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Age, Hf isotope and trace element signatures of detrital zircons in the Mesoproterozoic Eriksfjord sandstone, southern Greenland: are detrital zircons reliable guides to sedimentary provenance and timing of deposition?

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Abstract – The Gardar Rift in southern Greenland developed within Palaeoproterozoic rocks of the Ketilidian orogen, near its boundary with the Archaean craton. The Eriksfjord Formation was deposited at *c*. 1.3 Ga on a basement of *c*. 1.8 Ga Julianehåb I-type granite. Detrital zircons from the lower sandstone units shows a range of ages and ε_{Hf} compatible with proto sources within the Archaean craton and the Nagssugtoquidian mobile belt north and east of the craton; zircons that can be attributed to juvenile Ketilidian sources are less abundant. This suggests a predominance of distant sources, probably by recycling of older and no longer preserved cover strata. A significant fraction of *c*. 1300 Ma zircons have ε_{Hf} between 0 and −38. Rather than originating from a hitherto unknown igneous body within the Gardar Rift, these are interpreted as Palaeoproterozoic to late Archaean zircons that have lost radiogenic lead during diagenesis and post-depositional thermal alteration related to Gardar magmatism. Although the sediments originate from sources within Greenland, the age and initial Hf isotope distribution of Palaeoproterozoic and Archaean zircons mimics that of granitoids from the Fennoscandian Shield. This may reflect parallel evolution and possible long-range exchange of detritus in Proterozoic supercontinent settings. The lesson to be learned is that detrital zircon age data should not be used to constrain the age of sedimentary deposition unless the post-depositional history is well understood, and that recycling of old sediments, long-range transport and parallel evolution of different continents make detrital zircons unreliable indicators of provenance.

Keywords: Gardar Rift, Eriksfjord Formation, detrital zircon, U–Pb ages, Lu–Hf isotopes, trace elements in zircon.

1. Introduction

Zircon is a very robust mineral whose U–Pb age, Lu–Hf isotope signature and trace element distribution pattern can survive repeated cycles of erosion, sedimentary transport, diagenesis, metamorphic recrystallization and even crustal anatexis (e.g. Williams, 2001; Barr *et al*. 2003; Hawkesworth & Kemp, 2006; Røhr, Andersen & Dypvik, 2008; Kurhila, Andersen & Rämö, 2010; Røhr *et al*. 2010; Andersen *et al*. 2011). It is therefore commonly assumed that detrital zircon grains in a clastic sediment will retain a memory of the age and composition of the (generally) granitic intrusions in which the zircon originally formed, and the use of radiogenic isotope data and trace element distribution patterns from detrital zircon has become a popular tool in provenance analysis (e.g. Fedo, Sircombe & Rainbird, 2003). There is, however, also evidence that diagenetic processes in a clastic sediment may cause loss of radiogenic lead from zircon (Willner *et al*. 2003), but it is unlikely that this will also affect the more robust Lu–Hf isotope system (e.g. Amelin, Lee & Halliday, 2000). Isotope data from detrital zircons have been used successfully in studies of first-order global processes such as the extraction, growth and preservation of continental crust (e.g. Belousova *et al*. 2010; Condie *et al*. 2011; Voice, Kowalewski & Eriksson, 2011; Lancaster *et al*. 2011), as well as in regional studies where the purpose is to identify the source of material in a given sedimentary sequence (e.g. Lahtinen, Huhma & Kousa, 2002; Veevers *et al*. 2006).

The age and composition of detrital zircon grains transported out of a first-generation continental source terrane reflect the age and initial Hf isotope character of the igneous rocks (mainly granite) exposed to erosion. In sedimentary provenance studies, sufficiently large numbers (Vermeesch, 2004; Andersen, 2005) of analyses of individual crystals are pooled to produce a sample-specific distribution pattern of ages and initial Hf isotopic compositions. This distribution pattern is assumed to reflect the 'event signature' of the (in most cases geologically complex) environment in which the zircons formed (aka the protosource of the zircons). To be useful in provenance analysis, different protosource terranes must show specific patterns of age, rock type and Hf isotopic compositions, which are inherited by the detrital zircons. This justifies a *qualitative approach* to provenance analysis, which has the identification of different sources as its main

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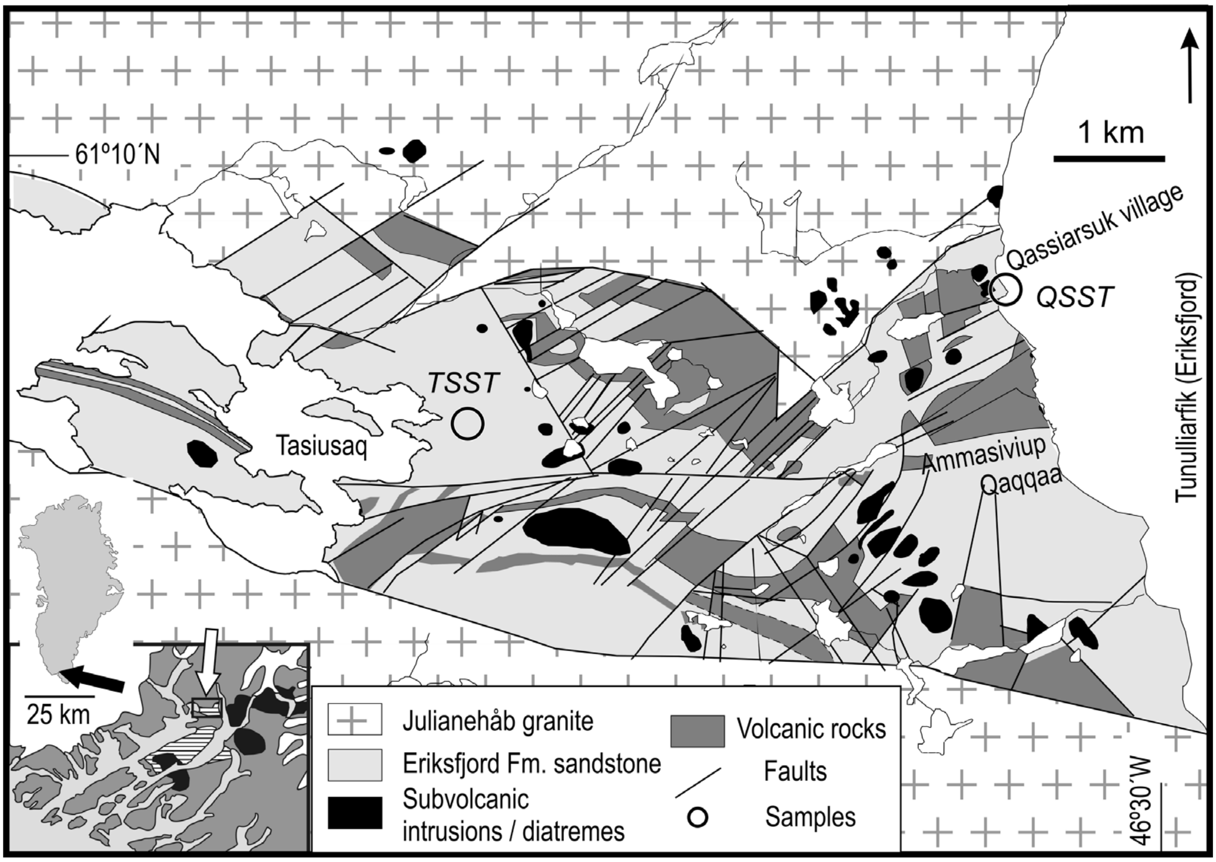

Figure 1. Simplified geological map of the Qassiarsuk–Tasiusaq graben, Gardar Rift, southwestern Greenland (Andersen, 2008). Post-carbonatite dykes and most minor faults have been left out. (Inset) The Qaqortoq–Narsarsuak region, southwestern Greenland. Key: Dark grey – Palaeoproterozoic granitic basement (Julianehåb granite). Black – Major nepheline syenite intrusions (Ilímaussaq and Igaliko complexes). Horizontal ruling – sandstones and volcanic rocks of the Eriksfjord Formation.

purpose (e.g. Fedo, Sircombe & Rainbird, 2003). If the relative abundance of different age and isotopic fractions in a set of analytical data can be assumed to reflect the corresponding distribution of fractions in the sediment, the relative importance of contributions of different protosources to the sedimentary basin can in principle be estimated. This is the *quantitative approach* of Fedo, Sircombe & Rainbird (2003). Furthermore, since a zircon must necessarily have formed before it can be transported and deposited in a sedimentary basin, the age of the youngest significant zircon age fraction in a detrital zircon population is commonly used as a maximum limit for the age of deposition (e.g. Knudsen *et al.* 1997; Bingen *et al.* 2001, 2005; Williams, 2001; Fedo, Sircombe & Rainbird, 2003).

