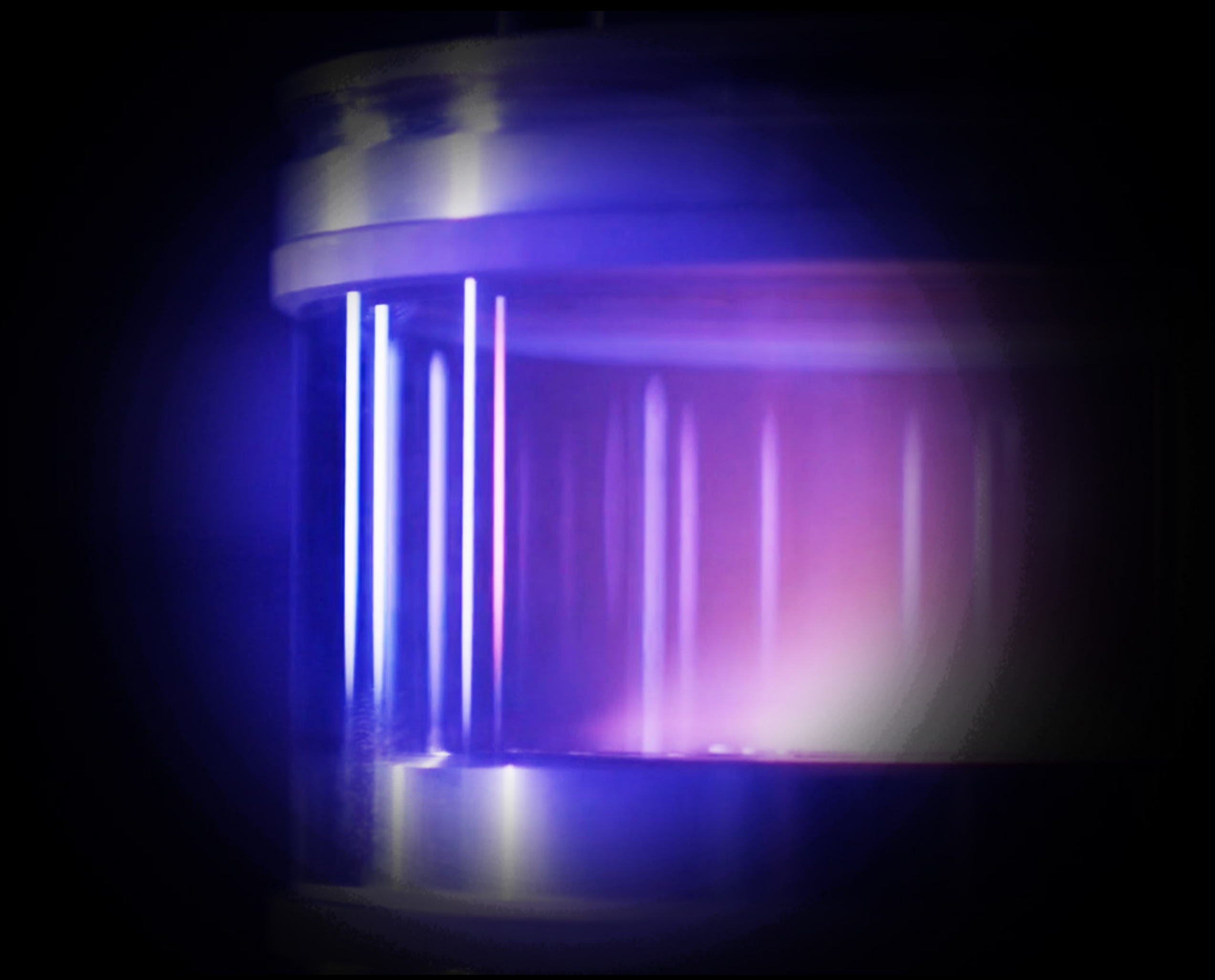
PART 3

HOW VARIOUS PARAMETERS INFLUENCE THE SPUTTER COATING PROCESS



OPERATING PRESSURE

A sputter coating process is performed in a vacuum. In general, the use of a lower working pressure (better vacuum) gives a better coating quality. Typical values of working pressure during sputter coating are between 2 x 10-2 mbar (2 Pa) and 2 x 10-1 mbar (20 Pa). Many commercially available sputter coaters use a dual stage rotary pump for the entire process.

