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Novel, low-cost hardware for ‘STEM in SEM’ imaging

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Background

Scanning transmission mode imaging can offer better contrast and resolution than secondary electron imaging in nano, soft and biological materials[1]. The concept of ‘STEM in SEM’ is to replicate the improved image quality of scanning transmission electron microscopy (STEM) in lower-cost scanning electron microscopes (SEM). In the hardware discussed in this work, a conversion plate allows the transmitted electrons to produce a signal using the Everhart-Thornley Detector (ETD). This enables bright field STEM imaging to be added to most SEMs through using only an additional sample holder.

Although this is not a new concept [2], little work has been published on the optimisation of the design and operational parameters. A higher efficiency allows a lower probe current to be used. In the SEM, the spot size is limited by the probe current and the brightness of the source. Due to this the improved efficiency in ‘STEM in SEM’ results in improved resolution.

The schematic (a) shows the basic concept of the ‘STEM in SEM’ holder. A standard TEM grid is clamped between plates at the top of the holder. The electron beam passes through the sample and reaches a conversion plate. Here transmitted electrons generate secondary electrons within the plate which may be detected by the ETD. Furthermore, an aperture may be inserted between the sample and the conversion plate to control the acceptance angle of the detector.

Methods

The optimisation process consisted of a series of experiments analysing the effect of each parameter in isolation.

The efficiency of the conversion of transmitted electrons to secondary electrons was simulated using CASINO, a Monte Carlo method simulation program [3] . The modelling indicated an improved conversion yield when an oxide forming metal coats the surface. The model was validated by comparing the ETD signal produced when a range of coated glass substrates were scanned with a focussed electron beam. The increase in secondary electron yield due to a sputtered aluminium surface coating was verified experimentally. As secondary electrons only escape from within a few nanometres of the surface, only a coating of high-yield material is needed.

This was implemented at a low cost by sputtering a gold-aluminium bilayer coating onto glass substrate.

Significant geometrical optimisations in both the angle of the conversion plate and the working distance were found. The image brightness was measured whilst varying the conversion plate angle. It was found that the efficiency of the plate has a significant peak at the optimised angle. It is thought that this is for two reasons. Firstly, as the angle increases, the transmitted electrons travel a greater distance within the material at a depth at which secondary electrons can escape. Secondly, the alignment of the emitted electrons to the ETD improves the detection efficiency.

One of the challenges with 'STEM in SEM' is the convolution of the desired transmitted signal with undesirable secondary electrons from the sample. The difference in detection efficiency of the ETD at different working distances was used to combat this. The ETD has poor detection efficiencies close to the polepiece. When using a sample working distance of 3mm and a converter plate working distance of 18mm, the signal was dominated by the transmission mode. The noise was further reduced by a reduction of the ETD cage bias from the 250-400V typically used to 50V. This limited the detected electrons to those emitted aligned closely to the detector.

The overall performance of the 'STEM in SEM 'converter was evaluated by imaging drop cast 40nm and 20nm nominal size silver nanoparticles on a holey carbon TEM grid. Ostwald ripening of the nanoparticles produced a broad distribution of particle sizes for resolution testing. The samples were imaged at 30kV using a tungsten filament SEM. The other microscope parameters were adjusted to suit the two imaging methods.

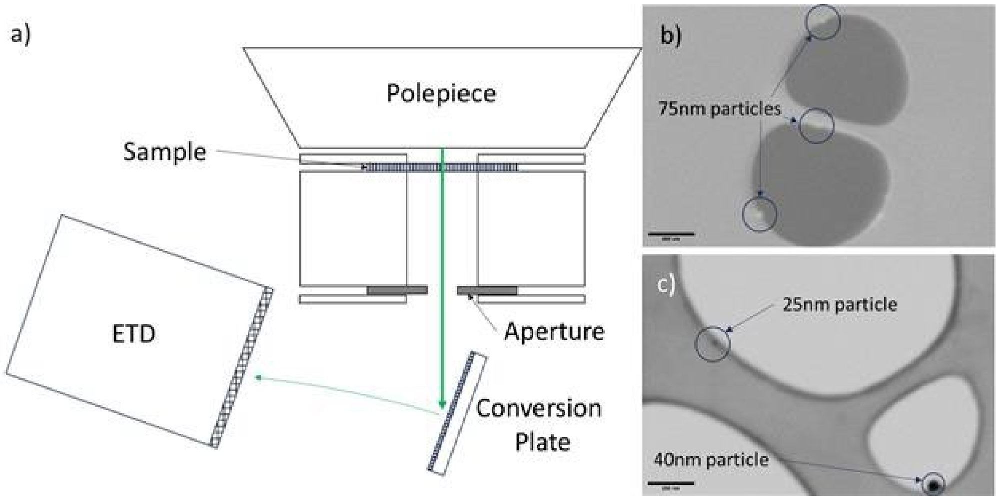
Results

The figure shows silver nanoparticles on a holey carbon film observed whilst using a tungsten filament SEM in (b) secondary electron mode at 30kX magnification and (c) using the STEM converter at 75kX magnification. The contrast was improved using the optimised 'STEM in SEM' converter when compared to secondary electron imaging. An improvement in contrast to noise ratio was seen in the STEM images. The improved contrast enables faster and easier imaging of the nanoparticles in the STEM mode. The smallest particles seen in secondary electron mode were 75nm in diameter. With the STEM converter, this was improved to 25nm.

Conclusion

The optimised 'STEM in SEM' converter presented here is a low-cost method to enable transmission mode imaging in the SEM. It has been shown this can be utilised to improve the image quality when imaging nanoparticles. Although, this does not represent new functionality compared to dedicated silicon STEM detectors, it is a much more accessible method of adding STEM capabilities to an SEM.

Graphic:



Keywords:

STEM, SEM, nanoparticles, low-voltage STEM

Reference:

[1] U. Golla-Schindler, B. Schindler, G. Schneider, Contrast and spatial resolution enhancement with the transmission mode in SEM, Microscopy and Microanalysis 27 (2021) 1820–1822. https://doi.org/10.1017/s1431927621006668.
[2] U. GOLLA, B. SCHINDLER, L. REIMER, Contrast in the transmission mode of a low-voltage scanning electron microscope, J Microsc 173 (1994) 219–225. https://doi.org/10.1111/J.1365-2818.1994.TB03444.X.
[3] H. Demers, N. Poirier-Demers, A.R. Couture, D. AJoly, M. Guilmain, N. de Jonge, D. Drouin, Three-dimensional electron microscopy simulation with the CASINO Monte Carlo software, Scanning 33 (2011) 135–146. https://doi.org/https://doi.org/10.1002/sca.20262.