Rate of growth of the preserved North American continental crust: Evidence from Hf and O isotopes in Mississippi detrital zircons

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Abstract

Detrital zircons from the Mississippi River have been analyzed for U–Th–Pb, Lu–Hf and O isotopes to constrain the rate of growth of the preserved North American continental crust. One hundred and forty two concordant zircon U/Pb dates on grains mounted in epoxy, obtained by Excimer laser ablation ICP-MS method, resolved six major periods of zircon crystallization: 0–0.25, 0.3–0.6, 0.95–1.25, 1.3–1.5, 1.65–1.95 and 2.5–3.0 Ga. These age ranges match the ages of the recognized lithotectonic units of the North American continent in the hinterland of the Mississippi River. Ninety-six zircons mounted on tape, which show no age zonation and were within 7.5% of concordance, were selected to represent the six U/Pb age time intervals and analyzed for Lu–Hf and O isotope by laser ablation MC-ICP-MS and SHRIMP II, respectively. The $\delta^{18}\text{O}$ values of the zircons show a small step increase in the maximum $\delta^{18}\text{O}$ values at the Archean-Proterozoic boundary from 7.5‰ in the Archean to 9.5‰, and rarely 13‰, in the Proterozoic and Phanerozoic. However, the average value of $\delta^{18}\text{O}$ in zircons changes little with time, showing that the increase in the maximum $\delta^{18}\text{O}$ values between 2.5 and 2.0 Ga, which can be attributed to an increase in the sediment content of the source regions of younger granitoids, is largely balanced by an increase in zircons with anomalously low $\delta^{18}\text{O}$, which can be attributed to hydrothermally altered crust in the granitoid source region.

ε_{Hf} values for the zircons range from 13.1 to –26.9. Zircons derived from juvenile crust, which we define as having mantle $\delta^{18}\text{O}$ (4.5–6.5‰) and lying within error of the Hf depleted mantle growth curve, are rare or absent in the Mississippi basin. The overwhelming majority of zircons crystallized from melted pre-existing continental crust, or mantle-derived magmas that were contaminated by continental crust. The average time difference between primitive crust formation and remelting for each of the recognized lithotectonic time intervals, which is defined as crustal incubation time in this study, is 890 ± 460 Myr. There is also a suggestion that the crustal incubation time increases with decreasing age in the Mississippi basin, which is consistent with the declining role of radioactive heat production in the lower crust with time.

The average Hf model age (1.94 Ga), weighted by fraction of zircons in the river load is in reasonable agreement with the Nd model age (1.7 Ga) for the Mississippi River. However, if the zircons are weighted by the area of North America covered by the six recognized periods of zircon crystallization the average model age is 2.35 Ga, which compares favorably with an area weighted Nd model age of 2.36 Ga. Our preferred approach is to use the measured O isotope values to constrain variations in the $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of the granitic source region from which the zircons crystallized, making the assumption that zircons with mantle-like O isotopic ratios have higher $^{176}\text{Lu}/^{177}\text{Hf}$ than zircons with higher O isotope values. This method gives an average Hf model age of 2.53 Ga, which is 180 Myr older than the constant $^{176}\text{Lu}/^{177}\text{Hf}$ calculation.

The area weighted zircon Hf model ages show two distinct periods of crust formation for the North American continent, 1.6–2.2 and 2.9–3.4 Ga. At least 50% of the preserved North American continental crust was extracted from the mantle by

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2.9 Ga and 90% by 1.6 Ga. Two similar periods of crustal growth are also recognized in Gondwana (Hawkesworth C. J. and Kemp A. I. S. (2006) Using hafnium and oxygen isotopes in zircons to unravel the record of crustal evolution. *Chem. Geol.* **226**, 144–162.), suggesting that these may be periods of global continental crustal growth. However, we stress that more data from other continents are required before the hypothesis of episodic global continental growth can be accepted with confidence.

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1. INTRODUCTION

The rate of growth of the continental crust remains controversial. There are two end-member hypotheses. The most widely accepted view is that the crust began to form a little before 4.0 Ga and has been growing largely irreversibly since then (Moorbath, 1977; McLennan and Taylor, 1982; Fyfe, 1978; Reymer and Schubert, 1984). The distribution of the preserved continental crust supports this view. Old crust covers only a small fraction of the Earth's surface and the fraction of crust covered by younger crust increases progressively with time. The other end-member hypothesis, is the steady-state model of Armstrong (1981, 1991), in which the mass of the continental crust that formed between 4.0 and 4.5 Ga was similar to today's and has been in a steady-state since then with continental crust being recycled into the mantle as fast as it forms. According to this hypothesis recycling has destroyed all of the crust that formed between 4.0 and 4.5 Ga.

Isotopic studies have been unable to distinguish between the continuous and steady-state hypotheses for the growth of the continental crust. On one hand, Nd isotopic studies of modern sediments give model ages between 1.4 and 1.8 Ga (Jacobsen, 1988; Goldstein and Jacobsen, 1988), which suggests that the bulk of the continental crust formed after 2.5 Ga. On the other hand, the steady-state hypothesis appears to be supported by Sm and Hf isotopic analyses of the Earth's oldest rocks, which require the Earth's early mantle to have Sm/Nd and Lu/Hf ratios well above the chondritic value (McCulloch and Bennett, 1994; Vervoort et al., 1996). If the observed Sm/Nd and Lu/Hf values are due solely or mainly to extraction of the continental crust (Bowring et al., 1989; McCulloch and Bennett, 1994), the amount of continental crust that is required to have been extracted from the mantle between 4.0 and 4.5 Ga is comparable to the mass of the modern continental crust. However, if extensive continental crust formed prior to 4.0 Ga, little evidence for its existence is preserved in Sm–Nd model ages of the Earth's oldest sediments (Jacobsen and Dymek, 1987) or in the zircons extracted from them (Nutman, 2001). Traces of zircon as old as 4.3 Ga in sediments from the Archean Yilgarn Craton of Western Australia (Wilde et al., 2001) show that the continental crust existed from 4.3 Ga but not prove that it was extensive. An alternative explanation of the high Sm/Nd (and Lu/Hf) ratio of the pre-4.0 Ga upper mantle is that it is due to removal of basaltic crust (Chase and Patchett, 1988; Galer and Goldstein, 1991). Extraction of oceanic (basaltic) crust raises the Sm/Nd ratio of the residual mantle and mass balance considerations indicate that only one third of the increase in Sm/Nd in the modern upper mantle is due to extraction

of continental crust, with the remaining two thirds being due to extraction of basaltic crust (Campbell, 2003). It is likely that most of the enrichment in Sm/Nd in the 4.0–4.5 Ga mantle was due to removal of basaltic crust, which must have been the dominant process during the first 500 Myr of Earth's history (Campbell, 2003; Davies, 1988, 2002). Lunar samples, after all, have high ϵ_{Nd} values requiring the moon's mantle to have developed a high Sm/Nd early in its history without the involvement of a continental crust.

Isotopic analyses of detrital zircons from the Earth's major rivers provide a new perspective on the growth rate of the continental crust. Zircons crystallize from crustal melts and, once formed, are resistant to destruction. They have low solubility in silicate melts so that they often partially survive crustal melting, recording evidence of earlier crust melting events when they do so. More importantly, in the present context, they are resistant to chemical weathering and mechanical abrasion so they survive weathering from their granitic or high-grade metamorphic source rock, transport down rivers, often through one or more cycle of sedimentation, to river mouths where a single sample can provide an effective sample of an entire river basin. Furthermore, individual zircons can be analyzed for three isotopic systems: U–Th–Pb, Lu–Hf and O isotopes, which provide different and complementary information.

U–Th–Pb analyzes date the time of crystallization of the zircon, normally the age of a crustal melting event but occasionally the age of high-grade metamorphism. The $^{176}\text{Hf}/^{177}\text{Hf}$ ratio preserved in zircon is close to the ratio of the melt from which the zircon crystallized. From this ratio a model age can be calculated that gives the time when the crustal source region, which melted to form the granitic magma from which the zircon crystallized, separated from the mantle. The zircon Hf model age is complementary to Nd model ages obtained from river sediments. Whereas Nd model ages of river sediments give an estimate of the average mantle extraction age for the preserved continental crust in a river basin, the zircon Hf models ages can resolve individual periods of crustal growth, which is important in distinguishing between continuous growth and episodic growth. Finally, O isotopes provide a measure of the involvement of low-temperature processes in the magma source region. In practice this can be taken to be a measure of the fraction of sediment in the source region (Hawkesworth and Kemp, 2006). The combination of Hf and O isotopes can be used to distinguish zircons derived from melting of igneous crust from those derived from melting of continental crust that contains a sedimentary component and we have used the measured O isotope values to con-

strain the $^{176}\text{Hf}/^{177}\text{Hf}$ ratio of the zircon source region, which is expected to be highly variable.

