# Lu-Hf systematics of 4.0 – 2.3 Ga old zircons from the Turee Creek Group (Pilbara Craton, W. Australia): Implications on the rise of atmospheric oxygen and global glaciation during the Paleoproterozoic

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## Abstract

We investigated the Hf isotopic systematics of detrital zircons in a succession of siliciclastic sediments and glacial diamictites from the early Paleoproterozoic sequence of the Boolgeeda Iron Formation (Hamersley Group) and overlying Turee Creek Group of the Pilbara Craton, Western Australia. About 400 detrital zircons yielding > 95% concordant U-Pb ages were analyzed for Hf isotopes to constrain their magmatic sources. 70% of the analyzed zircons display super-chondritic initial Hf isotopic compositions, demonstrating crystallization in mantle-derived magmas. Most of the data are comprised between model age lines at ~2.5 and 3.2 Ga, which suggests a sub-continuous crust generation by extraction from the depleted mantle during this time period. A single grain yields a 4.0 Ga age, which represents the first Hadean age for a zircon from the Pilbara Craton. Our results are significantly different from zircon Hf isotope data of the Glenburgh Terrane, in the southern border of the Turee Creek Group, or older successions of the Pilbara, Kaapvaal and Superior cratons, but show overlap with some of the Yilgarn Craton. This together with the occurrence of a Hadean zircon crystal preserved in the Boolgeeda glacial diamictite with similar Hf isotopic signature than the Jack Hills zircons makes the Yilgarn Craton a possible source material for the Boolgeeda glacial horizon. Alternatively, the majority of the zircons analyzed show ages which are consistent with those of the underlying 2.45 – 2.78 Ga Hamersley and Fortescue groups, formed by sedimentary successions interleaved with thick subaerial volcanic sequences associated with the emplacement of Large Igneous Provinces. Such subaerial volcanic rocks could account for the relatively juvenile character of the zircon analyzed. A local provenance of the siliciclastic material delivered to the Turee Creek Basin would support the role of large subaerial magmatic provinces as triggers of the rise of atmospheric oxygen and the onset of glaciations at the beginning of the Proterozoic.

***Keywords***

Zircon; Hafnium isotopes; Turee Creek Group; Great Oxidation Event; Paleoproterozoic glaciations; Hadean.

## 1. Introduction

The Archean-Proterozoic transition was marked by important redox and climatic changes, specifically the rise of atmospheric oxygen, the so-called Great Oxidation Event (GOE) (Holland, 2002), and several Paleoproterozoic glacial events (e.g., Rasmussen *et al.*, 2013; Caquineau *et al.*, 2018). The causal relationships between these events remain largely debated (e.g., Kopp *et al.*, 2005; Teitler *et al.*, 2014). One model proposed by Kump and Barley (2007) explained the rise of atmospheric oxygen by a change in the eruptive dynamic of the Earth at ca. 2.5 Ga that shifted the main style of volcanism from marine to subaerial. This increase in the subaerial proportion of Large Igneous Provinces (LIPs) would have buffered the greenhouse gases content of the atmosphere due to carbon dioxide consumption through enhanced silicate weathering, thus enabling atmospheric oxygen to build up and surface temperature to cool down, leading to global glaciation (Cox *et al.*, 2016). In addition, subaerial eruptions are known to produce more oxidized lavas and gases (Holland *et al*., 2002; Gaillard *et al*., 2011), which could also have contributed further to reduce oxygen sinks.

Zircon is a very resistant mineral, of which Hf is a major constituent (~ 1wt%), that represents the only witness of eroded and unknown source protoliths (Iizuka *et al.*, 2017). The incorporation of significant amounts of mantle-derived volcanic material within the upper continental crust can generate a large increase of juvenile Hf isotopic signatures in the zircons from the sediments. Glacial diamictites are considered to record the average chemical composition of the upper continental crust from which they formed by erosion (Gaschnig *et al.*, 2014). Consequently, U-Pb-Hf systematics in zircons sampled in these sediments appears as a valuable strategy to test the possible occurrence of materials derived from mantle sources. Here, we performed a Lu-Hf isotopic study of detrital zircons previously analyzed for U-Pb geochronology (Caquineau *et al*., 2018) from a suite of glaciogenic and non-glaciogenic sediments of Paleoproterozoic age that spans the GOE. The rocks investigated are from the Boolgeeda Iron Formation, which represents the uppermost succession of the Hamersley Group, and the Turee Creek Group (TCG) in the Hamersley Basin at the southern margin of the Pilbara Craton, Western Australia (Fig. 1). The main objective of this study is to (i) identify the major sources and further constrain the formation's model of this sedimentary sequence and (ii) to test the hypothesis of massive subaerial LIPs emplacement in the Pilbara Craton and its implications on the GOE and the Paleoproterozoic glaciations.

