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MICROANALYSIS BY ELECTRON ENERGY LOSS SPECTROSCOPY AT 300kV*

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Some features of the anticipated improvements in microanalysis by electron energy loss spectroscopy (EELS) at 300kV compared to operation at 100kV have been studied with the use of a Philips EM430T equipped with Gatan 607 EELS and EDAX 9100/70 systems. Our main interest in operation at higher accelerating voltage is in the expected increase in the usable specimen thickness which is particularly important for elemental analysis of structural materials. With increased accelerating voltage, multiple scattering should be reduced, collection efficiency should be increased as a result of smaller scattering angles, and decreases in ionization cross sections should be more than offset by increases in gun brightness.

Detailed measurements have been made on a range of specimens, with emphasis on boron nitride and silicon carbide and with particular care being taken in the choice of optimum collection angles (β) and illumination angles (α). There is some conflict in this area. The illumination angles are almost independent of voltage since they are largely controlled by the geometry of the illumination system (aperture sizes and lens focal lengths), but the Bragg angles and characteristic scattering angles at 300kV are about half of the 100kV values. This nonequivalence is an added complication for comparisons and quantification when $\alpha > \beta$.

As expected, for thin specimens little difference in peak-to-background (P/B) ratios was observed between 100 and 300kV (see Figs. 1 and 2). Also, a significant increase (>2 times) in the usable specimen thickness at higher accelerating voltage was confirmed. The precise dependence of P/B ratio on specimen thickness and accelerating voltage was found to be a function of ionization edge and material composition. For example, in boron nitride the nitrogen P/B ratio decreases slowly with increasing thickness, since the background is mainly due to the tails of the boron edge, whereas the boron P/B ratio decreases rapidly with increasing thickness. The carbon P/B ratio for specimens of silicon carbide was similarly almost constant with increasing specimen thickness since the background is mainly from the silicon L edge. Much of the SiC data was more complex, possibly as a result of elastic scattering effects from large d-spacings. Values for the absolute partial ionization cross sections determined for SiC ranged between 0.9 and 3 times the calculated cross sections. For these measurements the contributions of surface films were mitigated by a novel method that combines X-ray microanalysis, convergent beam electron diffraction and EELS measurements.

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DISCLAIMER

During this work it became apparent that the EELS instrumentation was not of optimal design, especially for 300kV operation. There is a severe hole count (see Figs. 3 and 4) which causes difficulties in thickness measurement from the low loss region and results in poorer P/B ratios than expected. Surprisingly, the edges most affected are at large losses at both 100 and 300kV operation.

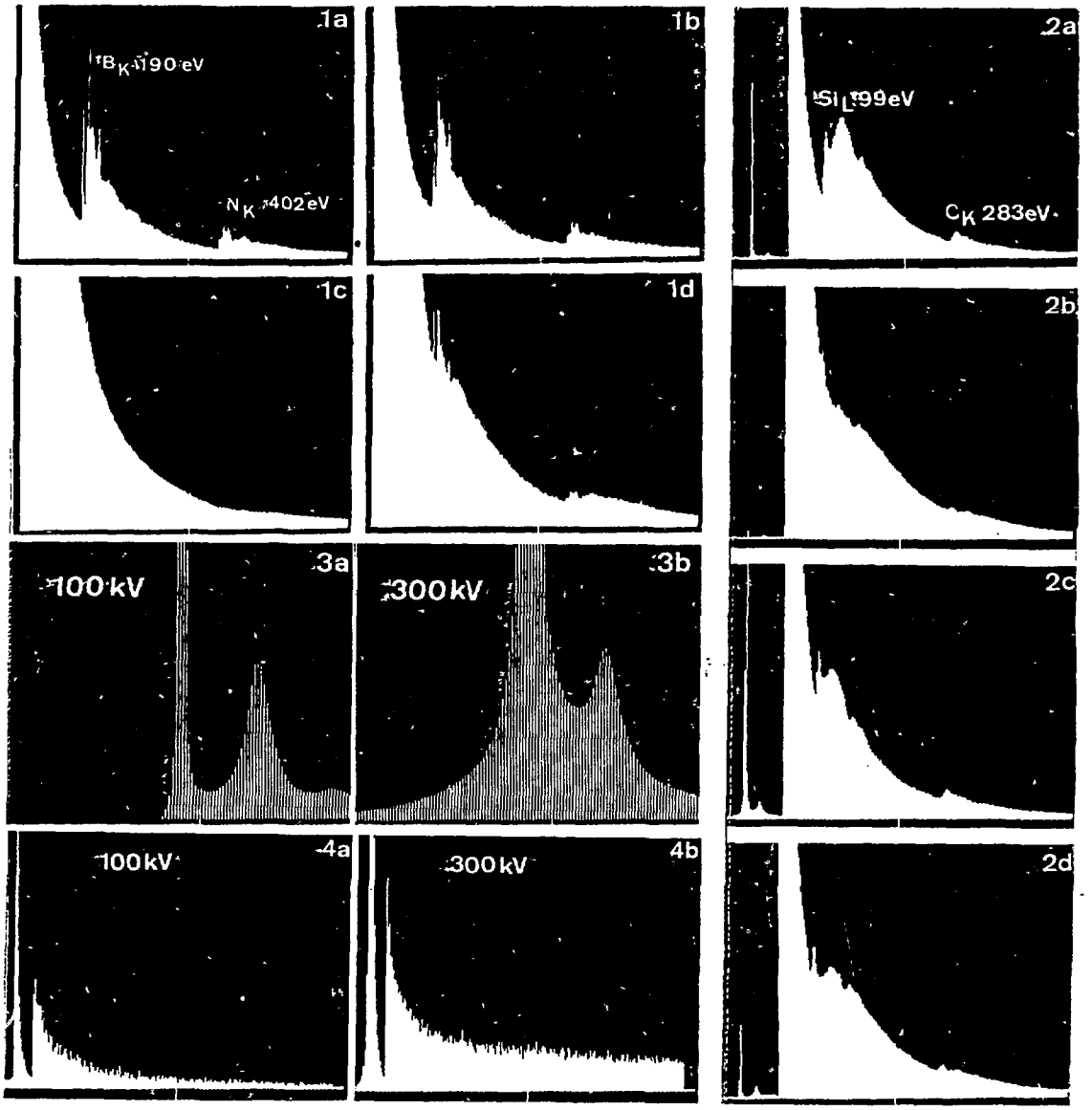

Fig. 1. Energy loss spectra of BN at two thicknesses t. (a) 100kV, t/λ_p ~ 0.1, (b) 300kV, t/λ_p ~ 0.1, (c) 100kV, t/λ_p ~ 0.7, (d) 300kV, t/λ_p ~ 0.7, where λ_p is the plasmon mean-free-path length at 300kV. β = 12mrad (100kV) and 4mrad (300kV). Fig. 2. Energy loss spectra of SiC, β = 10mrad. (a) 100kV, t = 24nm, (b) 100kV, t = 165nm, (c) 300kV, t = 34nm, (d) 300kV, t = 165nm. Fig. 3. Low loss region from 40nm-thick B_4C showing symmetric tails. Fig. 4. Hole-count spectra, 0-3kV, 1nA probe current, zero loss = 128 times full scale.