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Quantitative X-ray Microanalysis of Bare Insulating Materials

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Charging of non-conducting specimens can cause serious problems for quantitative x-ray microanalysis in electron beam instruments. Negative surface charging can reduce the landing energy of the probe electrons to such a degree that the resulting quantitative analysis is inaccurate by as much 20% relative to a known standard. The traditional fix for this problem is to destructively coat the specimen surface with a grounded conductive thin film. However, the charging problem can be eliminated without coating by precise neutralization of the negative surface charge in a variable-pressure SEM (VP-SEM) or in an environmental SEM (ESEM). Precise neutralization [1] can be combined with pressure-extrapolation corrections [2,3] that mitigate the loss of x-ray spatial resolution in variable-pressure SEMs.

Precise neutralization involves creating an appropriate number of positive ions in the chamber gas of the microscope. To ionize the correct number of chamber gas molecules for precise neutralization, a variable positive potential is placed on an electrode above the specimen that can create an avalanche of electrons and ions. Electrons are attracted to the positive electrode, while the positive ions drift to the specimen surface to neutralize the negative surface charge. To create a positive ion flux to just neutralize the surface, the specimen surface potential must be sensed by a wire above the specimen that is connected to ground through a sensitive ammeter. The potential on the electrode above the specimen is adjusted until the flow of current in the ammeter is zero [1].

To test this analysis method on an insulating material of known composition, particles of K-411 glass (NIST SRM 2066) [4] were pressed into a lead layer on an SEM specimen stub (Fig. 1). Pressure-extrapolation correction was accomplished by admitting 1.5 to 3 torr of water vapor into the 2-mm gap between the specimen and the pressure-limiting aperture of an FEI XL-30 FEG ESEM. X-rays generated with a 20-keV beam were detected with an EDAX x-ray spectrometer (Fig. 2). The precise charge neutralization method was used to eliminate surface charging for the x-rays collected at each pressure step. For the ZAF matrix-correction procedure, the x-ray count for each element was extrapolated to zero pressure (Fig. 3). Six particles, from 7 μm to 70 μm, were analyzed in this manner. Regardless of the pressure extrapolation method used, that of Doehne or of Gauvin, the concentration of each element was within the error bars of the known NIST-measured composition for SRM 2066 glass spheres (Table 1) [5].

References
[1] R. A. Carlton et al., *Microsc. Microanal.* 10 (2004) 753.
[2] E. Doehne, *Scanning* 19 (1997) 75.
[3] R. Gauvin, *Scanning* 21 (1999) 388.
[4] R. Marinenko et al., *Microsc. Microanal.* 6 (2000) 542.
[5] The author acknowledges support for Robert Carlton from Rhone-Poulenc-Rorer. Instrumental support was provided by Ralph Knowles (FEI) and Robert Anderhalt (EDAX).

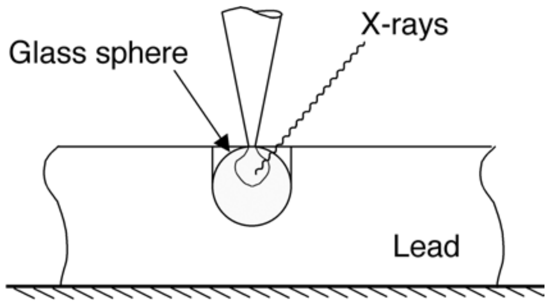

Fig. 1. Glass sphere pressed in lead.

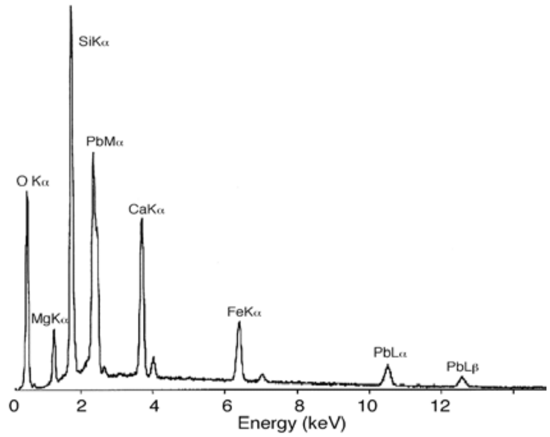

Fig. 2. X-ray spectrum from 22-µm particle

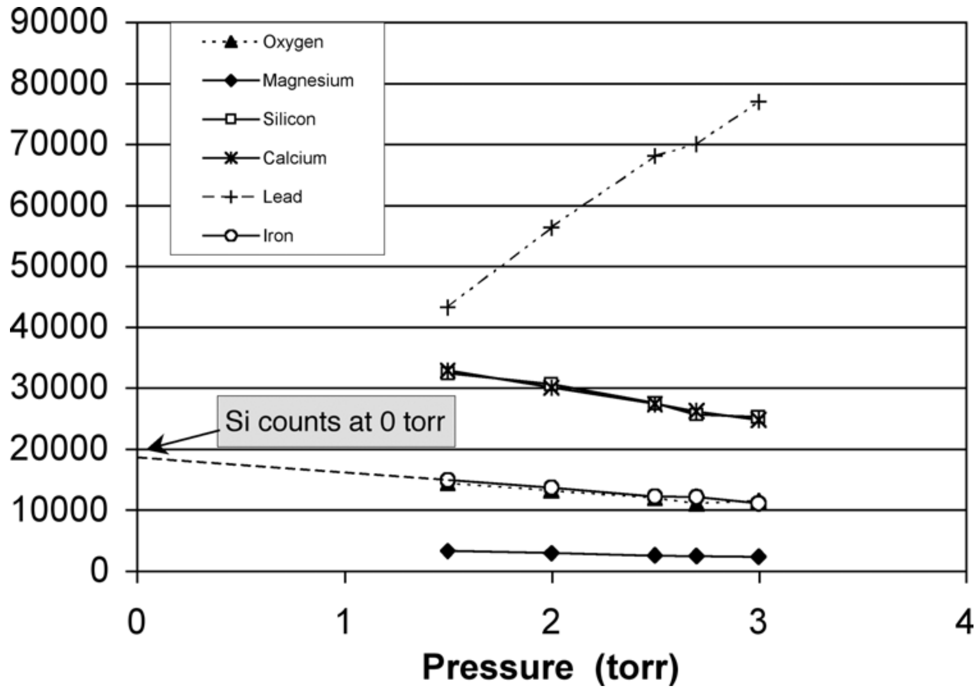

Fig. 3. Extrapolation of x-ray counts to zero pressure (40-µm glass sphere in lead).

Table 1. Results from Two Pressure-Correction Methods using Charge Neutralization
Mean Composition of measurements on SRM 2066 in wt % (Standard Deviation %)

	Mg	Si	Ca	Fe	O (diff.)
Doehne	9.0 (0.5)	24.5 (2.2)	9.9 (0.4)	10.0 (0.5)	46.6 (2.7)
Gauvin Linear Regression	9.1 (0.6)	24.7 (2.3)	10.0 (0.4)	10.1 (0.5)	46.1 (2.8)
NIST Sphere Values	9.15 ± 1.4	25.65 ± 1.7	11.15 ± 2.4	11.2 ± 2.2	42.9 ± 1.2
NIST Bulk	*8.85*	*25.38*	*11.06*	*11.21*	*42.36*

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