The strategy adopted in this study has been to analyze zircons separated from a single sample, collected near the mouth of the Mississippi River (Fig. 1), for U–Th–Pb, Lu–Hf and O isotopes, making the assumption that the sample is representative of the average crust within the river basin. This assumption has obvious limitations and a second series of calculations were made in which the analyzed zircons were weighted according basement area covered by the various U/Pb age population recognized in the sample, as discussed further below. The data are used to calculate the rate of growth of the preserved continental crust in the Mississippi basin, to identify periods of crustal growth, and to determine the time lag between crust formation and cratonisation (the crustal incubation time).

2. GEOLOGICAL BACKGROUND

Most of the North American continent is covered by sedimentary basins, which are underlain by a crystalline basement composed of a core of the Precambrian Laurentian Shield rimmed by the Neoproterozoic to Phanerozoic orogenic belts (Hoffman, 1988). Fig. 1 is a sketched map

showing the tectonic units of the North American continent crust.

The Laurentian Shield includes six Archean provinces, the Superior (2.7–3.0 Ga, Card, 1990), Wyoming (2.55–2.8 Ga, Frost et al., 1998), Hearne (3.3–2.6 Ga, Hoffman, 1988), Rae (2.7–2.87 Ga, St-Onge et al., 2007), Slave (2.7–2.65 Ga, Kusky, 1989) and Nain (3.8–2.8 Ga, Ryan, 1995) provinces. The Superior and Wyoming provinces are drained by the Mississippi River (Fig. 1). The Superior province is composed of upper amphibolite- and granulite-facies gneisses of supracrustal and plutonic origin, and low-grade greenstone belt metavolcanic–metasedimentary sequence (Card, 1990). The Wyoming province is composed primarily of the late Archean potassium-rich granitic rocks (Frost et al., 1998). Most Archean provinces, for example, the Superior province, are interpreted to have formed from tectonic amalgamation of smaller, exotic terranes (Card, 1990).

Paleoproterozoic orogenic belts form a curvilinear network surrounding the Archean provinces (Rivers, 1997) and comprise four ~1.98–1.79 Ga collisional orogens, the Trans-Hudson, Ungava, New Quebec and Torngat orogens and the slightly younger (~1.90–1.62 Ga) Penokean, Makovik, Central Plains, Yavapai and Mazatzal accretionary

Fig. 1. Regional geology map showing the tectonic units in the North American continent (Modified after Bally et al., 1989; Goodge et al., 2004; Whitmeyer and Karlstrom, 2007).

orogens (Rivers, 1997). The Mississippi River system drains a region that mainly overlies Yavapai and Mazatzal origin basement. The accretionary orogens are interpreted to be Andean-type magmatic arcs and/or closure of back-arc basins on southern Laurentia (Hoffman, 1989), whereas the collisional orogens are thought to be a combination of arc-continent and continent-continent collisions. These events culminated in creation of the Laurentia supercontinent (Hoffman, 1989; Van Kranendonk et al., 1993).

The ~1.5–1.34 Ga Granite–rhyolite belt in southeastern Laurentia comprises principally felsic igneous rocks that cut obliquely across the Paleoproterozoic orogens (Rivers, 1997).

The Grenvillian orogeny took place between ~1.19 and 0.98 Ga and consists of three distinct pulses of crustal shortening at ~1.19–1.14 Ga (Shawinigan orogeny), 1.08–1.02 Ga (Ottawan orogeny) and 1.00–0.85 Ga (Rigolet), separated by periods of extension (Rivers, 1997). The orogenic events generated anorthosite–mangerite–charnockite–granite suite and A-type hornblende granites and were responsible for the deformation and metamorphism of all rocks older than 1.08 Ga (McLelland et al., 1996). The Mid-Continent Rift served as the Grenville Front and preserved the abundant supracrustal succession of 1.11–1.09 Ga Keweenaw Supergroup, which consists of terrigenous sedimentary rocks and continental tholeiites (Green et al., 1987; Hoffman, 1989). Note that the total time interval for dated tectonic events, 0.98–1.19 Ga, is appreciably less than the range of ages for crustal melting (0.95–1.25 Ga) recorded by detrital zircons (Fig. 3).

The Appalachian orogeny to the southeast of Laurentia is generally considered to be the product of five discrete tectonomagmatic events: Avalonian–Carolinian (580–640 Ma), Potomac (~500 Ma), Taconian (~430–500 Ma), Acadian (~350–400 Ma) and Alleghanian (~265–325 Ma) (Hatcher, 1989; Hibbard and Samson, 1995; Goldberg and Dallmeyer, 1997). These five late Neoproterozoic/Paleozoic events were associated with the amalgamation of Laurentia and Pangea (Eriksson et al., 2003). The Appalachian Mountains are composed of strongly metamorphosed to unmetamorphosed Neoproterozoic to Paleozoic sedimentary rocks, with subordinate volcanic rocks and some Paleozoic granitoids (Rankin et al., 1993).

The Cordilleran orogeny to the west of Laurentia was formed by an arc-continent collision and consequent tectonic escape of the central and northern Cordillera to the north. Two major episodes of collision are recognized, the first between 187 and 164 Ma involved the collision of oceanic crust fragments with North America, and the second is linked to the oblique collision between a more westerly terrane with the Cordilleran margin between 100 and 40 Ma (Ross et al., 2005). The switch from collision to escape happened between 105 and 85 Ma and formed two belts of magmatism, one from subduction and the other from the underthrusting of North American crust to the west (Umhoefer, 2003). The main periods of silicic magmatism and caldera formation in the Cordillera were: 214–201, 172–148, 119–86 and 32–27 Ma; Sierra Nevada plutonism occurred from 217–80 Ma (Ward, 1995).

3. ANALYTICAL METHODS

The zircons were separated using conventional magnetic and heavy liquid separation techniques. Analyzes of U/Pb ages, and O and Hf isotopic compositions for the chosen zircons were carried out at the Research School of Earth Sciences, Australian National University. The analytical results are summarized in Electronic Annexes.

3.1. U/Pb age dating

Excimer Laser Ablation Inductively Coupled Plasma Mass Spectrometry (ELA-ICP-MS) was used to date the zircons in two different analytical sessions with different goals. The integration time was 40 ms for the three Pb isotopes, U and Th, and 10 ms for all other isotopes with a total mass sweep time of 0.385 s. ELA-ICP-MS analyzes require a larger ablation pit than an ion micro-probe analyzes, typically 30 μm across and 20 μm deep. As a consequence, each ablation consumes a large amount of material that may include inclusions of minerals such as apatite or material that is age-zoned. The response time of the ANU ablation cell, which we define as the time the signal takes to decay by a factor of 10, is ~2 s. The rapid response time of our cell allows us to monitor down-hole age zoning and to edit out sections of the profile that are age-zoned or that contain inclusions. Recognition of age zoning is particularly important. The zircons we dated included an appreciable percentage of the age-zoned grains. Failure to recognize age zoning results in reporting of meaningless mixed ages, which in turn results in: (i) the grains being plotted on an incorrect position on the U/Pb age histogram, broadening of age peaks and (ii) a significant error in the ϵ_{Hf} calculations (Harrison et al., 2005) and Hf model ages. Laboratories reporting ELA-ICP-MS U/Pb ages should demonstrate that they can resolve down-hole age zoning and that they are not producing meaningless mixed ages.

Campbell et al. (2005) describe the analytical method used in this study. The amount of common Pb was determined by the ^{208}Pb method (Compston et al., 1984) and the calculated amount subtracted, assuming a common Pb composition from the age-dependent Pb model of Cumming and Richards (1975). Whether a common Pb-uncorrected age or a common Pb-corrected age was chosen depended on whether or not the common Pb correction brought the analysis closer to Concordia. The criteria for selecting a grain are that its U–Th–Pb age is 95% concordant, including 2σ errors and that the observed to expected error ratio is less than 2 (see Campbell et al. (2005) for details). $^{206}\text{U}/^{238}\text{Pb}$ ages were used for zircons younger than 0.9 Ga, and $^{207}\text{Pb}/^{206}\text{Pb}$ ages for older grains.

