Guide to Coating for SEM Imaging

Imaging non-conductive samples in scanning electron microscopy (SEM) can be challenging. When exposed to the electron beam, non-conductive samples accumulate electrostatic charge, which can result in image distortions, defocussing, or bright spots on the SEM image. To prevent charge accumulation, techniques such as coating with a thin conductive layer, variable pressure SEM, low-kV imaging, beam deceleration, and environmental SEM (ESEM) can be employed.

This application note highlights the importance of coating in SEM sample preparation and offers practical guidance on target material selection, recommended coating thickness, and the impact of vacuum pressure and current on coating quality across a wide range of applications.

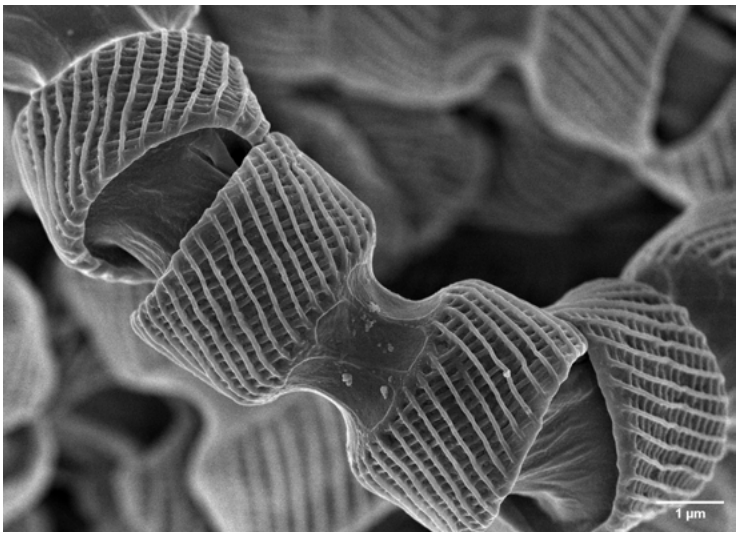


Figure 1. Spider web collected on a metal circular holder with epoxy resin as an adhesive medium. Prepared via critical point drying and coated with 8 nm Pt.

Reasons to Coat

1. Remove/reduce Charging from the Sample

In SEM, an accelerated electron beam is focused through a series of lenses and apertures under vacuum conditions. The electrons interact with atoms on the surface of the sample, which results in the emission of secondary electrons (SE), backscattered electrons (BSE) and X-rays from the sample. These signals are then recorded by detectors, and this information is used to generate high-resolution images.

For conductive materials, the electrons are dissipated if the sample is correctly grounded. However, when non-conductive materials are exposed to an electron beam, charge accumulation occurs. This accumulation results in an artefact known as charging, whereby blurring, streaking, or distorted images are observed (Figure 2/3). Coating samples with a thin conductive layer, can reduce charging by providing a pathway for the electrons to dissipate.

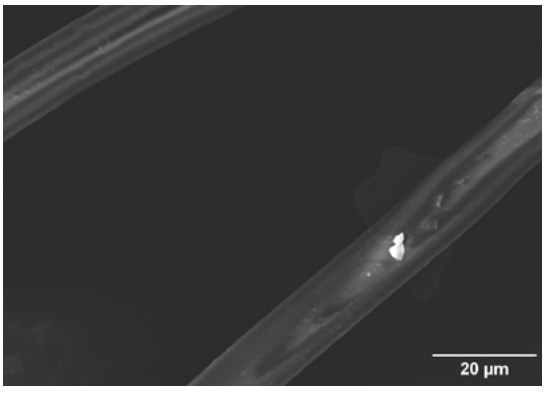


Figure 2. Uncoated spider silk with charging.

