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LITHOLUND THESIS 24

# Evolution of continental crust in the Proterozoic

growth and reworking in orogenic systems

Andreas Petersson



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DOCTORAL DISSERTATION

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KLIMATKOMPENSERAT  
PAPPER



# Abstract

To understand the growth of continental crust, the balance between juvenile mantle derived extraction, infracrustal reworking and crustal recycling, needs to be estimated. Since the beginning of the century, the use of coupled in situ zircon U–Pb, Lu–Hf and O isotope analyses as a tool to address these questions have increased exponentially. Numerous compilations of ever growing datasets have been presented, leading to new, and sometimes contrasting models of continental growth. Many of these models, however, suffer from a number of assumptions, including a mantle reservoir that has been homogeneously and linearly depleted since the Hadean. Further, the use of (mainly) detrital zircon, taken out of their geological context, and the application of their depleted model-ages clearly hamper the validity of these models.

To accurately address the question regarding continental crustal growth using combined zircon U–Pb–Lu–Hf–(O) isotope data, one needs to have contextual control and minimise the uncertainties of the applied models.

In papers included in this thesis such an approach has been used on three different Palaeo- to Meso-Proterozoic orogenic belts; in Fennoscandia, in North American Grenville and in the Birimian terrane of the West African craton.

The eastern part of the Sveconorwegian Province, located in the southwestern part of the Fennoscandian Shield, is made up of granitoid rocks that were emplaced through sequential tapping of a reservoir that formed through mixing between a 2.1–1.9 Ga juvenile component and Archaean crust. Between 1.7 and 1.4 Ga the continental crust of the Eastern Segment was reworked with little or no generation of new crust.

Further to the west, in the Idefjorden terrane of the Sveconorwegian Province, 1.65 to 1.33 Ga rocks have isotopic signatures that indicate reworking of older continental crust, including sediments. However, overall the isotopic signatures in the Idefjorden terrane indicate an increase in juvenile material with time, consistent with development of an extensional back-arc rift geotectonic setting, accommodating deposition of the local metasedimentary basin, Stora Le-Marstrand.

Isotope data from rocks within the Grenville orogen in subsurface Ohio suggest a common c. 1.65 Ga juvenile source to a majority of the sampled bedrock. Emplacement of this juvenile crustal contribution was followed by sequential reworking of that reservoir with little or no additional contribution to the source.

The c. 2.31–2.06 Ga Birimian terrane in Ghana, West African craton, is a commonly cited example of plume initiated crustal growth, that is known to have largely juvenile signatures. However, we can show that reworked Archaean crust contribute in a much larger extent than previously known, once again highlighting the importance of infracrustal reworking during emplacement of continental crust. Further, the emplacement of felsic rocks during the Eoeburnean pre-dates suggested plume related rocks, contradicting a suggested plume initiated crustal growth.

Collectively, these studies highlight the importance of infracrustal reworking in Palaeo- to Meso-Proterozoic accretionary orogens. These studies also provide good examples of combined zircon U–Pb–Lu–Hf–(O) isotope analyses on rocks and rock suites with known affinity where the validity of chosen models can be justified.

TO MY FAMILY  
LIV, OLOF & MÄRTA

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## List of papers

This thesis is based on the four papers listed below, which have been appended to the thesis. Paper I is reprinted under permission of the Lyell Collection. Paper II and III are reprinted under permission of Elsevier Limited. Paper IV has been submitted to the journal for consideration.

Paper I: Petersson, A., Scherstén, A., Andersson, J., Möller, C., 2013. Zircon U–Pb and Hf – isotopes from the eastern part of the Sveconorwegian Orogen, SW Sweden: implications for the growth of Fennoscandia, in Roberts, N. M. W., Van Kranendonk, M., Parman, S., Shirey, S. and Clift, P. D., eds., *Continent Formation Through Time*. London, Geological Society [London] Special Publication 389, 281–303. <http://dx.doi.org/10.1144/SP389.2>

Paper II: Petersson, A., Scherstén, A., Bernard, B., Gerdes, A., Whitehouse, M.J., 2015a. Mesoproterozoic continental growth: U–Pb–Hf–O zircon record in the Idefjorden terrane, Sveconorwegian orogen. *Precambrian Research* 261, 75–95. <http://dx.doi.org/10.1016/j.precamres.2015.02.006>

Paper III: Petersson, A., Scherstén, A., Andersson, J., Whitehouse, M.J., Baranoski, M.T., 2015b. Zircon U–Pb, Hf and O isotope constraints on growth versus reworking of continental crust in the subsurface Grenville orogen, Ohio, USA. *Precambrian Research* 265, 313–327. <http://dx.doi.org/10.1016/j.precamres.2015.02.016>

Paper IV: Petersson, A., Scherstén, A., Kristinsdóttir, B., Kemp, A. I. S., Kalvig, P., Solomon Anum, S., 2015c. Zircon U–Pb–Hf evidence for reworking of Archaean crust within the Birimian Palaeoproterozoic source rocks of the West African Craton, SE Ghana, Submitted to *Precambrian Research*, Manuscript.





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Most mornings when I got to work, the day started of with a cup of coffee and a few minutes of chat with my principal supervisor and friend Anders Scherstén. These chats did not only cover geology, but anything and everything. Our morning conversations, have at large formed the basis of this thesis. This is where new, often wild and not seldom crazy, but sometimes fruitful ideas have been put forward and discussed. I would like to acknowledge the importance of these morning chats and take the opportunity to humbly thank Anders for ALWAYS (not only during morning coffee) keeping his door open, and taking his time to explain, listen, discuss and guide. The guidance I have received reaches far outside the field of geology and the scope of Anders obligations. I could not have asked for a better supervisor.

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During my first days of geology studies in 2005, Ulf Söderlund was the one who made me realise that hard-rock geology is a fascinating field. I long thought that if I ever did a Ph.D, he would be my main supervisor. Things turned out differently, but throughout my years at the department Ulf has always acted as a mentor to me. Ulf's enthusiasm, encouragements and easy going attitude towards his work has inspired me many times, and without Ulf at the department I question if I would ever have ended up where I am today. For this, I am forever grateful.

Those first days of geology studies in 2005 is also when I first met Mimmi Nilsson. She has since been my "partner in crime" at the department. Mimmi has been an invaluable asset throughout my studies, both as a fellow student, as a co-worker, but mostly as a friend. Nobody can as mercilessly as Mimmi, simultaneously refute one of my crazy hypothesis and insult both me and my family (without causing any hard feelings).

Mimmi, I became doctor before you!

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Love you guys.



# Abbreviations

|        |  |
|--------|--|
| A-type | Anorogenic-type  |
| CHUR   | Chondritic Uniform Reservoir                                       |
| cPb    | Common Pb, non radiogenic Pb incorporated into the crystal lattice |
| I-type | Igenous-type   |
| IUGS   | The International Union of Geological Sciences                     |
| MASH   | Melting, Assimilation, Storage and Homogenization                  |
| MORB   | Mid Ocean Ridge Basalt   |
| REE    | Rare Earth Elements  |
| S-type | Sedimentary-type   |
| SIMS   | Secondary Ion Mass Spectrometer                                    |
| SMOW   | Standard Mean Ocean Water  |



# 1. Introduction

This thesis is based on three published papers and one manuscript, which are re/pre-printed in appendices I-IV. Included papers and manuscript are case-studies of Proterozoic terranes from different parts of the globe where the lowest common denominator is growth of the continental crust as revealed by combined zircon U–Pb and Lu–Hf or U–Pb, Lu–Hf, O isotopes systematics in the mineral zircon. To test whether these Proterozoic granites and gneisses formed at the time of crystallisation or if they were generated through reworking of older crust, is one of the main aims of this study.

The research was funded through grants from Fysiografen and Per Westlings Minnesfond to Andreas Petersson, and grants from the Swedish Research Council to Anders Scherstén, which are all gratefully acknowledged.

## 2. Evolution of continental crust

### 2.1 On the growth of continents

It is irrefutable that the continents have grown from non-existence to its current mass, but how and when this happened is a matter of debate and fundamentally important for our understanding of planet Earth. A central theme in this research is to what extent the uniformitarian paradigm can be applied back in time, i.e. have current processes been operating through Earth's history in the same way and at the same rates as today?

For example, the existence of modern style plate tectonics during the first half of the Earth's evolution (Hadean–Archaean) is still a matter of debate, (for review see Furnes et al. 2013; Kusky et al. 2013). Wilson-cycle plate tectonics have been suggested for the generation of Archaean crust, as far back as 3.2 Ga (e.g. Heubeck and Lowe, 1994 and Van Kranendonk et al., 2010) or perhaps even further.

Early attempts to estimate the cumulative growth of the continental crust through time are based on a number of different approaches. These include e.g. isotope geochemistry (primarily U–Pb, Rb–Sr, Sm–Nd and K–Ar); radiometric age datings; isotopic and chemical signatures of sediments and shales and crustal addition and subtraction rates (e.g. Hurley and Rand 1969; Veizer 1976; Fyfe 1978; Brown 1979; DePaolo 1980; Armstrong and Harmon 1981; Allégre and Rousseau 1984). Their estimates

for the Proterozoic range from positive (net growth) to steady state (neither net growth or loss) to negative (net loss).

