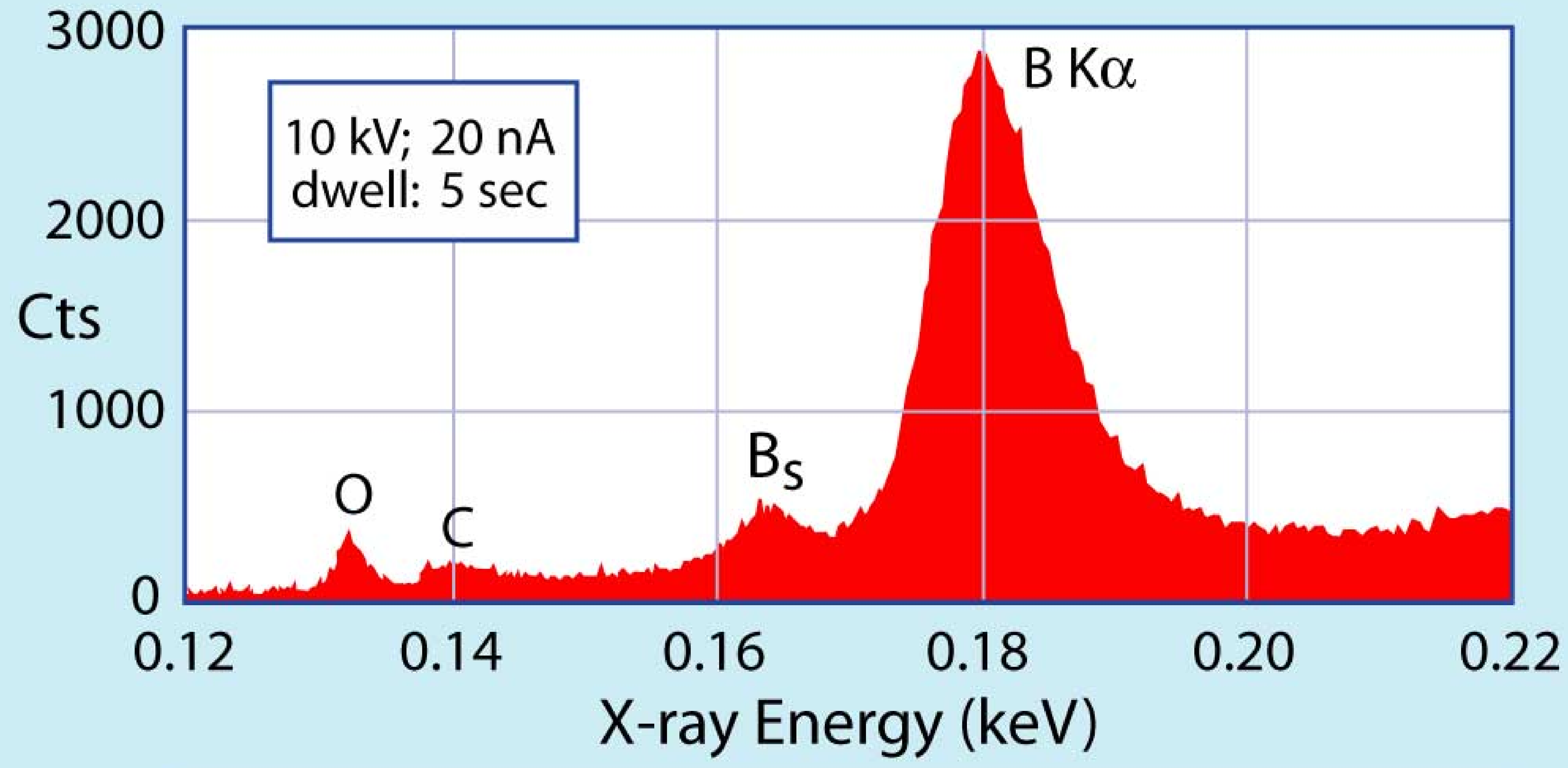
McSwiggen & Associates

TECH Notes

Boron

Boron (B, Z=5) is one of the more difficult elements to analyze. It has a very low count rate and it experiences peak shifts and shape changes from one material or standard to another. The low energy nature of the boron X-ray makes it easily absorbed by the sample and the detector. This produces the low count rate, and because the production of the boron X-ray involves a valance electron, its peak can shift considerably with any change in the nature of the atomic bonding. Therefore there can be peak shifts or shape changes between the standard and the material being analyzed.

