Supplemental Item A: Supplemental Analytical Methods forSubduction Initiation recorded in the Dadeville Complex of Alabama and Georgia, USA

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# 1. ANALYTICAL METHODS

## 1.1. Mineral Separation Procedures

Samples selected for zircon U-Pb analysis were crushed using a stainless-steel ring-and-puck mill in a FRITSCH Pulverisette 9. Crushing was done in 5 second intervals at 600 rpm and samples were sieved to < 250 µm. Crushed samples were washed using soap and water and decanted to clean of clay particulates, then placed in a ~60 °C drying oven until completely dry. Dry samples were then subjected to magnetic separation using Frantz magnetic separator angled at 7° and 15°, incrementally increasing the voltage until 1.4 A was reached.

The non-magnetic fraction at 1.4 A was then separated using sodium polytungstate (SPT) heavy liquid separation following the method of Andò (2020). Approximately 15 grams of sample material and 40 mL of SPT (density >2.95 g/cm3) were placed into 50 mL falcon tubes and agitated by hand before placed in an ultrasonic bath for 5 minutes to ensure wetting of all grains. Samples were then agitated by hand and centrifuged for 6 minutes at 300 rpm. The agitating and centrifuging step was repeated in triplicate and tubes were set aside to allow grains to settle. Once settled, the floating grains were removed by flash-freezing the bottom of the tube with an ethanol-dry ice bath (T < -70 ° C) until the SPT mixture solidified and decanting the remaining sample into a Büchner funnel consisting of two layers of filters sitting atop a vacuum flask attached to a WaterSaver faucet aspirator. The tube was rinsed in triplicate to remove all floating grains and the frozen portion allowed to thaw. Once fully thawed, the remaining sample was poured into a second Büchner funnel setup and rinsed in triplicate to remove all grains.

Heavy mineral separates were then inspected using a Leica EZ4W petrographic microscope and zircons were picked by hand. Selected zircons were placed on double sided tape and a clear PVC ring of outer inch diameter was placed around the grains. Epoxy resin was poured into the ring and allowed to cure for >24 hours. Epoxy mounts were polished to expose cores of zircon grains using 12 µm, 9 µm, 5 µm and 3 µm grit and washed in an ultrasonic bath between each grit. Alumina polishing paste of 0.1 µm grit was used to finalize polish for LA-ICP-MS analysis.

## 1.1.2. U-Pb Geochronology CA-TIMS

U-Pb dates were obtained by the chemical abrasion isotope dilution thermal ionization mass spectrometry (CA-TIMS) method from analyses composed of single zircon grains (Appendix B), modified after Mattinson (2005). Zircon was removed from the epoxy mounts for dating based on CL images and LA-ICPMS data, and then annealed in a muffle furnace at 900 °C for 60 hours in quartz beakers.

Zircon was put into 3 ml Teflon PFA beakers and loaded into 300 µl Teflon PFA microcapsules. Fifteen microcapsules were placed in a large-capacity Parr vessel and the zircon partially dissolved in 120 µl of 29 M HF for 12 hours at 190 °C. Zircon was returned to 3 ml Teflon PFA beakers, HF was removed, and zircon was immersed in 3.5 M HNO3, ultrasonically cleaned for an hour, and fluxed on a hotplate at 80°C for an hour. The HNO3 was removed, and zircon was rinsed twice in ultrapure H2O before being reloaded into the 300 µl Teflon PFA microcapsules (rinsed and fluxed in 6 M HCl during sonication and washing of the zircon) and spiked with the Boise State University mixed 233U-235U-205Pb tracer solution (BSU-1B). Zircon was dissolved in Parr vessels in 120 µl of 29 M HF with a trace of 3.5 M HNO3 at 220 °C for 48 hours, dried to fluorides, and re-dissolved in 6 M HCl at 180 °C overnight. U and Pb were separated from the zircon matrix using an HCl-based anion-exchange chromatographic procedure (Krogh, 1973), eluted together and dried with 2 µl of 0.05 N H3PO4.

Pb and U were loaded on a single outgassed Re filament in 5 µl of a silica-gel/phosphoric acid mixture (Gerstenberger and Haase, 1997), and U and Pb isotopic measurements made on a GV Isoprobe-T multicollector thermal ionization mass spectrometer equipped with an ion-counting Daly detector. Pb isotopes were measured by peak-jumping all isotopes on the Daly detector for 160 cycles and corrected for 0.16 ± 0.03%/a.m.u. (1σ) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on 204Pb and 207Pb, disappeared within approximately 60 cycles, while ionization efficiency averaged 104 cps/pg of each Pb isotope. Linearity (to ≥1.4 x 106 cps) and the associated deadtime correction of the Daly detector were determined by analysis of NBS982. Uranium was analyzed as UO2+ ions in static Faraday mode on 1012 ohm resistors for 300 cycles, and corrected for isobaric interference of 233U18O16O on 235U16O16O with an 18O/16O of 0.00206. Ionization efficiency averaged 20 mV/ng of each U isotope. U mass fractionation was corrected using the known 233U/235U ratio of the Boise State University tracer solution.

U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007), calibration of BSU-1B tracer solution of 235U/205Pb of 77.93 and 233U/235U of 1.007066 for, U decay constants recommended by Jaffey et al. (1971), and 238U/235U of 137.818 (Hiess et al., 2012). 206Pb/238U ratios and dates were corrected for initial 230Th disequilibrium using DTh/U = 0.20 ± 0.05 (1σ) and the algorithms of Crowley et al. (2007), resulting in an increase in the 206Pb/238U dates of ~0.09 Ma. All common Pb in analyses was attributed to laboratory blank and subtracted based on the measured laboratory Pb isotopic composition and associated uncertainty. U blanks are estimated at 0.013 pg.

A weighted mean 206Pb/238U date is calculated from equivalent dates (probability of fit >0.05) using Isoplot 3.0 (Ludwig, 2003) with error at the 95% confidence interval. Error is computed as the internal standard deviation multiplied by the Student's t-distribution multiplier for a two-tailed 95% critical interval and n-1 degrees of freedom when the reduced chi-squared statistic, mean squared weighted deviation (MSWD) (Wendt and Carl, 1991), takes a value less than its expectation value plus its standard deviation at the same confidence interval, which is when MSWD is <1+2\*sqrt[2/(n-1)]. This error is expanded via multiplication by the sqrt(MSWD) when the MSWD is ≥1+2\*sqrt[2/(n-1)] to accommodate unknown sources of over dispersion. Error on the weighted mean 206Pb/238U date is given as ± x / y / z, where x is the internal error based on analytical uncertainties only, including counting statistics, subtraction of tracer solution, and blank and initial common Pb subtraction, y includes the tracer calibration uncertainty propagated in quadrature, and z includes the 238U decay constant uncertainty propagated in quadrature. Internal error should be considered when comparing our date with 206Pb/238U dates from other laboratories that used the same tracer solution or a tracer solution that was cross-calibrated using EARTHTIME gravimetric standards. Error including the uncertainty in the tracer calibration should be considered when comparing our date with those derived from other geochronological methods using the U-Pb decay scheme (e.g., laser ablation ICPMS). Error including uncertainties in the tracer calibration and 238U decay constant (Jaffey et al., 1971) should be considered when comparing our date with those derived from other decay schemes (e.g., 40Ar/39Ar, 187Re-187Os). Errors on dates from individual analyses are at 2σ.

# 3. REFERENCES

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