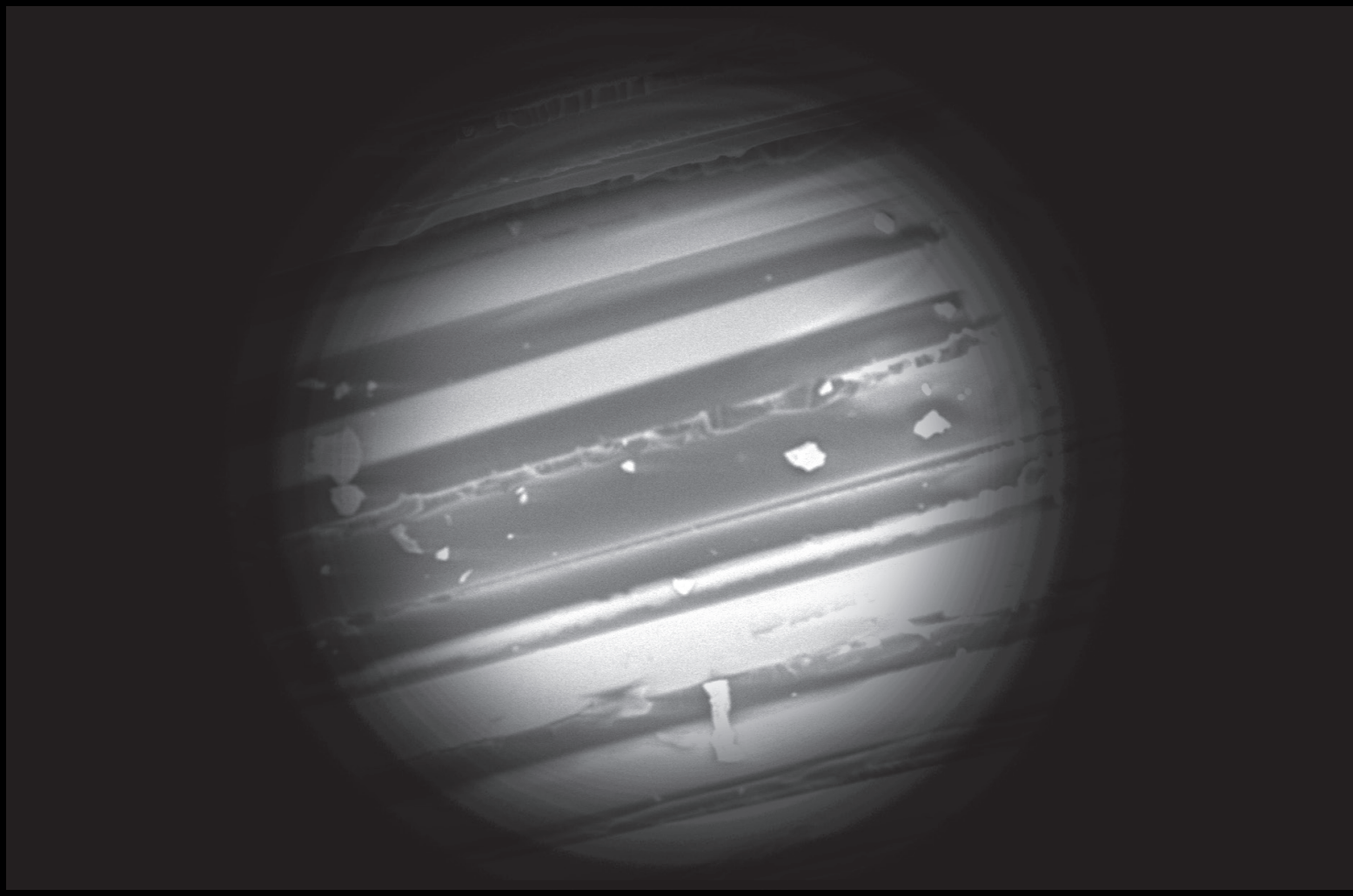
PART 1

CHARGING EFFECTS IN ELECTRON MICROSCOPY, AND HOW TO AVOID THEM

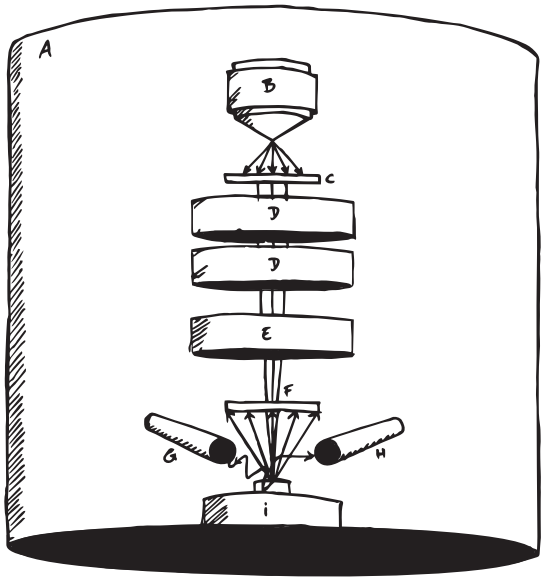


