Maniitsoq Geochronology Metadata

The full dataset of U-Pb zircon ages is reported in Olierook et al. (2021).

SHRIMP

SIMS U–Pb analysis was performed using Shrimp B in the John de Laeter Centre following analytical protocols matching those discussed in Wingate and Kirkland (2015). During the range of sessions these samples were analysed in, BR266 (Stern, 2001) was used as the primary reference material and at least ten analyses of this material were made interspersed with unknowns per session (Stern, 2001). Indicated external spot-to-spot (reproducibility) uncertainty was always better than 1% (1σ), and $^{238}U/^{206}Pb*$ calibration uncertainty was always better than 0.5 % (1σ). Calibration uncertainties are included in the errors of $^{238}U/^{206}Pb$ ratios and dates in Appendix Table 1. Analyses of the OG1 (3465.4 ± 0.6 Ma Stern, et al., 2009) standard yielded a $^{207}Pb/^{206}Pb$ weighted mean age of 3464 ± 3 Ma identical to the accepted value, and no $^{207}Pb/^{206}Pb$ fractionation correction is deemed necessary. Common-Pb corrections were applied to all SIMS analyses using contemporaneous isotopic compositions determined according to the Pb evolution model of Stacey and Kramers (1975).

LA-ICPMS

Zircon

Zircon mineral fractions were also analysed using laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the GeoHistory Facility, John de Laeter Centre, Curtin University. Targeted portions of individual zircon grains were ablated using a Resonetics M-50 193nm ArF excimer laser ablation system. In samples 000313 and 000319 isotopic intensities were measured using a Nu-plasma II ICP-MS, with high purity Ar as the plasma gas, with all isotopic masses except ^{238}U measured using ion counters. Instrumental mass bias, drift and inter-element fractionation corrections were performed by standard-sample bracketing. Following an integration time of 10 seconds for background analysis, samples were spot ablated for 30 seconds at a 7 Hz repetition rate in an ultrahigh purity He-N2 atmosphere using a 15 μm beam and laser energy of 2.5 J/cm^2. Plešovice reference zircon (337 Ma; Sláma et al., 2008) was used as the primary age standard in this study. Additionally, the following reference materials were run to verify the procedure: OG1 zircon (3465 Ma; Stern et al., 2009), and GJ1 (601.7 ± 1.4 Ma; Jackson et al., 2004). Analyses with $f^{204}\%$ >+2% and >10% discordance are not used for placing age constraints. Data were reduced in iolite using the Geochron3 reduction scheme and in-house excel macros. Errors for each spot are propagated by quadratic addition of within-run errors and the reproducibility of GJ1. ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb were measured for 0.03 seconds, ^{232}Th for 0.0125 seconds and ^{238}U for 0.0125 seconds. Reference materials were run after 10 unknowns with OG1 (3465 Ma; Stern et al., 2009) used as a primary standard with secondary standard GJ1 (601.7 ± 1.4 Ma; Jackson et al., 2004) used to verify the procedure. No common-Pb correction is applied due to low ^{204}Pb counts. Data reduction used the U/Pb Geochronology3 data reduction schemes in Iolite (Paton et al., 2011) and in-house Microsoft Excel macros.

Hf isotopes on selected samples were analysed using the Curtin GeoHistory Facility, reported in Gardiner et al. (2019), and the implications for the regional geology are discussed in relevant sections of these notes. Samples were analysed in split stream configuration for U-Pb and Lu-Hf isotopes. Zircon was ablated using a Resonetics RESOlution M–50A–LR system, incorporating a COMPex 102 193 nm excimer UV laser. Following a cleaning pulse and a 40 s period of background analysis, samples were spot ablated for 35 s at a 10 Hz repetition rate using a 50μm beam and laser energy at the sample surface of 2.0 J/cm^2. An additional 40 s of baseline was collected after ablation. The sample cell was flushed with ultrahigh purity He (300 mL/min) and N$_2$(1.0 mL/min) and high purity Ar was employed as the plasma carrier gas.

Six U–Pb isotopes were measured using an Agilent 7700s quadrupole ICPMS, with high-purity Ar as the plasma gas (flow rate 0.98 L min^{-1}). The dwell time on the mass stations was ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb (0.03 s each), ^{232}Th (0.0125 s), and ^{238}U (0.0125 s). During time-resolved processing, contamination resulting from inclusions and compositional zoning was monitored, and only the relevant part of the signal was integrated. The primary reference material used for U–Pb dating in this study was zircon standard OG1 (3465.4 ± 0.6 Ma Stern, et al., 2009), which has an ablation response similar to the Archean grains of interest. Secondary standard zircon 91500 (1062.4 ± 0.4 Ma; Wiedenbeck et al., 1995), GJ-1 (601.7 ± 1.4 Ma; Jackson, et al., 2004), and Plešovice (337.13 ± 0.37 Ma; Sláma, et al., 2008) yielded ^{206}Pb/^{238}U ages within 1% of the accepted value when reduced using appropriate matrix matched standards. The time-resolved mass spectra were reduced using the U/Pb Geochronology data reduction schemes in IoliteTM (Paton, et al. 2011), and in-house Microsoft Excel macros. No common lead corrections were deemed necessary due to generally low ^{204}Pb counts. Those analyses with elevated ^{204}Pb counts are not used in placing age constraints. All ages are reported with ±2σ uncertainties unless specifically stated otherwise.The ablated split for Lu–Hf analysis was measured on a Nu Plasma II multi-collector inductively coupled plasma mass spectrometer. All isotopes (^{180}Hf, ^{179}Hf, ^{178}Hf, ^{177}Hf, ^{176}Hf, ^{175}Lu, ^{174}Hf, ^{173}Yb, ^{172}Yb and ^{171}Yb) were counted on the Faraday collector array. Time-resolved data were baseline subtracted and reduced using Iolite (DRS after Woodhead et al., 2004), where ^{176}Yb and ^{176}Lu were removed from the 176 mass signal using ^{176}Yb/^{173}Yb = 0.7962 and ^{176}Lu/^{175}Lu = 0.02655 with an exponential law mass bias correction assuming ^{172}Yb/^{173}Yb = 1.35274 (Chu et al., 2002). The interference corrected ^{176}Hf/^{177}Hf was normalized assuming ^{179}Hf/^{177}Hf = 0.7325 (Patchett and Tatsumoto, 1980) for mass bias correction. The mass spectrometer was initially tuned using Yb and Lu-doped JMC475 Hf solutions introduced using the Aridus II desolvating nebulizer, with the goal of maximizing Hf detection efficiency while minimizing oxide production. A range of different zircon crystals from reference standards were analysed together with the samples in each session to monitor the accuracy of the results. In addition, the corrected ^{180}Hf/^{177}Hf ratio was calculated to monitor the accuracy of the mass bias correction and yielded an average value of 1.886754, which is within the range of values reported by Thirlwall and Anczkiewicz (2004). Calculation of εHf values used the decay constant of Scherer et al. (2001) and the Chondritic Uniform Reservoir (CHUR) values of Blichert-Toft and Albarède (1997).

