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Ion imaging of ancient zircon

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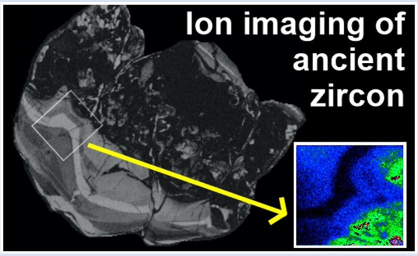
Abstract



The Idiwhaa gneiss, part of the Acasta Gneiss Complex, Canada, is a key source of information concerning formation of continental crust on the early Earth. However, zircon crystals from this oldest dated felsic crust were affected by multiple stages of alteration and metamorphism, leading to difficulties in disentangling primary from secondary processes. These grains provide an opportunity to understand the alteration processes that affect ancient zircon crystals. Ion imaging reveals pervasive recrystallisation fronts extending inwards from the margins of grains. Ahead of these recrystallisation fronts, grain cores contain isolated pockets of amorphous, but concordant, 3.99 Ga zircon that evidently escaped post-magmatic modification of U and Pb. The transport of these elements, involving the decoupling of parent and daughter isotopes, is highly heterogeneous over space and time within metamict zircon, yet localised domains still retain primary age information. Our data indicate that metamictisation of zircon alone does not lead to radiogenic Pb loss, which requires interaction with fluid.

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Introduction

The rarity of unaltered igneous rocks that formed earlier than three billion years ago (>3 Ga) leads to considerable uncertainty in tracking the development of nascent continental crust and in understanding the establishment of, and recycling between, long-lived geochemical reservoirs that ultimately maintained life (Ward and Brownlee, 2000; Willbold *et al.*, 2015). In our quest to better understand the early Earth (Hadean to Eoarchean; ≥3.6 Ga), analysis of the date, trace element, and isotopic composition of zircon crystals has been fundamental (Harley and Kelly, 2007; Valley *et al.*, 2015; Trail *et al.*, 2016). Although U–Pb isotopic ratios in ancient zircon grains are potentially easier to measure due to protracted radiogenic Pb ingrowth, such grains frequently show evidence for radiation damage that variably may disturb their primary crystal chemical and isotopic compositions (Pidgeon *et al.*, 2017). Radiation damage generates pathways for fluids whose passage through the crystal may modify the composition of zircon by removing Pb and/or facilitating uptake of other non-formula elements (Nasdala, 1998). However, the effects of radiogenic Pb loss can be difficult to disentangle from other secondary processes, including recrystallisation, diffusion, and growth of new zircon, all of which may occur in response to tectonothermal disturbance and/or fluid ingress. The effects of these secondary processes can alter the primary chemical composition of zircon and are mostly cumulative such that their effects increase with age.

The Acasta Gneiss Complex (AGC) in Northwest Territories, Canada (Supplementary Fig. S-1), contains the oldest known evolved rocks on Earth, the tonalitic Idiwhaa gneiss, which contains zircon grains preserving U–Pb crystallisation ages as old as *ca.* 4.03 Ga (Stern and Bleeker, 1998; Bowring and Williams, 1999; Reimink *et al.*, 2014, 2016). However, the rocks preserve evidence for a complex Pb-loss history such that the primary processes involved in their formation are difficult to disentangle (Moorbath *et al.*, 1997; Reimink *et al.*, 2014, 2016; Kirkland *et al.*, 2020). Here, we investigate a sample (AC13) of the Idiwhaa gneiss using a multi-technique approach combining optical, secondary electron, electron-backscattered diffraction (EBSD), cathodoluminescence (CL) imaging, and secondary ionisation mass spectrometry spot analyses of zircon grains with detailed ion imaging (mapping) of selected grains. Previous ion imaging studies of ancient zircon elsewhere have implied variable intra-crystalline radiogenic Pb mobility (Kusiak *et al.*, 2013; Ge *et al.*, 2018, 2019). Data from the Idiwhaa gneiss provide insight into the processes of zircon growth and modification of Earth's oldest known continental crust.

Sample and Method

Sample AC13 contains mainly quartz and plagioclase, with less biotite, hornblende, and garnet, and accessory magnetite, ilmenite, apatite, and zircon. The rock is a banded gneiss with felsic (quartzofeldspathic) and more mafic layers, the latter dominated by hornblende, with minor garnet, and biotite, that define a foliation. The matrix comprises subequal proportions of quartz and plagioclase interspersed with finer-grained biotite. Limited greenschist-facies alteration is evident through sericitisation of plagioclase and partial chloritisation of biotite along cleavage planes (Supplementary Fig. S-2).

