APPLICATION NOTE

The influence of coating quality on SEM imaging of PVdF electrospinning fibres

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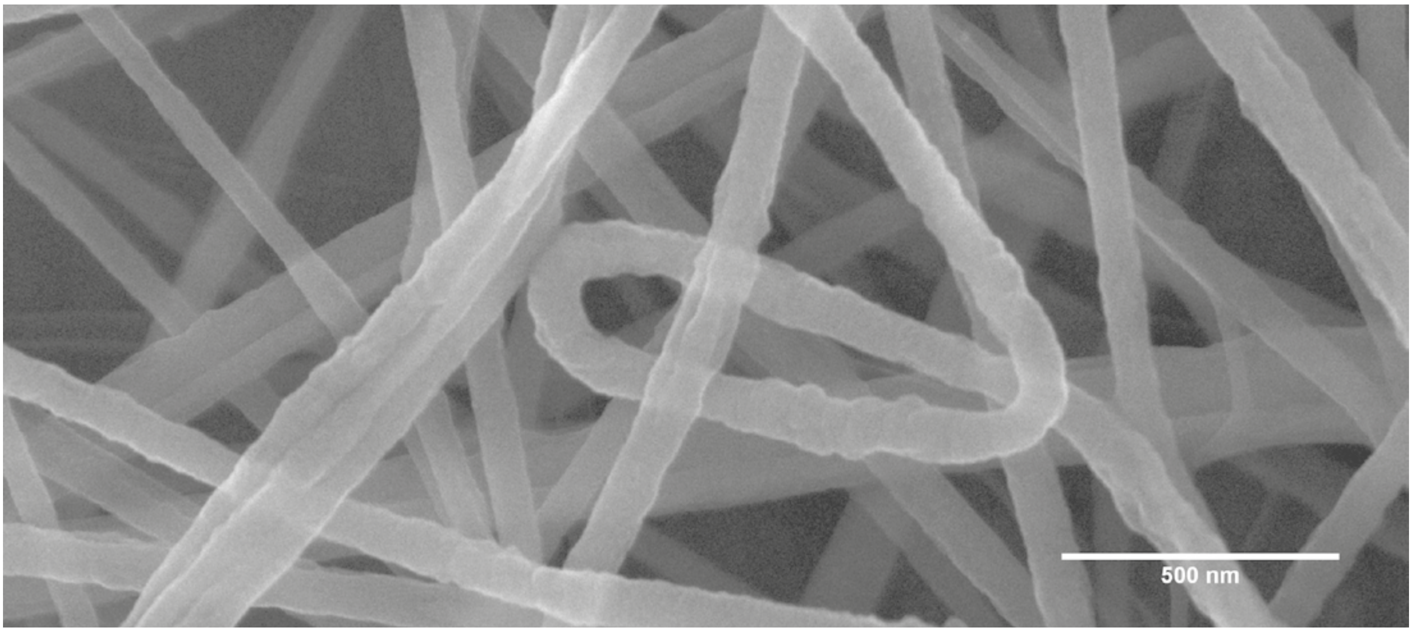
The increasing amount of applications for nano-fibres, especially those produced in the **electrospinning** process, creates a need to image them in a way that allows users to examine not only their alignment and diameter, but also their discrete morphology.

Electrospinning fibres are used in many fields from energy storage (solar cells, fuel cells), environmental engineering (membranes and filters) to healthcare (tissue engineering and drug delivery). Usually they are processed further through chemical modification, which equips their surface with active molecules. Before this can happen it is essential to examine their native surface to govern the post-treatment process in the desired way.

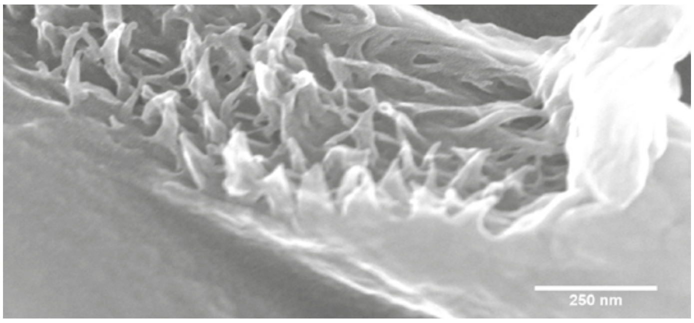
These fibres can have a variety of diameters from hundreds to a few nanometres. They can also be smooth or exhibit porosity.

