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A new 3.59 Ga magmatic suite and a chondritic source to the east Pilbara Craton

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ABSTRACT
The Pilbara Craton, Western Australia hosts one of the best preserved Paleoarchean granite-greenstone terrains on Earth, and is inferred to have developed on an older (> 3.8 Ga), possibly Hadean, continental substrate. Such ancient crust has, however, never been identified in outcrop. Here, we show that metamorphosed gabbroic, leucogabbroic and anorthositic rocks of the South Daltons area, in the western part of the Shaw Granitic Complex, formed at 3.59–3.58 Ga and were intruded by granitic magma at 3.44 Ga. The 3.59–3.58 Ga gabbroic rocks, here named the Mount Webber Gabbro, represent the oldest, unambiguous igneous rock emplacement in the Pilbara Craton and significantly predates the oldest volcanic activity of the 3.53–3.23 Ga Pilbara Supergroup within the East Pilbara Terrane. We interpret the Mount Webber Gabbro samples to represent fragments of a dismembered layered mafic intrusion. Mantle-like zircon δ¹⁸O and Hf isotope signatures indicate derivation from a chondritic to near chondritic mantle at ~3.59 Ga, and do not support the existence of a > 3.8 Ga basement to the East Pilbara Terrane. These results strengthen the notion of an approximately chondritic > 3.5 Ga mantle beneath the Pilbara Craton, and provide further evidence that recent estimates of Archean stabilised continental volumes, based on the assumption of crust extraction from a global, convecting depleted mantle reservoir, may be overestimated.

1. Introduction
Granite-greenstone terranes are the hallmark of Archean cratons, but it is debated whether they formed entirely from juvenile mantle-derived material (e.g. Hiess et al., 2009; Kemp et al., 2015; Fisher and Vervoort, 2018) or developed through reworking of an older continental substrate (e.g. Hickman, 1983; Collins et al., 1998; Kamber et al., 2005; Smithies et al., 2005; Van Kranendonk et al., 2007; Gardiner et al., 2017). This debate has implications for the initial growth, volume and composition of the earliest continental nuclei on Earth.

The East Pilbara Terrane of the Pilbara Craton, Western Australia, hosts one of the best preserved Paleoarchean granite-greenstone terranes on Earth (Fig. 1). The komatiitic to basaltic lavas, stratigraphically part of the Pilbara Supergroup, are inferred to have extruded onto an older (> 3.53 Ga) substrate of continental crust (e.g. Van Kranendonk et al., 2007; Tessalina et al., 2010). This interpretation is based on several, largely indirect, lines of evidence. For example, in the Warrawagine Granitic Complex, tonalitic gneiss enclaves containing 3.66–3.58 Ga zircon cores are found within younger ~3.42–3.24 Ga monzogranite and granodiorite (see Kemp et al., 2015). Enclaves of ca. 3.58 Ga gabbronoritc anorthosite in 3.43 Ga granitic rocks of the Shaw Granitic Complex have also been reported (McNaughton et al., 1988, in a conference abstract). Although the gabbronorite enclaves are not necessarily of continental affinity, a magmatic event of this age is corroborated by trondhjemitic and granodioritic gneisses in the Mucan Granitic Complex, dated to 3.591 ± 0.036 Ga and 3.576 ± 0.022 Ga respectively (zircon U-Pb, Wiemer et al., 2018). A 3.724 Ga xenocrystic zircon in the Panorama Formation (Thorpe et al., 1992) and 3.71–3.50 Ga detrital zircon grains in the Farrel Quartzite (Sheppard et al., 2017) could suggest the existence of even older crust within the east Pilbara Craton. Sedimentary successions across the Pilbara Craton contain detrital zircon that predates the most ancient rocks in the area by up to 300 million years (Van Kranendonk et al., 2007; Hickman et al., 2015).
An ancient continental crust has also been inferred from trace element modelling (Green et al., 2000) and from Nd isotope compositions of basaltic (Van Kranendonk et al., 2007; Tessalina et al., 2010; Gardiner et al., 2017) and granitic rocks (e.g. Hamilton et al., 1981; Jahn et al., 1981; Gruau et al., 1987; Bickle et al., 1989, 1993; Smithies et al., 2003; Smithies et al., 2007; Champion, 2013; Champion and Huston, 2016; Gardiner et al., 2017). Eoarchean (ca. 3.8–3.6 Ga) depleted mantle model ages based on zircon Hf isotopes are also reported from granitic rocks of the Mount Edgar Batholith (Gardiner et al., 2017; see their Table 1). So far, the oldest (>3.6 Ga) Nd and Hf model ages have been reported from northwestern Yule, northern Shaw, Mount Edgar, Muccan, and eastern Warrawagine granitic complexes (see Van Kranendonk et al., 2007; Gardiner et al., 2017). Granitic rocks of >3.22 Ga granitic supersuites in the East Pilbara Terrane (Callina, Tambina, Emu Pool and Cleland) yield Nd model ages of ~4.1–3.4 Ga (Smithies et al., 2003, 2007;
Champion, 2013) and some of the more recent studies suggest that the cryptic ancient crust might even be Hadean (4.5–4.0 Ga; e.g. Tessalina et al., 2010).

It is notable, however, that outcrops of unambiguously Eoarchean to Hadean continental crust have never been identified in the Pilbara Craton; the previous existence of such ancient crust is largely inferred from the older detrital zircon relics noted above (which are of uncertain origin; Kemp et al., 2015), and model crust formation ages that hinge on the interpretation of continental separation from a depleted (MORB-source) mantle from 4.5 Ga to the present day (e.g. McCulloch and Wasserburg, 1978; DePaolo, 1980). Such an assumption of continental formation from a > 3.5 Ga strongly depleted mantle, particularly for Lu-Hf isotopes, is based on few robust observational data and might lack justification (e.g., Kemp et al., 2015; Fisher and Vervoort, 2018). If continental crust was extracted from a less depleted mantle source reservoir, such as convecting asthenosphere of chondritic composition, these model ages, and the inferences of ancient basement in the Pilbara Craton, would be invalid. More widely, a number of studies (e.g., Vervoort and Kemp, 2016) have noted that, for Hf isotopes, the assumption of a strongly depleted mantle source will produce a bias towards model crust formation ages that are too old, leading to erroneously high estimates of ancient continental volumes.

Here, we report new zircon U–Pb–O–Hf isotope data from meta-igneous (mostly gabbroic) rocks within the South Daltons pluton in the western part of the Shaw Granitic Complex, East Pilbara Terrane. With these results, we identify a new 3.59–3.58 Ga magmatic event in the Shaw Granitic Complex, here named the Mount Webber event, which predates the previously oldest known single component igneous rocks in the Pilbara Craton. The 3.59–3.58 and ca. 3.44 Ga intrusions have zircon O and Hf isotope signatures that are consistent with generation from a near-chondritic mantle source. These results add another significant element to the magmatic history of the Pilbara Craton and provide no evidence for the existence of a > 3.8 Ga basement.

2. Regional geology

2.1. The Pilbara Craton

The Pilbara Craton consists of three main lithotectonic elements, the East Pilbara Terrane (3.53–3.22 Ga), the West Pilbara Supergroup (3.28–3.07 Ga), and the De Grey Superbasin (3.06–2.93 Ga; Figs. 1 and 2). About 60,000 km² of the craton is exposed in the northeast, but three-quarters of the craton is unconformably overlain to the south by volcanic and sedimentary Neoarchean–Paleoproterozoic rocks of the Mount Bruce Supergroup (Hickman, 2004).

The East Pilbara Terrane is composed of three volcano-sedimentary groups (Warrawoona, Kelly, and Sulphur Springs) deposited between ~3.53 and 3.23 Ga, collectively known as the Pilbara Supergroup, that flank and overlie large (35–120 km across), granitic complexes. Each individual domal structure of the East Pilbara Terrane comprises a central granitic complex flanked by an adjacent greenstone belt (Van Kranendonk et al., 2002). Eleven such granite–greenstone domes make up the East Pilbara Terrane, and are separated by boundary faults, most of which are located between the greenstone belts of adjacent domes (Van Kranendonk and Collins, 1998). This crustal architecture forms the archetype of the dome-and-keel pattern of Archean granite-greenstone terranes (Van Kranendonk et al., 2007).

The largely coherent eruptive units of the Pilbara Supergroup are dominated by tholeiitic and komatiitic basalts interlayered with thin sedimentary horizons and locally including thick felsic volcanic units; komatiite units are less common, although locally up to 400 m thick (e.g. Hickman et al., 1990; Smithies et al., 2007; Van Kranendonk et al., 2007; Hickman, 2012).

The lowermost contact of the supergroup is intruded out by younger granitic bodies, and the uppermost parts of several of the eruptive units have been eroded, such that the maximum original depositional thickness is unknown. However, no stratigraphic repetition has been demonstrated (Van Kranendonk et al., 2001, 2004, 2007; Hickman and Van Kranendonk, 2004), despite numerous geochemical traverses across the major greenstone belts (Glikson and Hickman, 1981a, 1981b; Glikson et al., 1986; Smithies et al., 2007) and geochronological mapping of the Pilbara Supergroup (Van Kranendonk et al., 2002, 2006, Fig. 2).

