

Shafaii Moghadam, H., Stern, R.J., Griffin, W.L., Khedr, M.Z., Kirchenbaur, M., Ottley, C.J., Whattam, S., Kimura, J.-I., Ghorbani, G., Gain, S., O'Reilly, S.Y., and Tamura, A., 2019, Subduction initiation and back-arc opening north of Neo-Tethys: Evidence from the Late Cretaceous Torbat-e-Heydarieh ophiolite of NE Iran: GSA Bulletin, <https://doi.org/10.1130/B35065.1>.

Data Repository

SUPPLEMENTARY DATA

Supplementary Table DR1. Representative compositions of minerals in the Torbat-e-Heydarieh ophiolites.

Supplementary Table DR2. Trace elements composition of minerals from THO mantle peridotites and cumulate gabbros.

Supplementary Table DR3. Whole rock data of the THO mantle peridotites and gabbros.

Supplementary Table DR4. Whole rock analysis of the THO magmatic rocks.

Supplementary Table DR5. Whole rock Nd-Hf isotope data for the Torbat-e-Heydarieh ophiolitic rocks.

Supplementary Table DR6. SHRIMP U-Pb data for zircon from magmatic rocks of the Torbat-e-Heydarieh ophiolites.

Supplementary Table DR7. LA-ICPMS U-Pb data for zircon from magmatic rocks of the Torbat-e-Heydarieh ophiolites.

Supplementary Table DR8. Lu-Hf isotope data for zircon from magmatic rocks of the Torbat-e-Heydarieh ophiolites.

ELECTRONIC APPENDIX A

Major element compositions of minerals were analyzed using JEOL wavelength dispersive electron probe X-ray micro-analyzer (JXA 8800R) at Kanazawa University. Accelerating voltage, beam current, and beam diameter for the analyses were 20 kV, 20 nA, and 3 μm , respectively. Representative mineral compositions are reported in Supplementary Table 1.

Trace-element contents of minerals from peridotites and gabbros were analyzed on polished thick-sections in the Geochemical Analysis Unit (GAU) at CCFS/GEMOC, Macquarie University; Sydney, Australia. An Agilent 7700 laser ablation system has been used to analyze trace element abundances in minerals. The Agilent 7700 was coupled with a New Wave UP-266 nm Nd:YAG laser microprobe. Data were collected and processed using the GLITTER software, which allows for the cleanest part of the time-resolved spectrum to be selected, avoiding inclusion phases and host silicate phases. NIST-612 and BCR-2 glasses were used as an external calibration. The trace-element compositions of minerals are reported in Supplementary Table 2.

Major and trace element analyses of pillow lavas, massive lavas, plagiogranites and dikes were carried out using ICP-AES and ICP-MS at CNRS-SARM, Nancy University (France), using BR, DR-N, UB-N, AN-G and GH standards. For major elements, the uncertainty (1 sigma) is better than 2% for concentrations higher than 5 wt.% and better than 5% in the range 0.1-5 wt.%. For trace elements and REEs, the precision is 5% in the range 1-100 ppm and 10% in the range 0.1-1 ppm. The bulk rock major and trace elements analyses for pillow lavas, massive lavas, plagiogranites and dikes are shown in Supplementary Table 4.

Major elements of the Torbat-e-Heydarieh mantle gabbroids and peridotites were analyzed using ICP-AES and ICP-MS at ACME Analytical Laboratories Ltd, Canada. Concentrations of trace elements in Torbat-e-Heydarieh gabbroids and peridotites were determined by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) using a Thermo Scientific X-Series 2 in the Department of Earth Sciences at the University of Durham, following a standard nitric and hydrofluoric acid digestion (Ottley et al., 2003). Sample preparation was undertaken in clean air laminar flow hoods. Briefly the procedure is as follows; into a Teflon vial 4ml HF and 1ml HNO₃ (SPA, ROMIL Cambridge) is added to 100 mg of powdered sample, the vial is sealed and left on a hot plate at 150 °C for 48 h. The acid mixture was evaporated to near dryness, the moist residue has 1 ml HNO₃ added and evaporated again to near dryness. 1 ml HNO₃ was again added and evaporated to near dryness. These steps convert insoluble fluoride species into soluble nitrate species. Finally, 2.5 ml HNO₃ was added and diluted to 50 ml after the addition of an internal standard giving a final concentration of 20 ppb Re and Rh. The internal standard was used to compensate for analytical drift and matrix suppression effects. Calibration of the ICP-MS was via international rock standards (BHVO-1, AGV-1, W-2, and NBS688) with the addition of an in-house standard (GP13) (Ottley et al., 2003). These standards and analytical blanks were prepared by the same techniques as for the THO samples. To improve the signal-to-noise threshold for low abundances of incompatible trace elements in ultramafic rocks, instrument dwell times were increased (Ottley et al., 2003). The composition of the reference samples (W-2, AGV-1, BHVO-1, BE-N, NBS688) was analyzed as unknowns during the same analytical runs. For the analyzed elements, reproducibility of these reference samples is generally better than 2% and the measured composition compares favorably with that