Andersen (2005) demonstrated that a quantitative approach to detrital zircon data is generally unjustified. In this paper, new U–Pb and Lu–Hf isotope data from sandstones of the Eriksfjord Formation from the Mesoproterozoic Gardar Rift in SW Greenland are presented. These data illustrate that the qualitative approach to the interpretation of detrital zircon data may also be problematic, and that a combination of long-range transport and reworking of sediment, post-depositional alteration and parallel evolution of continents set severe limits on the applicability of the method to depositional chronology and sedimentary provenance analysis.

2. Geological setting

The Gardar Rift is a Mesoproterozoic continental rift cutting Palaeoproterozoic rocks of the Ketilidian orogen in southernmost Greenland (Emeleus & Upton, 1976; Kalsbeek, Larsen & Bondam, 1990). The Eriksfjord Formation comprises *c.* 1800 m of continental sandstone and *c.* 1600 m of volcanic rocks (Poulsen, 1964; Emeleus & Upton, 1976; Upton *et al.* 2003) deposited on a basement made up by the 1.80–1.85 Ga Julianehåb I-type granite (Garde *et al.* 2002). The volcanic rocks of the Eriksfjord Formation are mainly basalts (Halama *et al.* 2003), with minor alkaline pyroclastic rocks and lavas (Stewart, 1970; Emeleus & Upton, 1976; Andersen, 2008 and references therein). The sandstones sampled for the present study occur within the Qassiarsuk–Tasiusaq graben, which is a subsidiary E–W-trending structure within the NE–SW-trending Gardar Rift, crossing the Narsaq peninsula (Fig. 1). The volcanic and sedimentary strata in this graben compose the lower parts of the Eriksfjord Formation, i.e. the Majût sandstone member and the lower parts of the overlying Mussartût volcanic member with sedimentary interlayers (Poulsen, 1964). Unlike

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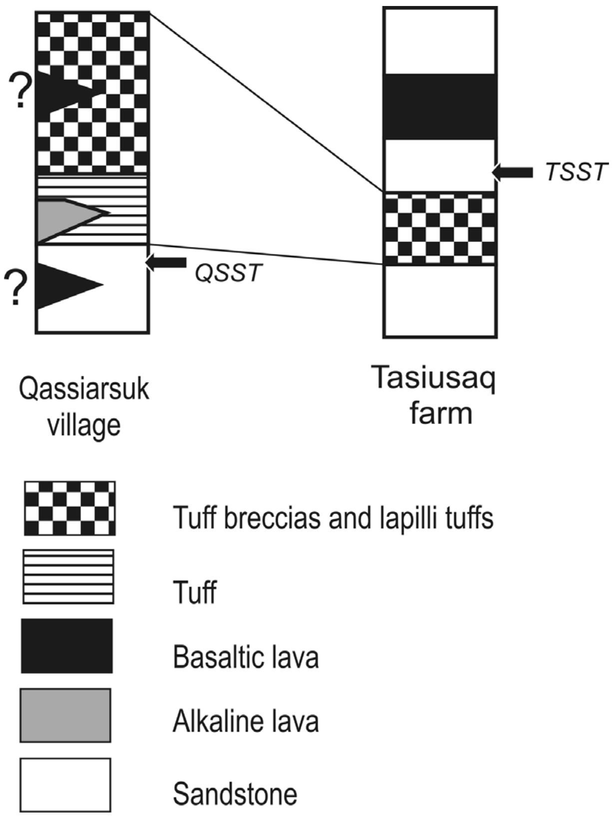

Figure 2. Simplified stratigraphic columns of the Eriksfjord Formation at Qassiarsuk and Tasiusaq, from Andersen (2008). The question marks in the Qassiarsuk village column refer to the position of an isolated outcrop of pahoehoe basalt of uncertain stratigraphic position.

in the basaltic sequence of the Mussartût type profile, some 10 km SW of Qassiarsuk (Emeleus & Upton, 1976), the first volcanic rocks in the Qassiarsuk–Tasiusaq graben are alkaline lavas and carbonatite, and melilitic pyroclastic rocks; basalts are interlayered with these at a slightly higher stratigraphic level (Fig. 2). The sandstones are penetrated by diatremes of alkaline silicate and carbonatitic composition, some of which were feeders to identifiable volcanic strata (Andersen, 2008), as well as by numerous mafic dykes belonging to the main Gardar dyke swarm (Upton *et al.* 2003 and references therein).

The sandstones of the Eriksfjord Formation vary in composition from arkose to quartz arenite, and in colour from red to white. Abundant channel cross-bedding and other primary depositional features suggest deposition on floodplains and in shallow lakes within a developing graben system; the sand is thought to have originated from sources within the Julianehåb granite (Poulsen, 1964).

The age of deposition of the basal Eriksfjord Formation sediments is bracketed by the *c.* 1.8 Ga crystallization age of the underlying Julianehåb granite (Garde *et al.* 2002) and the age of the oldest igneous rocks of the Gardar Rift. Rift-related magmatism can be divided in 'Early Gardar' and 'Late Gardar' periods. Most of the large alkaline plutons in the rift formed during the Late Gardar period, lasting from *c.* 1185 Ma to *c.* 1144 Ma, whereas the Early Gardar magmatism produced the extrusive rocks of the Eriksfjord Formation, their associated diatremes and subvolcanic rocks, mafic dykes and gabbroic to syenitic intrusions (Upton *et al.* 2003 and references therein). Based on Rb–Sr, Sm–Nd and Pb–Pb whole-rock isochrons, Andersen (1997) and Passlick *et al.* (1993) reported ages of *c.* 1200 Ma for volcanic rocks of the Qassiarsuk complex (belonging to the Mussartût member) and the stratigraphically higher Ulukasik volcanic member, respectively. These relatively young ages are difficult to reconcile with field relationships suggesting that both sandstones and lavas of the Eriksfjord Formation have been intruded by Early Gardar plutons of the Motzfeldt complex (Emeleus & Harry, 1970), dated to 1273 ± 6 Ma (U–Pb on zircon, McCreath *et al.* 2012). The *c.* 1200 Ma ages for volcanic rocks of the Eriksfjord Formation probably reflect post-magmatic hydrothermal processes rather than primary crystallization (see discussion by Upton *et al.* 2003 and Andersen, 2008).

Sample QSST was taken from a shore exposure near the quay at Qassiarsuk village (Figs 1, 2). The sandstone is a cross-bedded, white, quartz cemented quartz arenite underlying the first alkaline pyroclastic rocks. The existence of angular clasts of sandstone incorporated at the base of the pyroclastic rocks shows that the sandstone was consolidated by the time of the first volcanism. A diatreme consisting of alkaline silicate rocks penetrates the sandstones *c.* 100 m from the sampling locality. Sample TSST was taken from a quartz arenite overlying the alkaline volcanic rocks east of Tasiusaq farm (Figs 1, 2). The sandstone is similar to the lower strata sampled at Qassiarsuk, but its deposition must postdate the first phase of volcanism in this part of the Gardar Rift.

3. Zircon petrography

Zircon occurs as an abundant heavy mineral in the sandstone samples. Crystals are well rounded to sub-rounded (Fig. 3), and very commonly show short-wavelength oscillatory zoning, typical of magmatic zircon (e.g. Corfu *et al.* 2003). U–Pb ages of these zircons range from (rare) Meso- to Palaeoarchaean ages to *c.* 1300 Ma (below). Some of the Archaean zircons occur as distinct xenocrystic cores in Proterozoic grains (Fig. 3a), but in general, Archaean and Palaeoproterozoic zircons have the character of normal, igneous crystals (Fig. 3b, c). In the youngest of the age groups, domains of variable cathodoluminescence intensity interfere with, and partly obscure, the oscillatory zoning, which, however, remains visible (Fig. 3d–f).