Multiple pulses of voluminous magmatic intrusions (3.49–3.46, 3.45–3.42 Ga, 3.32–3.29 Ga, 3.25–3.22 Ga, 2.95–2.92 Ga, and 2.85–2.83 Ga) over hundreds of millions of years produced the granitic complexes of the east Pilbara Craton. The oldest suites of voluminous granitic magmatism (3.49–3.42 Ga) are mainly represented by tonalite–trondhjemite–granodiorite (TTG). They are especially well preserved within the Shaw Granitic Complex, but also form parts of the Carlindi, Yule, Mount Edgar, Corunna Downs, Warrawagine and Muccan Granitic Complexes (Figs. 1 and 2; Buick et al., 1995; Nelson, 1999a; Bagas et al., 2005; Van Kranendonk et al., 2007; Gardiner et al., 2017). These older Paleoarchean granitic rocks vary from being moderately deformed (e.g. the northern parts of the Shaw Granitic Complex; Bickle et al., 1993), to highly deformed migmatitic gneisses (e.g. Shaw and Mt. Edgar Granitic Complexes; Bickle et al., 1993; Collins, 1993). Such gneisses are found as numerous enclaves in younger granitic rocks. The pre-3.42 Ga granitic rocks have been divided into the 3.49–3.46 Ga Callina Supersuite and the 3.45–3.42 Ga Tambina Supersuite: the Callina Supersuite is contemporaneous with the felsic extrusive units within the Duffer Formation; the Tambina Supersuite and rhyolitic Panorama Formation also form a contemporaneous intrusive/extrusive pair (Van Kranendonk et al., 2006).

Younger Paleoarchean granitic intrusions of the East Pilbara Terrane became increasingly potassic, and are represented by granodiorite and monzogranite of the 3.32–3.29 Ga Emu Pool Supersuite and by monzogranite and syenogranite of the 3.25–3.22 Ga Cleland Supersuite. Following rifting and breakup of the East Pilbara Terrane at ~3.20 Ga, margins of the separated terranes were locally intruded by tonalite and granodiorite of the Mount Billroth Supersuite (Hickman, 2012, 2016).

Three younger terranes make up the West Pilbara Supergroup, the ~3.28–3.25 Ga Karratha, the ~3.20 Ga Regal, and the ~3.13–3.11 Ga Sholl Terranes (Fig. 1). The Regal Terrane (basaltic crust formed in a rift basin) was obducted onto the Karratha Terrane between 3.16 and 3.07 Ga, and the Sholl and Karratha Terranes were tectonically juxtaposed along the Sholl Shear Zone between 3.09 and 3.07 Ga. The West Pilbara Supergroup then collided with the East Pilbara Terrane at ~3.07 Ga (Van Kranendonk et al., 2010).

Two main extensional events have affected the Pilbara Craton. The first was initiated at ~3.2 Ga, separating the Karratha Terrane from the East Pilbara Terrane, and is associated with deposition of the ~3.19 Ga Soanewsie Group clastic succession, which in turn is overlain by a substantial basaltic volcanic sequence. Following the amalgamation of the East Pilbara Terrane and West Pilbara Supergroup, a second extensional event led to accumulation and deposition of volcanic and sedimentary rocks of the 3.06–2.93 Ga De Grey Supergroup (Fig. 1).

2.2. The Shaw Granitic Complex

The Shaw Granitic Complex is a > 50 km diameter assemblage of granitic intrusions with emplacement ages spanning ~650 Ma (Fig. 3A). Stratigraphic younging occurs within the greenstone sequences away from the granitic complex. At the present level of exposure, the complex approximates to a cross-section in which the granitic margins grade downward into progressively more sheared variants forming a sheeted granitic complex, interleaved with older greenstones.

The 3.5–3.3 Ga calc-alkaline plutonic suite in the Shaw Granitic Complex is predominantly highly sheared, folded and metamorphosed to the upper amphibolite facies (Bickle et al., 1980; Bickle et al., 1989; Pawley et al., 2004; Van Kranendonk et al., 2007). This has formed a
Fig. 2. Space-time chart outlining the evolution of the exposed Pilbara Craton (modified after Hickman et al., 2010). The stratigraphic positions of samples analysed in this study are indicated by a red star symbol. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
greyish, largely migmatitic gneiss, which is intruded by several large plutons of variably deformed granitic rocks and post-tectonic granites in the central part of the complex (Fig. 3). However, the northern part of the Shaw Granitic Complex includes relatively undeformed biotite granodiorite, with subordinate tonalite and monzogranite, usually with preserved igneous textures (Bickle et al., 1983). These rocks also have sharp, discordant intrusive contacts with the surrounding greenstone formations.

Bickle et al. (1983) determined the age of older, predominantly granodioritic rocks of the Shaw Granitic Complex to 3.499 ± 0.022 Ga using a Pb-Pb whole-rock isochron, and McNaughton et al. (1988) dated rocks from the same suite to 3.493 ± 0.004 Ga and 3.467 ± 0.006 Ga respectively using ion-microprobe U-Pb on zircon. A granodiorite in the South Daltons area in the western part of the Shaw Granitic Complex was dated by McNaughton et al. (1993) to 3.431 ± 0.004 Ga using ion microprobe on zircon. These igneous events are not restricted to the Shaw Granitic Complex, and the ~3.45 Ga event is coeval with formation of the Tambina Supersuite and eruption of the Panorama Formation. Furthermore, the North Pole Monzogranite, which intrudes a greenstone belt 20 km north of the Shaw Granitic Complex, is virtually coeval with the older North Shaw Suite intrusions at 3.459 ± 0.018 Ga (U-Pb, zircon: Thorpe et al., 1992).

Granitic rocks of the Tambina Supersuite constitute a major part of the Shaw Granitic Complex, and are interpreted to be derived through melting of Callina Supersuite protoliths (Pawley et al., 2004). There is a close temporal relationship between the less deformed intrusive rocks of the North Shaw Suite and the grey gneisses within the complex (Williams et al., 1983; Pawley et al., 2004). Williams et al. (1983) dated a zircon core from the grey gneiss to 3.485 ± 0.030 Ga (SIMS, zircon U-Pb). This suggests a common source to these rocks, and that the grey gneisses are derived from the North Shaw Suite, a relationship otherwise obscured by poor exposure.

Two regional scale metamorphic events have been identified in the Shaw Granitic Complex (Bickle et al., 1985), a ~3.2 Ga high-P kyanite-sillimanite event and a ~2.95–2.84 Ga lower-P andalusite-sillimanite event (Wijbrans and McDougall, 1987).

The gabbroic samples examined in the present study comprise a raft several hundred metres in length within the South Daltons pluton in the western part of the Shaw Granitic Complex (Fig. 3). These rocks are described below.

Fig. 3. A) Simplified geology of the Shaw Granitic Complex. Modified after Bickle et al. (1983). B) Simplified geology of the South Daltons area, western north Shaw Granitic Complex. Based on the interactive geological map of the Geological Survey of Western Australia (GeoVIEW.WA).
3. Analytical methods

3.1. Major and trace element analyses

All samples were analysed by Bureau Veritas Minerals Pty Ltd. in Perth. The samples were cast using a 66:34 (Lithium Tetraborate 66%/Lithium Metaborate 34%) flux with 4% Lithium nitrate added to form a glass bead. Major elements were determined by X-ray Fluorescence. Trace elements were determined by Laser Ablation Inductively Coupled Plasma Mass Spectrometry on a fused bead. Loss on Ignition was determined using a robotic TGA system with furnaces set to 110 °C and 1000 °C (LOI1000). A sub-sample was digested with sulphuric and hydrofluoric acids and FeO determined via titration. Accuracy and precision of the analyses were monitored using geochemical reference rock powders (Kerba Monzogranite and Bunbury Basalt; Morris, 2007), inserted as unknown samples. All major and trace element data, including that obtained from the reference materials, are found in Supplementary Table A.

3.2. Zircon U-Pb SIMS age determination

Secondary ionisation mass spectrometry (SIMS) U–Th–Pb isotopic analyses were carried out using a large geometry Cameca IMS1280 instrument at the Swedish Museum of Natural History. The instrument set up follows that described by Whitehouse et al. (1999), Whitehouse and Kamber (2005) and references therein. An O2+ primary beam with 23 kV incident energy (−13 kV primary, +10 kV secondary) was used for sputtering. For this study, the primary beam was operated in aperture illumination (Köhler) mode yielding a ~15–20 μm spot. Pre-sputtering with a 25 μm raster for 120 s, centring of the secondary ion beam in the 4000 μm field aperture (FA), mass calibration optimisation, and optimisation of the secondary beam energy distribution was performed automatically for each run, FA, and energy adjustment using the 90Zr2+O16 peak at nominal mass 196. Mass calibration of all peaks in the mono-collection sequence was performed at the start of each session; within run mass calibration optimisation was performed using the 90Zr2+O16 peak, applying a shift to all other species. A mass resolution (M/ΔM) of ~5400 was used to ensure adequate separation of Pb isotope peaks from nearby HSIs+ species. Ion signals were detected using the axial ion-counting electron multiplier. All analyses were run in fully automated chain sequences. Data reduction assumes a power law relationship between Pb+/U+ and UO2+/U+ ratios with an empirically derived slope in order to calculate actual Pb/U ratios based on those in the 91500 reference zircon (206Pb/238U = 0.17917 ± 8; Wiedenbeck et al., 1995). U concentration and Th/U ratio are also referenced to the 91500 standard (81.2 ppm U, Th/U = 0.344; Wiedenbeck et al., 1995).