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Zircon crystals from sample AC13 were analysed for U–Pb isotopes using the SHRIMP II ion probe at Curtin University. Following SHRIMP analysis, ion imaging of zircon was performed on a Cameca 1280 ion microprobe at the University of Western Australia. The detailed analytical procedures are described in the Supplemental Information. The U–Pb data table and apparent dates based on ion mapping are given in Supplementary Data Tables S-1 and S-2, respectively. All uncertainties within the text are quoted at the 2σ level.

Zircon U–Pb Date and Internal Textures

Zircon grains from sample AC13 are subhedral, have moderate length-to-width ratios and are brown under transmitted light. Based on cathodoluminescence (CL) images, their internal features can be simplified into three distinctive textural components (CL types 1–3) (Fig. 1). Highly-metamict cores (CL type 1) generally show low response and mottled CL emissions, contain abundant inclusions, and are commonly traversed by high CL response fractures. Electron backscattered diffraction (EBSD) analysis reveals the cores to be largely composed of low-crystallinity zircon, with patchy areas of more crystalline zircon within the mottled CL texture domains. The cores are bordered by discontinuous zones (CL type 2) up to 30 μm thick that parallel feint ('ghost') oscillatory zoning interpreted to reflect primary magmatic growth (Geisler *et al.*, 2007; Harley *et al.*, 2007). Analysis by EBSD shows that CL type 2 zircon is highly crystalline, typically comprising a low CL response inner band with indistinct, broad zoning that transitions to an outer band with a discrete high CL response. The internal texture of CL type 2 reveals inward-facing cuspate textures at sites where fractures are now centred (Fig. 1). Domains of CL type 2 are surrounded by more homogeneous low CL response rims (CL type 3) that form the outermost edge of the grains. The inner edge of CL type 2 has a convoluted margin against CL type 1, but a more regular contact against CL type 3 rims (Fig. 1).

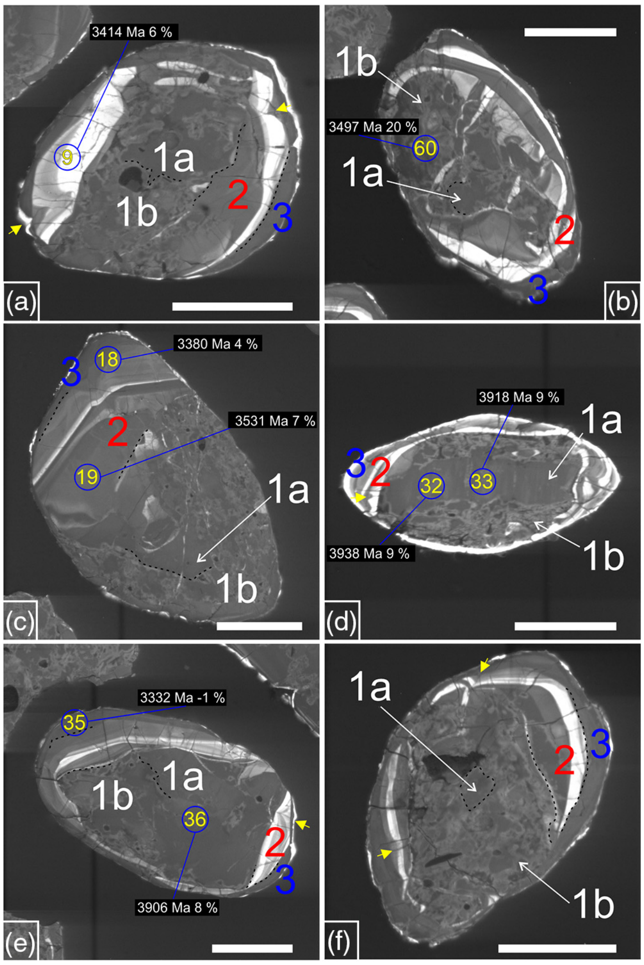

Figure 1 (a–f) Representative CL images of AC13 zircon. U–Pb spots are blue ellipses. Numbers indicate the CL types 1–3 (white, red, and blue font). Yellow arrows indicate cuspate zonation centred on fractures. $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates and discordance % in white font. Scale bars are 100 μm.

