The effect of low-temperature annealing on discordance of U–Pb zircon ages

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Discordant U–Pb data of zircon are commonly attributed to Pb loss from domains with variable degree of radiation damage that resulted from α-decay of U and Th, which often complicates the correct age interpretation of the sample. Here we present U–Pb zircon data from 23 samples of ca. 1.7–1.9 Ga granitoid rocks in and around the Siljan impact structure in central Sweden. Our results show that zircon from rocks within the structure that form an uplifted central plateau lost significantly less radiogenic Pb compared to zircon grains in rocks outside the plateau. We hypothesize that zircon in rocks within the central plateau remained crystalline through continuous annealing of crystal structure damages induced from decay of U and Th until uplifted to the surface by the impact event ca. 380 Ma ago. In contrast, zircon grains distal to the impact have accumulated radiation damage at shallow and cool conditions since at least 1.26 Ga, making them vulnerable to fluid-induced Pb-loss. Our data are consistent with studies on alpha recoil and fission tracks, showing that annealing in zircon occurs at temperatures as low as 200–250 °C. Zircon grains from these samples are texturally simple, i.e., neither xenocrysts nor metamorphic overgrowths have been observed. Therefore, the lower intercepts obtained from regression of variably discordant zircon data are more likely recording the age of fluid-assisted Pb-loss from radiation-damaged zircon at shallow levels rather than linked to regional magmatic or tectonic events.

The U–Pb decay system in zircon is the most widely used geochronometer for the determination of radiometric ages, due to its general robustness in geological systems and the direct control of closed-system behaviour through the dual decay of uranium. In addition, analytical techniques such as Secondary Ion Mass Spectrometry (SIMS) and Laser Ablation-Inductive Coupled Plasma-Mass Spectrometry (LA-ICP-MS) enables high-spatial resolution analysis and age determination of multiple discrete events from a single zircon grain e.g.1,2. Accumulation of radiogenic Pb over time is linked to the 238U-206Pb, 235U-207Pb and 232Th-208Pb decay chains, which is associated with recoil during emission of alpha particles and radiation damage e.g.3. Despite the robustness of the U–Pb system in zircon, concordant data are relatively rare even in pristine igneous rocks4. The geological cause of discordance is generally attributed to the loss of radiogenic Pb through solid-state diffusion, fluid-assisted element mobility in radiation-damaged zircon, or recrystallization of metamict domains5 (and references therein). Solid-state diffusional Pb-loss from crystalline zircon requires temperatures of at least 900–1000 °C6,7. Loss at significantly lower temperatures occurs more readily in radiation-damaged zircon, especially in the presence of fluids8–11.

Experimental studies show that radiation-damaged zircon is able to recover through annealing when the temperature is sufficiently high. Depending on the amount of radiation damage, the annealing temperature in highly damaged zircon on short timescales (minutes to days) has been estimated to 850 and 1200 °C under dry conditions12,13 which exceed those of the deep crust and silicic magmas. However, over geological time scales, structural defects, such as alpha damage (point defects and recoil tracks14) and fission tracks, anneal at significantly lower temperatures although estimates vary. Annealing of fission tracks have been suggested to occur at temperatures between 200 and 250°C15–17 while alpha damage anneals over a broader temperature range14,18. This leads to the hypothesis that Pb-loss is enhanced at shallow, relatively cool, crustal levels due to the accumulated

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effects of radiation damage and fluid-assisted element mobility, as suggested by 19. However, this hypothesis has yet to be empirically tested on natural zircon.

We have analysed the U–Pb systematics in zircon from 23 near-surface samples of Paleoproterozoic basement rocks in and around the Siljan impact structure in northern Dalarna, central Sweden (Fig. 1), using LA-ICP-MS, supported by Field-Emission Scanning Electron Microscopy (FE-SEM). The Siljan impact structure is located at the boundary between the Transscandinavian Igneous Belt (TIB) and the westernmost domain of the Svecofennian crust (Fig. 1). The structure has an estimated rim-to-rim diameter of about 52 km and a 28–30 km wide central plateau that is dominated by Järna and Siljan granite20,21 (Fig. 1). Traditionally both granite types are assigned to the 1.81–1.65 Ga-old TIB rocks22. However, a recent re-organization of the lithotectonic framework of Sweden by23 refers Järna granite to syn-Svecokarelian rocks, formerly addressed as Svecofennian crust23, whilst Siljan granite builds part of the post-Svecokarelian rocks. The central plateau is surrounded by an annular depression that is partly filled by lakes and down-faulted Paleozoic sediments24–27 (Fig. 1).