Zircons were U–Pb dated by two different methods. First, 169 zircons were mounted in a conventional electron microprobe-style epoxy mount to determine the U/Pb age distribution of the zircons in the sample. The normal precautions were taken to minimize sampling bias during mounting and analyzes. The heavy mineral concentrate was placed on a weighing paper that was folded and pinched so that a sub-population could be spilled onto a

glass plate that was placed under a binocular microscope. All recognized zircons in the sub-sample were picked by hand and analyzed regardless of appearance. One hundred and fourth eight of the analyzed grains met our selection criteria (see [Electronic Annex I](#) and [Fig. 2](#)), 73 requiring no common Pb correction and 75 requiring a small correction that was normally less than 2%. Twenty one grains failed the selection criteria. The data are plotted as an age histogram in [Fig. 3](#). Filled bars show concordant grains and open bars discordant grains. Six distinct time peaks can be recognized in the histogram: 0–0.25, 0.3–0.6, 0.95–1.25, 1.3–1.5, 1.65–1.95 and 2.5–3.0 Ga ([Fig. 3](#)). Six concordant grains fell outside these defined age ranges.

A second set of zircons (397 grains) were soaked in concentrated HF acid overnight at room temperature to remove metamict and fractured grains that could compromise their O isotope, U–Pb or Lu–Hf systematics. Zircons with minimum dimensions of 50 μm were selected, mounted on tape and dated by the rim-piercing method described by [Campbell et al. \(2005\)](#). The laser holes were drilled through the top $\sim 20\ \mu\text{m}$ of unpolished grains and provide a continuous profile of the age variation from the rim of the grain towards its center. This method is ideal for identifying grains with U/Pb age zoning, which were excluded for O and Hf isotope analyzes in this study. Finally 96 grains were selected for further analyzes from the six recognized time intervals as follows: 21 grains with ages between 0 and 0.25 Ga, 12 grains between 0.3 and 0.6 Ga, 19 grains between 0.95 and 1.25 Ga, 12 grains between 1.3 and 1.5 Ga, 14 grains between 1.65 and 1.95 Ga, and 18 grains between 2.5 and 3.0 Ga. The results are listed in detail in [Electronic Annex II](#). The selected grains are better than 92.5% concordant, have a homogeneous Ti and P distributions, show no age zonation and have no inclusions of apatite, rutile or titanite. These grains were then mounted in an epoxy mount especially designed for high precision SHRIMP O

Fig. 2. Concordia plot of the 169 zircons set in epoxy and dated by laser ICP-MS. “Selected” grains lie within 95% of concordance, including 2 σ errors and have an observed to expected error ratio of better than 2. Zircon sampling was designed to avoid bias, thus we assume that dated zircons represent the sand as a whole.

Fig. 3. U/Pb age histogram of the data plotted in [Fig. 2](#) showing the six age periods that can be recognized in the dates: 0–0.25, 0.3–0.6, 0.95–1.25, 1.3–1.5, 1.65–1.95 and 2.5–3.0 Ga.

isotope analyzes and analyzed for first O and then Hf isotopes. This approach deliberately biased zircon selection towards grains most suitable for accurate O and Hf analyzes.

3.2. O isotope analyzes

Zircon grains were mounted in 25 mm diameter epoxy and polished to expose midsections. Reflected light photomicrographs and cathodoluminescence images for individual grains were used as maps to select appropriate domains for O isotope analyzes. The disk was coated with 10 nm Al prior to SHRIMP analysis. Oxygen isotope analyzes were carried out on SHRIMP II in static multicollection mode for ^{18}O and ^{16}O . A 1–3 nA primary $^{133}\text{Cs}^+$ beam was accelerated to 15 KV, and focused to around 25 μm , with O^- secondary ions extracted. The count rate for $^{16}\text{O}^-$ was typically 1×10^9 c/s. Charge neutralization was carried out with a moderate-energy (ca. 1.2 keV) electron beam focused to about 100 μm and with a 45° incident angle. The contribution from electron-induced ionization was not significant ([Ickert et al., 2008](#)).

Chips of TEM-2 ($\delta^{18}\text{O} = 8.20 \pm 0.01$ (1 σ)‰ VSMOW; [Valley, 2003](#)) were embedded into each mount and analyzed to bracket every three measurements of unknown zircons. Another standard material R33 ($\delta^{18}\text{O} = 5.55 \pm 0.04$ (1 σ)‰ VSMOW; [Valley, 2003](#)) was also analyzed as a secondary standard to check the analytical procedures ([Electronic Annex III](#)). No grains that was closer than 50 μm from the edge of the mount was analyzed. The standard data were fitted to a straight line or simple curve (no more than second order polynomial) and this fit to the standard calibration curve, which lies outside counting statistics, is the principal source of analytical uncertainty. Rough fractionation factors were determined by electrometer calibration. Measured, background corrected oxygen isotope ratios are corrected for mass bias and gain simultaneously using the relationship:

$$\frac{R_{\text{unknown}}^{\text{true}}}{R_{\text{unknown}}^{\text{measured}}} = \frac{R_{\text{std}}^{\text{true}}}{R_{\text{std}}^{\text{measured}}}$$

where $R = {}^{18}\text{O}/{}^{16}\text{O}$. This procedure simultaneously takes care of both machine drift and fractionation. The internal errors for individual analyzes in February were 0.2–0.5‰ (95% c.l.) and 0.1–0.3‰ in May. The spot-to-spot reproducibility assuming isotopic homogeneity of TEM-2 is $\pm 0.25\%$ (1 σ) in February, and $\pm 0.48\%$ (1 σ) in May. Summing the variance to the individual uncertainties gives mean R33 values of $+5.4 \pm 0.2\%$ (2 σ_m ; MSWD = 0.35) for February, and $+4.9 \pm 0.5\%$ (2 σ_m ; MSWD = 1.83) for May. The $\delta^{18}\text{O}$ values for 96 zircon grains are listed in [Electronic Annex IV](#).

3.3. Hf isotope analyzes

Hf isotopic compositions of the zircon grains were measured using a ThermoFinnigan Neptune MC-ICP-MS coupled to a second-generation ANU HelEx laser ablation system featuring an ArF excimer ($\lambda = 193$ nm) laser and fast response two-volume laser ablation cell ([Eggins et al., 2005](#)). The laser ablation site for Hf isotope analysis was centered as closely as possible to the spot where O isotope analysis was obtained, such that the region of the zircon analyzed for both Hf and O isotopes was almost identical. Data were acquired by ablating a 62 μm (diameter) laser spot, and occasionally a 47 μm laser spot in the case of small grains. The basis of our analytical method has been described previously in detail by [Harrison et al. \(2005\)](#) but has been extensively modified. Yb and Lu interference corrections are now made using the Yb and Lu isotopic ratios reported by [Chu et al. \(2002\)](#), and measured isotope intensities are corrected for differing amplifier response times on each of the faraday cups. Data were normalized to ${}^{179}\text{Hf}/{}^{177}\text{Hf} = 0.7325$, using an exponential correction for mass bias ([Maréchal et al., 1999](#); [Chu et al., 2002](#)). The Lu–Hf data for four reference zircon obtained in this study are presented in [Electronic Annex V](#). Direct measurement of ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ in four zircon standards gave results that are ~ 0.7 ϵHf units lower than those reported by [Woodhead and Hergt \(2005\)](#). A detailed assessment of all laser ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ measurements made in our laboratory over the last four years reveals this offset to be typical of our MC-ICP-MS. We confirmed the systematic bias in our instrument by measuring a standard solution of the Hf isotope reference material JMC 475. The value obtained for ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ was 0.282142 ± 5 , which is 0.000018 below the accepted value of 0.282160. Accordingly we have normalized our ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ measurements to the weighted average of the 91500, Mud Tank and Temora standards measured in each analytical session to the solution values reported by [Woodhead and Hergt \(2005\)](#). We did not include our measured values for the Monastery zircon standard in this normalization as the difference between the measured ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ values for this standard and those reported by [Woodhead and Hergt \(2005\)](#) is significantly and consistently greater than for the other standards. We attribute this difference to our particular Monastery zircon standard having a lower ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ value than that measured by [Woodhead and Hergt \(2005\)](#). We observe no evidence

for correlation between the size of applied Yb and Lu corrections and measured ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ values, despite the size of these corrections ranging from <0.1 to 20%. Measured ${}^{178}\text{Hf}/{}^{177}\text{Hf}$ values average 1.437235 ± 45 (1 σ , $n = 375$) for all standards and unknowns. Similarly, measured ${}^{174}\text{Hf}/{}^{177}\text{Hf}$ values average 0.008663 ± 21 (1 σ , $n = 375$), despite the variable and large size of the Yb correction (from 2 to almost 95%). A summary of results obtained for the standards, including normalized and un-normalized values for ${}^{176}\text{Hf}/{}^{177}\text{Hf}$, is given in [Electronic Annex V](#).

