LANXOR

INTRODUCTION TO METAL COATING TECHNOLOGY FOR ELECTRON MICROSCOPY

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ABOUT US

Aptco Technologies is a manufacturer of measurement instruments and testing equipment for academic and industrial labs. Its LUXOR series of SEM metal sputter coaters uses the unique A^2 coating technology and algorithms to obtain thin and homogeneous metal coatings in a fully automated and user-independent manner.

INTRODUCTION

This document is intended to provide the reader with insights into the sputter coating sample preparation technique that is used extensively in electron microscopy (EM) labs worldwide. Metal sputter coating not only prevents sample charging by improving electrical and thermal conduction, but also improves secondary electron emission, reduces electron beam penetration in the sample, providing better edge resolution, and offers a better protection of electron beam sensitive samples.

Although sputter coating is well known and accepted as a sample preparation technique for electron microscopy, the fine details of this technology are not always well understood. This document gives you an insight into the different aspects, basic principles and parameters of sputter coating.

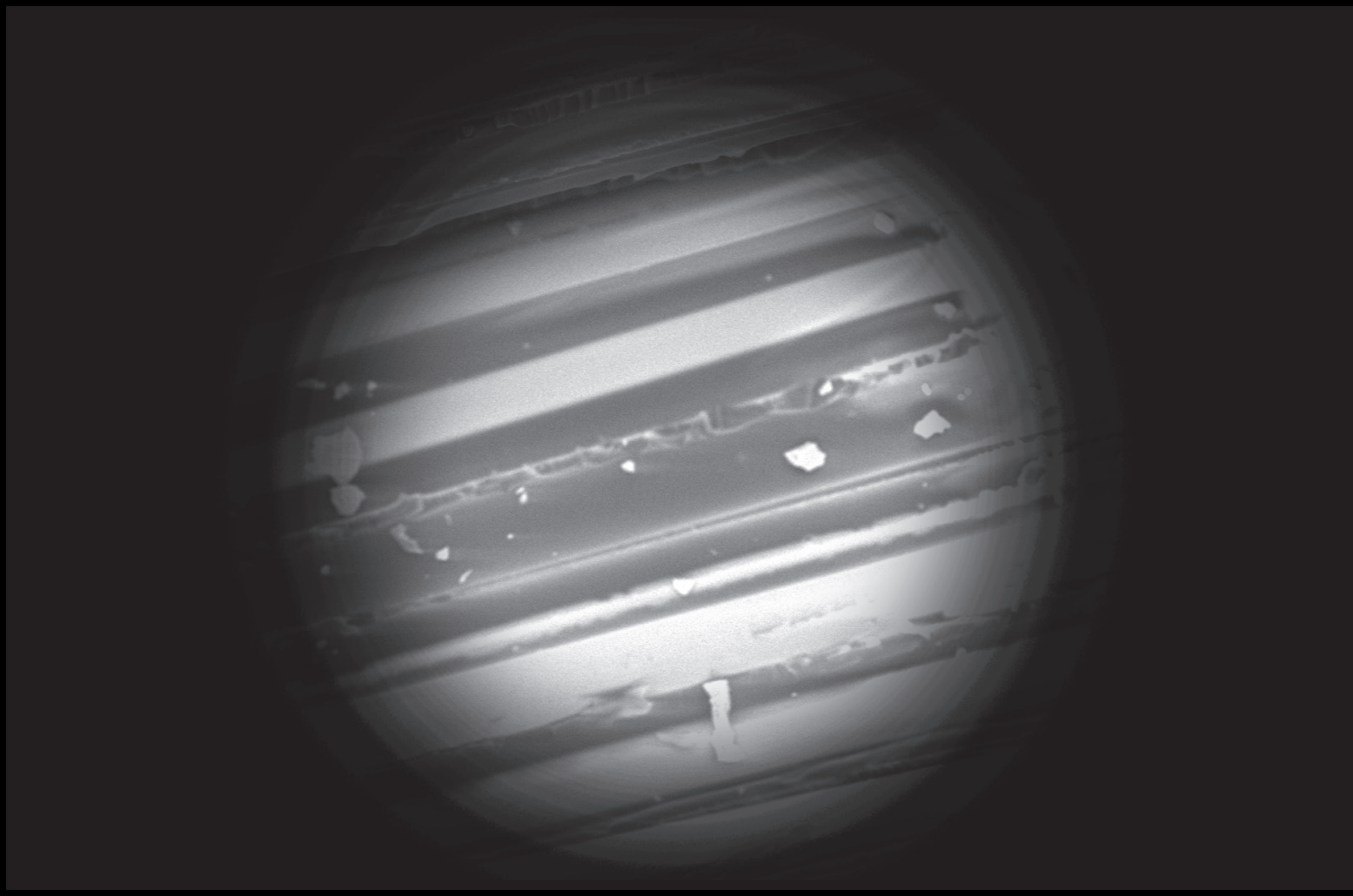
Next you can read how the LUXOR engineers have used and adapted these elements to develop the new generation of LUXOR metal sputter coaters, and how the A^2 unique technology that forms the heart of the new LUXOR series of metal sputter coaters guarantees thin, high quality coatings that allow you to get the best images from your SEM.

The content of the 4 chapters looks as follows:

Part 1: Charging effects in electron microscopy, and how to avoid them
Part 2: Basic principles of sputter coating
Part 3: How various parameters influence the sputter coating process
Part 4: LUXOR A² technology: SEM coating made smart and easy

PART 1

CHARGING EFFECTS IN ELECTRON MICROSCOPY, AND HOW TO AVOID THEM

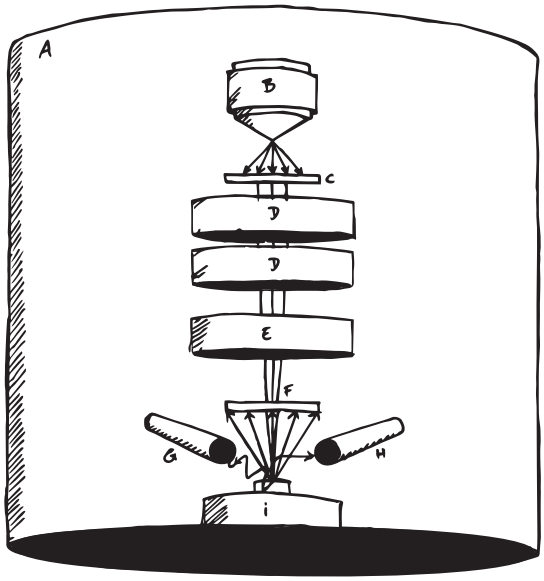


WHY USE METAL COATING IN EM

In an electron microscope, samples are placed in a chamber under vacuum and then fired upon with an electron beam.

As a result, non- or weakly conducting samples are electrically charged, because the number of emitted electrons is different (larger or smaller) from the number of incident electrons at certain locations of the sample. This effect is called “charging”, and it causes abnormal image contrast.

To illustrate the effects of charging and the different methods to control it, we looked at uncoated and coated tissue paper in an EM.



A vacuum chamber
B electron gun
C anode
D electron optics (magnetic lenses)
E scanning coils
F backscattered electron detector
G x-ray detector
H secondary electron detector
i sample stage

