Analytical perils (and progress) in electron microprobe trace element analysis applied to geochronology: Background acquisition, interferences, and beam irradiation effects

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ABSTRACT

Electron probe microanalysis (EPMA) of accessory minerals such as monazite, xenotime, and thorite for minor- and trace-element concentrations and geochronology, requires consideration of beam irradiation effects (increasing heat and charge) as higher current densities and lengthy counting times are employed, and requires careful, detailed assessment of background intensities and interferences. A carbon coat (250 Å thickness) is generally inadequate for prevention of absorbed current fluctuation and beam damage when using the high current densities applied for high precision (e.g., 200 nA, focused beam). Beam irradiation effects include element mobility in monazite, resulting in P loss relative to REE. Coating materials of higher electrical and thermal conductivity are indicated, and use of gold (≥100 Å) is strongly suggested. Systematic compositional and, therefore, age variability can result simply from analytical effects, requiring evaluation of all aspects of data acquisition. The spectra relevant to measurement of Y, Th, Pb, and U are complex, especially in REE-bearing minerals. Acquisition of detailed spectral wavelength scans allows recognition of background and peak interferences, as well as curvature. Background intensities can be extracted directly from scan data by regression. X-ray mapping allows delineation of domains, guiding background acquisition and detailed quantitative analysis. Minor substitution or fluorescence of unexpected elements can compromise analyses, one documented effect being the fluorescence of K in monazite adjacent to, or hosted by, K-feldspar or micas. This effect, clearly evident within 10 micrometers from K-feldspar, can result in erroneous U concentrations leading to misinterpretation of rim "ages" as younger overgrowths. Absorption edges associated with Th also become relevant to the measurement of U at high Th concentrations. Because background intensity is sensitive to variation in average atomic number, backgrounds must be acquired from each identified domain, with particular attention being paid to Th variation. Misapplication of background intensities can result in large age discrepancies, for example, application of backgrounds obtained from a high-Th domain in monazite to measurement of a low-Th domain (1/3 of the amount in the high-Th domain) results in an overestimation of the U$M\beta$ background intensity of 0.008 cps/nA, and an overall "increase" in age of 70 m.y.