The Hf chondritic value of [Blichert-Toft and Albarede \(1997\)](#) was used for the calculation of ϵHf values and the depleted mantle ${}^{176}\text{Hf}/{}^{177}\text{Hf}$ growth line was calculated assuming a present-day ${}^{176}\text{Hf}/{}^{177}\text{Hf} = 0.28325$, similar to that of average mid-ocean ridge basalts, ${}^{176}\text{Lu}/{}^{177}\text{Hf} = 0.0384$ ([Griffin et al., 2002](#)) and a decay constant of $1.867 \times 10^{-11} \text{ yr}^{-1}$ for ${}^{176}\text{Lu}$ ([Scherer et al., 2001](#)). Hf model ages were calculated from the measured Hf isotopic compositions and U/Pb ages of the zircons and projecting the $({}^{176}\text{Hf}/{}^{177}\text{Hf})_i$ of zircons back to the depleted mantle growth curve, assuming a ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ for the crustal source region that melted to form the granitic melt from which the zircon crystallized. Two model ages were calculated. The first (T_{DM}) assumes a mean crustal ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ value of 0.0115 ([Rudnick and Gao, 2003](#)) for the zircon source region. The second (T_{DM}^{O}) uses a ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ that varies with the measured O isotopic composition of the zircon. The ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ ratio of the crust is highly variable and there are appreciable differences in this ratio between upper (0.00831), lower (0.0187) and average continental crust (0.0115) ([Rudnick and Gao, 2003](#)). Oxygen isotopes can be used to reduce uncertainty in the choice of ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ for individual grains. Zircons with near-mantle O isotope values are assumed to have crystallized from a melt derived by melting igneous rocks or a mixture between such a melt and mantle-derived melt. Zircons with a high O isotope value are assumed to have crystallized from a magma derived from a crustal source region with a high sedimentary component. We have assumed a ${}^{176}\text{Lu}/{}^{177}\text{Hf} = 0.021$ for the samples with the most primitive O isotope values ($\delta^{18}\text{O} < 5.5\%$), a value appropriate for mafic lower crust ([Kemp et al., 2006](#)) and 0.0083 for zircons with $\delta^{18}\text{O} > 9.5\%$, a value appropriate for the upper crust. ${}^{176}\text{Lu}/{}^{177}\text{Hf}$ values for zircons lying between these extremes have been determined by linear interpolation. Hf model ages are local average crustal formation ages because the zircon source region may include igneous rocks of more than one age, sediments and juvenile magma from the mantle. The Lu–Hf isotope ratios, ϵHf_i values and calculated T_{DM} and T_{DM}^{O} ages for 96 grains are listed in [Electronic Annex IV](#).

4. RESULTS

4.1. Major time intervals of zircon U/Pb ages

As noted earlier six time intervals can be recognized in the histogram of U/Pb ages: 0–0.25, 0.3–0.6, 0.95–1.25, 1.3–1.5, 1.65–1.95 and 2.5–3.0 Ga ([Fig. 3](#)). Only a few grains ($n = 6$) fall in the gaps between these time intervals. No grain older than 3.35 Ga was found and only two grains have ages older than 3.0 Ga ([Fig. 3](#)).

The six time intervals shown by the U/Pb ages of the zircons match the lithotectonic units of the North American continent in the hinterland of the Mississippi River (Fig. 1). Zircons with ages of 2.5–3.0 Ga come from the Archean Superior and Wyoming provinces, those with ages between 1.65 and 1.95 Ga were mainly produced during the Paleoproterozoic Yavapai and Mazatzal orogens, 1.3–1.5 Ga zircons are from the Granite–Rhyolite belt, 0.95–1.25 Ga grains are from the Grenville orogen, and a small group of zircons with ages between 0.3 and 0.6 Ga were produced by the Appalachian orogen. The youngest grains, with ages <0.25 Ga, formed during the Cordilleran orogen.

The percentage of the concordant grains in each of the selected time intervals, from the youngest to the oldest, is 15.5, 4.9, 38.0, 14.8, 13.4 and 13.4%. If both concordant and discordant grains are considered for each of the time intervals, these percentages change by less than 1%. We have therefore used concordant grains (because their ages are better defined) to calculate the fraction of the zircons in each of the time intervals, which is then used as a weighting factor to calculate the Hf model age for formation of the continental crust in the Mississippi basin. As noted in Section 3.1 discordant grains were not analyzed for O or Hf isotopes.

4.2. O isotope

All of the grains selected for O isotope are magmatic zircons with concordant ages that have survived overnight attack by HF acid to minimize the possibility that their $\delta^{18}\text{O}$ values were affected by post-magmatic hydrothermal alteration (e.g., Valley, 2003; Cavosie et al., 2005). As a consequence, we assume that the $\delta^{18}\text{O}$ values faithfully preserved the $\delta^{18}\text{O}$ values of the original source magmas from which the zircons crystallized.

A compilation of $\delta^{18}\text{O}$ (zircon) versus U/Pb ages, for the selected zircons for each of the time intervals, is shown in Fig. 4a. Compared with other time intervals, the Archean zircons, with U/Pb ages ranging from 2.5 to 3.0 Ga, have a small range of $\delta^{18}\text{O}$ with all but two grains lying between 5.05 and 7.35‰. The two exceptions have $\delta^{18}\text{O}$ values of 7.91 and 8.75‰. The data yield an average of $\delta^{18}\text{O}$ (zircon) = 6.40 ± 1.03 (1 σ)‰. The average $\delta^{18}\text{O}$ values for zircons from the other five time intervals are: 6.92 ± 1.55 ‰ for 1.65–1.95 Ga, 6.56 ± 1.46 ‰ for 1.3–1.5 Ga, 7.36 ± 1.84 ‰ for 0.95–1.25 Ga, 6.71 ± 1.08 ‰ for 0.3–0.6 Ga, and 6.78 ± 1.87 ‰ for <0.25 Ga (Fig. 4b), indicating that the Proterozoic and Phanerozoic zircons have more variable $\delta^{18}\text{O}$ values than the Archean grains. The majority of the Proterozoic and Phanerozoic grains have $\delta^{18}\text{O}$ values below 9.5‰ except for three grains with very high $\delta^{18}\text{O}$ values. Our data suggest a step at 2.0 Ga in the maximum common $\delta^{18}\text{O}$ from 7.5‰ in the Archean to 9.5‰ in younger zircons.

4.3. Hf isotope

As noted earlier, a Hf model age (T_{DM}) is assumed to be the average age of the crustal source region that melted to form the host granitic magma from which the analyzed zircon crystallized. We therefore interpret the T_{DM} and T_{DM}^{O}

ages to date crustal growth. Histograms, showing the comparison of T_{DM}^{O} ages and U/Pb ages for each of the time intervals, are given in Fig. 5. Compared with much more variable T_{DM}^{O} ages of the young grains (U/Pb age <0.25 Ga, Fig. 5a), zircons in other time intervals have a relatively small range in T_{DM}^{O} ages and their T_{DM}^{O} ages are closer to their U/Pb ages (Fig. 5b–f). It follows that young grains are also more variable in εHf_i values than the grains in the other five age ranges (Fig. 6).

The effectiveness of our procedure can be assessed by comparing our results for 12 1.3–1.5 Ga zircons from the Mississippi with the Goodge and Vervoort (2006) study of 33 1.3–1.5 Ga anorogenic granites from southern North America. If their data are recalculated, using a $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of 0.0115, the average Hf model age obtained is 1.94 ± 0.14 Ga (1 σ), which is similar to our average T_{DM} age of 1.96 ± 0.25 Ga and T_{DM}^{O} age of 2.13 ± 0.37 Ga.

