Crustal evolution of the South Mayo Trough, western Ireland, based on U–Pb ages and Hf–O isotopes in detrital zircons

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Abstract: Ordovician strata of the South Mayo Trough in western Ireland contain clastic deposits that represent materials eroded from a large and diverse continental area over a time scale that spans much of the Earth’s history. Therefore, it is a useful region to use detrital zircons to construct a continental crustal growth model. Here, we report integrated U–Pb, Lu–Hf and O isotope measurements obtained from in situ analyses of 160 zircons from the South Mayo Trough. U–Pb zircon crystallization ages define three major magmatic episodes of crustal reworking in the Archaean (Lewisian), Mesoproterozoic (Grenville), and Ordovician (Grampian). These data, together with oxygen isotope data and Hf model ages, suggest that crustal growth, recorded in the strata of the South Mayo Trough, started at ~4 Ga and continued until 1.4 Ga, with two major growth periods at 2.3–2.1 and 2.0–1.5 Ga. We find that the crustal incubation time is decoupled from the duration of supracrustal alteration processes; some zircons with very long crustal incubation times have pristine mantle δ18O signatures suggesting minimal low-temperature surface processing in their source regions. Identifying such zircons is the key for future studies in constructing realistic net continental crustal growth models unaffected by crustal recycling.

Supplementary materials: Data tables for U–Pb, Lu–Hf and oxygen isotopes for detrital zircons from South Mayo Trough, as well as plots of values for zircon standards (δ18O for r33, U–Pb ages for 91500 and R33, and 176Hf/177Hf for 91500, GJ-1, and Plešovice) and reverse concordia plots of zircon samples are available at www.geolsoc.org.uk/SUP18543.

The South Mayo Trough in western Ireland is unique in the Appalachian–Caledonian arc–continent collision system (Dewey 2005). Provenance studies, including U–Pb geochronology in zircons and Ar–Ar dating in white micas, have revealed that sediment in the South Mayo Trough derived from igneous and metamorphic complexes from the British Isles, Greenland, Laurentia, and Baltica, and that zircon ages cluster around three important periods of crustal evolution; the Lewisian (Archaean), Grenville orogeny (Mesoproterozoic) and Grampian orogeny (Ordovician) (Friedrich et al. 1999; Flowerdew et al. 2005; Chew et al. 2007; Clift et al. 2009; McConnell et al. 2009; Mange et al. 2010). The Ordovician strata of the South Mayo Trough were deposited as a conformable sequence, first in the forearc of a north-facing oceanic arc, then in a synorogenic basin above the Grampian orogen. This was followed by deposition in an extensional hanging-wall basin above the exhuminig Dalradian metamorphic complex and, finally, in an Andean-type margin successor basin (Mange et al. 2010). This depositional history and the relationship of the South Mayo Trough to the Grampian orogeny, Laurentian basement and the pre- and post-collision arcs make it an ideal location for the study of crustal evolution using detrital minerals.

The recent rapid development of new analytical techniques has allowed the analyses of U–Pb ages, Lu–Hf and O isotopes on a single zircon (Hawkesworth & Kemp 2006; Kemp et al. 2006, 2007; Li et al. 2009, 2010a,b), which provide us with new information on the growth rate of continental crust. The crustal building process is considered to proceed in two stages: (1) extraction of primitive basaltic juvenile crust from the mantle (e.g. mid-ocean ridges); (2) remelting of primitive crust to form felsic crust in island and continental arc settings. Crustal remelting leaves cold refractory residues devoid of heat-producing elements (K, U, Th) in the lower crust, which leads, eventually, to continental cratonicization (Campbell & Hill 1988; Wang et al. 2011). The age of both stages can be uncovered, potentially, by analysing zircons. Hf model ages of zircons are widely accepted to date the first stage, whereas U–Pb ages of zircons date the second stage. The age difference between the two is known as the crustal residence time (e.g. Iizuka et al. 2010) or crustal incubation time (Wang et al. 2009, 2011). For the rest of the paper, we shall adopt the term ‘crustal incubation time’. In our opinion, this term fully captures the image of crustal maturation time and processes. ‘Crustal residence time’ may be perceived as the material no longer residing in the crust after this time interval, which would be incorrect.