## 2. Geological setting

The Turee Creek Group (TCG) is a ca. 4 km-thick continuous sedimentary succession conformably overlying the Boolgeeda Iron Formation of the Hamersley Group and unconformably overlain by the Beasley River Quartzite and the Cheela Springs Basalt of the lower Wyloo Group (Figs. 1, 2; Martin, 1999; Van Kranendonk *et al.*, 2015). From bottom to top, the TCG consists of siliciclastic sedimentary rocks, minor stromatolitic carbonates and glaciogenic strata of the Kungarra Formation, quartz-rich sandstones of the Koolbye Formation, and shales and stromatolitic marine carbonates of the Kazput Formation (Fig. 2b; Trendall, 1981; Martin, 1999; Van Kranendonk *et al.*, 2015). The TCG sedimentary sequence has been described as a broad shallowing upward profile from deep-water iron formation of the Boolgeeda Iron Formation, through fine-grained siliciclastic deposits of the Kungarra Formation, to fluvial and shallow marine strata of the Koolbye and Kazput formations (Martin, 1999; Mazumder *et al.*, 2015; Van Kranendonk *et al.*, 2015). The Boolgeeda Iron Formation overlies the Woongarra Rhyolite and Weeli Wooli Formation, which have been interpreted as a subaerial bimodal mafic and felsic volcanic sequence emplaced at 2450 Ma (Barley *et al.*, 1997). This intense volcanic event would have produced large volumes of lavas (>30 000 $km^3$, Barley *et al.*, 1997) covering the continental surface at the time of the glacial event recorded in the Boolgeeda Iron Formation, and possibly during the subsequent deposition of the overlying TCG. The unconformably overlying Beasley River Quartzite represents a terrestrial deposit composed of a basal fluvial conglomerate and overlain by an unit of fine-grained quartzite (Mazumder and Van Kranendonk, 2013). Metamorphism of the group did not exceed sub-greenschist facies conditions (Rasmussen *et al.*, 2005).

Several sections of the TCG have been described in the southwestern margin of the Hamersley Basin (Martin, 1999; Van Kranendonk *et al.*, 2015; Krapež *et al.*, 2017). These include from East to West: the Turee Creek, Hardey and Duck Creek synclines, as well as the Yeera Bluff area (Fig. 1b). Glaciogenic horizons were used as reference markers to establish stratigraphic correlations among these different sections. The main glacial body is the ~400m thick Meteorite Bore Member (MBM) that was originally described in the Hardey Syncline section (Trendall, 1981; Figs. 1, 2b and e). More recently, Van Kranendonk and Mazumder (2015) reported a second level of massive diamictites located above the reference Meteorite Bore Member in the Hardey Syncline; in the following, we will refer to this horizon as the 'second Kungarra diamictite'. A third diamictite horizon associated with sandstones and siltstones was described in the Duck Creek Syncline at the Boundary Ridge locality and at Yeera Bluff located 50 and 150 km north westward from the Hardey Syncline, respectively (Fig. 1b, Martin, 1999; Van Kranendonk *et al.*, 2015). These diamictites, associated with deep marine sediment of the Boolgeeda Iron Formation, were interpreted as distal equivalents of the Meteorite Bore Member formed as sediment gravity flows and rain-out from rare icebergs (Martin, 1999; Van Kranendonk *et al.*, 2015). However, Caquineau *et al.* (2018) and Philippot *et al.* (2018) recently reported the occurrence of a 2m-thick diamictite horizon within the underlying Boolgeeda Iron Formation in the Hardey Syncline (drill core TCDP1, see below and Fig. 2d). Because this diamictite layer is stratigraphically located ~1500 meters below the Meteorite Bore Member, it thus represents a different and older glacial event. Based on new geochronological (see below) and stratigraphic constraints, Caquineau *et al.* (2018) and Philippot *et al.* (2018) showed that this diamictite layer can be correlated with the diamictites horizons reported in the Boolgeeda Iron Formation at Yeera Bluff and the Boundary Ridge locality. This glacial horizon is named hereafter the 'Boolgeeda diamictite'.

Time constraints on the deposition of the sedimentary sequence of interest include: (i) a U-Pb zircon age of 2450 ± 3 Ma for the underlying Woongarra Rhyolite (Figs. 1, 2a and b; Trendall *et al.*, 2004), (ii) a U-Pb detrital zircon maximum age of 2460 ± 9 Ma for the Boolgeeda diamictite (Caquineau *et al.*, 2018), (iii) a Re-Os age of 2312 ± 6 Ma obtained on diagenetic pyrite from the Meteorite Bore Member diamictite (Philippot *et al*., 2018) in agreement with a U-Pb maximum age on detrital zircon of 2340 ± 22 Ma (Caquineau *et al*., 2018) for this glacial formation, and (iv) a U-Pb zircon age of 2209 ± 15 Ma for the overlying Cheela Springs Basalt (Martin *et al.*, 1998). A baddeleyite U-Pb age of 2208 ± 10 Ma was obtained on dolerite sills that intrude the TCG and are truncated by the unconformably overlying Beasley River Quartzite (Müller *et al.*, 2005). Thus, the significance of the age of the Cheela Springs Basalt has been questioned (Müller *et al.*, 2005). Accordingly, the age of the Beasley River Quartzite remains loosely bracketed between ~2210 Ma and 2031 ± 6 Ma (Fig. 2a, Martin *et al.*, 1998; Müller *et al.*, 2005).

