

EPMA methods:

Monazite Analyses

Monazite grains were mapped on the Cameca SX100 using conditions of 15kV, 100nA focused beam, and 100ms dwell time. Quantitative analysis was performed using the CAMECA SX-Ultrachron, which uses an optimized CeB6 source and large and very large monochromators (Allaz et al, 2020). All analyses were done using a 15kV, 200nA focused beam (Jercinovic et al, 2008). The analysis includes the full major and minor element analysis of monazite domains identified in grain maps, as well as high sensitivity analysis of U, Th, and Pb for age analysis, with counting times of 500s for U, 400s for Th, and 700s for Pb. The analysis was implemented using Probe for EPMA (Probe Software, Inc.) in conjunction with Cameca's PeakSight control software. Matrix corrections for quantitative analysis were implemented via the PAP method (Pouchou and Pichoir, 1984). Moacyr monazite (ca. 508 Ma) was used as a consistency reference material periodically throughout the analytical sessions.

Silicate Analyses

Silicate minerals were mapped on the Cameca SX100 at 15 kV, 100nA, and 50ms dwell time. Silicate analyses are performed on the five-wavelength spectrometer Cameca SX100. This instrument has an integrated Bruker EDS and is automated with Cameca's Peaksight software. Analyses were performed at 15 kV, 20 nA, using a 2-micrometer beam diameter. Appropriate oxide and silicate standards were used. Matrix corrections were done via the PAP method (Pouchou and Pichoir, 1984).

Zr-in-rutile Analyses

Full thin section maps, which included a channel for Ti to locate rutile, were acquired on the Cameca SX100 at 15kV, 300nA, and 20ms dwell time. Zirconium-in-rutile quantitative analyses were performed on the Cameca SXfive Tactis. This is a five-wavelength spectrometer instrument with fully integrated Bruker EDS and LaB₆ source. Analyses were performed at 15kV, 200 nA, with a focused beam. Zirconium was measured by simultaneously integrating counts from four large PET (LPET) monochromators. Zr metal was used for calibration. Acquisition was done using Cameca's Peaksight automation software, and matrix corrections were done via PAP (Pouchou and Pichoir, 1984). Background acquisition was done by the multipoint method (Allaz et al., 2019) as implemented by Cameca. Rutile grains with nearby zircon grains were avoided to ensure no boundary fluorescence.

References

- Allaz, J., Williams, M.L., Jercinovic, M.J., Goemann, K., and Donovan, J. (2019). Multipoint Background Analysis: Gaining precision and accuracy in microprobe trace element analysis. *Microscopy and Microanalysis* 25 30-46.
- Allaz, J.M. Jercinovic M.J., and Williams, M.L. (2020) U-Th-Pb total dating of REE-phosphate by electron microprobe: Review and progress. IPT Conf. Series: Materials Science and Engineering 891 (2020) 012001 doi:10.1088/1757-899X/891/1/012001.
- Jercinovic, M.J., Williams, M.L., and Lane, E.D. (2008) In-situ trace element analysis of monazite and other fine-grained accessory minerals by EPMA. *Chemical Geology* 254, 197-215.
- Pouchou, Jean-Louis and Pichoir, Françoise (1984) A new model for quantitative X-ray microanalysis. 1. Application to the analysis of homogeneous samples. *La Recherche Aérospatiale*, Vol 3, pp. 167-192.