"TURBO MOLECULAR PUMPS ACHIEVE THE REQUIRED VACUUM MORE QUICKLY AND ARE EASIER TO REGULATE, BUT ARE MORE EXPENSIVE TO BUY AND MAINTAIN"

These pumps can reach an ultimate vacuum of 10-3 mbar (0.1 Pa). Sometimes such a pump is used in combination with a turbo-molecular pump, which can reach an ultimate vacuum of up to 5 x 10-5 mbar (0.005 Pa). In this case one speaks of a primary vacuum for the dual stage rotary vane pump and secondary vacuum for the turbo pump. Turbo pumps achieve the required vacuum more quickly and are easier to regulate, but are more expensive to buy and maintain. In addition, the effective working pressure for sputter coating is more in the working range of the dual-stage rotary vane pump.

INERT GAS

As discussed earlier, the free electrons and ions of an inert gas are attracted to the cathode and anode in a cathode tube arrangement. With increasing voltage, electrons collide with gas atoms. When the voltage exceeds a critical value self-sustaining glow discharge occurs. During this phenomenon, the cathode is bombarded by gas ions, causing plasma sputtering.

The gas used is preferably inert (it will not decompose in the glow discharge), and has a relatively high atomic weight. Argon, with an atomic weight of 39.95, is an ideal candidate. It is freely available from a large number of manufacturers, and inert. The high-purity variant known as N4.8 (Zero Grade) 99.998% purity is ideally suited for high resolution imaging sputter applications.

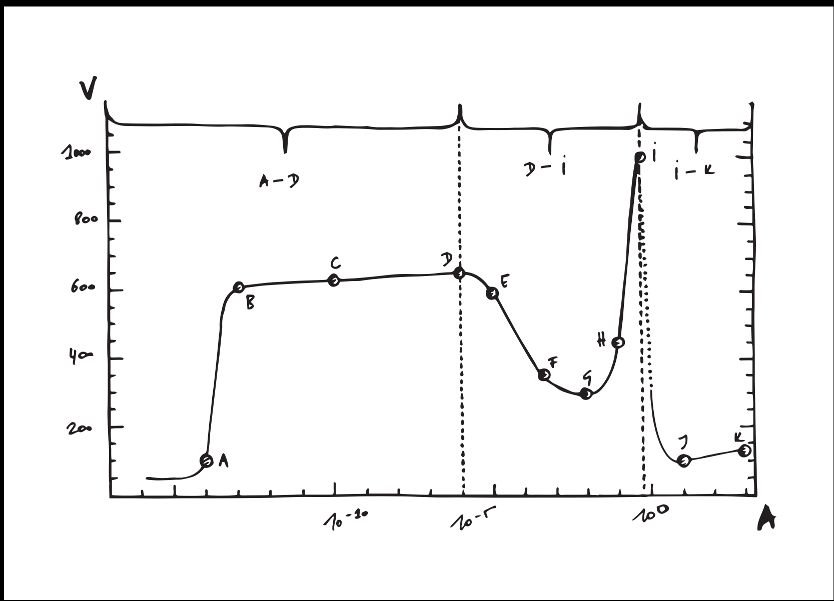
pressure regulator with 2 pressure dial gauges. The left gauge shows the pressure in the gas cylinder, while the right gauge allows for a fine adjustment towards the sputter coater

“SOMETIMES NITROGEN, WHICH IS AVAILABLE IN MANY LABORATORIES, IS USED AS A PROCESS GAS, OR EVEN AMBIENT AIR WHEN WORKING AT LOW MAGNIFICATIONS”

However, other gases are also used for sputter coating with gold where the magnifications are moderate (< 50.000x) and there is no danger of a reaction of decomposing gas with the samples. Sometimes nitrogen is used, which is available in many laboratories, or often just ambient air.