WHY USE METAL COATING IN EM

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

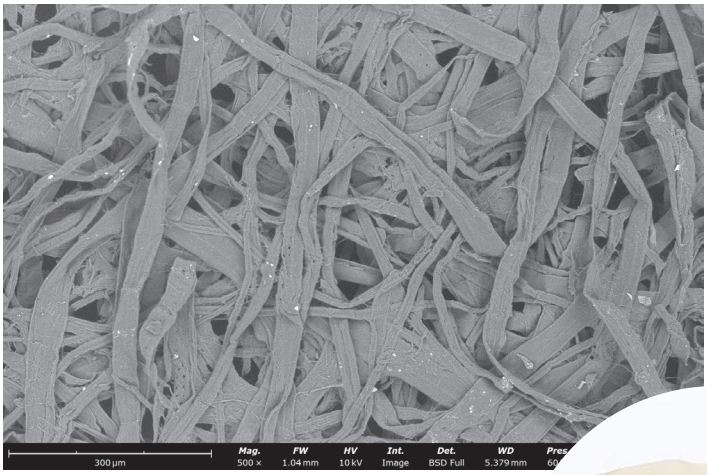
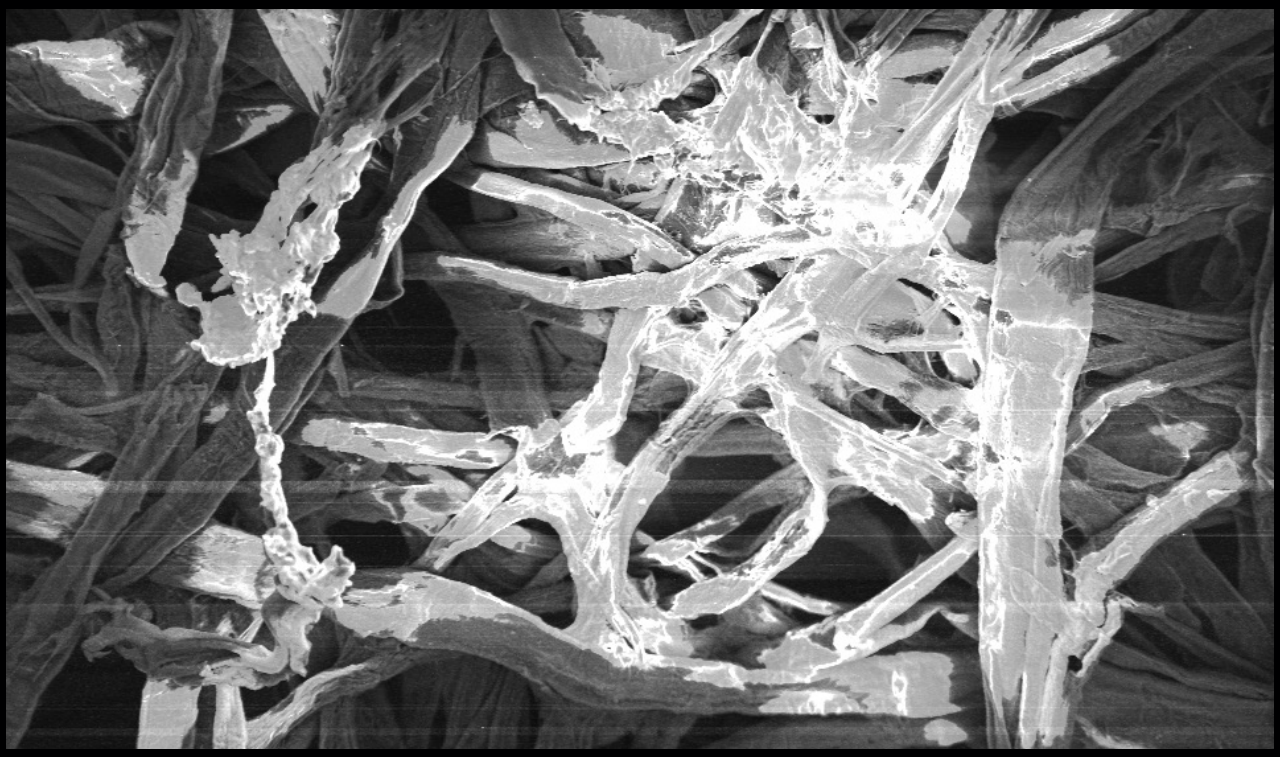