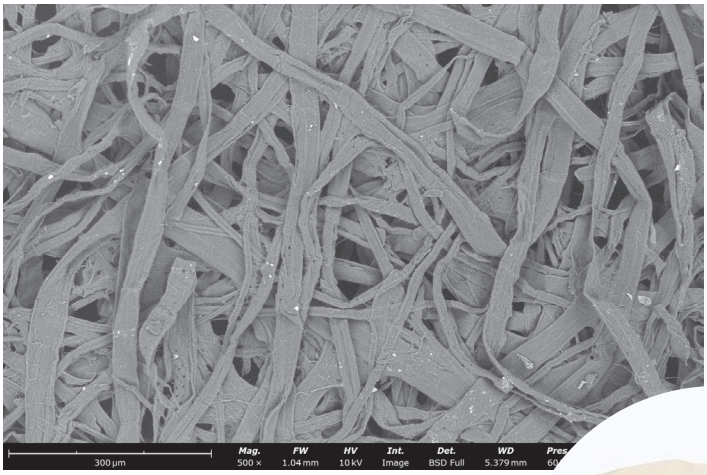
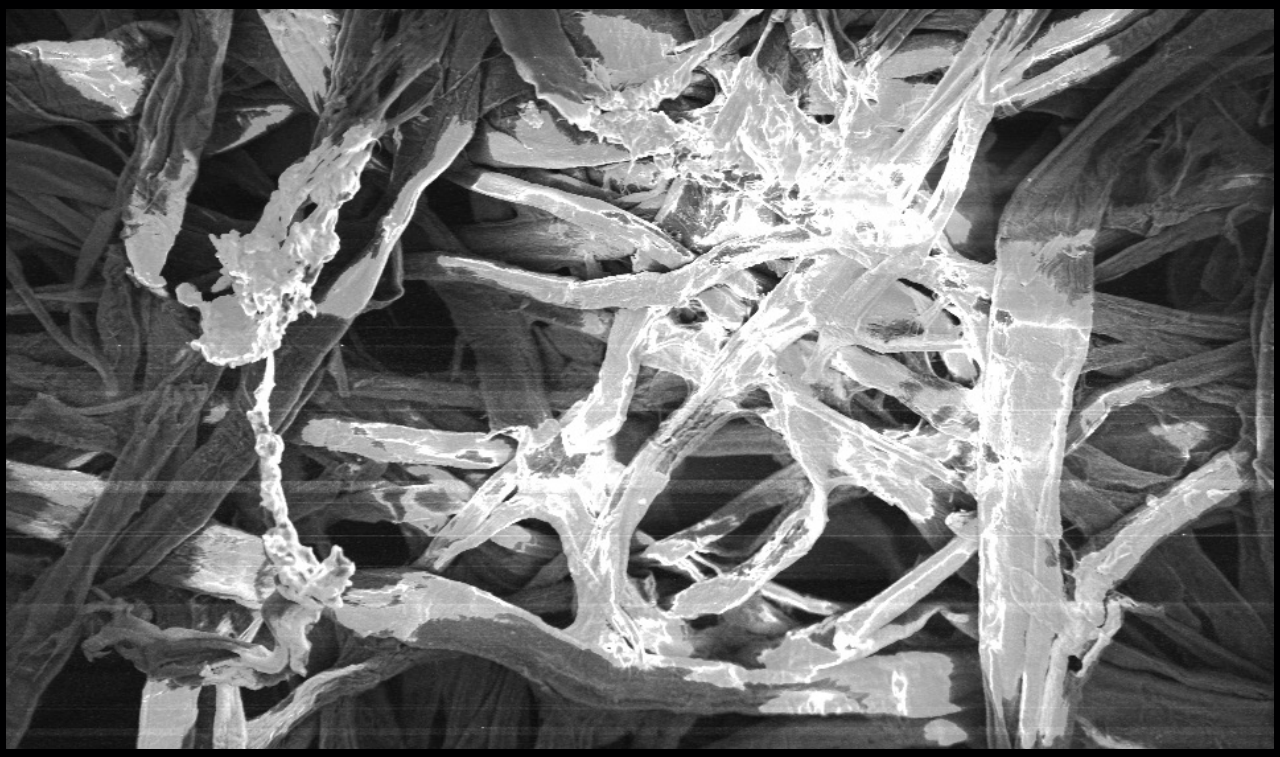


image of tissue paper in an electron microscope

tissue paper samples on 12 mm aluminium sample holders fixed with double sided carbon tape



extreme charging of tissue paper. Notice zones where the sample seems to melt, and lines in the scanning direction

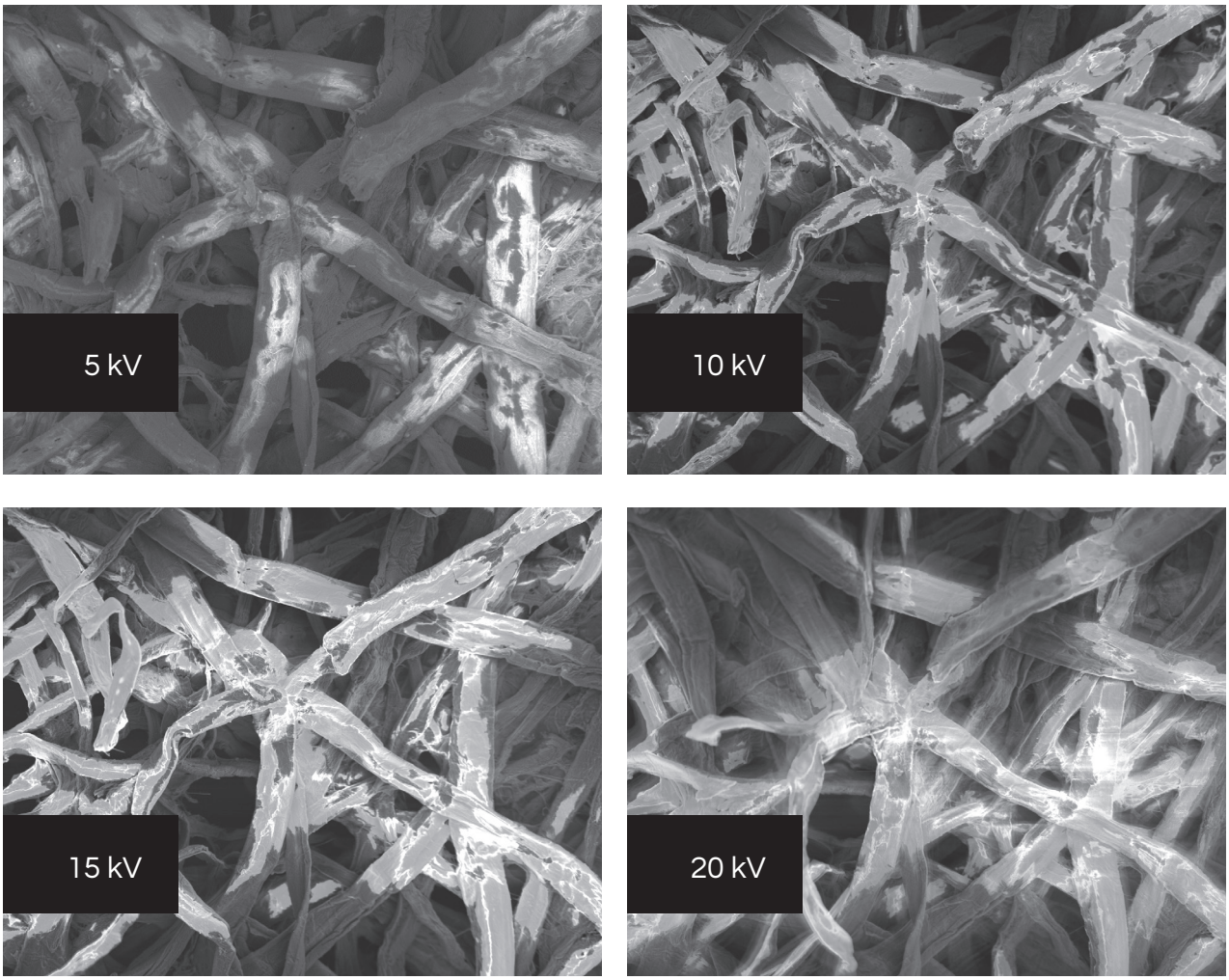
Different charging effects can occur, depending on the degree of charging, and they can result in one or more of the following phenomena:

- Certain samples (e.g. powders or thin fibres) may melt, disintegrate or break up as a result of the electrical charge
- Images may show extremely light and dark areas. This effect is sometimes combined with lines in the scanning direction of the electron probe
- Image distortion, blurring or drift
- Lower image contrast, resulting in less topographical information

On the other hand, electrically conductive samples will pass excess electrons to the underlying conductive material of the sample holder in a scanning electron microscope (SEM), preventing charging.
Charging of samples can be controlled by a number of methods. Here we will briefly discuss four of these methods and look how image quality changes as they are applied.

