Supplementary materials - Methods and procedures for $^{40}Ar/^{39}Ar$ analysis

Mineral separation

Mineral separation for argon geochronology is based on microstructure analysis on rock samples. Once the target microstructure is identified, rock specimens were dissected to separate specific microstructures for direct dating, with special care to collect only fresh grains for analysis. When white mica deformation fabrics are intergrown and unable to be separated, more than one generation of white mica will be included in the analysis.

Any weathered edges of rock sections were removed, and fresh rock sections were then reduced to 1 cm thick pieces or less by the anvil-top manual rock splitter. The pieces were then milled to < 420 μm grains by the ring mill. Grains are then sieved into four size fractions: 355-420 μm; 250-355 μm; 150-250 μm and < 150 μm. Grains are then checked under the microscope to ensure selection of 'intact' grains. Mineral grains with 355-420 μm diameter were then selected for analysis, and grains were then subjected to magnetic separation using the FRANZ. This procedure separates the biotite-rich fraction, the targeted white mica-rich fraction and the non-magnetic fraction. All separates are packed, the white mica-enriched fraction is then hand-picked under microscope in the microscope room of the Structural and Tectonics group, ANU. Hand picking aims to isolate a pure (~ 99%) fraction of single mineral type for analysis, white mica grains with slight weathering and iron staining from inclusion minerals are removed until visually pristine grains are achieved.

Sample irradiation details

Irradiation of samples for $^{40}Ar/^{39}Ar$ analysis is required, this was undertaken at the University of California Davis McClellan Nuclear Research Centre, CA, US. The samples in this study were done in three irradiation batches, numbered ANU CAN #31, ANU CAN #32 and ANU CAN #33 respectively. For each sample, calculated amounts of grains were weighed, recorded and wrapped in labelled aluminium packets in preparation for irradiation. The sample filled foils were placed into a quartz irradiation canister together with aliquots of the flux monitor GA1550. The GA1550 standards are dispersed throughout the irradiated cannister, between the unknown age samples. In addition, packets containing K_2SO_4, CaF_2 and KCl were placed in the middle of the canister to monitor correction factors and ^{40}Ar production from potassium. Irradiated samples were unwrapped upon their return to the Australian National University, and then rewrapped in tin foil in preparation for analysis in the mass spectrometer.

$^{40}Ar/^{39}Ar$ procedures and analysis information

Samples and standards were analysed in the Argon Laboratory at the Research School of Earth Science, The Australian National University, Canberra, Australia using a Thermo Fisher ARGUS-VI multi-collector mass spectrometer. A furnace step-heating technique was used to extract argon isotopes from the samples to ensure 100% release of ^{39}Ar, while the flux monitors crystals (GA1550 biotite) were fused using a CO_2 continuous-wave laser; gases extracted from both the samples

and standards were analysed in the Argus VI mass spectrometer. Samples had been wrapped in tin foil so as to melt the tin and pump away the gases prior to the sample analysis. The furnace was degassed 4 times at 1,450°C for 15 minutes and the gas pumped away prior to the loading of the subsequent sample. Gas released from flux monitors and each step of sample analyses was exposed to three different Zr-Al getters to remove active gases for 10 minutes and the purified gas was isotopically analysed in the mass spectrometer. Samples were analysed with 30 (micas) to 42 steps (feldspars) and with temperatures of the overall schedule rising from 450° to 1,450°C. The $^{40}Ar/^{39}Ar$ dating technique is adapted from McDougall and Harrison (1999) and described in Forster and Lister (2009). The background levels were measured and subtracted from all analysis, laser and furnace. For example, backgrounds were measured prior to every step of the sample analysis and subtracted from the isotope intensities for ^{40}Ar, ^{39}Ar, ^{38}Ar, ^{37}Ar and ^{36}Ar. The nuclear interfering values for the correction factors for the isotopes are listed below. These are measured for the reactions and uncertainties of $(^{36}Ar/^{37}Ar)Ca$, $(^{39}Ar/^{37}Ar)Ca$, $(^{40}Ar/^{39}Ar)K$, $(^{38}Ar/^{39}Ar)K$ and $(^{38}Ar)Cl/(^{39}Ar)$ K, and were calculated prior to sample analysis.

Flux Monitor: GA 1550 @ 98.5 ±0.8 Ma (Spell & McDougall, 2003)

Lambda ^{40}K5.5430E-10

Table S1 – Irradiation details

Correction Factors	CAN #31	CAN #32	CAN #33
$(^{36}Ar/^{37}Ar)_{Ca}$	2.24512E-04	1.238E-04	1.936E-04
$(^{39}Ar/^{37}Ar)_{Ca}$	7.65305E-04	9.431E-04	8.398E-04
$(^{40}Ar/^{39}Ar)_{K}$	6.60898E-03	2.809E-02	2.713E-02
$(^{38}Ar/^{39}Ar)_{K}$	1.15553E-02	1.136E-02	1.116E-02
$(^{38}Ar)_{Cl}/(^{39}Ar)_{K}$	8.15099E-02	8.026E-02	8.080E-02
Ca/K conversion	1.90	1.90	1.90
Discrimination factor	1.00623 ± 0.20%	1.00568 ± 0.18%	1.00568 ± 0.18%

All canister irradiated at the UC Davis MNRC, CA, US. Irradiation power for CAN #31 to #33 were 20.02 MWh in October 2018. 8.03 MWh in December 2018 and 18.03 MWh in July 2019 respectively. Canister had 1.0 mm cadmium shielding (Tetley et al. 1980).

Table S2 – Sample details

Sample	Foil	J-Factor	J-Factor uncertainty	Mineral	Measurement Date
IO17-05	H2	3.95817E-03	0.4782	White Mica	15 Jan 2019
IO17-04	H3	3.96051E-03	0.4781	White Mica	17 Jan 2019
IO17-03	H4	3.96286E-03	0.4779	White Mica	21 Feb 2019
IO18-01	H5	1.57300E-03	0.2506	White Mica	02 Jun 2019
IO18-01	H9	1.57417E-03	0.2505	K-feldspar	10 Jun 2019
IO18-05	H7	1.57359E-03	0.2505	White Mica	22 Apr 2019

^{40}K abundances and decay constants are those of Steiger & Jager (1977). Stated precisions for ^{40}Ar/^{39}Ar ages include all uncertainties in the measurement of isotope ratios and are quoted at the one sigma level and exclude errors in the age of the fluence monitor GA1550 (98.5 ±0.8 Ma). The reported data have been corrected for system backgrounds, mass discrimination, fluence gradients and atmospheric contamination. GA1550 standards were analysed and a linear best fit was then used for the calculation of the J-factor and J-factor uncertainty.

Data reductions were done with an adapted version of Noble Software (2020, written and adapted by the Australian National University Argon Laboratory). The data reduction was based on optimising MSWD (the mean square of weighted deviates) of isotope intensities with an exponential best fit methodology. The discrimination factor was calculated by analysing Air Shots analysis on either side of sample analysis, based on the atmospheric 40Ar/36Ar ratio (298.57; see Lee et al 2006), and the calculation of the 1amu was used for the discrimination factor.

Tables 1-2, which includes details on: the heating schedule, Argon isotope abundances and uncertainty levels, %Ar*, ^{40}Ar*/^{39}Ar(K), Cumulative ^{39}Ar%, Age and uncertainty, Ca/K, Cl/K, J-factor and J-factor uncertainty, noting that the fractional uncertainties are shown as %, and are stated in the headings of the appropriate columns. Uncertainty levels of the calculated ages are at one sigma.

References

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