Eighty-six U–Pb SIMS spot analyses were obtained from 44 zircon grains. Results are listed in Supplementary Data Table S-1 and illustrated in Figure 2. The analyses are concordant to strongly discordant and scatter away from several apparently concordant Hadean to Archean components. Sixty-five analyses targeting a range of internal textures within the CL type 1 cores, characterised by mottled and patchy CL zonation, are >5 % discordant (Group D). The most discordant of these analyses are sited within inclusion-rich metamict zircon. Based on internal textures and U–Pb systematics, zircon grains within 5 % of concordia can be grouped into at least three components. Group 1a comprises three analyses on the core of a single grain with homogenous CL type 1 textures and low crystallinity (Supplementary Data Table S-1), data from which yield a weighted mean $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ date of 3990 ± 2 Ma (MSWD = 0.4). Ten analyses on more heterogeneously-textured CL type 1 core domains (Group 1b) have $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates ranging from 3973–3780 Ma. Group 2 comprises four analyses targeting CL type 2 transgressive veins and fronts that yield $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates of 3520–3380 Ma. A single analysis (Group 3) on a homogeneous CL type 3 rim yields a $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ date of 3332 ± 12 Ma.

Ion and EBSD imaging of two zircon grains that preserve the various CL textures discussed above was undertaken (Supplementary Fig. S-3). Grain X comprises an amorphous core with $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ date of 3990 Ma (Group 1a) and is transgressed by high-CL response, low-U veins associated with $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates of 3973–3780 Ma (Group 1b). The edge domain of this grain (CL type 2) has a more homogeneous CL response and higher EBSD band contrast due to a higher degree of crystallinity (Fig. 3).

Grain 53 has many of the same features as Grain X. It comprises a CL type 1 core with low CL-response, and high U and Th concentrations that transition into a broad CL type 2 domain with lower U and Th, centred on a high CL response front (Fig. 3; Supplementary Fig. S-4). An ion imaging apparent date profile through the rim into the core reveals a CL type 3 edge domain (step 0–14 μm) with concordant to normally-discordant apparent $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates as old as 3895 Ma, decreasing towards *ca.* 1800 Ma at the extreme edge of the profile (Fig. 4). Moving inwards through the crystal, a CL type 2 zone with highly-variable apparent dates corresponds to the high CL response front (step 14–33 μm). At a distinct low-U front, apparent $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ ratios generally decrease and apparent $^{238}\text{U}/^{206}\text{Pb}^*$ ratios increase to produce extreme reverse discordance, implying U loss uncoupled to the degree of Pb mobility. Apparent $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates within this reversely discordant front increase from ~3000 Ma at the rim ward edge to ~4000 Ma at the core side of the front. In the core, zircon with homogenous CL type 1a textures, higher U content, and low crystallinity has broadly concordant and less-variable $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates (step 33–46 μm) of around 4000 Ma. The mottled CL type 1b area of the core of the grain is dominated by normal discordance with apparent $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ dates as low as ~2400 Ma (Fig. 4).