published information in (Potts et al., 1992). The bulk rock major and trace elements analyses for cumulate gabbroids and peridotites are shown in Supplementary Table 3.

Eight whole-rock samples were analyzed for their Nd and Hf isotope compositions (Supplementary Table 5). Chemical separation of Nd and Hf used c. 100 mg of sample powder that was spiked with mixed ^{176}Lu - ^{180}Hf and ^{149}Sm - ^{150}Nd tracers and subsequently digested on a hotplate in a 1:1 mixture of concentrated HF-HNO₃ (24 hours, 120°C). Subsequently, the acid mixture was dried and the samples were digested in a 3:2 mixture of concentrated HF-HNO₃ in steel-jacketed Parr pressure vessels (3 days, 180°C). Subsequently, fluorides were decomposed by drying with 1 ml of perchloric acid and three additional dry-down steps with concentrated HNO₃. The chemical separation of Hf and Lu from the matrix follows the procedure of (Münker et al., 2001). Samarium and Nd were separated from the residue after Hf separation, using Bio-Rad AG50W-X8 cation resin (200-400 mesh) and Ln-Spec resin (Pin and Zalduendi, 1997).

The Nd and Hf isotope compositions as well as Lu-Hf and Sm-Nd concentrations were determined using a Thermo-Finnigan Neptune multi-collector ICP-MS at the joint clean lab facilities in Cologne/Bonn. Data was collected in static multi-collection mode. Values of $^{143}\text{Nd}/^{144}\text{Nd}$ and $^{176}\text{Hf}/^{177}\text{Hf}$ were corrected for mass fractionation using the exponential law and $^{146}\text{Nd}/^{144}\text{Nd}=0.7219$ and $^{179}\text{Hf}/^{177}\text{Hf}=0.7325$, respectively. Repeated analyses of the standards La Jolla Nd and Hf AMES (isotopically identical to JMC-475) yield mean values of $^{143}\text{Nd}/^{144}\text{Nd}=0.511835$ (n=2) and $^{176}\text{Hf}/^{177}\text{Hf}=0.282156$ (n=18). The external long-term reproducibility is c. ± 40 ppm for Nd and Hf isotopes (2 RSD). Reported values are given relative to 0.511859 for La Jolla and 0.282160 for Hf AMES. Procedural blanks were typically below 60 pg for Hf and Nd. The external precision of the Nd and Hf measurements was further assessed by multiple digestions of six samples (see Supplementary Table 5). External precision (2 RSD) for the replicates was better than 30 ppm for $^{143}\text{Nd}/^{144}\text{Nd}$ and 40 ppm for $^{176}\text{Hf}/^{177}\text{Hf}$.

In order to have precise ages for the Torbat-e-Heydariyeh ophiolite, zircons from plagiogranites (4 samples) and diabasic (1 sample)-rodingitized gabbroic dike (1 sample) within the mantle harzburgite were analyzed by SHRIMP at the Korea Basic Science Institute, Ochang, South Korea and LA-ICPMS at Geochemical Analysis Unit (GAU), CCFSG/GEMOC, Macquarie University. For SHRIMP analysis, zircon grains were mechanically separated using conventional mineral separation techniques that employed crushing, grinding, sieving, and magnetic separation steps, followed by handpicking of zircons under a binocular microscope. The internal structure of these zircon grains was studied using transmitted and reflected light optical microscopy, and by scanning electron microscope-cathodoluminescence (CL) imaging. Analytical procedures including instrumental set-up, and the acquisition and treatment of data employed in this SHRIMP U-Pb zircon dating study, are outlined in (Compston et al., 1984). For the reduction of raw data and in making the final age calculations, we used the programs SQUID and Isoplot/Ex (Ludwig, 2003, 2009). The SHRIMP U-Pb zircon analytical data are summarized in Supplementary Table 6.