TARGET VOLTAGE

At low voltages, only the free ions and electrons will be attracted to the electrodes in a cathode gas tube (A). As voltage is increased some ionisation is produced by collision of electrons with gas atoms (B-D). If the voltage is increased further then a self-sustaining glow discharge occurs - characterised by the typical luminous glow. The target value for sputter coating has been reached (D-H). At higher voltages, an unstable glow discharge occurs, which may eventually lead to arc discharge (I-K).



typical voltage-current characteristics of electrical discharge

Typical values for the target current in sputter coating vary from a few hundred volts to a few thousand volts. These values depend mainly on the design of the sputter chamber and the target material used. In general, sputter coating at a low target voltage is preferred because of the lower energy input and heat generation.

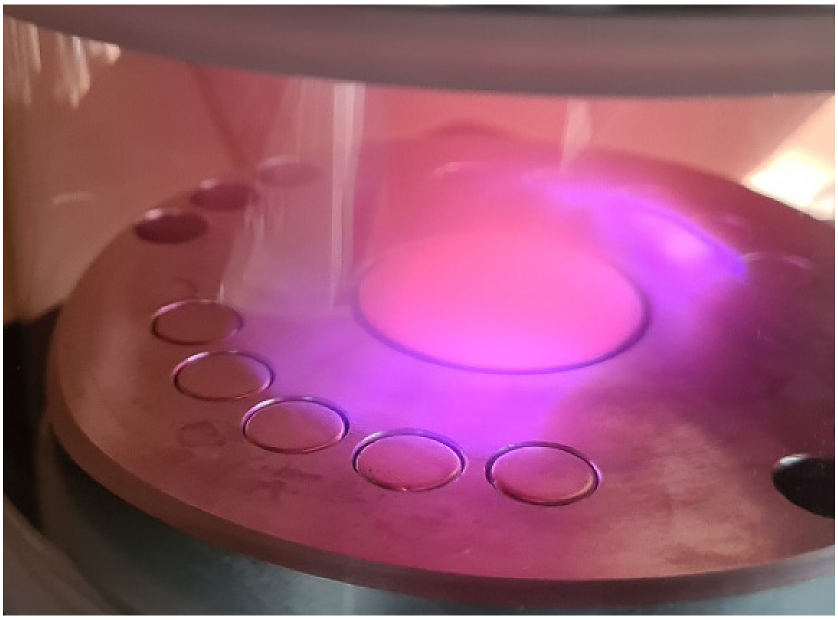
SPUTTERING CURRENT

In sputter coating processes, sputter current values in the range of 0 mA to several tens of mA are generally used. Sputtering with low current values is generally preferred, because it leads to a smaller grain size of the sputtered target material, which improves the quality of the produced coatings in terms of homogeneity and coverage.

Low sputtering currents produce smaller grain sizes resulting in thin coatings with high density and low surface roughness. Also, when coating three-dimensional structures such as porous materials and nanofiber samples, it is important to coat with a smaller grain size so that hollow spaces and gaps or heaps of material are coated.