1. REDUCE CHARGING BY WORKING AT LOWER ACCELERATION VOLTAGES

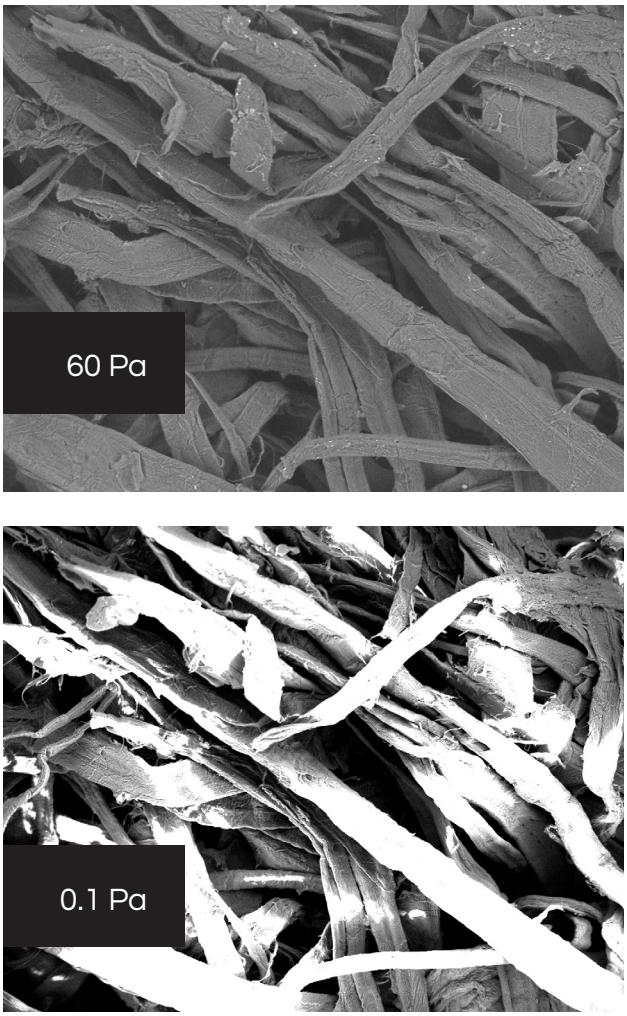
Firstly, charging can be partially avoided by reducing the speed at which electrons hit the sample. This is possible by working at lower acceleration voltages (unit is kV). At high acceleration voltages, electrons will penetrate deeper into the sample and will have more difficulty escaping. At lower acceleration voltages, more electrons can escape, so that less charging occurs. A disadvantage of working at lower acceleration voltages, however, is that the image quality in most cases also decreases. Moreover, for some samples charging already occurs at low acceleration voltages, making imaging very difficult or impossible in any case.



SEM imaging of tissue paper at 5, 10, 15 and 20 kV. Magnification 1.000x, 0.1 Pa, BS detector

2. REDUCE CHARGING BY WORKING AT REDUCED VACUUM

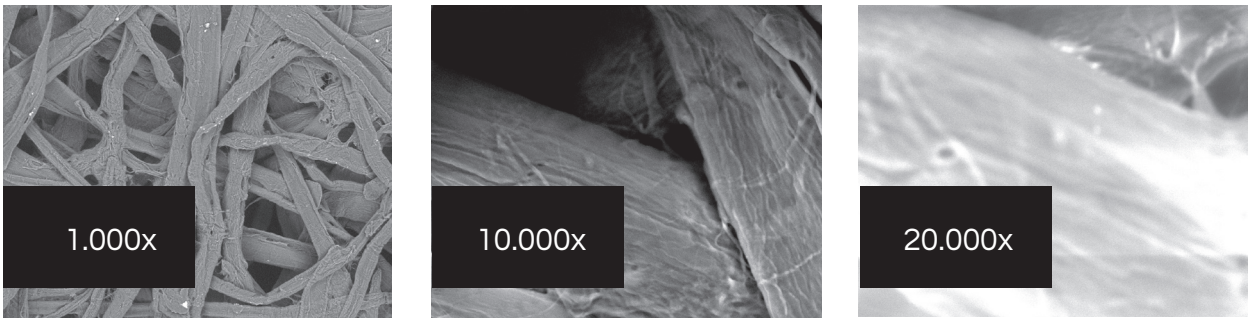
Another method to prevent charging is to work in a reduced (less strong) vacuum. This is done by introducing a gas, air or water vapour into the sample chamber. The gas molecules contact the charged sample surface, become negatively charged by collecting an electron, and then discharge that electron somewhere in the grounded chamber. A disadvantage of having a gas entering the chamber is that there is an interaction of the electron beam with the gas, which can also lead to reduced image quality.



SEM imaging of tissue paper at 60, 10 and 0.1 Pa. Magnification 1.000x, 10 kV, BS detector

3. REDUCE CHARGING BY WORKING AT LOWER MAGNIFICATIONS

Some samples that are poor conductors can still be imaged at low magnifications. At higher magnifications, this becomes more problematic, even when using appropriate acceleration voltage and pressure values.



effect of magnification on charging of tissue paper: to reduce charging effects the pressure was set at 60 Pa while a 10 kV acceleration voltage was used. With these settings, acceptable image quality is still available at a 1.000x magnification. At 10.000x magnification the sample starts deforming and slightly charging, while at 20.000x magnification the sample is obviously charging

4. REDUCE CHARGING BY METAL SPUTTER COATING

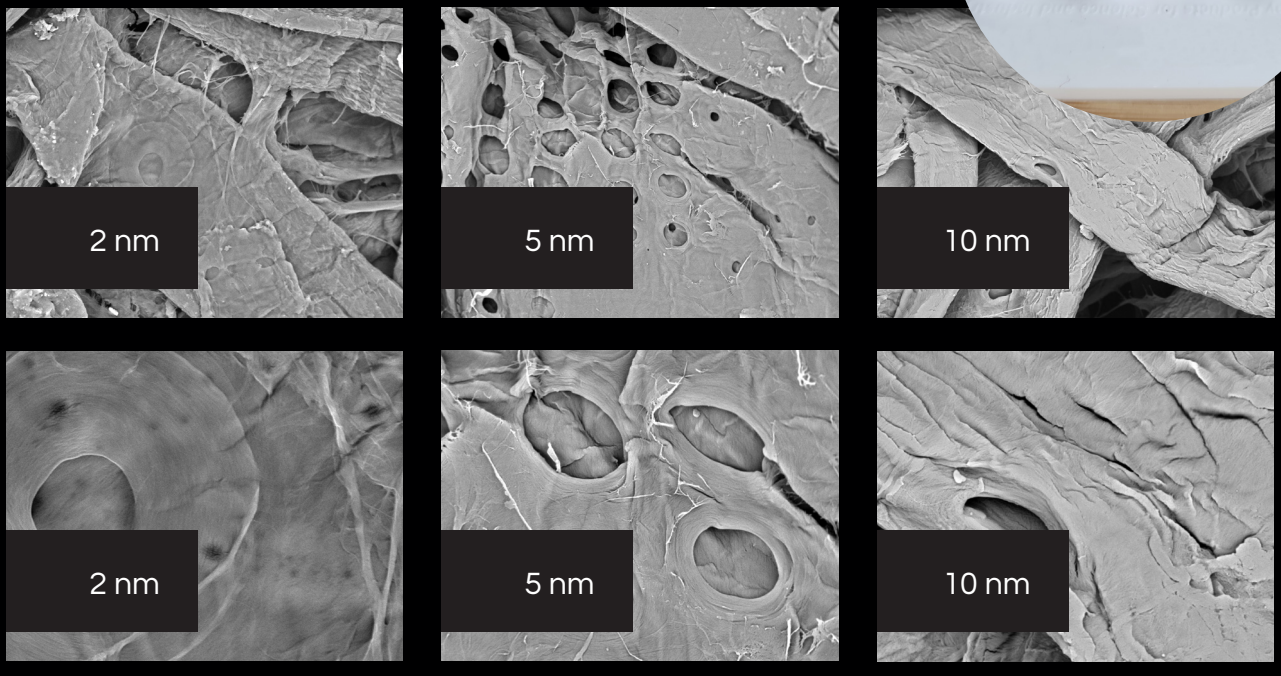
The best image quality of non-conducting materials is obtained by metal sputter coating. A metal sputter coater deposits a very thin layer (typically 1 nm to 10 nm) of conducting material (most commonly gold or platinum) over the surface of the sample.

" THE BEST IMAGE QUALITY OF NON-CONDUCTING MATERIALS IS OBTAINED BY METAL SPUTTER COATING."

Excess electrons in the sample then have a path to ground, so charging is essentially eliminated.