The sedimentation of the TCG is considered to have occurred in the McGrath Trough, an asymmetric basin structurally linked with the migration to the northeast of a geanticline or a thrust-fold belt system (e.g., Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015). This basin has been interpreted either as a retroarc foreland basin (Martin *et al.*, 2000; Müller *et al.*, 2005; Krapež *et al.*, 2017) or as an intracontinental basin (Van Kranendonk *et al.*, 2015). It is generally accepted that the Turee Creek Group formed in a long-lived basin between ~2.45 and 2.2 Ga (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015). An alternative view (Krapež *et al.*, 2017), which proposes that the TCG formed in a short-lived foreland basin that closed by ~2.43 Ga or shortly after, can be ruled out by recent geochronological constraints on the MBM diamictites mentioned above (Caquineau *et al*., 2018; Philippot *et al*., 2018). The provenance of the sediments forming the TCG is subject of debate, mainly because it relies on controversial paleocurrent data, which established that the detritus was principally derived from the south and the southwest (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015; Krapež *et al.*, 2017). The major source terranes include the Pilbara Craton, the Hamersley Province to the north (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015), and the Glenburgh Terrane to the south (Krapež *et al.*, 2017), a continental exotic fragment that is thought to have collided with the southwestern margin of the Pilbara Craton ca. 2.2 Ga ago (Johnson *et al.*, 2011; 2013). The occurrence of three discrete glacial diamictite horizons, and new age constraints of the TCG led Caquineau *et al*. (2018) to propose that the ca. 2.45 Ga-old Boolgeeda diamictite can be correlated with (1) the pre-2.43 Ga Makganyene Formation of the South African Transvaal Supergroup (Gumsley *et al.*, 2017), (2) the basal glaciogenic horizon of the Ramsay Lake Formation of the North American Huronian Supergroup (Rasmussen *et al.*, 2013), and the ca. 2.43 Ga old Fennoscandian glacial diamictites (Melezhik *et al.*, 2013). This early glacial event at about 2.45 Ga recognized in five different cratons could therefore have been global in extend (i.e., Snowball Earth). Caquineau *et al.* (2018) further proposed that the two diamictites of the Kungarra Formation (the MBM and the second Kungarra diamictite) can be correlated with the pre-2.31 Ga Gowganda Formation (North America) and upper Duitschland (South Africa) glacial diamictites (Rasmussen *et al.*, 2013), and the ca. 2.26 Ga old upper Timeball Hill (North America) and Ditojana (South Africa) diamictites (Rasmussen *et al.*, 2013), as well as the Enchantment Lake Formation (North America).

## 3. Samples

The samples analyzed in this study have been described in detail in Caquineau *et al.* (2018). These include drill core samples collected in the course of the Turee Creek Drilling Project (TCDP1 and TCDP2) obtained at the base and intermediate level of the Turee Creek Group, respectively, as well as surface samples collected in lithostratigraphic levels not intercepted by the drill cores (Fig. 2). Six samples were selected from different stratigraphic levels in drill cores TCDP 1 and TCDP 2 and eight complementary samples were collected in the field (Fig. 2; drill core samples are noted T1-X or T2-X, with X referring to the depth; surface samples are labelled with Pi- followed by the sampling year and the sample number). These include from bottom to top: Three glaciogenic dropstone-bearing sandstones of the Boolgeeda glacial diamictite (T1-152 and Pi-09.07 and Pi-09.08 of the Boundary Ridge Locality), a sandstone layer at the base of TCDP 2 (T2-332) and four samples of the main glacial diamictite deposit of the Meteorite Bore Member (T2-272, T2-205, T2-169, T2-130), three samples of the second Kungarra glacial diamictites (Pi-15.15, Pi-15.16, Pi-15.17), two samples from meter-scale massive sandstone bars of the upper part of the Kazput Formation (Pi 15.41, Pi-15.42), and one sample of the Beasley River Quartzite (Pi-15.02).

The Hf isotopic composition was measured in 382 detrital zircon grains from diamictites, sandstones and quartzites that previously gave > 95% concordant U-Pb dates (Fig. 2 and Table 2). These include: (1) 82 zircons from the Boolgeeda diamictite both from the Hardey (drill core sample T1-152) and Duck Creek (surface samples Pi 09.07 and Pi 09.08) synclines (Fig. 2c and d), (2) 108 zircons from the Meteorite Bore Member diamictite (drillcore samples T2-130, T2-169, T2-205 and T2-272) and an underlying sandstone bar (T2-332) (Fig. 2e), (3) 101 zircons from the second Kungarra diamictite (surface samples Pi 15.15, Pi15.16, Pi 15.17), (4) 59 zircons from two sandstone layers of the upper part of the Kazput formation (surface samples Pi 15.41, Pi 15.42) (Fig. 2f), and (5) 32 zircons from the Beasley River Quartzite (surface sample Pi 15.02).

## 4. Methods

The cathodoluminescence image (CL) of the Hadean mounted zircon grain was generated using a Zeiss Sigma FEG-SEM equipped with a detector operating at 15kV current. Mineral separation involved crushing and sieving at the threshold of 300 μm, Wilfley table, Frantz magnetic separator and heavy liquids. U-Th-Pb isotopic data on zircons were obtained by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the Laboratoire Magmas & Volcans (LMV, Clermont-Ferrand, France). Detailed analytical techniques are reported in Hurai *et al.* (2010), Paquette *et al.* (2014) and Caquineau *et al.* (2018).

Hafnium isotope analyses were performed with a Thermo Scientific Neptune Plus Multicollector (MC) ICPMS coupled to a Resonetics M50E 193nm Excimer laser system at the LMV (Clermont-Ferrand). The operating conditions and instrument settings are described in Moyen *et al.* (2017) and Paquette *et al.* (2017) and detailed in S1. Repeated analyses of both natural (91500, GJ-1 and Mud-Tank) and synthetic Hf and REE-doped (Fischer et al., 2011) zircon reference materials are reported in Table S1. More than 50 replicates of 91500, GJ-1 and Mud Tank zircon reference materials yielded $^{176}Hf/^{177}Hf$ of 0.282307 ± 16, 0.282011 ± 15 and 0.282520 ± 12, respectively, which well agree with reference values (eg. Hu *et al.*, 2012). Mean and two standard deviation values obtained on MUNZirc 0-2a ($^{176}Hf/^{177}Hf$ = 0.28213 ± 3), MUNZirc 1-2b (0.28214 ± 2); MUNZirc 3-2b (0.28215 ± 2) and MUNZirc 4-2b (0.28216 ± 3) are fully consistent with reference values (Fisher *et al.*, 2011). The $\varepsilon_{Hf}$ values are calculated as parts per 10,000 deviation of the $^{176}Hf/^{177}Hf$ of the zircons at the time of their formation relative to the Chondritic Uniform Reservoir (CHUR) defined by the $^{176}Lu$ decay constant of 1.867 x $10^{-11}yr^{-1}$ (Söderlund *et al.*, 2004) and the CHUR parameters ($^{176}Hf/^{177}Hf$ = 0.282793; $^{176}Lu/^{177}Hf$ = 0.0338; Iizuka *et al.*, 2015). A $^{176}Lu/^{177}Hf$ ratio of 0.015 typical of bulk continental crust (Griffin *et al.*, 2004) has been used for representing model age lines.