A common Pb correction was applied if the 206Pb signal statistically exceeded average background and assumed a 207Pb/206Pb ratio of 0.83 (equivalent to present day Stacey and Kramers, 1975, model terrestrial Pb). Decay constants follow the recommendations of Jaffey et al. (1971) and compiled by Steiger and Jäger (1977). All age calculations were done in Isoplot 4.15 (Ludwig, 2008) and quoted at 2σ uncertainty level. All internal errors and external uncertainty in the standard analyses are propagated into the final quoted uncertainties. Systematic uncertainties highlighted by interlaboratory experiments are not propagated. Zircon OGC/OG1 from the Owens Gully Diorite (207Pb/206Pb age = 3465.4 ± 0.6 Ma; Stern et al., 2009) was also periodically analysed to monitor data quality and accuracy. The Owens Gully Diorite, from the Mount Edgar Granitic Complex (Nelson, 2005), is one of the oldest and best-studied intrusive rocks of the Pilbara Craton, and thus is an appropriate comparator for the present study. Sixteen analyses from these zircons returned a 207Pb/206Pb age of 3465.0 ± 2.2 Ma (95% conf., MSWD = 1.5), identical to the values provided by Stern et al. (2009). All results are presented in Supplementary Table B.

3.3. Ion microprobe oxygen isotope analysis in zircon

Oxygen isotope ratios (18O/16O) in zircon were determined using a Cameca IMS 1280 multi-collector ion microprobe hosted by the Centre for Microscopy, Characterisation and Analysis (CMCA), University of Western Australia (UWA). After U-Pb analyses, the gold coating was removed by light polishing. The sample mounts were then carefully cleaned with detergent, distilled water and ethanol in an ultrasonic bath and re-coated with gold (30 nm in thickness) prior to SIMS O isotope analyses.

The sample surface was sputtered over a 10 × 10 μm area with a 10 kV, Gaussian Cs⁺ beam with an intensity of ~3 nA and total impact energy of 20 keV. An electron gun was used to ensure charge compensation during the analyses. Secondary ions were energy filtered using a 30 eV band pass with a 5 eV gap towards the high-energy side. 16O and 18O were collected simultaneously in Faraday cup detectors fitted with 1010Ω (L2) and 1011Ω (H1) resistors, respectively, and operating at a mass resolution of ~2430. The magnetic field was regulated using NMR control.

Each analysis included pre-sputtering of a 15 × 15 μm area during 30s, and automatic centring of the secondary ions in the field aperture, contrast aperture and entrance slit. Each analysis then consisted of 20 four-second cycles, which gave an average internal precision of ~0.16‰ (2 SE). The analytical session was monitored in terms of drift using at least two bracketing standards every 5 to 6 sample analyses. Instrumental mass fractionation (IMF) was corrected using zircon Temora 2 (8.2‰; Black et al., 2004) following the procedure described in Kita et al. (2009). The spot-to-spot reproducibility was 0.2–0.3‰ (2 SD) on Temora 2 during the analytical session.

Uncertainty on each δ18O spot was calculated by propagating the errors in the instrumental mass fractionation determination, which includes the standard deviation of the average oxygen isotope ratio measured on the primary standard during the session, and internal uncertainty of each sample data point. Raw 18O/16O ratios and corrected δ18O (quoted with respect to Vienna Standard Mean Ocean Water, VSMOW) are presented in Supplementary Table C.

3.4. LA-ICP-MS zircon Lu-Hf analyses

Lu-Hf analyses were carried out during two sessions at the School of Earth Sciences, The University of Western Australia, using a 193 nm Cetac Analyte G2 excimer laser installed with a two-volume HelEx2 sample cell, and a Thermo-Scientific Neptune Plus MultiCollector ICP-MS. Where possible, the Lu-Hf spot overlapped pits from both the U-Pb and O isotope analysis. Circular spots with a diameter of 40 μm and 50 μm were used. Each analysis was initiated by a 30 s electronic onpeak baseline followed by an ablation period of 60 s involving 60 integration cycles of one second each. A laser pulse repetition rate of 4 Hz was used and the laser energy was held at ~5.5 J/cm2, which equates to an ablation rate of ~0.05 μm per pulse for zircon. Helium carrier gas (1.0 l/min) was used to transport the ablated particles from the sample chamber. This was combined with argon gas (flow rate c. 0.6 l/min) and nitrogen (~0.012 l/min) further downstream before entering the argon plasma torch. Masses 171Yb, 173Yb, 175Lu, 176(Hf + Lu + Yb), 175Hf, 179Hf and 180(Hf + W + Ta) were measured simultaneously by Faraday detectors. Isobaric interference of 177Yb and 176Lu on 176Hf was calculated using the measured intensities of 171Yb and 175Lu along with known isotopic ratios of 176Yb/177Yb = 0.897145 (Segal et al., 2003) and 176Lu/177Lu = 0.02655 (Vervoort et al., 2004). Mass bias corrections were calculated using the exponential law. For calculations of βHf, measured intensities of 175Hf and 177Hf and a canonical 175Hf/177Hf ratio of 0.7325 were used (Patchett and Tatsutomo, 1980). εYb was calculated using the measured intensities of 172Yb and 171Yb and a 172Yb/171Yb ratio of 1.130172 (Segal et al., 2003). Mass bias behaviour
of Lu was assumed to be identical to Yb. Five zircon references were used for quality control, FC-1, Mud Tank, Temora 2, 91500 and OGC. Analysed $^{176}\text{Hf}/^{177}\text{Hf}$ ratios of the sample zircon were normalised based on a comparison between the average of analysed $^{176}\text{Hf}/^{177}\text{Hf}$ of Mud Tank zircon measured in a given session and its reported $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.282507 ± 0.000006 determined by solution analysis (Woodhead and Hergt, 2005), itself reported relative to JMC475 $^{176}\text{Hf}/^{177}\text{Hf}=0.282160$. Session 1, yielded a Mud Tank zircon $^{176}\text{Hf}/^{177}\text{Hf}=0.282491 ± 0.000011$ (n=18, 2SD) and session 2 yielded a $^{176}\text{Hf}/^{177}\text{Hf}=0.282491 ± 0.000011$ (n=26, 2 SD), both within the uncertainties of the recommended values of Woodhead and Hergt (2005). All analyses were screened for within run variability to decipher any heterogeneities within the analysed zircon grains. Calculations of $\varepsilon\text{Hf}$ were done using the interpreted pooled igneous crystallisation age of the rock (Whitehouse and Kemp, 2010), $\lambda_{^{176}\text{Lu}}=1.867 \times 10^{-11}\ \text{a}^{-1}$ (Scherer et al., 2001; Söderlund et al., 2004), $(^{176}\text{Lu}/^{177}\text{Hf})_{\text{CHUR}}=0.0336$ and $(^{176}\text{Hf}/^{177}\text{Hf})_{\text{CHUR}}=0.282785 ± 11$ (Bouvier et al., 2008). All zircon Lu-Hf data are given in Supplementary Table D (unknowns) and Supplementary Table E (zircon reference materials).

4. Results

4.1. Field relations

Five samples were collected from a small (200 m × 200 m) outcrop area within the middle portion of the South Daltons pluton. Here, strongly foliated and complexly interspersed leucogabbro, gabbroic and dioritic to tonalitic exposures flank thin strips of ultramafic rock, which define a low, approximately E–W trending ridge that persists for several hundred metres (Fig. 4). This lithological assemblage is crosscut by sheets of leucogranite, and enclosed by a coarse-grained, foliated biotite granodiorite, in which pinkish K-feldspar phenocrysts impart a ‘speckled’ appearance.

The leucogabbroic rocks appear to enclose the ultramafic pods and are restricted to within ~ 50 m either side of these (a sample from the westernmost ultramafic pod is dominantly hornblende, with aligned sheaves of clinohlore). Leucogabbros are lithologically heterogeneous on outcrop scale, varying in grain size and in the proportions of mafic versus felsic minerals. The dominant variety is medium to coarse-grained and comprises ~60% altered plagioclase and 40% blocky hornblende (see below). Variants range from coarser grained and rutile-bearing to medium grained with spindly hornblende; some of these show a diffuse layering and grade locally into anorthosite with decreasing hornblende content (Fig. 5A). The tight packing of constituent plagioclase prisms is visible on the pale brownish-orange weathered surfaces of these rocks. Coarser, pegmatitic domains (5–10 cm long) with hornblende prisms to several centimetres long occur in several places in the leucogabbros, particularly immediately adjacent to the ultramafic bodies, where the leucogabbros have a ‘hybird’ appearance (Fig. 5B). Hornblende-rich metagabbro crops out as several prominent elongate lenses (up to 40 m long) in the central part of the outcrop tract (Fig. 5C–D), whereas amphibolite defines small bodies (several m²). Boulders of the biotite granodiorite occur on the north and south sides of the ultramafic–leucogabbroic strip, although the nature of the contact between these and the leucogabbros is not clear. On the south side of the ultramafic pods, dark, even-grained diorite to tonalite slabs crop out sporadically and contain thin felsic veins that are locally intensely folded (Fig. 5E). Dioritic-tonalitic rocks are interlayered with, and intruded by, metre-scale leucogranitic sheets, and are covered by alluvium further downslope (south) from the ultramafic pods. All gabroic samples studied here are metamorphosed and contain deformation fabrics, but the meta- prefix is omitted below for brevity. Photographs of representative hand-samples of each rock type analysed in this study are in Supplementary Fig. A.