Oxygen isotopes are sensitive to low-temperature processes such as erosion and sedimentation (Valley 2003; Hawkesworth & Kemp 2006), which raise the δ18O in terrestrial supracrustal materials. For example, sedimentary rocks have δ18O values between 10 and 40‰ relative to VSMOW (Vienna Standard Mean Ocean Water), whereas hydrothermally altered oceanic crust has δ18O values between 10 and 20‰ (Eiler 2001). This means that zircons that...
crystallized from a mixed magma source of mantle-derived and sedimentary components have higher δ18O values than the canonical mantle zircon δ18O value of 5.3 ± 0.3‰. Thus, the combination of oxygen isotope data and Hf model ages derived from zircon analyses can provide important constraints on crustal origins (i.e. juvenile crustal extraction from the mantle versus recycling of pre-existing continental crust).

This paper reports new U–Pb, Lu–Hf, and O isotope data for detrital zircons separated from six samples. The goal is to use the Ordovician sediments of the South Mayo Trough as a window to gauge their provenance and major global crustal growth periods.

Materials and methods

Study area and sampling

The South Mayo Trough in western Ireland (Fig. 1) is a broadly simple syncline of Ordovician strata lying between strips of Silurian strata that conceal the contacts between Ordovician and Dalradian rocks. The southern limb of the syncline preserves a thinner sequence of Lower Ordovician arc, proximal forearc, and successor basin strata, whereas the northern limb consists of a thicker, more steeply dipping southward-younging, distal forearc sequence (Fig. 1). Between the Dalradian of North Mayo and the northern strip of Silurian rocks, the Clew Bay Complex is an assemblage of sediments, ophiolitic mélanges and blocks, and polyphase-deformed turbidites and slates probably developed as a subduction–accretion complex to the north-facing Ordovician arc (Dewey & Shackleton 1984; Dewey & Ryan 1990; Dewey & Mange 1999). Greywackes of the Clew Bay Complex are characterized by detrital, deep purple zircons, dated to indicate a Laurentian Grenville Province source (Mange et al. 2010). In contrast to all fossiliferous and unambiguous Silurian strata in western Ireland (Harkin et al. 1996), metamorphic index minerals are absent from the Killadangan Formation (Mange et al. 2003), which is a part of the Clew Bay Complex (Fig. 1). Its low heavy mineral diversity and high purple zircon abundance suggest that the Killadangan Formation is the oldest assemblage in the South Mayo Trough (Mange et al. 2003). In the Ordovician upper Derrylea Formation, Dalradian metamorphic index minerals appear, marking the progressive erosional unroofing of both the obducted ophiolite and its subjacent Dalradian metamorphic complex. Based on the above geological context, two samples, one from the Killadangan complex (WPG90/4) and the other from the upper Derrylea Formation (DL90/7), were selected (see Fig. 1 for sample location). For our crustal evolution study, the Killadangan sample probably provides the oldest populations of detrital zircons, whereas the upper Derrylea sample provides the most representative sampling of provenances of northern limb strata (Fig. 1).

The tiny island of Inishturk (Fig. 1) was mapped (Graham et al. 1989) as a continuous southward-younging sequence of Sheeffry Formation turbidites to the north, and Derrylea Formation turbidites to the south, as in the main South Mayo Trough north limb (Fig. 1), with a series of intraformational isoclines. Four samples of the tuffaceous horizons on Inishturk from the north–south traverse of Mange et al. (2003) were selected for further isotopic studies.
One analysis takes c. 4 min consisting of pre-sputtering (c. 120 s), automatic beam centring (c. 60 s) and integration of oxygen isotopes (20 cycles × 4 s, total 80 s). Uncertainties on single analyses are usually better than 0.2−0.3‰ (1σ).