## 5. Zircon Hf isotopic compositions

Hf isotopic data are presented in Table 1. The zircons from the Boolgeeda diamictite yielded a range of $^{207}Pb/^{206}Pb$ ages between 2436 and 3216 Ma, as well as a single Hadean grain (discussed below), and initial $\varepsilon_{Hf}(t)$ values between -12.4 to +6.2 (Fig. 3a). About half of the analyzed zircons display a supra-chondritic Hf isotopic composition (i.e. > 0) reflecting the addition of material derived from partial melting of the depleted mantle at an unknown time. In contrast, the remaining zircons analyzed shows a sub-chondritic Hf initial isotopic composition (i.e. < 0) indicating that zircons parental melts recycled an evolved crust.

Zircons from the MBM diamictite yielded a range of $^{207}Pb/^{206}Pb$ ages between 2359 and 3000 Ma and initial $\varepsilon_{Hf}(t)$ values between -5.2 and +7.6 (Fig. 3b). The mantle-derived component is proportionally more important in the MBM diamictite than in the Boolgeeda diamictite and the time interval appears shifted toward slightly younger ages. Zircons from the second Kungarra diamictite yielded a range of $^{207}Pb/^{206}Pb$ ages between 2430 and 3350 Ma and initial $\varepsilon_{Hf}(t)$ values between -11.8 and +6.7 (Fig. 3 c). Again the addition of a mantle-derived component appears predominant as recorded by chondritic to supra-chondritic Hf initial isotopic compositions. This pattern appears very similar to that of the Booldeega diamictite. Zircons from the sandstones of the upper part of the Kazput Formation yielded a range of $^{207}Pb/^{206}Pb$ ages between 2430 and 3230 Ma and initial $\varepsilon_{Hf}(t)$ values between -7.4 and +6.4 (Fig. 3d). Most of the data display supra-chondritic Hf initial isotopic composition, which demonstrates a prevailing input of mantle-derived component. These Hf isotope data share similarities with those of the MBM diamictite (Fig. 4b).

Zircons from the Beasley River Quartzite yielded a range of $^{207}Pb/^{206}Pb$ ages between 2450 and 3230 Ma and initial $\varepsilon_{Hf}(t)$ values between -10.4 and +5.9 (Fig. 3e). Almost equal proportions of sub- and supra-chondritic zircon Hf isotopic composition were recorded. The results present similar ages and Hf initial isotopic signatures than the other formations.

When all the analyses are plotted together (Fig. 3f), two major points are evidenced: (1) the majority of the U-Pb ages range from 2.45 Ga to 3.20 Ga, and (2) more than 70% of the analyses plot in the supra-chondritic field, which demonstrates the prevailing occurrence of mantle-derived materials. Considering a $^{176}Lu/^{177}Hf$ ratio of 0.015 typical of bulk continental crust (Griffin et al., 2004), the data can be confined between two model age lines at *c.a.* 2.54 Ga and 3.20 Ga. As discussed by Vervoort and Kemp (2016), these ages are dependent of a number of factors such as mantle source models and Lu/Hf values and therefore should not be taken at face values and only considered as indicative of the time when the source of the zircon's protoliths separated from the mantle.

## 6. The first record of a Hadean zircon in the Pilbara Craton

Four analyses of a single zircon crystal (120 x 60 µm in size) from sample Pi 09.07 of the 2.45 Ga Boolgeeda diamictite at the Boundary Ridge locality yielded an upper intercept at 4013 ± 69 Ma. One of the analyses is concordant at 3998 ± 15 Ma (Fig. 4a and Table 2). The Th/U ratios (0.2 to 0.8) favour a magmatic origin for the crystal. This zircon crystal represents the first record of a Hadean relict within the Pilbara Craton. The zircon shows a negative initial $\varepsilon_{Hf}(t)$ value of – 3.3 (Fig. 4b), which is consistent with the filtered Hf isotope dataset for Jack Hills zircons in the Yilgarn Craton after Whitehouse et al. (2017). These authors interpreted these data to represent zircon growth events during the Hadean, although the nature of these events remains unconstrained. As discussed by Kemp et al. (2010), it is therefore tempting, although speculative, to propose the crystallization of this Hadean zircon in a magma source derived from the Depleted Mantle and carrying an evolved crustal component earlier than 4.0 Ga. Depending on the nature of the crust (i.e. felsic or mafic) from which the zircon crystallized, the Hf model ages are 4.27 or 4.49 Ga (Fig. 5b), which are in agreement with Nd model ages obtained on samples from the 3.5 Ga Dresser Formation in the Pilbara Craton (Tessalina *et al.*, 2010).

