Quantitative X-ray Microanalysis of Particles at Low Beam Energy

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The results of a previous study indicate that reducing the accelerating voltage of the electron beam for quantitative particle analysis significantly reduces the magnitude of the particle mass and absorption effects.[1] By reducing the beam energy to 10 keV it was possible to quantitatively analyze irregularly shaped shards of K-411 glass (Table 1) using bulk standards and a conventional ZAF correction procedure without normalization or corrections for particle effects. As shown in Fig 1, at 10 keV the spread of RD values[*] is from +0.08 to –0.13 with a standard deviation of 0.054. This compares to a spread of RD values for 25 kV, from +0.26 to –0.60 with a standard deviation of 0.238.

In the current study, shards of analytical glass K-3189 (Table 1) were analyzed at 5 keV and 10 keV in an analytical scanning Auger microscope, ASAM, which has a UHV chamber at 2×10^{-8} Pa. To avoid contamination during the analysis, the samples were prepared using the following procedure: 1) The bulk glass was dry ground to produce shards in the 1 micrometer to 10 micrometer size range. 2) The shards were dispersed in ethanol and deposited onto a polished C substrate. 3) The preparation was baked in a vacuum oven at 100° C for 30 min. 4) The sample was removed from the oven immediately carbon coated and placed into the instrument.

Fig 2 shows the ratios of the Fe L x-ray intensities from two glass shards with similar sizes and shapes. The analytical area, indicated by the small square, for the top shard is positioned on the downward slope of a fracture surface facing away from the detector. The arrow in the upper left of the image indicates the detector direction. The analysis area for the second shard, although also on the downward slope of a fracture surface, is positioned nearer the top edge of the fracture. Comparing the particle-to-bulk ratios for the Fe L x-ray intensities indicates that the small difference in position of the analysis area relative to the particle surface and x-ray detector direction produces a very large difference in the two ratios. The variations are due to differences in the x-ray absorption between the two particles as a result of the different particle morphologies. Rather than reducing the effects of particle morphology, lowering the beam energy to 5 keV and utilizing the Fe L x-ray lines instead of the Fe Kα x-ray line, has actually resulted in a much larger particle absorption effect. The effect is large enough that for this glass composition, quantitative analysis would have a higher accuracy performed at 10 keV and utilizing the Fe Kα line.

References

[1] J.A. Small, J. of Res. of the Natl. Inst. of Stds. and Tech. 107 (2002) (in press).

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[*]The relative differences (RDs) between the particle values and the certified bulk values for all elements were calculated from the following equation: $RD = \left[\left(exp. - known \right) \middle/ known \right]$.

Table 1. Compositions of Analytical Glasses K-411 and K-3189

glass/element	O	Mg	Al	Si	Ca	Ti	Fe
K-411	0.4236	0.0885	-	0.2538	0.1106	-	0.1121
K-3189	0.427	0.060	0.074	0.187	0.10	0.012	0.14

Unnormalized Data
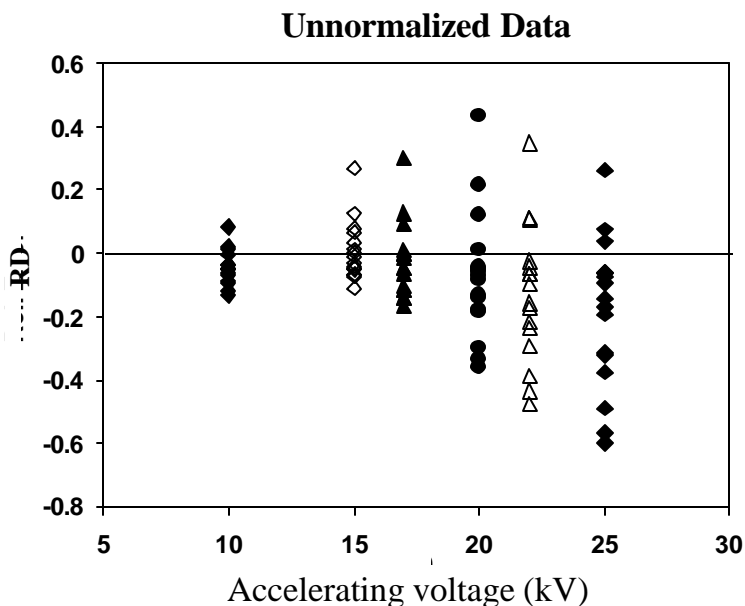
Fig 1. RD values for glass shards *vs* electron beam accelerating voltage.

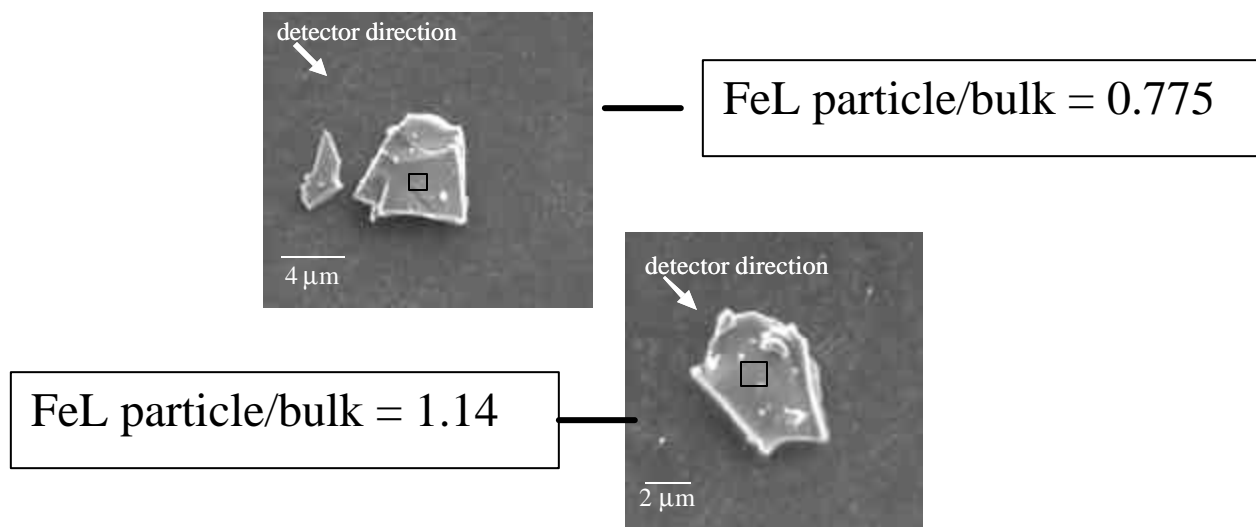FeL particle/bulk = 0.775

FeL particle/bulk = 1.14

Fig 2. Secondary electron images and Fe L particle-to-bulk x-ray intensity ratios for two shards of K-3189 glass similar in size and shape.