Zircon U–Pb crystallization ages of Järna and Siljan granite range between 1.9 and 1.7 Ga28–30. In northern Dalarna where the Siljan impact structure is located, dolerite dykes and sills have yielded concordant to near concordant U–Pb baddeleyite ages that fall in the 1462–1461 Ma, 1271–1264, and 978–946 Ma age intervals31. Previous K–Ar whole rock ages32 and Rb–Sr whole rock-biotite dates33 performed on the two younger generations

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**Figure 1.** Geological map modified after21 of the Siljan impact structure. Sample localities of Järna and Siljan granite inside and outside the central plateau are shown. The inset map shows the location of the Siljan impact structure in Sweden (red point). (Map modified with Adobe Illustrator version v. 25.0.1, https://www.adobe.com/products/illustrator.html?promoid=PGRQQLFS&mv=other#).
of dolerite yielded similar age estimates. These mafic rocks are pristine with no signs of post-magmatic metamorphism or fluid-induced alteration. Considering that both the Rb–Sr system and baddeleyite are sensitive to such events (e.g., in conjunction with the general low closure temperature for the isotopic K–Ar system (250–550°C), there cannot have been significant thermal heating or fluid activity in the Siljan region after at least ca. 1260 Ma until the impact event occurred which has been dated at 380.9 ± 4.6 Ma through 40Ar/39Ar on melt dikelets and melt breccias. Dolerite dykes and sills that intruded the Siljan region about 1000–900 Ma are scarce (Fig. 1) and could only have caused local heating of host rocks in the immediate contact to the intrusions. In general, the TIB plutonic rocks in Sweden are associated with abundant volcanic rocks of the same age, which in Dalarna are represented by the so-called Dala porphyries and ignimbrites (~1.8 to ~1.7 Ga, e.g.,) that are abundant immediately northwest of Siljan. The co-existence of plutonic and volcanic rocks at present-day near-surface temperatures remains unaffected in zircon from the target rocks at shock pressures up to 20 GPa e.g.,. Zircon neoblasts tend to form at sites with crystal structure defects (e.g., radiation-damaged domains from 2 to 16 GPa for the rocks inside the central plateau. In contrast, the basement rocks outside the central plateau more commonly have metamict domains (about 45% of the dated grains) with higher concentrations of non-stoichiometric elements (e.g., Na, Ca, Fe) compared to zircon grains within the central plateau where about 6% of the dated grains show textural evidence for metamictization (see Supplementary Fig. S2 and S3). Zircon and quartz from the central plateau display planar features, that are exclusively impact-generated. Planar features, indicative for recrystallization into neoblastic zircon is absent in our samples. Planar features, defined by plotting 206Pb/204Pb against the 207Pb/206Pb as a lower intercept of 206Pb/204Pb and was culled (e.g., see Supplementary Fig. S5). The lower limit of 206Pb/204Pb varies between the samples but commonly range between 10^5 and 10^4. Spot analyses that do not match the criteria were rejected from the age calculations.

Data was plotted in Wetherill concordia diagrams and linear regressions were performed of each population, revealing a range of discordant to discordant analyses (see Supplementary Fig. S4). The regression typically yielded mean square weighted deviates (MSWD) between 0.4 and 17.0. Upper intercept dates fall between 1.9 and 1.7 Ga, which corresponds with the known age range of the Siljan and Järna granites. Lower intercepts are < 617 Ma with no systematic variation between rocks from inside or outside of the central plateau (see Supplementary Fig. S6).