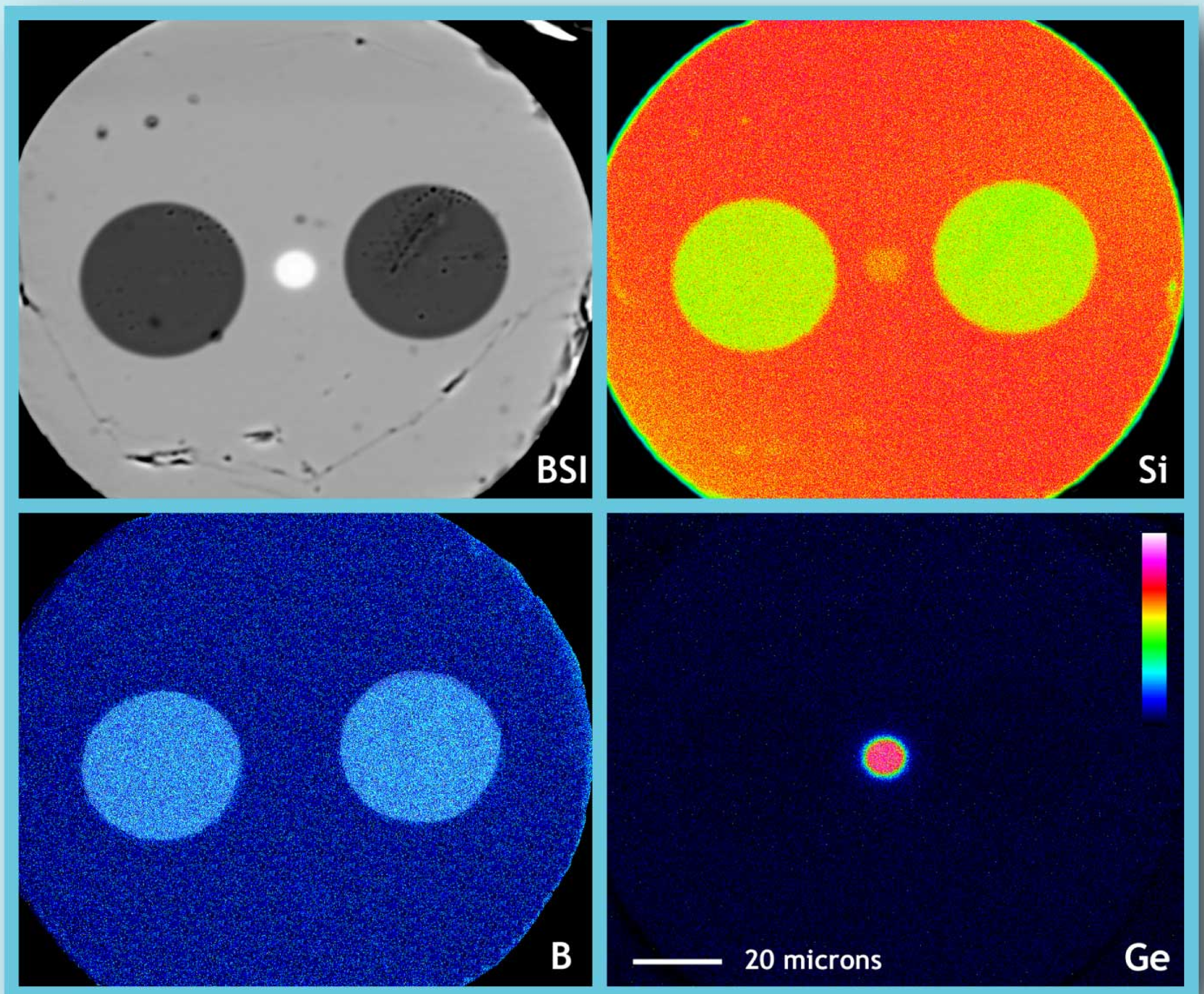
If the goal is to produce quality elemental X-ray maps and precise quantitative analyses, it is important to use wavelength dispersive spectrometers (WDS). WDS systems can produce X-ray count rates that are at least 10x higher than those from most energy dispersive spectrometers (EDS), and they have an energy resolution that is 10x smaller. Therefore WDS can produce X-ray maps of excellent quality in a fraction of the time, and can easily resolve the boron peak from nearby neighboring peaks even when there are only a few tens of eV between them.



Wavelength dispersive spectrometer (WDS) scan showing the main boron peak at 0.18 keV. Due to the high spectral resolution of these spectrometers, the boron can easily be distinquised from other nearby peaks.

