Pushing the Limits of Environmental Scanning Electron Microscopy

FELMI-ZFE

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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]. Especially applications in the kPa regime are limited by poor image quality (e.g. wetting experiments).
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]).

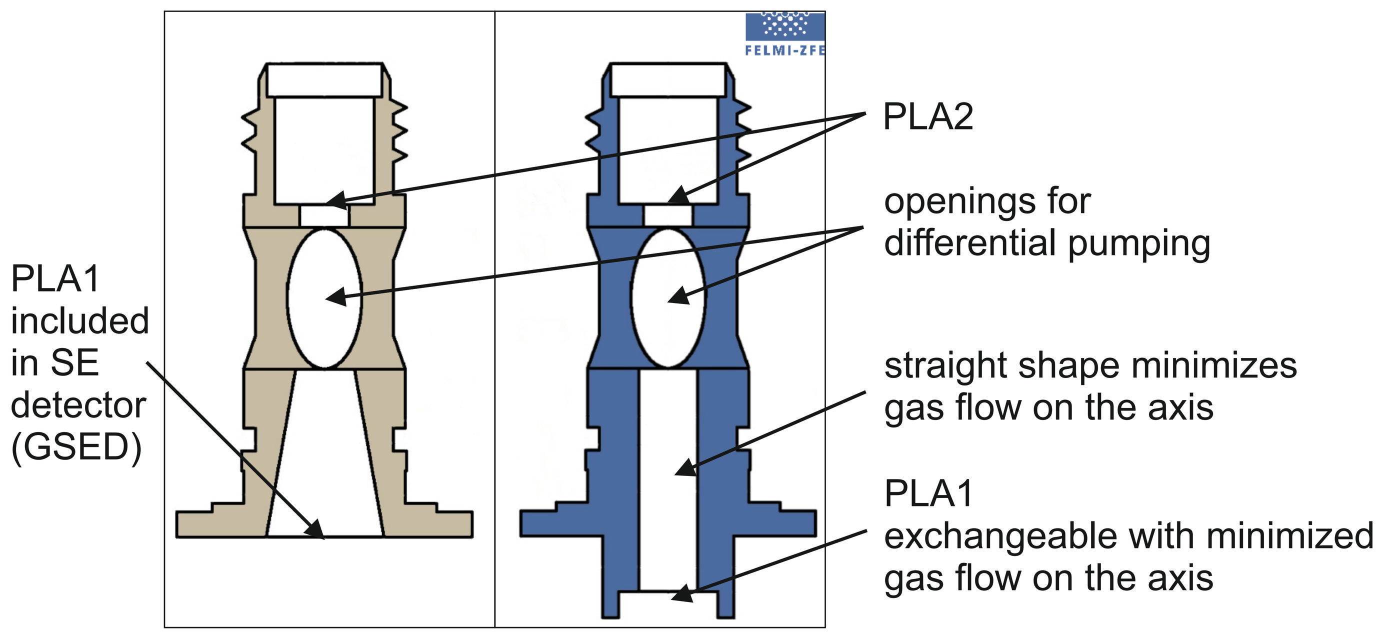

Fig. 1 Schematic drawing of the pressure limiting aperture holder
left: original design; right: new design

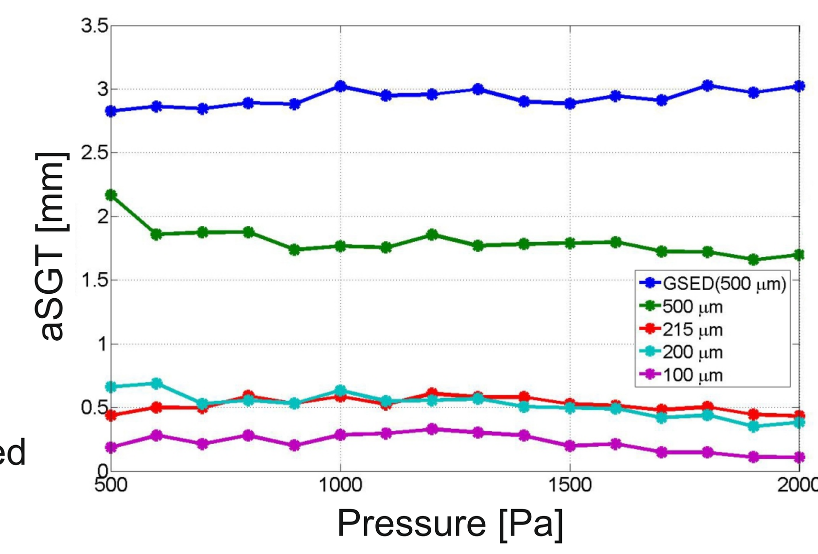

Fig. 2 aSGT [mm] as a function of pressure [Pa] for different PLA1 diameters

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 (see figure 3).
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.