4. Analytical methods

Heavy mineral fractions were isolated from crushed sandstone by Wilfley table washing and heavy liquid separation using sodium polytungstate solution; zircon was retrieved from these impure separates by hand

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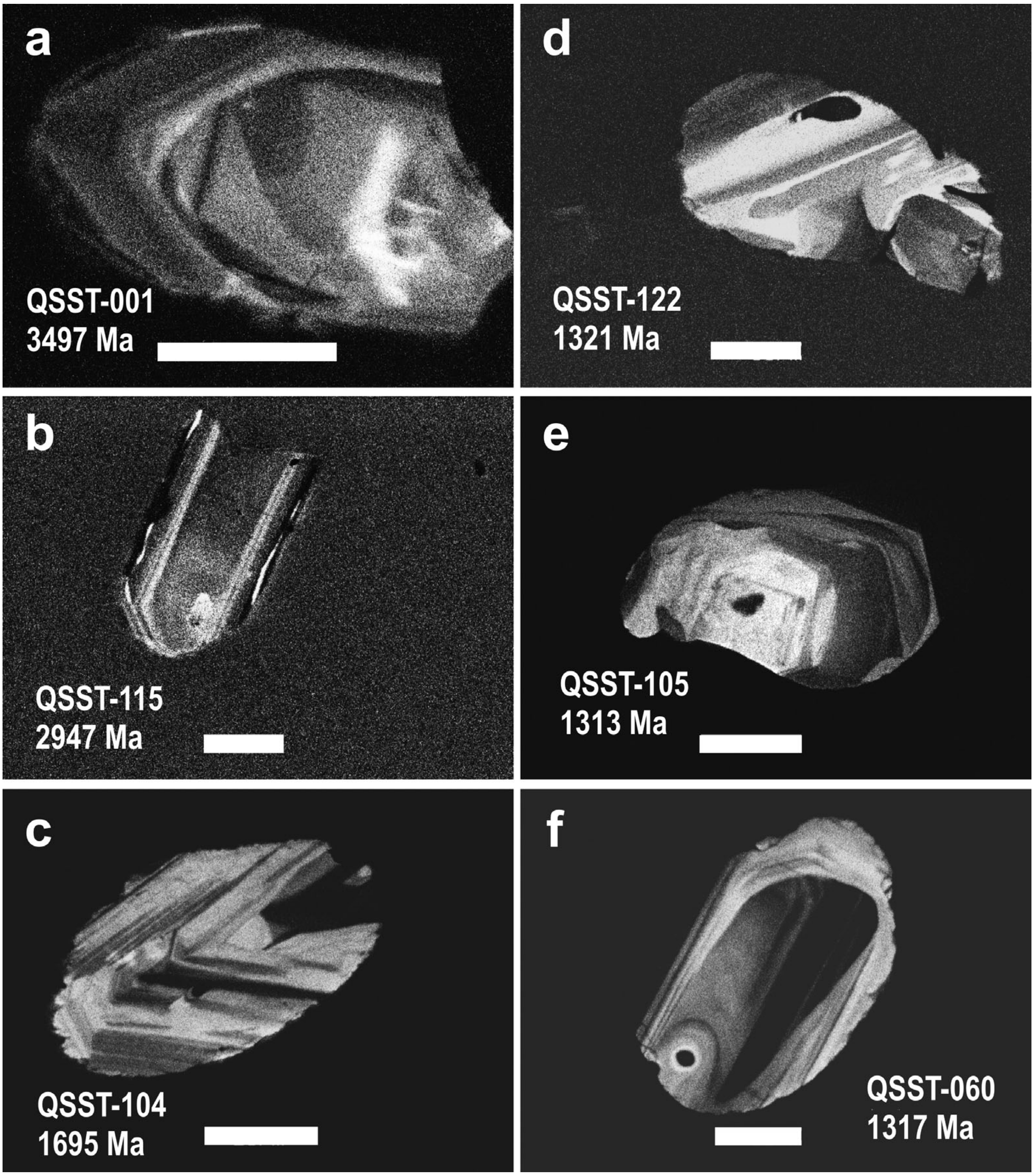

Figure 3. SEM–CL images of detrital zircons from the Eriksfjord sandstone. Analysis numbers refer to online Supplementary Table S1 (at http://journals.cambridge.org/geo), and the ages given are ^{207}Pb–^{206}Pb ages. Length of scale bars is 50 μm. (a) Palaeoarchaean core with patchy variations in CL brightness in a CL dark (undated) grain. (b) Intact magmatic zoning in a Mesoarchaean, sub-rounded grain. (c) Intact oscillatory magmatic zoning in Palaeoproterozoic zircon. (d) Young zircon with zones of enhanced CL brightness overprinting oscillatory zoning. (e) Young zircon with increased CL brightness in the lower left part. Traces of oscillatory magmatic zoning are visible within the CL bright area. (f) Young zircon with increased CL brightness along the edges. The limit of the CL bright zone is discordant to the trace of oscillatory magmatic zoning, which can be discerned in both dark and bright areas.

picking. This approach was chosen to avoid experimentally induced bias caused by magnetic separation (Sircombe & Stern, 2002; Andersen *et al.* 2011). Randomly chosen grains were embedded in epoxy resin, polished, examined under the binocular microscope and imaged by cathodoluminescence (CL) and backscattered electrons, using a JEOL JSM 6460LV scanning electron microscope (SEM) at the Department of Geosciences, University of Oslo.

U–Pb and Lu–Hf analysis was done by laser ablation inductively coupled plasma source mass spectrometry (LA-ICP-MS), using a Nu Plasma HR multicollector mass spectrometer equipped with a NewWave LUV 213 Nd-YAG laser microprobe. Analytical protocols

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followed those of Rosa *et al.* (2009) and Andersen *et al.* (2009) for U–Pb, and Heinonen, Andersen & Rämö (2010) for Lu–Hf. Standard laser operating conditions were beam diameter 40 μm in aperture imaging mode, pulse frequency 10 Hz and beam energy density *c.* 0.06 J cm^{-2} for U–Pb and beam diameter 55 μm (aperture imaging mode), pulse frequency 5 Hz and beam energy density *c.* 2 J cm^{-2} for Lu–Hf, in both cases using static ablation. Nu Instruments online software was used for reduction of Lu–Hf data, whereas U–Pb raw data were reduced using an in-house interactive spreadsheet program built on Microsoft Excel 2003. In both methods, isotopically homogeneous parts of the time-resolved signal were interactively selected for integration.

During the period these samples were analysed, repeated analyses of the Mud Tank zircon yielded $^{176}Hf/^{177}Hf = 0.282509 \pm 44$, n = 831, and Temora-2 0.282679 ± 61, n = 460; the latter (± 2 epsilon units) is accepted as a conservative estimate of the precision of the method. The decay constant of ^{176}Lu of Söderlund *et al.* (2004), CHUR (chondritic uniform reservoir) parameters of Bouvier, Vervoort & Patchett (2008) and depleted mantle parameters of Griffin *et al.* (2000), modified to the CHUR and λ values, were used. Geochronological calculations and plotting was done using Isoplot 3.57 (Ludwig, 2003).

Trace elements in zircon were analysed by LA-ICP-MS, using an Agilent 7500 quadrupole ICP-MS with a NewWave LUV213 laser microprobe at the Department of Geology, University of Helsinki, Finland. The NIST SRM610 silicate glass was used as an external standard, and ^{28}Si as an internal standard. The laser beam diameter was 80–100 μm, and the laser energy setting was adjusted to get sufficiently high counts on standards and unknowns. A fast-scanning protocol was applied, measuring masses from 28 to 238. Raw data were reduced to concentrations offline, using an interactive routine written in Excel 2003/VBA. The GJ-1 reference zircon (Liu *et al.* 2010) was analysed as an unknown as a monitor of analytical quality. A summary of analyses of GJ-1 made over a two-year period (average, 2 standard deviations, 5th and 95th percentiles) is given together with data for unknowns in the online Supplementary Material (Supplementary Table S3 at http://journals.cambridge.org/geo). For U, Y, Ce and heavy rare earth elements (HREEs) (Gd to Lu) 2RSD uncertainty is less than 30 %. The higher uncertainty for the more abundant Hf is probably due to heterogeneity (zoning) in the Hf/Zr ratio of the crystal. The values reported for the low-concentration elements La and Pr should be regarded as semiquantitative. In general, the values of individual trace elements reproduce those published by Liu *et al.* (2010), with the exception of Pb, La, Pr and Ti, where the new analyses give significantly (?) higher concentrations.

Analytical data are given in the online Supplementary Material at http://journals.cambridge.org/geo.

5. Results

5.a. U–Pb dating

Detrital zircon crystals in both of the sandstone samples show large ranges of U–Pb ages, from near the presumed age of deposition to Meso- to Palaeoarchaean (Fig. 4, online Supplementary Table S1 at http://journals.cambridge.org/geo). Although some of the analyses, especially in the older range of the age spectrum are distinctly discordant, no discordance filter (e.g. Røhr, Andersen & Dypvik, 2008) has been applied to the data. This is considered permissible since no quantitative statistical methods are used.

A major fraction of grains in QSST have ^{207}Pb–^{206}Pb ages in the range from 1290 to 1330 Ma (Fig 4c); in TSST this age fraction is much smaller (Fig. 4d). Except for this fraction, the age spectra for the two samples show similar highs and lows, with a major group of Palaeoproterozoic zircons (1750–2000 Ma), and smaller groups in the late Archaean. Sample TSST has a small group of near-concordant zircons between 3000 Ma and 3500 Ma; these ages are absent in QSST, but instead a small group of discordant zircons with ^{207}Pb–^{206}Pb ages in the range 3400 to 3600 Ma was observed. Zircons with ^{207}Pb–^{206}Pb ages between 1750 Ma and 1330 Ma are scarce, and all are distinctly discordant.

