

Hunter-Bowen deformation in South Percy Island, northeastern Australia

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SUPPLEMENTARY PAPERS

Australian Journal of Earth Sciences (2018) 65 (2),
<http://dx.doi.org/10.1080/08120099.2018.1419506>

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METHODS AND ANALYTICAL PROCEDURES

Geochemical analysis

Prior to analysis, sawn slices of fresh rock were soaked in deionised water to remove sea salt, dried, and crushed in a tungsten carbide ring mill. X-ray fluorescence (XRF) major element, loss on ignition and As, Cu, Ni, Pb, Sc and Zn analyses were made in January 2016 by Spectrachem Analytical, Lower Hutt, New Zealand (job SA18623). Additional trace element data were obtained by fused bead inductively coupled plasma mass spectrometry (ICP-MS) by ALS Laboratories Brisbane, Australia in January 2017 (workflow ME-MS81, job BR16228150). Whole rock geochemical data for six South Island Terrane rocks are available in Appendix 1, and sample and analytical data have been lodged in the Petlab database (<http://pet.gns.cri.nz>; Strong et al. 2016).

We used MORB-normalised spider diagrams with elements in order of decreasing compatibility in igneous systems (Sun & McDonough, 1989) to make simple interpretations of the petrochemistry. Elements such as K, Cs, Sr, Rb, Ba, Pb and U are known to be especially mobile during metamorphism and have been omitted from the figures, and although there may be some issues with Th-addition, this does not greatly affect our interpretations.

U–Pb zircon geochronology

U–Pb geochronology of zircon crystals was conducted at the Central Analytical Research Facility, operated by the Institute for Future Environments at Queensland University of Technology. Zircon concentrates were obtained using conventional crushing, sieving, and washing, followed by magnetic and heavy liquid (diiodomethane) separation. Zircon crystals were mounted, polished, and imaged before analysis of isotopic ratios and trace element concentrations by LA-ICP-MS. Full methodology and instrumental configuration following the guidelines of Horstwood et al. (2016) are reported in Table A1.

The analytical procedure involved alternating between approximately 10 unknown zircon crystals and 4 reference material analyses. The NIST 610 glass (Pearce et al., 1997) was used as a reference material for trace element determination while Temora 2 (TIMS $^{206}\text{Pb}/^{238}\text{U}$ age = 416.78 ± 0.33 Ma, Black et al., 2004), R33 ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ age of 419.26 ± 0.39 Ma (Black et al., 2004) and Plešovice (ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ age = 337.13 ± 0.37 Ma, Sláma et al., 2008) were the primary and two monitor zircon reference materials, respectively. We report a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age for the R33 reference material of 425.3 ± 7.2 Ma (95% conf., $n_{\text{conc}} = 12/27$, MSWD = 0.52, prob. = 0.89) and a coherent but slightly discordant $^{206}\text{Pb}/^{238}\text{U}$ – $^{207}\text{Pb}/^{235}\text{U}$ bivariate weighted mean age ('concordia age', Ludwig, 2003) of 421.5 ± 4.1 Ma ($n = 27$, MSWD_{equiv.} = 0.85, prob_{equiv.} = 0.77, MSWD_{conc.} = 5.8, prob_{conc.} = 0.016), which are both within error of the accepted age. The reported weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age for the Plešovice reference material is slightly too young at 331.0 ± 3.6 Ma (95% conf., $n_{\text{conc}} = 23/26$, MSWD = 0.56, prob. = 0.95). However, the Plešovice data form a coherent but slightly discordant $^{206}\text{Pb}/^{238}\text{U}$ – $^{207}\text{Pb}/^{235}\text{U}$ bivariate weighted mean age ('concordia age', Ludwig, 2003) of 328.7 ± 2.7 Ma ($n = 26$, MSWD_{equiv.} = 0.85, prob_{equiv.} = 0.77, MSWD_{conc.} = 5.2, prob_{conc.} = 0.023), which is in good agreement with the accepted age.