# 7. Discussion

## 7.1 Summary of the zircon Hf isotopic signatures

Zircon Hf isotopic data appear relatively consistent throughout the sedimentary sequence of the Boolgeeda Iron Formation and the Turee Creek Group. About 70 % of the analyzed zircons are characterized by chondritic to supra-chondritic Hf isotopic compositions, with 30% of the analyses showing $\varepsilon_{Hf}(t)$ values higher than +3. Most of the data plot between model age lines at ~2.5 and 3.2 Ga (Fig. 3f). Although these ages are not representative of the time of extraction of juvenile magmas from the depleted mantle (Vervoort and Kemp, 2016), the lack of evidence for a crustal contribution older than 3.2 Ga indicates that the majority of the data is consistent with a sub-continuous or multi-episodic crust generation through depleted mantle differentiation after about 3.2 Ga. Eo- and Paleo-Archean crustal rocks did not contribute significantly to the sedimentary material of the Boolgeeda diamictite and TCG sediments, in agreement with zircon age peaks at 2.54, 2.68, 2.82, 2.95 and 3.15 Ga (Caquineau *et al.*, 2018). The recognition that the zircon Hf isotopic compositions of the Beasley River Quartzite (unconformably overlying the Turee Creek Group) are undistinguishable from the Boolgeeda and Turee Creek zircon Hf compositions further support the notion of a relatively homogeneous source over a substantial long period of time between ~2.45 and 2.0 Ga.

## 7.2 Paleogeographic significance of the zircon Hf isotopic data

To evaluate the main sources of the Boolgeeda diamictite and the TCG sediments we have considered our isotope data in the broader context of published radiogenic isotope data for igneous rocks from several representative cratons in Australia (Glenburgh Terrane and Pilbara and Yilgarn cratons), South Africa (Kaapvaal Craton) and North America (Superior Craton).

### *7.2.1 GlenburghTerrane*

The Glenburgh Terrane has been defined as an exotic continental fragment that collided with the southwestern margin of the Pilbara Craton during the ~2.22 – 2.15 Ga Ophtalmian orogeny (Blake & Barley, 1992; Martin *et al.*, 2000; Johnson *et al.*, 2011). Zircons from the 2430–2555 Ma old Halfway Gneiss, which represents the oldest component of the Glenburgh Terrane, have a variety of Paleoproterozoic and Archean ages and a relatively narrow range of sub-chondritic Hf isotopic compositions interpreted to reflect reworking of an older 2600-2730 Ma crust (Johnson *et al.*, 2011). Some of the ages and Hf isotopic data overlap those of the Boolgeeda diamictite and TCG detrital zircons (Fig. 5a), but only a few of the zircons from the Glenburgh Terrane match the supra-chondritic values recorded in our samples. Hence, while the Glenburgh Terrane may have contributed to Turee Creek basin (Krapež *et al.*,2017), it cannot be considered as a major source material of the Boolgeeda diamictite and TCG sediments.

### *7.2.2 Pilbara Craton.*

Magmatic zircons from the Pilbara Craton mainly crystallized at about 3.2-3.3 Ga or before (Fig. 5b; Gardiner *et al.*, 2017; 2019; Petersson *et al.*, 2019 and references therein). These TTG suites are interpreted as derived from partial melting of basaltic crust with the contribution from older felsic components (e.g. xenoliths of the Strelley Pool Formation up to 3.7 Ga; Gardiner *et al.*, 2019). After ca. 3.3 Ga, the trend toward more negative Hf values is interpreted by these authors as recording more extensive crust reworking. In the eastern

Pilbara Craton, multiple pulses of magmatism produced voluminous granitic complexes from 3.5 Ga to 2.95-2.92 Ga and 2.85-2.82 Ga. This age range is much older than the one recorded for the Boolgeeda and TCG detrital zircons. As shown in figure 5b, the Hf isotope data from both central and eastern Pilbara do not overlap with those of the TCG, ruling out any significant contribution of the Pilbara Craton to this sedimentary succession.

### *7.2.3 Northern Yilgarn Craton*

The Yilgarn Craton located south of the Pilbara Craton and Glenburgh Terrane consists of several distinct tectonic entities (the Narryer, Youanmi and South West terranes and the Eastern Goldfields Superterrane) that have been accreted to the amalgamated Pilbara Craton–Glenburgh Terrane during the ~1.96 – 2.0 Ga Glenburgh orogeny (Occhipinti *et al*. 2004; Johnson *et al.*, 2011, 2013), about 200 Ma after deposition of the TCG (i.e. 2210 Ma, Müller *et al.*, 2005). Available Hf isotope data of magmatic zircons from the Yilgarn Craton (Griffin et al., 2004) show similarities with our results. Both display supra-chondritic values during Neoarchean times, a quite similar range of ages and only a few zircon grains older than 3.2 Ga (Fig. 5c). However, the Boolgeeda sample also contains younger grains with supra-chondritic Hf isotopic compositions down to 2.4 Ga compared to the Yilgarn zircons (2.6 Ga). Nevertheless, the strong overlap between our Hf isotope data and those of the Yilgarn Craton together with the occurrence of a Hadean zircon crystal in the Boolgeeda diamictite, similar in age and Hf isotopic signature to the Jack Hills zircons (Whitehouse *et al*., 2017 and references therein) makes the Yilgarn Craton a possible source for the Boolgeeda glacial horizon. This conclusion is in apparent contradiction with the cratonisation history and paleogeographic reconstruction of the Yilgarn and Pilbara cratons, which are considered to be part of two different supercratons during the early Paleoproterozoic (Cheney, 1996; de Kock *et al.*, 2009; Smirnov *et al.*, 2013), and to have accreted during the ~1.96 – 2.0 Ga Glenburgh orogeny

(Johnson *et al.*, 2013), that is about 200 Ma later than the upper age limit of the TCG (Müller *et al.*, 2005; see chapter 7.4 below for further discussion).