4.2. Petrography

4.2.1. Leucogabbros (12TKPB01, 15TKPB26, 15TKPB27)

Sample 12TKPB01 is from the central part of the leucogabbroic strip and is representative of the main leucogabbroic rock type in the area. This is a moderately coarse, even-grained rock dominated by altered, chalky to greenish plagioclase, with a strong foliation defined by prisms (to 6 mm) and clotty aggregates of hornblende (Fig. 6A). Sheaves and plates of coarse white mica line some foliation surfaces. In thin section, plagioclase is almost completely altered to sericite and includes granules of epidote and ragged plates of muscovite and chlorite (Fig. 6A). Former blocky plagioclase laths show euhedral faces against hornblende masses (generally unaltered, pale yellow-green to blue-green), which comprise aggregates with polygonal internal grain boundaries. Irregularly-shaped blebs of quartz and rutile are sparsely disseminated. Metamict allanite grains are enclosed within hornblende.

![Fig. 4. Simplified sketch map of the sample area showing the general distribution of the various lithologies analysed in this study.](image-url)
Zircon grains are subhedral to anhedral, equant to very elongate, and vary in size from 50×100μm to 150×400μm. Many of the grains are irregularly shaped without sharply defined crystal faces, although most appear to be fragments of larger grains. They vary in colour from pale brown to dark brown and turbid, with fine growth zoning visible in the outer parts of the grain in transmitted light. In cathodoluminescence (CL) images, these zircon grains have unzoned centres with faint oscillatory zonation preserved towards grain boundaries.

Sample 15TKPB26 is lithologically similar to 12TKPB01 but is distinctly coarser grained, such that lenticular hornblende aggregates reach 3 cm in length (Fig. 6A). In three dimensions, these aggregates are discoid, and the constituent hornblende blades show no linear preferred orientation. Small pinkish granules (to 0.5mm) and elongate aggregates of rutile are conspicuous. In thin section, these are seen to be associated with distorted, pale brown flakes of biotite, which are now extensively replaced along cleavage traces to birefringent white mica and form clots with epidote. Rutile grains are interstitial to blocky plagioclase grains, and metamict allanite is common (Fig. 6). Zircon grains are subhedral to anhedral and vary in size from 70×100μm to 150×350μm. They show very similar characteristics to zircon grains of 12TKPB01 (Fig. 7).

Medium-grained leucogabbro 15TKPB27 differs from the other samples in containing a higher proportion of plagioclase (aligned pale greenish polycrystalline masses up to 1 cm long) and in that hornblende occurs as small, strongly aligned spindly grains (rarely to 3 mm) or, less...
commonly, small clots of polygonal grains (Fig. 6A). Green-tinted mica flakes are sparse and oriented in the foliation–in thin section, these are either chlorite or white mica strips replacing pale orange biotite (Fig. 6A). Elongate quartz grains, and to a lesser extent rutile, are common (Fig. 6A). The constituent zircon grains are euhedral to subhedral and commonly elongate, although equant grains occur. They vary in size from $50 \times 130 \mu m$ to $200 \times 450 \mu m$ and are light to dark brownish in colour. In CL, two main growth phases are evident, a darker primary phase (cores) and a brighter secondary phase seen as thick rims (e.g. #16, #19 and #25, Fig. 7), both unzoned.

### 4.2.2. Gabbro (15TKPB25, 15TKPB23)

Gabbro 15TKPB23 comprises a discrete east–west oriented body ~40 m long and 5 m wide that lies along the interface between coarse leucogabbro and the biotite granodiorite. It is a dark grey to black, medium, even-grained rock dominated by hornblende, which defines a strong lineation, with subordinate plagioclase. In thin section, hornblende is pale khaki to deep blue-green and forms blocky, irregular-shaped and unaltered grains (most ~1mm) intergrown with heavily seritised plagioclase (Fig. 6B). Quartz is conspicuous as interstitial blebs and inclusions in hornblende. Irregularly shaped aggregates of titanite, commonly with small epidote granules and opaque blebs, are disseminated throughout (Fig. 6B).

Zircon grains in 15TKPB23 are in general slightly rounded and subhedral, but euhedral grains with sharp terminations occur. Most grains are around $50 \times 150 \mu m$ in size and colourless to slightly pale brown. In CL, the zircon grains are broadly zoned varying from concentric (e.g. #04 and #05, Fig. 7) to irregular zoning (e.g. #09 and #11, Fig. 3). Thin (<10μm) CL bright rims are present in most grains and attributed to secondary growth.

Gabbro 15TKPB25 occurs as a series of low (<1 m) boulders that define a pod 10 m long and 6 m wide enclosed within the central part of the leucogabbro tract. It is a dark grey, medium-grained rock comprising aligned hornblende prisms and plagioclase grains. A diffuse layering on the scale of ca. 5–6 cm is defined by variations in modal hornblende abundance (Figs. 5D and 6B). Rutile granules are conspicuous and abundant in hand sample. In thin section, these form irregularly-shaped aggregates interstitial to both hornblende and plagioclase, that are intimately intergrown with an opaque mineral (Fig. 6B). Zircon grains are subhedral to anhedral with very few flat crystal planes. They vary in size from $70 \times 100 \mu m$ to $200 \times 450 \mu m$ and are light to dark brownish in colour. In CL images, a weakly luminescent broad zoning is preserved (e.g. #26, Fig. 7). Slightly more luminescent zircon occurs as thin rims (e.g. #15 and #26, Fig. 7) and patchy domains in most grains (e.g. #01 and #17, Fig. 3).

### 4.3. Geochemistry

A summary of sample locations and geochemical analyses is in Table 1. Major and trace element data are in Supplementary Table A, and zircon U-Pb, O and Lu-Hf data are in Supplementary Tables B–E.

#### 4.3.1. Major and trace elements

The South Daltons gabbroic samples have basaltic SiO2 contents, ranging from 48 to 51 wt%, low to moderate TiO2 of 0.3–1.3 wt% (except 15TKPB25, which has 4.1% TiO2), high Al2O3 of 14–24 wt%, MgO of 4.5–7.0 wt% and K2O ranging between 0.9 and 2.6 wt%. The MgO is slightly lower and K2O is markedly higher in the leucogabbros.
(12TKPB01, 15TKPB26 and 15TKBP27) compared to the hornblende-rich gabbros 15TKPB23 and 15TKPB25. The bulk-rock Mg numbers (Mg# = 100 × molar MgO/(MgO + FeOtot)) are variable and range from 48 (15TKPB23) to 77 (15TKPB27), as are Cr (25–159 ppm) and Ni (52–98 ppm). The conspicuously low Mg# in hornblende-rich gabbro 15TKPB23 reflects greater Fe enrichment (12.1% FeOt) than in the other samples, although this rock has the highest Cr content. Sr contents are variable, from 161 ppm in hornblende-rich gabbro 15TKPB23 to a maximum of 535 ppm in coarse leucogabbro 15TKPB26. Based on their low Zr/P2O5 all samples are tholeiitic in character (Winchester and Floyd, 1976). However, in an AFM plot all leucogabbros plot in the calc-alkaline field, while 15TKPB23 has clear tholeiitic affinity and 15TKPB25 falls on the dividing line between calc-alkaline and tholeiitic (Irvine and Baragar, 1971). The FeO*/MgO versus SiO2 diagram of Miyashiro (1974) classifies all leucogabbros as calc-alkaline, while the hornblende-rich gabbros 15TKPB23 and 15TKPB25 plot as tholeiitic (Fig. 8A–B).

Primitive mantle normalised trace element patterns are LILE enriched and have negative anomalies for Nb, Ta and P relative to neighbouring elements, and positive anomalies for K, Pb and (except gabbro 15TKPB23) Sr (Fig. 9a). Rb varies over a considerable range (35–172 ppm), and Th is enriched (0.98–3.21 ppm), with all samples besides 15TKPB23 having Th concentrations > 2.5 ppm. All rocks are strongly enriched in Cs. Sample 15TKPB25 has a pronounced positive spike in Ti, and subtler negative Nb-Ta anomaly, in accordance with conspicuous modal rutile. The enrichment in fluid-mobile elements is further highlighted on a MORB-normalised diagram using the ordering scheme of Pearce (1983) (Fig. 9B), although here the depletion in Nb-Ta relative to adjacent elements is much less evident; the normalised abundance of these elements instead largely conforms to a sloping negative trend from Rb to Yb.

All leucogabbro samples, and gabbro 15TKPB25, show steeply sloping chondrite normalised REE patterns with enrichment in the LREE and variable relative depletion in the HREE (Fig. 9); leucogabbros 12TKPB1 and 15TKPB27 show the lowest middle to HREE abundances. Gabbro 15TKPB23 is distinctly different in having a much flatter REE pattern. 12TKPB01 and 15TKPB27 have minor positive Eu-anomalies with Eu/Eu* of 1.16 and 1.24, respectively, while remaining samples have negligible Eu anomalies.
4.3.2. U-Pb, O and Lu-Hf isotopes

4.3.2.1. Leucogabbro 12TKPB01. Individual U-Pb analyses of twenty-five grains yield mainly discordant data (n = 21/25 at the 2σ level). One concordant analysis (#11) plots slightly below the main cluster of concordant analyses indicating slight Pb-loss. The five analyses with the oldest 207Pb/206Pb-dates, ranging between 3.593 and 3.583 Ga, including two < 2% discordant analyses (#05 and #12), yield a 207Pb/206Pb-weighted average of 3.588 ± 0.005 Ga (95% confidence, MSWD = 1.4). This is interpreted as the igneous crystallisation age of this sample (Fig. 10).

