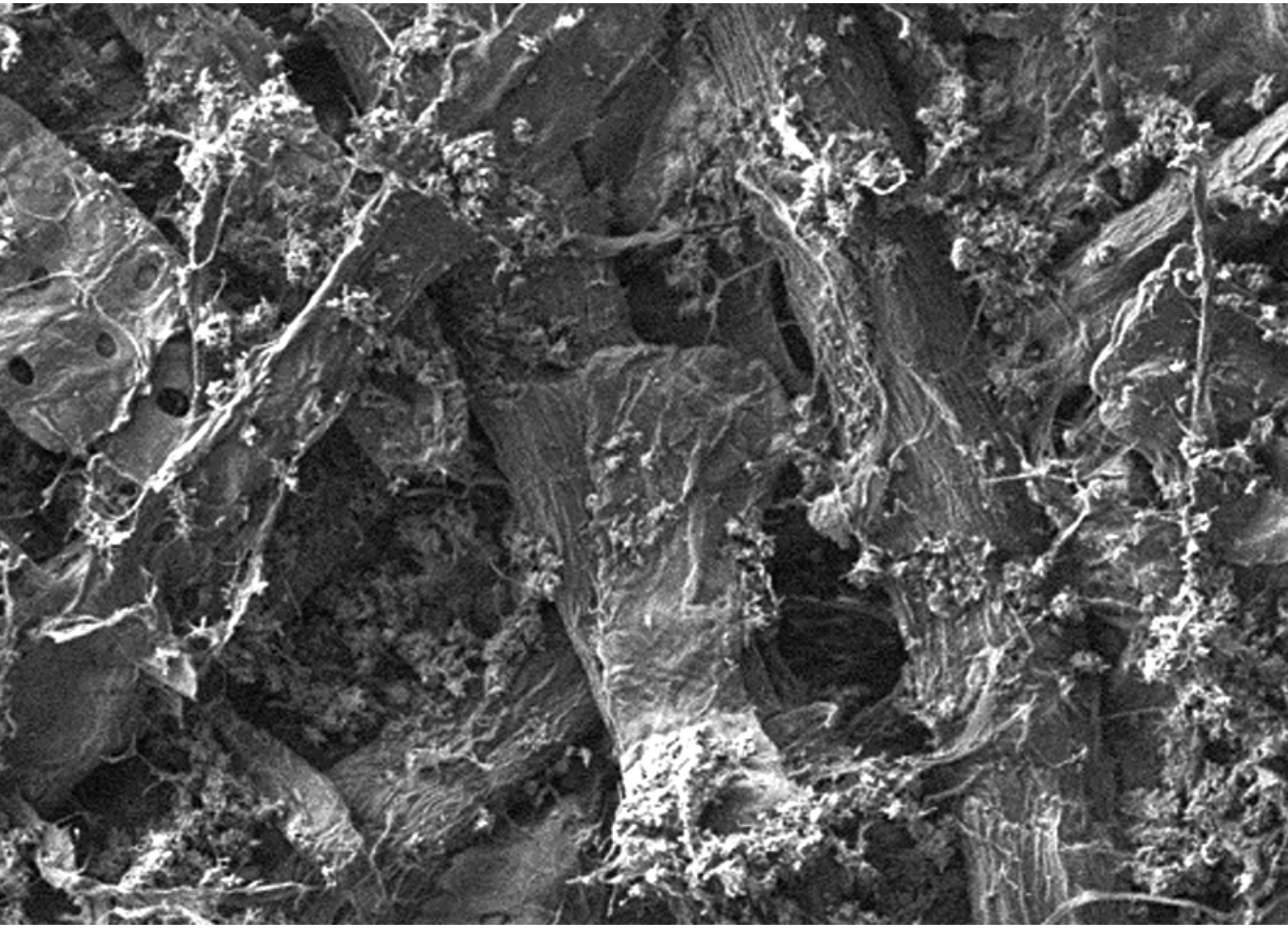
Application Note



Imaging Solutions for the Paper Technology Industry

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Imaging Solutions for the Paper Technology Industry

