

Supplemental Material

$^{40}\text{Ar}/^{39}\text{Ar}$ geochronology and the diffusion of ^{39}Ar in phengite-muscovite intergrowths during step-heating experiments *in vacuo*

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Appendix A - Analytical Procedures

Microstructural analysis was undertaken on each sample to determine the history, the different generations of potassium-bearing minerals and the intra-grain character of each of the minerals analyzed with argon geochronology.

Mineral separation was undertaken after microstructural analysis of thin sections. Separation procedures were undertaken firstly by physical segregation of structures, for example zones where white mica defined a specific fabric commonly only several millimetres wide. White micas were then separated with minimal crushing, only to eliminate composites, followed by significant hand picking procedures for identification of different grain generations and to purify the material analyzed.

Irradiation of samples for $^{40}\text{Ar}/^{39}\text{Ar}$ analysis was undertaken at the HIFAR nuclear reactor of the Australian Nuclear Science and Technology Organization under the support of the *AINSE Award Grant 04P046*. Separates were irradiated for 144 hours in position X33 or X34 (adjacent to the core of the reactor), and inverted three times to reduce the neutron flux gradient along the length of the can; a cadmium liner was used to minimize interference from thermal neutrons. Irradiation procedures followed those described by McDougall and Harrison (1999). GA1550 biotite (98.5 ± 0.8 Ma) has been used as the fluence monitor (Spell and McDougall, 2003). Samples and fluence monitors were wrapped in aluminum for irradiation.

The $^{40}\text{Ar}/^{39}\text{Ar}$ analysis procedures followed those described by McDougall and Brown (2006). Aluminum foil was removed and samples were rewrapped in tin so as to be able to melt the wrap and pump away the contaminated gases prior to the analysis of a sample. Grains were then analyzed using the step-heating method using the furnace technique with temperature control monitored via a thermocouple at the base of a tantalum crucible within a double vacuum resistance furnace. White micas on a VG1200 gas sources mass spectrometer with a Daly detector operating with sensitivities of approximately 5×10^{17} mol/mV⁻¹ and for the fluence monitors on the VG3600 at 7.6×10^{17} mol/mV⁻¹. Machine discrimination was determined from repeated analysis of atmospheric argon, and line blanks were measured at different temperatures prior to analysis. Backgrounds calculations are subtracted for every step. White micas were analyzed with **20–22 steps** rising in temperature from 550°C to 1350°C. The heating schedule was deliberately set to outgas contaminants at lower temperatures so as to minimize mixing with radiogenic argon released from low retentivity sites. Temperature increases in the schedule were increased in small temperature jumps so as to minimize mixing of different gas populations on each step.

Fluence monitors were analyzed using an argon-ion continuous wave laser and the VG3600 Mass Spectrometer. Gas released from each step is exposed to Zr-Al getters to remove active gases for 12 minutes, the purified gas then being isotopically analyzed in the mass spectrometer. The furnace was decontaminated between samples. Corrections for argon produced by interaction of neutrons with K and Ca were made using the following correction factors: $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.5 (\pm 0.14) \times 10^{-4}$, $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7.86 (\pm 0.01) \times 10^{-4}$ and $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.7 (\pm 0.23) \times 10^{-2}$ (Tetley et al., 1980). ^{40}K abundances and decay constants are taken from standard values recommended by the IUGS subcommission on Geochronology (Steiger and Jager, 1977). Stated precisions for $^{40}\text{Ar}/^{39}\text{Ar}$ ages include all uncertainties in the measurement of isotope ratios and are quoted at the 1sigma level.

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