Fifty-one out of the 63 grains defining the young zircon fraction in QSST form a near-concordant cluster with a concordia age of 1304 ± 3 Ma, albeit with an elevated mean square weighted deviation (MSWD) (Fig. 5a) caused by a slight and uniform tendency to normal discordance. The clustering is very tight, with a MSWD for combined concordance and equivalence of 0.93. The 12 remaining grains fall on a lead-loss line from this cluster towards a young lower intercept; the three young zircons from sample TSST fall on this discordia line (Fig. 5b).

5.b. Lu–Hf isotopes

Lu–Hf isotope data (online Supplementary Table S2 at http://journals.cambridge.org/geo) have been recalculated to initial $^{176}Hf/^{177}Hf$ and plotted at their corresponding ^{207}Pb–^{206}Pb ages in Figure 6a. Initial $^{176}Hf/^{177}Hf$ show a wide range (0.29036 to 0.28192 or $\varepsilon_{Hf} = -38$ to $+6$), which is not correlated with age, so that all of the main age groups except the Mesoarchaean and Palaeoarchaean show 30 epsilon units or more internal variation. Distinctly positive epsilon values ($^{176}Hf/^{177}Hf >$ CHUR(t) in Fig. 6a) are found mainly in the older part of the Palaeoproterozoic age fraction (t = 1800–2000 Ma) but even in this age interval, zircons with positive ε_{Hf} are less abundant than those with negative ε_{Hf}.

The least radiogenic Proterozoic zircons ($^{176}Hf/^{177}Hf_i \leq 0.2810$) fall along the trend expected from continental crust ($^{176}Lu/^{177}Hf = 0.015$) isolated from the mantle in Palaeoarchaean time.

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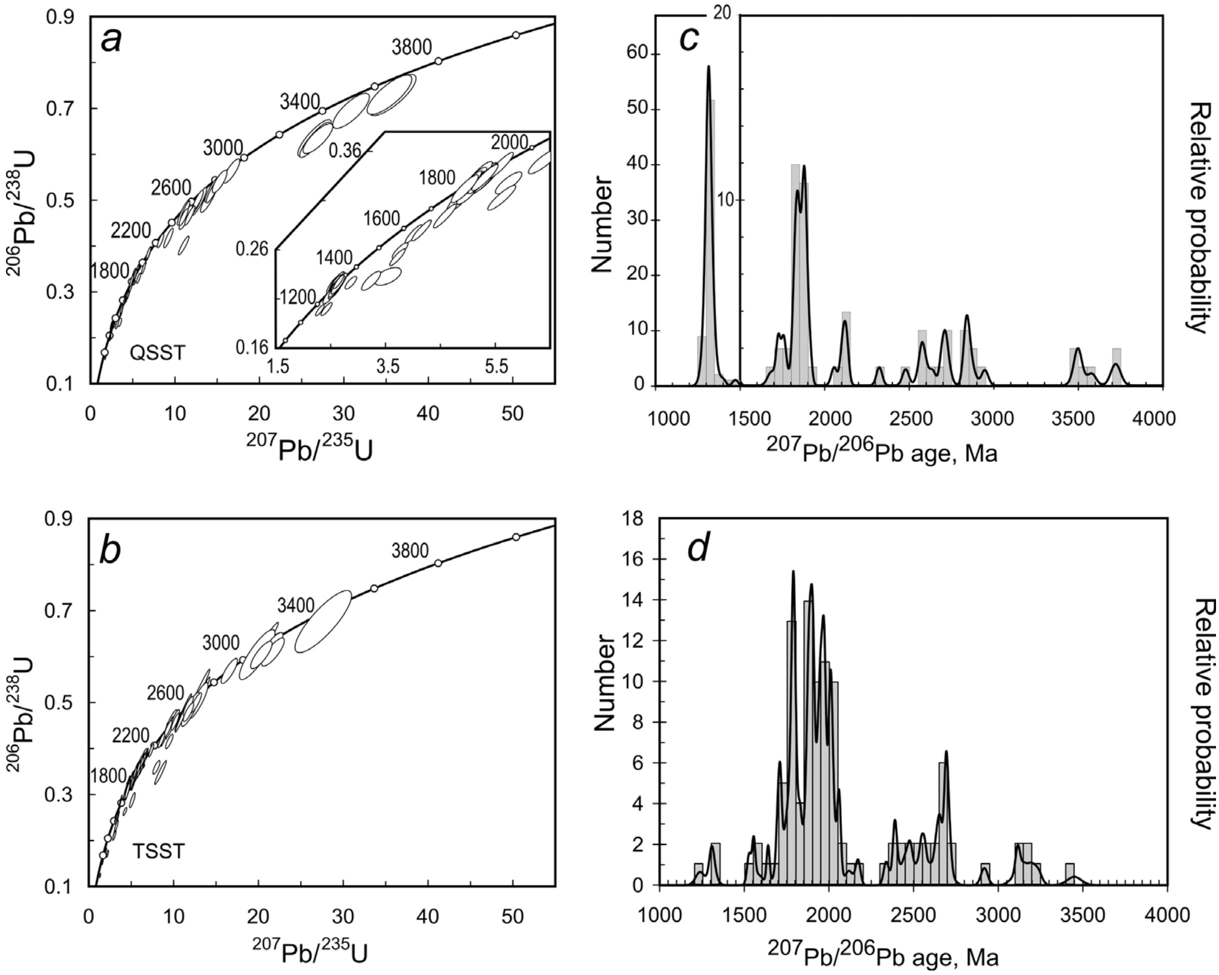

Figure 4. U–Pb data from detrital zircons in Eriksfjord sandstone. Data from online Supplementary Table S1 at http://journals.cambridge.org/geo. (a) U–Pb concordia diagram for detrital zircons in sample QSST from Qassiarsuk. Error ellipses are 2SE reported on the individual points. Inset – The lower age range of zircons in this sample, illustrating the concordant cluster of Early Gardar-age zircons (see also Fig. 5). (b) U–Pb concordia diagram for detrital zircons in sample TSST from Tasiusaq. Error ellipses are 2SE reported on the individual points. (c) Accumulated probability plot of $^{207}Pb–^{206}Pb$ ages from sample QSST from Qassiarsuk. Note change of scale at 1500 Ma. (d) Accumulated probability plot of $^{207}Pb–^{206}Pb$ ages from sample TSST from Tasiusaq.

The *c*. 1300 Ma ('Gardar age') zircons show a range of initial Hf compositions, with ε_{Hf} from −38 to 0. A similar range in initial $^{176}Hf/^{177}Hf$ is seen in the U–Pb discordant zircons with $^{207}Pb–^{206}Pb$ ages between 1750 and 1400 Ma (Fig. 6b).

5.c. Trace elements

Trace elements were analysed in selected zircons from sample QSST, mainly to detect any systematic difference between the *c*. 1300 Ma age fraction and the older zircons (Fig. 7, data from online Supplementary Table S3 at http://journals.cambridge.org/geo). The chondrite-normalized trace element distribution patterns are typical for zircons, with positive anomalies for Hf, U, Th and Ce, and negative anomalies for Ti, Nb and Eu. Each of the main age groups (Gardar-age, Palaeoproterozoic, Archaean) shows significant concentration ranges, which overlap completely for elements from Gd to P in Figure 7. The upper part of the light rare earth element (LREE) concentration ranges of Archaean and Palaeoproterozoic zircons resemble the relatively flat and LREE-enriched patterns typically seen in granitoids (Belousova *et al*. 2002), but many grains from these age groups fall significantly below this field. The Gardar-age zircons are lower in LREEs and Pb than the Palaeoproterozoic zircons, and overlap only with the lower range of LREEs seen in the Archaean zircons.

6. Discussion

6.a. Young zircons: young source or post-depositional lead loss?

A conventional interpretation of the young age fraction in QSST is that these zircons have been derived from one or more source rocks formed at a well-defined age of 1304 ± 3 Ma, i.e. in the initial phase of the Early Gardar period (Upton *et al*. 2003). The only information on the Hf isotope characteristics of Early Gardar igneous rocks available to date are LA-ICP-MS Hf isotope analyses of zircon from an altered syenite in the Motzfeldt complex (J. A. McCreath, unpub.

