302
doi:10.1017/S1431927616002361
Microsc. Microanal. 22 (Suppl 3), 2016
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Strategies for Obtaining High Spatial Resolution in Imaging and Spectroscopy of Beam-sensitive TEM Specimens

R.F. Egerton[1]

[1.] Department of Physics, University of Alberta, Edmonton, Canada T6G 2E1.

Spatial resolution is the central aim in microscopy, and is determined by four main physical effects in the case of TEM imaging and EDX or energy-loss spectroscopy:

1. Instrumental factors, including the lens aberrations of the imaging lenses (in CTEM, EFTEM) or probe-forming lenses (STEM, spectroscopy) [1] .

2. Beam spreading (in STEM and spectroscopy) arising from incident-beam convergence and from elastic scattering of the primary electrons. This effect is significant for thicker specimens or lower accelerating voltages but can sometimes be limited by use of an angle-limiting aperture; see Figure 1.

3. Delocalization of the inelastic scattering, particularly in the case of EELS and EFTEM imaging. The delocalization distance is roughly (15nm)/$E^{3/4}$, where E = energy loss, giving near-atomic dimensions for core losses, a few nm for valence losses and several tens of nm for vibrational losses [2]. In a crystal, channeling effects complicate the situation but may reduce the amount of delocalization.

4. Statistical factors, including electron-beam shot noise and readout noise. Shot noise depends on electron-source brightness and recording time in the case of a robust specimen, but is limited by radiation damage for a beam-sensitive specimen. Statistical factors then predominate over all others and the dose-limited resolution (DLR) is given by [3]: $\delta = (\mathrm{SNR})\,(\mathrm{DQE}\; F\; D_e)^{-1/2}\,(C^2+3C+2)^{1/2}\,/|C|$, where SNR ~ 5 is the *required* signal/noise ratio, DQE represents the noise performance of the electron (or x-ray) recording system, F is the signal/primary-electron count ratio, D_e is a characteristic fluence (in electrons per unit area) beyond which the signal is lost due to radiation damage, and C is the contrast ratio between resolution elements (related to peak/background ratio, for spectroscopy); see Figure 2.

Electrically-conducting specimens suffer knock-on displacement damage at higher accelerating voltages, with a characteristic dose D_e of typically10^8 e/nm^2, giving DLR ~ (10^{-3}nm).F which permits atomic resolution if the imaging mode is efficient or the analysis signal is sufficiently strong. However, atomic displacement at surfaces or grain boundaries is frequently observed and the solution may be to use a lower accelerating voltage, subject to limitations 1 and 2 above.

Poorly conducting specimens damage by radiolysis, which is often much more efficient than knock-on displacement [5]. For a typical organic material, $D_e \sim 10^3$ e/nm^2 and DLR can be several nm for bright-field scattering-contrast TEM imaging ($F > 0.1$ but low C) or HAADF STEM imaging (large C but low F). Phase–contrast imaging represents an attempt to achieve high C and F ~ 1 (in CTEM imaging) but is inefficient (F<<1) in STEM. Reducing the specimen temperature from 300 K to 100 K reduces D_e by a factor of typically 4 to 40, giving a 2 to 6-fold improvement in spatial resolution.

For energy-loss spectroscopy, $F = t/\lambda_i = n_a t \sigma_i$, where λ_i and σ_i are a mean free path and scattering cross section for the inelastic process involved, n_a is the appropriate atomic density and t is the specimen

https://doi.org/10.1017/S1431927616002361 Published online by Cambridge University Press

thickness. For x-ray emission spectroscopy, $F = n_a t\, \sigma_i\, \eta$ where η is the detector collection efficiency and ω is a x-ray fluorescence yield. Typically the spatial resolution is limited by photon statistics for a thin specimen and by beam spreading in a thicker specimen.

The DLR equation above applies to the situation where some feature or chemical element is present in a localised area that differs from its surroundings. But usually such a feature is repeated in many places and its spatial distribution is of interest. Principal-component analysis (PCA) of spectrum-image data can then be used to reject noise components and mitigate the effect of radiation damage. Compressive sensing might serve a similar purpose. Since radiolysis is hard to control, a strategy for obtaining high spatial resolution should include a suitable choice of imaging technique [5].

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[5] The author thanks the Natural Sciences and Engineering Research Council of Canada for funding.

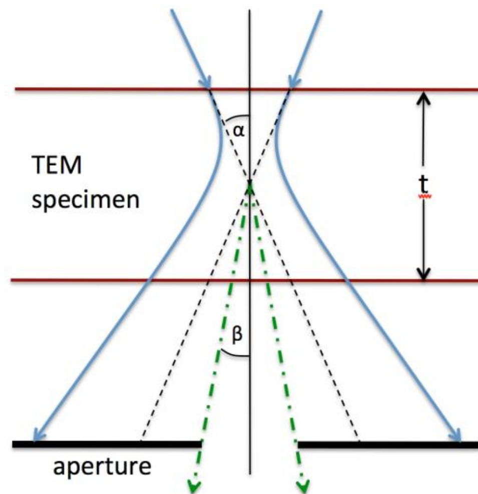

Figure 1. Volume of specimen giving rise to x-ray emission (outlined by blue curve) and the energy-loss signal (green dash-dot cone) for scattering at the mid-plane of a TEM specimen.

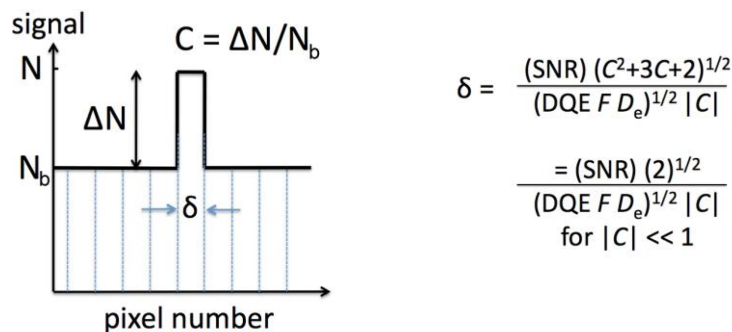

Figure 2. Weber contrast *C* and dose-limited resolution δ for a TEM image or elemental map.

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