Nine Lu-Hf isotope analyses, all in different zircon grains, yield

$^{176}$Lu/$^{177}$Hf < 0.003, $^{176}$Yb/$^{177}$Hf < 0.07 and $^{176}$Hf/$^{177}$Hf ranging between 0.28050 and 0.28060. The εHf (3.588 Ga) values range between −0.7 and +0.2 with a weighted average of $-0.3 ± 0.3$ (95% confidence, MSWD = 0.4).

4.3.2.2. Gabbro 15TKPB23. Sixteen U-Pb analyses produced exclusively concordant data-points. A 207Pb/206Pb-weighted average of all dated points yields a 3.436 ± 0.004 Ga date (95% confidence, MSWD = 1.8). However, the thirteen analyses with the oldest 207Pb/206Pb-dates define a tight cluster while three analyses (n=778-2, #7 and #15) indicate slight Pb-loss as they plot below the main cluster. The thirteen coherent analyses result in a Concordia age of 3.437 ± 0.003 Ga (2σ, MSWD = 1.1) and a 207Pb/206Pb-weighted average of 3.438 ± 0.003 Ga (95% confidence, MSWD = 0.5), which is interpreted as the igneous crystallisation age of this sample (Fig. 10).

Twenty analyses of 15 different dated grains yield δ18O values between 4.5 and 5.8‰, with an arithmetic average of 5.3 ± 0.3‰ (2SD) and weighted average of 5.4 ± 0.4‰ (95% conf., MSWD = 7.1). Twelve zircon Lu-Hf isotope analyses in the nine least discordant (0–17%) dated grains yield $^{176}$Lu/$^{177}$Hf < 0.003, $^{176}$Yb/$^{177}$Hf < 0.07 and $^{176}$Hf/$^{177}$Hf ranging between 0.28050 and 0.28062. The εHf (3.575 Ga) values range between −1.3 and +0.3 with a weighted average of $-0.4 ± 0.2$ (95% confidence, MSWD = 1.1).

4.3.2.3. Leucogabbro 15TKPB25. Thirty-four U-Pb analyses in 31 grains yield mostly discordant data (n = 31/34 at the 2σ level) with 207Pb/206Pb-dates ranging from 3.587 to 2.141 Ga, indicating both recent and ancient Pb-loss. A regression of all 34 data points yields an upper intercept of 3.514 ± 0.058 Ga. However, as the data dispersion suggests multiple Pb-loss events this date seems geologically insignificant. A regression of the 13 data points with the oldest 207Pb/206Pb-dates defines an upper intercept of 3.582 ± 0.013 Ga (MSWD = 4.9) and the five oldest analyses yield a 207Pb/206Pb-weighted average of 3.580 ± 0.008 Ga (95% confidence, MSWD = 12).

Among the concordant and nearly concordant data points, a group of three analyses (n=778-8, #18 and #26) form an overlapping cluster, which yields a 207Pb/206Pb-weighted average of 3.575 ± 0.007 Ga (95% confidence, MSWD = 2.0). This age is within the uncertainty of the upper intercept age cited above and is interpreted to best represent the age of igneous crystallisation of this rock from the available data (Fig. 10).

Seven analyses on six different dated grains result in δ18O values between 4.5 and 5.8‰, with an arithmetic average of 5.3 ± 0.3‰ (2SD) and weighted average of 5.4 ± 0.4‰ (95% conf., MSWD = 7.1). Twelve zircon Lu-Hf isotope analyses in the nine least discordant (0–17%) dated grains yield $^{176}$Lu/$^{177}$Hf < 0.003, $^{176}$Yb/$^{177}$Hf < 0.07 and $^{176}$Hf/$^{177}$Hf ranging between 0.28050 and 0.28062. The εHf (3.575 Ga) values range between −1.3 and +0.3 with a weighted average of $-0.4 ± 0.2$ (95% confidence, MSWD = 1.1).

4.3.2.4. Coarse-grained leucogabbro 15TKPB26. Twenty-seven U-Pb analyses in 23 different dated grains yield 11 discordant and 16 concordant data points (at the 2σ level). The data loosely define a discordia indicating ancient Pb-loss and yield an upper intercept date of 3.567 ± 0.012 Ga. Due to apparent multiple Pb-loss events and large scatter in highly discordant analyses, the nominal lower intercept of 0.897 ± 0.086 Ga probably lacks geological significance. The nine analyses with the oldest 207Pb/206Pb-dates, all concordant and without obvious indication of Pb-loss, define a Concordia age of 3.580 ± 0.001 Ga (95% confidence, MSWD = 3.1) and a virtually identical 207Pb/206Pb-weighted average of 3.580 ± 0.002 Ga (95% confidence, MSWD = 3.7). This is interpreted to best represent the igneous crystallisation age of this rock from this dataset (Fig. 10).

Eighteen zircon oxygen isotope analyses from seventeen different dated grains, all ≤5% discordant, yield δ18O values between 3.2 and 6.2‰. Omitting one analysis (#21, discarded due to the ion beam sampling a crack), the δ18O range contracts to 5.4–6.2‰, with an average of 5.9 ± 0.2‰ (2SD) and a weighted average of 5.9 ± 0.2‰ (95% conf., MSWD = 1.3).
Twelve zircon Lu-Hf isotope analyses yield $^{176}\text{Lu}/^{177}\text{Hf}$ ≤ 0.003, $^{176}\text{Yb}/^{177}\text{Hf}$ < 0.1 and $^{176}\text{Hf}/^{177}\text{Hf}$ ranging between 0.28052 and 0.28066. The $\varepsilon_{\text{Hf}}$ (3.580 Ga) values range between −0.9 and +0.3 with a weighted average of −0.4 ± 0.2 (95% confidence, MSWD = 1.0).

**4.3.2.5. Leucogabbro 15TKPB27.** Forty-four U-Pb analyses in 36 different grains yield ten concordant data points and 34 that are discordant at the 2σ level. The data indicate multiple Pb-loss events with concordant to almost concordant analyses with $^{207}\text{Pb}/^{206}\text{Pb}$-dates ranging from 3.581 to 3.383 Ga. A regression using the entire data set results in an upper intercept date of 3.521 ± 0.028 Ga (MSWD = 1.41). This intercept is markedly younger than the concordant data points, so we interpret this date to be geologically meaningless. Given the scatter evident among the concordant analyses, presumably due to ancient Pb-loss, the five concordant analyses comprising the oldest and most coherent group are favoured and yield a Concordia age of 3.579 ± 0.001 Ga (95% confidence, MSWD = 7.6). This age is identical within uncertainty to the $^{207}\text{Pb}/^{206}\text{Pb}$-weighted average of the same five spots at 3.580 ± 0.001 Ga (95% confidence, MSWD = 1.0), which is interpreted to best represent the age of igneous crystallisation of this rock (Fig. 10). No geological significance is attached to the lower intercept (0.768 ± 0.0039 Ga) of the scattered discordia array.

Nineteen oxygen isotope analyses, all in different grains of which fourteen have been dated, yield $\delta^{18}\text{O}$ values between 0.9 and 5.7‰. Sixteen of these analyses have coherent values between 5.3 and 5.7‰, and three analyses (15TKPB27-14, #25 and #30) deviate, with values of 0.9, 4.3 and 2.6‰ respectively. Analysis 15TKPB27-30 is considered unreliable as it overlapped a crack. It is also noteworthy that analysis 15TKPB27-14, yielding the anomalous $\delta^{18}\text{O}$ value of 0.9‰ is from a 0.5% discordant grain that yields a $^{207}\text{Pb}/^{206}\text{Pb}$-date that indicates slight Pb-loss. A weighted average of all remaining analyses yields 5.1 ± 0.5‰ (95% conf., MSWD = 38) while the coherent group, excluding the two remaining statistical outliers, yields an average of 5.4 ± 0.3‰ and a weighted average of 5.4 ± 0.1‰ (95% conf., MSWD = 0.6).

Fourteen zircon Lu-Hf isotope analyses in as many different dated grains yield $^{176}\text{Lu}/^{177}\text{Hf}$ ≤ 0.002, $^{176}\text{Yb}/^{177}\text{Hf}$ < 0.07 and $^{176}\text{Hf}/^{177}\text{Hf}$ ranging between 0.28052 and 0.28060. The $\varepsilon_{\text{Hf}}$ (3.580 Ga) values range between −0.9 and +0.3 with a weighted average of −0.2 ± 0.2 (95% confidence, MSWD = 0.5).

**4.3.2.6. Owens Gully Diorite, OGC/OG1**

Thirty-two O isotope analyses of the OGC zircon yield very coherent $^{18}\text{O}/^{16}\text{O}$ between 0.0020161 and 0.0020176 and a weighted average of 0.0020171 ± 1 and $\delta^{18}\text{O}$ of 5.92 ± 0.07‰ (95% conf., MSWD = 1.2, Supplementary Table C). Although the oxygen isotope characteristics of zircon OGC have not previously been documented, these results can be compared with those generated during an earlier analytical session using a Cameca IMS1280 ion microprobe at the Swedish Museum of Natural History (instrument setup and analytical procedures similar to those of Whitehouse and Nemchin, 2009). Twenty-two analyses, of which one statistical outlier was discarded, provided very consistent $^{18}\text{O}/^{16}\text{O}$ between 0.0020159 and 0.0020177 and a weighted average of 0.0020169 ± 2 and $\delta^{18}\text{O}$ of 5.83 ± 0.01‰ (95% conf., MSWD = 2.2). These values are similar to results obtained from the present study (Fig. 11, Supplementary Table C).