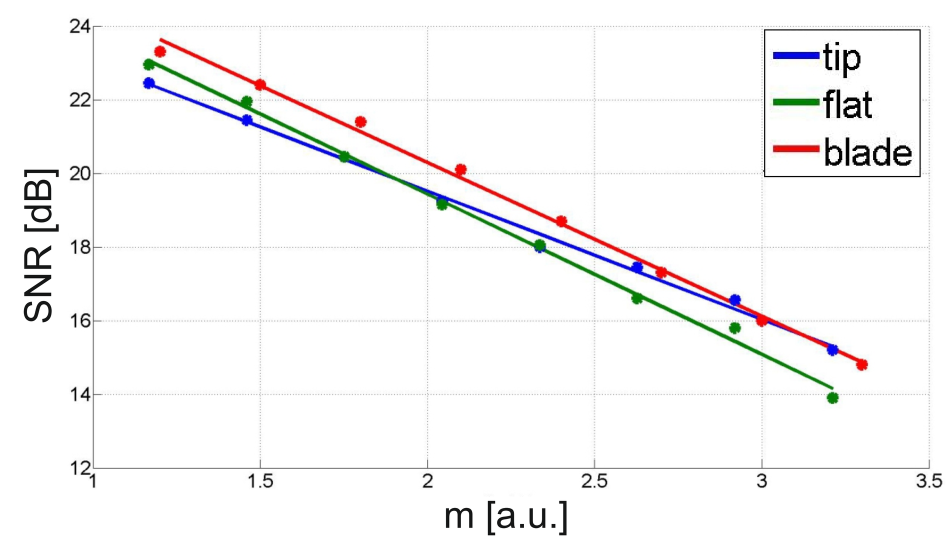

Fig. 3 SNR [dB] as a function of the average number of interaction per electron m [a.u.] for different detector geometries

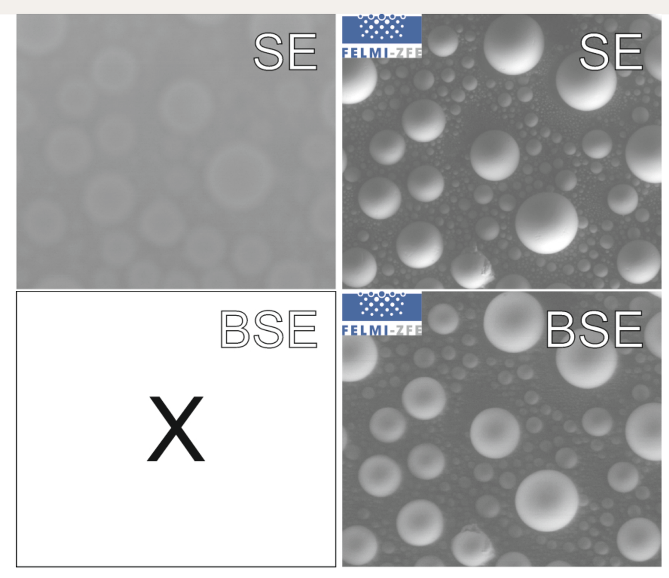

Fig. 4 SE images of tin spheres on carbon (500 Pa (H_2O), E = 5 keV, I = 1 nA, DT = 30 µs)
left: original design; right: new design

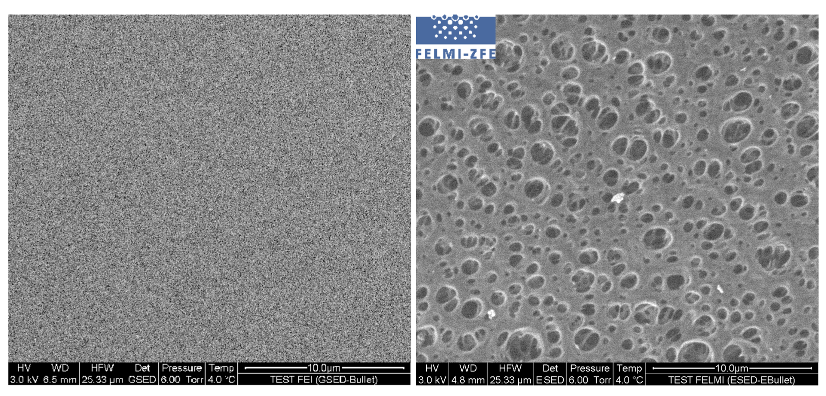

Fig. 5 SE image of a membrane (800 Pa (H20); E = 3 keV; 4°C; I = 0,8 nA; DT = 10 µs)
left: original design; right: new design

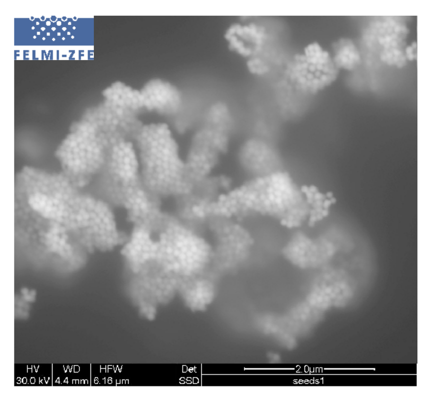

Fig. 6 BSE image of gold nanoparticles in oil at 10 kPa chamber pressure

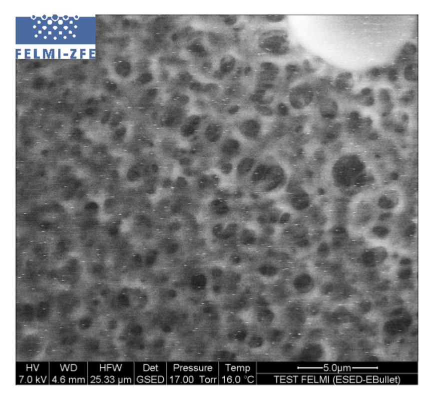

Fig. 7 SE image of a membrane (2,3 kPa (H20); 16°C; I = 0,8 nA; DT = 10 µs)

Conclusion

The new design enables higher pressure, shorter dwell time and lower electron energy ESEM applications. The overall improvements can be seen in figure 4.
With this outstanding signal to noise ratio limits of conventional ESEM technology can be crossed. Backscatter imaging at high pressure, wetting experiments at low acceleration voltages and low dwell times are possible as well as imaging liquid samples without cooling (see figure 5,6,7).

References/ Literature

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