Data reduction and interpretation was conducted using Lolite (Paton, Hellstrom, Paul, Woodhead, & Hergt, 2011), Isoplot v4.15 (Ludwig, 2003), and VizualAge (Petrus & Kamber, 2012). The time resolved signal for each ablation period was inspected for anomalous isotope ratios and trace element concentrations, which likely correspond to inclusions, cracks, disturbed/discordant zones, or multiple growth zones, were excluded from the selection and that the reported data is representative of a coherent part of the analysed zircon crystal. Further quality control of the data in Excel included the exclusion of analyses with anomalously high P, Ti, and La contents, defined as greater than the third quartile range for zircon crystals from granitoids (Belousova, Griffin, O'Reilly, & Fisher, 2002). Mean weighted ages were only calculated from dates that were doubly concordant within 2SE (i.e. where three or more of the geochronometers were indistinguishable within the analytical precision). All age uncertainties reported herein are presented as propagated 2SE, with mean weighted age errors presented at the 95% confidence level. Error propagation inflates the age uncertainty of an individual unknown zircon by accounting for any excess uncertainties within the primary zircon standard population (Paton et al., 2011).

Table S1. LA-ICP-MS instrumental conditions and analysis metadata

Laboratory and sample preparation	
Lab name	Central Analytical Research Facility, Institute for Future Environments, Queensland University of Technology
Mineral	Zircon
Sample preparation	Crushed and sieved to <455 μm , RP4 'Wilfley' table, Frantz Isodynamic magnetic separator (1 A, 25° pitch, 20° roll), methylene iodide, 25 mm resin mount, 1 μm polish
Imaging	Zeiss Sigma SEM (VPSE-G3 detector, 15kV beam current, 0% gain, 60 μm aperture, ~10 mm WD) with carbon coating
Laser ablation system	
Make, Model and type	ESI New Wave Research NWR 193
Ablation cell and volume	Trueline Mk 2, non-cantilevered small-volume ablation cell
Laser wavelength	193 nm
Pulse width	5 \pm 1 ns
Fluence	2 J cm ⁻² at laser exit with approximately 70% degradation through lenses
Repetition rate	8 Hz
Ablation duration	30 s
Spot diameter	30 μm
Sampling mode	Static spot ablation
Carrier gas flow	1.0 L min ⁻¹ Ar
Cell carrier gas flow	0.6 L min ⁻¹ He
ICP-MS Instrument	
Make, Model and type	Agilent 8800 ICPMS
Sample introduction	He (+ ablated material) and Ar (carrier gas) mix via gas smoother directly into torch
RF power (W)	1300 W
Make-up gas flow	None
Detection system	Discrete dynode electron multiplier
Masses measured	29, 31, 49, 91, 93, 139, 140, 146, 153, 163, 175, 177, 181, 206, 207, 208, 232, 238
Integration time per peak/dwell times (ms)	10 ms for isotopes except Ti (25 ms), and U–Th–Pb (30 ms)
Total integration time per output data point	0.3556 s (including "dead time")
'Sensitivity' as useful yield (% , element)	(<i>N.B. this should represent the time resolution of the data</i>) NIST 610 mean ²³⁸ U counts (background corrected) = 12500 cps for 462 ppm U (27.1 counts/ppm)
IC Dead time (ns)	Detector dead time 31 ns; overhead on mass switching is ~17%
Data Processing	
Gas blank	25 s with shutter closed and laser firing
Calibration strategy	TEM 2 used as primary reference material, Plešovice and R33 used as monitor reference materials. NIST 610 used as primary concentration reference material. Lab procedure consisted of 10 unknown zircon analyses bracketed between 4 reference material analyses.
Reference Material info	TEM 2 (Black et al., 2004) Plešovice (Sláma et al., 2008) R33 (Black et al., 2004) NIST 610 (Pearce et al., 1997)
Data processing package used / Correction for LIEF	lolyte (Paton et al., 2011) for instrumental drift, data normalisation, downhole fractionation correction, and age calculation.
Mass discrimination	Correction of measured reference material value to the certified reference material values
Common-Pb correction	No common-Pb correction applied to the data.

