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Supplemental Material

Supplementary Information: Geochronology Methods

Table S1.⁴⁰Ar/³⁹Ar isotopic data for white mica, Kalymnos and Telendos islands, Dodecanese, Greece

Table S2. ⁴⁰Ar/³⁹Ar isotopic data for white mica, Kalymnos and Telendos islands, Dodecanese, Greece

1 Supplementary Information

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Paleogeographic position of the central Dodecanese, southeastern Greece: the push-pull of Pelagonia

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14 Introduction

- 15 Herein are the analytical methods used, and data tables of, the geochronological analyses of the rocks
- 16 from Kalymnos and Telendos islands, Dodecanese, Greece. See Figure 9 of the manuscript for sample
- 17 locations.
- 18

19 Tables

- Table S1. ⁴⁰Ar/³⁹Ar isotopic data from multiple single-grain total fusion for white mica, Kalymnos and
 Telendos islands, Dodecanese, Greece
- Table S2. ⁴⁰Ar/³⁹Ar isotopic data from step heated experiments for white mica, Kalymnos and Telendos
 islands, Dodecanese, Greece
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25 Text S1. Analytical Methods

26 Sample preparation

27 Conventional procedures were used to extract and select the most appropriate minerals from rock 28 samples in preparation for (U-Th)/He and 40 Ar/ 39 Ar dating. Each sample was cleaned with a wire brush 29 and water to remove any surface contamination. The sample was allowed to air dry and was then crushed 30 to a size fraction of ≤ 1 cm with the use of a standard jaw crusher. The material was then passed through 31 a series of sieves to obtain grain size fractions >250 µm, 63-250 µm and <63 µm. The 63-250 µm fraction 32 was then rinsed through a decantation process to remove any remaining fine powder adhering to the grains and dried for 24 h at a temperature of 30°C using a heat lamp. Heavy mineral separation was then conducted using methylene iodide (SG: 3.0 g/cm³) in order to obtain a more concentrated fraction of zircon. The heavy mineral separates were then individually passed through a Frantz magnetic mineral separator as needed to further isolate zircon. White mica was obtained from the >250 µm size fraction using a mortar and pestle to further crush the sample while preserving the integrity of the mica. Samples were sieved to obtain a 150 µm-250 µm size fraction. In the case of white mica and zircon grains were then picked using tweezers under a binocular microscope.

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41 ⁴⁰Ar/³⁹Ar geochronology: multiple single-grain total fusion analysis

Once samples were picked and characterized, ⁴⁰Ar/³⁹Ar analytical work was performed at the University of Manitoba (Winnipeg, Canada) using a multi-collector Thermo Fisher Scientific ARGUS VI mass spectrometer, linked to a stainless steel Thermo Fisher Scientific extraction/purification line and Photon Machines (55 W) Fusions 10.6 CO² laser. Argon isotopes (from mass 40 to 37) were measured using Faraday detectors with low noise 1 x 1013 Ω resistors and mass 36 was measured using a compact discrete dynode detector. The sensitivity for argon measurements is ~6.312 x 1017 moles/fA as determined from measured aliguots of Fish Canyon Sanidine (Dazé et al., 2003; Kuiper et al., 2008).

49 Standards and unknowns were placed in 2 mm deep wells in 18 mm diameter aluminium disks, with 50 standards placed strategically so that the lateral neutron flux gradients across the disk could be evaluated. 51 Planar regressions were fit to the standard data, and the ⁴⁰Ar/³⁹Ar neutron fluence parameter, J, 52 interpolated for the unknowns. Uncertainties in J are estimated at 0.1-0.2% (1s), based on Monte Carlo 53 error analysis of the planar regressions (Best et al., 1995). All specimens were irradiated in the cadmium-54 lined, in-core CLICIT facility of the Oregon State University TRIGA reactor. The duration of irradiation was 55 12 h and using the Fish Canyon sanidine (Kuiper et al., 2008) and GA1550 biotite (Spell and McDougall, 56 2003) standards.