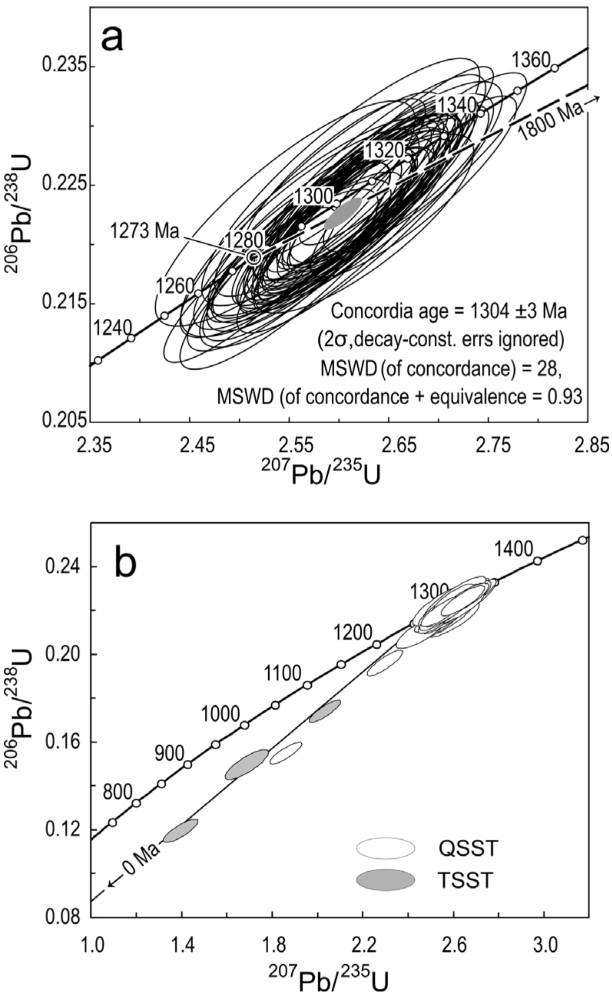

Figure 5. U–Pb systematics of young zircons in the Eriksfjord sandstone. (a) Concordia diagram for young concordant zircons from sample QSST from Qassiarsuk (N = 51). The grey ellipse represents the weighted average of the analyses, as reported by Isoplot (Ludwig, 2003). The broken line is a reference cord from 1273 Ma, assumed to represent Early Gardar intrusive magmatism (McCreath *et al.* 2012) to an upper intercept at 1800 Ma. (b) Lead-loss line from the cluster in (a) to a lower intercept at zero defined by discordant zircons in QSST and TSST (shaded). This suggests that some zircons in the young age group have suffered recent lead loss.

Ph.D. thesis, Univ. St Andrews, 2009), which indicate ^{176}Hf/^{177}Hf between 0.28193 and 0.28226 (ε_{Hf} = −1 to +11) at 1273 Ma (A. A. Finch, pers. comm.). These values are in general more radiogenic than those of the Gardar-age zircons in the sandstone, with only marginal overlap between the two groups (Fig. 6b); this intrusion is therefore not a likely source for the Gardar-age zircons in the sandstone.

The wide range of ε_{Hf} values in the Gardar-age zircon fraction (ε_{Hf} = 0 to −38) would imply that the source rock was derived from a very heterogeneous crustal source. An S-type granite could probably fit the description, except that the LREE levels of the Gardar-age zircons are lower than what is commonly expected for granites (e.g. Belousova *et al.* 2002). The large size of this age fraction suggests an uncommonly well-defined maximum age limit for the deposition of the sediment. This 'standard' interpretation of these detrital zircon data implies the existence of extrusive or intrusive rocks of Early Gardar age that, in contrast to most other Gardar igneous rocks, which are clearly mantle derived (e.g. Blaxland *et al.* 1978; Paslick *et al.* 1993; Andersen, 1997; Halama *et al.* 2003; Coulson *et al.* 2003), must have been formed by melting of a very heterogeneous crustal source. The existence of such a source rock would have had far-reaching consequences for the evolution of the Gardar Rift. Unfortunately, no rocks meeting these criteria have been reported from southern Greenland (e.g. Kalsbeek, Larsen & Bondam, 1990; Henriksen *et al.* 2000; Upton *et al.* 2003).

Potential distant source rocks could be found in the Grenville province of North America, where 1300–1400 Ma igneous rocks of continental arc and anorogenic origin do occur (Hanmer *et al.* 2000; McLelland, Selleck & Bickford, 2010 and references therein). Sm–Nd isotope data suggest a generally juvenile character for these rocks (e.g. DePaolo, 1985), and North American detrital zircons in the 1.2 to 1.5 Ga age range in general show positive ε_{Hf} (Belousova *et al.* 2010; Condie *et al.* 2011) in contrast to the negative values in the Eriksfjord Formation. Since the young zircons are scarce in the younger sandstone unit, the *c.* 1300 Ma hypothetical source was more likely local than distant, and must either have been removed by erosion, or blanketed over by the earliest Eriksfjord sandstones and the volcanic rocks of the Qassiarsuk complex.

The 1304 ± 3 Ma age calculated for the Gardar-age zircons falls within the range of possible depositional ages for the Eriksfjord Formation, and an alternative explanation for the age is that it reflects loss of radiogenic lead from older detrital zircons after deposition of the host sediment. Low-temperature diagenetic processes have been shown to cause significant lead loss in detrital zircons (Willner *et al.* 2003), but in the Gardar Rift, a thermal event caused by the first Early Gardar magmatism is a more likely cause. The sandstone in Qassiarsuk village is immediately overlain by the lavas and tuffs of the Qassiarsuk complex and is penetrated by several diatremes, one of which is exposed close to the position of sample QSST (Andersen, 1997, 2008). Sample TSST was taken from a sandstone unit deposited after the culmination of the early magmatism in the Qassiarsuk–Tasiusaq graben, and has escaped similar local heating after deposition. Southern Greenland was geologically quiet from the end of the Ketilidian activity at *c.* 1740 Ma (Garde *et al.* 2002) to the start of the Gardar rifting event (e.g. Kalsbeek, Larsen & Bondam, 1990). Discordant zircons with ^{207}Pb–^{206}Pb ages between *c.* 1740 Ma and *c.* 1330 Ma most likely record partial lead loss in the same thermal event (Fig. 6b).

The effects of ancient and recent lead loss on the U–Pb and Lu–Hf isotope systems of zircon are well understood (e.g. Amelin, Lee & Halliday, 2000): both of the processes will cause normal discordance of the U–Pb ages, but recent lead loss acting alone will

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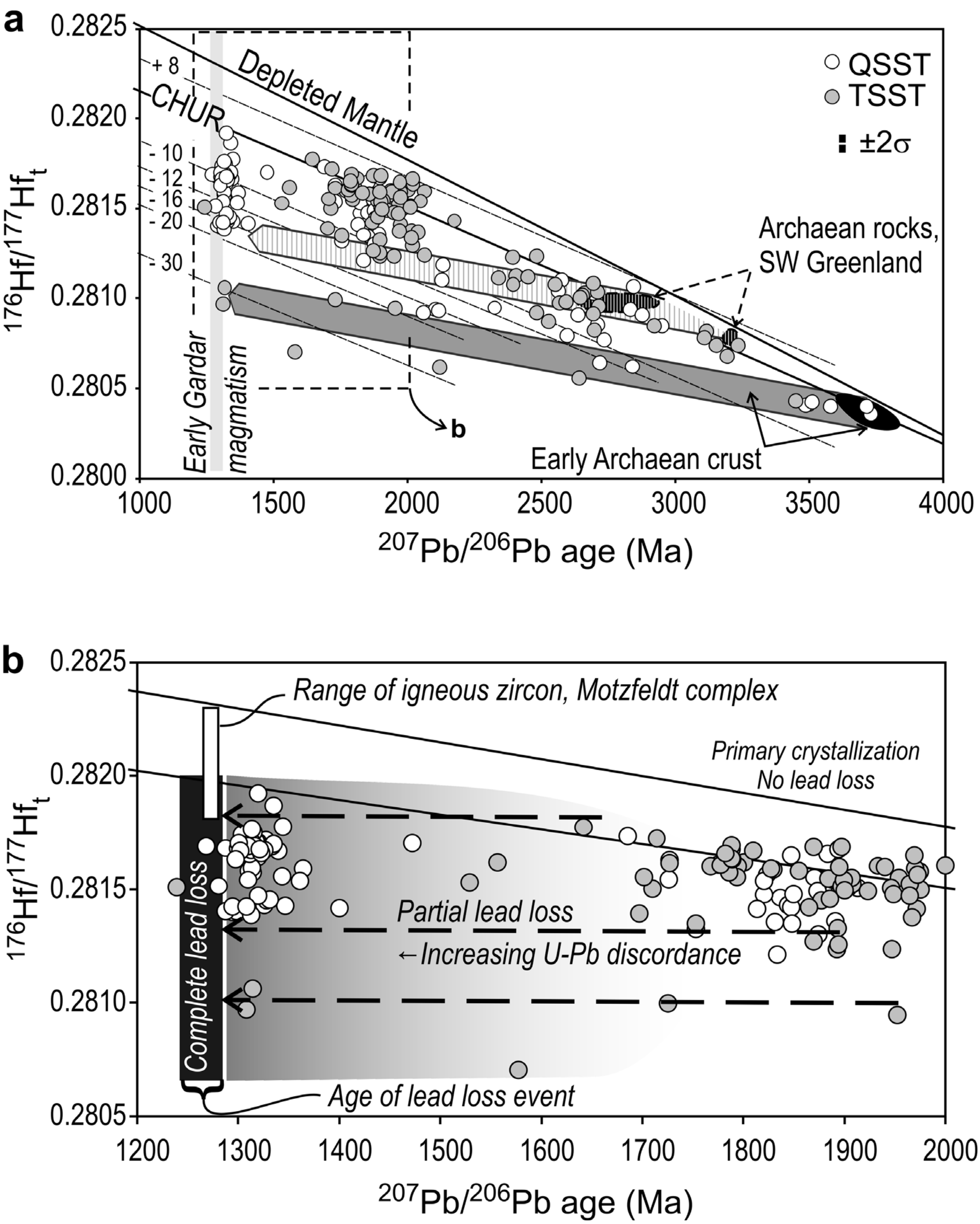