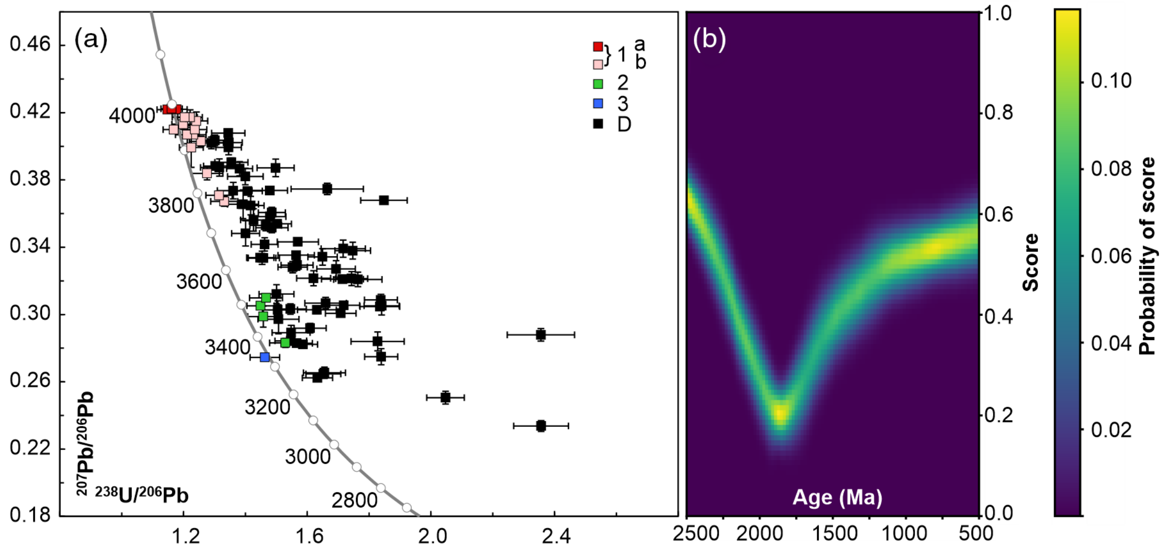

Figure 2 U–Pb geochronology of Acasta Gneiss zircon. **(a)** Inverse concordia. Colours denote interpreted CL groups. Red squares indicate Group 1a (resistive primary zircon core), pink squares indicate Group 1b (altered zircon core), green squares indicate Group 2 (analyses including younger zircon components), blue square indicates Group 3 (younger zircon overgrowth), black squares indicate Group D (>5 % discordant). Uncertainty shown at the two-sigma level. **(b)** Kolmogorov–Smirnov distance score between modelled upper intercept date for discordant *versus* concordant zircon population (within two sigma of concordia), calculated across all possible radiogenic-Pb loss times (see Kirkland *et al.*, 2020). The lowest distance score indicates greatest similarity in date structure and most likely time of Pb mobility. Colour scale is the probability estimate for KS score from bootstrapping.

Primary age Signature

An important question in the interpretation of the U–Pb geochronology of the studied zircon crystals from Acasta is the degree of secondary (post-magmatic) modification of primary isotopic ratios. Specifically, whether the various (near) concordant Eoarchean to Mesoarchean dates reflect new zircon growth or variable ancient radiogenic-Pb loss.

The oldest dates in this study comprise three concordant analyses of the homogeneous core domain (Group 1a) of Grain X, which yield a weighted mean date of 3990 ± 2 Ma. Under CL, this grain reveals a homogeneous rim with high-CL-response front (CL type 2) and a low-CL-response core (CL type 1). The core contains both homogeneous domains (CL type 1a) and mottled domains with reticulated alteration patterns (CL type 1b). The extremely low EBSD band-contrast response of this core suggests a low degree of crystallinity. Notwithstanding, homogeneous sites within the core preserve concordant U–Pb systematics (CL type 1a).

The concordant core analyses have high U (>891 ppm) and Th (>737 ppm) contents and, based on alpha dose calculations (Murakami *et al.*, 1991), are predicted to be in a highly metamict state (with 23.6–28.6 x 10^{15} alpha events assuming no post-crystallisation annealing). However, at 3520–3380 Ma, the best estimate for the time of their recrystallisation some 500 Ma after their magmatic growth (CL type 2; Fig. 2), calculations indicate that this core would have still been crystalline (1.8 x 10^{15} alpha events). Despite the extreme accumulated radiation damage implied by the EBSD data, the core of Grain X has concordant U–Pb systematics. We interpret the apparent lack of radiogenic-Pb loss in this core to indicate that it did not interact with secondary fluids, either at the time of recrystallisation or later. These observations are consistent with the concept that diffusive transport of U and Pb in zircon requires both a diffusion network and the presence of fluids (Pidgeon *et al.*, 1966; Geisler *et al.*, 2002; Herrmann *et al.*, 2021). Importantly, in isolation, radiation damage seems not to affect mobility of U or Pb.

In contrast to the concordant CL type 1a core domains that preserve Eoarchean dates, CL type 1b core domains are characterised by a mottled CL response; EBSD analysis reveals variable and highly-convoluted patterns (Fig. 3). The ion image for Grain 53 reveals a small CL type 1a core domain with consistent U/Pb* and Pb*/Pb* dates of *ca*. 4.0 Ga. This domain is in textural continuity with reticulated CL type 1b core domains that show high- to very-high normal discordance and are interpreted to have lost radiogenic Pb. Thus, small type 1a areas, as in the core of Grain X, appear to represent relict metamict core regions that remained isolated from fluids, and which consequently preserve concordant earliest Eoarchean dates.