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Introduction

Those industries concerned with the processing of paper face a range of imaging challenges to provide high quality information to allow them to reach their own targets on technology, productivity, and ultimately profitability. The scanning electron microscope (SEM) is widely used to provide imaging solutions owing to its unique ability to produce images over a wide range of magnifications and specimen environments. One of the principal areas of interest is the interaction of paper fibres with water, which occurs during all phases of the useful life of paper, and conventional SEMs are not able to stabilise liquid water on a specimen. However, thanks to extended pressure ranges, the specimen environment in a SEM can now be controlled so that liquid water can be condensed onto materials of interest.

Instrumentation

In the SEM a focused electron beam is scanned across the specimen. Electrons emitted from the specimen are detected and presented as a grey scale image. With a very high depth of field and a large range of magnifications from only a few times to beyond 100,000 times, the modern SEM provides a flexible and productive tool for surface imaging. The Extended Pressure EVO LS SEMs provide the means to introduce water vapour into the chamber so that truly wet specimens can be imaged. At sufficiently high water vapour pressures, and in combination with a cooled specimen, liquid water can be condensed onto the specimen leading to studies of the interaction of materials with water.

Figure 1 *ZEISS EVO LS15 Extended Pressure SEM.*

The imaging of paper in the SEM with the use of gas to compensate for "charging"

The analysis of paper has always provided a challenge for the high vacuum (HV) only SEMs as the accumulation of electrons within the surfaces of the fibres leads to imaging artefacts referred to as "charging".
The complex three-dimensional microstructure of interlocking fibres frustrates attempts to apply the traditional remedy of sputtering a thin gold coating on the surface of the fibres.
As a partial solution to this problem, the relatively recent introduction of variable pressure (VP) (sometimes referred to as "low vacuum") SEMs that introduce a controlled gas atmosphere at the specimen, has largely overcome the charging problem. For several years, insulating materials have been routinely examined in VP SEMs by using a variable pressure (circa 50 Pa) of air in the specimen chamber. Ionisation of the gas by secondary electrons provides a source of positively charged gas ions that are attracted to the negative charge in surface layers.

This charge balancing mechanism is so effective that the majority of SEMs are now supplied with the capability to operate in this mode. Electron detection in a VP SEM is achieved either by using a detector for backscattered electrons or, in ZEISS microscopes, by the patented variable pressure secondary electron detector (VPSE) described in our literature. The VPSE detector provides true secondary electron imaging necessary for characterising the microscopic surface layers responsible for many macroscopic properties such as feel and friction. One limiting consequence of introducing gas along the electron optical axis of the column is that some fraction of the primary electrons is scattered by gas molecules before reaching the specimen. The effect is minimised in VP instruments by selecting high beam energy to achieve a low scattering probability. Unfortunately surface detail is then lost as the penetration depth of the electron beam becomes very large compared to the characteristic thickness of layers of interest.

This is especially true for low atomic weight specimens often met in the paper industry. In order to make use of the surface imaging provided by low beam energies, the distance over which the primary electron beam is exposed to gas molecules, known as the Beam Gas Path Length (BGPL), should be made as small as possible. Whilst in conventional VP and LV instruments this value may be 10-20 mm, this is usually 1 or 2 mm in the EP instruments. This combination of secondary electron detection, low beam energy, and short BGPL, is a feature of the EVO Series of instruments. Some examples of how this combination of features can be used to provide new imaging solutions for the paper industry are described. The image in Fig. 2 shows the overwhelming influence of unstable surface potentials (“charging”) that electron microscopists have traditionally faced with uncoated insulating specimens even at low beam energies of 3 kV.