Radiometric ages of igneous rocks (or minerals such as zircon) show an uneven distribution with peaks and troughs through time (Fig. 1). This pattern can be interpreted in different ways. Either, the peaks represent periods of high rates of crustal growth (i.e. mantle derived melts) and troughs periods of crustal growth quiescence (e.g. Condie, 1998). This implies a non-uniformitarian Earth with respect to rates or processes. Alternatively, the age peaks and troughs are an effect of preservation or sampling bias. Interestingly, the age peaks correlate with supercontinent assembly (Fig. 1; e.g. Condie, 1998; Campbell and Allen, 2008; Parman, 2015). Either supercontinent assembly is associated with greater growth rates (e.g. Condie, 1998) or higher preservation potential (Hawkesworth et al., 2009).

If the age peaks reflect higher rates of juvenile crust formation, then this should be associated with mantle-like isotope signatures, while preservation bias might be associated with both juvenile and ancient isotope signatures. These questions can be addressed on both global (e.g. Reymer and Schubert, 1984; Campbell, 2003; Dhuime et al., 2012; Belousova et al., 2010) and regional scale (e.g. this thesis).

Since at least the Archaean, convergent plate margins play a central role in the growth of continental crust (e.g. Kamber et al., 2003; Martin et al., 2005). Understanding the balance between juvenile mantle derived magmatism, reworked and recycled crust and loss of continental crust into the mantle (c.f. Mišković and Schaltegger, 2009; Belousova et al., 2010; Hawkesworth et al., 2010; Dhuime et al., 2012) is important to evaluate and understand the rate of crust generation in volcanic arc settings (Armstrong, 1971; Plank and Langmuir, 1993; Mišković and Schaltegger, 2009, Fig. 2).

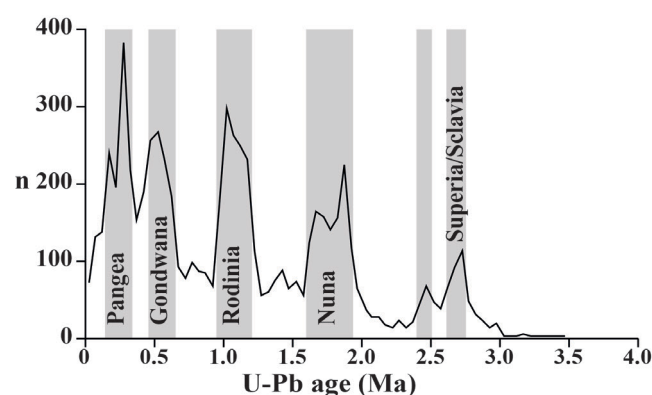
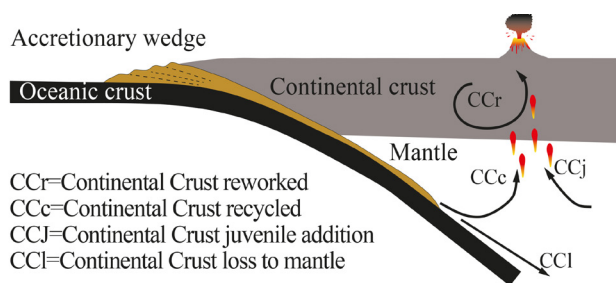


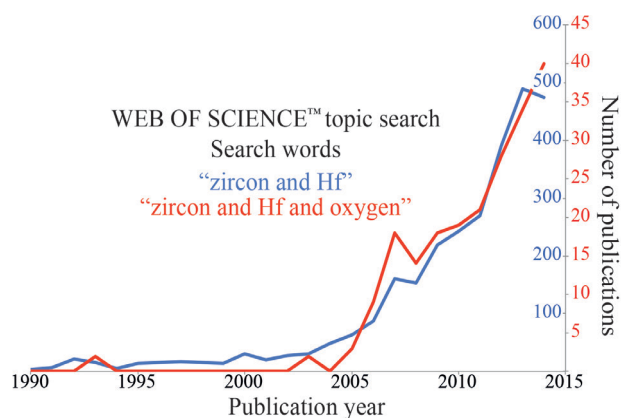
Figure 1. Density plot of U–Pb ages of mainly concordant detrital zircon (Black line). Time of supercontinent assemblies are superimposed as grey fields. Redrawn from Campbell and Allen, (2008).

Since the beginning of this century, coupled U–Pb, Lu–Hf and U–Pb, Lu–Hf and O isotope studies on detrital zircon and zircon from granite intrusions have proven a powerful tool, which is reflected in the dramatic increase in publications since 2004 (Fig. 3). Ten years down the road, “zirconology” (i.e. combined zircon U–Pb–Hf–O) has become a major data archive in the puzzle to solve crustal growth issues as it allows for discrimination between juvenile (high  $^{176}\text{Hf}/^{177}\text{Hf}$  and  $\delta^{18}\text{O}$  c. 5.3‰) and reworked (low  $^{176}\text{Hf}/^{177}\text{Hf}$  and  $\delta^{18}\text{O}$  >5.3‰) crust (e.g. Hawkesworth and Kemp, 2006).

Recycling continental crust back through the mantle by subduction processes will decrease the proportion of radiogenic Hf in subsequent suprasubduction magmas, giving them more evolved signatures (Fig. 2; e.g. Hawkesworth et al., 1979; Griffin et al., 2000; Kemp et al., 2006; Hawkesworth et al., 2010). Likewise, melting, assimilation, storage and homogenisation (MASH) or

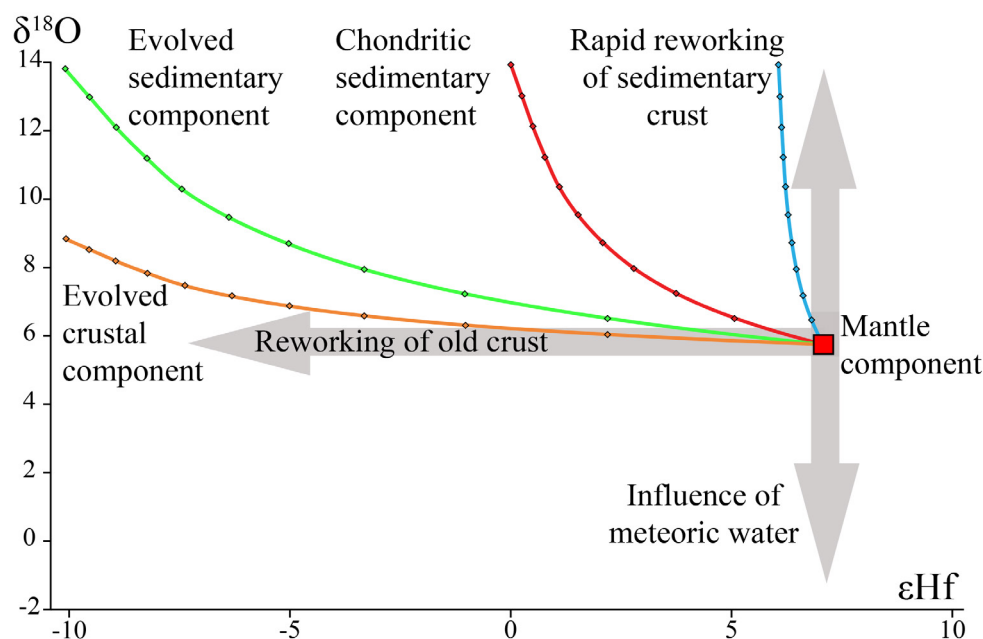


**Figure 2.** Schematic conceptual model illustrating different processes connected to growth and decrease of the continental crust.



**Figure 3.** Number of publications found on Web of Science per year including the topic search words “zircon and Hf” and “zircon and Hf and oxygen” respectively, showing a vast increase in publications from the beginning of the 21st century.

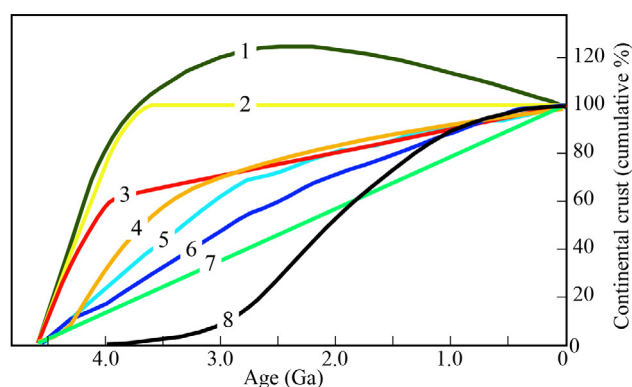
similar processes within the lower crust above a subduction zone will also yield more evolved Hf isotope signatures if the crust in the upper plate is significantly older than the mantle derived magmas. Similarly, as different geological reservoirs have a wide span in  $\delta^{18}\text{O}$  whereas the mantle has a narrow  $\delta^{18}\text{O}$  value of  $5.3 \pm 0.3$ ‰, reworking of sediments or altered oceanic crust into magma will affect its  $\delta^{18}\text{O}$  signature (Valley et al., 2005). As non-metamict zircon generally preserves its  $\delta^{18}\text{O}$  from crystallisation, the O-isotope composition of zircon is an ideal tracer of crustal contamination processes (Valley et al., 2005). Combined zircon O–Hf isotope signatures can therefore provide information on the nature and age structure of the processes outlined above (Fig. 4).



**Figure 4.** Schematic mixing model describing mixing between a juvenile component (red square;  $\epsilon\text{Hf}=7.0$  and  $\delta^{18}\text{O}=5.7$ ‰), and altering evolved; chondritic and juvenile, igneous and sedimentary components. A  $\delta^{18}\text{O}$  of 5.7‰ and 14‰ represent crust with mantle-like  $\delta^{18}\text{O}$  and the average values of sediments respectively. Ticks represent 10% mixing increments.

