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The "Great VPSEM Gotcha": Great VPSEM Imaging Does Not Imply Great VPSEM X-ray Microanalysis! Degraded Spatial Resolution Is Always Imposed by Gas Scattering

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The subject of this paper is not new! Indeed, the problem of the degraded spatial resolution of electron-excited X-ray microanalysis due to elastic scattering of beam electrons with gas atoms was recognized at the very dawn of the emergence of variable pressure scanning electron microscopy (VPSEM) [1-4]. However, based upon repeated inquiries that we continue to receive, it is apparent that many users of VPSEM, an operational capability of more than 90% of SEMs delivered in the last ten years, are the unknowing victims of the "Great VPSEM Gotcha". The "Gotcha" is the profound and misleading difference in the impact of gas scattering upon electron imaging and X-ray microanalysis. VPSEM images with backscattered and secondary electrons can retain essentially all of the spatial resolution achievable in conventional high-vacuum SEM, even if only a small fraction of the original beam current remains within the focused beam after gas scattering. Although the beam electrons scattered into the broad skirt interact with specimen features that are different from those encountered by the unscattered electrons that remain in the focused beam, these remote skirt events are spatially uncoordinated and occur with extremely low area density compared to the focused beam. The BSE and SE produced in the skirt thus effectively contribute only noise to the image. The reduction in the signal-to-noise ratio can be readily compensated by increasing the pixel dwell time to recover image visibility. However, these same skirt electrons create characteristic and continuum X-rays from whatever atoms they encounter. These remotely produced X-rays are not excluded by the collimator and cannot be distinguished in the measured energy dispersive spectrum (EDS) from those X-rays generated within the interaction volume produced by the focused beam. The impact of gas scattering on EDS spectra depends strongly on the beam energy, the gas species, and the gas path length (the product of pressure and distance). To estimate these effects, Danilatos developed analytical expressions for the radius of the gas scattering skirt and the fraction of the unscattered beam remaining [5]. While these equations are useful for describing the extent of the skirt, the impact of gas scattering on the EDS spectrum depends in detail on the distribution of scattered electrons within the skirt and the particular materials encountered. Monte Carlo electron trajectory simulation can model gas scattering for specific VPSEM operating conditions and the particular specimen geometry situation [6]. Examples of the electron distribution and the EDS spectra calculated with the Monte Carlo simulation embedded in NIST DTSA-II are shown in Figures 1 and 2 for a 5 µm cube of FeS_2 on Al at $E_0 = 10$ keV. For typical conditions encountered in VPSEM operation, the gas skirt extends hundreds of micrometers, and 90% or more of the measured EDS spectrum can originate outside the focused beam interaction volume. Even at the threshold of useful VPSEM operation where the elevated chamber pressure just suppresses charging of uncoated insulating specimens, the gas scattering skirt extends tens of micrometers and may contain 30% or more of the original beam current. Thus, VPSEM operation always compromises X-ray measurements to the degree that microanalysis at a spatial resolution equivalent to high vacuum operation is not possible. Rather, the analyst must realize that the "analysis footprint" inevitably creates a composite spectrum with remote contributions that often overwhelm the information generated within the nanometer-to-micrometer dimensions of the interaction volume created by the focused beam.

References:

[1] R. Bolon, C. Robertson, E. Lifshin, Microbeam Analysis-1989, ed. P. Russell (San Francisco Press) p. 449.
[2] G. Danilatos, Scanning Microscopy **4** (1990), p. 799.
[3] E. Doehne, D. Stulik, Scanning Microscopy **4** (1990), p. 275.
[4] E. Doehne, N. Bower, Microbeam Analysis **2(S35)** (1993)
[5] G. Danilatos, Micros. Res. & Tech. **25** (1993) p. 354.
[6] N. Ritchie, DTSA-II available free at: www.cstl.nist.gov/div837/837.02/epq/dtsa2/index.html