Figure 2 *Secondary electron image of the internal structure of copy paper obtained in high vacuum and without prior preparation. The analysis conditions are 3 kV, Everhart-Thornley SE detector. The "charging" artefacts include the white horizontally streaked areas to the top and right of the field together with a distortion of the image. The horizontal field of view is 140 µm.*

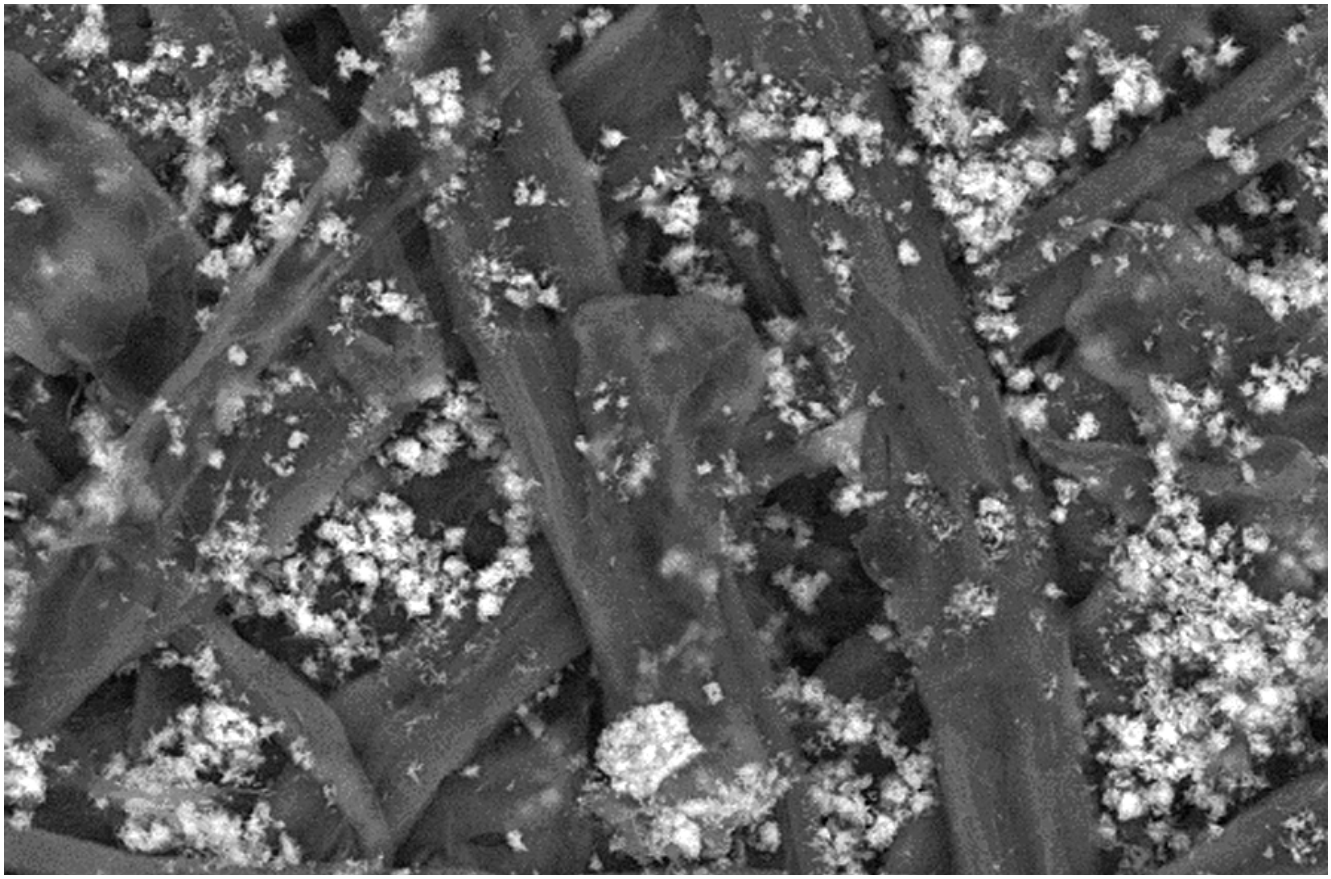

Figure 3 *Backscattered electron image of the internal structure of copy paper obtained with a conventional VP SEM and without prior preparation. The imaging conditions are 20 kV, solid state BSD, 11mm BGPL, and 40 Pa air. The horizontal field of view (identical to Fig. 2) is 140 µm.*

