



## **Sputter Coating**

## **Technical Brief**



Document Number TB-SPUTTER

Issue 2 (01/02)

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# 1 Introduction

When a target is bombarded with fast heavy particles, erosion of the target material occurs. The process, when occurring in the conditions of a gaseous glow discharge between an anode and cathode is termed sputtering. Enhancement of this process for scanning electron microscopy (SEM) sample coating is obtained by the choice of a suitable ionisation gas and target material. Sputtered metal coatings offer the following benefits for SEM samples:

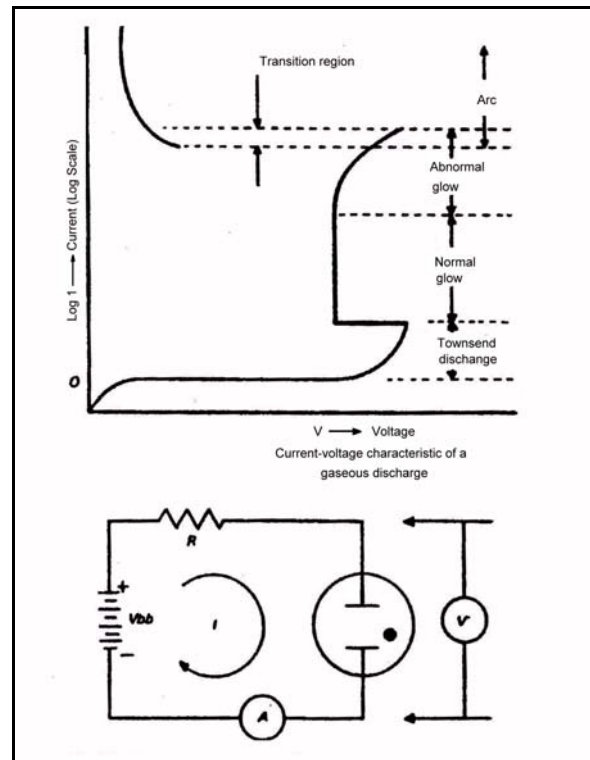
- Reduced microscope beam damage.
- Increased thermal conduction
- Reduced sample charging (increased conduction).
- Improved secondary electron emission
- Reduced beam penetration with improved edge resolution
- Protects beam sensitive specimens

Increase in electrical conductivity of a sample is probably the single most common requirement for SEM, though all factors come into play with FEG SEM. Low voltage SEM operation can still benefit in many cases from a thin coating.

The development of Sputter Coater systems embodies significant empirical design, however, an understanding in classical terms of glow discharge characteristics enhance such designs and may assist in the comparison of differing systems.

## 2 Gaseous Condition

If an inert gas such as argon is included in a cathode gas tube, the free ions and electrons are attracted to opposite electrodes and a small current is produced. See Figure 1.



A = Ammeter V = Voltmeter

**Figure 1 - Circuit to determine the current-voltage characteristics of a cold cathode gas tube**

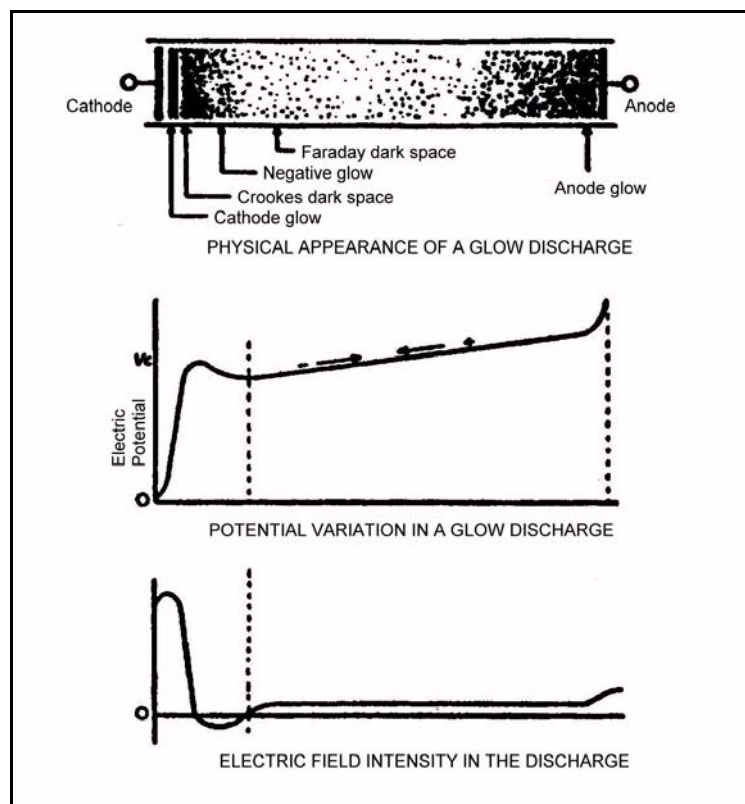
As voltage is increased some ionisation is produced by collision of electrons with gas atoms, named the "Townsend" discharge. When the voltage across the tube exceeds the breakdown potential, a self sustaining glow discharge occurs - characterised by a luminous glow.