Uncertainty level and propagation	Ages are quoted at 2SE absolute, propagation is by quadratic addition. Reproducibility and age uncertainty of reference material are propagated where appropriate.
Quality control / Validation	Plešovice (331.0 ± 3.6 Ma, 95% conf., $n_{\text{conc}} = 23/26$, MSWD = 0.56, prob. = 0.95) R33 (425.3 ± 7.2 Ma, 95% conf., $n_{\text{conc}} = 12/27$, MSWD = 0.52, prob. = 0.89) Systematic uncertainty for propagation is 2% (2s). Time resolved signal validation in <i>Iolite</i> (Paton et al., 2011) with <i>VizualAge</i> (Petrus & Kamber, 2012).
Other information	Signal smoothing accomplished by one small expansion volume and 3 m of transfer tubing. This “triple quad” system was run in “single quad” mode with no reaction gas.

⁴⁰Ar/³⁹Ar thermochronology

Four samples of hypabyssal igneous rocks (PE1503, PE1508, PE1510, PE1519a) were crushed and passed through a Frantz Magnetic Barrier Separator to obtain feldspar concentrates, followed by hand-picking of the most pristine single crystals. The samples were loaded into a 21-pit Aluminium disk along with the neutron fluence monitor Fish Canyon Sanidine (age 28.201 ± 0.046 Ma; Kuiper et al. 2008), following the geometry illustrated in Vasconcelos et al. (2002). The irradiation disks were closed with aluminium covers, wrapped in aluminium foil and vacuum heat sealed into quartz vials. All samples were irradiated for 14 hours over the period 2 December 2016 to 5 December 2016 in the Cadmium-lined B-1 CLICIT facility, a TRIGA-type reactor, Oregon State University, USA. All ages are reported using the decay constants of Steiger and Jäger (1977).

Following a decay period after irradiation, samples were analysed by laser ⁴⁰Ar/³⁹Ar heating following procedures detailed in Vasconcelos et al. (2002). Before analysis, the samples and fluence monitors were baked-out under vacuum at ~200 °C for ca. 12 hours. Each sample was heated incrementally with a continuous-wave Ar-ion laser with a 2-mm wide defocused beam. The fraction of gas released was cleaned through a cryocooled cold-trap ($T = -125$ °C) and two C-50 SAES Zr–V–Fe getters and analysed for Ar isotopes in a MAP215-50 mass spectrometer equipped with a third C-50 SAES Zr–V–Fe getter. Full system blanks and air pipettes were determined before and after each sample. Automation and analytical procedures followed are described in Deino and Potts (1990), and Vasconcelos et al. (2002). The data were corrected for mass discrimination, nucleogenic interferences, and atmospheric contamination following the procedures in Vasconcelos et al. (2002), using the software “MassSpec Version 8.133” developed by Alan Deino of the Berkeley Geochronology Centre, USA. A ⁴⁰Ar/³⁶Ar value of 298.56 ± 0.31 Ma for atmospheric argon was used for the calculation of the mass spectrometer discrimination (Renne et al., 2009). The mass spectrometer sensitivity was calculated based on the analysis of an air pipette (1.634×10^{-13} moles ⁴⁰Ar) on the Faraday detector (4.257 mV) equipped with a 1×10^{11} Ohms resistor, yielding a Faraday sensitivity of 3.84×10^{-9} moles/nA. The current multiplier sensitivity measured on a Balzers 217 Electron Multiplier, operated with a gain of ~145 000 is $\sim 4.5 \times 10^{-14}$ moles/nA.