## 2.2 Proterozoic – growth and reworking in orogenic systems

How did we get from 0% continental crust to the 100% we have today? Numerous models have been published the last decades proposing vastly varying results (Fig. 5). However, relatively fast continental growth during the Archaean is advocated by an increasing number of publications (e.g. Fyfe, 1978; Armstrong and Harmon, 1981; Reymer and Schubert, 1984; Campbell, 2003; Dhuime et al., 2012). These models normally have an inflection point between 4.0 Ga and 2.5 Ga where growth rate decreases during the Proterozoic Eon (Fig. 5). This decrease in crustal growth might in part reflect an increase in infracrustal reworking as a result of an onset of modern style plate tectonics. How well do these results correlate to regional studies of Proterozoic orogenic systems? This question permeates this thesis and the studies within. Shedding some new light to whether infracrustal reworking or juvenile contribution is the dominant process during the Proterozoic is the main aim of this thesis.



**Figure 5.** Growth curves for the continental crust as proposed by several different models, with 100% representing the present-day cumulative volume of crust. Key: 1. Fyfe (1978), 2. Armstrong & Harmon (1981), 3. Reymer and Schubert (1984), 4. Campbell (2003), 5. Dhuime et al. (2012), 6. Belousova et al. (2010), 7. Linear growth, 8. Allège and Rousseau (1984).

In this thesis three different orogenic systems are studied; an accretionary arc system in the Fennoscandian Shield (Paper I and II); a continent-continent collisional orogeny in subsurface North American Grenville (Paper III); and the Birimian terrane of the West African craton, sometimes considered to be an example of plume initiated crustal growth (Paper IV).

A large number of post Archaean terranes have been interpreted as juvenile, including e.g. southwestern Fennoscandia and the Birimian terrane of the West African craton (e.g. Abouchami et al., 1990; Boher et al., 1992; Brewer et al. 1998; Åhäll and Connelly, 2008; Tapsoba et al., 2013), which contradicts reworking dominated systems. This apparent contradiction is discussed in paper I, II and

IV. In this thesis the growth evolution of these three different Proterozoic shield areas will be discussed individually based upon their combined zircon U–Pb–Lu–Hf or U–Pb–Lu–Hf–O isotope record in granitoids. Collectively these papers shed new light into questions regarding growth of the continental crust in these regions, showing the importance of both infracrustal reworking and juvenile mantle derived contribution during the Palaeo- to Meso-Proterozoic.

## 2.3 Crust formation in a Palaeo- to Meso-Proterozoic accretionary orogeny

Papers, I and II focus on the southwestern parts of the Fennoscandian Shield, where paper I is aimed at the Eastern Segment and paper II at the Idefjorden terrane (Fig. 1 in Paper I and II). The two crustal segments are separated by a several km wide shear zone interpreted as a mid-crustal ramp that accommodated relative eastward-directed transport of the Idefjorden terrane onto the Eastern Segment (e.g. Viola et al., 2011).

The current view of crustal growth in southwestern Fennoscandia involves c. 330 million years of semi-continuous, juvenile subduction-related magmatism, including episodic continental crust formation in nine pulses between 1.85 Ga and 1.55 Ga, all tied to a persevering convergent margin system (Brewer et al., 1998; Åhäll and Connelly, 2008). The testing of this model using coupled zircon U–Pb–Lu–Hf–O isotopes led to paper I and II.

## 2.4 Crustal growth in a Mid-Proterozoic continent-continent collisional orogeny

The Grenville Province in North America is one of the most studied Precambrian orogens on Earth. It is, however, poorly known in its entirety since it only surfaces in limited areas south of Canada. Current knowledge about the orogen is largely based on data from the Canadian portion, and our understanding of the vast unexposed subsurface Grenville Province is based on geophysical surveys and a few basement penetrating deep drill cores (c.f. Rivers, 2012). Recent work on the evolution of Grenville aged rocks of the mid-continent have focused on zircon U–Pb and Sm–Nd analyses on the few available exposed outcrops (i.e. Daly and McLelland, 1991; McLelland et al., 1993; Van Schmus et al., 1996; Rohs and Van Schmus, 2007; Fisher et al., 2010). In order to improve our understanding of the evolution of this region, paper III present U–Pb, O and Hf isotope data from zircon in drill-core samples from the subsurface basement of Ohio.



## 2.5 A classic example of assumed plume related continental crustal growth

The crustal growth evolution of the Birimian terrane in West Africa overlaps with a time period otherwise related to global magmatic quiescence (2.4–2.2 Ga; Condie et al., 2009). Crystallisation ages from the Birimian bedrock of Ghana range between c. 2.31 and 2.06 Ga, with a predominance of ages between 2.21 and 2.06 Ga (e.g. Gasquet et al., 2003; de Kock et al., 2011).

The Birimian crust is a commonly cited example (e.g. Arndt, 2013) of plume-related crustal growth. Boher et al. (1992) propose a model where the Birimian crust initially formed an oceanic plateau around which subduction zones subsequently reworked the oceanic plateau before it was accreted to the Archaean nucleus of the Man Shield.

The formation of the Birimian crust is an example of rapid crustal growth, as large volumes of juvenile continental material was emplaced during a short time-span (Abouchami et al., 1990). The Birimian terrane is however poorly investigated using methods that discriminate between juvenile and reworked crust, and indication of a reworked ancient crustal component is known from the Winneba pluton in southeastern Ghana where Nd-model ages of c. 2.6 Ga indicate the involvement of an Archaean source (Leube et al., 1990).

The apparent lack of data, combined with the indication of a possibly less juvenile origin of the Birimian terrane in Ghana is treated in paper IV.

## 3. Methods

### 3.1 Zircon

Zircon (Fig. 6) has the chemical composition of  $\text{ZrSiO}_4$  and is a tetragonal orthosilicate with  $\text{SiO}_4$  tetrahedra sharing edges with intervening  $\text{ZrO}_8$  dodecahedra. Zircon is common in most igneous rocks of intermediate to quartz-saturated composition. Had it not been for trace elements such as Hf, U and Th (+REE and Ti), which substitute for  $\text{Zr}^{4+}$  or  $\text{Si}^{4+}$ , zircon would not be as powerful geochemical tool as it is. It is the impurities and substitutions that occur in zircon that makes it so useful. Zircon commonly contain trace amounts of P, Y, U, Th and lanthanides. Among these,  $\text{U}^{4+}$  and  $\text{Th}^{4+}$  are radioactive and decay to isotopes of Pb. The benefit here is that Pb is not incorporated into zircon as it forms divalent ions in most magmatic systems, rendering its ionic radius and

charge unfit for the zircon structure. Thus, zircon U–Th–Pb forms a perfect hourglass geochronometer where the amount of Pb in the zircon is directly proportional with the time since zircon crystallisation. Furthermore, zircon is stable up to 1690 °C (1963 K) at atmospheric pressure where the U–Pb clock will remain undisturbed up to temperatures of around 1000 °C (Finch and Hanchar, 2003). Another important property of zircon is its low diffusivity of anions (Valley, 2003). This allows zircon to preserve its chemical signatures of crystallisation (Cherniak and Watson, 2003). Together with the fact that zircon is hard and refractory makes it uniquely robust. So robust that it can withstand its host rock being thoroughly metamorphosed, molten and/or mechanically weathered and still preserves its isotopic information (Scherer et al., 2007).

### 3.2 Zircon U–Pb system

Pb in zircon is produced from the radioactive isotopes  $^{238}\text{U}$ ,  $^{235}\text{U}$  and  $^{232}\text{Th}$  that with time all decay through a chain of alpha and beta emissions into stable Pb isotopes.  $^{238}\text{U}$  decays into  $^{206}\text{Pb}$ ,  $^{235}\text{U}$  into  $^{207}\text{Pb}$  and  $^{232}\text{Th}$  into  $^{208}\text{Pb}$ . All three radioactive parent isotopes have a predetermined half-life (i.e. time required to decay the radioactive parent into half of the initial amount) fixed by the IUGS Sub-commission on Geochronology. Since Pb is not readily incorporated into zircon and the decay rate of the radioactive U and Th-parents are known, U–Th–Pb isotope analyses of zircon can be used to calculate the time passed since crystallisation by recording the proportions between the parent-daughter isotopes. The amount of initial common Pb (cPb, non radiogenic Pb incorporated into the crystal lattice) in a crystalline newly formed zircon is normally negligible. Nevertheless, initial-Pb can be incorporated in zircon or occur in inclusions or

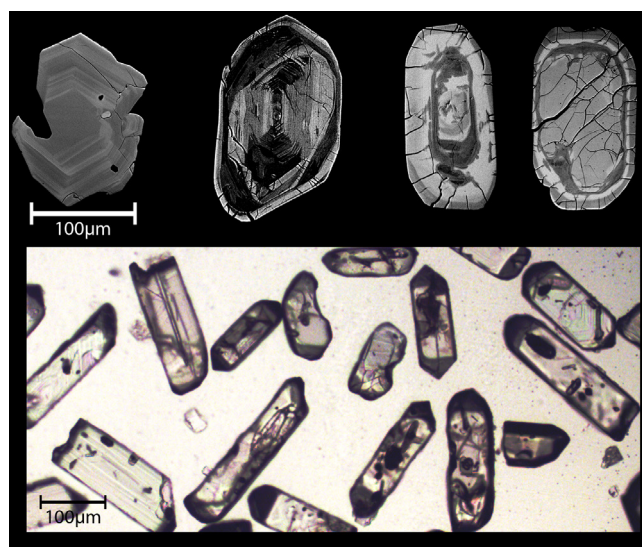


Figure 6. Backscattered Electron Images (BSE; above) and optical microscope images (below) of different varieties of zircon grains.