4.4. Comparison with previous work

A study of U/Pb ages and Hf isotopes in zircons from the Mississippi River by Izuka et al. (2005) concluded that crustal reworking was an important process in the evolution of the North American continental crust and identified the period between 2.0 and 1.3 Ga as one of rapid crustal growth periods. However, Izuka et al. (2005) made a virtue of analyzing all of the zircon grains they separated for both U/Pb and $^{176}\text{Hf}/^{177}\text{Hf}$ to avoid bias whereas we only analyzed grains with no age zoning for Hf isotopes. Izuka et al. (2005, Fig. 2A) identified three peaks in their U/Pb age data, including a sharp <0.2 Ga peak, a broad 0.9–1.8 Ga peak and a small 2.6–2.8 Ga peak, whereas we resolved six distinct peaks, which correspond to known tectonic events, i.e., <0.25 Ga corresponding to the Cordilleran orogen, 0.3–0.6 Ga to the Appalachian orogen, 0.95–1.25 Ga to the Grenville orogen, 1.3–1.5 Ga to the Granite–Rhyolite belt, 1.65–1.95 Ga to the Paleoproterozoic Yavapai and Mazatzal orogens, and >2.5 Ga to the Archean terranes. It is unlikely that this difference is due to sampling because the samples were collected from similar locations. We are confident that the peaks we resolved are real as they correspond to known tectonic events. Another important difference between our study and that of Izuka et al. (2005) is that we analyzed zircons for O isotopes to help constrain the $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of the zircon source region whereas they did not.

5. DISCUSSION

5.1. Sediment component in each of time intervals

The oxygen isotopic composition of igneous zircons provides a record of intracrustal recycling and crust–mantle interaction (Valley et al., 2005). Zircons in equilibrium with pristine mantle-derived melts theoretically have $\delta^{18}\text{O}$ values of 5.3 ± 0.3 ‰ (Valley et al., 1998). Zircons with $\delta^{18}\text{O} < 5.0$ ‰ are assumed to have protoliths or contaminants that have exchanged with surface waters at high-temperature (Valley et al., 2005). The dominantly high $\delta^{18}\text{O}$ reservoir on Earth is supracrustal materials, which include

Fig. 4. (a) Plot of $\delta^{18}\text{O}_{\text{‰}}$ versus U/Pb ages for the detrital zircons from the Mississippi River and (b) the average O isotope compositions for the zircons from each of the time intervals.

sedimentary rocks with $\delta^{18}\text{O} = 10\text{--}40\text{‰}$ and hydrothermal altered oceanic crust with $\delta^{18}\text{O} = 10\text{--}20\text{‰}$ (Eiler, 2001 and references therein). Therefore zircons crystallized from a mixed source magma, which included both mantle-derived and sedimentary components, will have $\delta^{18}\text{O}$ values above the mantle $\delta^{18}\text{O}$ value of $5.3 \pm 0.3\text{‰}$. However, the variability of $\delta^{18}\text{O}$ in supracrustal materials makes it impossible estimate the percentage of sediment and/or altered oceanic crust in the source region of the magmas from which the zircons crystallized.

In the Mississippi basin, the Archean zircons show little variation with most grains lying between 5.05 and 7.35‰ , comparable with the $\delta^{18}\text{O}$ values of the zircons from the Archean rocks elsewhere (e.g., Valley et al., 2005). The lower maximum $\delta^{18}\text{O}$ values of the Archean zircons, compared with those of the Proterozoic and Phanerozoic zircons, suggest that Archean zircons have crystallized from melts that

originated from a source region with less sediment produced by low-temperature weathering than their Proterozoic and Phanerozoic equivalents.

Proterozoic and Phanerozoic zircons show an increase in their maximum $\delta^{18}\text{O}$ to 9.5‰ , with rare grains having values as high as 13‰ . Two factors contribute to the high maximum $\delta^{18}\text{O}$ in Proterozoic and Phanerozoic zircons: (1) after 2.5 Ga supracrustal sequences became dominated by shales, chemical sediments, limestones and cherts with $\delta^{18}\text{O}$ higher than altered Archean basalts (Land and Lynch, 1996; Shields and Veizer, 2002; Perry and Lefticariu, 2003), and (2) an increase in the ratio of sedimentary to igneous rocks in the source regions of the magmas from which zircons crystallized (c.f., Valley et al., 2005).

The change of $\delta^{18}\text{O}$ in this study is similar to the trend described by Valley et al. (2005) and Kemp et al. (2006).

Fig. 5. Histogram of T_{DM}^O model ages and U/Pb ages for each of the selected time intervals.

A compilation by Valley et al. (2005) also found that most Archean zircons have $\delta^{18}O < 7.5\text{‰}$ and most Phanerozoic and Proterozoic zircons have $\delta^{18}O < 10\text{‰}$. However, instead of the continuous increase in the maximum $\delta^{18}O$ doc-

Fig. 6. Plot of ϵHf_i versus U/Pb ages.

umented by Valley et al. (2005) and Kemp et al. (2006), our data suggest a small step increase in the range of observed $\delta^{18}O$ values from 2.5 to 2.0 Ga, i.e., most zircons older than 2.5 Ga have $\delta^{18}O$ lying between 5 and 7.5‰ whereas most zircons younger than 2.0 Ga vary more widely from 4.5 to 9.5‰ with a few high and low $\delta^{18}O$ exceptions (Fig. 4a). This may be because the data set reported by Valley et al. (2005) is biased towards samples of geological interest with higher $\delta^{18}O$, whereas the detrital data set reported here are more likely to be random. However, the difference may also be due to our smaller data set.

A feature of the data in this study is that there is little change in the average $\delta^{18}O$ value for the selected time intervals, if $\delta^{18}O$ values less than 4.5‰ are disregarded. This point is illustrated in Fig. 4b, which shows that the mean for other five selected time intervals lies only $0.2\text{--}1.0\text{‰}$ higher than the average $\delta^{18}O$ for the Archean zircons (6.4‰), implying that there has been no significant increase in the average $\delta^{18}O$ zircon magma source region with time. It is therefore the range, rather than the mean of $\delta^{18}O$ values, that increases in rocks younger than 2.5 Ga, implying a greater diversity in the source region of younger granites, with both sediment and high-temperature altered crust becoming increasingly important in younger granite source regions. A notable exception is the Grenville-aged zircons, which have a significantly higher average $\delta^{18}O$ than zircons from any other period, implying an unusually high sedimentary fraction in the source region for these zircons. This feature was also documented by Valley (2003).

5.2. Identifying juvenile continental crust

Juvenile continental crust, by definition, is the crust derived directly from the mantle. Zircons derived from juvenile crust should have $\delta^{18}O$ values lying within the mantle range of $5.3 \pm 0.3\text{‰}$ and ϵHf_i values that lie within uncertainty of the depleted mantle growth curve for Hf. Izuka et al. (2005) found that 8% of the zircons they analyzed from the Mississippi lay within two ϵHf units of the depleted mantle growth curve and concluded that there were significant additions of juvenile material to the North American continental crust between 1.3 and 2.0 Ga and

after 2.5 Ga. Our data do not support this conclusion. Fig. 6 shows variations in $\varepsilon_{\text{Hf}_i}$ for zircons with different $\delta^{18}\text{O}$ plotted as a function of their U/Pb age. None of the Mississippi zircons have $\varepsilon_{\text{Hf}_i}$ consistent with derivation from juvenile crust. We therefore conclude that zircons derived from juvenile continental crust are rare or absent in the Mississippi basin and that the overwhelming majority of grains crystallized from remelted continental crust. Note this interpretation does not preclude the possibility that the North American craton contains mafic rocks that did not crystallize zircon or that many of the granitic intrusions include a significant component of mantle-derived magma that has mixed with crustal melts. The grains with mantle-like $\delta^{18}\text{O}$ values are interpreted to have crystallized from remelting of mantle-derived precursors where the time difference between primitive crust formation (mantle extraction) and remelting that led to zircon crystallization can be 100's of Myr.

5.3. Crustal incubation time

Basaltic melts, when they leave the mantle, have a SiO_2 activity defined by the olivine-orthopyroxene buffer, which is below that of the baddeleyite–zircon buffer. As a consequence, baddeleyite is the expected Zr-bearing phase in basaltic melts that are not contaminated by continental crust and this prediction is consistent with observations. Zircon crystallization therefore requires a minimum of two stages in melt generation: (1) extraction of a primitive, zircon-free basaltic crust from the mantle and (2) melting of that primitive crust to form a felsic magma from which zircon can crystallize. The heat required to melt the primitive crust can be transferred from the mantle by conduction or via injection of juvenile basaltic into the crust.