Metal coating can be problematic when used in combination with energy-dispersive X-ray spectroscopy (EDS) for element analysis, because the element used for coating will also show up in the EDS spectrum. However, this signal tends to be small because of the very thin layer applied, and most EDS software modules include subtraction of spectra that contain only the metal coating. Reducing the acceleration voltage or working at lower vacuum also have a negative effect on the accuracy of the EDS signal. Moreover, sputter coated samples are easy to image, so this method is often preferred, especially by non-expert users and high throughput labs that analyse large amounts of samples.

from left to right 2 nm, 5 nm and 10 nm gold coated paper tissue samples on aluminium stubs



5.000x (top row) and 20.000x (bottom row) magnification with 2 nm, 5 nm and 10 nm gold coating thickness (pressure = 0.1 Pa and acceleration voltage = 10 kV). Note that the 2 nm coating avoids sample charging while showing more surface topography details at higher magnifications

PART 2

BASIC PRINCIPLES OF SPUTTER COATING

INTRODUCTION TO THE SPUTTERING PROCESS

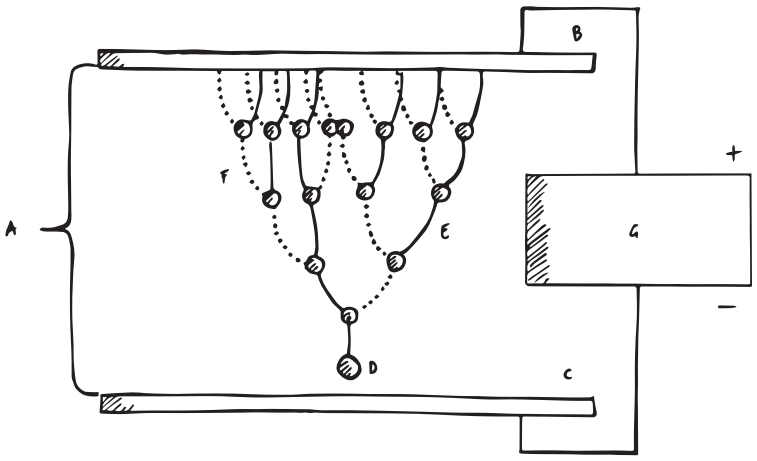
When a metal surface is hit by heavy particles with high speed, decomposition and abrasion of the surface material occurs.

We are talking about “sputtering” when this process occurs by a gaseous glow discharge between a cathode and an anode. In this setup, the surface, which is called the “cathodic target”, is hit by ions generated from an inert gas that has a relatively high atomic weight. The target material is commonly gold or platinum, while the gas is commonly argon.

As a result, a deposition of microscopic target material “particles” will occur in all directions. This material will form a coating on the surface of the sample.

One of the elements that is crucial for understanding the basic principles of sputter coating is understanding the principle of gaseous glow discharge, as it is applied in gas discharge lamps. Electric discharge in a gas appears when it is ionised by applying a voltage that is sufficient to cause electrical conduction in the gas.

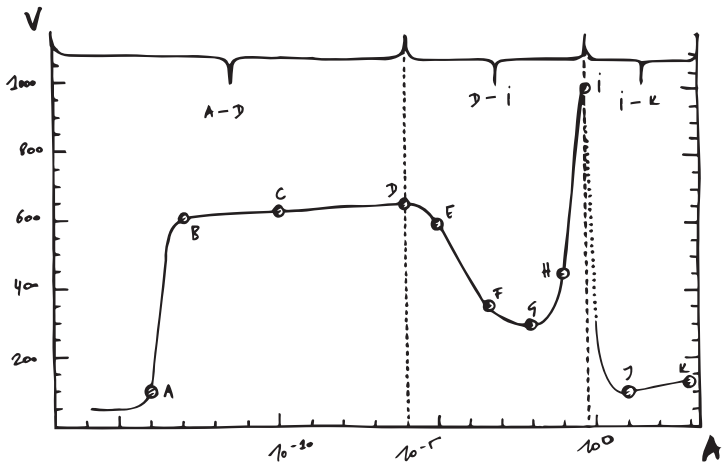
A electric field
B anode
C cathode
D original ionisation event
E ionising electron path
F liberated electron path
G DC voltage source



visualisation of a Townsend Avalanche

This phenomenon, which is typically observed in a gas-filled tube containing electrodes, is known as **Townsend discharge**: when a critical value of electric field strength is reached, a sustained multiplication of electron flow by ion impact occurs.

As the electric field is increased various phases of discharge are encountered:



A random pulses by cosmic radiation
B saturation current
C Townsend avalanche
D self-sustained Townsend discharge
E unstable region: corona discharge
F sub-normal glow discharge
G normal glow discharge
H abnormal glow discharge
i unstable region: glow-arc transition
J electric arc
K electric arc

image: Typical voltage-current characteristics of electrical discharge

The A-D region is called dark discharge; there is some ionisation, but the current is very low and there is no significant amount of radiation produced.

The D-G region exhibits a negative differential resistance

The G-I region is a region of glow discharge; the plasma emits a faint glow that occupies almost all the volume of the tube; most of the light is emitted by excited neutral atoms.

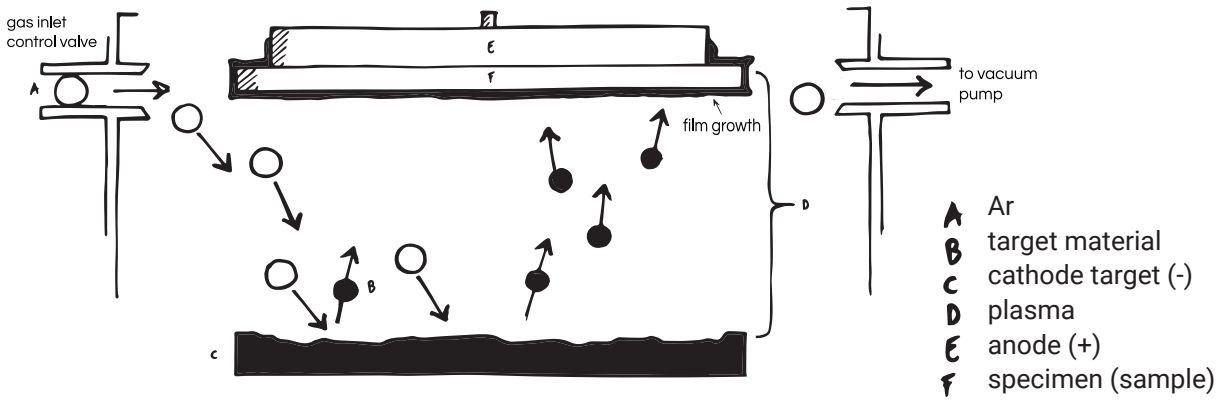
The I-K region is a region of arc discharge; the plasma is concentrated in a narrow channel along the centre of the tube; a great amount of radiation is produced.

The voltage required to initiate and sustain a glow discharge is dependent on the pressure and type of gas. Once the condition for a sustained discharge is met, the tube exhibits the characteristic glow discharge. It has been established that free ions and electrons are attracted to opposite electrodes producing a discharge.

Under conditions of glow discharge in an argon filled tube, an ion bombardment of the cathode target material by the relatively high atomic weight argon ions will occur. This will result in the decomposition of the cathode material and is termed plasma sputtering, with the subsequent deposition of the sputtered material forming a coating of the original cathode material on the surface of the sample and sputter chamber.

HOW DOES A SPUTTER COATER WORK, AND WHICH PARAMETERS INFLUENCE THE PROCESS?