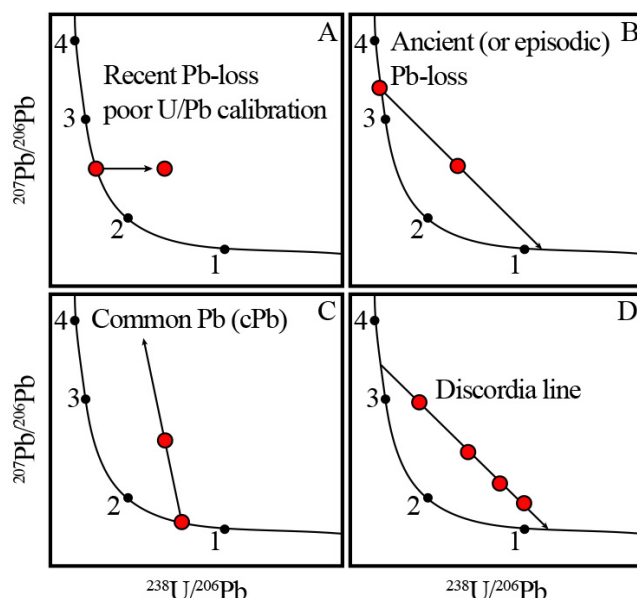
introduced and hosted in cracks or radiation damaged areas. Common Pb contains different proportions of  $^{204}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$  and  $^{208}\text{Pb}$  where only  $^{204}\text{Pb}$  is non-radiogenic and unique to the common Pb component. For this reason, only  $^{204}\text{Pb}$  can be used to monitor the presence of cPb in the zircon. If present, the cPb-contribution to  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$  and  $^{208}\text{Pb}$  has to be subtracted, which can be done using an assumed age of the cPb and a growth model of that Pb (e.g. Stacey and Kramers, 1975). If the common Pb has been corrected for or is absent, the age of a zircon crystal can be calculated using the following equations (1–2):

$$[\text{Eq. 1}] \quad ^{206}\text{Pb}/^{238}\text{U} = e^{\lambda^{238}\text{t}} - 1$$

$$[\text{Eq. 2}] \quad ^{207}\text{Pb}/^{235}\text{U} = e^{\lambda^{235}\text{t}} - 1$$

As the uranium decay constants ( $\lambda^{238}\text{U}$  and  $\lambda^{235}\text{U}$ ) are known while  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{207}\text{Pb}/^{235}\text{U}$  in a sample (e.g. zircon) are measured using a mass spectrometer, each equation can be solved for  $t$ . If  $t_{206/238}$  and  $t_{207/235}$  are identical the analysis is said to be concordant, while if  $t_{206/238} \neq t_{207/235}$  the analysis is discordant. Plotting an infinite number of concordant values generates a concordia curve. True concordance requires a closed system with respect to U, Th, Pb and all intermediate daughters since zircon crystallisation.

Discordance might have several causes, but is usually attributed to Pb-loss, or incorporation of common-Pb (cPb). Recent Pb-loss is usually assumed to be due to Pb-loss in a more or less metamict (non-crystalline) non-annealing crystal lattice at cool surface conditions. In an inverse Tera-Wasserburg diagram (used in this thesis), this is seen as horizontal displacement of the zircon to the right of the concordia curve (Fig. 7A). Alternatively, apparent recent Pb-loss or Pb-enrichment/U-loss might be caused by incorrect U/Pb calibration, which will result in horizontal data displacement towards the right and left respectively. Notably, for any recent Pb-loss, the  $^{207}\text{Pb}/^{206}\text{Pb}$  date is unaffected and will represent a minimum age. Ancient Pb-loss (Fig. 7B) might occur due to e.g. a metamorphic event or other processes that caused Pb-loss. For incomplete processes, partial Pb-loss will be recorded as a point located in between the initial crystallisation age and the Pb-loss event. Importantly, such data cannot be distinguished from recent Pb-loss in the case of a single data point. This example highlights that discordant  $^{207}\text{Pb}/^{206}\text{Pb}$  dates are minimum ages. Common Pb has high  $^{207}\text{Pb}/^{206}\text{Pb} \approx 1$ , which will displace the data point along a steep path towards high  $^{207}\text{Pb}/^{206}\text{Pb}$  dates (Fig. 7C). If different zircon fractions lose different amounts of Pb in a single episodic Pb-loss event, they will plot on a straight line with the upper intercept defining the time of crystallisation and the lower intercept defining the time of the Pb-loss event. This line is called a discordia (Fig. 7D). For a discordia to be meaningful, the U/Pb ratios must be accurately measured and the effects of cPb and recent Pb-loss must be negligible.



**Figure 7.** Inverse (Tera-Wasserburg) U–Pb concordia diagram illustrating the effects of (A) recent Pb-loss or U/Pb calibration, (B) episodic Pb-loss, (C) common Pb contamination and (D) discordance due to either episodic Pb-loss or mixing of two age components. Numbers with black dots represent ages in Ga.

### 3.3 Secondary Ion Mass Spectrometry (SIMS)

The Secondary Ion Mass Spectrometer (SIMS) is a powerful instrument for the in situ analysis of the chemical and isotopic compositions of small volumes of solid materials. A beam of high energy primary ions are focused onto the area of interest with a spatial precision of 10–25  $\mu\text{m}$  across. The penetration depth is less than 2  $\mu\text{m}$  and the mass sputtered is in the range of  $10^{-10}$  to  $10^{-9}$  g (Stern, 2009). The SIMS analysis can hence be regarded as a surface analytical technique. Atoms and molecules sputtered by the high energy primary ions are focused into a secondary ion beam, later captured by a detector after passing a mass analyser (Fig. 8). The primary beam is produced through a cold-cathode duoplasma and the ion of choice, predominantly  $^{16}\text{O}^+$  or  $\text{O}_2^+$ , is filtered out. The primary beam is focused onto the sample surface, which sputters ions from the sample surface and generates secondary ions. The secondary ions are focused through electrostatic lenses and deflectors into a curved electrostatic energy filter, which bend low energy ions more than high energy ones. (Ireland and Williams, 2003) The apparatus filter the energy spectrum of interest and those ions enter a magnetic sector, i.e. the mass analyser. This sector is also curved and separate ions by mass/ $z$  ratio, where  $z$  is the ionic valence. An ion multiplier captures the separated ions. The ions are measured sequentially through dynamic peak jumping where each mass is counted for a short time period before it switches to the next mass (Benninghoven et al., 1987).

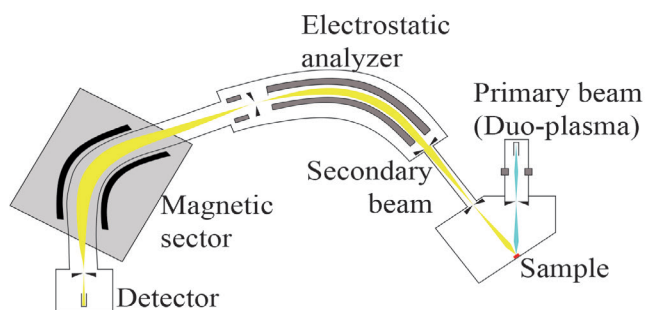


Figure 8. Simplified schematic illustration of a Cameca IMS 1280 ion probe.

### 3.4 SIMS U–Pb dating

For SIMS (Secondary Ion Mass Spectrometry) U–Pb dating of zircon, U, Th, Pb,  $^{204}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{208}\text{Pb}$ , ThO,  $\text{Zr}_2\text{O}$  and either  $^{238}\text{U}$  or its oxides UO and/or  $\text{UO}_2$  are measured (Benninghoven et al., 1987).  $\text{Zr}_2\text{O}$  is measured as an internal standard for calculation of concentrations of different elements. The Pb isotope ratios are measured for age calculations and common Pb corrections. U is measured for concentrations and U/Pb and ThO is used for Th/Pb. Resolving isobaric interferences between different ions or ion complexes is important, and high mass resolution is therefore required. For example,  $\text{HfO}^{2+}$  and  $\text{HfSi}^+$  have similar mass to the Pb ions as well as some of the REE hydroxides,  $\text{Hg}^+$  and  $\text{WO}^+$ . Some of these interferences can produce apparent high cPb concentrations resulting in overcorrected isotopic Pb-ratios.

For normalising data and to calibrate element ratios reference materials are required that are chemically well characterised. Zircon grains with uniform and known distribution of Pb, U and Th content and exactly determined isotopic ratios are used as standard material. These standards are continuously measured before, in between, and after analysis of natural samples to detect variations and fluctuations in the measurements. This procedure enables the correction of mass bias, mass fractionation and other fluctuations (Ireland and Williams, 2003).

In these studies SIMS zircon analysis was made at the Nordsim facility, at the Swedish Museum of Natural History, using a Cameca IMS 1280. Analytical procedures largely followed Whitehouse et al. (1999) and Whitehouse and Kamber (2005). Decay constants are from Jaffey et al. (1971).

### 3.5 Zircon O-isotope system

O is the most abundant element on Earth with three naturally occurring isotopes,  $^{16}\text{O}$ ,  $^{17}\text{O}$  and  $^{18}\text{O}$ . Their abundances are  $^{16}\text{O}$ : 99.763%,  $^{17}\text{O}$ : 0.0375% and  $^{18}\text{O}$ : 0.1995%. Since they are all stable and non-radiogenic, mass-dependent fractionation is the foremost process that creates variation in oxygen isotope ratios, although other physio-chemical processes such as kinetic effects are also important (Hoefs, 1997).