The instrumental mass fractionation factor (IMF) was corrected using the R33 zircon standard following the procedures of Li et al. (2009, 2010a,b). Measured 18O/16O ratios were normalized to VSMOW composition with 18O/16O=0.0020052, and then corrected for the IMF as follows:

\[
\delta^{18}\text{O}_M = \left[ \frac{18\text{O}}{16\text{O}} \right]_M - 1 \times 1000(\%o) \\
IMF = \delta^{18}\text{O}_{(\text{standard})} - \delta^{18}\text{O}_{(\text{standard})} \\
\delta^{18}\text{O}_{(\text{sample})} = \delta^{18}\text{O}_M - IMF
\]

where \(\delta^{18}\text{O}_{(\text{standard})}\) is a per mil deviation of the measured 18O/16O ratio for R33 zircon standard relative to VSMOW using equation (1), \(\delta^{18}\text{O}_{(\text{sample})}\), is a nominal value for R33 reported relative to VSMOW, which is 5.55±0.04‰ (Valley 2003; Black et al. 2004), and \(\delta^{18}\text{O}_{(\text{sample})}\) is the IMF corrected value for unknown zircons, which is our final reported value for the samples.

The external reproducibility of 18O/16O is better than 0.4‰ (2SD). The average δ18O value of 5.50±0.40‰ (2SD) for R33 is within error of the nominal value of 5.55±0.34‰ (2SD) reported by Valley (2003), Black et al. (2004), and Kita et al. (2009).

U–Pb dating. U–Pb isotopes were measured in situ using a 193 nm excimer laser (Photon Machines Analyte 193H) in conjunction with a Thermo Element Xr high-resolution single-collector ICP-MS system at UC Davis. The Analyte 193H is equipped with a dual-volume Helex Cell, which negates any fractionation associated with differential flow in traditional laser cells and decreases the signal response time. The Element XR was initially tuned in solution mode, with typical intensities in low-resolution mode of c. 2.5 × 10^6 c.p.s. for a 1 ppb solution of uranium. After connecting the laser, the laser gas flows were then adjusted for maximum sensitivity and stability. Typical laser parameters involved a 30−40 μm spot size, with a repetition rate of 5 Hz, laser energy of 5 mJ and fluence of 1.2 J cm−2. Each analysis measured 204Pb, 206Pb, 207Pb, 208Pb, 232Th and 239U with a total analysis time for one sample of 75 s. The first 20−25 s of each analysis was an on-peak gas blank, which was later subtracted during data reduction, leaving typical analysis times of 50−55 s. All measurements were performed at low resolution and exclusively in counting mode to prevent any added uncertainty associated with the cross-calibration of counting and analogue modes on the Element XR. Data reduction was performed off-line using the lolite data reduction package, of which more details have been given by Paton et al. (2010). We report 206Pb/239U ages for zircons younger than 1 Ga, and 206Pb/207Pb ages for zircons older than 1 Ga.

During each analysis period, two zircon standards were also measured to account for fractionation associated with the ablation, instrumental mass bias, and to counter any instrumental drift during an analytical session. We used 91500 as a calibration standard, and assessed the accuracy of our results by repeat analyses of the R33 zircon standard. A total of 18 analyses of R33 gave an average age of 417±10 Ma (2SD). This is within error of the standard age of 419.26±0.39 Ma (Black et al. 2004) determined by isotope dilution thermal ionization mass spectrometry (ID-TIMS). Further details of analytical procedures have been given by Tollstrup et al. (2012).
Hf isotopes. Hafnium isotopes were also measured using the Analyte 193H laser ablation system in conjunction with a Thermo Neptune Plus multi-collector ICP-MS system at UC Davis. The Neptune Plus was initially tuned in solution mode, in low resolution, prior to attaching the laser. Typically, a 20 ppb solution of JMC-475 gave an intensity of 8 V of $^{180}$Hf using an X-skimmer cone, Jet-sampling cone and an Apex desolvating nebulizer. After optimizing the signal on $^{180}$Hf, the laser was connected and laser gas flows were adjusted for maximum sensitivity and stability. Typical spot sizes for zircon analyses were between 50 and 60 μm, and other laser parameters were similar to those used for U–Pb measurements. Intensities were measured simultaneously on Faraday cups with 1011 Ω resistors. The Neptune's cup configuration was set up with $^{177}$Hf in the centre cup, $^{172}$Yb, $^{175}$Lu and $^{176}$Hf measured in the low mass cups, and $^{178}$Hf, $^{179}$Hf and $^{180}$Hf measured in the high mass cups. Each zircon analysis consisted of 200 measurements with an integration time of 0.25 s. On-peak baselines were measured at the start of each analytical session and were automatically subtracted from the measured data by the Neptune software. Isotope ratios were corrected for instrumental mass bias by applying an exponential correction assuming a constant value for $^{179}$Hf/$^{177}$Hf of 0.7325. Interferences of $^{176}$Yb and $^{176}$Lu on $^{176}$Hf were corrected offline assuming exponential mass bias and using $^{172}$Yb/$^{173}$Yb = 1.35272, $^{176}$Yb/$^{172}$Yb = 0.588673, and $^{176}$Lu/$^{172}$Lu = 0.02655 (Vervoort et al. 2004).

