

Ion imaging of ancient zircon

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Supplementary Information

The Supplementary Information includes:

- Analytical Methods
- Tables S-1 and S-2
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- Supplementary Information References

Analytical methods

SIMS U–Pb

Sample AC13 was collected by Stephen Moorbath from the University of Oxford in July 1995, from 500 metres NNW of the Acasta camp. Zircon crystals from sample AC13 were analysed for U–Th–Pb isotopes using the SHRIMP II ion probe at Curtin University following standard operating procedures (Wingate and Kirkland, 2014). The zircon surface was sputtered with a primary, mass-filtered (O_2^-) beam with ~ 2 nA current, focused to a ~ 15 μm spot. The mass resolution, $M/\Delta M$, was better than 5000. Twenty-two analyses of the 91500 zircon reference material (Wiedenbeck *et al.*, 1995) were obtained during the session, all of which indicate an external spot-to-spot (reproducibility) uncertainty of 1.33% (1σ) and a $^{238}\text{U}/^{206}\text{Pb}^*$ calibration uncertainty of 0.45% (1σ). These calibration uncertainties are included in the calculated uncertainties on $^{238}\text{U}/^{206}\text{Pb}^*$ ratios and dates listed in Table S1. The OG1 zircon reference material was analysed as an unknown and yielded a weighted mean $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ age of 3458 ± 7 Ma (MSWD = 0.51, $n = 5$), within accepted values (Stern *et al.*, 2009). No fractionation correction on $^{207}\text{Pb}/^{206}\text{Pb}$ was deemed necessary. Common-Pb corrections were applied to all analyses using contemporaneous common Pb determined according to the model of Stacey and Kramers (1975) based on ^{204}Pb counts. The Excel-based program Squid 2 (Ludwig, 2001) was used for data processing and data were plotted using Isoplot (Ludwig, 2003).

SIMS ion imaging

Ion imaging to map the U and Pb isotopic composition of zircon was performed with a Cameca 1280 ion microprobe equipped with a high-brightness Oregon Physics Hyperion-II RF-plasma oxygen ion source at the University of Western Australia. Prior to analysis, the sample mount was cleaned with ethanol, dried, and a ~ 10 nm gold coating applied. A mass filtered $^{16}\text{O}^-$ 500 pA primary ion beam was focussed in Gaussian mode and rastered over a 50×50 μm area. A larger 65×65 μm area was pre-sputtered for 15 minutes to remove gold coating and limit the effects of surface contamination. This procedure was followed by automatic centring of the secondary ion beam in the 3000 μm field aperture and optimisation of mass calibration, performed

automatically for each run using the $^{90}\text{Zr}_2^{16}\text{O}^+$ species at a nominal mass of 196. During analysis, the sample chamber was flooded with oxygen to enhance Pb ion yields (Schuhmacher *et al.*, 1994). The secondary ions of $^{177}\text{Hf}^{16}\text{O}$, $^{90}\text{Zr}_2^{16}\text{O}$, ^{204}Pb , ^{206}Pb , ^{207}Pb , ^{208}Pb , background, ^{238}U , $^{232}\text{Th}^{16}\text{O}$, $^{238}\text{U}^{16}\text{O}$ and $^{238}\text{U}^{16}\text{O}_2$ were measured utilising a mass-skipping routine. All masses were measured on the H2 detector operating at a mass resolving power of ~ 4800 , except for $^{177}\text{Hf}^{16}\text{O}$ and $^{90}\text{Zr}_2^{16}\text{O}$ that were measured on the L1 and H2 detectors, respectively, and Pb isotopes that were measured on L1 (^{204}Pb), C (^{206}Pb), H1 (^{207}Pb) and H2 (^{208}Pb). Images (resolution 512 x 512) were integrated over 100 planes, resulting in a total measurement time of 1 hour 14 minutes per image. The Cameca WinImage2 software was used to process the images (in plane accumulation mode) and to extract U–Pb ratios along profiles of interest via a 5 by 5 μm area moving along the profile line. Profile lines were normalised to counts along a line in the same orientation on the 91500 reference zircon (Wiedenbeck *et al.*, 1995). Common Pb correction was applied to all ratios using measured ^{204}Pb counts and the terrestrial Pb evolution model of Stacy and Kramers (1975). Apparent dates across transects are given in Table S2.

Electron backscatter diffraction microstructural and cathodoluminescence imaging

CL imaging and EBSD mapping was conducted using a Tescan Mira3 field emission scanning electron microscope with an Oxford Instruments AZtec/Nordlys EBSD+EDX acquisition system in the John de Laeter Centre, Curtin University. Panchromatic CL images were collected on untilted samples using 10 kV acceleration voltage, whereas EBSD data were collected at a 70° tilt, 20 kV acceleration voltage, and 1.5 nA beam current. EBSD data were processed and visualised using AZtecCrystal, including a ‘wildspike’ correction to remove isolated erroneous pixels. Individual zircon grains were mapped using the match unit cell parameters of Hazen and Finger, (1979).