The current density and voltage drop remains relatively constant, the increase in total current being satisfied by the area of the glow increasing. Increasing the supply voltage increases current density and voltage drop, this is the abnormal glow region.

Further increase in supply voltage concentrates the glow into a cathode spot and arc discharge is apparent. The operating parameters of sputter coaters are within the glow discharge regions of the characteristic described.

### 3 Glow Discharge

Once the condition for a sustained discharge is met, the tube exhibits the characteristic glow discharge, so called because of the associated luminous glow. It has been established that free ions and electrons are attracted to opposite electrodes producing a discharge - however for a discharge to be self-sustaining requires regeneration of the electrons by the positive ion bombardment of the cathode. This produces secondary electrons and enhances ionisation. The resulting positive ion excess creates a positive space charge near the cathode. The voltage drop experienced is termed the cathode fall. If the discharge is established in a long narrow tube it exhibits the characteristics indicated.



**Figure 2**

The positive ion density in the "Crookes dark space" is very high; as a result a significant voltage drop is experienced between it and the cathode. The resulting electric field accelerates the positive ions which produce secondary electron emission from the cathode.

These electrons accelerated in the direction of the anode cause ionisation, generating positive ions to sustain the discharge. Subsequently, excitation of the gas results in intense illumination in the negative glow region. From this stage the electrons have insufficient exciting or ionising energy, resulting in the "Faraday dark space". Towards the anode a small accelerating field can produce ionisation and excitation, the gas again becoming luminous.

## 4 Sputter Coating

It has been indicated that under conditions of glow discharge, ion bombardment of the cathode will occur. This results in the erosion of the cathode material and is termed plasma sputtering, with the subsequent omni-directional deposition of the sputtered atoms forming coatings of the original cathode material on the surface of the sample and work chamber.

This process is enhanced in sputter coaters for use in Scanning Electron Microscopy where one objective is to provide an electrically conductive thin film representative of the specimen to be viewed. Such films inhibit "charging", reduce thermal damage, and enhance secondary electron emission.

The most common arrangement for a D.C. (Direct Current) sputter coater is to make the negative cathode the target material to be sputtered (typically gold, platinum or with high vacuum sputter coaters, metals such as chromium and iridium), and to locate the specimens to be coated on the anode (which is usually "earthed" to the system, so the specimens are effectively at "ground" potential).

The desired operating pressure is obtained by a pump (usually a two-stage rotary vacuum pump, or a turbomolecular pumped "backed" by a rotary pump), with an inert gas, such as argon admitted to the chamber by a fine control (leak) valve.

## 5 Operating Characteristics

The glow discharge in sputtering is significantly dependent on the work function of the target material and pressure of the environmental gas. A range of target materials are used including gold, gold-palladium, platinum and silver. Although gold is still a common sputtering metal, having the most effective electrical conduction characteristics, it does however, have the largest grain size and is not always suitable for high resolution coating. For this reason gold-palladium and platinum are now widely used as their grains sizes are smaller than gold. Films with even smaller grain sizes can be achieved using metals such as chromium and iridium, but both require the use of a high vacuum (turbomolecular pumped) sputtering system.

