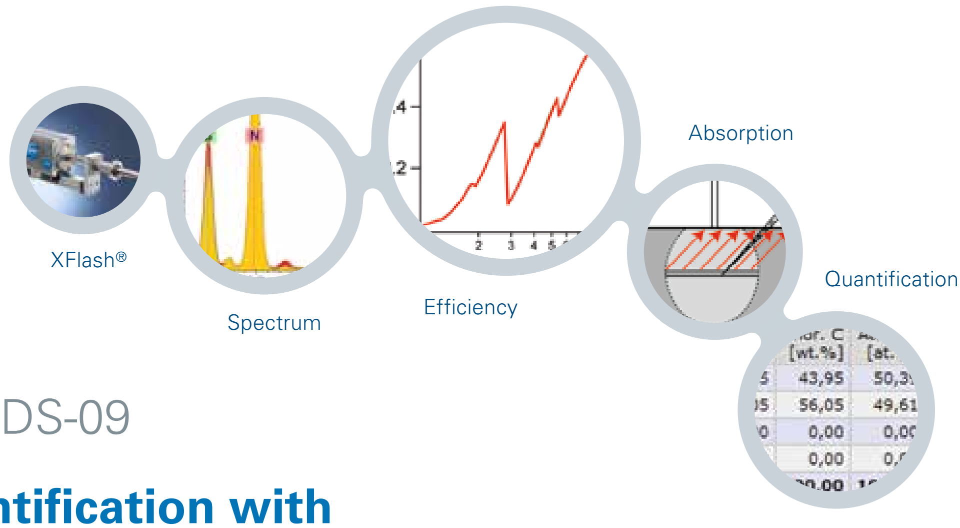

Application Note # EDS-09

Light element quantification with TQuant: Analyzing boron nitride (BN) at different acceleration voltages

Boron nitride (BN) with an element ratio of 50 : 50 atomic% displays 2 peaks, the B-K peak at 183 eV and the N-K peak at 392 eV. Both are in the very low energy range, which means that a range of factors influencing peak intensities have to be considered. As will be shown, this makes it futile to believe that the B-K and the N-K peak have the same intensity (height), just because they have the same atomic concentration of 50%. First of all, Castaing's "first approximation to quantitative analysis" applies to the weight concentration and not the atomic concentration. Secondly, and here more important, this approximation only applies when absorption and efficiency can be neglected, e.g. for a Fe-Ni system where K peaks with an energy of ~ 6 keV are used.

One important factor affecting the visible peak height is the efficiency of the detector. Efficiency is defined as the rate of X-rays detected versus the rate of incoming X-rays. Efficiency in the light element / low energy range below 1 keV is typically in the order of 25% to 50% (see Fig. 1). It is almost independent of whether a silicon drift detector (XFlash®) or a Si(Li) is used. Influences playing a role in the low energy range are the detector window and the reduced efficiency of the detector chip itself (Si dead layer, contact layer, etc.)

The second important factor affecting peak height is absorption of generated radiation within the sample already. Depending on the density of the sample, the energy of the radiation and the detector take off angle more or less of the generated radiation will be able to leave the sample for detection (see Fig. 2). In this case around 90% will be absorbed in the sample and only 10% can be detected.

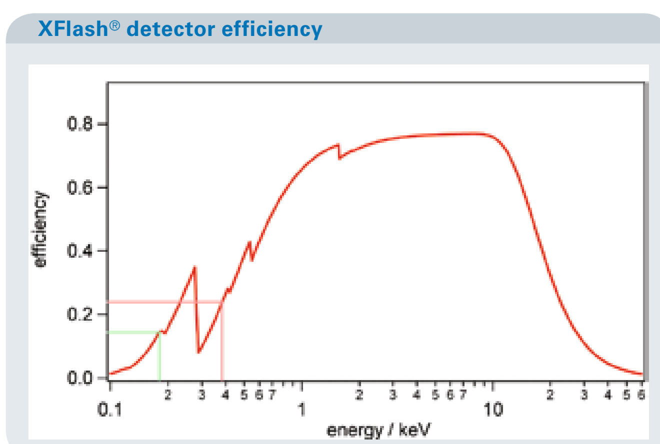

Fig 1 Efficiency curve for a detector with light element window. Highlighted are the efficiency for B (green), which is around 15%, and N (pink), around 25%.