PVdF-poly (vinylidiene fluoride) is one of the materials used as part of the electrolyte in lithium-ion batteries, due to its high dielectric constant, and strong chemical and electrochemical resistance.

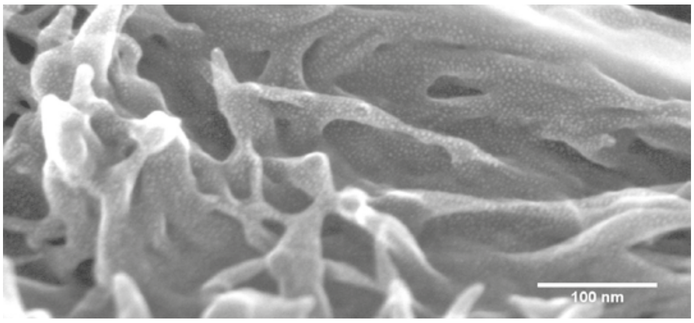
PVdF electrospinning fibres coated in a high vacuum with 1nm of gold using the Q150V Plus coater



Tear on PvDF elctrospinning fibre



Zoom in on the structure of a damaged fibre



The Q150V Plus coater allows the user to coat **electrospinning fibres** in such a way that it is possible to image even very small fibres and examine their morphology. **The parameters** that are most important for metal sputter coating are: **vacuum, sputtering current and sputtering gas pressure.**

This applies to any target material used. The factor which plays the most significant role in the metal sputtering process is the **vacuum.** The higher the vacuum, the smaller the grain size, and also a less contaminated coating is produced. This allows the user to cover the electrospinning fibre samples with a minimal thickness of metal and yield good contrast SEM images, which clearly show their surface morphology.

The influence of coating parameters on coating quality, when gold is used as a sputtering material.

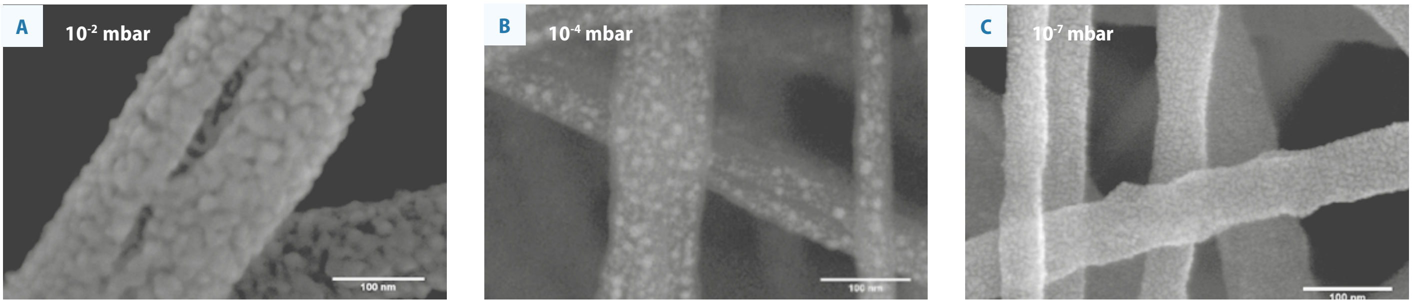
When applying a conductive coating for SEM imaging, it must have two key features. First, the coating layer needs to be thin enough not to attenuate sample morphology. Secondly, it must be dense enough to provide conductivity, which allows for the removal of any accumulating charge from the specimen surface. Different metals have intrinsic SE yield and have different grain sizes. A gold coating was chosen here to present the influence of sputtering parameters on the grain size and ultimately the coating thickness.

