Optimization of the Gun, Lenses, and Detectors for High-Vacuum SEM Studies of Non-Conductive Specimens

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Classically, SEM imaging of the topography of uncoated bulk, insulating specimens relies on the optimization of accelerating voltage of the incident beam. At one of two 'quiescent' gun voltages, an uncoated, perfectly non-conductive sample accumulates neither a net negative nor positive static surface charge. In the 1980s, a variable-pressure approach (VP-SEM) was developed whereby an injected 'imaging gas' produces positive and negative ions that compensate an accumulated surface charge permitting SE imaging over a wider-range of accelerating voltages. More recently, advances in SEM gun, lens, and detector designs afford high-vacuum SEM users additional strategies to achieve ultra-high resolution topographical imaging of non-conductive specimens over a range of 'landing energies' ranging from the conventional low-energy range (1-5kV) all the way down into the ultra-low energy regime of 10s of volts.

The ideal high-vacuum SEM for insulating engineering- and soft-materials provides flexible hardware and software that permit the SEM operator to minimize and/or stabilize surface charging, as well as to filter-out those signals containing charge-related distortions. Figure 1a-c shows an in-lens SE detector comparison of a cuttlebone sample imaged in the acceleration voltage regime classically associated with distortions arising from surface charge effects. Figure 1a shows typical charging effects while the injection of oxygen as an imaging gas to locally raises pressure to compensate surface charging (figure 1b).

However, a comparison of the sharpness of the injected gas image with that of an image produced using only a monochromated incident beam (figure 1c) reveals the true cost of using an imaging gas as lost topographical detail of the surface of the cuttlebone. SEM micrographs in figure 2 reveal the morphological features of an uncoated surface of a beam sensitive dehydrated protein foam. In this case, an annular detector is inserted in the gap between the lens and the sample. The concentric rings of the annular detector segment four separate signals in real-time. Charging is largely confined to the inner-most detector ring, with the outer rings revealing topographical features without any obvious distortion.

Ultra-high-resolution imaging of the true surface of non-conductive samples is possible with landing energies as low as 20V (figure 3). Tuning the ratio of gunto- cathode lens biasing enables the operator to vary the dispersion of the secondary signal across the detector for optimized information content. At any time in the image acquisition process, a magnetic immersion field may be deployed to explore lower beam currents and increase resolution in both the in- and ex-lens signals without sacrificing any signal-to-noise.



Figure 1a-c: HV-SEM, TLD-SE micrographs of cuttlebone showing charging (a) disappearing with (b) injected oxygen and (c) a monochromated incident beam. Acquisition conditions for figs. 1a, b: 5kV, 25pA, fig. 1c: 2kV and 3.1pA.



Figure 2a-d: Simultaneously acquired HV-SEM micrographs from dehydrated foam. The DBS detector rings segment the SE signal by angle of departure from the surface enabling undistorted imaging of non-conductive samples



Figure 3a, b: Low-kV HV-SEM micrographs of uncoated pollen captured at lowand ultra-low landing energies using the DBS detector in cathode lens mode. The DBS low-angle signals exclude charging and the cathode lens adjusts landing energy and the angular dispersion of the SE signal across the detector.