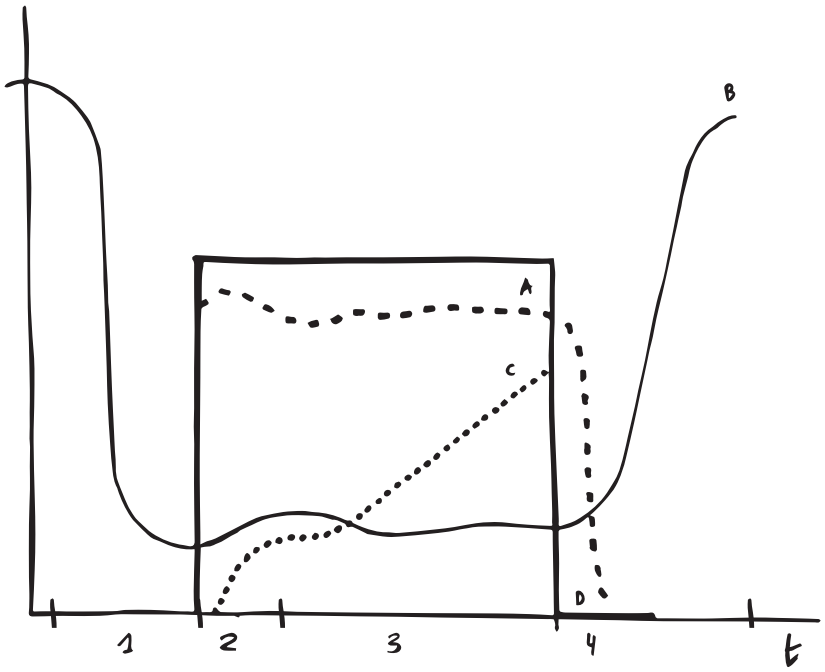
We just learned that under conditions of glow discharge, an ion bombardment of the cathode by (argon) ions will occur. This will result in the decomposition of the cathode material, with the subsequent formation of a coating of the cathode material on all surfaces.
What would a basic setup of a sputter coater system look like? The sputtering process takes place in a closed glass cylinder, the “sputtering or plasma chamber”, which houses an anode and a cathode. Furthermore, there is an opening towards a vacuum pump, and an opening for injection of the inert process gas.



The negative cathode (C) will be the target material to be sputtered (typically gold or platinum), while the specimen (F) to be coated (the substrate) will be on the anode (E).

The desired operating pressure is obtained by a vacuum pump, while an inert gas, such as argon, is admitted to the chamber by a control valve.

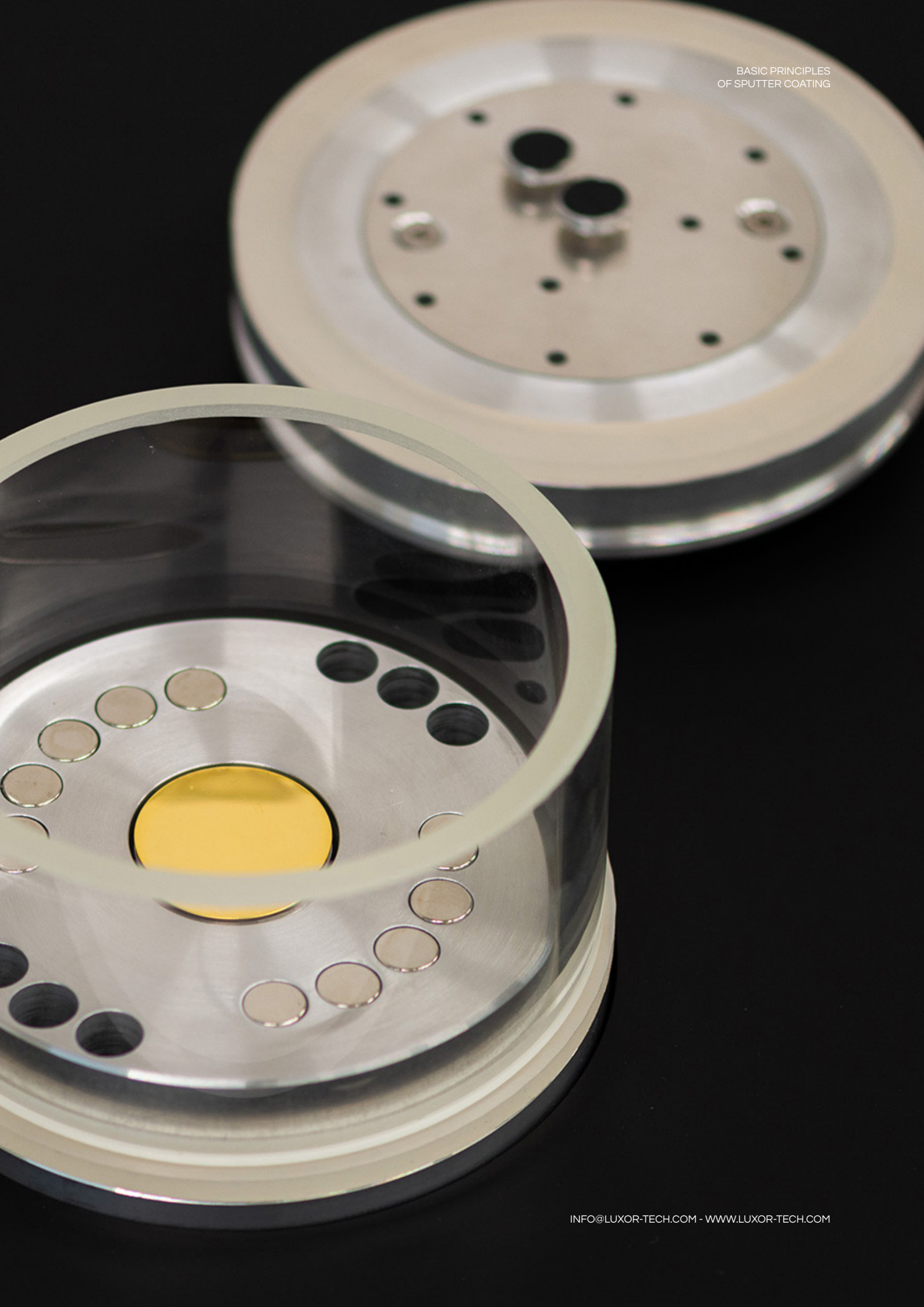
Next is a graphical presentation of a basic sputter coating process using gold as a sputter material and argon as an inert process gas. The graph shows how the current, voltage, pressure and coating thickness are varying during the process.



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

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

The combination of vacuum, voltage, current, time and distance between target and sample determine the growth rate, homogeneity and fineness of the coating on the sample.



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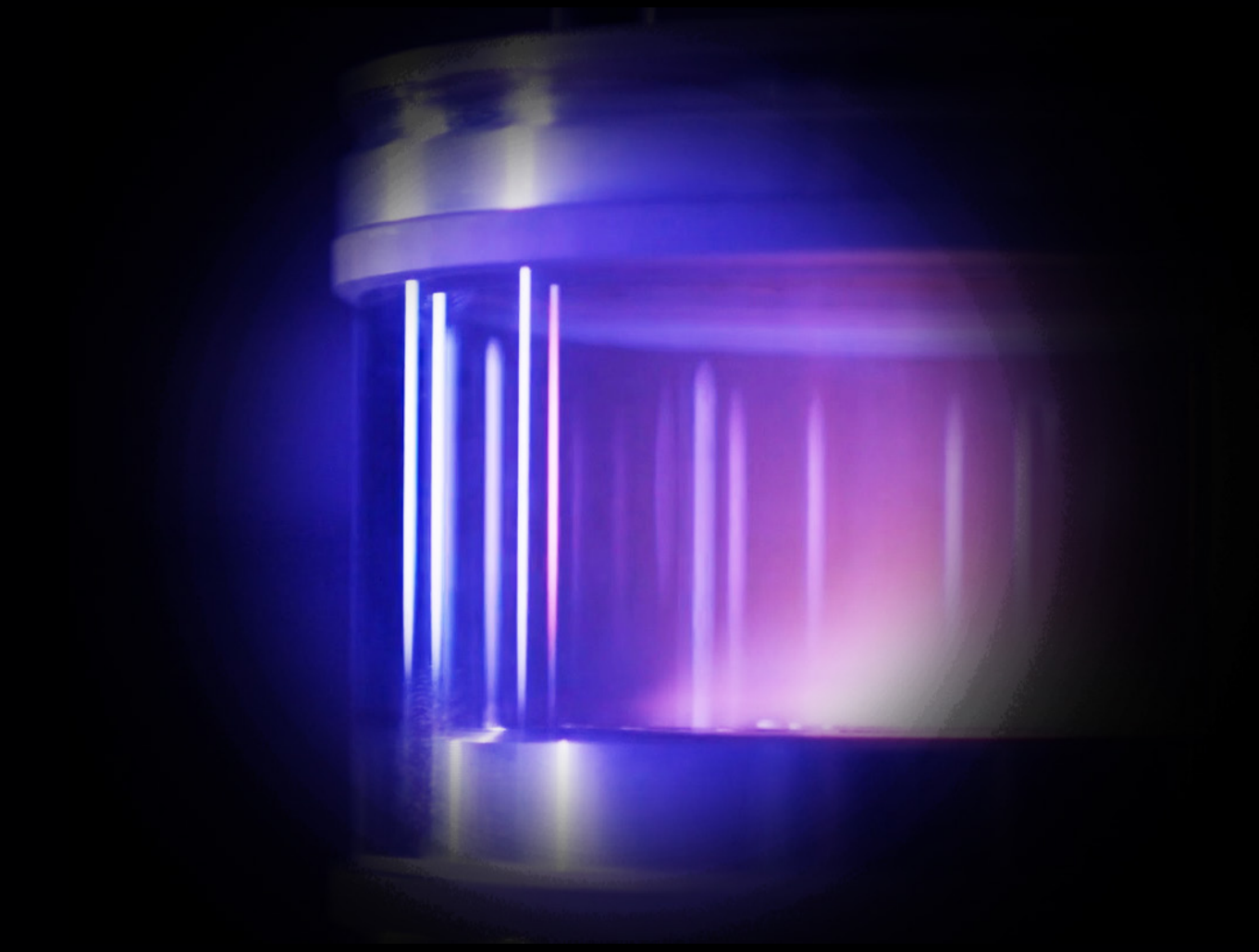
PART 3

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.