Discussion

The effect of the Siljan impact event on the zircon U–Pb system. It has been shown that impact events can successfully be dated by U–Pb of so-called neoblastic zircon, which preferentially occur in impact melt rocks e.g.,. Zircon neoblasts tend to form at sites with crystal structure defects (e.g., radiation-damaged domains) at temperatures between 1100 and 1200 °C. This typically results in strongly discordant data sets for which the lower intercept ideally represents the age of the impact e.g.,. Although the 380.9 ± 4.6 Ma age of the Siljan impact event falls within our lower intercept range of ~617 to 7 Ma (see Supplementary Fig. S6), textural features indicative for recrystallization into neoblastic zircon is absent in our samples. Planar features, formed at T < 900 °C and P < 20 GPa, suggest that the temperatures were insufficient to cause any resetting of the U–Pb system. As shown in previous studies, the U–Pb system through diffusional or recrystallization processes remains unaffected in zircon from the target rocks at shock pressures up to 20 GPa e.g.,.

We have divided the zircon grains into two subsets, one comprising zircon in rocks from outside the central plateau (Fig. 3a) and one comprising zircon in rocks inside the central plateau (Fig. 3b). Impact-generated hydrothermal activity that might enhance Pb-loss, especially in radiation-damaged zircon, was more prevalent...
The effect of annealing on discordance in zircon. We assess the cause and difference of the observed discordance in zircon through plotting the U concentration against the degree of discordance (Fig. 3c). In order to obtain overlapping data for the two subsets, we have screened the effective U (eU) concentration to between 200–400 ppm for the subset from outside the central plateau than inside (Fig. 3c). Number of analyses included in the calculation of 1 S.E. and 1 S.D. of the mean discordance for each eU concentration interval can be found in Table S3.

Table 1. Summary of the U–Pb data, including the upper and lower intercept dates, MSWD, n = number of analyses, coordinates of sample localities and granite type.

| Sample number | Lower intercept age (Ma) | Upper intercept age (Ma) | MSWD | n | Location | Lat (°N) | Long (°E) | Description |
|---------------|--------------------------|--------------------------|------|---|---------|----------|-----------|-------------|
| Outside central plateau | | | | | | | | |
| 12 | 210 ± 52 | 1881 ± 27 | 3.80 | 30 | 60° 54.503′ | 15° 00.197′ | Järna granite |
| 23 | 198 ± 170 | 1833 ± 29 | 7.90 | 32 | 60° 54.964′ | 14° 37.008′ | Järna granite |
| 24 | 255 ± 62 | 1804 ± 15 | 2.30 | 27 | 60° 53.896′ | 14° 38.071′ | Järna granite |
| 25 | 286 ± 180 | 1852 ± 66 | 17.00 | 17 | 60° 51.087′ | 14° 36.016′ | Siljan granite |
| Inside central plateau | | | | | | | | |
| 21 | 106 ± 350 | 1815 ± 21 | 8.70 | 23 | 60° 59.569′ | 14° 46.760′ | Siljan granite |
| 31 | 162 ± 190 | 1773 ± 33 | 3.50 | 20 | 61° 04.258′ | 14° 57.545′ | Siljan granite |
| 32 | 554 ± 1100 | 1756 ± 35 | 2.20 | 9 | 61° 05.236′ | 15° 02.643′ | Siljan granite |
| 34 | 128 ± 400 | 1747 ± 13 | 1.30 | 11 | 61° 07.016′ | 14° 59.630′ | Siljan granite |
| 36 | 617 ± 480 | 1753 ± 26 | 1.30 | 17 | 61° 07.133′ | 14° 48.314′ | Siljan granite |
| 54 | 1733 ± 19 | 2.80 | 38 | 61° 00.117′ | 14° 54.557′ | Järna granite |
| 55 | 1701 ± 17 | 0.77 | 17 | 61° 00.885′ | 14° 53.894′ | Siljan granite |
| 57 | 67 ± 360 | 1736 ± 19 | 1.40 | 18 | 60° 57.721′ | 14° 53.894′ | Järna granite |
| 58 | 1747 ± 15 | 1.70 | 18 | 60° 57.409′ | 14° 54.355′ | Järna granite |
| 59 | 1805 ± 16 | 0.92 | 8 | 60° 58.524′ | 14° 52.866′ | Järna granite |
| 61 | 11 ± 480 | 1752 ± 20 | 2.10 | 26 | 61° 01.268′ | 14° 53.411′ | Järna granite |
| 62b | 7 ± 340 | 1727 ± 5.8 | 1.09 | 21 | 60° 59.524′ | 14° 52.030′ | Järna granite |
| 64a | 65 ± 350 | 1783 ± 10 | 0.60 | 14 | 61° 04.046′ | 14° 50.075′ | Järna granite |
| 65 | 321 ± 1100 | 1815 ± 28 | 2.90 | 6 | 61° 03.671′ | 14° 50.729′ | Järna granite |
| 66 | 426 ± 280 | 1727 ± 29 | 2.10 | 8 | 61° 03.190′ | 14° 52.150′ | Järna granite |
| 67 | 1713 ± 18 | 1.20 | 12 | 61° 04.146′ | 14° 53.946′ | Järna granite |
| 68 | 1714 ± 9.4 | 1.60 | 20 | 61° 02.111′ | 14° 54.799′ | Järna granite |
| 69 | 37 ± 680 | 1726 ± 14 | 0.69 | 14 | 61° 03.051′ | 14° 53.612′ | Järna granite |
| 71 | 1714 ± 28 | 0.40 | 12 | 61° 01.604′ | 15° 02.803′ | Järna granite |
residence times might explain the observed difference in discordance between the two subsets if the temperature was sufficiently high for annealing of zircon prior to the uplift of the central plateau. A geothermal gradient of 29–32 °C/km, correspond to a temperature of 256–232 °C for 8 km uplift46, which is considerably lower than the annealing temperatures of 600–650 °C postulated by19. However, the latter temperature estimates rely on annealing of already radiation-damaged zircon during metamorphism or experiments of short duration, which are not applicable in the present context. It is known that the annealing temperature of fission tracks in zircon is estimated to between 200 and 250 °C15–17, whereas annealing of alpha damage may require slightly higher temperatures (> 250 °C14) although lower estimates have also been suggested (100–160 °C18). With respect to these annealing temperature estimates, we find a remarkably good agreement with the temperature estimates for the uplifted central part of the Siljan structure, which is in consistency with the observed difference in discordance between the two subsets. Our results suggest that annealing of alpha recoil and fission tracks in zircon from Siljan were operating at 200–250 °C, prohibiting accumulation of radiation damage in zircon and hence significant Pb-loss prior to uplift.