Water evaporation enriches the lighter  $^{16}\text{O}$  isotope in the vapour phase because of its lower mass. This leads to a difference in isotopic composition between the vapour and residual liquid phase. The isotopic difference is reported as the relative deviation from Standard Mean Ocean Water (SMOW) in parts per mil; a standard defined by Craig (1961) and is calculated according to the equation:

$$[\text{Eq. 3}] \delta^{18}\text{O} = [({}^{18}\text{O}/{}^{16}\text{O})_{\text{spl}} / ({}^{18}\text{O}/{}^{16}\text{O})_{\text{smow}} - 1] \times 10^3$$

Different geological reservoirs range over different spans of  $\delta^{18}\text{O}$  values as outlined by Hoefs (1997) who lists the most important geological reservoirs and their respective spans of  $\delta^{18}\text{O}$  values: viz. meteoric water c. -45‰ to 10‰, ocean water c. 0‰, sedimentary rocks c. 5‰ to 40‰, metamorphic rocks c. 0‰ to 25‰, granitic rocks c. -5‰ to 15‰ and basaltic rocks c. 5‰ to 10‰. Chondritic meteorites, bulk Earth and MORB all have values around 5.7‰  $\delta^{18}\text{O}$  (Fig. 9; Rollinson, 1993). As noted above, continental crust displays greater variation in  $\delta^{18}\text{O}$  relative to the Earth's mantle. The oxygen isotope variation in igneous rocks can be traced to incorporation of supracrustal material strongly affecting their  $\delta^{18}\text{O}$  values, as supracrustal material commonly has heavier oxygen signatures than primitive mantle magmas. This, and the fact that non-metamict zircon generally preserve their  $\delta^{18}\text{O}$  values of crystallisation (Valley et al., 2005), makes it possible to recognise crustal recycling and to get an indication of the composition of the host rock. However, zircon O-isotope ratios are only applicable with textural control (Valley, 2003). Overgrowths, mixed, inherited and new age domains or radiation-damaged areas can all cause misleading results.

In these studies zircon O-isotopes were analysed using the Cameca IMS 1280 SIMS at the NORDSIM facility, at the Swedish Museum of Natural History in Stockholm, largely following the analytical procedures of Whitehouse and Nemchin (2009).



### 3.6 Zircon Lu–Hf system

$^{176}\text{Lu}$  decays via  $\beta^-$  to  $^{176}\text{Hf}$  with a mean  $\lambda^{176}\text{Lu}$  of  $1.867 \pm 0.008 \times 10^{-11} \text{yr}^{-1}$  (Scherer et al., 2001; Söderlund et al., 2004). During partial melting, Lu/Hf fractionation is driven by the higher incompatibility of Hf over Lu. This has led to a depleted mantle with high (supra-chondritic)  $^{176}\text{Hf}/^{177}\text{Hf}$  and a crust with low (sub-chondritic)  $^{176}\text{Hf}/^{177}\text{Hf}$ . At the time of Earth formation, the Hf isotope composition was chondritic and has since fractionated into its present reservoirs. The divergence of the Hf isotopic composition is measured as parts per ten thousand deviations from a Chondritic Uniform Reservoir (CHUR) and is commonly expressed using the  $\epsilon$ -notation.  $\epsilon\text{Hf}$  is calculated using the equation below:

$$[\text{Eq. 4}] \epsilon\text{Hf} = \left[ \left( \frac{^{176}\text{Hf}/^{177}\text{Hf}}{(^{176}\text{Hf}/^{177}\text{Hf})_{\text{chondrite}}} - 1 \right) \times 10^4 \right]$$

Zircon is very resistant to Hf mobility and contamination since Hf forms an integral part of the zircon lattice. Hf concentrations in zircon is commonly c. 10 000 ppm which yield low Lu/Hf meaning that the initial  $^{176}\text{Hf}/^{177}\text{Hf}$  remains constant over time reflecting the source magma at crystallisation. This enables us to interpret the composition and age of the source rock as well as model the time of mantle extraction.

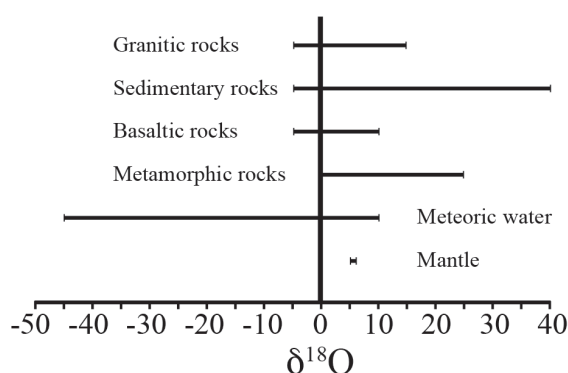


Figure 9. Different geological reservoirs and their respective  $\delta^{18}\text{O}$  span. Data from Rollinson (1993).

### 3.7 (LA)-MC-ICP-MS

The (Laser-Ablation) Multiple Collector Inductively Coupled Plasma Mass Spectrometer (MC-ICP-MS) is a double-focusing instrument with an array of Faraday collectors (Fig. 10). One of the main benefits of using a high temperature plasma discharge is the ability to generate a significant numbers of positively charged ions from a wide variety of elements in particular those with a high ( $>10\text{eV}$ ) first ionisation potential (e.g. Lu, Yb, Hf). The sample (from a liquid or a solid) is pumped into a spray chamber/nebuliser system creating an aerosol and is then injected into an Ar-plasma for ionisation. When reaching approximately 6000–8000 K at the analytical zone of the plasma, the sample consists of excited atoms and ions, representing the elemental composition of the sample. Once ionised the sample is transported into the mass spectrometer through an interface consisting of metallic cones with very small orifices that are maintained at vacuum. The ionisation procedure in the plasma generates ions with a large spread in kinetic energies. This would result in, if untreated, an optical signal disturbed by ions of different kinetic energy but identical mass. Hence, the first step in the mass spectrometer is energy focusing through a  $90^\circ$  electrostatic analyser that provide the desired energy focus necessary. After the electrostatic analyser, the ion beam is transported through a flight tube into a large magnet that provides mass dispersion large enough to facilitate the use of a multiple Faraday-cup set-up.

When coupling a laser ablation (LA) system to a MC-ICP-MS one can combine the benefits from the high ionisation potential of the ICP and the in situ analyses capabilities of the laser.

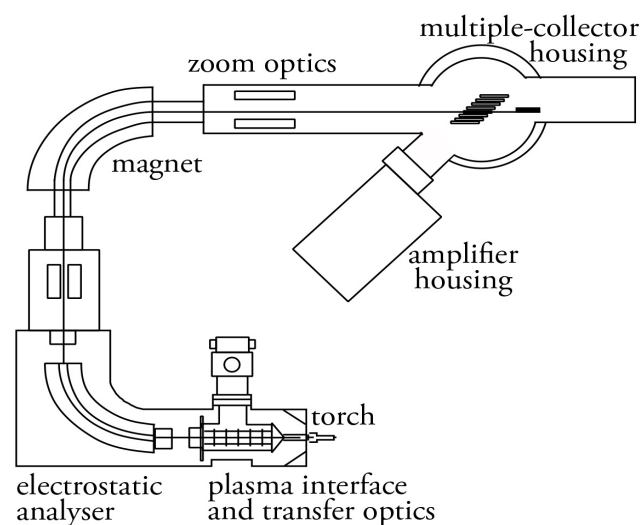


Figure 9. Simplified schematic illustration of a Neptune MC-ICP-MS.