"LOW AND CONSTANT SPUTTERING CURRENTS PRODUCE SMALLER GRAIN SIZES RESULTING IN THIN COATINGS WITH HIGH DENSITY AND LOW SURFACE ROUGHNESS"

Sputter coaters with an algorithm that monitors and adjusts the current during the coating process are able to coat with low current values from 1 to 5 mA, which contributes greatly to the quality of the coating produced. In general, manual coaters, where the current value is only initially set by the operator and is not further adjusted, need to work with higher current values (typically 10 mA - 30 mA) to prevent instabilities and fluctuations from causing the coating process to fail. A direct consequence of this is that manually controlled coaters also produce poorer quality coatings and can only be used for a limited number of applications.



coating process in a LUXOR coater. Note the typical donut shape of the plasma and the circularly positioned magnets

SPUTTERING TIME

The time required to create a metal coating depends mainly on the desired coating thickness and the coating current used.

A higher current causes a faster increase of the coating thickness, but often also leads to a less homogeneous or dense coating, making it necessary to apply a thicker coating in order to prevent charging. This in turn increases the coating time.

When using a low coating current, the growth rate of a gold or platinum coating is typically 0.5 nm/min to 3 nm/min, with a gold coating typically being formed up to 2 times faster. In practice, this means that a coating process with a homogeneous and dense gold or platinum coating of 2 to 10 nm takes approximately 2 to 5 minutes. If we add to this the preliminary vacuum step and venting at the end of the process, a coating process typically takes 5 to 8 minutes. Multiple samples can be coated within this timeframe, so this sample preparation step should never be a bottleneck for SEM analyses.

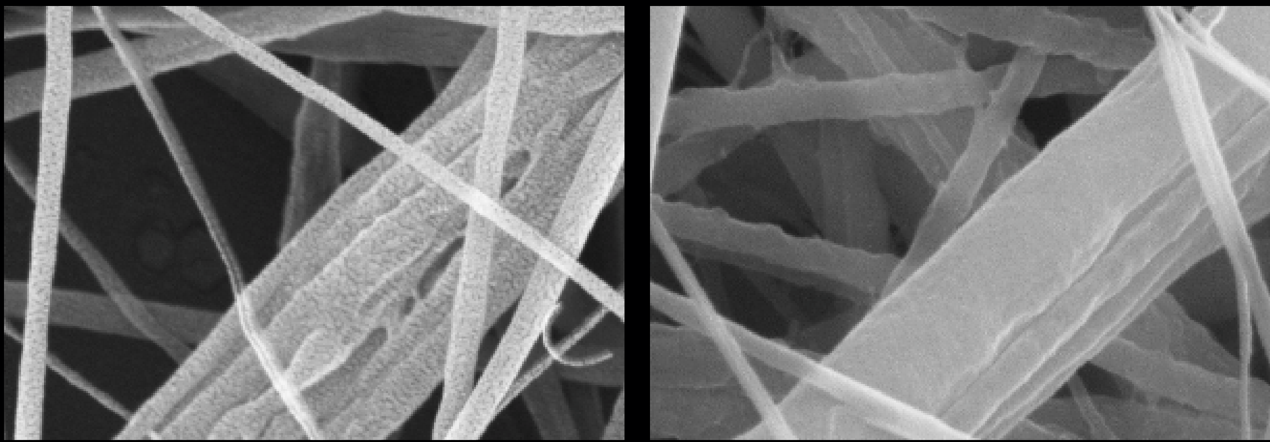
TARGET MATERIAL

To coat SEM samples with a sputter coater, a large number of conductive metals can be used as target materials. These target materials have a (much) higher secondary electron (SE) yield than the non-conducting sample materials, resulting in a much sharper and more detailed image.

Typically targets have a thickness of 0.1 to 0.5 mm and a diameter of 20 to 80mm. Because most sputter coater systems have a system of permanent magnets incorporated to avoid heat generation (see below - “Avoiding heat generation”), the target is mainly consumed in a certain zone, the so-called “racetrack”. Because of this preferential consumption, targets must be replaced when only a limited part of the target material has been effectively consumed, and today preference is increasingly given to targets with smaller diameters of 30 or 40 mm.

