Progress and applications of scanning electron microscopy in the presence of a gas

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Following on from the development the scanning electron microscope (SEM), the past couple of decades have seen a proliferation of commercially available SEMs that do not require high vacuum conditions in the specimen chamber. Whilst the exact technological specifications vary, the unique feature of this genre of SEM is the presence of one or more gases in the region of the specimen. The excitation and ionization of gas atoms or molecules to produce electrons and photons and the movement of both positive and negative charge carriers are processes of great importance in providing detectable signals, while the subsequent formation of positive ion by-products allows insulating specimens to be imaged without the need for a conductive coating.

Air and water vapour are commonly used as imaging gases, and water vapour can be used to control the thermodynamic stability of moist or liquid specimens as well as having a role in dynamic hydration and dehydration experiments, although alternative gases such as nitrogen and helium may be selected for a variety of other reasons. Applications vary widely, from nanometrology and materials science examples to biological and soft materials, and include all manner of *in situ* experiments from tensile testing to high temperature redox reactions. There has been much progress in imaging for both the 'low vacuum' or 'variable pressure' case, where the gas pressure need only be sufficient to mitigate negative charging and for the 'environmental' case where the gas pressure is a factor in maintaining some form of specimen equilibrium or to stimulate a reaction or to investigate the behaviour and properties of the specimen.

One important area that continues to progress is our understanding of the physical basis for imaging and related processes in a gas. This includes elucidation of the behaviour of positive ion by-products, consideration of the specimen and its dielectric properties as an integral part of the system and the role of beam-gas interactions in controlling surface chemistry. Such knowledge has led to much improved signal collection (see figure 1) and interpretation of contrast, which is well documented in the literature (see [1] and references therein) as well as suggesting a means for keeping specimens free of contamination [2]. Indeed, electron beam-gas interactions are beginning to find practical use for *in situ* etching and/or chemical vapour deposition to yield nanofabricated structures [3-6].

Technological developments include the biasing of specimens to reduce the primary electron landing energy, originally used in the high vacuum SEM [7, 8], to enable high resolution, high contrast surface-sensitive imaging at low energies [9], and the introduction of secondary electron detectors that operate at pressures of up to 40 kPa (30 torr), allowing experiments to be carried out at pressures and temperatures at or closer to real conditions. This has implications for carrying out reactions such as catalysis and observing biological cells and tissues. Figure 2 shows a sample of gypsum imaged at high humidity and room temperature (corresponding to around 100% relative humidity). Thin specimens can be imaged by collecting transmitted electrons and, if the detector offers specimen cooling in

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conjunction with a water vapour imaging environment, gives access to nano-suspensions in the liquid state [10] and charge-free, highly detailed imaging of polymeric materials [11].

1. D.J. Stokes, *Principles and practice of variable pressure/environmental scanning electron microscopy (VP-ESEM)*, 2008. John Wiley & Sons.
2. M. Toth *et al*, Journal of Applied Physics, 2009. In press.
3. K. Molhave *et al*, Nano Letters, 2003. **3**(11) p. 1499-1503.
4. B.D. Myers and V.P. Dravid, Nano Letters, 2006. **6**(5) p. 963-968.
5. M. Toth *et al*, Journal of Applied Physics, 2007. **101**(5).
6. M. Toth *et al*, Nano Letters, 2007. **7**(2): p. 525-530.
7. I. Mullerova and L. Frank, Scanning, 1993. **15**: p. 193-201.
8. L. Frank *et al*, Journal of Applied Physics, 2006. **100**(9).
9. D. Phifer *et al*, Microscopy Today, July 2009.
10. A. Bogner *et al*, Ultramicroscopy, 2005. **104**(3-4): p. 290-301.
11. L.Staniewicz *et al*, *Proceedings of Microscopy & Microanalysis 2009*. In press.
12. M. Toth *et al*, Applied Physics Letters, 2006. **88**(2): p. Article no. 023105.
13. B.L. Thiel *et al*, Review of Scientific Instruments, 2006. **77**(3): p. Article No. 033705.

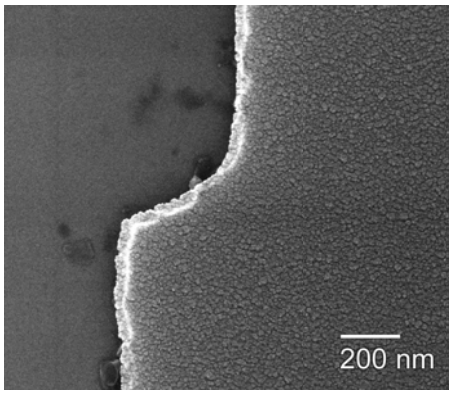

Figure 1. Ultra-high resolution (~1 nm) image of electrically-insulating photolithographic mask. The texture of the chrome oxide surface layer is clearly visible. To the left, the quartz substrate is seen. Secondary electron imaging with 40 Pa (0.3 torr) water vapour: the electron amplification gas pathway is maximized at a short working distance by using both electrostatic and magnetic fields, while excess positive ions are removed, to ensure highly efficient signal collection. Courtesy of M. Toth. (See also [12, 13]).

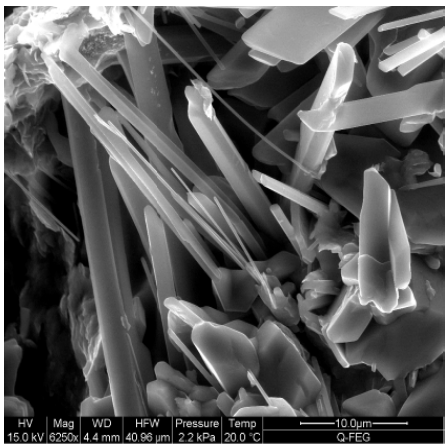

Figure 2. Secondary electron image of gypsum under conditions of high pressure: 2.2 kPa (16.5 torr) water vapour and a specimen temperature of 20°C, to give ~100% relative humidity. Courtesy of E. Baken.

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