Supplementary Tables

Table S-1 Supplementary U–Pb data table (.xlsx)

Table S-2 Supplementary ion imaging traverse data table (.xlsx)

Table S-1 and S-2 (.xlsx) are available for download from the online version of this article at <https://doi.org/10.7185/geochemlet.2332>



Supplementary Figures

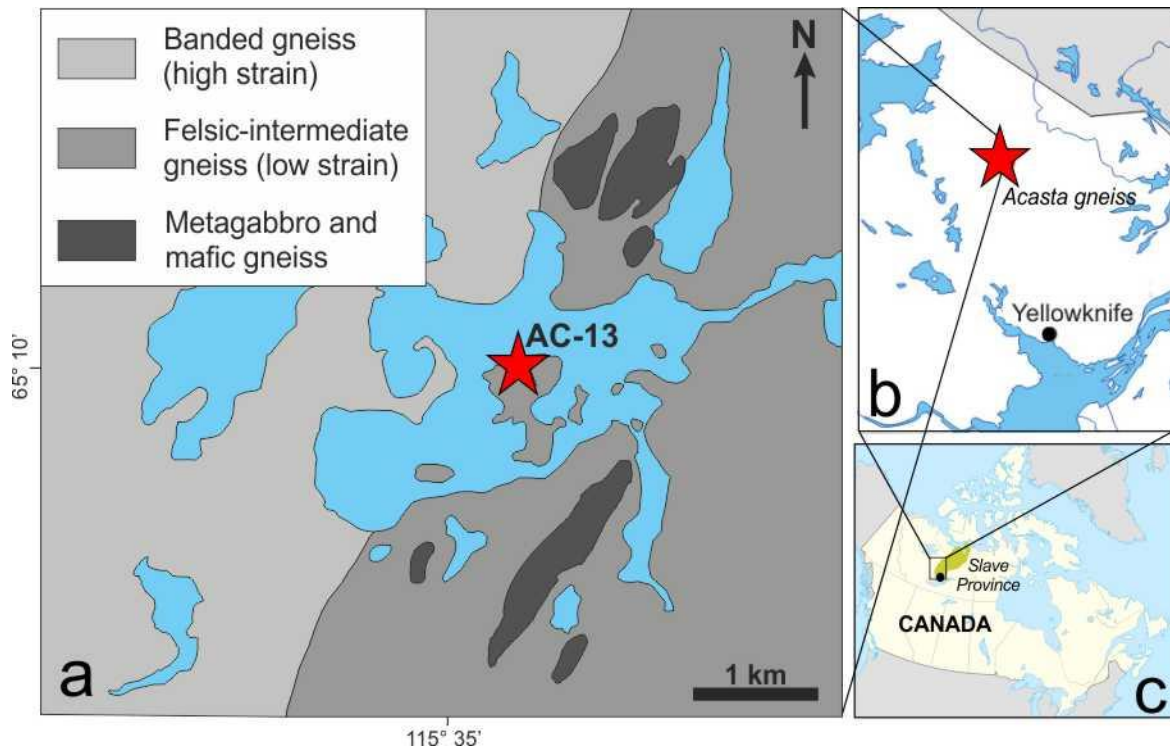


Figure S-1 Maps of the Acasta Gneiss Complex region and wider surrounds. **(a)** Sketch map of the Acasta Gneiss Complex. The red star marks the location of the investigated sample. **(b)** Regional map of the Northwest territories, Canada, with the AGC shown as a red star. **(c)** Map of Canada showing the location of the Slave Province.