Fig. 3 shows an image that is representative of VP SEMs using high primary beam energy, long BGPLs, and backscattered electron detection. The image shows the structure of the larger paper fibres and appears to give a useful, representative, and complete image of the specimen. In contrast, Fig. 4 shows an image from the same field of the specimen when examined in an EVO instrument using low beam energy, a short BGPL, and true secondary electron detection (with the VPSE detector). The surface detail on the specimen is clear. The analysis conditions for Fig. 4 were 3 kV beam energy and 64 Pa of air with a 2 mm (BGPL). The original magnification in all of the images was 2500 x relative to an 18" display. The image (Fig. 3) obtained using the BSD shows very little surface detail, owing to the generation of BSEs from a greater excitation volume, and so such images should be regarded as complementary to those provided by a true secondary electron detector (Fig. 4).

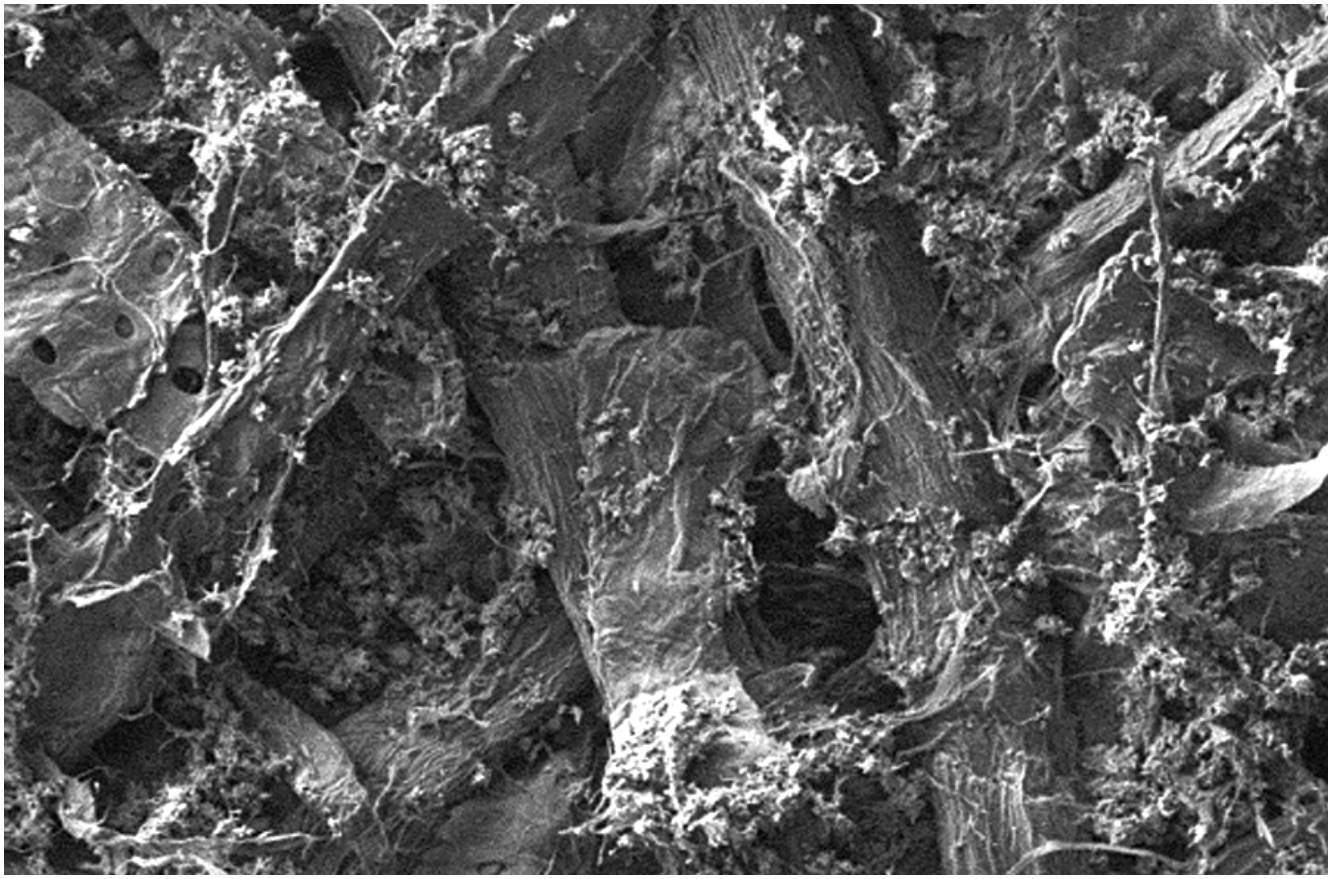

Figure 4 *A secondary electron image of the internal structure of copy paper obtained with an EVO LS Extended Pressure SEM with no prior preparation. The imaging conditions are 3 kV, VPSE detector, 2 mm BGPL, and 64 Pa air. The field of view is identical to Fig. 2 and 3.*

The effect of water on paper fibres

The mechanisms by which paper changes its morphology as a consequence of changes in humidity, and therefore its mechanical strength and stability, is an important consideration for the paper industry. These processes can be observed in real-time in the EVO