57 Irradiated samples were placed in a Cu sample tray, with a KBr cover slip, in a stainless steel high 58 vacuum extraction line and baked with an infrared lamp for 24 h. Single crystals were either fused or step-59 heated using the laser, and reactive gases were removed, after ~3 min, by three GP-50 SAES getters (two 60 at room temperature and one at 450°C) prior to being admitted to an ARGUS VI mass spectrometer by 61 expansion. Five argon isotopes were measured simultaneously over a period of 6 min. Measured isotope 62 abundances were corrected for extraction-line blanks, which were determined before every sample 63 analysis. Line blanks averaged ~5 fA for mass 40 and ~0.022 fA for mass 36. Mass discrimination was monitored by online analysis of air pipettes, which gave during two separate sessions mean of D: 1.0063 ± 0.0001 and D: 1.0089 ± 0.0005 per amu, based on 67 aliquots interspersed with the unknowns. A value of 295.5 was used for the atmospheric ⁴⁰Ar/³⁶Ar ratio (Steiger and Jäger, 1977) for the purposes of routine measurement of mass spectrometer discrimination using air aliquots, and correction for atmospheric argon in the ⁴⁰Ar/³⁹Ar age calculation. Corrections are made for neutron-induced ⁴⁰Ar from potassium, ³⁹Ar and ³⁶Ar from calcium, and ³⁶Ar from chlorine (Roddick, 1983; Renne et al., 1998; Renne and Norman, 2001). Results are reported in **Table S1**.

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72 ⁴⁰Ar/³⁹Ar geochronology: incrementally heating analysis

73 Individual mineral separates were loaded into 2-3 mm-deep aluminum foil packets, which were 74 subsequently stacked vertically into 35-mm long foil tubes and placed into the tubular holes of an aluminum cylinder. Several flux monitor grains of Fish Canyon tuff sanidine (Renne et al., 1998; Steiger 75 76 and Jäger, 1977) were loaded into each sample packet. The vessel was irradiated for 160 MWH in medium 77 flux position 8A at the research nuclear reactor of McMaster University (Hamilton, Canada). Neutron fluence was $\sim 1.08 \times 10^{13}$ neutrons/cm² operating at a 2.5 MW power level. Correction factors for typical 78 79 interference species produced by thermal neutrons during irradiation are 0.058 (40 Ar/ 39 Ar_k), 0.000743 80 $({}^{39}\text{Ar}/{}^{37}\text{Ar}_{\text{Ca}})$ and 0.000258 $({}^{36}\text{Ar}/{}^{37}\text{Ar}_{\text{Ca}})$.

81 Analytical conditions follow the protocol of Kellett and Joyce (2014). Single grain aliquots and 82 monitors were loaded into separate 1.5 mm diameter pits in a copper planchet and placed under vacuum. 83 Individual grains were progressively heated and analyzed using a Photon Machines Ltd. Fusion 10.6 55W 84 CO₂ laser coupled to the all-metal extraction line and a Nu Instruments Noblesse multicollector mass 85 spectrometer operated at the Geological Survey of Canada (Ottawa, Canada). Laser heating was homogenized over a beam radius of 2 mm for a total of 40 s, after which the released gas was exposed to 86 87 SAES NP-10 (~400°C) and HY-STOR 201 (25°C) getters in the extraction line for three minutes. Following 88 gettering, sample gas was expanded into the mass spectrometer. The Nu Noblesse is a single-focussing, 89 Nier-source, magnetic sector multicollector noble gas spectrometer equipped with two quadrupole lens 90 arrays. Argon ions were measured with a fixed array of three ETP discrete dynode ion-counting multipliers 91 (IC0, IC1, IC2). Data collection occurred in two multicollection cycles: cycle $1 = {}^{40}Ar_{IC0}$, ${}^{38}Ar_{IC1}$ and cycle 2 =³⁹Ar_{IC0}, ³⁷Ar_{IC1}, ³⁶Ar_{IC2}. Blanks were run every five analyses, in an identical manner to unknowns. Air shots 92 were analyzed every 10 analyses to monitor efficiency and mass fractionation. Relative collector efficiency 93 and mass bias corrections were made for IC1 and IC2 collectors relative to IC0 using ⁴⁰Ar_{IC0}/³⁶Ar_{IC1} and 94 ⁴⁰Ar_{IC0}/³⁶Ar_{IC2} measurements of air. Mass bias in ICO was not corrected as it would be equally applied to 95