Twenty-seven Lu-Hf isotope analyses of zircon OGC, obtained over two different analytical sessions, yield $^{176}\text{Lu}/^{177}\text{Hf}$ < 0.002, $^{176}\text{Yb}/^{177}\text{Hf}$ < 0.06 and $^{176}\text{Hf}/^{177}\text{Hf}$ ranging between 0.28060 and 0.28070, with an average value of 0.280639 ± 0.000021. The age-corrected $^{176}\text{Hf}/^{177}\text{Hf}$ (3.467 Ga) has an average of 0.280556 ± 0.000004, 95% confidence MSWD = 0.9, which is identical to the 0.280554 ± 0.000007 solution value reported by Kemp et al. (2017). The $\varepsilon_{\text{Hf}}$ (3.467 Ga) values range between +0.1 and +1.5 with a weighted average of +0.4 ± 0.2 (2σ, MSWD = 0.9).
5. Discussion

5.1. 3.59–3.58 Ga magmatism in the Pilbara Craton

The 3.588 ± 0.005 Ga zircon U-Pb age obtained from leucogabbro 12TKPB01 predates the oldest known (3.53 Ga) eruptive rocks in the East Pilbara Terrane by almost 70 Ma. Merging all data from the gabbroic samples 12TKPB01, 15TKPB25, 15TKPB26 and 15TKPB27, and calculating an age from the most coherent group of 207Pb/206Pb-dates (n = 25), yields a weighted average of 3.578 ± 0.001 Ga (95% confidence, MSWD = 1.7). This is in line with the ages estimated from the individual samples and, together with the similar major and trace element characteristics and Hf-O isotope signatures (see below) is consistent with a mutual igneous evolution to these rocks. The distinctly younger zircon age of 3.438 ± 0.003 Ga for 15TKPB23 is compatible with the age range of the Tambina Supersuite, which is an extensive component of the Shaw Granitic Complex (Van Kranendonk et al., 2006). It also corroborates the 3.431 ± 0.004 Ga age (sample UWA 98053) quoted by McNaughton et al. (1993) from the northern part of the South Daltons area. The younger emplacement age, together with the distinctly different REE pattern and zircon Hf-O systematics, preclude a simple petrogenetic link with the 3.59–3.58 Ga gabbros and leucogabbros, despite the geographical proximity. Given that 15TKPB23 also contains the pervasive, hornblende-defined foliation present in the older leucogabbros, the age of deformation and amphibolite facies metamorphism in this part of the Shaw Granitic Complex is constrained to be younger than 3.44 Ga.

The 3.59–3.58 Ga South Daltons gabbroic rocks are coeval with the 3.591 ± 0.036 Ga and 3.576 ± 0.022 Ga TTG gneisses reported from the Muccan Granitic Complex (zircon U–Pb; Wiemer et al., 2018). Zircon grains of this age have also been previously recorded within two gneiss samples separated by 15 km in the Warrawagine Granitic Complex (heterogeneously banded tonalitic gneiss GSWA 142870 and GSWA 180057, both forming enclaves in 3.42 Ga granodiorite of the Tambina Supersuite, Nelson, 1999b; Wingate et al., 2009; Kemp et al., 2015). As this group of igneous rocks comprises the oldest magmatic episode recorded in the Pilbara Craton, we propose that the magmatic episode is referred to as the Mount Webber event, and that the South Daltons gabbroic rocks are collectively named the Mount Webber Gabbro (Fig. 2). Zircon crystals of this age are also commonly encountered in younger sedimentary successions across the Pilbara Craton. For example, the majority (n = 90/104) of the detrital and inherited pre-Pilbara Supergroup zircon grains analysed by Kemp et al. (2007) yield 207Pb/206Pb-dates between 3.65 and 3.55 Ga. If these grains originate from within the Pilbara Craton, then they further strengthen the notion of a widespread ~3.58 Ga magmatic event predating the oldest rocks in the East Pilbara Terrane. Such an event is in line with a proto-continental basement for the Pilbara Craton, as proposed by Van Kranendonk et al. (2007). The limited available dataset does not allow for areal delineation of such a putative basement except that it is represented in three of the East Pilbara Terrane granitic complexes, at localities separated by 170 km.
It is noteworthy that older (to 3.66 Ga) zircons in a gneiss enclave in the Warrawagine Granitic Complex (Kemp et al., 2015), >3.7 Ga xenocrystic zircon in the Panorama Formation and Farrel Quartzite (Thorpe et al., 1992; Sheppard et al., 2017) and ancient >3.8–3.6 Ga detrital zircon grains within sedimentary basins of the Pilbara Craton (Van Kranendonk et al., 2007; Hickman et al., 2010; Kemp et al., 2015) could imply the existence of even older crust within the Pilbara Craton.

5.2. Origin of the Mount Webber gabbroic rocks

The Mount Webber leucogabbros, gabbros and associated ultramafic rocks are of limited outcrop extent and have been substantially modified by metamorphism, deformation and pervasive alteration. Nonetheless, as described above, the 3.59–3.58 Ga samples locally retain relict igneous textural features and mineralogical variations reminiscent of cumulate igneous layering in both leucogabbro-anorthosite and hornblende-rich gabbro, as well as an overall basaltic major element composition, viz., 48–51 wt% SiO₂ and 4.5–7.0 wt% MgO. Such lithological assemblages (i.e., leucogabbros, anorthositic gabbros and ultramafic rocks) and bulk compositions are commonly encountered in layered mafic intrusions (e.g., Bushveld, Yuan et al., 2007; Sept Iles; Namur et al., 2010; Skåegaard, Irvine et al., 1998), where they are interpreted to form by differentiation of ponded basaltic parental magmas. This origin is favoured for similar rock types within large anorthosite complexes (e.g., Laramie; Frost et al., 2010) and Archean layered intrusions (e.g., Fiskenaasset, Polat et al., 2010; Manfred, Myers, 1988, see also Ashwal and Bybee, 2017). The small, pegmatitic domains observed in the Mount Webber gabbro are also a feature of layered anorthosite complexes, and attributed to trapping of evolved residual melts against less permeable layers in the cumulate pile (e.g., Mitchell et al., 1996).

By analogy, we therefore consider it most plausible that the Mount Webber gabbroic to anorthositic enclaves represent variably differentiated fragments of a layered mafic intrusion (Mount Webber Gabbro) formed by crystallisation of mantle-derived basaltic parent magma. This inference accords with the zircon O-Hf isotope data (Section 5.3).

An alternative, that the gabbros and associated ultramafic rocks are cumulates from an intermediate magma derived by re-melting young, hydrated mafic crust, is not supported by the zircon O isotope data. The latter provide no evidence for such a hydrated (i.e., altered) protolith, and thus for the involvement of a hydrous fluid necessary to flux melting (Section 5.3).

Although a basaltic progenitor magma is inferred, deducing the specific composition of this, and the tectonic setting in which it was generated, is made ambiguous by the metamorphosed and altered nature of the gabbros, as presently preserved. It is also clear from the moderate MgO and compatible trace element contents, and cumulate features, that the gabbroic rocks do not represent primary mantle-derived melts. Strongly elevated LILE relative to the HFSE is the hallmark of subduction zone processes (e.g., McCulloch and Gamble, 1991), although in the case of the Mount Webber gabbro samples enrichment in the mobile elements Cs, Rb and K is probably at least partly due to alteration, evidenced by conversion of plagioclase to fine sericite and epidote, and secondary growth of white mica and chlorite. Rb and K abundances also closely correlate with LOI (Supplementary Table A). This LILE enrichment serves to accentuate the negative Nb-Ta anomaly, although it remains that the gabbroic samples do exhibit low Nb-Ta relative to the LREE and Th. If primary, the origin of this feature, common in Archean anorthositic rocks (e.g., Polat et al., 2018), does not have a clear-cut explanation in the present, somewhat limited dataset. Possibilities include (1) the involvement of a Nb-Ta sequestering phase at an earlier stage in the magmatic evolution, such as rutile or amphibole (Foley et al., 2002), (2) derivation from a mantle source overprinted by a LREE-rich component, perhaps derived from recycled oceanic crust (e.g. as argued for Archean anorthositic rocks by Polat et al., 2018), or (3) intra-crustal contamination, potentially accompanying fractional crystallisation of the parental magma to the gabbroic to anorthositic enclaves. Concerning the latter possibility, the lack of correlation between Nb/Th and Hf isotope data precludes formation of the variable Nb-Ta anomalies solely by assimilation of significantly older continental crust (Fig. 5C). The zircon oxygen isotope data further argue that any contaminant was not of supra-crustal derivation.