Based on our data, the 3990 ± 2 Ma date for the three CL type 1a analyses is interpreted as the minimum crystallisation age of the magmatic protolith to the gneiss, consistent with the general distribution of data away from this point on the concordia diagram (Fig. 2). Given the evidence for multiple episodes of radiogenic-Pb mobility, the time at which radiogenic Pb loss occurred may be estimated using a Concordance–Discordance–Comparison test (Kirkland *et al.*, 2020). Application of this test indicates that the greatest similarity between the discordant and the concordant populations is achieved for a radiogenic Pb loss event at 1854 +101/−81 Ma (Fig. 2b). This age, which matches the youngest outermost concordant component of the CL type 3 domain on the ion image profile from Grain 53 (Fig. 4), is contemporaneous with the Paleoproterozoic Wopmay Orogeny that affected the western part of the Slave Province as part of the assembly of the Columbia supercontinent (Fisher *et al.*, 2020). Apatite (re) growth in the AGC has also been ascribed to this event (Antoine *et al.*, 2020; Fisher *et al.*, 2020). We interpret the U–Pb systematics to reflect a ≥3990 ± 2 Ma magmatic rock that underwent episodes of Pb mobility at 3965 Ma, 3520–3380 Ma, and *ca*. 1850 Ma within those portions of grains that had access to fluids (Iizuka *et al.*, 2007; Guitreau *et al.*, 2018; Kirkland *et al.*, 2020).

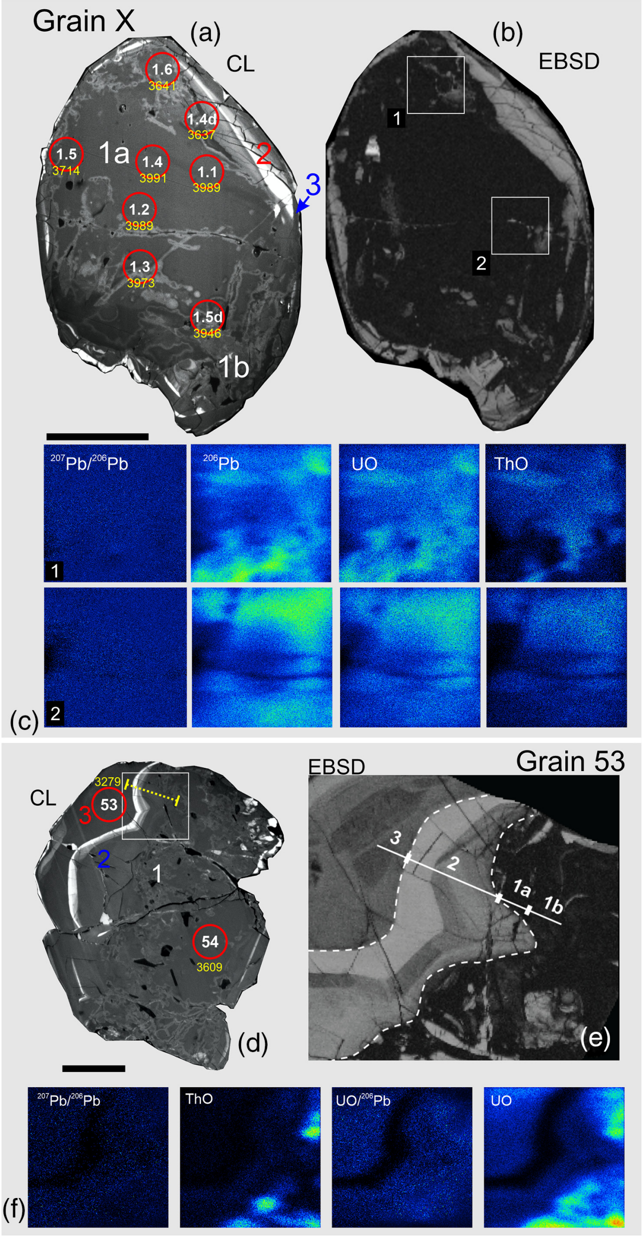

Figure 3 Images of Acasta Gneiss zircon. **(a and d)** Cathodoluminescence (CL). **(b and e)** Electron Backscatter Diffraction (EBSD) band contrast. **(c and f)** Ion images for the indicated isotopic masses and ratios. Brighter colours denote higher counts. Coloured text (1, 2, and 3) on the CL image denotes different CL types. U–Pb zircon analytical spots are shown as red circles (with $^{207}Pb^*/^{206}Pb^*$ dates in yellow font). Scale bars are 50 µm. Yellow dashed line in d is the apparent date transect shown in Fig. 4.