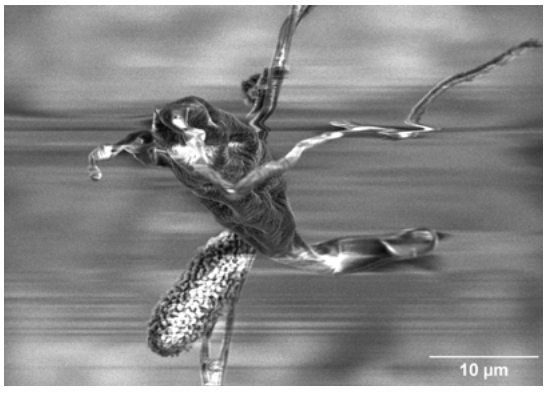


Figure 3. Uncoated spider web with trapped pollen and protozoa.

2. Improve Secondary Electron Emission and Signal to Noise Ratio

Low atomic number (low-z) elements, such as carbon, produce lower secondary electron (SE) and backscattered electron (BSE) emission yields. This results in lower contrast compared to higher-z elements, and a higher tendency to charging.

While low-voltage SEM can help reduce charging and prevent sample damage, it limits sample penetration (Figure 4), potentially resulting in signal loss from the surface.

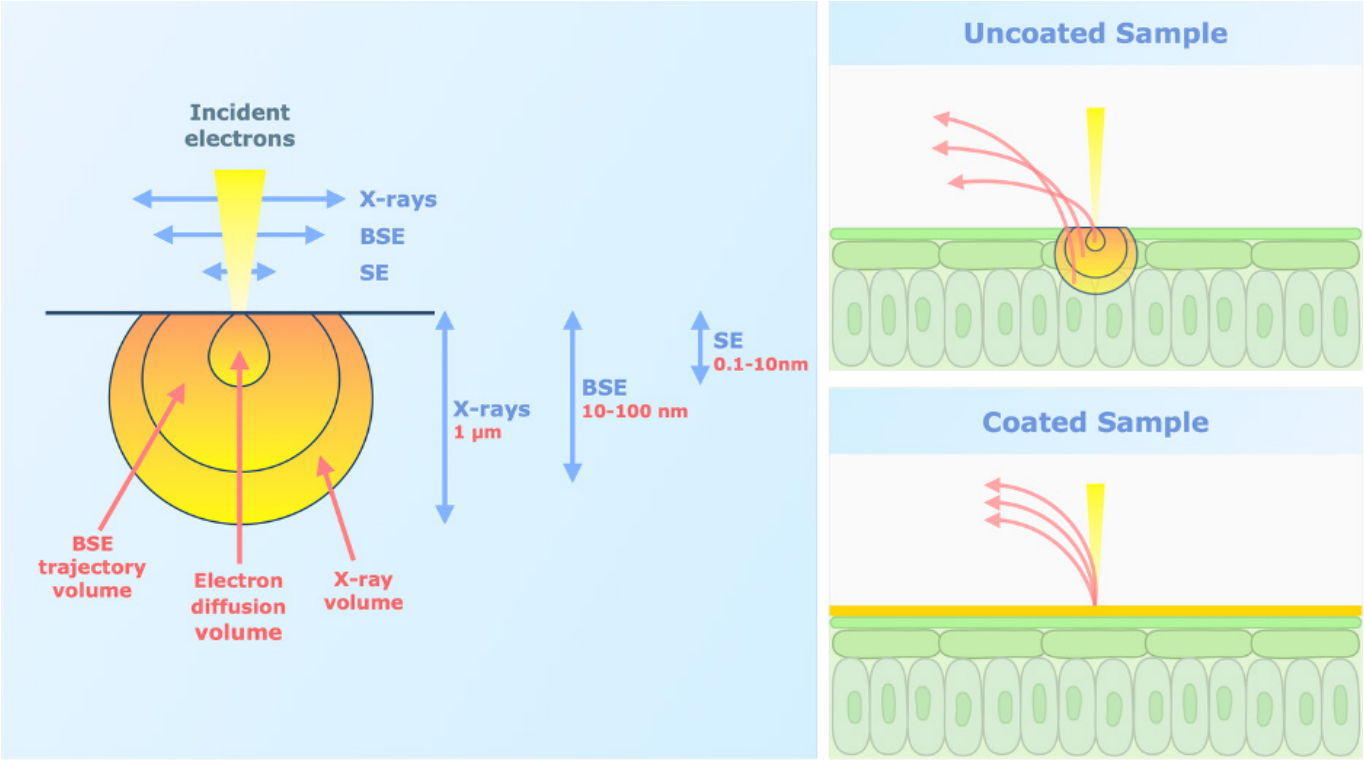


Figure 4. Electron beam interaction volume in EM and comparison of beam interation with coated and uncoated samples.