Other chemical features of the Mount Webber gabbros are easier to relate to those of the igneous protolith. The small positive Eu anomalies in leucogabbro 12TKPB1 and anorthositic sample 15TKPB27, in tandem with the Sr enrichment, are consistent with plagioclase accumulation and suggest that these samples do not represent melt compositions. This accords with the relict cumulate features in the leucogabbros noted above, and could also explain the lower overall REE content of these two samples. Accumulation, perhaps magmatic, of a titaniferous phase is implicated by the prominent Ti anomaly and smaller negative Nb-Ta anomaly in gabbro 15TKPB25. The high La/Yb and low Lu/Hf of all samples (besides gabbro 15TKPB23) could reflect deep melting in the garnet and/or hornblende stability field, or magmatic fractionation of these minerals, but geochemical trends from a larger compositional range of samples are required to assess this further (e.g., Davidson et al., 2007).

On a more definitive note, the markedly flatter REE pattern of the younger (3.44 Ga) gabbro 15TKPB23, together with the lower Sr but higher FeO and Cr, suggest that this younger intrusion was generated from a different mantle-source, or under different melting conditions. This is supported by the Hf isotope data (see below).

5.3. A 3.59 Ga chondritic basement to the East Pilbara Craton

The 3.59–3.58 Ga age of the Mount Webber gabbroic samples defines emplacement of the oldest single component igneous rocks recognised within the Pilbara Craton. As noted above, older zircon grains (to 3.66 Ga) are recognised in a banded tonalitic gneiss enclave in the Warrawagine Granitic Complex. These zircons have fine oscillatory zoning, consistent with crystallisation from melt, but younger zircon age populations are also present (3.61–3.57 Ga, 3.46–3.41 Ga), in part...
developed around the older cores (Kemp et al., 2015). This, together with the lithological heterogeneity, implies that the rock comprises different components that formed at different times; the actual emplacement age of the igneous protolith to the gneiss is therefore uncertain (see Kemp et al., 2015).

These 3.59–3.58 Ga gabbro and leucogabbro components of the Mount Webber Gabbro are significantly older than the surrounding South Daltons granodiorite. Hence, these gabbroic rocks provide the first direct evidence in the Shaw Granite Complex for part of the inferred basement on which the Pilbara Supergroup was deposited. Furthermore, in a progressively inflating granite dome where younger magmas exploit the same structural magma conduits, older components are displaced towards the batholith margins (Van Kranendonk et al., 2007); hence it is not surprising that the oldest constituent of the Shaw Granite Complex is found along its outer margins, as is also true for the Mount Edgar and Muccan granitic complexes (Van Kranendonk et al., 2007; Wiemer et al., 2018).

From an isotopic perspective, the 3.59–3.58 Ga Mount Webber Gabbro samples have zircon $\delta^{18}$O values consistent with crystallisation in equilibrium with mantle oxygen. These results corroborate previously presented zircon $\delta^{18}$O isotope data from > 3.3 Ga Pilbara rocks (Fig. 12, Kitajima et al., 2012; François et al., 2014; Van Kranendonk et al., 2015). This argues against reworking of an $^{18}$O-enriched supra-crustal source component by the 3.59–3.58 Ga magmas (i.e., materials that have interacted with surface waters). Additionally, a weighted average of all zircon $\delta^{18}$O values for the Mount Webber gabbro samples yields $-0.01 \pm 0.13$ (95% conf., MSWD = 2.4), within error of CHUR, as defined by Bouvier et al. (2008). There is no obvious correlation between zircon $\delta^{18}$O and $\epsilon_{Hf}(t)$ in any of the samples, but as illustrated in Fig. 13, a majority of the analyses from the > 3.5 Ga samples yield isotopic signatures within the mantle range for both O and Lu-Hf isotopes (Fig. 13). The zircon Hf isotope data from the younger gabbro 15TKPB23 also overlap CHUR, suggesting a single, homogeneous source to this rock.

It could be argued that the zircon Hf isotope signatures of the Mount Webber gabbroic rocks reflect interaction between a depleted mantle-like component (i.e., $\epsilon_{Hf} > 0$) and older crust ($\epsilon_{Hf} < 0$), where the resulting mixture fortuitously straddles CHUR. This is, however, difficult to reconcile with the consistent and tightly clustered zircon Hf isotope signatures (Figs. 13–14), together with the mantle-like zircon $\delta^{18}$O and the gabbroic bulk rock composition of these samples. We instead interpret the geochemical and isotope data to indicate derivation of the ~3.59–3.58 Ga Mount Webber gabbros from an approximately chondritic mantle, with minimal influence from older source materials. In this interpretation, the emplacement of the ~3.59 Ga Mount Webber gabbroic rocks signifies an early crust generation episode within the nascent Pilbara Craton.

5.4. Implications for cratonic evolution

It is instructive to consider the new isotope data presented here in the broader context of published radiogenic isotope data for igneous rocks from the Pilbara Craton. Whole rock Nd isotopes from granites, granodiorites and tonalites from the North Shaw Suite (McCulloch, 1987; Bickle et al., 1993) yield $\epsilon_{Nd}(3.45–3.47 \text{ Ga})$ of $+1.3$ to $-1.3$ with an average of $0.4 \pm 0.5$ (2SD, $n = 6$). As with the zircon Hf isotope signatures of the Mount Webber Gabbro, these values are in agreement with a chondritic source. Nebel et al. (2014) presented whole-rock Hf isotope data from ~3.5–2.9 Ga komatiites and basaltic komatiites from different stratigraphic units of the Pilbara Craton. These data have a large spread of Hf isotope signatures ranging from $\epsilon_{Hf}(3.46 \text{ Ga})$ of $+8.2$ to $\epsilon_{Hf}(3.35 \text{ Ga}) = -14.6$ (Fig. 14A). Fisher and Vervoort (2018) argue that Hf isotope data for igneous rocks should be treated in similar fashion to U-Pb data and assigned a weighted average instead of being presented as a range. However, due to lack of coherence within the dataset of Nebel et al. (2014), no weighted average has been calculated for these data. Younger Paleoarchean granitic supersuites, e.g. Emu Pool (3.32–3.28 Ga) and Cleland (3.27–3.22 Ga), reported by Gardiner et al. (2017), show scattered zircon Hf isotopic signatures, $\epsilon_{Hf}(3.35 \text{ Ga}) = +4.4$ to $-5.1$, when recalculated using recent reference values for CHUR and $^{176}$Lu decay constant of Bouvier et al. (2008) and Söderlund et al. (2004) respectively. This has been interpreted to indicate subsequent reworking of older TTG crust, with trends towards slightly less radiogenic signatures with time (see Fig. 14A; Gardiner et al., 2017). It should, however, be noted that our recalculations of the Hf data in Gardiner et al. (2017) gives a weighted average of all 3.48–3.29 Ga zircon $\epsilon_{Hf}(t)$ values of the Tambina and Emu supersuites of $+0.17 \pm 0.20$ (n = 243, 95% conf., MSWD = 11), and 63% of individual analyses (n = 152/243) yield values within error of CHUR. Amelin et al. (2000) analysed zircon from different 3.45–3.32 Ga quartz porphyries from the Marble Bar region that yield Hf isotope signatures
indistinguishable from CHUR, with weighted average \( \varepsilon_{Hf}(t) \) values of +1.0 \( \pm \) 1.0 (Fig. 14A). Similarly, Guitreau et al. (2012) analysed zircon from 3.48 to 3.30 Ga Pilbara TTGs yielding Hf isotope signatures with a \( \varepsilon_{Hf}(t) \) weighted average of +0.4 \( \pm \) 0.9. Kemp et al. (2017) characterised the 3.467 Ga zircon from Owens Gully Diorite of the Mount Edgar Granitic Complex, obtaining an average \( \varepsilon_{Hf}(t) \) of +0.6 \( \pm \) 0.7 (Fig. 14A, B). In fact, all available zircon Hf isotope data from Paleoproterozoic felsic igneous rocks of the East Pilbara (i.e., from 3.6 to 3.3 Ga) Terrane yield average values within error of CHUR. This observation is further reinforced by the inherited and detrital zircon data presented by Kemp et al. (2015, Fig. 14B). The ca. 3.47–3.3 Ga igneous rocks (including gabbro 15TKPB23 of the present study) cannot be derived solely from reworking of the 3.59–3.58 Ga Mount Webber gabbron and leucogabbros, which would be too unradiogenic at < 3.5 Ga (Fig. 14B). Instead, it appears that an overall chondritic Hf isotope signature prevailed in the eastern Pilbara Craton from 3.59 Ga to at least 3.3 Ga, suggesting that felsic magmatic episodes in this time span involved the addition of new crust. Quantifying the proportion of new versus reworked crust incorporated by the felsic igneous suites requires better knowledge of the isotope evolution of the Pilbara mantle over this period. This can potentially be provided by the basaltic and komatiitic rocks erupted in the East Pilbara Terrane from 3.53 to 3.3 Ga, since the Hf isotopic composition of these can constrain that of the mantle source.

Van Kranendonk et al. (2015) argued for a \( \sim \) 35 km thick > 3.5 Ga protocrust, a thickness approximately twice that of the entire Pilbara Supergroup (Hickman and Van Kranendonk, 2004; Hickman, 2012), as a source to the Paleoproterozoic TTG of the Pilbara and Kaapvaal cratons. Similarly, Wiemer et al. (2018) inferred a \( \sim \) 43 km thick, \( \geq \) 3.6 Ga ultramafic/mafic protocrust as the source for Eo- to Paleoproterozoic TTG within the Pilbara Craton. Extraction of an equivalent of \( \sim \) 35 km of crust prior to 3.6 Ga would lead to formation of an incompatible element depleted mantle residue, which would evolve over time towards superchondritic \( 176\text{Lu}/177\text{Hf} \) Hf. Such a radiogenic signature is not evident within the Mount Webber gabbron (of inferred mantle derivation), or in the Pilbara detrital and inherited zircon dataset (3.66–3.55 Ga) reported by Kemp et al. (2015), or, more widely, in Hf isotope datasets from the oldest, best preserved and best age-constrained igneous rocks.
from a number of Archean cratons, which cluster conspicuously around CHUR (Kemp and Hawkesworth, 2013; Kemp et al., 2015; Hiess and Bennett, 2016; Fisher and Vervoort, 2018; Fig. 14A, B). Collectively, these sample sets (and see also Bolhar et al., 2017 for the Zimbabwe Craton) therefore provide no evidence for a global depleted mantle reservoir before 3.6 Ga.