J-factors for each Al-disk were determined by the laser total fusion analyses of 15 individual aliquots of neutron fluence monitor, each aliquot consisting of one to three crystals of Fish Canyon sanidine. The 15 aliquots were distributed into five pits (one central, four peripheral) in the Al-disk as shown in Vasconcelos et al. (2002). The individual J for each aliquot was calculated by the Mass Spec program (Vasconcelos et al. 2002) based on the nominated age used for the Fish Canyon sanidine fluence monitor. A single J for each pit was calculated using the arithmetic mean of the three calculated J values from the three aliquots in each pit. The J for the Al-disk is the arithmetic mean of the five individual pit J values. The calculated error of the J of the Al-disk is the standard deviation of the five pit J values.

Two incremental heating spectra for the reference material Mt Dromedary GA-1550 biotite crystals yield reproducible spectra that define plateaus with dates of 99.13 ± 0.71 and 98.83 ± 0.19 Ma, both results indistinguishable from the published age (McDougall & Wellman, 2011). The recovery of correct date for this independent standard indicates that our reported $^{40}\text{Ar}/^{39}\text{Ar}$ dates are robust.

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Table S2. Geochemical data.

Sample #	P84899	P84901	P84912	P84908	P84900	P84906
SiO₂ (%)	50.16	49.34	49.14	65.72	64.1	55.44
TiO₂	1.72	1.41	1.59	0.04	0.25	0.32
Al₂O₃	14.76	15.92	15.89	19.22	18.82	18.73
Fe₂O₃T	11.24	9.58	10.18	1.19	2.76	5.84
MnO	0.21	0.15	0.17	0.12	0.05	0.18
MgO	6.96	6.62	5.98	0.41	1.48	4.56
CaO	8.80	10.71	10.17	0.33	4.34	5.29
Na₂O	3.88	2.88	2.86	7.57	5.08	2.06
K₂O	0.18	0.66	0.88	4.97	1.96	2.40
P₂O₅	0.20	0.15	0.17	0.12	0.12	0.10
LOI	2.20	2.13	2.29	0.44	1.55	4.60
Total	100.31	99.55	99.32	100.13	100.51	99.52
As (ppm)	bd	3	1	1	2	12
Ba	93.6	52.1	77.4	1230	291	465
Ce	12.9	11.5	7.30	32.6	17.1	14.8
Cr	260	298	264	bd	5	94
Cs	0.44	0.27	0.29	0.81	1.45	1.92
Cu	46	68	39	bd	5	9
Dy	7.10	5.31	6.18	3.07	1.11	1.49
Er	4.41	3.34	3.87	1.91	0.66	1.00
Eu	1.65	1.38	1.44	0.39	0.63	0.55
Ga	19.8	16.6	18.6	22.9	19.7	20.4
Gd	6.55	4.85	5.57	3.04	1.60	1.61
Hf	3.7	2.6	3.1	2.9	2.8	1.9
Ho	1.50	1.11	1.29	0.61	0.24	0.32
La	3.20	2.00	2.45	17.25	8.15	2.85
Lu	0.62	0.46	0.54	0.31	0.12	0.17
Nb	3.75	3.20	3.35	10.0	1.75	1.55
Nd	15.0	10.7	12.6	14.7	9.1	7.1
Ni	53	56	57	4	2	36
Pb	5	3	4	21	7	25
Pr	2.83	1.92	2.37	4.17	2.24	1.64
Rb	5.05	13.4	18.6	75.5	49.8	82.9
Sc	34	40	39	3	3	24
Sm	4.62	3.32	3.85	2.92	1.73	1.45
Sr	198	162	151	71.9	370	258
Ta	0.3	0.2	0.2	1.2	0.1	0.1
Tb	1.06	0.80	0.94	0.50	0.21	0.25
Th	0.33	0.13	0.82	10.15	1.665	1.475
Tm	0.61	0.47	0.56	0.29	0.10	0.16
U	1.12	0.05	0.11	2.77	0.27	0.71
V	296	260	281	1	41	176
Y	41.5	30.6	36.2	20.7	5.85	9.50
Yb	4.07	2.99	3.46	2.02	0.67	1.05
Zn	89	74	82	48	36	106
Zr	146	100	122	65	100	72

bd, below detection