For LA-ICPMS analysis, zircons were separated following electrostatic disaggregation (selFrag) of the rock sample, then using standard gravimetric and magnetic techniques; grains were picked under a binocular microscope and mounted in epoxy discs for analysis. All grains were imaged by CL and BSE to provide maps to guide the choice of analytical spots. Zircon U-Pb ages were obtained using a 193 nm ArF EXCIMER laser with an Agilent 7700 ICP-MS system. Detailed method descriptions have been given by

(Jackson et al., 2004). The ablation conditions included beam size (30 μm), pulse rate (5Hz) and energy density (7.59 J/cm²). Analytical runs comprised 16 analyses with 12 analyses of unknowns bracketed by two analyses of a standard zircon GJ-1 at the beginning and end of each run, using the established TIMS values (²⁰⁷Pb/²⁰⁶Pb age= 608.5 Ma, (Jackson et al., 2004)). U-Pb ages were calculated from the raw signal data using the on-line software package GLITTER (Griffin et al., 2008). U-Pb age data were subjected to a common-lead correction, except for those with common-Pb concentrations lower than detection limits. The results were processed using the ISOPLOT program of (Ludwig, 2003). The external standards, zircons 91500 and Mud Tank, gave mean ²⁰⁶Pb/²³⁸U ages of 1063.5 \pm 1.8 Ma (MSWD=1.3) and 731.1 \pm 1.2 Ma (MSWD=0.77), respectively, which are similar to the recommended ²⁰⁶Pb/²³⁸U ages of 1062.4 \pm 0.4 Ma and 731.9 \pm 3.4 Ma respectively (Chang et al., 2006; Woodhead and Hergt, 2005; Yuan et al., 2008). LA-ICPMS U-Pb zircon analytical data is summarized in Supplementary Table 7.

In situ zircon Lu-Hf isotopic analyses were performed using a Nu Plasma multi-collector ICP-MS, coupled to a Photon Machines 193 nm ArF excimer laser system at CCFS (Macquarie University). The analyses were carried out using the Nu Plasma time-resolved analysis software. The methods, including calibration and correction for mass bias, are described by (Griffin et al., 2004; Griffin et al., 2000). The ablation spots (55 μm) for the Hf isotope analyses were situated close to the U-Pb analysis positions on each grain. The accuracy of the Yb and Lu corrections during LA-MC-ICPMS analysis of zircon has been demonstrated by repeated analysis of standard zircons with a range in ¹⁷⁶Yb/¹⁷⁷Hf and ¹⁷⁶Lu/¹⁷⁷Hf. Four secondary standards (Mud Tank and Temora) were analyzed between every ten unknowns to check instrumental stability. ¹⁷⁶Hf/¹⁷⁷Hf ratios of the Mud Tank zircon gave an average of 0.2825355 \pm 0.0000041 (2SD; n=122); those of Temora gave 0.2826971 \pm 0.0000078 (2SD; n=42). These values are identical to those recommended for Mud Tank (0.282507 \pm 0.000003) and Temora (0.282693 \pm 0.000052) (Fisher et al., 2014). The isobaric interferences of ¹⁷⁶Lu and ¹⁷⁶Yb on ¹⁷⁶Hf are very limited, because of the extremely low ratios of Lu/Hf and Yb/Hf in the measured standard zircons. The interference of ¹⁷⁶Yb on ¹⁷⁶Hf was corrected by measuring the interference-free ¹⁷²Yb isotope and using ¹⁷⁶Yb/¹⁷²Yb to calculate ¹⁷⁶Yb/¹⁷⁷Hf. The appropriate value of ¹⁷⁶Yb/¹⁷²Yb was determined by successive spiking the JMC475 Hf standard (1 ppm solution) with Yb, and iteratively finding the value of ¹⁷⁶Yb/¹⁷²Yb required to yield the value of ¹⁷⁶Hf/¹⁷⁷Hf obtained on the pure Hf solution (Griffin et al., 2004; Griffin et al., 2000). Zircon Hf isotope data are presented in Supplementary Table 8.