image of tissue paper in an electron microscope

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

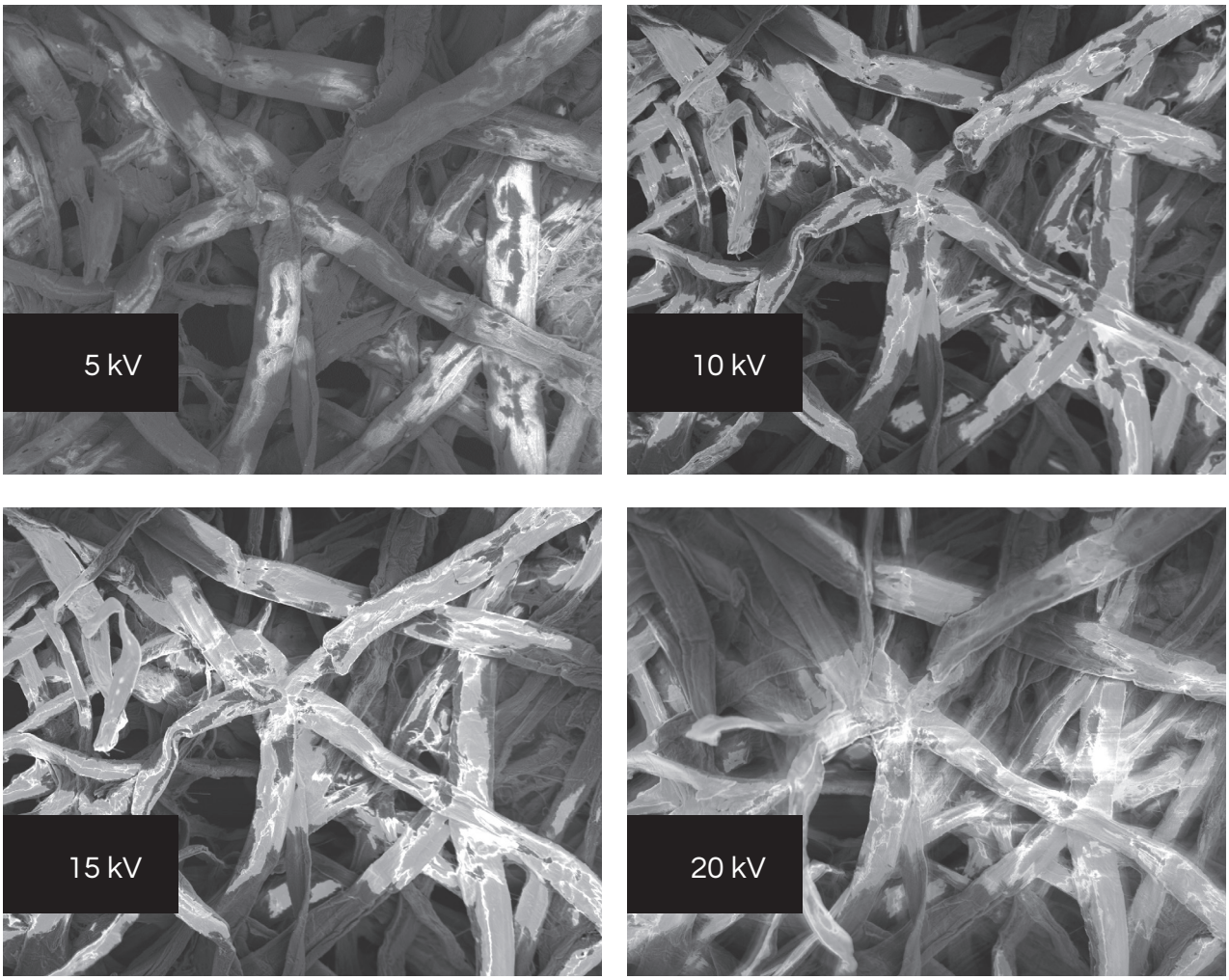
Different charging effects can occur, depending on the degree of charging, and they can result in one or more of the following phenomena:

