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K409: A potential reference material for sub-micron X-ray resolution by EPMA

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With advances in electron beam instrumentation, there has been a trend toward higher resolution electron gun sources for electron microprobes. JEOL has been marketing field-emission gun (FEG) microprobes since 2003 (8530F), and CAMECA introduced their SX5FE microprobe in 2011. However, there remain questions about the full utilization of such tight beams as those afforded by the FEG applied to common rock-forming minerals (e.g. silicates, oxides, carbonates, phosphates, glasses), because the desired improvement in X-ray spatial resolution for quantitative determination of the compositions of sub-micron size objects necessitates operation at lower accelerating voltages and use of low-energy X-ray lines. The physics of electron scatter and ionization energies under such conditions is of primary concern regarding the spatial resolution of field-emission EPMA.

In the 1970s, the U.S. National Bureau of Standards (now National Institute of Standards and Technology) developed a series of glass reference materials for microanalysis. The two glasses K411 and K412 were certified in 1982 (Marinenko, 1982), and both contain SiO2, MgO, CaO and FeO/Fe2O3, with K412 additionally containing Al2O3. Both glasses were independently characterized and each found to be homogeneous. The composition of K411 is equivalent to stoichiometric pyroxene (augite). Decades later, with interest in microanalysis of particles, microspheres (2-40 um) of K411 composition were developed (Marinenko et al., 2000). Recently, a vial of NBS glass "K409" was unearthed at the University of Wisconsin; it was apparently a "failed experiment" for a sodium-rich microanalysis standard (D. E. Newbury, personal communication) with a nominal composition of SiO2 (55 wt%), Al2O3 (15 wt%), FeO (20 wt%) and Na2O (10 wt%). Close inspection of this sample by SEM reveals a plenitude of equant euhedral iron oxide microlites ≤ 1000 nm in diameter. This "failed experiment" fortuitously created a potential standard for higher resolution X-ray microanalysis, which we document here for the first time. Our investigations of the K409 material using a JEOL 8530F include Si K α , Fe K α and Fe L α line profiles several microns in length across the sharp grain boundaries of the Fe-oxides and provide some initial results for X-ray spatial resolution based on the lateral distance over which the intensity shifts between ~10 and 90 % of maximum. Spatial resolution for Si K α improves from >500 nm at 10 keV to ~200-300 nm between 5-8 keV; resolution for Fe K α improves from ~300 nm to ~200 nm with a drop from 10 to 8 keV. The Fe L α line profile acquired at 5-7 keV likewise yields resolution in the ~200 nm range. Results of further examination of K409 and other materials (e.g. a Fe-Si couple) that constrain both beam and X-ray spatial resolution of EPMA using the field-emission gun will be presented.

References:
Marinenko, R.B., 1982. Preparation and Characterization of K-411 and K-412 Mineral Glasses for Microanalysis: SRM 470, Natl. Bur. Stand. (U.S.) Spec. Pub. 260-74.
Marinenko, R.B., Roberson, S., Small, J.A., Thorne, B.B., Blackburn, D. and Kauffman, D., 2000. Preparation and Characterization of K-411 Glass Microspheres, Microsc. Microanal. 6 (6), 542-50.