Optimizing the Environmental Scanning Electron Microscope for In Situ Applications

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Introduction

In environmental scanning electron microscopy (ESEM) the imaging gas inside the chamber suppresses charging and outgassing of the sample but it also decreases the signal to noise ratio (SNR) [1]. *In Situ* applications in the kPa regime are limited by poor image quality (e.g. wetting experiments) because short dwell times and low acceleration voltages are necessary for image acquisition.
Recent publications on high pressure capabilities of state of the art microscopes (such as the FEI Quanta series microscopes) have shown that they are working far away from physical limits and that there is plenty of room for improvements [2,3].
The key to high image quality at high pressures is to reduce scattering of the primary beam electrons inside the imaging gas as far as possible while maintaining ideal operation conditions for the SE-detector [4].

Pressure Limiting Aperture Holder

In conventional ESEM the sample chamber is separated by a differential pumping system and two pressure limiting apertures (PLA) from the high vacuum inside the electron column.
A new aperture holder with an optimized design for high pressure applications was created that significantly reduces the additional stagnation gas thickness (the distance the electron beam has to overcome inside the gaseous environment before entering the sample chamber). In addition, PLA1 is exchangeable and optimized for high pressure applications (see figure 1 and 2, [4]).

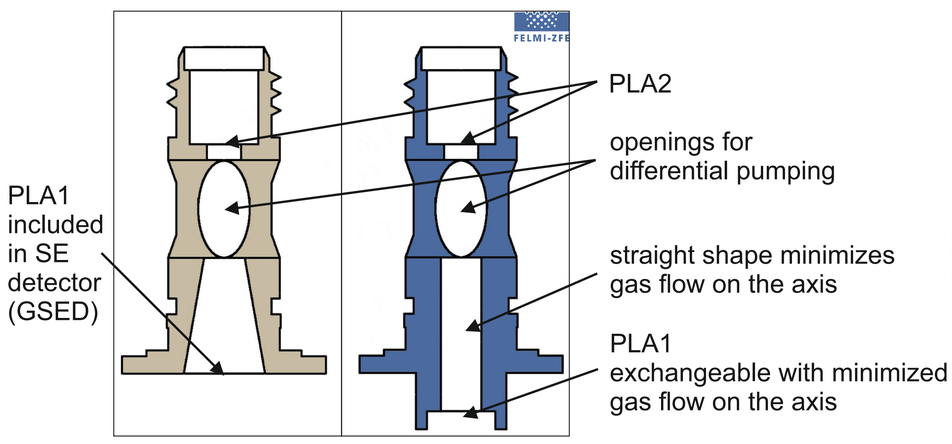

Fig. 1 Schematic drawing of the pressure limiting aperture holder
left: original design; right: new design

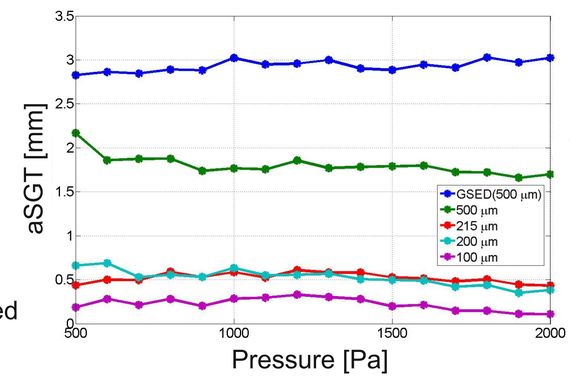

Fig. 2 aSGT [mm] as a function of pressure [Pa] for different PLA1 diameters

SE Detector and Examples

With increasing chamber pressure this SE signal amplification strongly decreases [1].
By repositioning and modifying the shape of the detector the high pressure performance can be optimized [4]. In comparison to a conventional detector the electric field nearby a needle detector with very small tip radius R < 10 µm) is strong enough for SE amplification and by positioning the needle on the sample table it operates at ideal conditions regardless of pressure and working distance. Extensive cooling of the sample to decrease the dew point is no longer necessary and experiments close to its natural state are possible.
A by-product of this design is that the conventional position of the backscatter electron detector (BSE) at the end of the column is no longer blocked by the SE detector.