- **Gold (Au)** can be used for low and medium magnification applications, typically up to a few 10.000x magnification. At higher magnifications we can see the grain size of the coating. Another advantage of gold as a coating material is that it can be used in combination with ambient air as process gas, whereas coating with other target materials often has to be done in an inert gas like argon. The typical grain size after coating with gold is about 8-12 nm. Typically, gold coatings of 5 - 15 nm thickness are applied

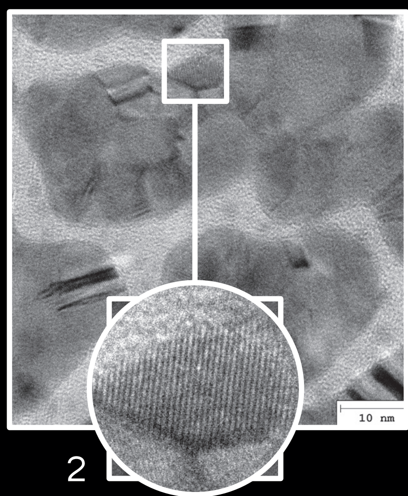
LUXOR Gold and Platinum metal coater targets. With these targets, the target material is pre-mounted on a holder, making them easy to mount or replace in the coater

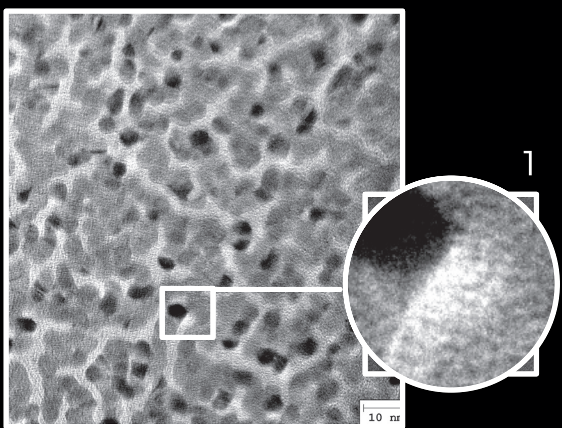


comparison of a 10 nm gold coating (left) and a 5 nm platinum coating (right) on a nanofiber sample at 100.000x magnification. Note that the gold particles become visible in this range of magnification, making gold less suitable for high resolution SEM imaging

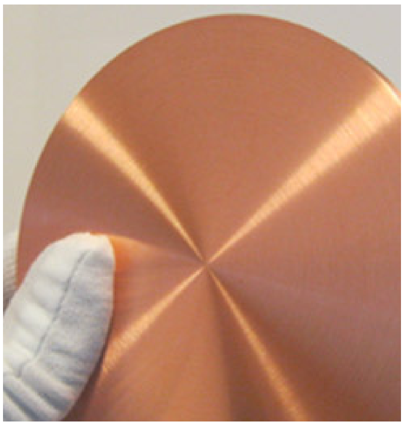
"THE GRAIN SIZE AFTER COATING WITH GOLD IS TYPICALLY ABOUT 8-12 NM. PLATINUM COATINGS HAVE A FINER GRAIN SIZE THAN GOLD AND ARE THEREFORE MORE SUITABLE FOR HIGHER RESOLUTION IMAGING."

- **Platinum** has a finer grain size than gold (2-3 nm) and is therefore more suitable for high resolution applications. 50.000x magnification is a practical limit above which Platinum is recommended. The sputtering rate for platinum is lower than for gold, but due to the smaller grain size one can work with thinner layers (of a few nm) making the entire coating process comparable. Platinum tends to be sensitive for stress cracking when oxygen is present, as may be the case in porous samples. That is why coating of platinum in a high purity gas that doesn't contain oxygen is preferred.





comparison of high-resolution TEM images (JEOL 2100Plus) of a 5 nm gold coating (top) and a 2 nm platinum coating (left). Notice the typical gold and platinum "islands" with the platinum coating showing smaller islands. Detail 1: Around the islands smaller particles of metal are also visible. These form a thinner coating that also prevents charging. Detail 2: at some regions crystal-like structures and lattices are also visible. (images courtesy of Dr. Benndorf and Prof. Oeckler, Leipzig University)