In contrast, metals possess higher atomic numbers and greater densities of free electrons. These properties improve SE emission and signal-to-noise ratios. A thin metal coating (1-5 nm) of Pt, Ir, Cr, or Au, can be applied to a sample, to localise SE and BSE signals to the exterior of the surface and reveal more detailed topographical features. Although thicker coatings can further increase SE yield, they may obscure sample details at magnifications above 100k.

3. Increase Mechanical Stability

Delicate samples, such as biological or soft materials, can be easily damaged by the electron beam. This can result in surface deformation, as well as drift.

Sample coating can help to increase mechanical stability of samples by increasing their rigidity, making their surface more robust.

4. Reducing Contamination

Contamination in SEM can interfere with image quality and data accuracy. Thin metal layers often trap fewer contaminants compared to non-conductive samples due to their chemical inertness, lower surface energy, and improved charge and heat dissipation. Reducing contamination not only improves image quality but also minimises contamination within the microscope, preserving its functionality.

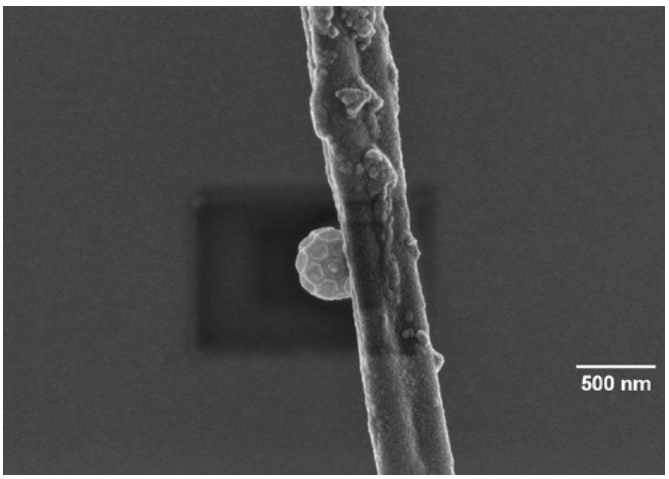


Figure 5. Spore attached to spider silk. Black squares observed as a result of hydrocarbons deposited from the outgassing sample.

5. Reducing Beam Damage

The high energy of the electron beam can induce thermal damage to samples. Metal coatings reduce beam damage, by providing effective charge dissipation, absorbing and redistributing electron energy and enhancing thermal conductivity of the sample.