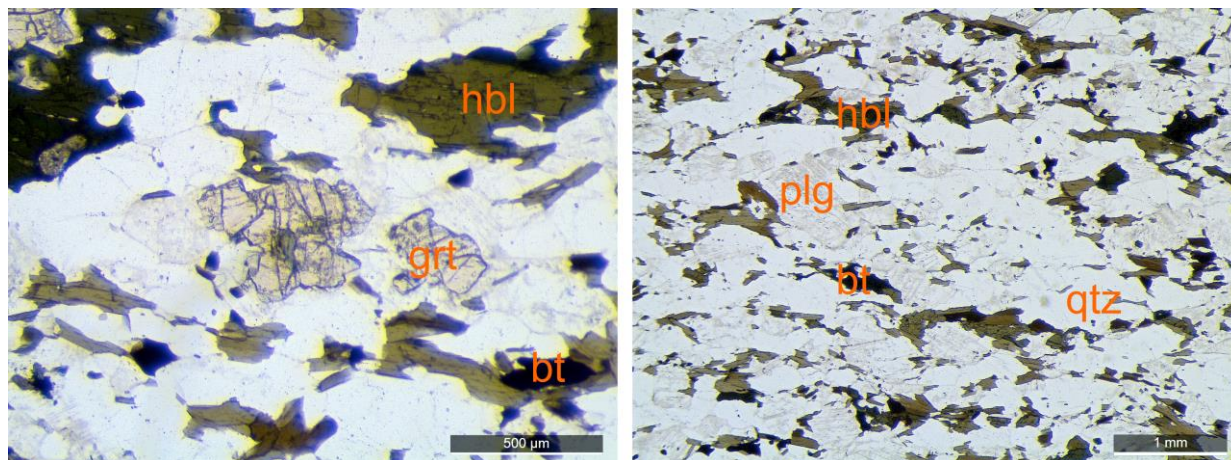


Figure S-2 Transmitted light photomicrographs of sample AC13 Idiwhaa gneiss, Acasta Gneiss Complex. Quartz (qtz), plagioclase (plg), biotite (bt), hornblende (hbl), and garnet (grt). Scale bars shown.