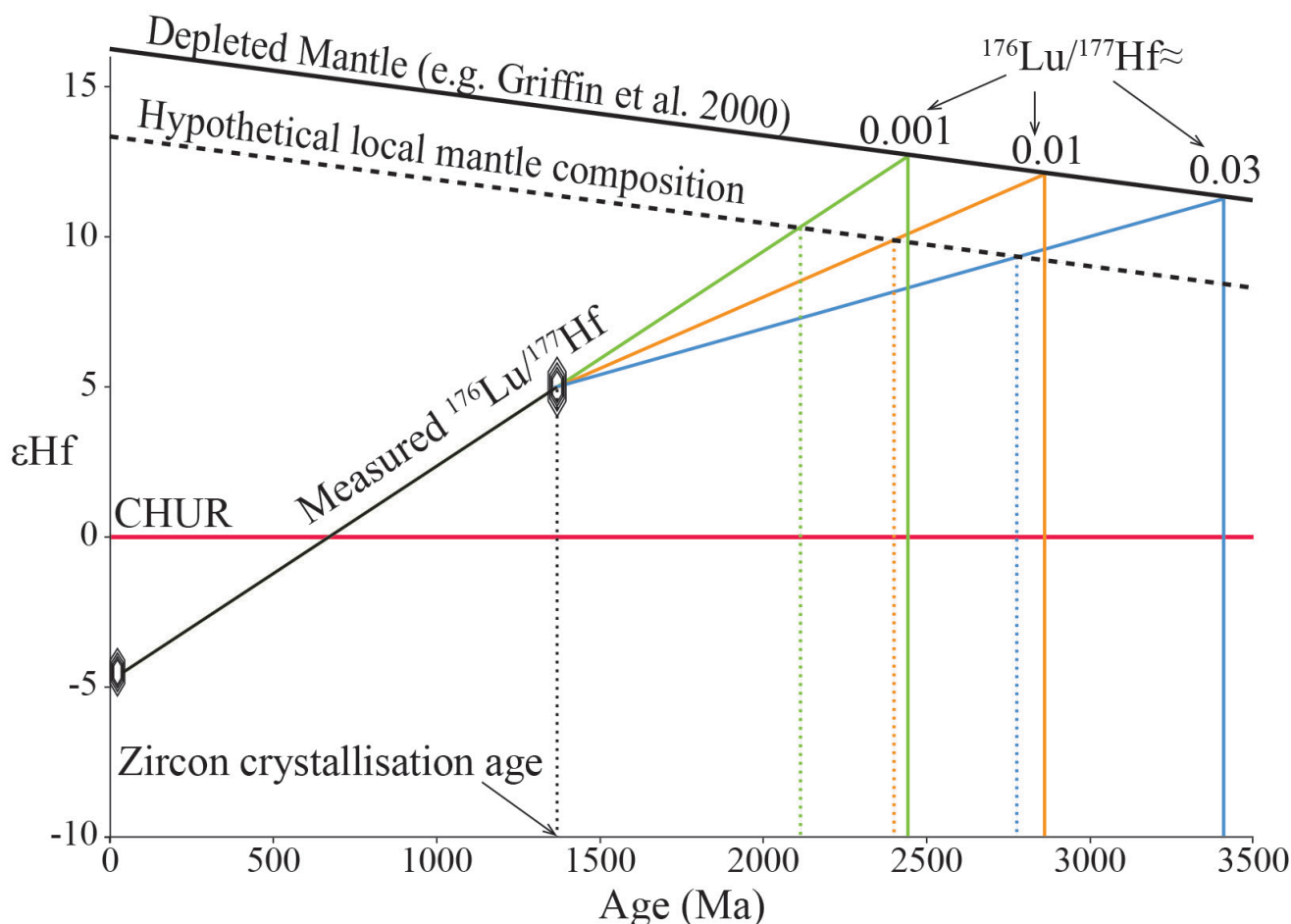
### 3.8 LA-MC-ICP-MS zircon Lu–Hf analyses

Zircon Lu–Hf analyses, included in this thesis were carried out at three different laboratories. At the Memorial University, St. Johns, Newfoundland, Canada, using a Finnigan Neptune multi collector ICP-MS connected to a GeoLas Laser ablation system (Paper I and III), at Goethe-University Frankfurt using a Thermo-Finnigan NEPTUNE multi collector ICP-MS coupled to a Resolution M-50 (Resonetics) 193 nm ArF excimer laser (ComPexPro 102F, Coherent) system (Paper II), and at the Advanced Analytical Centre at James Cook University in Townsville, Australia using a GeoLas 193-nm ArF laser and a Thermo-Scientific Neptune High resolution multi collector ICP-MS (Paper IV).

As the zircon Lu–Hf analyses were carried out at three different laboratories, slightly different analytical protocols were used. A description of a “common” analytical method is briefly summarised below.

Round laser spots with diameters between 25 and 60  $\mu\text{m}$  were drilled with repetition rate of c. 5 Hz and an energy density of c. 6 J/cm<sup>2</sup>. The Faraday cup set-up varied slightly but largely complied with masses 171 (Yb), 173 (Yb), 175 (Lu), 176 (Hf+Lu+Yb), 177 (Hf), 178 (Hf), 179 (Hf) and 180 (Hf+W+Ta). Isobaric interference of <sup>176</sup>Yb and <sup>176</sup>Lu on <sup>176</sup>Hf was calculated using the measured intensities of <sup>171</sup>Yb and <sup>175</sup>Lu along with known isotopic ratios of <sup>176</sup>Yb/<sup>171</sup>Yb = 0.897145 (Segal et al., 2003) and <sup>176</sup>Lu/<sup>175</sup>Lu = 0.02655 (Vervoort et al., 2004). Mass bias corrections were calculated using the exponential law. For calculations of  $\beta\text{Hf}$ , measured intensities of <sup>179</sup>Hf and <sup>177</sup>Hf and a <sup>179</sup>Hf/<sup>177</sup>Hf ratio of 0.7325 was used.  $\beta\text{Yb}$  was calculated using measured intensities of <sup>173</sup>Yb and <sup>171</sup>Yb and a <sup>176</sup>Yb/<sup>171</sup>Yb ratio of 1.130172 (Segal et al., 2003). Mass bias behaviour of Lu was assumed to be identical to Yb.

In addition, the stable Hf isotope ratios, <sup>178</sup>Hf/<sup>177</sup>Hf and <sup>180</sup>Hf/<sup>177</sup>Hf, were monitored since these should be constant within error throughout the measurements. External standards were in all cases used for quality control, and all data were adjusted relative to a standard reference.



**Figure 11.** Simplified  $\epsilon\text{Hf}$  versus crystallisation ages (in Ma) diagram, showing the influence of mantle composition and choose reservoir <sup>176</sup>Lu/<sup>177</sup>Hf on model-ages. Coloured solid lines show hypothetical model ages using a conventional depleted mantle model and reservoir <sup>176</sup>Lu/<sup>177</sup>Hf  $\approx$  0.001, Green; <sup>176</sup>Lu/<sup>177</sup>Hf  $\approx$  0.015, Yellow; <sup>176</sup>Lu/<sup>177</sup>Hf  $\approx$  0.030, Blue. Dashed lines indicate the same <sup>176</sup>Lu/<sup>177</sup>Hf-trajectories but using a hypothetical (less depleted) local mantle component.

Calculations of  $\epsilon_{\text{Hf}}$  use equation 4 and  $\lambda^{176}\text{Lu} = 1.867 \times 10^{-11} \text{yr}^{-1}$  (Scherer et al., 2001; Söderlund et al., 2004),  $(^{176}\text{Lu}/^{177}\text{Hf})_{\text{CHUR}} = 0.0336$  and  $(^{176}\text{Hf}/^{177}\text{Hf})_{\text{CHUR}} = 0.282785$  (Bouvier et al., 2008).

## 4. Methodological limitations

The zircon Hf-isotope two-stage model is the standard method for calculating model ages of felsic zircon bearing rocks, i.e. the time of extraction of crust from the mantle. The model age is hinging on two assumptions, namely knowledge of the  $^{176}\text{Lu}/^{177}\text{Hf}$  of the source rock and the degree of depletion of the mantle source as summarised in figure 11. A general depleted mantle evolution curve for the source (c.f. Griffin et al., 2000) and a precursor  $^{176}\text{Lu}/^{177}\text{Hf}$  ranging between 0.01 and 0.03 (c.f. Kemp et al., 2006; orange and dark blue lines respectively in figure 11) is commonly assumed. Using this approach and values, protolith crustal residence times from 100 to 400 million years are common, and have been explained by delayed silicic magma generation and infracrustal differentiation after initial crust extraction from the mantle (Kemp et al., 2006). However, it has been noted that, in relation to subduction zones, the underlying mantle is commonly less depleted than e.g. MORB, which form the basis of most depleted mantle curves (Nebel et al., 2011; Iizuka et al., 2013). The commonly used homogeneously and linearly depleted mantle models (e.g. Nowell et al., 1998; Griffin et al., 2000) are convenient and user-friendly mantle end-members to work with. However, the composition of the mantle source to most magmatic systems has shown to be very heterogeneous due to enrichment processes such as subduction of ancient crust/sediments (e.g. Hawkesworth et al., 1993; Plank and Langmuir, 1993; Elliott et al., 1997). Iizuka et al. (2013) show that the Hf isotope composition in modern island arc crust scatter by as much as c. 12  $\epsilon$ -units, which reflect variable enrichment of the mantle (and to a smaller extent local crustal contamination). It is thus reasonable to conclude significant mantle heterogeneity between different arc systems, and over time.

An important consequence of using an overly depleted mantle source is that model ages become too old and that the long residence times of the source rocks are simply model artefacts (Fig. 11). These model artefacts was for example shown by Nebel et al. (2011) in the Banda arc, East Indonesia where historic volcanic rocks yield Hf-model ages between 100 and >1000 Ma. A second consequence is that model ages are commonly used in the construction of continental crust growth curves, and will

result in too much crust generation too early in Earth's history. Furthermore, using model ages as the basis of distinguishing between juvenile and reworked crust in such a manner partly neglects a possible juvenile mantle derived contribution to rocks with extended mantle derivation ages.

Zircon crystallises predominantly from evolved magmas, usually with >60%  $\text{SiO}_2$  leading to a preservation and sampling bias towards differentiated rocks of the continental crust. This can hamper identification of rocks that were derived from juvenile sources. Zircon with depleted Hf isotope signatures that are similar to the mantle source are considered juvenile. Mantle depletion might vary however, and commonly has to be assumed, which hampers interpretation of the zircon data. This is particularly true in orogenic systems where the mantle source might be less depleted than MORB-like depleted mantle. Clearly, standard models outlined above fall short in this respect as a moderately depleted value might either be interpreted as a mixed component between a juvenile (depleted) and an ancient (enriched) reworked component, or simply as an old reworked component as outlined in the two-stage model above (Fig. 11). In conclusion, model ages must be interpreted with caution (c.f. Arndt and Goldstein, 1987).

