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Supplemental Material

Document 1. Supplementary Information: Analytical Methods

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Supplementary Information – Analytical Methods

Table S1 EPMA for pyroxene at FIU

Major elemental analysis of pyroxene from Unit IV was analyzed at the Florida Center for Analytical Electron Microscopy (FCAEM), Florida International University using JEOL 8900R superprobe instrument. Minerals were analyzed at accelerating voltage of 15 kV and 20 nA beam current using a 1 μm electron beam. Major elements were measured for 15-30 seconds and data were corrected using ZAF correction method. For calibration primary standards used were enstatite (Al, Mg, Si), diopside (Ca), chromite (Cr), olivine (Fe, Mn, Ti), plagioclase (Na), orthoclase (K). The accuracy of measurements for major oxides was better than 5%, trace elements in the minerals close to the detection limit had a higher bias. Minerals were analyzed in back-scattered electron mode to reduce the possibility of analyzing inclusions; two points per grain close to the core were analyzed and averaged. Repeated analysis of FIU standard enstatite STD 350 was carried out at the beginning, middle and end of the analysis to check for data quality and account for any instrument drift.

Table S2 EPMA for pyroxene and olivine at LEEDS

In-situ major element mineral chemistry was analyzed at the Univ. Leeds *LEMAS* Facility using JEOL JXA 8230 microprobe instrument. Clinopyroxenes and olivines were analyzed at accelerating voltage of 20kV and 30 nA beam current using a 3 μm -wide point beam. All major elements were measured for 20-45 seconds on peak and data were corrected using a ZAF (atomic number, absorption, fluorescence) correction procedure. For calibration we used primary standards almandine (Al), olivine USNM Springwater (Si, Mg, Fe), diopside (Si, Mg, Ca), rhodonite (Mn), rutile (Ti), Cr_2O_3 (Cr), Ni metal (Ni) and haematite (Fe). The accuracy of measurement was better

than 5%. Within each crystal, multiple points were analyzed to account for possible differences between core and rim. Analysis were corrected to account for drift in the probe current by using repeated analysis of the Smithsonian Institution NMNH standards diopside St 7308 GEO2 and San Carlos olivine (Fo₉₀) USNM 111312/444.

Table S3 EPMA and LA-ICPMS for amphibole at ANU

Electron microprobe analyses for major element were carried out on 40 amphiboles grains from E68R2W40 layer, using Cameca SX-100 electron probe with 4 spectrometer at the Research School of Earth Sciences (RSES) in Australian National University (ANU), Canberra, Australian. Operating conditions were 15 kV gun voltage, 20 nA sample current and 1 μ m electron beam. The counting times at the peaks were 30s. Geologic standards were used for calibration, and the results were calculated using the Link's ZAF matrix correction program.

In situ trace element concentrations of amphiboles were analyzed at the Key Laboratory of Mineralogy and Metallogeny, Guangzhou Institute of Geochemistry, Chinese Academy of Sciences using Laser ablation inductively coupled plasma-mass spectrometry. The LA-ICPMS system is composed of an Agilent 7900a ICP-MS coupled with a Resonetic RESOLution S155 ArF-Excimer laser source ($\lambda=193$ nm). Laser energy was 4 J/cm², with repetition rate of 6 Hz, spot size of 40 μ m in diameter and a total of 40 s ablation time. Both a double-volume sampling cell and Squid pulse smoothing device were used to improve data quality. Helium gas was used as carrier gas to the ICP source. NIST612 was used as an external, and ⁴³Ca as an internal standard. The trace element data were calculated by ICPMS DataCal 10.2 (Liu et al., 2008).

Table S4) Nd-, Hf- and Sr- isotope analysis of amphibole at USC.

See text and Yogodzinski et al. (2018) for analytical methods.

Table S5) $^{40}\text{Ar}/^{39}\text{Ar}$ dating

Ages of the amphibole separates from Unit IV of Site U1438E were determined using the $^{40}\text{Ar}/^{39}\text{Ar}$ dating facility at the Geological Survey of Japan/AIST. 20-25 g of amphibole-bearing sandstone from two different depth of the core were crushed using a SELFRAG mineral separator at National Museum of Nature and Science, and then sieved to 150 – 250 μm in size. Amphibole crystals were concentrated using heavy liquid and magnetic separator. Then the amphibole concentrates were examined under a microscope, and any impurities were removed. Sample irradiation was done at the CLICIT facility of the Oregon State University TRIGA reactor for 4 hours. Sanidine separated from the Fish Canyon Tuff (FC3) was used for the flux monitor and assigned an age of 27.5 Ma, which has been determined against our primary standard for our K-Ar laboratory, Sori biotite, whose age is 91.2 Ma (Uchiumi and Shibata, 1980).