The sputter head and sputter power supply should be effective over a range of anticipated target materials.

The deposition rate is current dependant, and if we operate in the correct glow region of the characteristic plasma discharge, as previously described, several fold changes in current should be available for a relatively small change in sputtering voltage. The deposition rate should not be sensitive to small changes in pressure which may be experienced in the system.

If the sputtering head is well designed and operating at low voltage and as a result, low energy input, then radiant heating from the target and high energy electrons (potentially the most significant sources of damage to delicate specimens) should be considerably reduced. There is also evidence to suggest that such a sputter head system may also produce finer grain size for a given target material.

The presence of an inert gas which will not decompose in the glow discharge is obviously desirable. Argon, having a relatively high atomic weight, provides a suitable source of ions for effective bombardment of the target material. Sputtering in air is best avoided.

The effectiveness is also dependent on the "mean free path" (m.f.p.) which is inversely proportional to pressure. If the m.f.p. is too short, insufficient energy will be gained for effective bombardment and will inhibit movement of sputtered material from the target.

If the m.f.p. is too long, insufficient collisions occur and, in addition, the flow of sputtered material may change from diffusion in the gas to free molecular flow with a reduction in the effectiveness of omni-directional deposition.

If these characteristics for sputter heads are achieved, then it should not be necessary to cool the specimen stage for the majority of applications. If not, however, such cooling will only serve to reduce the baseline temperature, the thermal conductivity of most specimens we are considering being relatively poor.

For sensitive specimens pre-cooling (Peltier, water or cryo cooled) and subsequent reduction of the baseline may still be desirable and there is also evidence to suggest a reduction in grain size of the coating. It may be apparent that Scanning Electron Microscopy requires a versatile system without compromising performance. Specifically, fine grain size, uniform coating and low heat input. Consideration of these characteristics in design and development should enable a suitable coating system to be realised.

A major disadvantage of simple diode sputter coaters in SEM is the excessive amount of heat generated in the sample. To overcome this problem, permanent magnets are utilised to deflect the high energy electrons generated in the glow discharge away from the sample.

The magnetic lines of force cause enclosed loops at the target surface, increasing the interaction path length of the high energy electrons in the discharge. Deflection and retardation of electrons result in increased ion yield and sputtering efficiency.



It was indicated previously that while imperial design may be in evidence, it should now be apparent that effective production of positive ions for glow discharge is required. The sputter head and its associated power supply should be a primary objective of design and development.

All modern SEM sputter coaters use heads fitted with an arrangement of magnets and often an associated shroud assembly, with a disc target. Power supplies generally employ solid state switching for applied voltage control.