Campbell and Hill (1988) recognized two stages in the formation of Archean granite–greenstone terrains: (1) separation of a primitive continental crust from the mantle and (2) cratonisation, which involves widespread melting of the primitive continental crust to form granites. McLaren and Sandiford (2001) have applied a similar hypothesis to the Proterozoic Mount Isa area of Australia. The first-stage primitive continental crust is expected to have a relatively uniform distribution of low-melting temperature components, including the heat producing elements K, Th and U. The distribution of K, Th and U are particularly important because about half of the Earth's heat producing elements are found in continental crust. If the concentration of these elements in the lower crust is high, a significant source of heat will be trapped under an upper crustal insulating blanket of basalt and sediment, which leads to heating of the lower crust. In an extreme case this could result in melting of the lower crust and the formation of granite, especially in the Archean, when radioactive heat production was the highest (West and Mareschal, 1979).

Granite production, however, is rarely random and is usually linked to specific tectonic events such as subduction or the arrival of a new mantle plume, suggesting that an external heat source is required to trigger periods of widespread melting of the lower continental crust (Campbell and Jarvis, 1984). Although radioactive heat production

in the lower crust is supplementary to asthenospheric heat sources (including basalt) in most granite melting events it nevertheless plays an essential role in preparing the melting of the lower crust by heating it close to its solidus temperature. The movement of granitic melt from the lower crust to the upper crust depletes the lower crust in heat producing elements, which has two fundamental implications for crustal evolution: (1) it reduces the temperature of the lower crust, which increases its strength (Sandiford et al., 2002; Sandiford and McLaren, 2002) and (2) removing both the heat producing elements and the low-melting temperature components from the lower crust makes further melting difficult or impossible (Campbell and Jarvis, 1984; Campbell and Hill, 1988; Sandiford et al., 2002). As a consequence, widespread melting of the lower crust results in cratonisation. The U/Pb and the Hf model ages of the zircons, and the time gap between them, are consistent with two principal stages in the evolution of continental crust. Hf model ages date stage one, separation of primitive crust from the mantle; whereas U/Pb ages date the stage two, widespread crustal melting and cratonisation. The difference between these ages, which we define as the *crustal incubation time*, is the time difference between initial formation of the continental crust and later cratonisation.

Fig. 7 shows the crustal incubation times for the six tectonic periods recognized in the Mississippi basin from the U–Pb dating. The average incubation time is 890 ± 460 Myr and it varies from 620 ± 320 Myr for the oldest period to 1270 ± 820 Myr for the youngest. Ignoring local variations in the concentration of heat producing elements in the primitive lower crust, the incubation time depends on a combination of the time taken for radioactive decay to raise the temperature of the lower crust to or close to its solidus temperature, and the time taken to expose it to an asthenospheric source of heat such as a subduction zone or mantle plume. The latter is essentially random and controlled by tectonic processes. The time taken to heat the lower crust to its melting temperature is controlled by the time available for radioactive decay and the age of the crust. The role of time is thus obvious. Heat released by radioactive elements continues to raise the temperature of

Fig. 7. Plot of crustal incubation time versus U/Pb age for each of the selected time intervals.

the lower crust until it reaches a steady-state when the rate of dissipation of heat, due to the combined effects of conduction and advection, is equal to the rate of heat generation. The rate of radioactive heat production decreases with time because the fraction of radioactive isotopes in the heat producing elements declines and is about a factor of two times less today than it was in the Archean. As a consequence old primitive lower crust heated faster than young primitive lower crust today.

We note that no zircons from the Mississippi basin have the crustal incubation time less than 100 Myr and that the average incubation times for the six U/Pb age time intervals lie within 325 Myr of 945 Myr, suggesting that accumulation of radioactive heat in the lower crust plays an important role in preparing the lower crust for melting in the Mississippi basin. This view is supported by the observation that there appears to be an overall increase in the incubation time with decreasing age (Fig. 7), which could be attributed to the decline in rate of radioactive heat production with time. However, this conclusion may be misleading because it is based on data from a single continent.

5.4. Comparison of Hf model age and Nd model age

The differences between Hf and Nd model ages for continental crust formation, for samples with model age of about 2.0 Ga, are illustrated in Fig. 8. For both systems the fractionation between parent and daughter isotopes is much greater between depleted mantle and continental crust than it is for intracrustal processes. It is therefore mantle-crust fractionation that controls both Hf and Nd model ages. Nd and Sm are geochemically similar and little fractionation of these elements occurs during weathering and crustal melting. Neodymium model ages for sedimentary rocks assume that the Sm/Nd measured in the sediments is the same as those of the Nd source region and that crustal processes do not change this ratio over the history of the region. The effect of an error in this assumption is illustrated in Fig. 8a. If the Sm/Nd ratio used in the calculation is changed from an upper crustal value of 0.174 to a lower crustal value of 0.254, the calculated model ages varies by only 42 Myr for a rock with a model age of about 2.0 Ga.

Lu and Hf on the other hand have distinct geochemical properties and their $^{176}\text{Lu}/^{177}\text{Hf}$ ratios vary significantly within the crust. The effect of assuming different $^{176}\text{Lu}/^{177}\text{Hf}$ ratios for the zircon source region, for two zircons with different U/Pb ages, is illustrated in Fig. 8b. A zircon with a U/Pb age of 1.5 Ga and a $^{176}\text{Hf}/^{177}\text{Hf}$ of 0.281789 gives a model age of 2.10 Ga if an upper crustal $^{176}\text{Lu}/^{177}\text{Hf}$ ratio age is assumed but 2.24 Ga if a lower crustal value is used, a difference of 140 Myr. For a zircon with the same $^{176}\text{Hf}/^{177}\text{Hf}$, but a U/Pb age of 0.5 Ga, the model age become 2.32 Ga if an upper crustal Lu/Hf ratio is used and 2.81 Ga for a lower crustal value, a difference of 490 Myr. Clearly Nd model ages of sedimentary rocks are more robust than Hf model ages because the former are less susceptible to errors in the estimated parent–daughter ratio of the isotopic system. They are therefore better suited to giving the average model age of sedimentary source regions

Fig. 8. Comparison between Nd and Hf model ages showing the differences between assuming upper and lower crustal parent/daughter ratios. The Nd model age assumes measured $^{143}\text{Nd}/^{144}\text{Nd}$ of 0.510191 and $^{147}\text{Sm}/^{144}\text{Nd}$ isotopic ratios of 0.1048 for the upper crust, 0.1174 for the average crust and 0.1532 for the lower crust. The assumed zircon $^{176}\text{Hf}/^{177}\text{Hf}$ for the Hf model age is 0.281789 and the upper and lower crust are assumed to have $^{176}\text{Lu}/^{177}\text{Hf}$ of 0.0083 and 0.0215, respectively.

but cannot resolve individual periods of crustal growth whereas Hf model ages can. Furthermore the shortcoming of Lu/Hf uncertainty can be minimized if O isotopes are used to constrain the Lu/Hf ratio of the crustal source region for the modeling mantle extraction ages of individual zircons.

Hf model crustal formation ages have been calculated for each of the U/Pb age time intervals recognized in the Mississippi basin (Table 1). Average Hf model crustal formation ages are obtained by weighting the model ages in each of the U/Pb age time intervals. Two methods have been used, both of which are described in the caption of Table 1: (1) weighting by the fraction of zircons in each of the six selected time intervals as determined by the conventional U–Pb dating method and shown in Fig. 3 and (2) weighting by the proportion of the basement areas of granitoids and high-grade metamorphic rocks in North America, which are sources of primary zircons, in each of the six tectonic units shown in Fig. 1. The average Hf T_{DM} and T_{DM}^{O} crustal formation age are 1.94 and 2.13 Ga when the data weighted by the fraction of zircons, and 2.35 and 2.53 Ga when weighted by area (Table 2). The average Hf model ages, weighted by the fraction of zircons in each of U/Pb

Table 1

Average variable Lu/Hf (T_{DM}^O) and fixed Lu/Hf (T_{DM}) model ages for each of the time intervals resolved by the U/Pb dates of zircons from the Mississippi River.