1 Base vacuum level

The base vacuum is a parameter that has an immediate impact on the coating quality. The better the vacuum, the finer the grains of the coated material.This is due to fewer particles that the sputtered metal can collide with during its way to the substrate surface. The same metal sputtered using a different base vacuum will result in different grain sizes. This change in vacuum is depicted below.

All coatings were generated using the same thickness of Gold, 2 nm, sputtering current (20 mA), and have the same pressure of sputtering gas (Ar), 10^{-2} mbar. However, all used a different base vacuum.

SEM images of PVdF electrospinning fibres coated with 2 nm Au, using various base vacuum levels



A: 10^{-2} mbar (Q150R Plus)   B: 10^{-4} mbar (Q150T Plus)   C: 10^{-7} mbar (Q150V Plus)

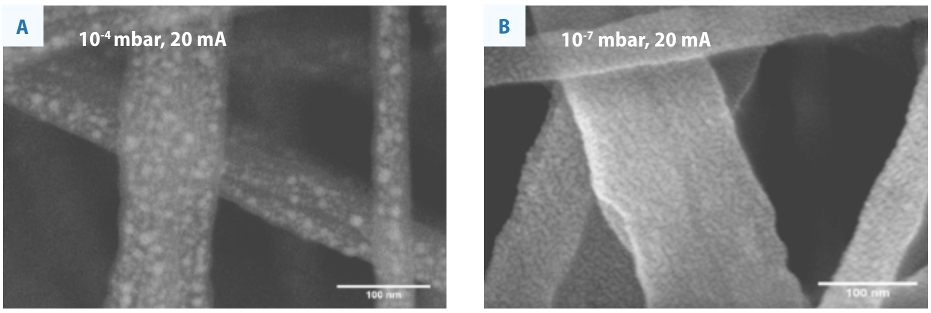
2 Sputtering current

Cold magnetrons in the Q Plus series coaters allow users to apply low currents when sputtering. This improves the coatings produced in terms of the coverage density and grain size. Low sputtering currents provide a smaller grain size and, if combined with a high vacuum base pressure, allows users to produce neat and very dense layers of minimal thickness.

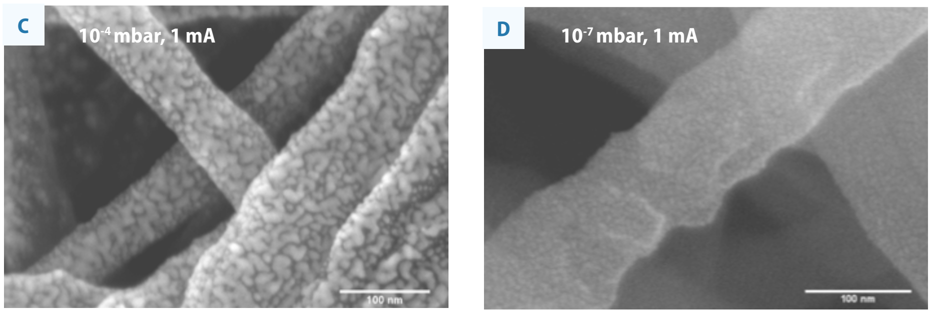
A low sputtering current is especially recommended where there is a requirement to access areas like cavities or stacks found in electrospinning fibres.

The images to the right present the influence of the sputtering current and base vacuum on the produced gold coatings. All coatings were made of the same 2 nm thickness.