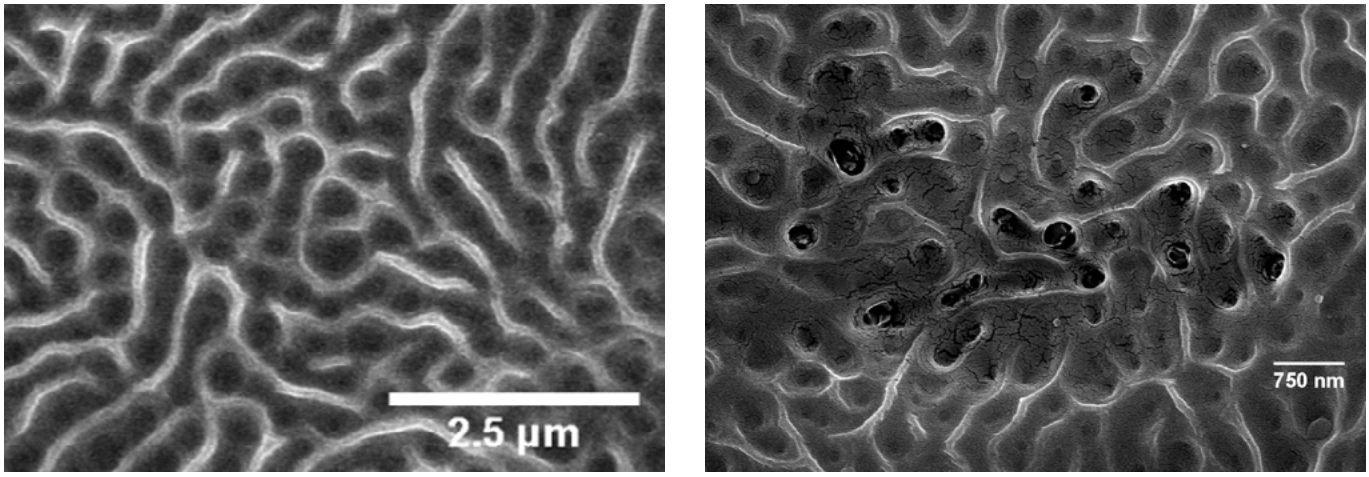
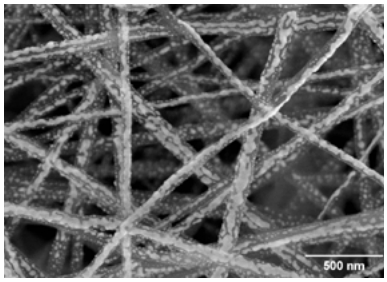
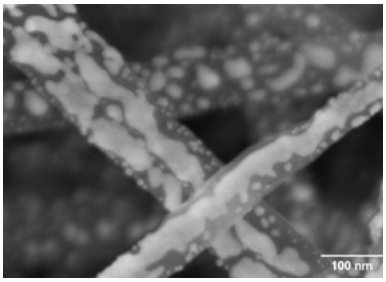
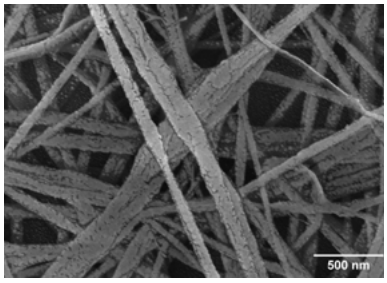
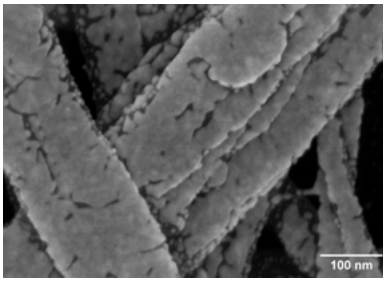
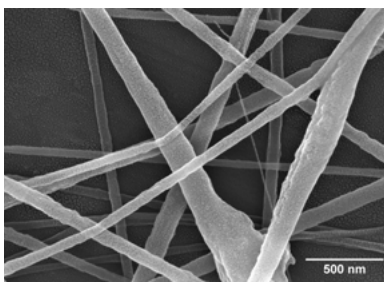
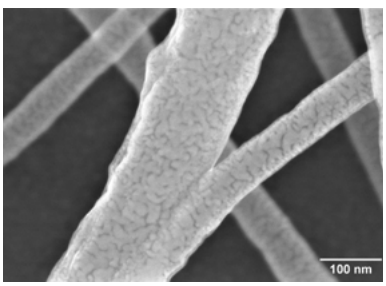


Figure 6. Snake skin where beam damage is visible.

The Impact of Vacuum Pressure and Current on Coating Quality

The ultimate vacuum level of a system significantly impacts the quality of coatings produced. Higher vacuums reduce gas contamination, improve material deposition, enhance adhesion, control coating properties, minimise oxidation, and maintain thermal stability. This results in smaller grain sizes.

Figure 7. Comparison of 5 nm Au coating on PVdF electrospinning fibres with varying initial vacuum.