Recrystallisation

Ion imaging reveals a spatial variation in apparent dates within the crystalline domain (CL type 2) surrounding the core (CL type 1) in Grain 53 (Fig. 3). This region contains significant variation in composition along the axis of the recrystallisation front, with deviations to reverse discordance coupled with low-U concentrations, compatible with the loss of U to a fluid. This apparent date pattern is consistent with the low-U front representing a zone of recrystallisation, with U flushed out to the grain margin (Nasdala *et al.*, 2010; Putnis and Austrheim, 2013). The region behind (rim-ward of) the recrystallisation front (CL type 3) shows less reverse discordance. This rim region may comprise several different growth domains, with one likely age component estimated by a concordant 3332 ± 12 Ma analysis (CL type 3), requiring either expulsion of all earlier-formed radiogenic Pb or new zircon growth. Thus, we interpret the apparent date cross section to image a frozen alteration front transiting through the grain from rim to core, leaving a homogenised rind (Geisler *et al.*, 2007) onto which later zircon precipitated (*e.g.*, CL type 3).

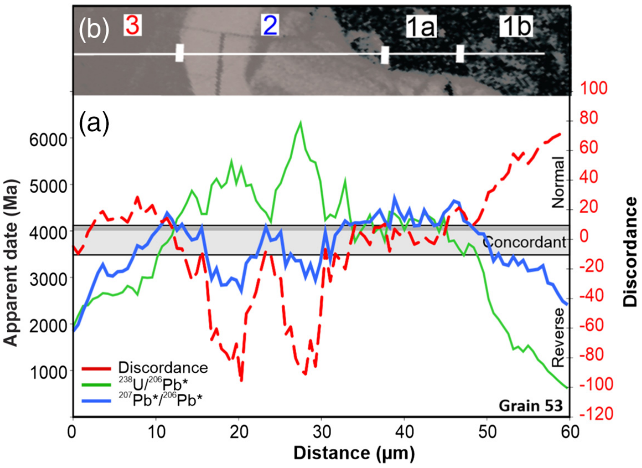

Figure 4 **(a)** Plot of apparent dates from ion image traverse. Dates calculated by moving a box (5 µm by 5 µm) along transect line shown in figure 3d. Primary age of Acasta indicated by thin dark grey box. Light grey box depicts +10 to −10 % U–Pb concordance. **(b)** Band contrast image of the zircon traverse line.

Figure 4 implies at least two distinct alteration processes: radiogenic Pb loss from metamict cores leading to normal U–Pb discordance, and U dissociation from Pb leaving a reversely discordant front. Generally, Pb is considered to be more mobile than U in zircon (Pidgeon *et al.*, 1966). However, hydrothermal experiments on zircon have demonstrated enhanced U mobility in saline solutions (Geisler *et al.*, 2003). The front shows no noticeable reduction in Zr, but a slight decrease in Hf (Supplementary Fig. S-4), supporting a dissolution–recrystallisation mechanism producing zircon with reduced trace element concentrations (Geisler *et al.*, 2007). Importantly, as this new zircon is reversely discordant, it incorporated some unsupported Pb directly from the core, decoupling parent U from radiogenic Pb (Mattinson *et al.*, 1995). This implies that metamict cores interacted with fluids, losing volume and mass to a metamorphic liquid that facilitated recrystallisation localised on the ancient grain margin.

This process has implications for U–Pb geochronology as, despite the lower U fronts in zircon being less susceptible to radiation damage, they are nonetheless discordant due to incorporation of disassociated radiogenic-Pb. Any analytical mixture with such a front and normally discordant core, dependent on the percentage of mixture, could result in an apparently concordant analysis, yet having no age significance. Importantly, ion imaging provides a way to understand the process of alteration within geochronometers, ultimately helping to isolate domains in metamict zircon that still retain primary isotopic significance.

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Additional Information

Supplementary Information accompanies this letter at https://www.geochemicalperspectivesletters.org/article2332.

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