Figure 6. Initial $^{176}Hf/^{177}Hf$ detrital zircons in the Eriksfjord sandstone plotted at the $^{207}Pb–^{206}Pb$ age of the individual zircon. (a) An overview of all the data (online Supplementary Table S3 at http://journals.cambridge.org/geo). The range of early Archaean rocks in Greenland (black) is based on data from Blichert-Toft *et al*. (1999) and Amelin, Lee & Halliday (2000); ranges for later Archaean rocks in SW Greenland are from Szilas *et al*. (2012*a,b*). The fields of Hf isotope evolution of early and late Archaean crust through time have been constructed assuming average crustal $^{176}Lu/^{177}Hf = 0.015$ (grey and ruled arrows, respectively). The box outlined by a broken line is the limit of the diagram magnified in (b). (b) Detail of (a) showing the effect of lead loss in Early Gardar time on Palaeoproterozoic and older zircons. Zircons that have suffered no lead loss are concordant at their primary crystallization ages (Palaeoproterozoic to Archaean). Zircons that have lost all their lead are also U–Pb concordant, but at the age of the secondary event. Crystals that have suffered partial lead loss are U–Pb discordant and thus give meaningless $^{207}Pb–^{206}Pb$ ages, but in the Hf isotope evolution diagram they plot along subhorizontal trends starting at the primary crystallization age and terminating at the age of the lead-loss event (broken arrows). Increasing shade suggests increasing loss of lead in Early Gardar time. The range of $^{176}Hf/^{177}Hf$ in zircon in syenite from the Motzfeld complex has been adapted from the unpublished Ph.D. thesis of J. A. McCreath (Univ. St Andrews, 2009).

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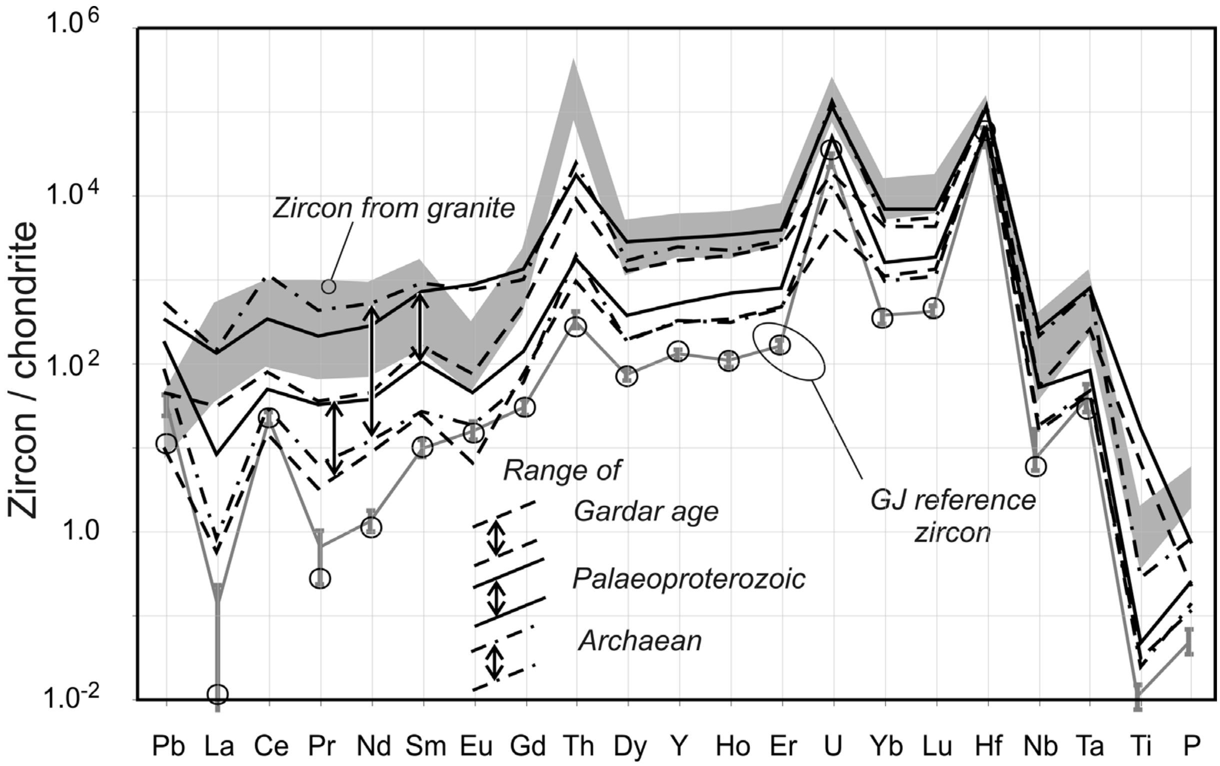

Figure 7. Ranges of trace element concentrations in detrital zircons from the Eriksfjord sandstone (sample QSST). The analyses (online Supplementary Table S3 at http://journals.cambridge.org/geo) have been normalized to chondritic values of McDonough & Sun (1995); the plotting sequence follows Belousova, Griffin & O'Reilly (2006). The average of repeated analyses of the GJ-1 reference zircon are shown with 5 to 95 interpercentile ranges (vertical bars). Circles represent the average of eight mean analyses reported by Liu *et al*. (2010).

influence neither the ^{207}Pb–^{206}Pb age nor the $^{176}Hf/^{177}Hf$ calculated at the ^{207}Pb–^{206}Pb age. In contrast, ancient lead loss will generate a subhorizontal trend in a $^{176}Hf/^{177}Hf$ versus ^{207}Pb–^{206}Pb age diagram, whose slope is controlled by the $^{176}Lu/^{177}Hf$ ratio of the zircon, and which terminates at the age of the lead-loss event. An old zircon that has lost all its radiogenic lead in such an event will be U–Pb concordant at the younger age, but the corresponding $^{176}Hf/^{177}Hf$ ratio will remain close to the original value of the zircon, owing to the flat slope of the lead-loss line in the Hf isotope evolution diagram. The ^{207}Pb–^{206}Pb age of a zircon that has suffered partial lead loss in an ancient event is meaningless and serves mainly to constrain the lead-loss trend in the $^{176}Hf/^{177}Hf$ versus time diagram (Fig. 6b). If a group of detrital zircons with variable age and initial Hf isotopic composition were affected by complete lead loss after deposition of the host sediment, the result would be a vertical array in the $^{176}Hf/^{177}Hf(t)$ versus time diagram, at the age of the lead-loss event, similar to what is observed in the Eriksfjord Formation (Fig. 6b).

The large range of $^{176}Hf/^{177}Hf$ observed in the Gardar-age zircons in the Eriksfjord sandstone can be adequately explained by extensive loss of radiogenic lead in Early Gardar time from Palaeoproterozoic to late Archaean detrital zircons whose ε_{Hf} prior to lead loss ranged from low positive to highly negative values, as shown in Figure 6b. It is not possible to assign a primary crystallization age to these zircons or to relate them to a specific protosource rock. This age fraction thus has no implications for sedimentary provenance and does not define a maximum depositional age for the sediment. Post-depositional alteration has not affected the Lu/Hf ratio or the Hf isotope composition of the zircons. The trace element patterns suggest that LREEs may have been lost together with radiogenic lead; other elements were not affected by the process. No visible overgrowths of new zircon were formed, and the pre-existing oscillatory zoning pattern is largely preserved, although locally overprinted by an interfering, non-oscillatory cathodoluminescence signal (Fig. 3d–f).

6.b. Archaean and Palaeoproterozoic protosources: near or distant?