### *7.2.4 Kaapvaal Craton*

Magmatic zircons from the Kaapvaal Craton mainly crystallized from material that is much older than the one recorded for the Boolgeeda and TCG detrital zircons (Fig. 5e, Zeh *et al.*, 2009). Assuming the 'Vaalbara' hypothesis (Cheney, 1996; deKock *et al.*, 2009; see Eriksson *et al.*, 2011 for an alternative hypothesis), the post-2080 Ma Magaliesberg Quartzite, which is stratigraphically located at the top of the Transvaal Supergroup, could represent an analog of the TCG sediments. Two populations of zircons can be distinguished on the basis of U-Pb ages and initial $\varepsilon_{Hf}$(t) values (Fig. 5d, Zeh *et al.*, 2016). One is characterized by Archean age and both supra- and sub-chondritic Hf isotopic signatures; the second population shows Paleoproterozoic age and sub-chondritic Hf isotopic compositions (Fig. 5d). Our data from the Boolgeeda diamictite and TCG sediments mostly plot between these two zircon populations, which argue for distinctly different sources. The zircons of Archean age (population 1) of the Magaliesberg Quartzite have been attributed to the erosion of the Pietersburg Block of the Kaapvaal Craton. The absence of major overlap with the Boolgeeda and TCG zircons indicates that this terrane was not a major source of the Boolgeeda and TCG sediments. The source of the Paleoproterozoic zircons (population 2) is unclear. The dominant crustal recycling signature indicated by the strongly negative $\varepsilon_{Hf}$(t) values could be linked to the break-up of the Vaalbara Supercraton between ~2.45 and 2.2 Ga. Accordingly, U-Pb geochronology, Hf isotopes and paleogeographic reconstructions all argue against the Kaapvaal Craton as a possible source for the Boolgeeda diamictite and TCG sediments.

### *7.2.5 Superior Craton*

The Paleoproterozoic glacial events also affected the Superior Craton during the deposition of the Huronian Supergroup (Young, 1970). Available zircon Hf isotopic data from the Superior Craton are mostly supra-chondritic (Corfu and Noble, 1992; Corfu and Stott, 1993 and 1996; Davis *et al.*, 2005) and characterize samples older than 2.7 Ga. As shown in figure 5f, data from the Boolgeeda diamictite, the TCG and the Superior Craton are mostly different, likely due to different cratonization histories.

### 7.3 Implications for a prevailing mantle-derived magma source

The comparison of our detrital zircon $\varepsilon_{Hf}(t)$ data with those of the literature shows that the Glenburgh Terrane and the Pilbara, Kaapvaal, Zimbabwe and Superior cratons did not contribute significantly to the Boolgeeda and TCG sediments. Over 80% of the analyzed zircons yielded ages spanning those of the Hamersley and Fortescue groups between 2.45 and 2.78 Ga (Arndt *et al.*, 1991; Trendall *et al.*, 2004). This supports the interpretation that the Boolgeeda Iron Formation and the TCG successions mainly derived from the erosion of the underlying Hamersley and Fortescue groups. Martin et al. (2000) and Van Kranendonk et al., (2015), originally proposed this interpretation based on geological and stratigraphic reconstructions. As shown by their supra-chondritic Hf isotopic compositions, detrital zircons from the Boolgeeda diamictite and the TCG successions preferentially incorporated mantle-derived material. This is not typical of terrigenous sedimentation in cratonic settings, where erosion products generally record diverse lithologies of various ages reflecting the erosion of older crustal basement characterized by sub-chondritic Hf isotopic compositions. The marked differences in zircon Hf compositions between the Boolgeeda diamictite and TCG sediments, and the Pilbara Craton (Fig. 5b) and the Glenburgh Terrane (Fig. 5a) is particularly striking, as these two continental blocks are the most likely sources in addition to the Fortescue and Hamersley groups.

A possible explanation for the minor proportion of sub-chondritic Hf isotopic compositions in the Boolgeeda diamictite and TCG zircons can be a lack of emerged crustal blocks carrying a sub-chondritic Hf isotope record at the time of deposition. This is particularly critical for the Pilbara Craton to the north and the Glenburgh Terrane to the south, which have been considered as the main potential sources of the TCG successions (e.g., Krapez *et al*., 2017). The scarcity or lack of surface exposure of Pilbara Craton lithologies seems unlikely as it comprises a wide range of rocks of different ages between 2.7 and 3.5 Ga that crop out on thousands of $km^2$ (Hickman and Van Kranendonk, 2012). Additionally, the Mount Bruce Supergroup, which contains the TCG and the underlying Hamersley and Fortescue groups, was unconformably deposited on the basement of the Pilbara Craton (Arndt *et al.*, 1991). The lack of detrital zircon much older than 3.2 Ga within the TCG may also be related to the absence of efficient transport and/or erosion of Archean material. However, sandstones of the Koolbye Formation and of the Beasley River Quartzite (see Fig. 2) have been interpreted as fluvial to shallow marine, and fluvial to aeolian deposits, respectively (Mazumder and Van Kranendonk, 2013; Mazumder *et al.*, 2015), witnessing the activities of rivers and wind during the deposition of the TCG.

About 75% of the zircons analyzed here come from glaciogenic deposits (i.e.Boolgeeda, MBM and second Kungarra diamictites). Glaciogenic rocks typically derive from widespread physical erosion of the upper continental crust by icesheets and are therefore geographically dispersed (e.g., Gaschnig *et al.*, 2014). The supra-chondritic Hf isotopic composition recorded in the major part of the zircon populations from the Boolgeeda and Turee Creek sediments is at odd with a sub-chondritic Hf isotopic composition generally expected for the recycled upper continental crust (e.g., Condie, 2014). The Welli Wooli Formation of the Hamersley Group, comprising basalts, dolerites as well as the Woongarra Rhyolite, has been interpreted as a subaerial bimodal Large Igneous Province (LIP) emplaced at 2450 Ma (Barley *et al.*, 1997). This intense volcanic event would have produced large volumes of lavas (>30 000 $km^3$, Barley *et al.*, 1997) covering the continental surface at the time of the glacial event recorded in the Boolgeeda Iron Formation, and possibly during the subsequent deposition of the overlying TCG. Thus, it is suggested that the erosion of these volcanic materials by ice sheets and their incorporation as terrigenous detritus within the glacial diamictites of the Boolgeeda and Turee Creek sedimentary successions could account for the supra-chondritic Hf isotopic compositions of many analyzed zircons (Fig. 3). Similarly, zircons corresponding to age peaks at 2540 and 2680 Ma could have been produced by previous volcanic eruptions attending deposition of the Fortescue and Hamersley groups and preserved at the continental surface. It is noteworthy that the Hf isotopic compositions of zircons from non-glacial sediments, i.e the sandstones of the Kazput Formation and the Beasley River Quartzite, do not exhibit different $\varepsilon_{Hf}(t)$ vs. time patterns than the zircons from the glacial diamictites (Fig. 3d and 3e). This suggests that these sandstones and quartzites may represent the erosional products of the same kind of LIP-derived volcanic material involved in the formation of the glacial diamictites.