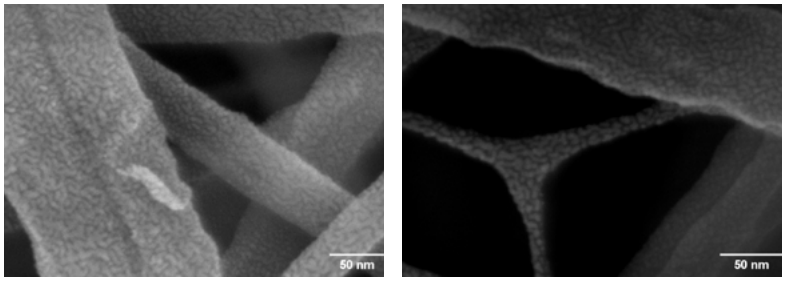
5 nm Au POOR VACUUM Poor coverage, large island of coating observed		
5 nm Au LOW VACUUM Improved coverage, but uneven size of islands		
5 nm Au HIGH VACUUM Good coverage, islands are evenly sized		

The current used during sputtering plays a significant role in determining the grain size of the deposited thin film. A higher current results in a higher rate of ionisation of particles and hence, a faster sputter rate. This increase in deposition rate leads to the formation of larger grains, as the material is deposited more rapidly. Therefore, to reduce grain size, a low sputtering current is recommended.

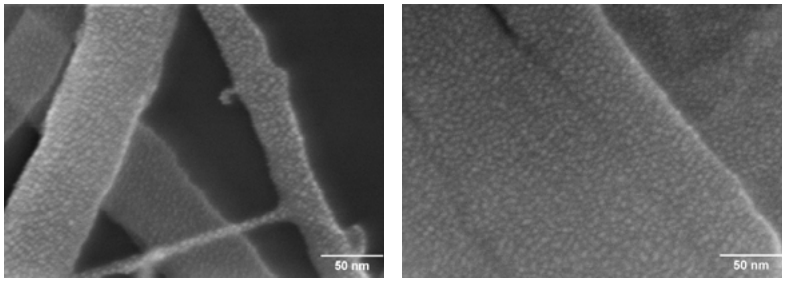
Figure 8 shows how low current and high vacuum can result in smaller grain size (1-3 nm).

Figure 8. PVdF electrospinning fibres coated with Au (high vacuum and low sputtering current).

2 nm Au
HIGH VACUUM, LOW CURRENT
Low current results in smaller grain size, and dense coating



1 nm Au
HIGH VACUUM, LOW CURRENT
Low current results in smaller grain size, and dense coating



Selecting a Target

The choice of coating material depends on the sample type and intended application. Table 1 suggests suitable targets for different applications. These are general recommendations, when completing elemental analysis the most suitable target will be dependent on the signal peaks from the sample.

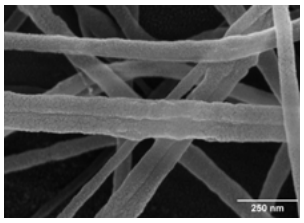
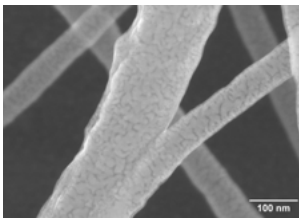
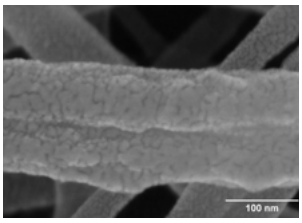
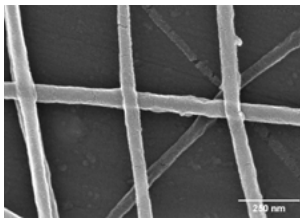
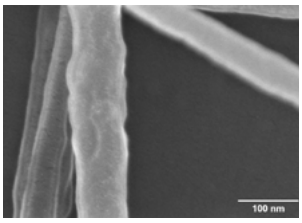
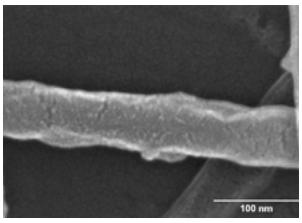
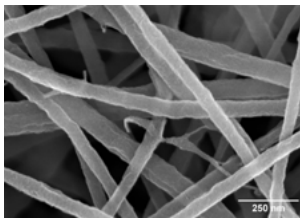
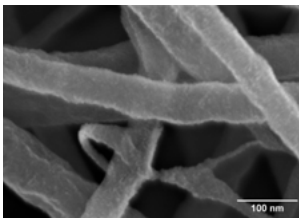
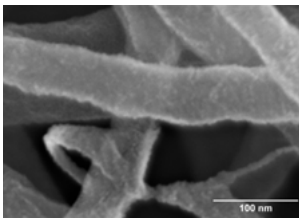
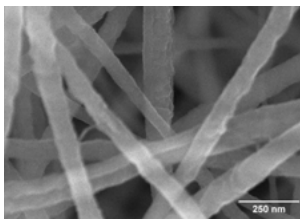
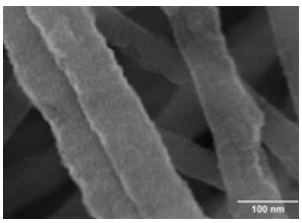
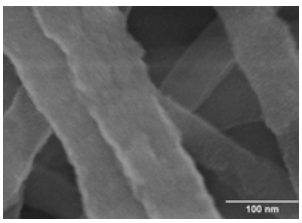
Table 1. Summary of frequently used targets and their applications.