Sample excitation and X-ray detection conditions

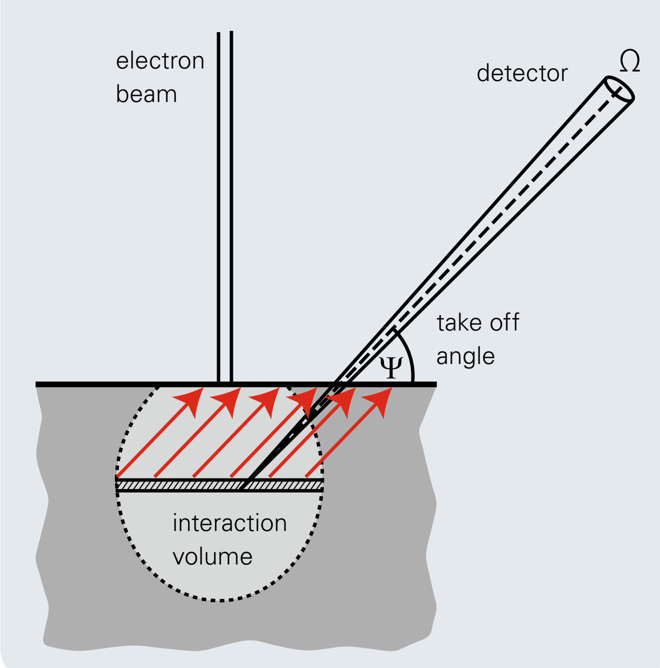

Fig. 2 The electron beam hitting the sample causes the production of X-rays within the interaction volume. Only those produced in the upper region have the chance to leave the sample (indicated by red arrows). The detector records a fraction of these X-ray under a take-off angle of Ψ and a solid angle of Ω.

A third factor affecting the appearance of a spectrum is the overvoltage, used for spectrum acquisition. Overvoltage is defined as the ratio of acceleration energy (for electrons: acceleration voltage in eV) versus peak energy. Ideally the overvoltage should be 2.5 to 3. Fig. 3 a, b shows how the overvoltage influences the peak heights in BN-spectra obtained at different acceleration voltages.

Quantification results of BN spectra

Acceleration voltage / kV	Boron / at.%	Nitrogen / at%
3	50.7	49.3
5	50.4	49.6
10	50.5	49.5
20	51.2	48.8

Normalized BN spectra

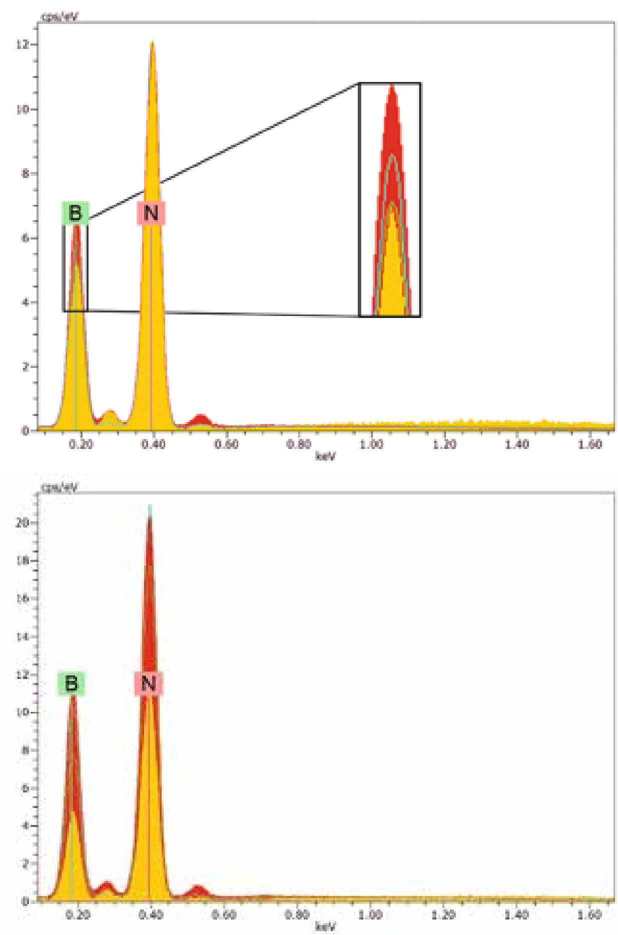

Fig. 3 Normalized spectra, red obtained at 3 kV, light blue outline at 5 kV, green outline at 10 kV, yellow at 20 kV. a) (Top) normalization with respect to the N peak makes changes in peak height ratios visible, especially visible at low acceleration voltages. b) (Below) normalization with respect to zero peak roughly indicates overall change of peak height with acceleration voltage.

This means that the only reliable method to judge the elemental content of BN and other light element spectra is to use a quantification routine that considers all factors and provides reliable results at low peak energies – TQuant. The table on the left contains results for the 4 spectra in Fig. 3.

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