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USE OF THE DISK-OF-LEAST-CONFUSION IN X-RAY MICROANALYSIS

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Whereas the spatial resolution for standard secondary electron (SE_i) imaging in a scanning electron microscope or electron probe microanalyzer is related to the incident probe diameter, the spatial resolution for x-ray microanalysis is related to the convolution of the probe diameter with the spatial extent of the analyzed volume for a point probe. The latter is determined by electron scattering in the specimen and the subsequent emission of excited x-rays from the specimen. As such, it is possible that "What you see is not what you get". This is especially true for instruments with high brightness electron sources (field emission). This problem is compounded by probe aberrations which at Gaussian image focus can produce significant electron tails extending tens of microns from the center of the probe.

If the main source of probe aberration is spherical aberration, the probe diameters at Gaussian focus and at the disk-of-least-confusion are given as $2 \cdot C_s \cdot \alpha^3$ and $0.5 \cdot C_s \cdot \alpha^3$, respectively (where C_s is the spherical aberration coefficient and α is the beam convergence half-angle). The optimum defocus¹ to achieve the disk-of-least-confusion is $-0.75 \cdot C_s \cdot \alpha^2$. Aperture sizes for x-ray microanalysis should be chosen such that the probe diameter at Gaussian focus is less than the spatial extent of the analyzed volume for a point probe. However, if the disk-of-least-confusion is used for analysis, a larger aperture ($\alpha_2/\alpha_1 \sim 1.6$) may be used with an attendant increase in probe current (possibly a 2.5-fold increase).

A Philips XL30/FEG scanning electron microscope equipped with both energy dispersive and wavelength dispersive spectrometers (EDS and WDS) was used in this study. A cleaved GaAs test specimen was used to measure the current distribution in electron probes at 5 and 20 kV as a function of aperture size and objective lens defocus. The generated Ga K_α x-ray intensity as measured by WDS was used to indicate the integrated current incident on the specimen as a function of distance relative to the specimen edge. Probe diameters can be estimated from profiles of the generated x-rays in the same manner that such diameters can be estimated from the integrated current profiles. Unfortunately, the x-ray intensity is not always proportional to the incident current. When an electron is incident on the GaAs near the cleaved edge, the x-ray yield is reduced when the electron escapes from the edge. Monte Carlo simulations for GaAs at 20 kV indicate that the normalized yield drops from 1 at $\sim 5 \mu\text{m}$ from the edge to ~ 0.2 near the edge. Experimental measurements show a similar decrease in normalized x-ray yield near the specimen edge. Both Monte Carlo simulations and measurements show that the magnitude of this effect is reduced at lower accelerating voltage as the spatial extent of the analyzed volume is decreased. X-ray intensities are normalized to that measured on the specimen at $10 \mu\text{m}$ from the cleaved edge.

The normalized profile of x-ray count rate for Gaussian focus at 20 kV is plotted in Figure 1 for a range of aperture sizes. Although probe currents $>500 \text{ nA}$ were measured for apertures of $600 \mu\text{m}$ diameter or greater, spherical aberration resulted in significant current at $>10 \mu\text{m}$ from the probe center. Probe diameters containing 90% of the current were estimated as $<0.2, 0.4, 4.4, 9.6$ and $18.8 \mu\text{m}$ for apertures 200, 300, 400, 600, and $1700 \mu\text{m}$ in diameter. Though large probe currents are available with the larger apertures (especially useful for WDS analysis), the spatial resolution would be unacceptable for many applications. Probe aberrations of similar magnitude were observed at 5 kV.

Defocussing the probe to the disk-of-least-confusion can provide a smaller incident probe with the same total current. Normalized profiles of x-ray count rate at 20 kV with a $400 \mu\text{m}$ diameter aperture are plotted in Figure 2 for a range of defocus. The diameters containing 90% of the probe current dropped from $\sim 4.4 \mu\text{m}$ at Gaussian focus to $\sim 2 \mu\text{m}$ at $-60 \mu\text{m}$ defocus. Further defocus to -70 and $-80 \mu\text{m}$ resulted in monotonic increase of the probe diameter. The factor of >2 improvement in probe diameter is slightly less than would be predicted from the simple equations given above. Other sources of probe aberration will be examined as possible origins for this discrepancy.

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If C_s and beam convergence are known, probe diameters and optimum defocus can be estimated. If C_s is unknown, the optimum defocus can be approximated in some cases by focussing for sharpest contrast change between two adjacent regions of significantly different contrast. Significant electron tails present at Gaussian focus will blur the transition from black to white; whereas the disk-of-least-confusion will give the sharpest transition. One problem with using the disk-of-least-confusion probe is that the spatial resolution of the SEM image is poor ($\sim 2 \mu\text{m}$ for a $400 \mu\text{m}$ aperture). However, that SEM image resolution better approximates the spatial resolution relevant for analysis than that of the Gaussian focus SEM image. Operationally, the Gaussian focus condition can be used for microstructural examination and positioning of the probe, whereas the disk-of-least-confusion condition can be used for analysis.²

REFERENCES

¹C. Van Essom et.al., J. Mater. Sci. 6 (1971) 213.

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FIG. 1 Normalized Ga K_{α} x-ray count rate versus distance of probe relative to cleaved edge of GaAs specimen for different aperture sizes. Experimental conditions: 20 kV, spot 6, Gaussian focus.

FIG. 2 Normalized Ga K_{α} x-ray count rate versus distance of probe relative to cleaved edge of GaAs specimen for differing defocus. Experimental conditions: 20 kV, 400 μm aperture, spot 6.

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