- 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

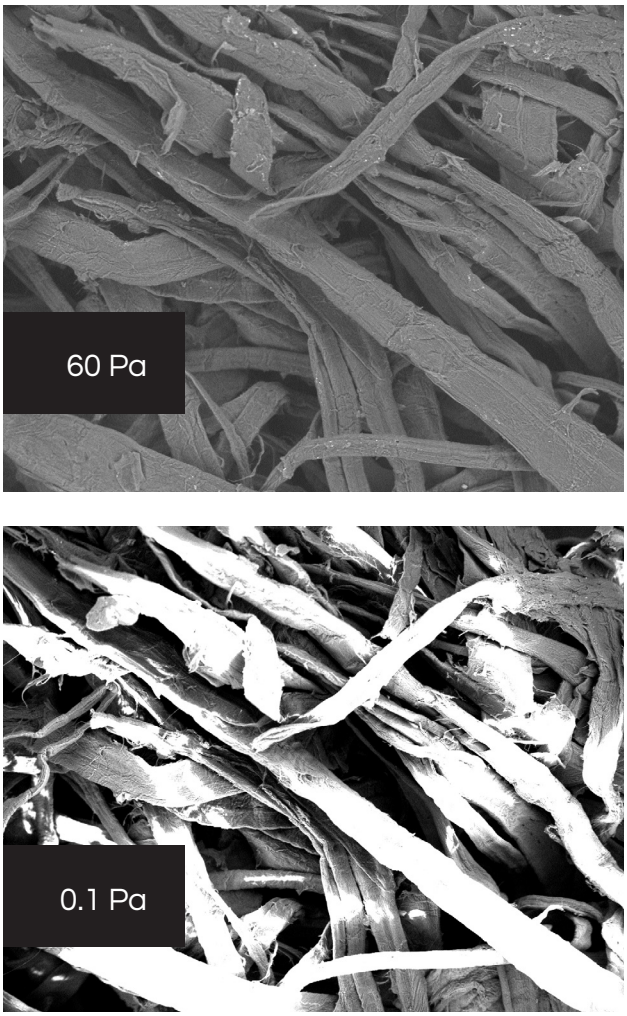
Firstly, charging can be partially avoided by reducing the speed at which electrons hit the sample. This is possible by working at lower acceleration voltages (unit is kV). At high acceleration voltages, electrons will penetrate deeper into the sample and will have more difficulty escaping. At lower 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