In general, Pb loss from radiation-damaged zircon is more efficient in the presence of fluids e.g.58,59. It is notable that rocks from the central plateau were heavily fractured and affected by hydrothermal activity due to the impact event46 (see Supplementary Fig. S1). This is in stark contrast to the rocks outside the central plateau, which were significantly less affected. Zircon from inside the central plateau should thus have been more affected by fluid interaction and Pb loss. This paradox can be explained if zircon inside the central plateau continuously underwent annealing prior to uplift (Fig. 4), which implies higher crystallinity and less vulnerability to hydrothermal alteration. We conclude that the zircon from the central plateau remained crystalline until uplifted and that surface exposure time remains the single most important factor in explaining the difference in discordance. Continuous Pb-loss near the surface will, with time, lead to excess scatter of analyses around the discordia and also explains the great variance for the subset outside the central plateau. Therefore, the lower intercepts obtained

Figure 2. Representative concordia plots for samples outside the central plateau (samples 12 = Järna granite and 25 = Siljan granite) and inside the central plateau (samples 36 = Siljan granite and 58 = Järna granite). (Plots created with with Adobe Illustrator version v. 25.0.1, https://www.adobe.com/products/illustrator.html?promoid=PGRQQLFS&mv=other#).
from regression of variably discordant zircon data are more likely recording the age of fluid-assisted Pb-loss from radiation-damaged zircon at shallow levels rather than linked to regional magmatic or tectonic events.