Replicates of zircon standards 91500, GJ-1 and Plešovice were measured throughout each analytical session to monitor quality. Analyses of 91500 were performed before and after a series of five unknowns. A total of 43 analyses yielded an average $^{176}$Hf/$^{177}$Hf of 0.282316 ± 33 (2SD), within error of previously published Hf isotope data measured in solution mode (e.g. Woodhead & Hergt 2005). For GJ-1 and Plešovice, we obtained $^{176}$Hf/$^{177}$Hf = 0.282032 ± 26 (2SD, $n$ = 9) and 0.282507 ± 17 (2SD, $n$ = 7), respectively. Further technical descriptions have been given by Tollstrup et al. (2012).

**Results**

U–Pb ages

All U–Pb ages are summarized in Figure 4. Representative CL images for zircons from each sample population are presented in
Figure 3, showing sampling position, typical zircon size and any zoning. Zircons in the Killadangan complex (WPG90/4) are all rounded, deep purple or purple–brown with a complex internal structure commonly encasing an earlier-formed core.

The Upper Derrylea Formation (DL90/7) contains a more varied population, from which the colourless to very pale pink, rounded or subrounded varieties, and those with a distinctive overgrowth or re-faceted grains were selected. Many of these zircons show a complex, multistage internal structure.

The tuff horizons on Inishturk (IT-5, -12, -17, -18) encase euhedral, doubly faceted zircons with oscillatory growth zoning, whereas the turbidite beds contain mostly rounded colourless, pale pink and a few deep purple zircons with a complex internal structure or with overgrowth rims.

A composite summary Tera–Wasserburg diagram for all the zircons investigated in this study and the histogram of all U–Pb ages obtained are given in Figures 4 and 5 respectively. Major U–Pb peaks correspond to Grampian synorogenic igneous zircons (450–498 Ma), minor Cambrian peak (500–526 Ma), Grenvillian peak (980–1250 Ma), and Lewisian basement formation (2587–2920 Ma), consistent with the interpretations of Mange et al. (2010). Age groups between 1650 and 2355 Ma most probably reflect Laxfordian-aged events (see Goodenough et al. 2010) affecting the Lewisian gneisses overlapping with the lower age limit of what is defined here. Our dataset from the South Mayo Trough captures three of the major global supercontinent forming orogenic events; Gondwana (650–400 Ma), Rodinia (1.25–0.95 Ga), and Superia/Scavia (2.75–2.6 Ga; Campbell & Allen 2008; Wang et al. 2009, 2011; Hawkesworth et al. 2010; Iizuka et al. 2010). Probably because of limited sampling, the Nuna peak (1.9–1.6 Ga) is less pronounced in our study (Fig. 5).

**Oxygen isotopes**

Oxygen isotopic compositions of detrital zircons are plotted against U–Pb ages in Figure 6a and Hf-model ages in Figure 6b. Of the 160 zircon grains examined, there are no zircons with δ18O >10‰. Three zircons, all from the Killadangan complex, give δ18O values that are lower than that of mantle zircon, and are interpreted to record alteration of their source rocks by interaction with hydrothermal or meteoric water. All the remaining zircons plot close to or above the mantle value, suggesting variable involvement of low-temperature supraclastic materials in their source regions. Roughly half of the zircons investigated show δ18O values between 5.3 and 6.5% (Fig. 6b), suggesting that they crystallized from juvenile magmas with insignificant involvement of supraclastic materials.