	U/Pb age time intervals	Average Hf T_{DM}^O (Ga) ^a	Average Hf T_{DM} (Ga) ^b	Fraction of zircon ^c	Proportion of outcrop area of source rocks ^d
1	0–0.25 Ga	1.40 ± 0.80	1.10 ± 0.55	0.155 (22 grains)	0.072 (cordillera orogen)
2	0.3–0.6 Ga	1.41 ± 0.24	1.18 ± 0.21	0.049 (7 grains)	0.043 (Appalachian orogen)
3	0.95–1.25 Ga	1.89 ± 0.39	1.74 ± 0.25	0.380 (54 grains)	0.215 (Grenville orogen)
4	1.3–1.5 Ga	2.13 ± 0.37	1.96 ± 0.25	0.148 (21 grains)	0.157 (Granite–rhyolite belt)
5	1.65–1.95 Ga	2.74 ± 0.62	2.50 ± 0.38	0.134 (19 grains)	0.120 (paleoproterozoic belt)
6	2.5–3.0 Ma	3.31 ± 0.28	3.16 ± 0.24	0.134 (19 grains)	0.393 (Archean provinces)

^a Hf model ages calculated with $^{176}\text{Lu}/^{177}\text{Hf}$ ratios that vary with the O isotopic values of the individual grains.

^b Hf model ages calculated with a fixed average crustal $^{176}\text{Lu}/^{177}\text{Hf}$ ratios of 0.0115.

^c The fraction of the concordant grains shown in Figs. 2 and 3 that lie within the designated U–Pb time intervals.

^d The factor used for weighting by area has been determined by measuring the area covered by each of the six tectonic units as shown in Fig. 1. Each of these areas includes both primary zircon-bearing rocks, such as granitoids and high-grade metamorphic rocks, and sedimentary rocks which contain zircons that have been recycled from older tectonic periods. Including the area covered by ancient sedimentary rocks in the area of a tectonic unit would lead to an over estimation of the amount of continental crust that formed during that period. The area covered by sediment has therefore been excluded and the weighting factor has been calculated for each of the recognized age units by multiplying its area by the fraction of primary zircons-bearing rocks it contains (granitoids + high-grade metamorphic rocks). The distribution of primary zircon-bearing rocks in each of the tectonic units is based on available information. Data source: Archean from Card (1990), Paleoproterozoic orogens (Yavapai and Mazatzal), Granite–rhyolite belt and Grenville from Whitmeyer and Karlstrom (2007), Appalachia from Sinha et al. (1989) and Cordillera from Bally et al. (1989).

age intervals, is a minimum estimate for the average age of crust growth for two reasons. First, older zircons are more likely to be destroyed than young zircons during transport erosion and sedimentation because they are more likely to suffer radiation damage and because they are more likely to be affected by multiple cycles of erosion and sedimentation. Second, rivers derive a disproportionate fraction of their load from rapidly eroding young orogenic areas that will normally have a high fraction of young crust. In the case of the Mississippi River young zircons from the western Cordillera are over represented in the river sedimentary load relative to old zircons from the Archean Canadian Shield. For these reasons we suggest that weighting by area gives a better estimate of the average age of formation of the North American continental crust. However this approach also has its limitations: there is considerable uncertainty in the area covered by the different tectonic periods identified from the U–Pb dating, with different authors showing different areas on their maps (e.g., Goodge et al.,

2004; Iizuka et al., 2005; Whitmeyer and Karlstrom, 2007), and as a consequence, the fraction of each of the areas covered by primary zircon-bearing igneous and high-grade metamorphic rocks is difficult to estimate.

The Hf model crustal formation ages that use an average crustal $^{176}\text{Lu}/^{177}\text{Hf}$ ratio (T_{DM}) are directly comparable to the Nd model ages for the Mississippi River. The average Hf T_{DM} age (1.94 Ga), weighted by the fraction of zircons (Table 2), is in good agreement with the Nd model age determined from the load of the suspended material in the Mississippi River (1.7 Ga) and from the suspended load of all rivers in the North America (1.8 Ga) (Goldstein and Jacobsen, 1988). Similarly, the average Hf T_{DM} age weighted by area (2.35 Ga) compares well with the average Nd model age of the basement rocks for North America (2.36 Ga) (Goldstein and Jacobsen, 1988). Average T_{DM}^O , which provide a better estimate of the average age of the preserved continental crust, are ca. 200 Myr older than the T_{DM} ages. We suggest that the mean Hf T_{DM}^O age of

Table 2

Comparison of average Hf model ages (T_{DM} and T_{DM}^O) and Nd model ages (T_{DM}) of the North American continental crust.

Average model crustal formation age	Hf T_{DM}^O and T_{DM} age (Ga)		Nd T_{DM} age (Ga) (after Goldstein and Jacobsen, 1988)		
	Detrital zircons from Mississippi		River suspended material in Mississippi	River suspended material in the North America	Basement rocks in the North America
Weighted by load ^a	2.13 ^c	1.94 ^d	1.7	1.8	
Weighted by area ^b	2.53 ^c	2.35 ^d	2.1	2.31	2.36

^a Hf model age is weighted by fractions of zircons in each of the identified U/Pb time intervals; Nd model ages determined by analyses of suspended material in the rivers.

^b Hf model age is weighted by the relative proportion of outcrop area of primary zircon-bearing rocks as discussed in Table 1; Nd model ages are weighted by area.

^c Hf model ages calculated with $^{176}\text{Lu}/^{177}\text{Hf}$ ratios that varying with the O isotopic value of the grain as discussed in the text.

^d Hf model ages calculated with a fixed average crustal $^{176}\text{Lu}/^{177}\text{Hf}$ ratio (0.0115).

2.53 Ga gives a better estimate of the average formation age of the North American continental crust.

5.5. Continuous or episodic continental crust growth?

A key unresolved question in the evolution of the continental crust is whether growth was continuous (Moorbath, 1977; McLennan and Taylor, 1982; Fyfe, 1978; Reymer and Schubert, 1984) or episodic (Condie, 1998). Nd model ages for clastic sediments give an average age at which the source material for the sediments was extracted from the mantle (O’Nions et al., 1983; Arndt and Goldstein, 1987). They therefore date *average* not specific mantle extraction events. Likewise, the Hf model age of a zircon, which crystallized from a magma produced by melting a sedimentary rock, also gives an *average* model crustal extraction age of the source regions that contributed material to the sediment; it is equivalent to the Nd model age for the sediment. Therefore, it is important to identify zircons that crystallized from a magma produced by melting a source rock with a low fraction of sediments when interpreting the Hf model ages of continental crust extraction events. Only

the zircons having O isotopic values lying within the mantle range ($\delta^{18}\text{O} = 5.3 \pm 0.3\text{‰}$) can be interpreted to have come from a magmatic precursor that contain no significant sedimentary component. The Hf model ages for such zircons date *specific* crustal extraction events; the time the continental crust was extracted from the mantle.

Ideally, only grains with $\delta^{18}\text{O}$ values between 5.0 and 5.6‰ should be considered in the identification of the model age of specific crustal extraction events. Incorporating zircon Hf model ages of grains with $\delta^{18}\text{O} > 5.6\text{‰}$ smears the crustal extraction ages by mixing the specific model crustal extraction age of the magmatic component in the continental crust with the average model age of the sedimentary component. In practice, the range has been extended to 4.5–6.5‰, taking into account the errors associated with measuring $\delta^{18}\text{O}$ by SHRIMP and recognizing that incorporating a small amount of sediment into the source region will produce minimal age smearing.