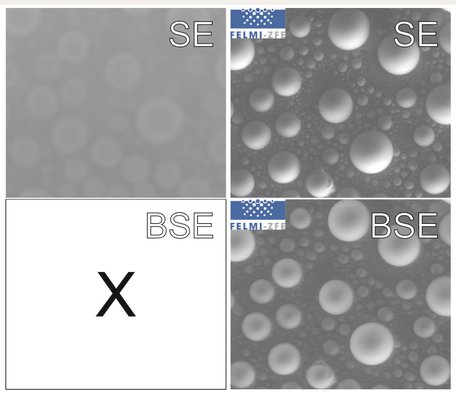

Fig. 3 SE images of tin spheres on carbon
(500 Pa (H2O), E = 5 keV, I = 1 nA, DT = 30 µs)
left: original design; right: new design

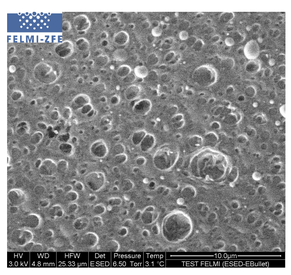

Fig. 4 SE image of a membrane with condensed water droplets
(870 Pa (H20); 3,1°C;
I = 0,8 nA; DT = 10 µs)

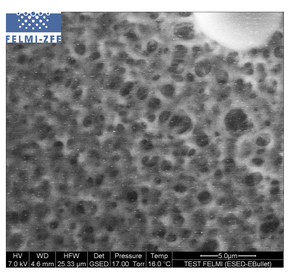

Fig. 5 SE image of a membrane
(2,3 kPa (H20); 16°C;
I = 0,8 nA; DT = 10 µs)

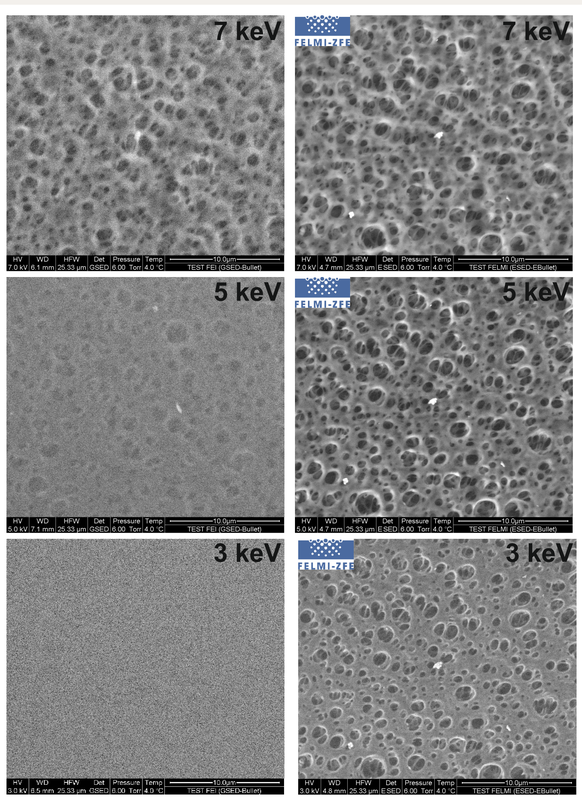

Fig. 6 SE image of a membrane
(800 Pa (H20); 4°C;
I = 0,8 nA; DT = 10 µs)
left: original design; right: new design

Conclusion

The new design enables higher pressure, shorter dwell time and lower electron energy ESEM applications. The overall improvements can be seen in figure 3.
With this outstanding signal to noise ratio limits of conventional ESEM technology can be crossed. Wetting experiments at low acceleration voltages and low dwell times are possible as well as imaging liquid samples without cooling (see figure 4,5,6).