There appears to be a stepwise increase in δ18O value from 2.5 Ga (7‰) to 2.0 Ga (10‰) in our dataset (Fig. 7a), possibly suggesting that low-temperature weathering processes were less important in the Archaean (Valley 2003; Kemp et al. 2006; Wang et al. 2009, 2011). However, two zircon grains in our sample set (IT-5-21 and IT-5-23) appear to violate this general trend, with δ18O values of 8.74 ± 0.21‰ and 9.75 ± 0.21‰, respectively.

**Lu–Hf isotopic systematics**

A Hf evolution diagram (ε176Hf v. U–Pb crystallization ages of zircons) is shown in Figure 7. The depleted mantle (DM)
Fig. 6. (a) Oxygen isotope composition of zircons, expressed as δ¹⁸O_VSMMOW (‰), plotted against U–Pb ages of zircons. Light grey bands represent zircon crystallization ages, clearly associated with major tectonic events locally and globally. (b) Zircon δ¹⁸O_VSMMOW data plotted against Hf model ages. Hf model ages for zircons with δ¹⁸O values below 6.5‰ (filled circles) with minimal supracrustal input give the best chance of providing juvenile crustal production from the mantle.

Fig. 7. Initial ε¹⁷⁶Hf plotted against U–Pb ages for all zircons. It should be noted that most zircons plotting close to the DM (grey line with ε¹⁷⁶HfDM = 13.4) have juvenile oxygen signatures (5.3‰ < δ¹⁸O < 6.5‰). Dotted lines are crustal evolution lines with average continental crustal (¹⁷⁶Lu/¹⁷⁷Hf)cc = 0.0115 (Rudnick & Gao 2003).
The significant offset between U–Pb crystallization and Hf model ages should be noted (Figs 5 and 8). This offset, as shown in Figure 8, indicates that crustal generation is not necessarily accompanied by zircon crystallization; significant mixing between crustal source regions (illustrated by dotted diagonal lines in Fig. 7) leads to smearing of Hf model ages. Crustal incubation times range between 100 and 2800 Ma. The overwhelming majority of Lewisian and Grenville zircons have a crustal incubation time between 100 and 1000 Ma. In contrast, the younger Grampian zircons have crustal incubation times that range between 1 and 2 Ga (Fig. 8). Oxygen isotopes can discriminate between crustal recycling and juvenile crustal production from the mantle (Kemp et al. 2006). It is noteworthy that most zircons with crustal incubation times <200 Ma all have 5.3‰ < δ18O < 6.5‰ (Fig. 8).

On the plot of U–Pb age v. ε176Hf (Fig. 7), it should be noted that the overwhelming majority of Lewisian and Grenville zircons have positive ε176Hf values, suggestive of input of source materials from the mantle. However, Grenville zircons also have the most variable oxygen isotopic compositions (Fig. 6a); how could these zircons obtain highly variable supracrustal oxygen input without acquiring or inheriting evolved crustal Hf isotope signatures? It is clear from Figures 7 and 8 that most of the Grenville zircons have a relatively short crustal incubation time. This suggests that the low-temperature alteration processes that affected and generated δ18O variations in the Grenville zircon source region were brief, because change in εHf variation requires a significant time to evolve given the long half-life of 176Lu. Similar observations and conclusions have been drawn in the Canadian Grenville Province and South China (Valley 2003; Wu et al. 2006).

The Grampian zircons have negative εHf and juvenile δ18O values, suggesting reworking of the Grenvillian igneous components without significant crustal contamination (Figs 7 and 8). The generation of some very negative ε176Hf (~30 to ~40) Grenville zircons by reworking of juvenile mantle-derived Lewisian materials must have proceeded with little crustal contamination, yet their crustal incubation time was long, between 1 and 2 Ga (Fig. 8). Our observation suggests that long crustal incubation time does not necessarily imply a long low-temperature alteration for the source region of zircon melts; clearly, the two time scales are not coupled.