Apatite

Apatite data is reported in Kirkland et al. (2018). Apatite grains (mounted and polished in 25 mm epoxy rounds) were ablated using a Resonetics RESOlution M-50A-LR system, incorporating a COMPex 102 193 nm excimer UV laser (the same laser as the zircon U-Pb analyses), with a 50 μm diameter laser spot, 5 Hz laser repetition rate, and laser energy of 1.4 J cm^{-2}. Isotopic intensities were measured using an Agilent 7700s quadrupole ICPMS, with high purity Ar as the plasma gas (1 L min^{-1}). The experiment used the following schedule: 2 x 1 second cleaning pulse; 10 seconds delay; 15 seconds background measurement;

30 seconds of sample measurement; 15 seconds of cell washout. The sample cell was flushed by ultrahigh purity He (350 mL min^{-1}) and N (3.8 mL min^{-1}). Apatite mass spectra were reduced using the VizualAge_UcomPbine data reduction scheme in Iolite (Chew et al., 2014; Paton et al., 2010) and in-house excel macros. The primary age standard used for LA-ICPMS apatite analysis was Mount McClure (524 Ma; Schoene and Bowring, 2006) with MAD (485 ± 1.7 Ma; Thomson et al., 2012) employed as a secondary standard to verify the procedure. Regressions from Stacey and Kramers, (1975) contemporaneous common Pb through the secondary standard, treated as an unknown, yielded an age within analytical uncertainty of the accepted value (MAD = 490 ± 4 Ma, MSWD = 0.4, n = 67).

Titanite

Titanite U–Pb and trace-element compositions were obtained at the GeoHistory Facility in the John de Laeter Centre, Curtin University in Perth, Australia and are reported in Kirkland et al. (2020). Titanite grains were ablated using a Resonetics RESOlution M-50A-LR system incorporating a COMPex 102, 193 nm excimer UV laser. Analyses were performed at a 5 Hz laser repetition rate with a laser fluence of 2.6 J·cm^{-2} as measured on the sample surface. Throughout the ablation the sample cell was flushed with ultrahigh purity He (350 mL·min^{-1}) and N (3.8 mL·min^{-1}). Further details on the titanite analytical protocol are given in Kirkland et al. (2018). Isotopic abundances were measured using an Agilent 8900 triple quadrupole ICPMS, with high purity Ar as the plasma gas (1 L·min^{-1}). A spot size of 50 µm was used for analyses on individual titanite grains mounted in epoxy resin and 10 µm spot size on thin sections. Ablation times of 30 s bracketed between two 20 s periods of background collection were used. Most trace elements were analysed for 0.01 s, but the dwell time was increased to 0.05 s for ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th and ^{238}U. U–Pb LA-ICP-MS data were reduced using VisualAge UComPbine data reduction scheme in Iolite and in-house excel macros (Chew et al., 2014; Paton et al., 2011). The primary age standard used for the titanite analyses was OLT1 (1015 ± 2 Ma; Kennedy et al., 2010). Titanite standard BLR-1 (1047.1 ± 0.4 Ma; Aleinikoff et al., 2007) was run as an unknown and a regression from contemporaneous Stacey and Kramers terrestrial model common Pb through the uncorrected data for BLR-1 yielded a date of 1047 ± 6 Ma (MSWD = 0.63, n = 15). We calculate f^{207} to assess the amount of common Pb within titanite analyses. f^{207} is the distance upwards along a mixing line towards common Pb from radiogenic Pb where 100% represents an analysis dominated by the common component. f^{207} can be readily calculated as the percentage difference between the U/Pb ratio predicted for concordance at the 207-corrected date versus the measured U/Pb ratio. Trace-element data were reduced using the Trace-element IS data reduction scheme in Iolite (Paton et al., 2010). MKED1 (Spandler et al., 2016) was used as the primary standard to calculate elemental concentrations (using 47Ti as the internal standard element) and to correct for instrument drift. 18.16 wt% Ti was assumed for titanite unknowns. Secondary trace element standards (OLT1, BLR-1, NIST 610, NIST 612) reproduce recommended values to within 2% for all elements when reduced using appropriate matrix matched standard.

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