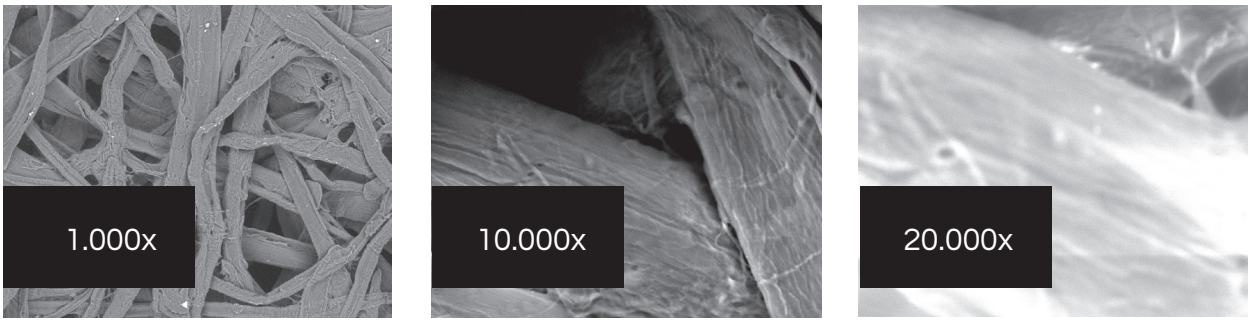
Another method to prevent charging is to work in a reduced (less strong) vacuum. This is done by introducing a gas, air or water vapour into the sample chamber. The gas molecules contact the charged sample surface, become negatively charged by collecting an electron, and then discharge that electron 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

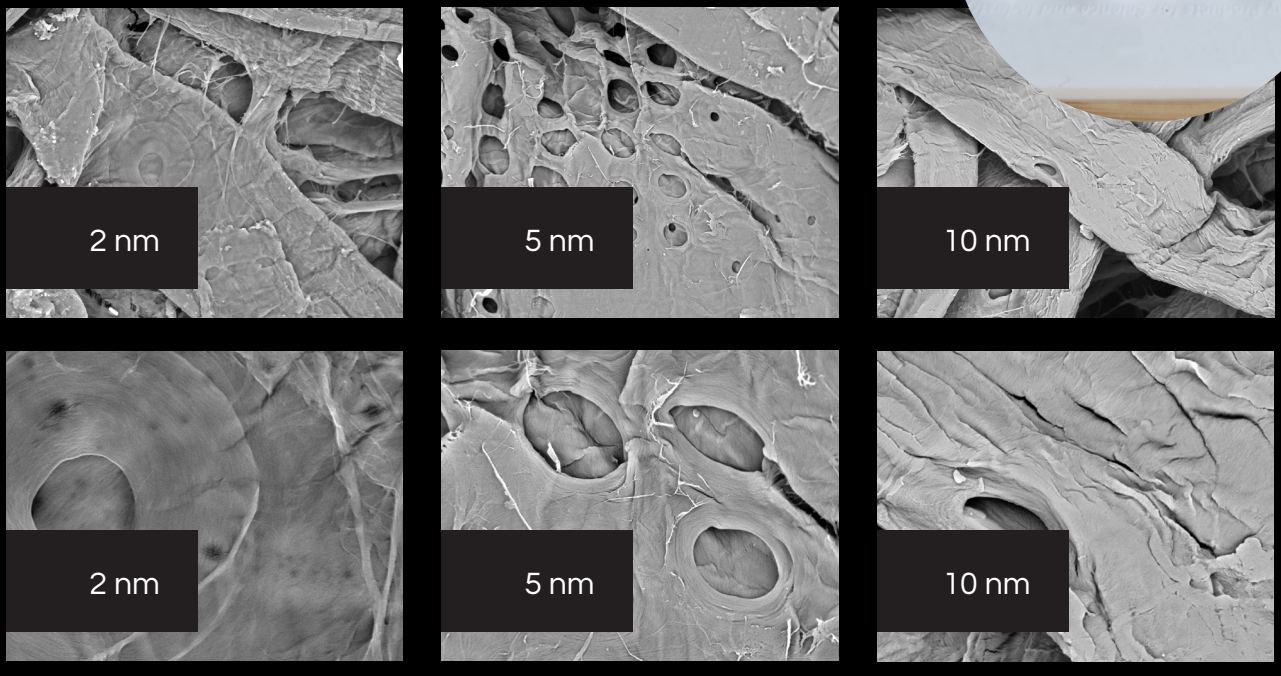
The best image quality of non-conducting materials is obtained by metal sputter coating. A metal sputter coater deposits a very thin layer (typically 1 nm to 10 nm) of conducting material (most commonly gold or platinum) over the surface of the sample.

" 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

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