A calculated cumulative crustal growth curve based on the sedimentary record of detrital zircons in the South Mayo Trough is shown in Figure 9. Hf model ages are weighted by zircon load (i.e. frequency of zircons falling within a particular age bin normalized to the total number of zircons analysed). Growth started at c. 4 Ga, the age of oldest known continental crust (Bowring & Housh 1995), and reached c. 20% of the present volume by 2.3 Ga, followed by a rapid increase to 60% between 2.3 and 2.1 Ga; the next major growth period was between 2.0 and 1.5 Ga, with effective completion of continental growth by 1.4 Ga. The remaining crustal processes mainly involved reworking and recycling, rather than building new crust. Our results, using the South Mayo Trough as a window to gauge the continental growth, appear to be most consistent with the models of Jacobsen (1988) and Taylor & McLennan (1985) (Fig. 9). Our model implicitly assumes that more continental growth means more crustal melt, and more zircon crystallizes from such melt, and the frequency of zircon populations in a particular age group is proportional to the volume of continental crust with such an age. The South Mayo Trough is a small local window to peep into the continental growth at large, but the basic growth structures captured by larger-scale studies (Campbell & Allen 2008; Pietranik et al. 2008;
Wang et al. 2009, 2011; Iizuka et al. 2010) are also apparent in our dataset. Whether zircon represents a biased record reflecting primarily crustal preservation rather than production (Hawkesworth et al. 2009) is unresolved from our dataset.

Conclusions

The following summarizes our major findings.

(1) U–Pb ages reveal that detritus delivered to the South Mayo Trough is represented by three major clusters of Archaean (Lewisian), Mesoproterozoic (Grenville), and Ordovician (Grampian) ages (Figs 4–8). These age clusters correspond to the ages of igneous and metamorphic complexes in the British Isles, Greenland, Baltica and Laurentia, which are thought to have supplied the sediments to the South Mayo Trough. These data support an earlier study by Mange et al. (2010) based on a smaller dataset (95 zircons).

(2) We applied oxygen isotope and Lu–Hf isotope systematics to the same zircons with known U–Pb ages to decipher the crustal growth history of the South Mayo Trough sediments. We find that the growth of juvenile continental crust started as early as Hadean time (c. 4 Ga), and continued through much of the Palaeoarchaean (3.5 Ga) and Neoarchaean (2.7 Ga), building up c. 20% of crustal volume by 2.3 Ga. A major period of crustal growth is then recorded from 2.3 to 2.1 Ga (c. 60%); the 480 Ma Grampian zircons were formed by recycling of these precursor materials (Fig. 9). The remaining crustal building process occurred from 2.1 to 1.5 Ga. By 1.4 Ga, continental crust formation processes were effectively complete.

(3) All zircons with crustal incubation times of less than 200 Ma have mantle-like δ^{18}O signatures between 5.0 and 6.5‰. Zircons with highly evolved crustal signatures with negative ε^{176}Hf, can contain mantle-like δ^{18}O, whereas zircons with depleted-mantle signatures with positive ε^{176}Hf, can contain highly variable δ^{18}O, indicating significant low-temperature surface processes involved in the source regions in which these zircons crystallized. This suggests that the time scales of crustal incubation and supracrustal alteration processes are not coupled.

Q.Z.Y., J.F.D., and M.A.M. are grateful to the University of California at Davis for facilities and financial support for the work. Q.Z.Y. acknowledges UC Davis Start-up funds and NASA Planetary Major Equipment grants as supplement to NNX08AG57G and NNG05G03G, which allowed the acquisitions of the instruments at UC Davis used in this study. This oxygen isotope work was supported in part by the State Key Laboratory of Lithospheric Evolution, IGG-CAS Grant Z201003. J. Wooden and F. Mazdab at the Stanford SHRIMP facility kindly helped M.A.M. in the summer of 2007 with the zircon mounting and SEM-CL imaging. We thank the two expert journal referees and the Chief Editor, G. Crowley, for their comprehensive and constructive reviews and efficient editorial handling, which have substantially improved the quality of the paper and its presentation.

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