## 5. Using detrital versus bedrock derived zircon

Coupled U–Pb–Lu–Hf–O isotope studies on detrital zircon have become an increasingly used tool for investigating the evolution of continental crust (e.g. Belousova et al., 2010; Condie et al., 2011; Voice et al., 2011 and Dhuime et al., 2012). The global data base of detrital zircon ages is large, (>200,000; Voice et al. 2011) and is thought to best represent the crustal rock record through time. There are a number of pros and cons to working with detrital zircon. These include the possibility to fast and easily sample large areas, but at an expense of contextual control. For example, Iizuka et al. (2005) analysed >400 zircon, extracted from one 3 kg sand sample collected at the Mississippi River mouth, allowing for interpretations on the growth evolution for large parts of the North American continent. Whether such samples are representative for the entire catchment area is another non-trivial issue where older bedrock (often situated in the central parts of cratons) become underrepresented when sampling at the river mouth. These problems can to some extent be dealt with by implementing

the K-factor as introduced by Allègre and Rousseau (1984) and recently tested by e.g. Dhuime et al. (2011a), but gets increasingly complicated when working with large data sets. Detrital zircon are commonly taken out of geological context and must be treated with caution. When dealing with detrital zircon each grain must be treated as a single sample and trends between different grains become very speculative (Fig. 12). This also complicates interpretations of isotopic signatures in these grains as discussed below.

It is commonly assumed that zircon with  $\delta^{18}\text{O}$  within error of the mantle value represent juvenile magmas derived from the mantle, while heavier oxygen signatures represent recycled or reworked supracrustal material (Fig. 9). These models assume that the zircon  $\delta^{18}\text{O}$  composition is an accurate proxy to distinguish between zircon that crystallised from juvenile magmas and zircon that crystallised from re-melted crust. However, as shown by e.g. Næraa et al. (2012), all reworked crust does not incorporate heavy oxygen, but can retain mantle value throughout the reworking process. Taken out of context, such data could appear to represent truly juvenile crustal contribution. Næraa et al. (2012) was able to identify these samples as derived from reworked ancient crust using Lu–Hf isotopes, but as shown in figure 4 reworking of an evolved crustal component without incorporation of supracrustal material does not influence the  $\delta^{18}\text{O}$  of the sample. Similar problems arise when working with Hf-isotopes in detrital zircon. Detrital zircon with prolonged Hf-model ages are assumed to have crystallised in re-melted older crust. However, it is (at least theoretically) as likely that the isotopic signature of a detrital zircon is the result of mixing between a juvenile and an ancient source (Fig. 12). To write these zircon of as

crystallised from merely reworked crust, neglects the juvenile contribution to this melt/crust. When working with zircon from bedrock samples e.g. granites, knowledge about the regional geology and the possibility to construct evolutionary arrays (see Paper I and III) through generically coupled samples helps distinguish between mixing and reworking (Fig. 12). Zircon from e.g. a granite will (if not perfectly homogenised) somewhat unpick the issue regarding mixing versus reworking. Excessive spread in the isotopic signatures (outside analytical error) hints toward contamination of the source i.e. mixing relations (Blue samples in Fig. 12). A detrital data set does not allow such interpretations as each analysis (e.g. zircon 1–5 in Fig. 12) must be treated as a single sample.

Further, trends between different samples (if corresponding to a reasonable  $^{176}\text{Lu}/^{177}\text{Hf}$  ranging between 0.01 and 0.03) allow for interpretation of a common source (Samples A–D in Fig. 12; Paper III), which combined with knowledge about the regional geology can provide certain robustness to once interpretations and mixing calculations. This is why contextual control is truly important, and why in situ sampling of bedrock e.g. granites, is better suited than detrital zircon when using the coupled zircon U–Pb–Lu–Hf–O isotope approach, at least at a regional scale. In situ sampling of bedrock has exclusively been used in the studies incorporated in this thesis, and is further discussed in section 7.

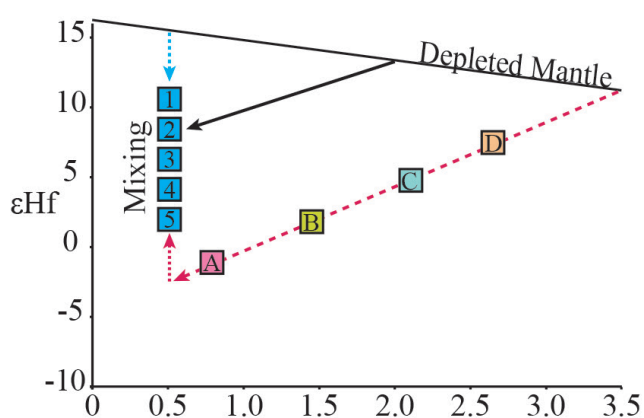
## 6. Summary of papers

### 6.1 Paper I

Zircon U–Pb and Hf–isotopes from the eastern part of the Sveconorwegian Orogen, SW Sweden: implications for the growth of Fennoscandia, by A., Petersson, A., Scherstén, J., Andersson, C., Möller, 2013. In: Roberts, N. M. W., Van Kranendonk, M., Parman, S., Shirey, S., Clift, P. D. (eds) *Continent Formation Through Time*. Geological Society, London, Special Publications, 389, first published online August 22, 2013, <http://dx.doi.org/10.1144/SP389.2>

Paper I presents new combined U–Pb and Lu–Hf isotopic data, from the Eastern Segment and the Idefjorden terrane of the Sveconorwegian Province, and suggests a revised model of crustal growth.

Currently accepted models for the growth of the continental crust in the southwestern part of Fennoscandia, including the eastern part of the Sveconorwegian Province, are largely based on U–Pb data and do not discriminate between juvenile and reworked crust.



**Figure 12.** Simplified  $\epsilon_{\text{Hf}}$  versus crystallisation ages (in Ga) diagram. Blue samples (1–5) illustrate internal spread within a sample population allowing for interpretations of mixing relations. However, if these analyses represent detrital zircon grains each analyses must be treated as a single sample and de-picking between mixing and reworking of a c. 2.0 Ga reservoir (as illustrated by the solid black arrow) becomes problematic. Analyses A–D illustrate different samples that allow for interpretation of a common source, provided the slope of the array correspond to a  $^{176}\text{Lu}/^{177}\text{Hf}$  between c. 0.01 and 0.03.



A majority of the basement in the southwestern parts of the Fennoscandian Shield was formed through mixing between a 2.1–1.9 Ga juvenile component and Archaean crust. Reworking of Archaean crust decreased with time between 1.9 and 1.7 Ga, homogenised and generated a mixed Svecofennian crustal reservoir. Subsequent magmatism between 1.7 and 1.4 Ga indicate sequential tapping of this reservoir with little or no generation of new crust.

Quartz to syenite magmatism with mildly depleted Hf-isotopic signatures at c. 1.2 Ga records juvenile contribution and crust net growth preceding the onset of the Sveconorwegian Orogeny.

Our new data indicating substantial reworking of 1.9–1.7 Ga crust within the arc system is in contrast to the previously proposed models of continental growth within the southwestern part of the Fennoscandian Shield. Long-term subduction along the margin of Fennoscandia is still preferred, but substantial reworking of existing continental crust and decreasing amounts of <1.9 Ga crustal growth is suggested.

## 6.2 Paper II

Mesoproterozoic continental growth: U–Pb–Hf–O zircon record in the Idefjorden terrane, Sveconorwegian orogen, by A. Petersson., A. Scherstén, B. Bingen., A. Gerdes., M. J. Whitehouse, 2014. *Precambrian Research* 261, 75–95. <http://dx.doi.org/10.1016/j.precamres.2015.02.006>

Paper II focuses on the Idefjorden terrane, located in the southwestern part of the Fennoscandian Shield and is thought to be an area of comparatively juvenile Mesoproterozoic crustal growth. Based on new, coupled zircon U–Pb–Lu–Hf–O isotopic data on thirteen samples of mafic to intermediate plutonic rocks from different domains of the Idefjorden terrane, we provide an improved model of crustal growth. The new data presented in this paper support alternating tectonics with a predominantly retreating volcanic arc system, with short pulses of accretion. This is seen through a gradual increase in  $\epsilon\text{Hf}$  from 3.5 to 5.4 in plutonic rocks that intruded between c. 1630 Ma and 1560 Ma. These isotopic signatures reflect an increased portion of juvenile magma with time, in the genesis of these rock suites. The trends seen in the zircon isotope data are consistent with development of an extensional back-arc rift geotectonic setting, accommodating deposition of the Stora Le-Marstrand greywacke dominated metasediment sequence. Variable amounts of metasediment incorporation during alternating tectonic regimes of contraction and extension in a mainly retreating subduction system, explains the heavy  $\delta^{18}\text{O}$  signatures of zircon in plutons intruding the

Stora Le-Marstrand metasediments. The detrital zircon record combined with the isotopic data presented in this paper suggest that the Idefjorden terrane was separated from the Fennoscandian Shield during deposition of the Stora Le-Marstrand metasediments.

From a crustal growth perspective, the isotopic signatures from zircon of the Idefjorden terrane indicate reworking of both older continental crust and sediments during crustal emplacement.

## 6.3 Paper III

Zircon U–Pb, Hf and O isotope constraints on growth versus reworking of continental crust in the subsurface Grenville orogen, Ohio, USA, by A. Petersson., A. Scherstén, J. Andersson, M. J. Whitehouse, M. T. Baranoski, 2014. *Precambrian Research*, (in press), <http://dx.doi.org/10.1016/j.precamres.2015.02.016>

As the basement in North America (Laurentia) is almost entirely covered by Palaeozoic cover-rocks its crustal evolution is poorly constrained. In paper III we present zircon U–Pb, O and Lu–Hf isotope data from drill-core samples from the subsurface basement of Ohio with the aim to improve our understanding of the evolution of this region. The isotope data suggest juvenile, mantle derived crustal addition at c. 1650 Ma followed by sequential reworking of a single reservoir. The c. 1650 Ma juvenile crust was reworked at c. 1450 Ma during the emplacement of the Granite-Rhyolite Province, and again during the Grenvillian orogeny.