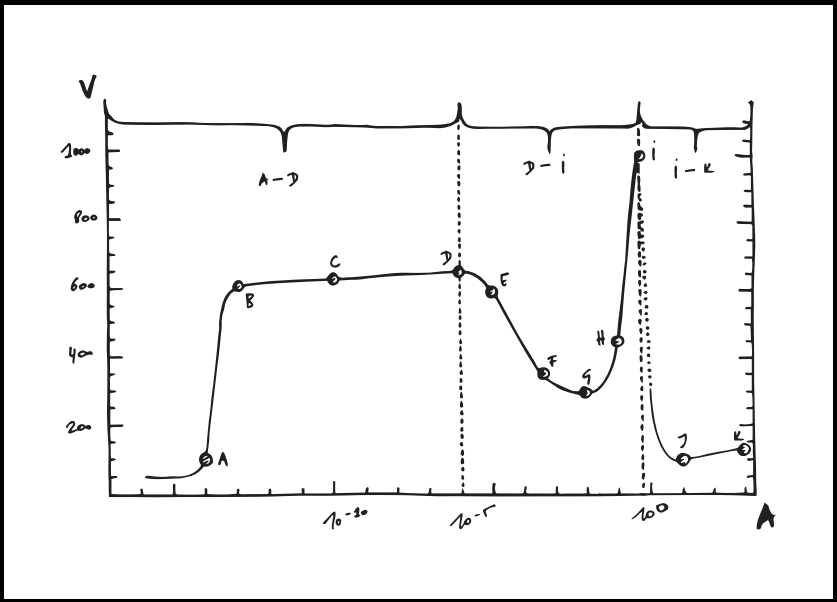
pressure regulator with 2 pressure dial gauges. The left gauge shows the pressure in the gas cylinder, while the right gauge allows for a fine adjustment towards the sputter coater

"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.

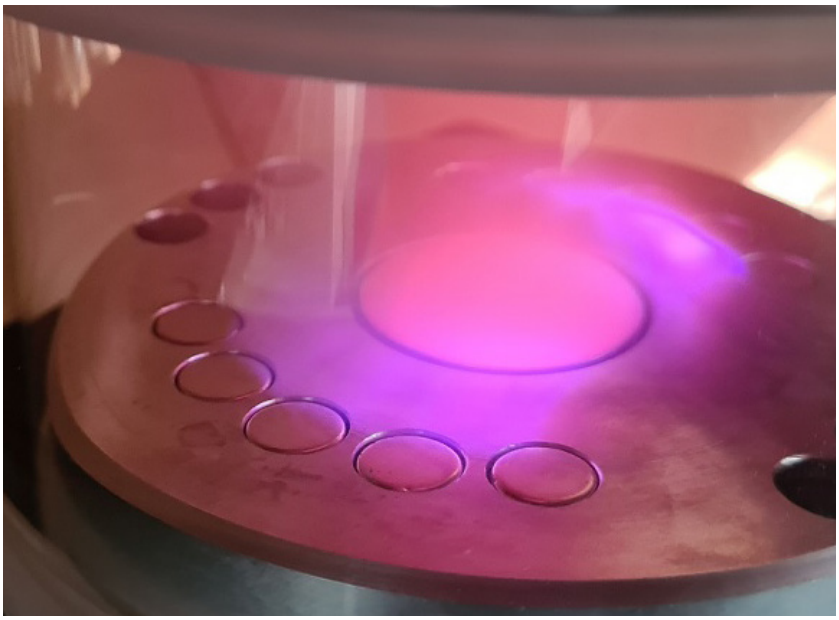
SPUTTERING CURRENT

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.

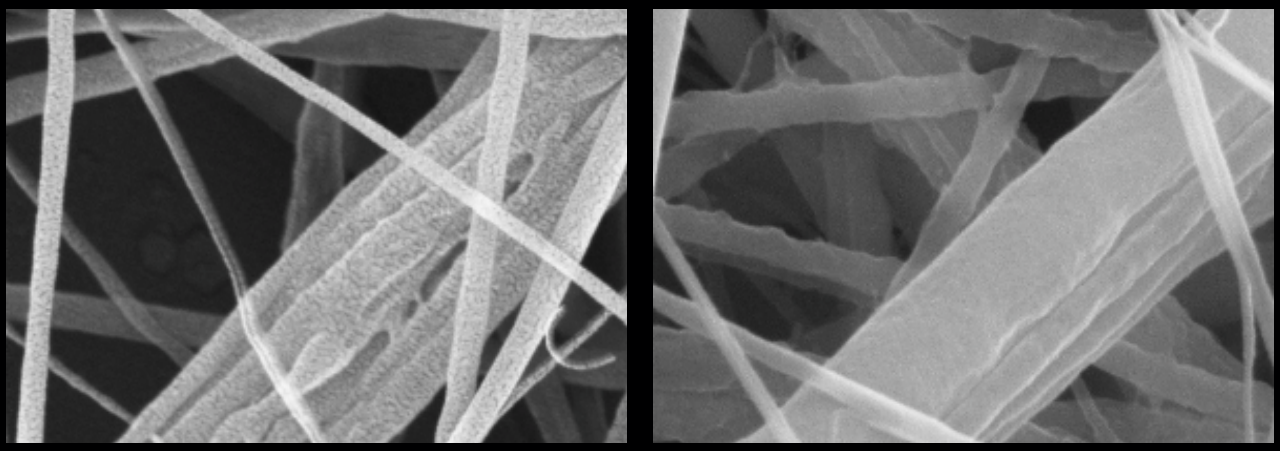
TARGET MATERIAL

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.

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

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.

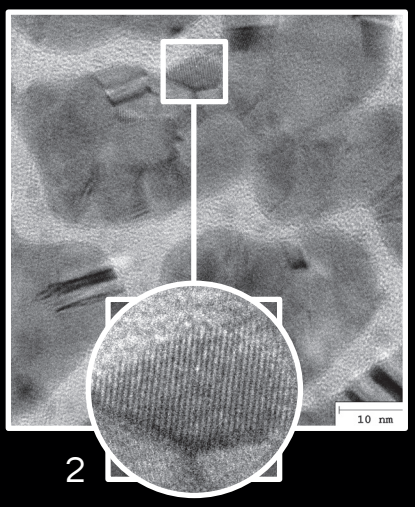
- **Gold (Au)** can be used for low and medium magnification applications, typically up to a few 10.000x magnification. At higher magnifications we can see the grain size of the coating. Another advantage of gold as a coating material is that it can be used in combination with ambient air as process 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

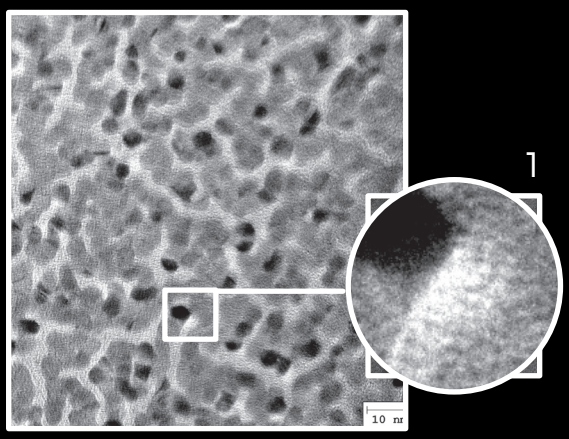


comparison of a 10 nm gold coating (left) and a 5 nm platinum coating (right) on a nanofiber sample at 100.000x magnification. Note that the gold particles become visible in this range of magnification, making gold less suitable for high resolution SEM imaging

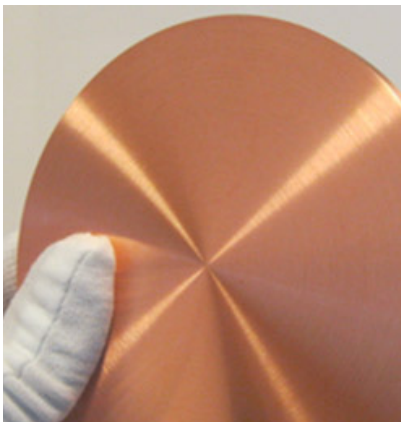
"THE GRAIN SIZE AFTER COATING WITH GOLD IS TYPICALLY ABOUT 8-12 NM. PLATINUM COATINGS HAVE A FINER GRAIN SIZE THAN GOLD AND ARE THEREFORE MORE SUITABLE FOR HIGHER RESOLUTION IMAGING."