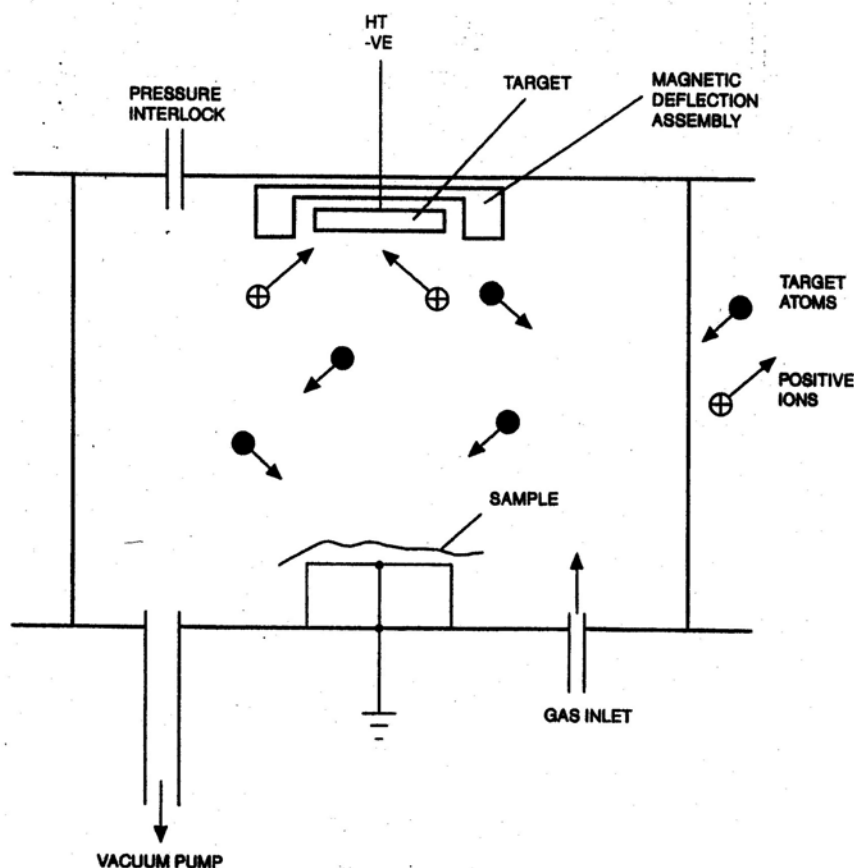


Figure 3 – Diagram of a “cool” sputtering head

The overall result is a low mean voltage head with low energy input. The possibility of thermal damage due to radiant heating and electron bombardment is considered negligible.

For a typical modern magnetron sputter coater

Vacuum	$8 \times 10^{-2}$	to	$2 \times 10^{-2}$ mbar
Sputtering Voltage	100V	to	3Kv
Current	0	to	50mA
Deposition	0	to	25 nm/min
Grain size	Less than 5nm		
Temperature rise	Less than 10C		

It is, of course, possible to satisfy very precise parameters by the selection of target material, 'voltage' 'deposition', 'current' and 'vacuum'. Under these conditions, it is possible to achieve thin films to 10nm with grain sizes better than 2nm and temperature rises of less than 1°C.

## 6 Choice of Sputtering Material

As stated many times, metal coating is an indispensable technique for SEM. The development of high resolution FEG SEMs has brought about more wide spread use of specialised techniques such as Ion Beam Sputtering, Penning Sputtering, E-Beam Evaporation and Planar magnetron ion-sputtering.

More lately Chromium coating has become the "fashionable" material to use. It offers a thin continuous film and emits less back scattered electrons than other sputter materials. However it is not free of its own problems. To operate it requires a high vacuum and ideally vacuum transfer (or vacuum storage) of the sample to avoid oxidation problem. Cr coated samples may often have a "see through" look as there is the possibility of images generated from electrons from sub surface structures. More recently iridium films have been shown to give excellent fine grain (sub nanometer) films that compare favourably with those generated with Cr. Both metals require high vacuum sputter coaters for effective deposition.

Application data collected has shown that a high quality well designed rotary pumped magnetron sputter coater, such as the Quorum K550X, is capable of producing a continuous Pt (platinum) film with a grain size in the order of 2 nm. It also has the benefit of being a good secondary electron emitter, unlike chromium. Some images of chromium show bright high contrast images. Many workers, and our own studies have led us to consider the possibility of each grain of chromium being oxidised before sample is coated and hence the film is not truly continuous and indeed each metal grain is individually charging. This is another reason to consider iridium as an alternative.

Silver as a sputter material is often ignored but is a very satisfactory method for ensuring conductivity of the SEM sample but has a major advantage the whole process is reversible as the metal may be removed by the neutral aqueous reagent known as "Farmers reducer". This enables many samples to be viewed and then returned to their original condition. **Beware.** ...Silver may form crystalline deposits on the surface of the sample in the presence of active Halogens

- Sputtered silver offers smaller grain size than evaporated silver.
- Sputtered Gold and Silver have similar grain size but the silver has larger reticulation after storage.
- Silver is the most conductive metal known.
- Silver has a high secondary electron coefficient.
- X-ray emission lines are well separated from the biologically important sulphur and phosphorous.
- Cost effective.

Gold/Palladium (80:20) targets are now a popular standard choice for the routine coating of a wide range of samples. The idea behind using this alloy is that the palladium will act as a physical barrier to the gold which will attempt to conglomerate into large islands and restrict ultimate resolution performance.