REFERENCES CITED

- Chang, Z.S., Vervoort, J.D., McClelland, W.C., and Knaack, C., 2006, U-Pb dating of zircon by LA-ICP-MS: *Geochemistry Geophysics Geosystems*, v. 7, <https://doi.org/10.1029/2005GC001100>.
- Compston, W., Williams, I., and Meyer, C., 1984, U-Pb geochronology of zircons from lunar breccia 73217 using a sensitive high mass-resolution ion microprobe: *Journal of Geophysical Research. Solid Earth*, v. 89, no. S02, <https://doi.org/10.1029/JB089iS02p0B525>.
- Fisher, C.M., Vervoort, J.D., and DuFrane, S.A., 2014, Accurate Hf isotope determinations of complex zircons using the “laser ablation split stream” method: *Geochemistry Geophysics Geosystems*, v. 15, no. 1, p. 121–139, <https://doi.org/10.1002/2013GC004962>.
- Griffin, W.L., Pearson, N.J., Belousova, E., Jackson, S.E., van Acherbergh, E., O’Reilly, S.Y., and Shee, S.R., 2000, The Hf isotope composition of cratonic mantle: LAM-MC-ICPMS

- analysis of zircon megacrysts in kimberlites: *Geochimica et Cosmochimica Acta*, v. 64, no. 1, p. 133–147, [https://doi.org/10.1016/S0016-7037\(99\)00343-9](https://doi.org/10.1016/S0016-7037(99)00343-9).
- Griffin, W.L., Belousova, E.A., Shee, S.R., Pearson, N.J., and O'Reilly, S.Y., 2004, Archean crustal evolution in the northern Yilgam Craton: U-Pb and Hf-isotope evidence from detrital zircons: *Precambrian Research*, v. 131, no. 3–4, p. 231–282, <https://doi.org/10.1016/j.precamres.2003.12.011>.
- Griffin, W., Powell, W., Pearson, N., and O'reilly, S., 2008, GLITTER: data reduction software for laser ablation ICP-MS: *Laser Ablation-ICP-MS in the earth sciences. Mineralogical association of Canada short course series*, v. 40, p. 204–207.
- Jackson, S.E., Pearson, N.J., Griffin, W.L., and Belousova, E.A., 2004, The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U-Pb zircon geochronology: *Chemical Geology*, v. 211, no. 1–2, p. 47–69, <https://doi.org/10.1016/j.chemgeo.2004.06.017>.
- Ludwig, K.R., 2003, User's Manual for Isoplot 3.00: A Geochronological Toolkit for Microsoft Excel.
- Ludwig, K.R., 2009, SQUID 2: A User's manual: Berkeley Geochronology Center Special Publications 4.
- Münker, C., Weyer, S., Scherer, E., and Mezger, K., 2001, Separation of high field strength elements (Nb, Ta, Zr, Hf) and Lu from rock samples for MC-ICPMS measurements: *Geochemistry Geophysics Geosystems*, v. 2, <https://doi.org/10.1029/2001GC000183>.
- Ottley, C., Pearson, D., and Irvine, G., 2003, A routine method for the dissolution of geological samples for the analysis of REE and trace elements via ICP-MS: *Plasma source mass spectrometry: Applications and emerging technologies*, no. 288, p. 221.
- Pin, C., and Zalduogui, J.F.S., 1997, Sequential separation of light rare-earth elements, thorium and uranium by miniaturized extraction chromatography: Application to isotopic analyses of silicate rocks: *Analytica Chimica Acta*, v. 339, p. 79–89, [https://doi.org/10.1016/S0003-2670\(96\)00499-0](https://doi.org/10.1016/S0003-2670(96)00499-0).
- Potts, P.J., Tindle, A.G., and Webb, P., 1992, *Geochemical reference material compositions: rocks, minerals, sediments, soils, carbonates, refractories & ores used in research & industry*, Taylor & Francis.
- Woodhead, J.D., and Hergt, J.M., 2005, A preliminary appraisal of seven natural zircon reference materials for in situ Hf isotope determination: *Geostandards and Geoanalytical Research*, v. 29, no. 2, p. 183–195, <https://doi.org/10.1111/j.1751-908X.2005.tb00891.x>.
- Yuan, H.L., Gao, S., Dai, M.N., Zong, C.L., Gunther, D., Fontaine, G.H., Liu, X.M., and Diwu, C., 2008, Simultaneous determinations of U-Pb age, Hf isotopes and trace element compositions of zircon by excimer laser-ablation quadrupole and multiple-collector ICP-MS: *Chemical Geology*, v. 247, no. 1–2, p. 100–118, <https://doi.org/10.1016/j.chemgeo.2007.10.003>.