- **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.

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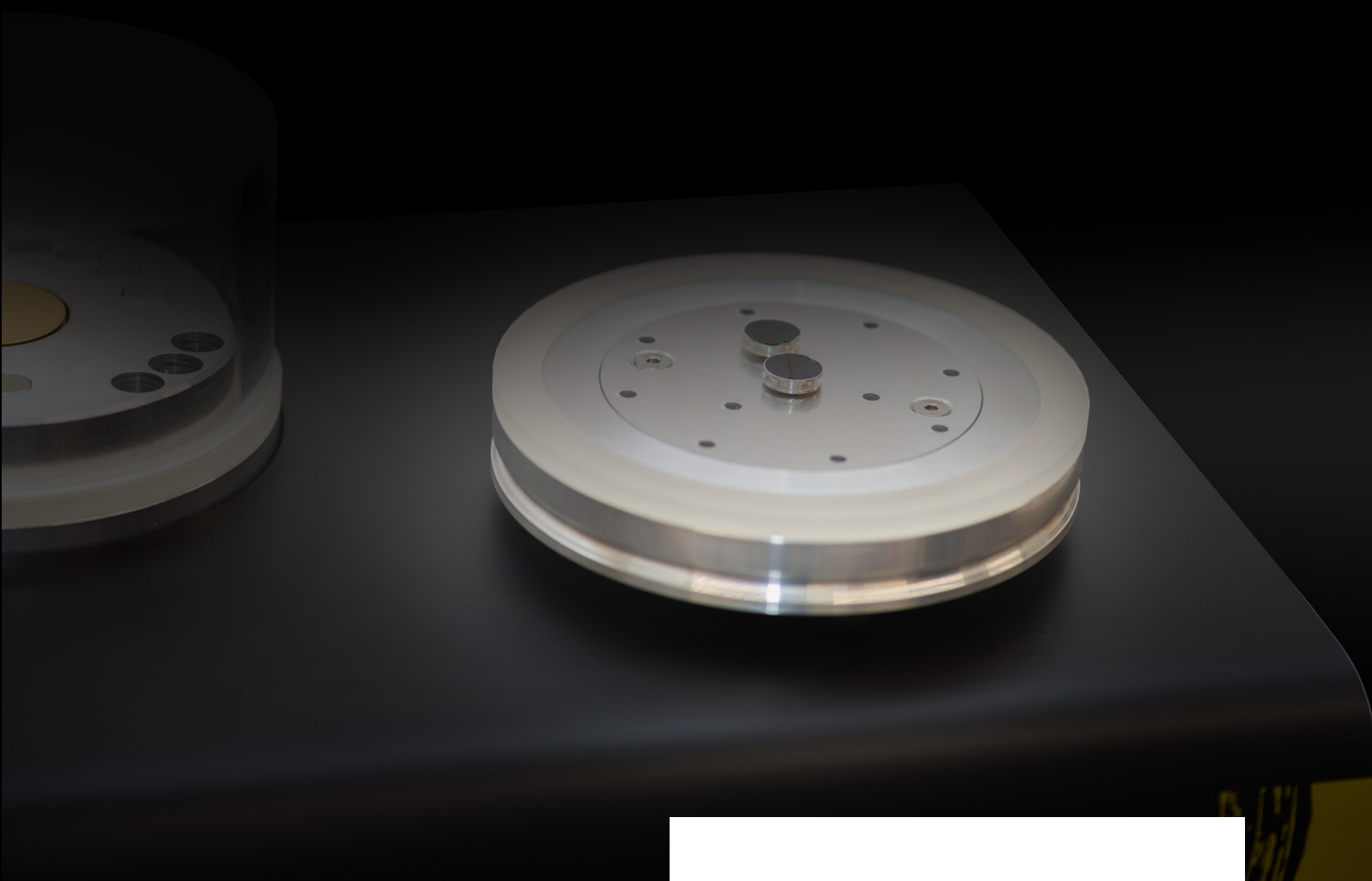
PART 4

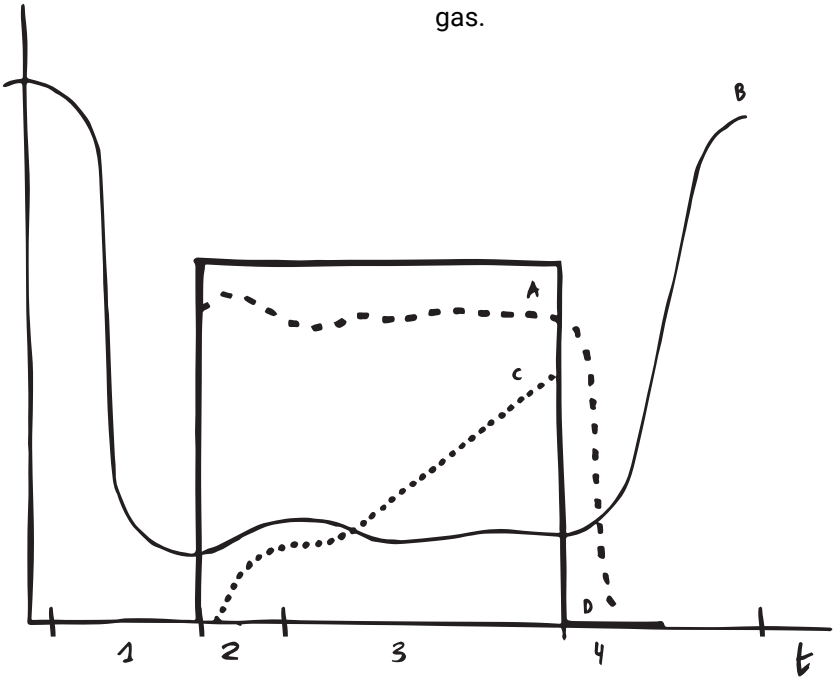
LUXOR A² TECHNOLOGY: SEM COATING MADE SMART AND EASY



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.



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

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

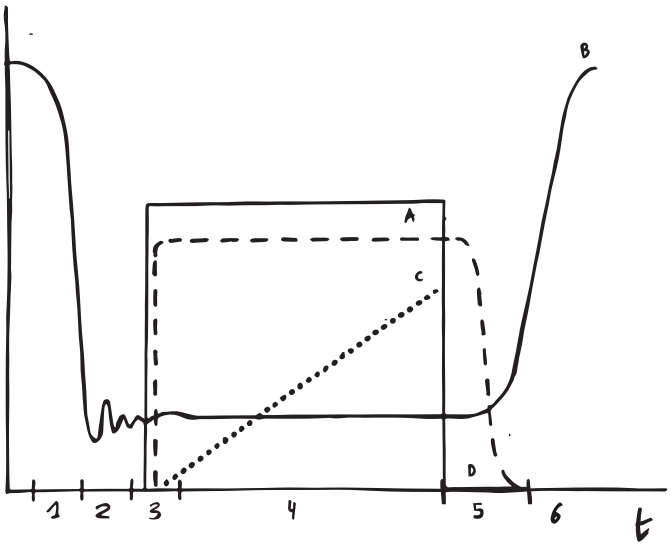
From this description we can conclude that for the accurate control of a sputter coating process the control of the vacuum and the sputtering current are essential. It is also clear that the lower the sputtering current can be kept, the finer the sputtering result will be.