SEM image of PVdF electrospinning fibres coated with 2 nm Au



A and C: 10^{-4} mbar, 20 and 1 mA respectively (Q150T Plus)

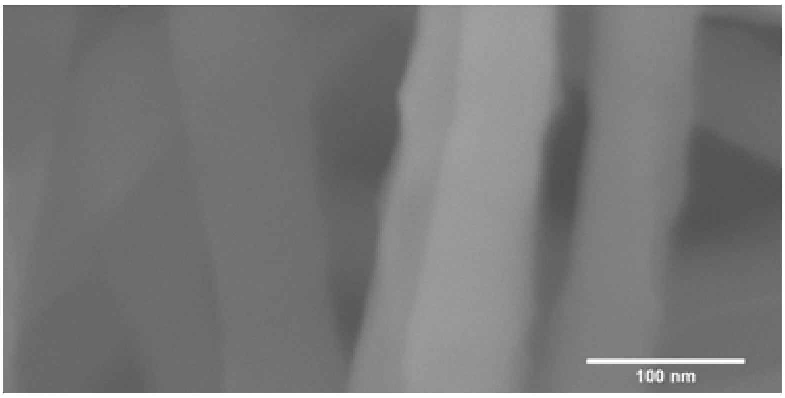


B and D: 10^{-7} mbar, 20 and 1 mA respectively (Q150V Plus)