The Gardar Rift is contained within the late Palaeoproterozoic Ketilidian orogen of southern Greenland, but relatively close to its boundary with the Archaean craton (Fig. 8). The Archaean craton of southern Greenland records events from *c*. 3.8 Ga to the Neoarchaean (Henriksen *et al*. 2000; Hölttä *et al*. 2008). Palaeoarchaean rocks give near-chondritic initial Hf isotope compositions at 3.6–3.8 Ga (Blichert-Toft *et al*. 1999; Amelin, Lee & Halliday, 2000); younger Archaean rocks in SW Greenland are near-chondritic at 2.7–3.0 Ga, with a hint of *c*. 3.2 Ga inheritance at $\varepsilon_{Hf} \approx +2$ (Szilas *et al*. 2012*a,b*). These two groups of Archaean rocks evolved to $\varepsilon_{Hf} \approx -30$ to -35 and

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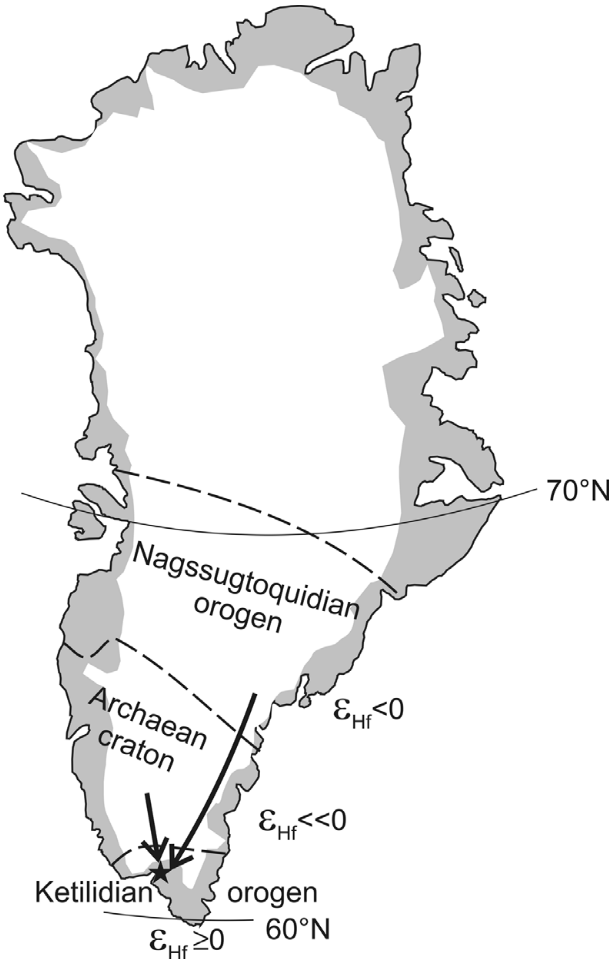

Figure 8. Simplified sketch of Greenland, showing the position of the Archaean craton and the Nagssugtoquidian and Ketilidian orogens

−15 to −20, respectively, at Early Gardar age, given a normal crustal ^{176}Lu/^{177}Hf ratio of 0.015 (Fig. 6a). No other Hf isotope data for rocks of late Archaean and Palaeoproterozoic age have so far been published, but Sr, Nd and Pb isotope data from rocks in the 2.7 Ga Skioldungen complex in SE Greenland (Blichert-Toft *et al*. 1995) and the Ketilidian orogen indicate a dominant contribution from juvenile material in late Archaean and Palaeoproterozoic time, including in the 1.80–1.85 Ga Julianehåb I-type granite batholith making up the basement in and around the Gardar Rift (van Bremen, Aftalion & Allaart, 1974; Patchett & Bridwater, 1984; Kalsbeek & Taylor, 1985; Garde *et al*. 2002). Because of the well-established positive correlation between Nd and Hf isotopes in crustal rocks (Vervoort & Patchett, 1996; Vervoort & Blichert-Toft, 1999), rocks with positive or near-zero ε_{Nd} are highly unlikely to produce zircons with strongly negative initial ε_{Hf} (Fig. 9). In contrast, rocks of the marginally older (1.92–1.83 Ga, van Gool *et al*. 2002) Nagssugtoquidian orogen north of the Archaean craton show a pronounced tendency to negative ε_{Nd} values (Fig. 9). Such rocks would be expected to produce zircons with a Palaeoproterozoic age and a distinctly negative ε_{Hf} signature.

The rare > 3500 Ma zircons in the Eriksfjord sandstone overlap in Hf isotopes with published Hf isotope data from Palaeoarchaean rocks in Greenland (Fig. 6). Near-chondritic zircons of 2.7–3.2 Ga age most likely originated in juvenile Archaean intrusions at some distance in East or West Greenland, whereas Neoarchaean and early Palaeoproterozoic zircons (2.2 to 2.6 Ga) with negative ε_{Hf} may have crystallized in this period from magmas derived from mixtures of Palaeoarchaean crust and juvenile material, with or without a contribution from late Archaean juvenile crust. Detrital zircons of *c*. 1800–1900 Ma age with low positive ε_{Hf} may have originated from protosources resembling the Julianehåb granite and other igneous rocks of the Ketilidian orogen. However, most of the Palaeoproterozoic zircons in the Eriksfjord sandstones have negative ε_{Hf} (Fig. 9), implying protosources with an extended crustal history at the time of zircon crystallization. From neodymium isotope data, such rocks are more likely to be found in the Nagssugtoquidian orogen. This suggests that far-transported Archaean and Palaeoproterozoic detritus derived from primary sources outside of the Ketilidian orogen has contributed significantly to the Eriksfjord sandstone.

It is unlikely that far-transported detritus has moved from primary sources in the Nagssugtoquidian orogen or the Archaean craton to the site of deposition in the Gardar Rift in a one-step process during Gardar time. More likely, Archaean and Palaeoproterozoic material was stored in intermediate reservoirs made up by older sedimentary rocks that have been recycled and re-deposited within the rift. Erosional remnants of pre-Gardar cover sequences that may have acted as intermediate repositories occur in the border zone between the Archaean and Ketilidian provinces of southern Greenland (Allaart, 1976).

Long transport distances and repeated events of recycling of detritus may be the rule rather than the exception. For example, Røhr, Andersen & Dypvik (2008) and Røhr *et al.* (2010) found that Cretaceous sandstones in northern Greenland and Arctic Canada contain significant contributions from recycled Proterozoic and Caledonian sediments. Furthermore, detrital zircons in Permian sandstone in the Oslo Rift, Norway, include fractions from sources in the central and eastern parts of the Fennoscandian Shield, which were brought to the final site of deposition through repeated cycles of sedimentation and re-deposition (Andersen *et al.* 2011).

6.c. Greenland or Fennoscandia: the problem of indistinct Proterozoic and Archaean protosource signatures

To use detrital zircon data to identify protosources for clastic sediments, potential source terranes must be different enough in age and Hf isotope signature so that they can be distinguished from each other. To illustrate that this may not always be the case, the combined set of data from the Eriksfjord sandstone is superposed on an accumulated set of data on igneous zircons

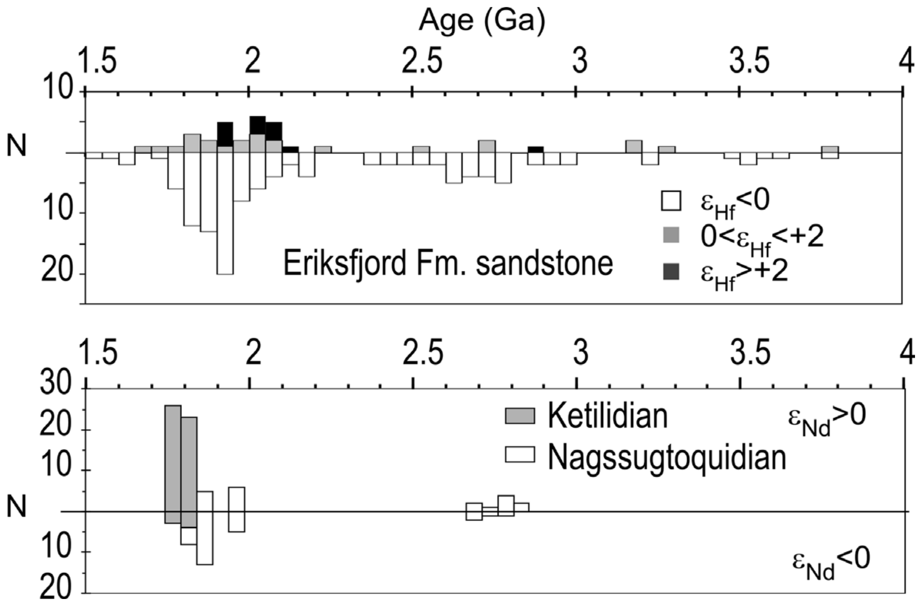

Figure 9. Initial Hf isotope signatures in Palaeoproterozoic and Archaean zircons from the Eriksfjord sandstone (this work) compared to Nd isotope data on potential protosource terranes. Sources of data: *Ketilidian* – Patchett & Bridgwater (1984) and Brown *et al.* (2003). *Nagssugtoquidian* – Kalsbeek, Pidgeon & Taylor (1987) and Whitehouse, Kalsbeek & Nutman (1998).

from Fennoscandian granitoids in Figure 10a. The pre-Gardar-age fractions of detrital zircons in the Eriksfjord sandstone show impressive overlap with the granitic zircons from Fennoscandia. Taken out of geological context, the similarity between the two patterns would indicate that the Fennoscandian granitoids were a likely protosource for the Eriksfjord zircons.