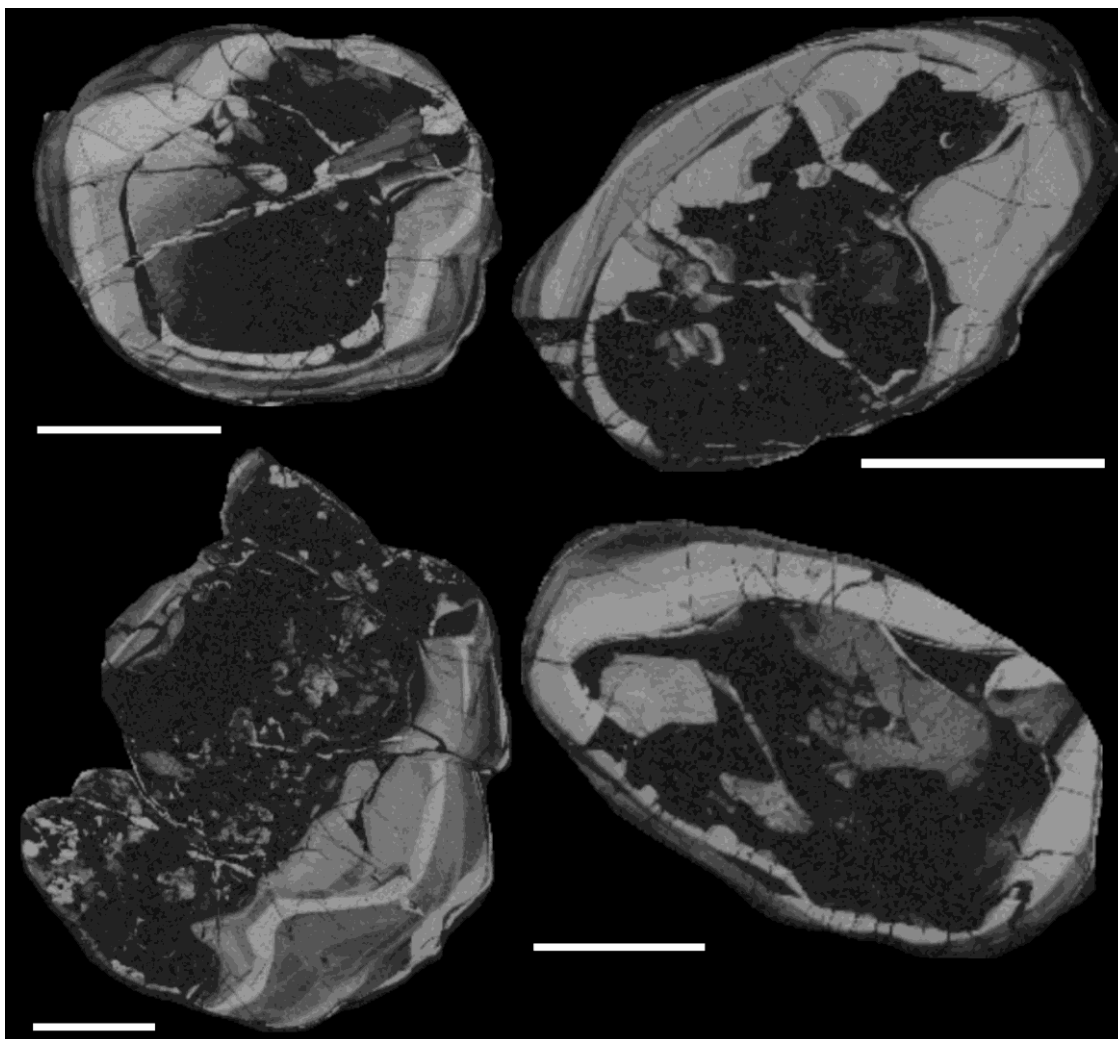


Figure S-3 Electron Backscatter Diffraction (EBSD) images of zircon grains. Representative EBSD band contrast images from zircon grains extracted from sample AC13, Idiwhaa gneiss, Acasta Gneiss Complex. Scale bar for each image is 50 μm .

Grain 53

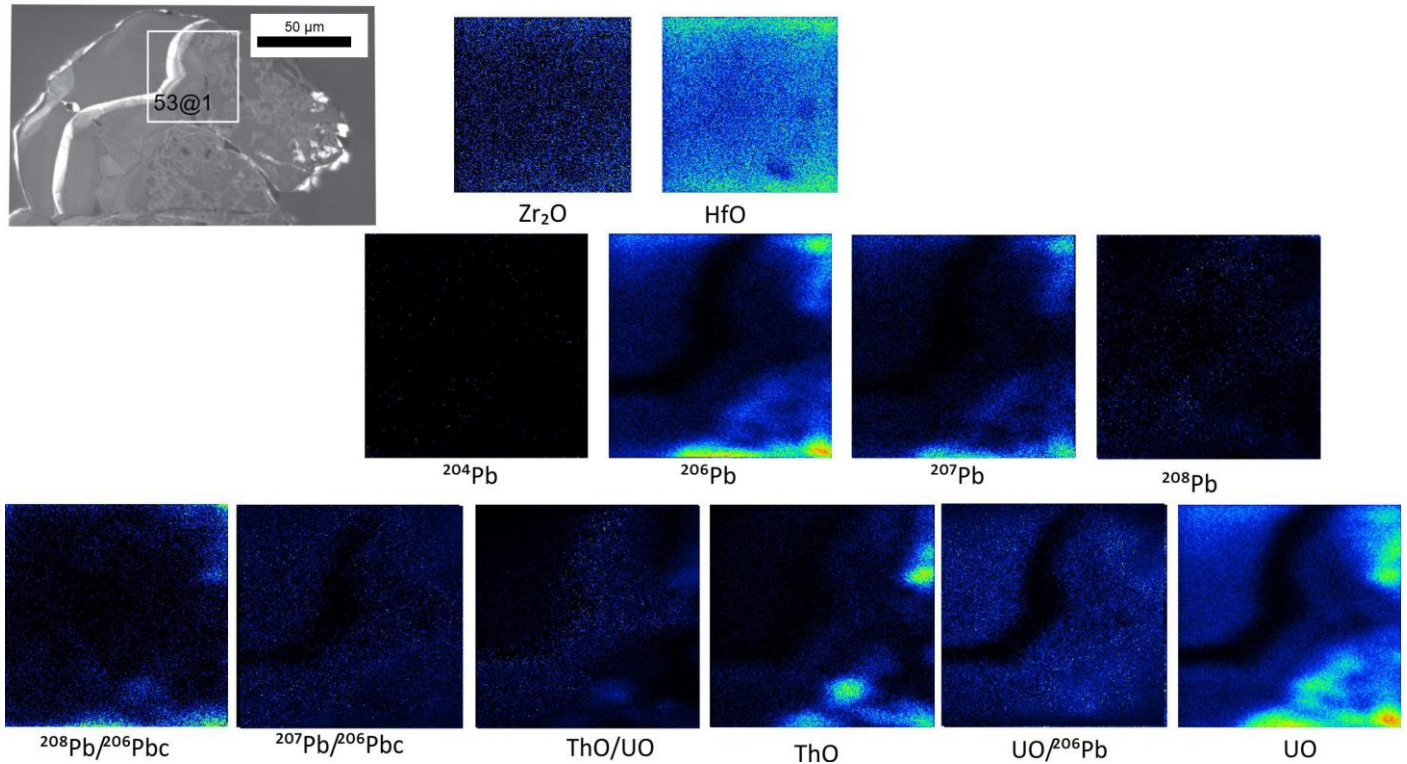


Figure S-4 Ion images of zircon Grain 53. Intensity of colour reflects cps for the indicated ratio, oxide, or isotope. Inset on the top left shows the location of the ion images on a reference cathodoluminescence image of the grain. A scale bar is provided in the cathodoluminescence image. All ion images are 50 x 50 μm.

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