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?

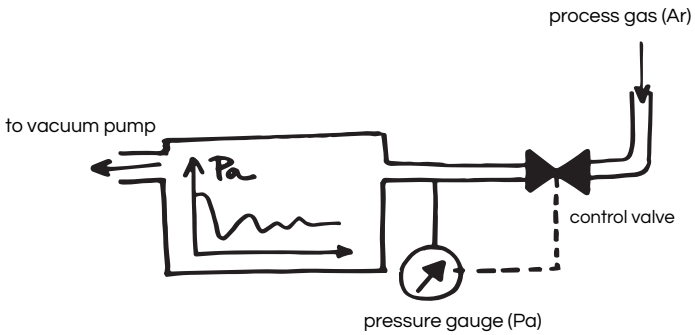
1. The air present is evacuated from the sputtering chamber by the vacuum pump and the chamber is filled/flushed with argon
2. The vacuum pump and vacuum valve that injects the argon bring the chamber to the required target vacuum
3. When the target vacuum is reached, the high voltage is switched on. 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.



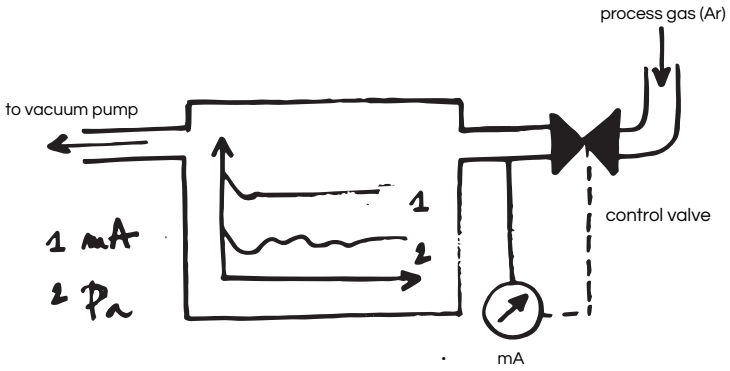
A current (mA)
B pressure (Pa)
C coating thickness (nm)
D voltage (V)

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 tto 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 = 1.0000 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 = 1.0000 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.

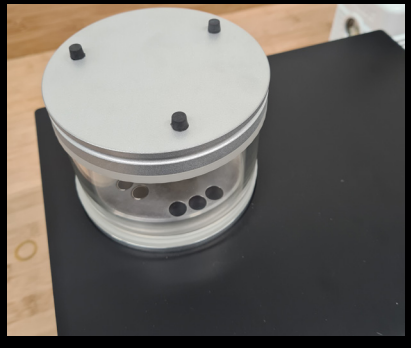
THE UNIQUE “UPSIDE DOWN” DESIGN

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.

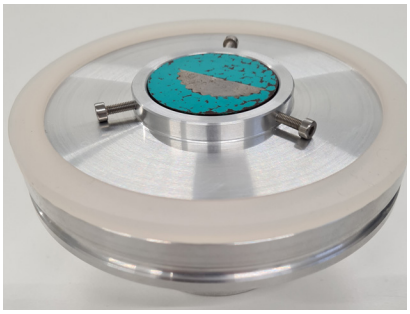
“IN LUXOR METAL COATERS, THE SAMPLES ARE MOUNTED UPSIDE DOWN IN THE LID OF THE PLASMA CHAMBER, WHILE THE TARGET IS LOCATED AT THE BOTTOM. ALTHOUGH THIS MAY SEEM A LITTLE CONTROVERSIAL AT FIRST, THIS CONCEPT ACTUALLY HAS MANY ADVANTAGES.”

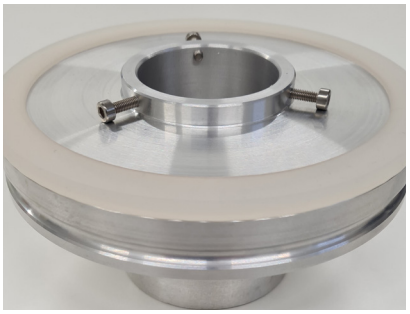
Next, the sample loading station is actually the lid of the coater, and is easily accessible which means samples can be inserted or removed without the need for special pliers or tweezers. This not only simplifies daily use, but also speeds up productivity.

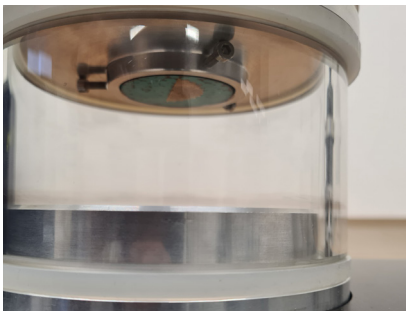


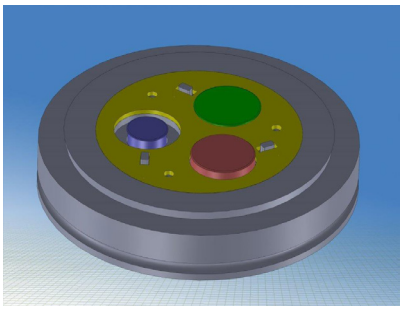
The loading station (or lid) of the LUXOR coater is equipped to work with standard pin stubs with a 3.15mm diameter and either 8 or 9.5mm pin length.

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

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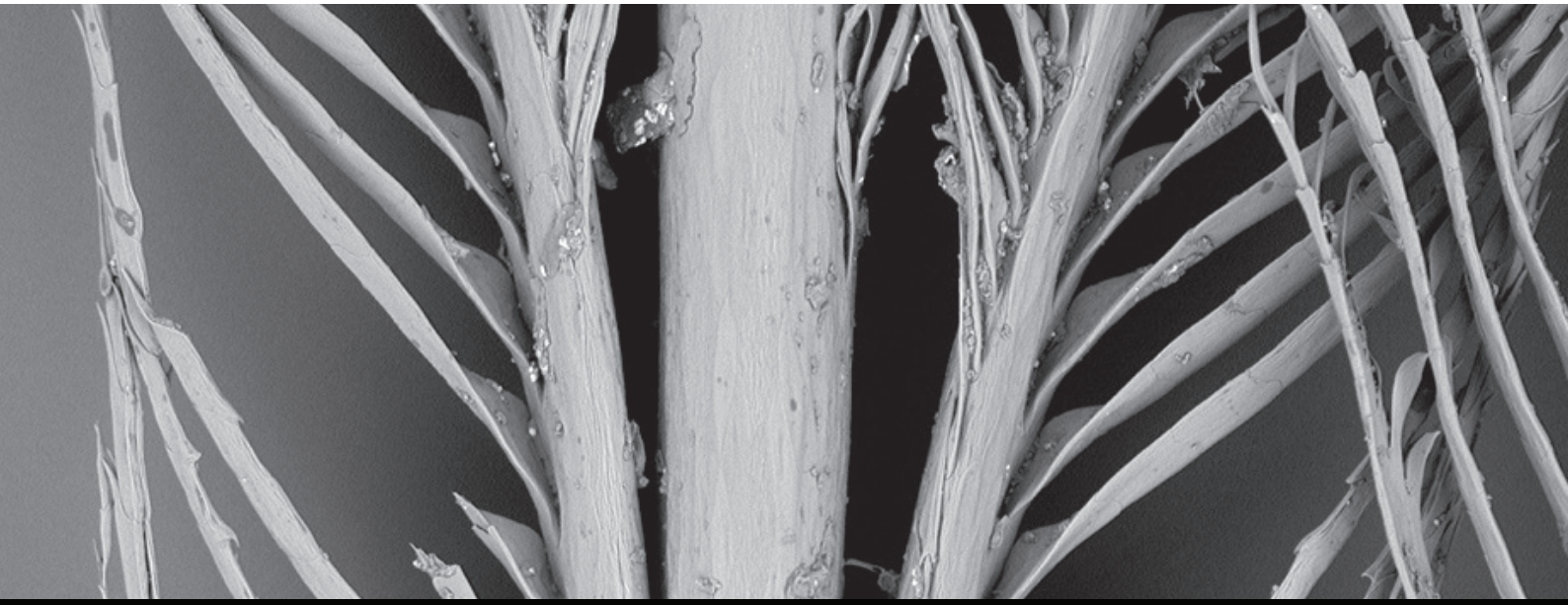

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