The grains with $\delta^{18}\text{O} = 4.5\text{--}6.5\text{‰}$ make up ~53% data set of this study. Fig. 9 shows histograms of Hf T_{DM}^{O} ages. Fig. 9a and c show the T_{DM}^{O} ages for all grains, whereas Fig. 9b and d show only grains with $\delta^{18}\text{O}$ between 4.5

Fig. 9. Histogram showing T_{DM}^{O} model ages for all selected zircons (a and c) and T_{DM}^{O} model ages for zircons with $\delta^{18}\text{O} = 4.5\text{--}6.5\text{‰}$ (b and d). The T_{DM}^{O} ages in (a) and (b) has been weighted according to the fraction of zircons in whichever of the six U/Pb age time intervals recognized in Fig. 3 as follows: weighting factor = (percentage of zircons in the U/Pb age time interval $\times$ 20)/number of zircons analyzed for Hf analyzes from that age interval. The T_{DM}^{O} in (c) and (d) has been weighted according to the relative proportion of outcrop areas of zircon-bearing rocks (granitoids and high-grade metamorphic rocks) in the six tectonic units corresponding to the same time intervals as follows: weighting factor = (proportion of outcrop areas of zircon source rocks in each of tectonic units $\times$ 20)/number of zircons analyzed for Hf analyzes from that age interval. The number 20 is arbitrary and is introduced to bring the minimum-weighting factor close to 1.

and 6.5‰. Fig. 9a and b are weighted by the fraction of zircon U/Pb ages in each of the six time intervals, whereas those in Fig. 9c and d are weighted by area of primary zircon-bearing rocks in each of six tectonic units. The data suggest continuous growth of the North American continental crust between 1.2 and 3.7 Ga but with two strong broad peaks in growth recognized in all histograms, one between 1.6 and 2.2 Ga, and the other between 2.9 and 3.4 Ga (Fig. 9a and c). The difference in the two weighting approaches affects the relative heights of the age peaks but not their position. The younger 1.6–2.2 Ga peak is enhanced relative to the older 2.9–3.4 Ga peak when the data are weighted by the fraction of zircons (Fig. 9a), whereas the older peak is enhanced when weighted by area (Fig. 9c). The two peaks recognized in this study are remarkably similar to two dominant peaks in continental growth of Gondwana identified by Hawkesworth and Kemp (2006) from four S-type granites and two Ordovician sandstones from Eastern Australia, which have peaks at 1.7–1.9 Ga and 2.9–3.1 Ga, suggesting that continental growth is episodic and that these were important periods of the crustal growth. However, we stress that more data are needed from other continents before the hypothesis of episodic global continental growth can be accepted with confidence.

Comparing the histograms of all zircons (Fig. 9a and c) with those only with mantle $\delta^{18}\text{O}$ values (Fig. 9b and d), the 2.9–3.4 Ga peaks are similar because the majority of Archean zircons have mantle $\delta^{18}\text{O}$ values. However, the 1.6–2.2 Ga peaks seen in Fig. 9b and d broadens to 1.3–2.4 Ga with the possibility of two sub-peaks when all zircons are considered (Fig. 9a and c), indicating some smearing occurs if all grains are included. However, the Hf model age distribution within the 1.6–2.2 Ga range remains broadly similar, suggesting that smearing has not had a major influence on the results. This conclusion contrasts with the results of Hawkesworth and Kemp (2006), in which the two tight Hf model age peaks seen for the grains with $\delta^{18}\text{O} < 6.5\text{‰}$, broaden significantly (become smeared) if all data are considered.

5.6. Zircon Hf model age constraints on crustal growth models

According to the Armstrong (1981, 1991) steady-state hypothesis for crustal growth, the continental crust has remained approximately constant or even decreased in volume since the early Archean (curve 1 in Fig. 10a and b), whereas most other hypotheses favored continuous growth models in which the crust has grown by incremental extraction from the mantle. The continuous growth models propose either regular increase in volume (Dewey and Windley, 1981), or that growth was more rapid in the Archean than in later times (McLennan and Taylor, 1982). Because the major crust extraction periods appear to be only slightly distorted by smearing as shown in Fig. 9 all grains were used to calculate cumulative Hf T_{DM} and T_{DM}^{O} model ages growth curves for the preserved continental crust in the Mississippi basin. Growth curves were calculated in two ways: (1) the data were weighted by the fraction of zircon

Fig. 10. Growth models of the North American continental crust weighted in two ways: (i) data are weighted by the fraction of zircons in each of time intervals (curve 4) and (ii) data are weighted by the area of primary zircon-bearing rocks in each of the tectonic units (curve 5). References for curves: 1-after Armstrong (1991); 2-after McLennan and Taylor (1982); and 3- after Goldstein and Jacobsen (1988). The upper figure (a) gives model ages that assume that the $^{176}\text{Lu}/^{177}\text{Hf}$ ratio varies with the measured O isotope value of the zircon, and the lower figure (b) uses a constant average crustal $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of 0.0115.

U/Pb ages in each of six time intervals (curve 4 in Fig. 10a and b), and (2) the data are weighted by the areas of primary zircon-bearing rocks in the same time intervals (curve 5 in Fig. 10a and b). All curves lie closer to the continuous growth model of McLennan and Taylor (1982) (curve 2 in Fig. 10a and b) than the Armstrong (1981, 1991) steady-state crustal growth curve. Fig. 10a and b also shows cumulative crustal growth curve of Goldstein and Jacobsen (1988) calculated from area weighted Nd model ages (curve 3). This curve is most closely comparable with the constant $^{176}\text{Lu}/^{177}\text{Hf}$ Hf model age (T_{DM}) curve and, as can be seen from Fig. 10b, there is excellent agreement between the two methods of calculating crustal growth for North America. The more realistic T_{DM}^{O} model Hf ages growth curve (Fig. 10a, curve 5) gives ages for a given fraction of continental growth that are 200–300 Myr older than T_{DM} model (compare curve 5 in Fig. 10a and b).

The growth curve most comparable with the Iizuka et al. (2005) study is the T_{DM} curve that was weighted by zircon fraction (curve 4 in Fig. 10b). This curve suggests that

about 30% and 80% of the preserved continental crust was extracted from the mantle by 2.3 and 1.4 Ga, respectively, and that there was only one major crustal growth period, between 1.6 and 2.4 Ga, similar to the results obtained by Iizuka et al. (2005). However, the weighted area T_{DM}^O curve indicates that 50% of the preserved continental crust was extracted from the mantle by 2.9 Ga and 90% by 1.6 Ga, respectively, and that there were two important periods of crustal growth; 3.4–2.9 Ga and 2.2–1.6 Ga. The curves weighted by zircon fraction give minimum estimates of the rate of growth of continental crust for the region because of the bias to the younger ages as discussed earlier, whereas the area weighted curves give more realistic estimates, despite their limitation. Note these results cannot preclude the Armstrong (1981, 1991) hypothesis if early-formed continental crust was recycled into the mantle before leaving isotopic evidence for its existence in the preserved continental crust.

6. CONCLUSION

Zircons derived from juvenile crust are rare or absent in the Mississippi basin. Two principal stages of crustal evolution are recognized; (i) separation of a primitive continental crust from the mantle and (ii) melting of the lower part of that crust to form granitoids. Once the low-melting temperature and heat producing elements have been removed from the lower crust it becomes refractory (cratonisation) and further melting is unlikely. Zircon Hf model ages date stage 1 and U/Pb ages stage 2. The time difference between stages 1 and 2, which we define as the crustal incubation time, averages 890 ± 460 Myr and increases with decreasing age in the Mississippi basin.

The zircon Hf model ages were weighted according to (i) the fraction of zircons in each of the tectonic periods recognized from the U–Pb dating and (ii) the area of primary zircon-bearing rocks in the Mississippi basin basement. The Hf T_{DM} , weighted by zircon age fraction, which is the most comparable with the Nd model age, is slightly older at 2.0 Ga than the 1.7 Ga Nd model age for the suspended river load of the Mississippi River. The T_{DM} age of 2.35 Ga, weighted by area agrees with the 2.36 Ga weighted by area Nd model age for the basement rocks of North America (Table 2), but our preferred variable $^{176}\text{Lu}/^{177}\text{Hf}$ T_{DM}^O age is 170 Myr older. Two distinct periods of continental crust growth can be recognized in the Hf T_{DM}^O ages; 1.6–2.2 and 2.9–3.4 Ga. These ages are remarkably similar to periods of growth in Gondwana recognized by Hawkesworth and Kemp (2006), suggesting that these may be periods of global continental crust growth, although we caution that this conclusion is based on limited data.

Finally, our study of oxygen isotopes in zircons from the Mississippi basin confirms the finding of Valley et al. (2005) and Kemp et al. (2006) that there is an increase in maximum $\delta^{18}\text{O}$ value of zircons at the Archean–Proterozoic boundary. However, there is little change in the average value of $\delta^{18}\text{O}$ with time, suggesting that an increase in the sediment content of the younger granitoid source regions is partially balanced by a high fraction of high-temperature hydrothermally altered low $\delta^{18}\text{O}$ crust.

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APPENDIX A. SUPPLEMENTARY DATA

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.gca.2008.10.037](https://doi.org/10.1016/j.gca.2008.10.037).

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