	Application	Au	Au/ Pd	Ag	Pt	Pt/ Pd	Ir	Cr	W	Ta	Pd	Ni	Cu	Ti	C
Imaging	SE, magnification < x 50k	•	•	•	•	•	•				•	•	•		•
	SE, magnification > x 50k	•			•	•	•	•	•	•					•
	SE, magnification < x 100k				•	•	•	•	•	•					•
	SE, signal boost (< 1 nm)	•			•		•								
	BSE [a]							•			•				•
Analytical	EBSD [d]											•	•	•	•
	Elemental analysis (EDS [b], WDS [c])	•			•		•		•						•

a Backscattered Electron
b Energy Dispersive Spectroscopy
c Wavelength Dispersive Spectroscopy
d Electron Backscatter Diffraction

Figure 9 provides recommendations for suitable targets based on different magnifications and applications.

Figure 9. Comparison of 5 nm coatings on PVdF electrospinning fibres with different materials (Au, Ir, Pt and Cr).

5 nm Au The larger grain size of the Au coating partially obscures the discrete morphology of the fibres.			
5 nm Ir The small grain size of the Ir coating reflects the fibre's morphology. Due to the brittleness of Ir, cracking can be observed on the fibres.			
5 nm Pt The small grain size of the Pt coating shows the morphology of the fibres.			
5 nm Cr While Cr produces very fine grain sizes, its tendency to oxidise can lead to reduced SE yield, compromising image contrast.			

Grain size is influenced by factors such as atomic size, melting point, solidification behaviour, nucleation rates, thermal stability, and recrystallization behaviour. The finest grains are produced by Ir, Cr, Ta, Pt, Pt/Pd and W, although it is possible to achieve smaller grain sizes with other metals if the vacuum level in the coater is sufficiently low.

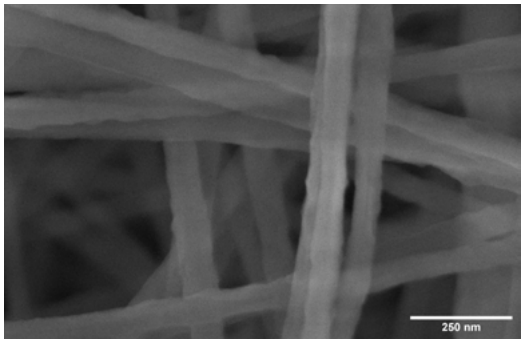
Another important factor to consider is that some metals are more prone to 'crack' over stress. For instance, when coating fragile scaffolds made from elastic polymers, iridium coatings may crack.

The thickness of the applied coating will also play a role in imaging. Additional thickness may obscure sample details and/or add unwanted 'decoration'. Therefore, it is best practice to use a thin coating layer (< 10 nm) to maintain image quality.

Figure 10. Comparison of Au coating thickness on PVdF fibres.