the ⁴⁰Ar/³⁹Ar ratio in unknowns and J-factors determined from monitors in the age calculation, thereby
cancelling out (e.g. Brumm et al., 2010). Error in J-factor values are conservatively estimated at ±0.6% (2s).
Sensitivity of the Nu Noblesse at the time of analyses was 7.1-7.5 Amps/mol. Data collection, reduction,
error propagation, age calculation and plotting were performed using the software MassSpec (version
7.93; Deino, 2001).

101 Analytical results are presented in **Table S2**; Figure 10 presents the data as spectra where the width 102 of each bar (thermal increment) represents the proportion of evolved gas, and the height represents the 103 uncertainty associated with the apparent age. The integrated (or total gas: Tg) age is an average age for 104 the sample calculated by summing the isotopic measurements of all steps with an uncertainty calculated 105 by quadratically combining errors of isotopic measurements of all steps. Plateau ages are conventionally 106 defined as the portion of an age spectrum composed of contiguous increments representing >70% of gas 107 released which result in concordant ages (Mahon, 1996). A preferred age (Tp), on the other hand, is 108 calculated as the weighted mean of a selection of mostly contiguous increments which represent >50% of 109 ³⁹Ar gas released and result in concordant ages. The calculated age uncertainties are relatively small because analytical precision in the age of each heating step is high. 110

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112 (U-Th)/He geochronology

The analytical portion of the (U-Th)/He experiments was conducted at the TRaIL (Thermochronology 113 114 Research and Instrumentation Laboratory) facility at the University of Colorado (Boulder, USA). Individual 115 mineral grains are handpicked using a Leica M165 binocular microscope equipped with a calibrated digital 116 camera and capable of both reflected and transmitted, polarized light. The grains are screened for quality, 117 including crystal shape and the presence of inclusions. The dimensions of the crystals are measured and converted to equivalent spherical radius (ESR) as this value is more readily incorporated into the equations 118 119 that govern the diffusion of He throughout the grain; from this point forward in the paper references to 120 grain size are measurements of ESR. After characterization, grains are placed into small Nb tubes that are 121 then crimped on both ends. This Nb packet is then loaded into an ASI Alphachron He extraction and 122 measurement line. The packet is placed in the UHV extraction line (~3x10⁻⁸ torr) and heated with a diode 123 laser to ~800-1100°C for 5 to 10 m to extract the radiogenic ⁴He. The degassed ⁴He is then spiked with 124 approximately 13 ncc of pure ³He, cleaned via interaction with two SAES getters, and analyzed on a Balzers 125 PrismaPlus QME 220 quadrupole mass spectrometer. Degassed grains are then removed from the line, and taken to a Class 10 clean lab for dissolution. Zircon are dissolved using Parr large-capacity dissolution 126 vessels in a multi-step acid-vapor dissolution process. Grains (including the Nb tube) are placed in Ludwig-127