A CO_2 laser heating system (NEWWAVE MIR10-30) was used at continuous wave mode for sample heating. A faceted lens was used to obtain a 3.2 mm-diameter beam with homogenous energy distribution to ensure uniform heating of the samples during stepwise heating analysis. Argon isotopes were measured on an IsotopX NGX noble gas mass spectrometer fitted with a Hamamatsu Photonics R4146 secondary electron multiplier in a peak-jumping mode.

Correction for interfering isotopes was achieved by analyses of CaF_2 and KFeSiO_4 glasses irradiated with the samples. The blank of the system including the mass spectrometer and the extraction line was 2.9×10^{-14} ml STP for ^{36}Ar , 1.4×10^{-13} ml STP for ^{37}Ar , 1.0×10^{-14} ml STP for ^{38}Ar , 1.2×10^{-14} ml STP for ^{39}Ar and 1.9×10^{-12} ml STP for ^{40}Ar . The blank analysis was done every 2 or 3 step analyses. All errors for $^{40}\text{Ar}/^{39}\text{Ar}$ results are reported at one standard deviation. Errors for ages include analytical uncertainties for Ar isotope analysis, correction for interfering isotopes

and J value estimation. An error of 0.5 % was assigned to J values as a pooled estimate during the course of this study. Results of Ar isotopic analyses and correction factors for interfering isotopes are presented in Supplementary Table DR5.

Plateau ages were calculated as weighted means of ages of plateau-forming steps, where each age was weighted by the inverse of its variance. The age plateaus were determined following the definition by Fleck et al. (1977). Inverse isochrons were calculated using York's least-squares fit, which accommodates errors in both ratios and correlations of errors (York, 1969). See also Fig. 10.

Results of stepwise-heating analyses of amphibole from Unit IV of Site U1438.

Analysis No.	Sample No.	steps	Total gas age ($\pm 1s$)	Plateau age ($\pm 1s$)				
			integrated age	weighted average	inv. isochron	$^{40}\text{Ar}/^{36}\text{Ar}$	MSWD	fraction of
			(Ma)	(Ma)	age (Ma)	intercept		^{39}Ar (%)
	1438E68R2							
17041	38-40 amp	49	45.51 \pm 0.09	47.03\pm0.12	46.8 \pm 0.4	292 \pm 5	1.22	55.8
	1438E68R2							
17158	46-48 amp	15	47.2 \pm 0.5	47.3\pm0.4	48.2 \pm 0.6	285 \pm 6	0.83	100.0

inv. isochron age: inverse

isochron age.

MSWD: mean square of weighted deviates ((SUMS/(n-2))^{0.5}) in York (1969).

Integrated ages were calculated using sum of the total gas released.

$I_b=4.962 \times 10^{-10} \text{Y}^{-1}$, $I_e=0.581 \times 10^{-10} \text{Y}^{-1}$, $^{40}\text{K}/\text{K}=0.01167\%$ (Steiger & Jager 1977).

Atmospheric

$^{40}\text{Ar}/^{36}\text{Ar}$: 295.5

Table S6) U/Pb in Zircons

In-situ U/Pb dating of individual zircon crystals within Unit IV (Table S6) was undertaken using a LA-ICP-MS Agilent mass spectrometer at the University of Tasmania. Ages based on isotopic

ratios were standardized against 91500 zircon (1062.4 Ma) (Wiedenbeck et al., 1995) and calibrations were checked using the Temora (Black et al., 2003) and Plesovice zircons. The weighted average age for these zircon standards, analyzed on the same day as the Unit IV zircons was 1067 ± 24 Ma for the 91500 zircon (weighted average of 9 zircons), 414 ± 15 Ma for 5 Temora zircons and 347 ± 8 Ma for 5 Plesovice zircons (95% confidence level).

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