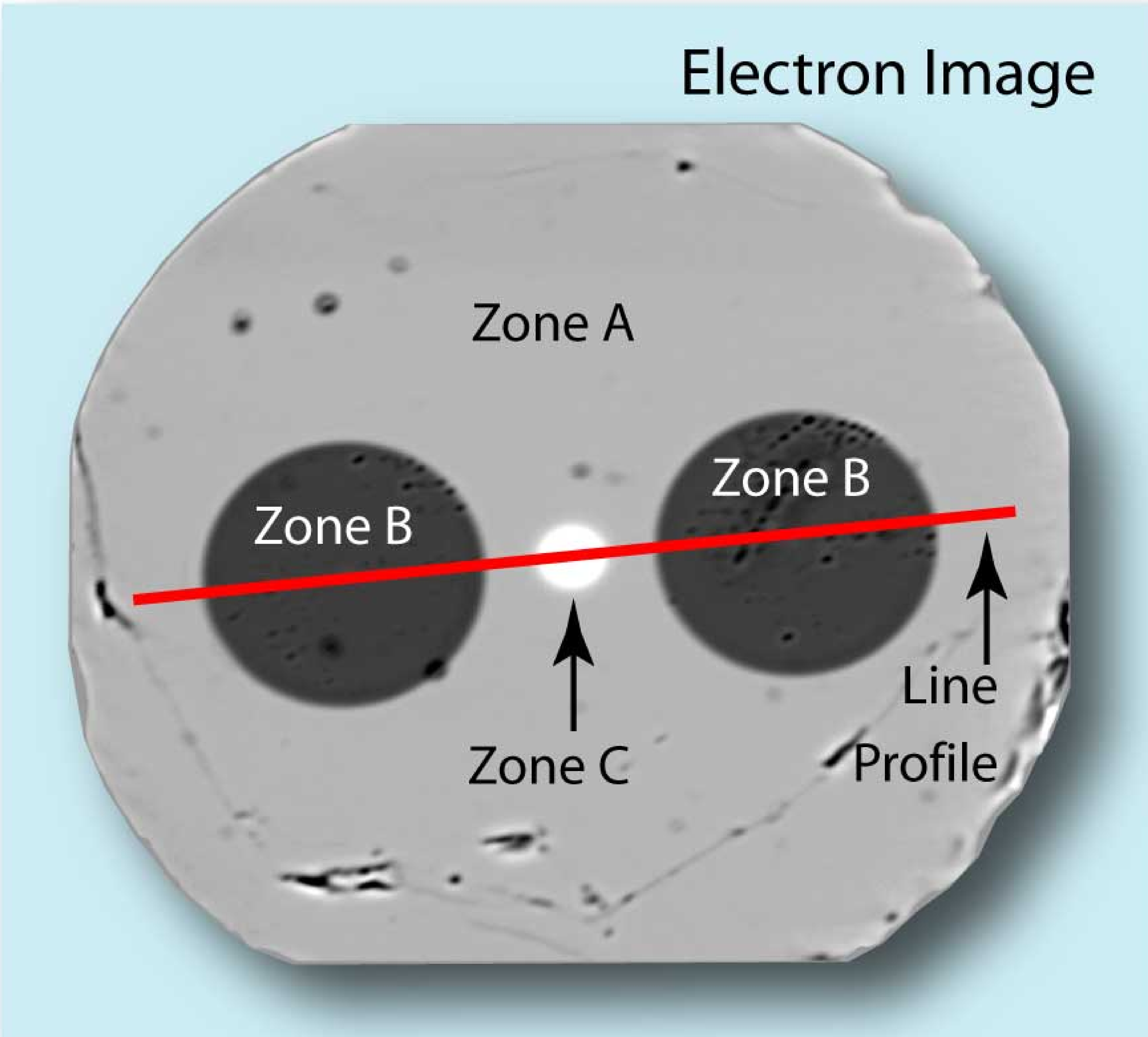
There are a number of other problems that can be associated with analyzing boron-rich materials. One is that boron can be very hydroscopic. Care must be taken during sample preparation to avoid contact with water or even moisture.

Optical Fiber



Element Maps

Backscattered electron image (BSI) and element maps for silicon (Si), boron (B), and germanium (Ge) of an optical fiber's end-face. The maps show that the core of the fiber is high in germanium and that there are two zones on each side of the core high in boron. These boron-rich zones cause the light passing through the core to maintain its polarization. X-ray maps such as these clearly show the distribution of each element and the nature of each zone. Quantitative analyses would then be required to determine how much of each element is present.

