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ULTRATHIN WINDOW DETECTOR AND DIGITAL PULSE PROCESSOR

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Borosilicate glass has been selected for immobilizing radioactive wastes because of its chemical durability and ability to incorporate many different elements.¹ Boron is an important component in the borosilicate waste glass: its addition facilitates the processibility of glass by lowering its viscosity. In addition, the release of boron is used to measure the corrosion progress during laboratory tests that determine the durability of a glass. Analysis of boron that is present in waste glasses and in the reaction products that form during the reaction of glass is important for understanding the reaction kinetics and mechanism of glass corrosion.

Waste glasses have been previously analyzed for B in our laboratory by wavelength-dispersive spectrometry (WDS) in SEM. As can be deduced from Fig. 1, WDS analysis of waste glasses with B concentrations up to 4 wt% yields a line with a slope of 0.4 counts/s/wt%, with a resolution of ~0.5 wt%. However, EDS is analytically more convenient and advantageous because its high detection efficiency and its ability to simultaneously acquire an entire spectrum. The innovation of the ultra-thin window has made it possible for an EDS to detect soft x-rays, which are absorbed by a normal Be window. Unfortunately, the detection sensitivity is still poor for elements lighter than C; boron is only detectable when analyzing B-rich (>20 wt%) samples, such as BN, with a traditional analog x-ray pulse processor (APP) (see Fig. 2). The technique cannot be readily employed to analyze B in waste glasses which have low B contents (typically <5 wt%). However, the sensitivity can be greatly improved by using the recently introduced digital pulse processor (DPP) (see Fig. 2), with which the preamplifier output is digitized and the resulting data stream is digitally processed.² In this paper, we report the detection of B in borosilicate waste glasses with a Noran Voyager III EDS system equipped with a DPP, which is interfaced with a Topcon ABT 60 SEM operating at 5 kV.

Two waste glasses selected for analysis include glass LD6-5412, developed by Pacific Northwest National Laboratory (PNNL) and glass EA, developed by Savannah River Technology Center (SRTC). These glasses contain 1.55 wt% and 3.47 wt% B, respectively. A blank test has also been carried out on a quartz sample containing no B to rule out possible artifacts. For waste glasses, the B peaks are clearly distinguishable in the EDS spectra acquired at 5 kV for 100 s (see Fig. 3a). To our knowledge, this probably is the lowest B concentration (1.55 wt%) detected with an EDS that has been reported. It should be noted, however, that the B peaks severely overlap with the C peaks, which are due to the carbon film coated on the sample surface to produce a conductive layer. The presence of the C peak could limit the detection of lower B concentrations, as evidenced in the spectrum for 1.55 wt% B where the B peak is only barely visible (Fig. 3a). This problem can be solved by subtracting the C peaks as shown in Fig. 3b, which still has a good peak-to-noise ratio for 1.55 wt % B. This result suggests that even lower B content could be detected by EDS with DPP if the C peaks are subtracted as done in Fig. 3b. By plotting the peak intensity against the weight percent of boron in the glass, we also found a nearly linear relationship, similar to that observed with WDS (see Fig. 1).³ Calibration of this curve will allow us to quantify the B contents in waste glasses.

References

1. J.C. Cunnane et al., DOE/EM-0177 (1994).
2. J.J. Friel and R.B. Mott, *Adv. Mater. & Proc.* 145(1994)35.
3. This research was sponsored by the U. S. Department of Energy, Office of Environmental Management, under Contract W-31-109-ENG-38.

FIG.1. Beer-Lambert plot showing number counts vs. B wt% in glass analyzed by WDS.

FIG.2. EDS spectra of BN comparing DPP vs. APP processing.

FIG.3. EDS spectra of BN and two waste glasses with less than 5 wt% B: (a) as-acquired spectra and (b) spectra with carbon peaks subtracted. The result demonstrates a detectability of less than 1.5 wt% B if C peaks can be properly subtracted.