**Methods**

Between 45 and 60 zircon grains were separated from each sample for LA-ICP-MS. All preparation and analyses were done at the Department of Geology, Lund University, Sweden. The zircon grains were separated through crushing, sieving, water-based density separation (Willey Table), hand-picking (100–200 grains per sample) with final size fraction of 80–250 µm, before being mounted in epoxy resin and polished until the central part of each grain was exposed. The zircon crystals are translucent with pinkish or yellowish colour to colourless, and mostly of prismatic and euhedral morphology as well as some rounded grains (see Supplementary Fig. S2).

The FE-SEM imaging was done on a Tescan Mira3 High Resolution Schottky FE-SEM equipped with an Oxford energy dispersive spectrometry (EDS) and a CL system. The accelerating voltage was set to 15 kV. The working distance ranged from 5 to 15 mm. Back-scatter electron (BSE) and cathodoluminescence (CL) of polished zircon crystals was used to guide the spot selection. Primarily, areas that occur homogeneous in BSE and CL were selected.

The LA-ICP-MS (n = 1284 spots) analyses were done using a Bruker Aurora Elite ICP-MS connected to a 193 nm Cetac Analyte G2 excimer laser. The running conditions are summarized in Supplementary Table S4. The repetition rate of the laser was set to 7–8 Hz, a square spot geometry with a size from 18 × 18 µm to 20 × 20 µm was used (area fixed in each sequence), and the fluence was set to ~ 5–6 J/cm². Samples and reference materials were placed in a two volume HelEx2 sample cell flushed with helium gas which was mixed with Ar and N₂ gas before entering the torch. NIST612 was used to tune the instrument, focusing on stable signals, low oxide production (< 0.5%), Th/U around 1, and high ²³⁷⁹Pb and ²³⁸⁰U signals. GI-1⁰⁰ and 91500⁶¹ served as primary and secondary reference material, respectively. The analytical sequence was done in automatic mode with a setup starting with 6 GJ-1, followed by analysing 10 unknowns and 3 GI-1, with up to 100–120 analyses per sequence. The masses ²⁰⁴Hg, ²⁰⁵⁸(Pb, Hg), ²⁰⁶⁸Pb, ²³²⁸Th, and ²³⁸⁰U (in some of the sequences ²⁰⁴Hg was left out) were measured in dynamic mode within a single collector system. Dwell times on each mass are listed in Supplementary Table S4. Raw data reduction, including base-line subtraction, drift correction and down-hole correction, was carried out with Iolite⁶² (version 3.63). The construction of the Wetherill concordia plots and age calculations was done by Isoplot Excel Add-in⁶³.
Figure 4. Cross-sections illustrating the evolution of the Siljan area prior (a), during (b) and after (c) the impact event. (a) Illustrates the crust in the period 1900–380 Ma. The different depth of zircon outside (black grain) and inside (white grain) the central plateau is shown. The zircon outside the central plateau resided near the surface where radiation damage accumulated since at least 1260 Ma. Zircon inside the central plateau resided at ~ 8 km depth and annealed continuously. (b) The impact event caused ~ 8 km uplift of the central plateau, exposing zircon to surface conditions since 380 Ma. (c) Present situation, zircon from both out- and inside the central plateau resides at surface (figure created with Adobe Illustrator version v. 25.0.1, https://www.adobe.com/products/illustrator.html?promoid=PGRQQLFS&mv=other#).

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Acknowledgements
S. Holm-Alwmark is supported by the Swedish Research Council (VR; 2017-06388). C. Alwmark acknowledges VR (621-2012-4504) and the Crafoord Foundation (20140617). We thank the anonymous reviewers and Sven Lukas for their time and effort in providing constructive feedback. We thank Klaus Mezger, Fernando Corfu, Noah McLean, and Ashley Gumsley for reading and commenting an early version of this manuscript.

Author contributions
M.H., C.A., U.S. and A.S. initiated the study. S.H.A. provided the material for the study. M.H., U.S. and A.S. wrote the first draft. M.H. did the petrographic and SEM analyses with assistance by C.A. T.N. and M.H. did the LA-ICP-MS analyses. All authors contributed to the interpretation of the data. All authors read and contributed on the text.

Funding
Open access funding provided by Lund University.

Competing interests
The authors declare no competing interests.

Additional information
Supplementary Information The online version contains supplementary material available at https://doi.org/10.1038/s41598-021-86449-y.

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