The hypothesis that the Earth's surface was covered by juvenile lavas during the Archean-Proterozoic transition, at least on the Pilbara Craton, may also have important implications on the concomitant rise of atmospheric oxygen (the GOE) and drastic cooling of the Earth's surface. As shown by Kump and Barley (2007), a rapid increase of the subaerial proportion of LIPs at ca. 2.5 Ga would have abruptly and permanently diminished the predominant sink for oxygen (submarine volcanism) and increased the sink for carbon dioxide (silicate weathering). This in turn would have enabled oxygen to build up into the atmosphere and triggered a glacial period (Cox *et al.*, 2016).

### 7.4 Geodynamic implications of a Hadean zircon in the Pilbara Craton

Several studies have suggested that the Pilbara Craton developed on a Hadean continental substrate (Smithies *et al.*, 2003, 2007; Champion, 2013; Tessalina *et al.*, 2010). Such ancient crust has, however, never been identified in outcrop. The oldest rock reported so far is the metamorphosed Mount Webber Gabbro located in the western part of the Shaw Granitic Complex, which formed at 3.59–3.58 Ga (Petersson *et al.*, 2019), and therefore predates the oldest known volcanic activity of the 3.53–3.23 Ga Pilbara Supergroup within the East Pilbara Terrane. Many Hadean zircons identified in Western Australia are from the Jack Hills meta-conglomerate of the Narryer Formation in the neighboring Yilgarn Craton (Wilde *et al.*, 2001, see above). The significant overlap between our Hf isotope data and those of the Yilgarn Craton, together with the occurrence of an Hadean zircon crystal preserved in the Boolgeeda diamictite with similar age and Hf isotopic signature than the Jack Hills zircons (Whitehouse *et al.*, 2017 and references therein) makes the Yilgarn Craton a possible source material for the Boolgeeda glacial horizon. Available paleogeographic reconstructions based on paleomagnetic data and cratonization history suggest that during the Paleoproterozoic, the Yilgarn and Pilbara cratons were part of two different supercratons, the Zimgarn comprising the Zimbabwe and Yilgarn cratons (Smirnov *et al.*, 2013), and the Vaalbara composed of the Kaapvaal and Pilbara cratons (Cheney, 1996; de Kock *et al.*, 2009). This argues against Yilgarn and Zimbabwe cratons as a source for the Turee Creek and Boolgeeda zircons (Fig. 5c and d). However, Gumsley *et al.* (2017) recently proposed that despite their dissimilar cratonization history, the Zimbabwe and Yilgarn cratons may have been part of a much larger supercraton, the Supervaalbara, which comprised Superior, Wyoming, Hearne, Kola, Karelia, Kaapvaal, and Pilbara. Considering that the 2.45 Ga old Boolgeeda glacial diamictite has been correlated with several glacial horizons in South Africa (pre-2.43 Ga Makganyene Formation of the Griqualand West region), North America (2.45 Ga Ramsay Lake Formation of the Superior Province) and Arctic Europe (2.43 Ga

Urkkavaara and Polisarka Formations of Fennoscandian Craton), Caquineau et al., (2018) proposed that it could represent a global glacial event. Accordingly, although the Yilgarn craton may have been located at a latitude very different from that of the Turee Creek Basin during the Paleoproterozoic, it cannot be excluded that the Hadean zircon preserved in the Boolgeeda diamictite has been transported by icesheets over significant distances.

## 8. Conclusions

Our zircon U-Pb-Hf isotopic study indicates that Neoarchean (to Mesoarchean) mantle-derived material was the major detrital component delivered to the Hamersley basin during deposition of the glacial and non-glacial sediments of the Boolgeeda Iron Formation and the Turee Creek Group, as well as the overlying Beasley River Quartzite. In this study, we discovered the first occurrence of a Hadean zircon grain in the Pilbara Craton. Rare materials older than 3.2 Ga were recycled within the Turee Creek basin. Our data highlight a much larger proportion of mantle-derived source materials than in comparable cratons and basins, for which zircon Hf isotopic data are available. The predominantly supra-chondritic Hf isotopic signatures can be explained by a relatively large proportion of subaerial volcanic rocks during deposition of the underlying 2.45 – 2.78 Ga Hamersley and Fortescue groups. A local origin of the material delivered to the Turee Creek basin is in agreement with an intracontinental depositional setting. The massive emplacement of subaerial LIPs in the Hamersley and Fortescue groups during the Neoarchean followed by subsequent erosion and alteration attending the formation of the Turee Creek basin may have triggered the rise of atmospheric oxygen and associated Paleoproterozoic glacial events.