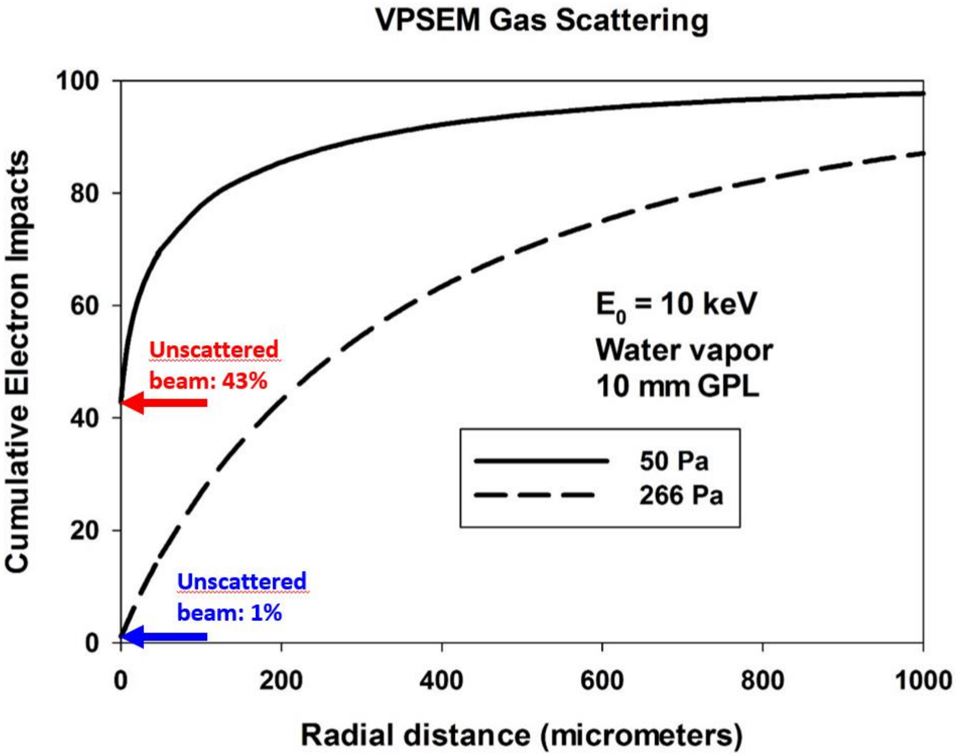

Figure 1. Electron impact profiles calculated with the Monte Carlo electron trajectory simulation in NIST DTSA-II.

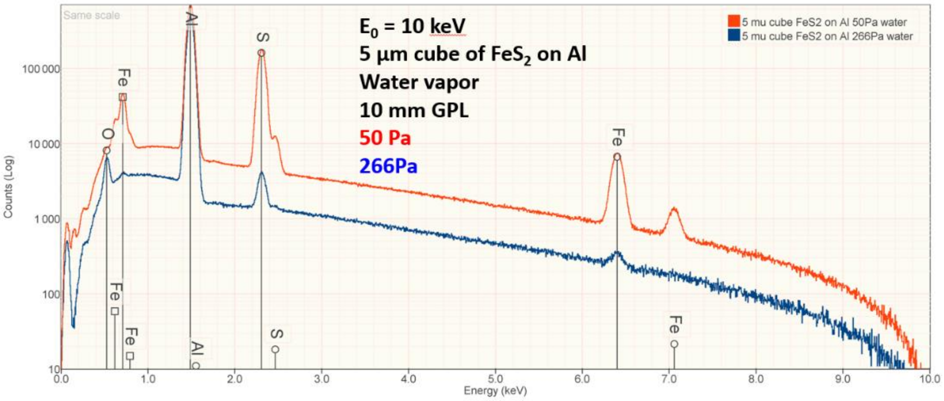

Figure 2. Corresponding EDS spectra calculated for a 5 µm cube of FeS_2 on an Al substrate.