The similarity between sediment from Greenland and granitoids in Fennoscandia may have three different causes: (1) it could be pure coincidence; (2) it could reflect parallel evolution in different continents, possibly related to supercontinent evolution in the geological past; or (3) the two continental blocks may have exchanged detritus when forming part of one or more old supercontinents. The observation that detrital zircons from sediments from Greenland and Arctic Canada, spanning a range of depositional ages from the Neoproterozoic to the Cenozoic, and modern sediment load in the Mississippi river show considerable overlap with the Fennoscandian granitoids argues against a purely coincidental explanation (Fig. 10b). On the other hand Fennoscandia and Laurentia, including Greenland, have formed part of Phanerozoic Pangaea, Mesoproterozoic Rodinia and a Palaeoproterozoic supercontinent (e.g. Zhao *et al.* 2004; Condie, 2005; Lahtinen, Garde & Melezhik, 2008). In some reconstructions, the Palaeoproterozoic granitoid complexes of Fennoscandia continued into those of the Nagssugtoquidian and Ketilidian provinces of Greenland (Karlstrom *et al.* 2001; Lahtinen, Garde & Melezhik, 2008). Parallel tectonomagmatic evolution in the two continents in Palaeoproterozoic time is therefore not at all unrealistic. The result would be overlapping U–Pb and Lu–Hf signatures that make the identification of source terranes from detrital zircon data difficult or even impossible.

Exchange of detritus between adjacent blocks in a supercontinent, in intracontinental basins that have since been broken up, or along a common continental shelf also cannot be ruled out. This would produce a more or less homogenized pool of sediment that can be eroded and re-sedimented in a later period. These processes add up to produce indistinct source terrane signatures, so that the assumption underlying the qualitative approach to detrital zircon analysis is violated. The data summarized in Figure 10 suggest that even combined age- and Hf isotope spectra cannot be used to answer a first-order question such as if a given sediment is derived from sources to the east or west of the North Atlantic.

6.d. The future of detrital zircon studies in stratigraphy and provenance analysis

The use of the youngest detrital zircon grain or fraction of grains in a sediment to define a maximum age of deposition has previously been criticized because there is no necessary connection between processes that generate zircons and those that release and eventually deposit them in a sedimentary basin (e.g. Andersen, 2005). Furthermore, instrumental U–Pb analysis is seldom precise enough to distinguish between undisturbed concordant zircons and zircons that have seen incipient lead loss (e.g. Knudsen *et al.* 1997). The present findings point to another possible source of bias in such data: even a significant and well-defined age fraction of detrital zircons may have been modified by loss of radiogenic lead after deposition of the sediment by the action of diagenetic fluids (Willner *et al.* 2003) or by a thermal event (this work). There are published examples in which the timing of sediment deposition is very tightly bracketed by

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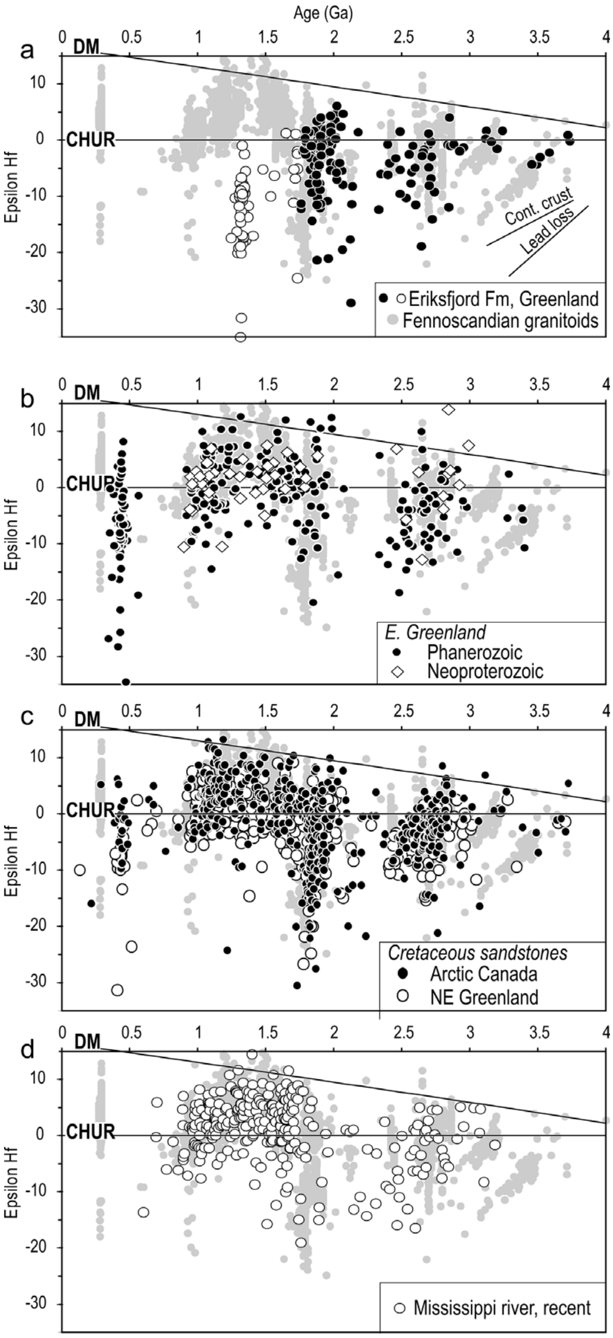

Figure 10. ε_{Hf} signatures of detrital zircons in the Eriksfjord sandstone compared to data on igneous zircons from Fennoscandian granitoids. Fennoscandian data from Patchett *et al.* (1981), Anderson, Griffin & Pearson (2002), Andersen *et al.* (2004, 2009), Anderson, Graham & Sylvester (2007, 2009), Heinonen, Andersen & Rämö (2010), Kurhila, Andersen & Rämö (2010), Heilimo *et al.* (2012), Lauri *et al.* (2011), Pedersen *et al.* (2009) and author's database. (a) Data from Eriksfjord sandstone (this work). (b) Detrital zircons from sedimentary rocks in eastern Greenland (Slama *et al.* 2011). (c) Detrital zircons from Cretaceous sandstones in Arctic Canada and NE Greenland (Røhr, Andersen & Dypvik, 2008; Røhr *et al.* 2010). (d) Detrital zircon in present-day sedimentary load of the Mississippi river (Condie *et al.* 2011).

the age of a significant detrital zircon fraction, and a well-defined, independently datable post-depositional tectonothermal event (e.g. Ordovician sediments of the Helgeland Nappe complex in Northern Norway; Barnes *et al.* 2007). Whereas post-depositional metamorphism or cross-cutting intrusions can generally be dated with confidence, the present results suggest that any model where the maximum age of deposition is based on detrital zircon data should be treated with extreme care, and only brought forwards where there are independent time constraints on sedimentation (e.g. biostratigraphic markers or datable diagenetic minerals).

Analyses of detrital zircons may still contribute to the understanding of sedimentary provenance, but to be used with confidence, the data must be combined with a broad range of other information about the rocks from which the zircons are separated, as well as a full understanding of the prior evolution of potential source terranes. Using detrital zircons as a first or unsupported approach is in general unjustified, and may in some cases lead to misleading or insignificant results. Probably, the method will be most useful in settings where one or more distinct and well-characterized point-sources may have contributed.

7. Conclusions

Although the use of combined U–Pb and Lu–Hf data on detrital zircons has contributed to our understanding both of crustal evolution on a global scale and evolution of sedimentary basins on a regional scale, the use of the method in stratigraphy and provenance analysis is fraught with problems that have not received sufficient attention. Some of these problems are highlighted by the data from the sandstones of the Mesoproterozoic Eriksfjord Formation in the Gardar Rift of southern Greenland.

(i) Young zircon age fractions may reflect post-depositional processes rather than processes in the source terrane, and are therefore useless as indicators of the maximum age of sedimentation.

(ii) Zircons may not have a clear and unique path from source to depositional sink: they may have been recycled through several intermediate reservoirs, which may have helped smooth out protosource signatures.

(iii) Different crustal source terranes may not necessarily have sufficiently different event signatures for zircons derived from them to be allocated to a source. For some continents, such as those bordering the North Atlantic, this is largely a result of processes of crustal evolution in former supercontinent settings.

Detrital zircon data may be most useful as a supplement to other information on the host sediments. Taken alone, they will have less predictive power than has generally been assumed.

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