LS Extended Pressure SEM by introducing water vapour into the chamber. At sufficiently high pressures, liquid water can be condensed onto the specimen and the effects observed. Fig. 5 shows the user menu that controls the temperature and pressure, and hence relative humidity, at the specimen. Images are usually taken using a Peltier cooled stage to reduce the specimen temperature to a few degrees Centigrade, which allows water to be condensed at chamber pressures (typically 650 Pa) significantly below the saturated water vapour pressure at room temperature (2600 Pa). In turn, the lower pressures reduce the adverse effects of beam scattering and therefore allow high contrast SEM images to be obtained.

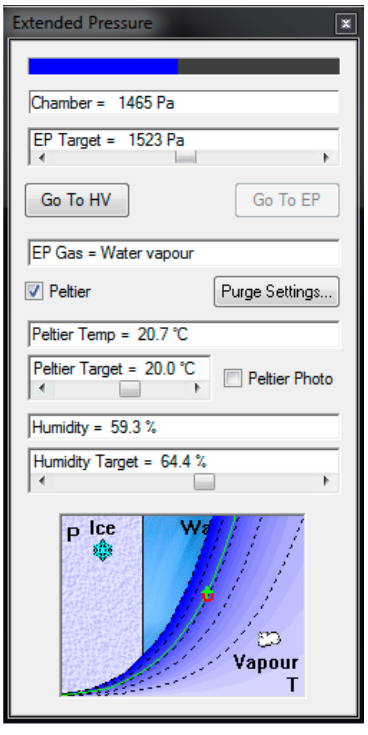

Figure 5
The control panel used to introduce water vapour into environment around the specimen.

One example of this type of experiment is shown in Fig. 6 using the cut edge of some paper. During the wetting phase (Fig. 6b) the paper absorbs water and individual fibres change their morphology. Immediately after the humidity is reduced to remove excess water (Fig. 6c), the fibres are more circular in cross section rather than the oval shape produced by the pressing process in manufacture. After an extended drying phase (Fig. 6d) the recovery processes can be followed in detail. These images were obtained using the BSE detector to provide fast topographic information so that images with high signal to noise ratios can be obtained. Such images can be collected using AVI capture software to record such dynamic processes.