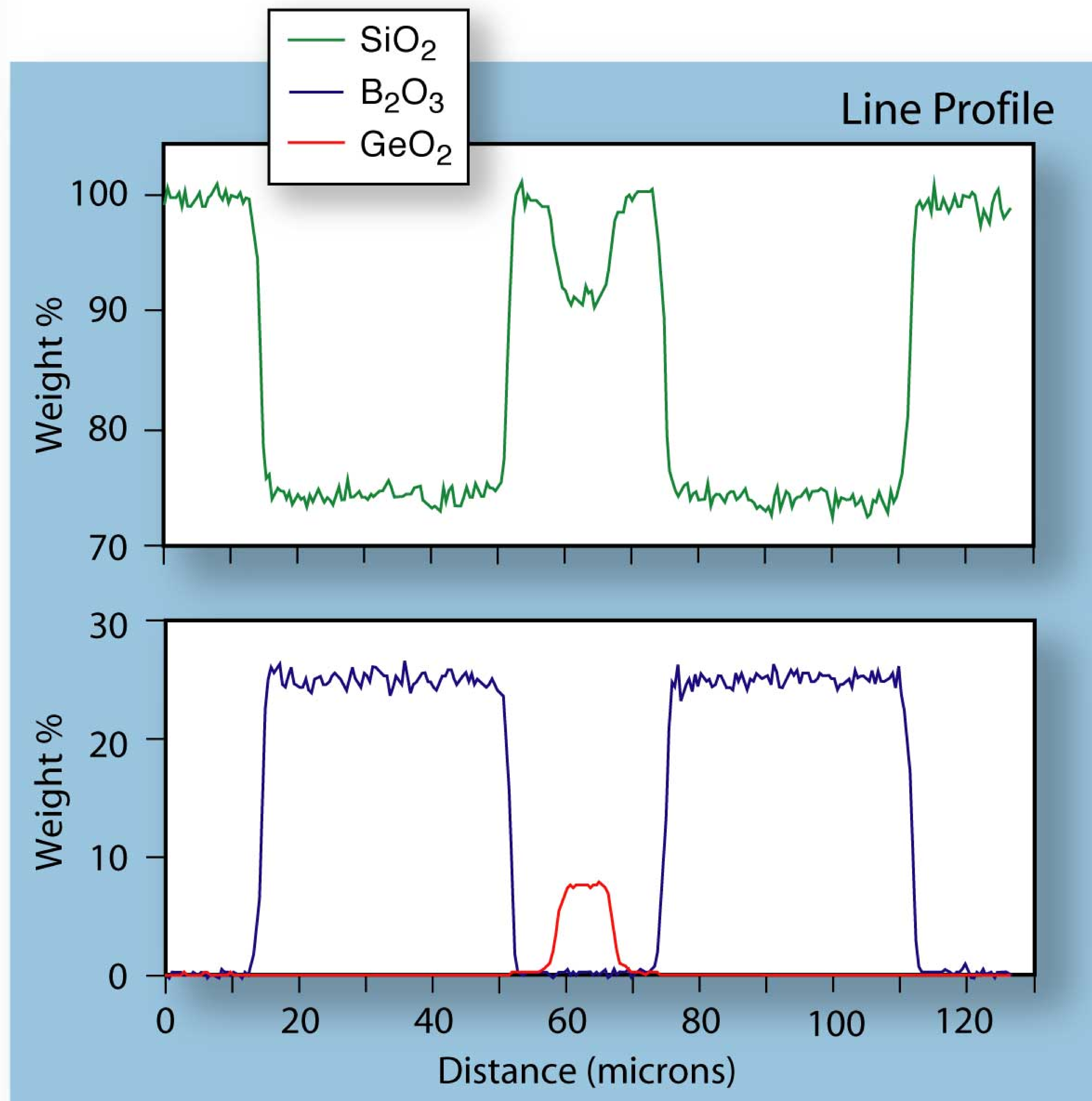


Boron-rich samples are also very susceptible to electron beam damage. To avoid this problem a low beam current should be used during the analyses.

In order to improve the production of boron X-rays, a low accelerating voltage should be used, when possible 10 kV. Higher accelerating voltages will produce a higher number of X-rays, but they will be produced deeper in the sample. Therefore they are absorbed by the sample before they can make there way to the surface. This lower accelerating voltage will also reduce the amount of beam damage during the analyses.

Above is the backscattered electron image of the optical fiber's end-face. Labeled are the three zones of interest, the core (Zone C), the boron-rich zones (Zone B), and the matrix glass (Zone A). The superimposed red line is the location of the line profile. The results of the line analyses are shown to the right. Line profiles have the advantage of showing the element distribution in the sample, but at a fraction of the time that it takes to produce a map. However, line profiles are typically semi-quantitative in nature because they tend not to be matrix-corrected.

For true quantitative analyses, it is essential that good standards be used, and that the appropriate corrections are made for the matrix effects. Standardless analyses should never be relied upon. It is also important that the results from the quantitative analyses not be normalized. Normalization typically hides errors that are contained within the data.



Quantitative Analyses

	Zone A			Zone B			Zone C		
	weight percent								
SiO_2	99.75	99.95	99.74	75.30	75.24	75.69	92.12	92.65	93.00
GeO_2	0.00	0.00	0.00	0.00	0.00	0.00	7.27	7.52	7.41
B_2O_3	0.00	0.06	0.00	23.85	23.77	24.07	0.00	0.18	0.00
Cl	0.19	0.15	0.12	0.17	0.16	0.18	0.08	0.09	0.09
Total	99.89	100.16	99.83	99.27	99.13	99.90	99.45	100.42	100.48