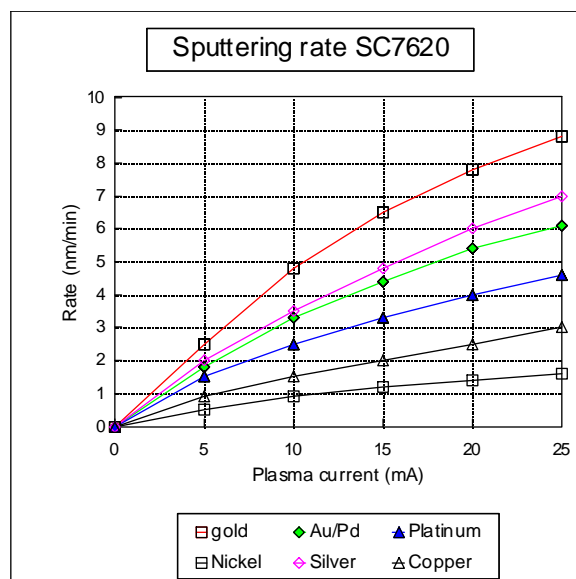
The minimal loss in secondary electron emission performance from the palladium is not seen as significant with current SEM's.

Other target choices are generally made based on the requirement for X-ray analysis of samples or back scattered electron detection.

## 7 Rates of Sputtering

A question regularly asked is what difference is there in sputtering rates for each of the target materials. The following list gives the variance of the materials in relation to gold, assuming gold to be: 1, it is impossible to give actual coating rates as this varies with sputtering conditions.

Au	Gold	1.0
Ag	Silver	1.2
Co	Cobolt	0.5
Cr	Chromium	0.5
Cu	Copper	0.7
Fe	Iron	0.5
Mo	Molybdenum	0.3
Ni	Nickel	0.5
Pd	Palladium	0.85
Pt	Platinum	0.6
Ta	Tantalum	0.2
W	Tungsten	0.2



**Figure 4 - Sputtering Rates for the SC7620**

## 8 Thickness of Coating

Experiments using interferometric techniques have shown that the thickness of Au/Pd coating sputtered in argon gas can be calculated at 2.5KV according to:

$Th = 7.5 I t$  (angstroms) ( $V = 2.5KV$ , target to specimen distance = 50mm)

$t$  = time in minutes

$I$  = current in mA

$Th$  = thickness in angstroms

Average coating times will be of the order of 2 -3 minutes using  $V = 2.5KV$  and  $I = 20$  mA

Platinum targets when fitted will give approximately half the deposition rate.

## 9 General Points for Improving Performance

1. **Cleanliness**, the work chamber must be kept clean! We advise that a separate carbon coater be used in applications where the maximum performance of the sputter coater is required
  - Clean the glass chamber with hot soapy water and dry thoroughly, solvents can be used but we have found this unnecessary and having greater danger to health and safety. If the deposit is stubborn, use a kitchen scouring pad such as the green Scotch Bright variety.
  - Use Isopropyl alcohol on metal surfaces, not acetone which has greater danger to health and safety. It will also take longer to out gas and reduce the vacuum performance.
2. **Vacuum**, Never leave the chamber under vacuum without isolating the roughing pump from the coater, this is usually done with a manual valve (Quorum high vacuum sputter coaters have useful "pump hold" facility that allows the vacuum chamber to be held under vacuum when the instrument is not in use). Failure to do so will increase the risk of suck back of hydrocarbons (pump oil) in to the sputter chamber and increase contamination.
  - Always ensure the system is dry and pumping to its correct vacuum level before working with samples, failure to do so will result in poor sputter rate and contamination.
  - Ballast rotary pumps on a regular basis and ensure they are serviced at regular intervals.
3. **Sputter gas**, Always use high purity argon gas of the grade known as "White spot" this will ensure fast sputter rate and good pump down time.
4. **Rotary planetary specimen stages** are essential for ensuring even coatings on specimens with irregular surfaces.