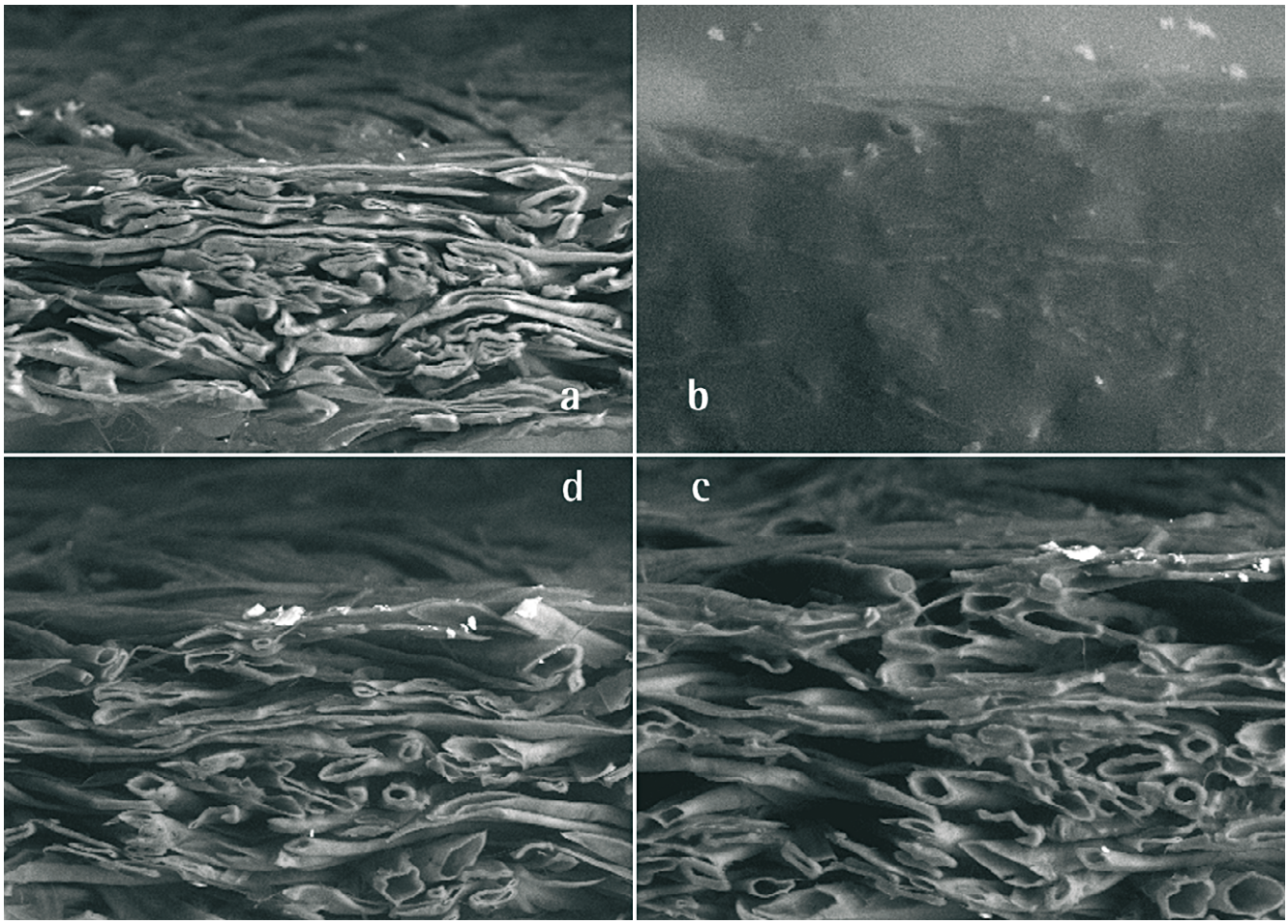

Figure 6a, b, c and d *The response of paper to liquid water.*
a: Cut paper edge.
b: Water flooding of the specimen.
c: Image of the paper immediately after removing excess water.
d: After complete drying at very low humidities. Note that paper has contracted and that individual the fibres have partially reverted to their initial form.
The horizontal field of view is 600 µm.

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