There are two different ways to reconcile this observation, depending on the prevailing mode of early Archean mantle dynamics. On the one hand, the paucity of a super-chondritic signal in ancient felsic rocks could suggest that early continental crust was sourced from convecting mantle, where the complementary highly depleted residues were physically isolated and not resampled by younger magmas. In this way, successive, large continental volumes could separate from a contracting primitive mantle reservoir and not record the chemical/isotope legacy of prior crust extraction. In such a model, the lack of super-chondritic Hf isotope signatures in the Mt. Webber rocks would not preclude the existence of local, highly depleted mantle domains, as could, for example, have been sequestered in a cratonic lithosphere root.

An opposing viewpoint, developed to account for positive εNd values in Archean rocks, is that Earth’s stable continental crust has grown from a progressively depleting mantle reservoir through time (e.g., McCulloch and Bennett, 1994; Bowring and Housh, 1995). Although the veracity of some of these Nd data has been questioned (e.g., Vervoort et al., 1996), it remains that the Hf isotope composition of juvenile felsic igneous rocks preserved on different cratons clearly diverges from CHUR towards radiogenic values after ca. 3.6 Ga (Fisher and Vervoort, 2018). This indicates that the ultimate mantle source of at least some juvenile Archean continental crust (and Archean anorthositic rocks; Ashwal and Bybee, 2017; Polat et al., 2018) recorded a long-term depletion in incompatible elements, as would be the case if the residues of crust extraction were, at least in part, back-mixed into ambient source mantle; indeed, crust generation from such a global, convecting depleted reservoir has continued to the present day (e.g., Vervoort and Blichert-Toft, 1999). In this circumstance, the apparent lack of positive εNd values before 3.6 Ga would be evidence that the volume of crust extracted and stabilised above the Moho in the Hadean to early Archean was too small to discernably influence the isotope evolution of a continuously tapped mantle source, which retained broadly chondritic 176Hf/177Hf (as reflected in the Mount Webber Gabbro).

For the Pilbara Craton, this scenario does not favour the models of Van Kranendonk et al. (2015) and Wiemer et al. (2018), both of which argued for substantial crust extraction prior to the eruption of the Pilbara Supergroup. More broadly, applying a pre-3.5 Ga chondritic mantle source to recent continental growth curves based on compilations of depleted mantle Hf model ages from zircons (e.g. Belousova et al., 2010 and Dhuime et al., 2012), would markedly lower estimates of global crustal generation rates during the first billion years of Earth’s history. If such a chondritic mantle model for pre-3.5 Ga crust extraction is correct, this would help sustain the view that the volumes of the earliest stabilised continents have been overestimated (Hiess and Bennett, 2016; Vervoort and Kemp, 2016; Fisher and Vervoort, 2018).

5.5. Interpreting the source of 3.44 Ga sub-mantle zircon δ18O

Taken alone, the low zircon δ18O values of the 3.44 Ga gabbro 15TKPB23 might suggest post crystallisation alteration involving high-grade metamorphism or hydrothermal alteration. All analysed grains are, however, concordant at the 2σ level and the U-Pb systematics of zircon from this rock indicate minimal disturbance. As shown by Pettersson et al. (2015) and Rubatto and Angiboust (2015), metamorphic alteration of zircon δ18O can be traced using the zircon Th/U, where a linear correlation between Th/U and δ18O indicate post crystallisation disturbance. However, there is no correlation between Th/U and δ18O within the 15TKPB23 zircons, and furthermore, the very coherent values with a spread of zircon δ18O less than one permil are not expected from post crystallisation alteration.

Relatively constant magmatic zircon δ18O values from primitive mantle melts though time compared to current mantle δ18O values led Valley et al. (2005) to propose a constant and homogeneous δ18O of the mantle back to ~4.4 Ga. Major oxygen isotope fractionation is constrained to near surface processes and so non-mantle-like values in igneous rocks are commonly explained by reworking of crustal material in the magma source or interaction between mantle derived melts and a crustal component during magma ascent (Eiler, 2001).

However, the lack of inherited zircon, combined with the tightly clustered zircon Lu-Hf and O isoce data for sample 15TKPB23 suggest a homogeneous single source to this rock. Low-δ18Oigneous zircons characteristically form in hydrothermal magmatic systems or at mid-ocean ridges, but rocks formed at such tectonic environments have low preservation potential, and sub-mantle zircon δ18O values are very uncommon in the Archean (Hammerli et al., 2018). As there is no obvious evidence for post crystallisation zircon alteration in 15TKPB23, it can be inferred that the gabbroic magma assimilated low δ18O crust prior to zircon crystallisation. Byerly et al. (2017) identified 3.27 Ga komatiites, from the Weltevreden Formation in the Barberton greenstone belt, Kaapvaal Craton in Southern Africa, that yield sub-mantle (~3‰) δ18O values from fresh olivine. These signatures are in line with sample 15TKPB23, ~2‰ below the 5.3 ± 0.6‰ mantle value of Valley et al. (1994, 2005) and suggest a low δ18O magma. This implies a heterogenous mantle derived source enriched by crustal material with sub-mantle δ18O-values. Oceanic crust affected by high-temperature hydrothermal alteration has most commonly been used as such component (Muehlenbachs, 1986). Assimilation of hydrothermally altered, juvenile (i.e., chondritic for Hf) mafic crust could potentially lower the δ18O of the source without markedly affecting the Hf isotopes, explaining the combined zircon O-Hf isotope signatures of 15TKPB23.

5.6. Oxygen isotope characterisation of the OGC/OG1 zircon

The increased popularity and use of zircon micro-analyses, such as U-Pb, O and Lu-Hf isotopes, increases the demand for high-quality and well characterised reference zircons as data quality monitors. The Paleoearchean Owens Gullly Diorite (OGC/OG1) zircon has been characterised for U-Pb isotopic systematics by Stern et al. (2009), and Hf isotopes by Kemp et al. (2017). The oxygen isotope data presented herein show excellent agreement between two different laboratories strengthening the validity of the results. Combining datasets from the two different laboratories yields a weighted average δ18O of 5.88 ± 0.06‰ (n = 53, 95% conf., MSWD = 1.7, Fig. 11). This represents the first characterisation of the 18O/16O in the OGC/OG1 zircon. The homogeneity (at the sampling scale) and reproducibility of the 18O/16O data (within the uncertainties of the techniques employed) suggests that the Owens Gully Diorite (OGC/OG1) zircon holds promise as an oxygen isotope quality monitor for ion microprobe (SIMS) micro-analysis.

6. Conclusions

Metamorphosed gabbroic, leucogabbroc and anorthositic rocks (Mount Webber Gabbro) of the South Daltons area in the western part of the Shaw Granitic Complex are here dated to 3.59, 3.58 and 3.44 Ga by zircon U-Pb isotopes. The 3.59–3.58 Ga gabbros comprise the oldest single component igneous rocks yet identified in the Pilbara Craton, significantly predating the eruption age of the oldest basaltic-komatiitic lavas. The recognition of these rocks thus adds another significant element to the magmatic history of the Pilbara Craton. Combined with existing U-Pb data from TTG gneisses of the Muccan Granitic Complex (Wiemer et al., 2018), detrital and inherited zircon (Kemp et al., 2015) and two gneissic tonalitic enclaves within the Tambina Superuite in the Warrawagine Granitic Complex (Nelson, 1999b; Wingate et al., 2009; Kemp et al., 2015), a widespread magmatic event across the Pilbara
Craton at ~3.59 Ga is established. We collectively refer to these gabbroic rocks as the Mount Webber Gabbro, and interpret the combination of field, petrographic, chemical and isotopic features to suggest that these represent variably differentiated fragments of a layered mafic intrusion. Given that the parental magma of the Mount Webber Gabbro was of mantle derivation, this episode resulted in addition of new crust in the early evolution of the Pilbara Craton. The actual amount of crust generated during this period, and whether this constitutes a component of an intra-continental basement to the <3.53 Ga volcano-sedimentary successions, remains to be established.

Zircon $^{180}$Hf and $^{176}$Hf isotope signatures in the 3.59 Ga Mt. Webber Gabbro do not support a contribution from significantly older crust or hydrated supracrustal materials, but are instead consistent with the tapping of a chondritic mantle source in this part of the Pilbara Craton ≥ 3.44 Ga. This finding is congruent with results from the oldest felsic igneous rocks in other Archean cratons, in providing no direct evidence for the involvement of a widespread, strongly depleted mantle in the formation of early Archean continental crust. One implication is that stabilised volumes of Hadean–Archean continental crust, estimated assuming that crust was extracted from a global, convecting depleted mantle reservoir, have been overestimated.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemgeo.2019.01.021.

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