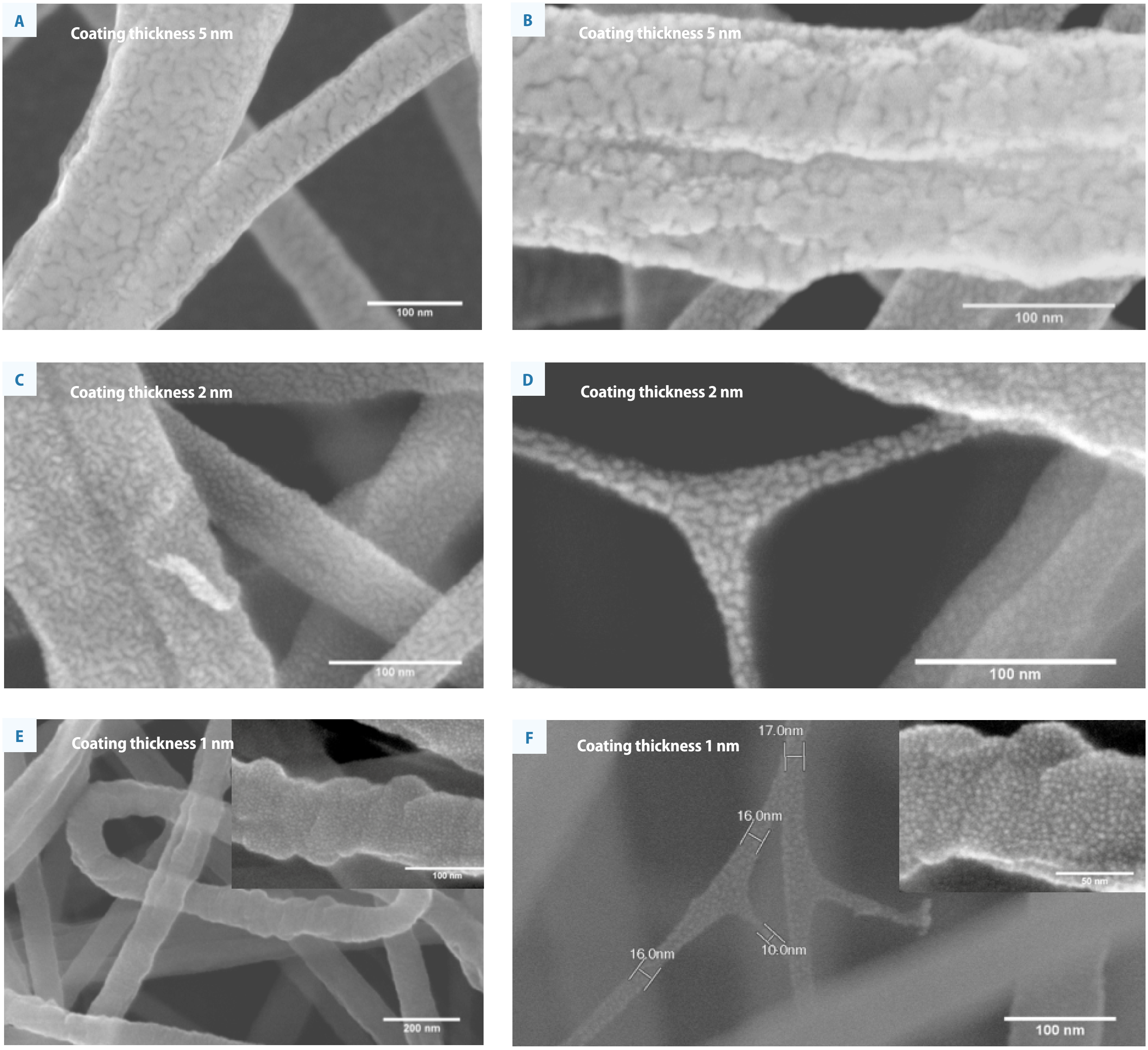
3 Coating thickness

Electrospinning fibres are made of polymers, they form stacks or scaffolding, where individual fibres touch each other but they are not connected. In SEM imaging such samples are the most challenging, as fibres gather charge and might move when subjected to the electron beam. For successful SEM imaging of electrospinning fibres a layer of conducting coating is a necessity. The coating has to be made of a metal, which exhibits a small **grain size** and has excellent **secondary electron (SE) yield.** Only when the key conditions are met can the coating be thin enough not to attenuate the discrete morphology of a single fibre and allow for crisp and clear imaging. The coating will also introduce some rigidity to the fibres allowing for easier imaging. In EM imaging the general rule says 'that less is more'. Thinner coatings allow users to see more surface details.

SEM image of uncoated sample of PVdF electrospinning fibres.



SEM image of PVdF electrospinning fibres coated under the same conditions: 10^{-7} mbar as a base vacuum and 1 mA sputtering current



Images show different thicknesses of gold. A, B: 5 nm C,D: 2 nm, E, F: 1 nm

Q150V PLUS

See how our high-vacuum coater delivers an ideal solution for coating electrospinning fibres

Recommended applications:

- Ultra-high-resolution magnification SEM
- Protective platinum layers for FIB
- R&D of corrosion-, friction-, and wear- protective layers
- Carbon coating of TEM grids
- BSE imaging
- EDX, WDS, EBSD analysis
- Carbon coating of replicas
- Nano-particles e.g. Zeolites
- Nano-brushes

The Q150V Plus is optimised for high-vacuum applications, with ultimate vacuum of 1×10^{-6}mbar or less possible. Together with the use of a wide-range Penning/Pirani gauge, this enables the sputtering of oxidising metals with ultra-fine grain sizes, which are suitable for high resolution imaging. The lower background pressure allows less contamination and thus finer grain structure in the deposition environment. Similarly, lower scattering allows for high purity, amorphous carbon films of high density.

Sputter coating of noble and oxidising metals using the Q150V S / ES Plus:

- Low and high magnification (up to x100,000) - Au, Au/Pd
- Ultra high magnification (above x100,000) - Pt, Cr, Ir
- For thin film applications ITO, Tungsten, Aluminium, Zinc
- Other targets are available

Q150V Plus features

User interface has been updated:

- Dual-core ARM processor for a fast, responsive display
- Capacitive touch screen is more sensitive for ease of use
- User interface software has been extensively revised, using a modern smartphone-style interface
- Comprehensive context-sensitive help
- USB interface allows easy software updates and backing up/copying of recipe files to USB stick
- Process log files can be exported via USB port in .csv format for analysis in Excel or similar. Log files include date, time and process parameters.
- 16GB of flash memory can store more than 1000 recipes

Allows multiple users to input and store coating recipes. New feature to sort recipes per user according to recent use.

Intelligent system logic automatically detects which insert is in place and displays the appropriate operating settings and controls for that process.

System prompts user to confirm target material and it then automatically selects appropriate parameters for that material

Intuitive software allows the most inexperienced or occasional operator to rapidly enter and store their own process data. For convenience a number of typical sputtering and carbon coating profiles are already stored but also allows the user to create their own.

Software detects failure to achieve vacuum in a set period of time and shuts down the process in case of vacuum leak, which ensures pump protection from overheating.

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