Au/Pd target

- **Gold/palladium (Au/Pd)**, with ratios 60/40 and 80/20, is often recommended as an alternative to gold that yields a smaller grain size. However, this is only the case when it is used in high-vacuum coating systems. In SEM sputter coaters, the difference is hardly visible, and a grain size of 4-8 nm is reported against 5-15 nm for pure gold. Moreover, it is less suitable for heat-sensitive materials and for EDS analysis due to the presence of a second material and thus a second set of peaks.

- **Silver (Ag)** is a good alternative for gold with a slightly lower cost and comparable or slightly larger grain size. The disadvantage is that in the presence of halogens it has a coarser grain size, and it also degrades faster in the presence of halogens, making it less suitable for long-term storage.

- **Iridium (Ir) and Chromium (Cr)** are both used for ultra-high resolution imaging in Field Emission Scanning Electron Microscope (FESEM). For both materials a grain size of 1-2 nm is reported. Both materials can currently only be used in combination with a turbo pumped, high vacuum sputter coater. Both metals should always be used in combination with argon since, especially for chromium, oxidation of the coating occurs in oxygen. Iridium is preferred today because the samples do not need to be kept under high vacuum, unlike those coated with chromium.

- **Carbon (C)** is not a material that can be applied in a thin layer via a DC sputter coater. It is applied by a carbon evaporator and is sometimes preferred when in EDS analysis it is necessary that none of the above materials are present.

DEPOSITION RATE

The deposition rate of a target material on a sample depends on a number of parameters, such as the sputter current, sputter voltage, pressure (vacuum) in the sample chamber, distance from target to sample, sputter gas and target material. This makes it impossible for manual sputter coaters, where a number of values are set manually at the start of the process and are not further tuned during the process, to determine a correct deposition rate or an exact coating thickness during or at the end of the process. Hence the use of a thickness measurement device is necessary.

setup with 10 circular permanent magnets
in the base of the LUXOR coater

The sputter current is the most important parameter for determining the deposition rate. Automatic sputter coaters monitor and control the sputter current at relatively low values (a few mA) giving deposition rates from 0.5 nm/min to 3 nm/min. With all other parameters closely monitored or established at the start and during the coating process, there is no need to use a thickness measurement device in the coating chamber.

"WITH ALL OTHER PARAMETERS CLOSELY MONITORED OR ESTABLISHED AT THE START AND DURING THE COATING PROCESS, THERE IS NO NEED TO USE A THICKNESS MEASUREMENT DEVICE IN THE COATING CHAMBER OF A LUXOR COATER"

Manual sputter coaters operate at higher sputtering currents (typically several tens of mA) resulting in less homogeneous, less dense and often thicker coatings of several tens of nm, with a consequent loss of sample surface detail in the SEM images.

setup with 10 circular permanent magnets in the base of the LUXOR coater

AVOIDING HEAT GENERATION

During the glow discharge, high-energy electrons are generated that can reach the sample and cause unwanted heating. When sputter coating for SEM applications, it is desirable to obtain a small grain size, homogeneous coating and as little heat generation as possible. In sputter coaters, the problem of heat generation is largely avoided by incorporating a system of permanent magnets that conduct the electrons away from the sample and contain the plasma in a doughnut shape above the target surface.

This results in an increased ion yield and sputtering efficiency, but it also means that the target is mainly consumed in a certain zone, the so-called "racetrack", and that replacement becomes necessary as soon as the plasma has "eaten" through the target. In fact, it is sufficient to work with a relatively small target (e.g. 30 or 40 mm), as long as the orientation and construction of the sputtering cell is such that a sufficiently large zone in which the samples are located is covered with a homogeneous coating of constant thickness.

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.

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