References/ Literature

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3. Fitzek H., Schroettner H., Wagner J., Hofer F., Rattenberger J., Experimental evaluation of environmental scanning electron microscopes at high chamber pressure, Journal of Microscopy Vol. 260, 133-139, 2015
4. Fitzek H., Schroettner H., Wagner J., Hofer F., Rattenberger J., High-quality imaging in environmental scanning electron microscopy – optimizing the pressure limiting system and the secondary electron detection of a commercially available ESEM, Journal of Microscopy Vol. 262, 85-91, 206

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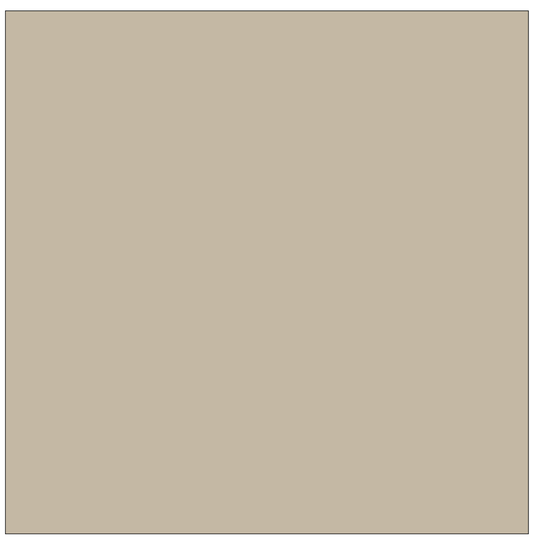
CORPORATE DESIGN FARBPALETTE



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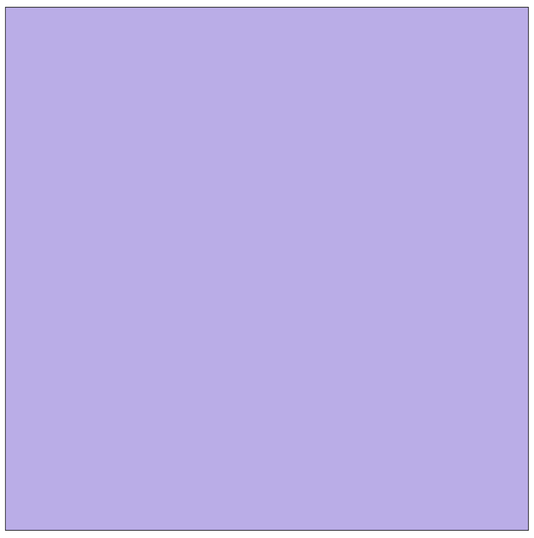

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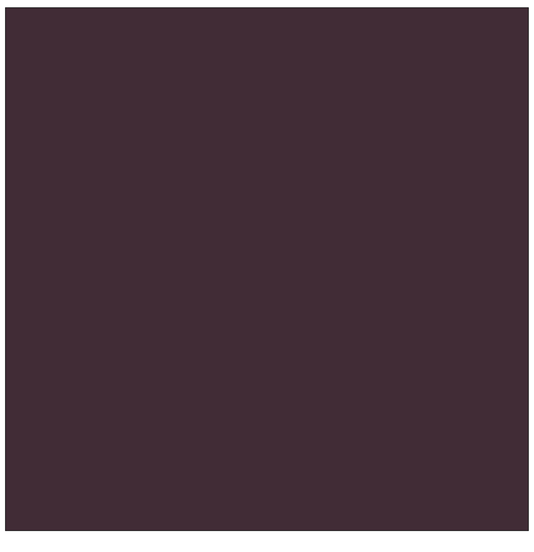

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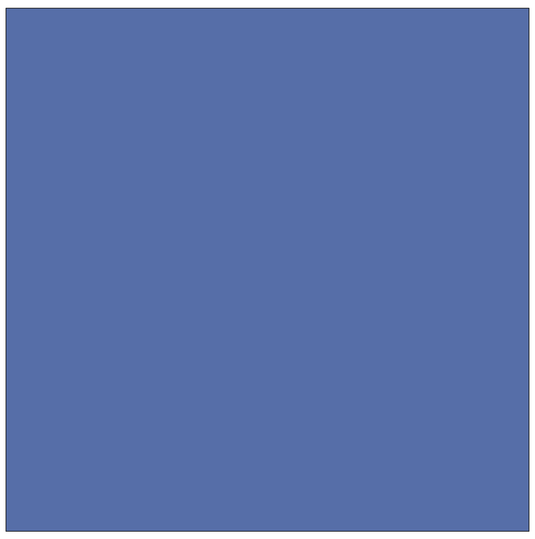
LOGOBLAU



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