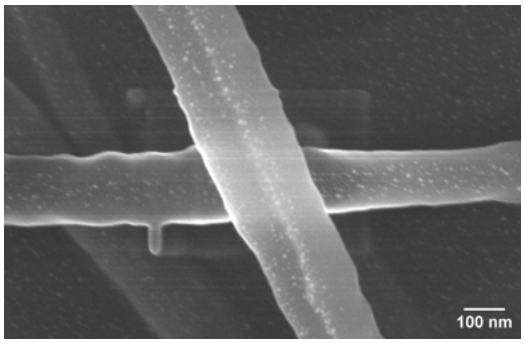
UNCOATED

Poor contrast and visible charging



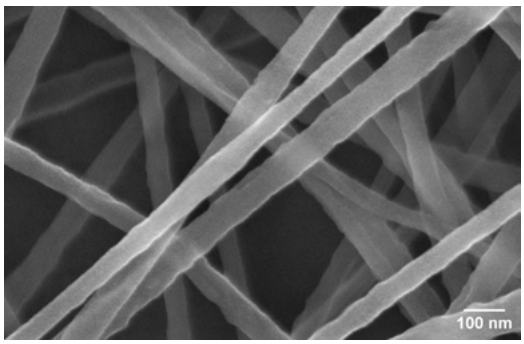
0.2 nm Au

Charging observed due to insufficient coating



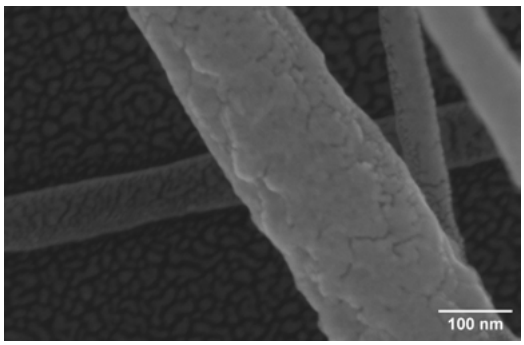
2 nm Au

Good contrast and no charging



10 nm Au

No charging but fibres are obscured by coating



Material	Application
Gold	Gold provides excellent electrical conductivity and a smooth, fine-grain coating ideal for high-resolution imaging in SEM. Often used for biological and soft samples.
Gold/Palladium	The Au/Pd alloy combines the conductivity of gold with the hardness of palladium, creating a fine, uniform coating with better resistance to sputtering wear. It is not suitable for EDS, as palladium will produce additional peaks in the spectrum that may overlap with sample element signals.
Silver	Silver provides high electrical conductivity and is useful for imaging soft, organic materials or when a reflective surface is needed in SEM or TEM. It is recommended for lower magnifications as it has a larger grain size than gold. Silver is ideal if the coating needs to be removed after imaging, as silver can be easily dissolved with a mixture of potassium ferricyanide and sodium thiosulfate.
Platinum	Platinum produces a very fine, uniform grain and is more durable than gold, making it suitable for samples requiring a thin, stable coating. For very elastic nano-structures it may not be suitable, as it can crack in the presence of oxygen.
Platinum/ Palladium	The Pt/Pd alloy provides a finer grain structure than pure platinum and is less prone to stress cracking. It is therefore ideal for high-resolution imaging.
Iridium	Iridium provides an extremely fine grain structure, ideal for ultra-high-resolution SEM. As it is brittle, it can be prone to stress cracking.
Chromium	Chromium has a fine grain size and so is suitable for high resolution imaging. However, due to its susceptibility to oxidise, the sample must be imaged immediately after coating. Chromium is also often used as a base or adhesion layer under other metals (e.g. Au) and works well for harder or denser samples that require a durable surface.
Tungsten	Tungsten has a higher SE yield than chromium and a finer grain size. However, it is prone to oxidation.
Palladium	Palladium has a low SE yield and is therefore only used for imaging up to x 50 k.
Nickel	Nickel is not suitable for SE imaging due to its low SE yield, magnetic properties, and significant electron scattering. However, it can be used for BSE and EDS analysis. Nickel coating can be removed with HCl or HNO_3.
Copper	Copper often contains copper oxide which makes it only suitable for low magnification SE imaging. The coating can be removed with HNO_3 or $FeCl_3$.
Titanium	Titanium is only suitable for EDS analysis. It can also be used as a base or adhesion layer under other metals (e.g. Au).
Carbon	Carbon is the only element that does not contribute to contrast in SE images because it is transparent to the electron beam. It is also conductive and chemically inert. Stability to the electron beam makes carbon suitable for analytical techniques such as EDS, WDS, and EBSD.