style Savillex vials, spiked with a ²³⁵U-²³⁰Th tracer, and mixed with 200 µl of Optima grade HF. The vials are 128 129 then capped, stacked in a 125 mL Teflon liner, placed in a Parr dissolution vessel, and baked at 220°C for 130 72 h. After cooling, the vials are uncapped and dried down on a 90°C hot plate until dry. The vials then 131 undergo a second round of acid-vapor dissolution, this time with 200 μ l of Optima grade HCl in each vial 132 that is baked at 200°C for 24 h. Vials are then dried down a second time on a hot plate. Once dry, 200 µl 133 of a 7:1 HNO₃:HF mixture is added to each vial, the vial is capped, and cooked on the hot plate at 90°C for 134 4 h. Once the minerals are dissolved, regardless of the dissolution process, they are diluted with 1 to 3 mL of doubly-deionized water, and taken to the ICP-MS lab for analysis. Mineral standards of Durango apatite 135 (31.5 Ma) and Fish Canyon Tuff zircon (28.2 Ma) are routinely analyzed (degassed and dissolved) in 136 137 conjunction with the samples with each run to ensure data integrity. Sample solutions, along with 138 standards and blanks, are analyzed for U, Th, and Sm content using a Thermo Element 2 magnetic sector 139 mass spectrometer. Once the U, Th, and Sm contents have been measured, He dates and all associated 140 data are calculated on a custom spreadsheet made by TRaIL staff. Results are reported in Table 1 of the 141 manuscript.

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143 References

Best, M., Christiansen, E., Deino, A., Grommé, C., and Tingey, D., 1995, Correlation and emplacement of a
 large, zoned, discontinuously exposed ash flow sheet; the ⁴⁰Ar/³⁹Ar chronology, paleomagnetism,
 and petrology of the Pahranagat Formation, Nevada: Journal of Geophysical Research, v. 100, p.
 24593-24609.

Brumm, A., Jensen, G.M., van den Bergh, G.D., Morwood, M.J., Kurniawan, I., Aziz, F., and Storey, M.,
2010, Hominins on Flores, Indonesia, by one million years ago: Nature, v. 464, p. 748-752.

- Dazé, A., Lee, J., and Villeneuve, M., 2003, An intercalibration study of the Fish Canyon sanidine and biotite
 ⁴⁰Ar/³⁹Ar standards and some comments on the age of the Fish Canyon Tuff: Chemical Geology, v.
 199, p. 111-127.
- Deino, A.L., 2001, Users-manual for Mass Spec v. 5.02: Berkeley Geochronology Center Special Publication
 v. 1a, 119 p.
- Jourdan, F., Verati, C., and Féraud, G., 2006, Intercalibration of the Hb3gr ⁴⁰Ar/³⁹Ar dating standard:
 Chemical Geology, v. 231, p. 77-189.

- Kellett, D., and Joyce, N., 2014, Analytical details of the single- and multicollection ⁴⁰Ar/³⁹Ar measurements
 for conventional step-heating and total-fusion age calculation using the Nu Noblesse at the
 Geological Survey of Canada: Geological Survey of Canada, Technical Note, v. 8, 27 p.
- Kuiper, K., Deino, A., Hilgen, F., Krijgsman, W., Renne, R., and Wijbrans, J., 2008, Synchronizing Rock Clocks
 of Earth History: Science, v. 320, p. 500-504.
- Ludwig, K., 2012, Isoplot 3.75: A Geochronological Toolkit for Microsoft Excel: Berkeley Geochronological
 Centre Special Publication no 5, 75 p.
- 164 Mahon, K., 1996, The new "York" regression; application of an improved statistical method to 165 geochemistry: International Geology Review, v. 38, p. 293–303.
- Renne, P., and Norman, E., 2001, Determination of the half-life of Ar-37 by mass spectrometry: Physical
 Review C, v. 63, doi: 10.1103/PhysRevC.63.047302.
- 168 Renne P., Swisher C., Deino A., Karner D., Owens T., and DePaolo D., 1998, Intercalibration of standards,
 absolute ages and uncertainties in ⁴⁰Ar/³⁹Ar dating: Chemical Geology, v. 145, p. 117-152.
- Roddick, J., 1983, High precision intercalibration of ⁴⁰Ar/³⁹Ar standards: Geochimica Cosmochimica et
 Acta, v. 47, p. 887-898.
- Steiger R., and Jäger E., 1977, Subcommission on geochronology: convention on the use of decay
 constants in geo- and cosmochronology: Earth Planetary Science Letters, v. 36, p. 359-362.
- Spell T., and McDougall I., 2003, Characterization and calibration of ⁴⁰Ar/³⁹Ar dating standards: Chemical
 Geology, v. 198, p. 189-211.