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## Figure captions

Figure 1: Localization of the Pilbara Craton in Western Australia (a). Geological maps of (b) the Pilbara Craton and Hamersley Basin in Western Australia with Turee Creek Group outcrops in yellow and (c) the Hardey Syncline area with locations of the TCDP drill cores and surface samples (showed with numbers from 1 to 5) analyzed in this study (Modified after Martin et al. (2000) and Van Kranendonk et al. (2015).

Figure 2: Stratigraphic logs of (a) the Mount Bruce Supergroup, which comprises the Fortescue, Hamersley, and Turee Creek groups, and the overlying lower Wyloo Group with geochronological constraints, (b) the complete section of Turee Creek Group in the Hardey Syncline, (c) the Turee Creek Group section at the Boundary Ridge locality, (d) drill core TCDP1, (e) drill core TCDP2, and (f) the upper Kazput cross section (see Fig. 1 b). (a) Modified after Trendall *et al.* (2004) and Philippot *et al.* (2018), (b, d, e) after Philippot *et al.* (2018) and (c, f) after Caquineau *et al.* (2018). Samples analyzed for their Hf isotope compositions include from bottom to top: (1) Boolgeeda diamictite (T1-152, Pi-09.07, Pi-09.08), (2) a sandstone layer of the Kungarra Formation (T2-332) and diamictites of the Meteorite Bore Member (T2-272, T2-205, T2-169 and T2-130), (3) second Kungarra diamictite MBM2 (Pi-15.41, Pi-15.42) (4) two massive sandstones of the upper Kazput section (Pi-15.15, Pi-15.16, Pi-15.17) and (5) Beasley River Quartzite (Pi-15.02).

Abbreviations: WoD, Wooly Dolomite, CSB, Cheela Springs Basalt, BRQ, Beasley River Quartzite, Ka, Kazput Formation, Kb, Koolbye Formation, MBM, Meteorite Bore Member, Ku, Kungarra Formation, TCG, Turee Creek Group, Bo, Boolgeeda Iron Formation, WR, Woongarra Rhyolite, WW, Weeli Wolli Formation, B IF, Brockman Iron Formation, MSW, Mount Mac Rae Shale – Mt. Sylvia Formation – Wittenoon Formation, M IF, Marra Mamba Iron Formation, Jee, Jeerinah Formation, Mad/Bu, Maddina Basalt/Bunjinah Formation, Tu/Py, Tumbiana Formation/Pyradie Formation, Ky/Bo, Kylena Formation/Boongal Formation, MRB, Mount Roe Basalt, Be, Bellary Formation. References for the geochronological constraints are: (1) Müller *et al.* (2005), (2) Martin *et al.*(1998), (3) Philippot *et al.* (2018), (4) Caquineau *et al.* (2018), (5) Trendall *et al.* (2004), (6) Arndt *et al.* (1991).

Figure 3: $\varepsilon_{Hf}(t)$vs age diagrams for each sample: (a) Boolgeeda diamictite, (b) MBM diamictite, (c) second Kungarra diamictite, (d) upper Kazput sandstones, (e) Beasley River Quartzite, (f) all data. CHUR, Chondritic Uniform Reservoir, DM, Depleted Mantle. The DM evolution line corresponds to a $^{176}Lu/^{177}Hf$ ratio of 0.03933 (Blichert-Toft and Puchtel, 2010) evolving from the chondritic initial composition starting at 4567 Ma (Iizuka *et al.*, 2015).The data can be confined between two model age lines at *c.a.* 2.54 Ga and 3.20 Ga. Hf model age lines are represented for a $^{176}Lu/^{177}Hf$ ratio of 0.015 typical of bulk continental crust (Griffin et al., 2004).

Figure 4: A Hadean zircon in the Pilbara Craton. (a) U-Pb Concordia plot of zircon analyses showing an upper intercept at 4013 ± 69 Ma and a Concordia date of 3998 ± 15 Ma. Inset: cathodoluminescence image of the grain with laser spots indicated. (b) $\varepsilon_{Hf}(t)$ vs. age diagrams of the Hadean zircon with model age lines for two different natures of crust, a felsic

crust with a $^{176}Lu/^{177}Hf$ ratio of 0.015 (Griffin *et al.*, 2004), and a mafic crust with a $^{176}Lu/^{177}Hf$ of 0.026 (Blichert-Toft and Albarède, 2008). The filtered domain of Jack Hills zircon analyses after Whitehouse *et al.* (2017) is represented by the yellow box.

Figure 5: Comparison of $\varepsilon_{Hf}$(t) data obtained in this study with literature data. The Boolgeeda diamictite and Turee Creek Group data are represented as grey squares in all diagrams. (a) Halfway Gneiss of the Glenburgh Terrane, (Johnson *et al.*, 2011), (b) Pilbara Craton, (Kemp *et al.*, 2015; Gardiner *et al.*, 2017 and 2019; Petersson *et al.*, 2019), (c) The northern part of the Yilgarn Craton, (Griffin *et al.*, 2004). (d,) South Africa: Magaliesberg Quartzite, (Zeh *et al.*, 2016), Kalahari Craton (Zeh *et al.*, 2009). (e) Superior craton (Corfu and Noble, 1992; Corfu and Stott, 1993 and 1996; Davis *et al.*, 2005).

## Tables Content

**Table1.** Zircon Lu-Hf isotope data obtained by in *situ* Laser Ablation MC-ICP-MS.

**Table 2.** Zircon U-Th-Pb data from the Hadean zircon crystal obtained by in situ Laser Ablation ICP-MS.

**Supplementary Table S1.** Detail of analytical conditions for Lu-Hf isotope analysis by *in situ* Laser Ablation MC-ICP-MS. Lu-Hf isotope data on natural GJ-1, 91500 and Mud Tank zircon reference materials and synthetic zircons doped with hafnium and rare earth elements (Fischer et al., 2011b) are included.