QCoat

Quorum

High-Vacuum Coating System

TurboQ

The TurboQ is an automated and versatile turbo pumped coater offering unparalleled ease of use, optimized for high-vacuum applications

Recommended applications include:

- Ultra-high resolution magnification FEG-SEM/FIB-SEM and FIB-SEM, up to and beyond 200K.
- Carbon support film for TEM grids
- EDS, WDS, EBSD analysis
- General thin film deposition

Features

- High vacuum instrument for ultra-high-resolution EM up to and beyond 200k
- Ultra fine grain size for high-resolution analysis
- Combined system capable of sputter and carbon coating
- Rapid cycle time for maximum sample throughput
- Tilt stage design giving uniform coating of complex 3D structures
- Film Thickness Monitor (FTM) to control deposition of coating
- Intuitive and responsive touchscreen colour panel design
- Multi-colour LED visual status indicator offering process updates
- Pre-set recipes for standard protocols resulting in sample reproducibility
- Customizable recipes for tailored applications
- Easy and quick set-up of profiles
- Multiple stage options based on application nee

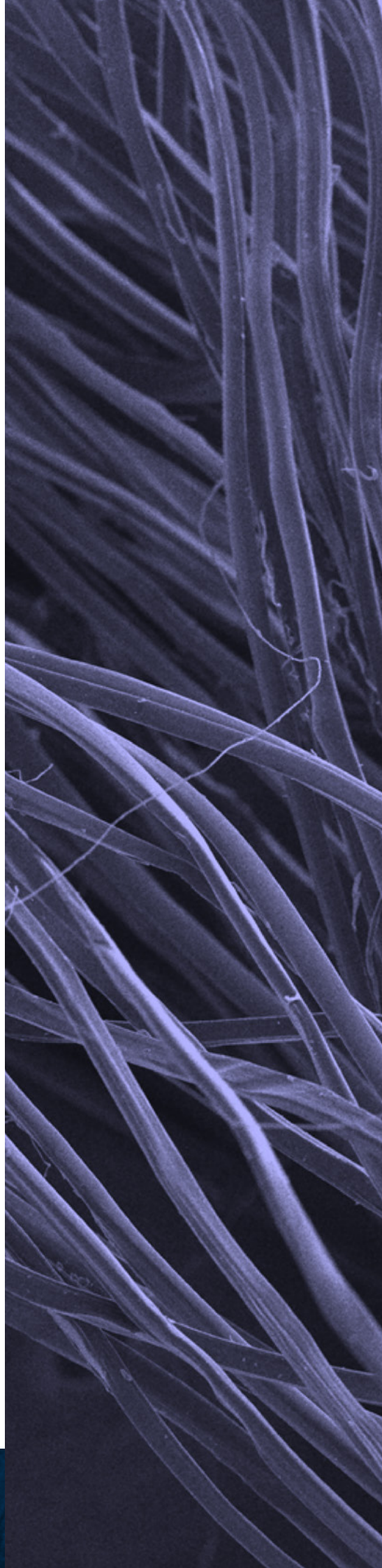
For more information visit:

TurboQ S – sputter coater for noble and/or oxidizing metals. Cr target offered as standard.

TurboQ E – evaporation coater for carbon/metal evaporation. Carbon rod source offered as standard.

TurboQ ES – combined coater offering both sputter coating and carbon and metal evaporation. Cr target and carbon rod source supplied as standard.

SPECIALISTS IN ELECTRON MICROSCOPY SAMPLE PREPARATION
E: SALES@QUORUMTECH.COM • **T:** +44 (0) 1323 810981 • **W:** QUORUMTECH.COM



Quorum Technologies
Judges House
Lewes Road
Laughton
East Sussex
BN8 6BN
United Kingdom

T: +44 (0) 1323 810981
E: sales@quorumtech.com
W: www.quorumtech.com

Products are for research use only.