The c. 1650 Ma protolith model ages of these samples combined with the presence of c. 1650 Ma magmatic rocks suggest an eastward extension of the Mazatzal Province and opens for a possible Mazatzal (or Mazatzal-like crust) protolith to the subsurface basement in Ohio and the surrounding Mesoproterozoic (i.e. Grenville-age) rocks. Such eastward extension of the c. 1650 Ma reservoir requires a revision of the >1.5 Ga crustal boundary, based on Nd isotope data, towards the east into Ohio. As the easternmost sample in this study has a more depleted Hf isotopic signature it limits the extent of >1.5 Ga basement in subsurface Ohio and constrains the location of the crustal boundary.

Also, c. 1050 Ma syn-orogenic magmatism suggests extrapolation of the Interior Magmatic Belt into subsurface Ohio. Heavy  $\delta^{18}\text{O}$  signatures in Grenvillian aged zircon and zircon domains suggest (re-)crystallisation of zircon in the presence of heavy  $\delta^{18}\text{O}$  fluids during the Grenville continent-continent collisional metamorphism.

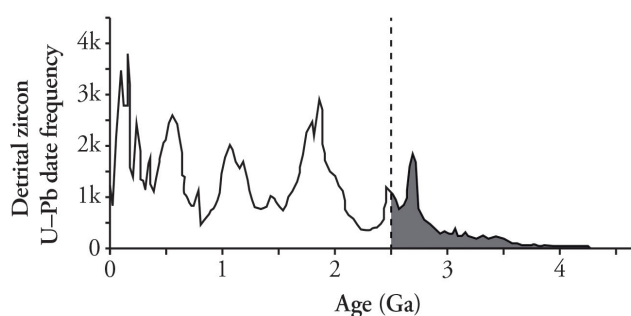


## 6.4 Paper IV

Zircon U–Pb–Hf evidence for reworking of Archaean crust within the Birimian Palaeoproterozoic source rocks of the West African craton, SE Ghana, by A. Petersson., A. Scherstén, B. Kristinsdóttir, A. I. S. Kemp, P. Kalvig, S. Anum., 2015. Submitted manuscript.

In paper IV we investigate the contribution of reworked Archaean crust to the Birimian bedrock of Ghana, West African craton, using zircon Lu–Hf isotopic composition of granites from the southern and northwestern parts of Ghana. Seven localities in southernmost Ghana and one locality in northwestern Ghana was sampled and separated for zircon. These zircon were analysed for combined U–Pb and Lu–Hf isotope data, and the results suggest predominantly juvenile crustal growth between 2.3 Ga and 2.1 Ga with a short but marked pulse of reworking of Archaean crust. Reworking of Archaean crust was previously poorly known and hinged on whole-rock Nd model-ages of the Winneba pluton in southern Ghana and sparse inherited zircon grains from mainly northwestern Ghana. The data presented in this paper suggest that reworking of Archaean crust was more extensive than previously known, but limited to a geologically short time interval between c. 2.141 and 2.126 Ga. This period was succeed by a fast return to juvenile crustal growth.

Coupled isotope data suggest a mainly retreating arc system with a shorter pulse of accretion between c. 2.18 and 2.12 Ga and a rapid return to slab retreat during the growth of the Birimian terrane. The accretionary phase initiated melting of sub-continental lithospheric mantle and the overlying Archaean crust, generating magma with sub-chondritic Hf signatures. Succeeding retreat of the slab led to trench-ward movement of the magmatic activity and a return to uncontaminated juvenile magma and supra-chondritic zircon Hf-isotope signatures.



**Figure 13.** Unfiltered global detrital zircon dates.  $n=199,358$  from Voice et al. (2011). Dashed line denote Archaean–Proterozoic boundary, and grey field denote pre-Proterozoic zircon.

Emplacement of the West Accra granodiorite (PK105) at 2.232 Ga requires felsic crust generation during the Eoerburnean. This predates the suggested plume-related rocks of Abuchami et al. (1990) and Boher et al. (1992) contradicting plume-initiated subduction during the formation of Birimian crust.

## 7. Combined zircon U–Pb–Lu–Hf-(O) isotopes as used in this thesis

As noted earlier, the mantle under a convergent margin can be variably enriched or depleted over time and along strike of an orogen. The potential effects of such heterogeneous mantle composition and an enriched or mildly depleted mantle are discussed in papers I, II and IV.

It has been argued by several authors that the syn-orogenic mantle underneath the proto-Fennoscandian crust was only mildly depleted due to long-term subduction-induced enrichment (e.g. Mearns et al., 1986; Valbracht, 1991a; Valbracht, 1991b; Claesson and Lundqvist, 1995; Andersson et al., 2006; Andersen et al., 2007; Rutanen et al., 2011 and Paper II). Similarly, Blichert-Toft et al. (1999) determined the local mantle component under the Birimian terrane of the West African craton at 2150 Ma to  $\epsilon_{\text{Hf}} = 6 \pm 2$ , about 3.5  $\epsilon$ -units below a MORB-like depleted mantle model of e.g. Griffin et al. (2000). This value is in good agreement with projected mantle depletion of arc systems (Dhuime et al., 2011b; Iizuka et al., 2013). As argued in section 4, applying a well-founded mantle model to one dataset (e.g. models from Dhuime et al., 2011b or Iizuka et al., 2013 to data derived from arc systems) is crucial when trying to differentiate juvenile versus reworked contribution to a suite of rocks. When studying a specific region, such as a pluton, orogen, arc or terrane, it is sometimes possible to link regionally associated rocks, and as outlined in section 5, to deduce an crustal evolution array in  $\epsilon_{\text{Hf}}$  versus time space. This might highlight specific reworking processes, pulses or periods of juvenile melt input (Sample A–D in Fig. 12, e.g. Paper I; Paper III). In paper I, we used combined zircon U–Pb and Lu–Hf data to show that 1.8–1.4 Ga granitoids in south Fennoscandia were drawn from a reservoir that formed through mixing between juvenile 2.1–1.9 Ga magmas and existing Archaean crust. This approach to combined zircon U–Pb and Lu–Hf isotope data can help unpicking the temporal evolution of a region, in particular with respect to derivation from a single or composite source and somewhat the

issue of mantle heterogeneity, although the mantle-composition remains an issue when/if calculating potential contribution ratios between the different sources (e.g. depleted mantle melts and reworked continental crust). Crustal evolution arrays can be argued to provide better constraints to mantle extraction ages than model-ages derived from scattered data sets or individual data points (e.g. detrital zircon) not compatible with a single array model. These models, however, still hinge on the assumption that the magmatic system remained closed during reworking.

In Paper III, a majority of the analysed samples from subsurface Ohio, allow for the construction of a crustal evolution array that was derived from a common source. The formation age of this source match the age of the c. 1.65 Ga juvenile Mazatzal Province (Whitmeyer and Karlstrom, 2007). This combined with the occurrence of 1.45 Ga A-type magmatism (commonly linked to the Mazatzal orogen) within subsurface Ohio allowed for the interpretation of the Mazatzal Province as a source of the basement of this region (e.g. Windley, 1993; Whitmeyer and Karlstrom, 2007).

The Hf-isotope data from rocks of the Idefjorden terrane (Paper II) does not form a simple crustal evolution arrays as in the Eastern Segment or subsurface Ohio. However, mirroring trends between Hf- and O-isotopes was interpreted in terms of increasing juvenile magmas, as shown by an increase in radiogenic Hf and a lowering of the zircon  $\delta^{18}\text{O}$ . These data in combination with the zircon U–Pb age constraints, is consistent with the development of an extensional back-arc rift geotectonic setting accommodating deposition of the local metasedimentary basin. Combined Hf- and O-isotopes further allowed mixing calculations for two-mica granites, between a local sedimentary component and a juvenile mantle end-member.

In Paper IV, covering the Birimian terrane of the West African craton in Ghana, sub-chondritic Hf-isotope signatures and internal spread vastly exceeding analytical error, indicate a composite source to many of the analysed samples. Mixing between an Archaean source and juvenile mantle derived magma during a short time-period (c. 2.14–2.13 Ga), was interpreted following the principles outlined in figure 12. Further, the coupled isotopic data, combined with regional geological features suggest an eastward, mainly retreating arc system with a shorter pulse of accretion between c. 2.18 and 2.12 Ga and a rapid return to slab retreat during the growth of the Birimian terrane. The accretionary phase initiated melting of sub-continental lithospheric mantle and the overlying Archaean crust, generating magma with sub-chondritic Hf signatures. Subsequent slab retreat led to trench-ward movement of the magmatic activity and the mixture of juvenile and Archaean crust was replaced by less contaminated juvenile magma.

## 8. Concluding remarks

Based on the different growth evolution curves proposed in figure 5, there appears to be no consensus on when the continental crust formed. However, most of the more recent zircon isotope based studies (i.e. the preserved crustal record) on the growth of the continental crust yield fast initial growth during the Hadean and Archaean, and reworking as the dominant process during post-Archaean times (e.g. Belousova, et al., 2010; Dhuime et al., 2012; Roberts and Spencer, 2014). Interestingly, incompatible trace elements of mantle derived rocks, which track the successive depletion (and enrichment) of the mantle yield similar results to those studies based on the zircon record (Sylvester et al., 1997; Campbell, 2003; Condie, 2003). The four studies in this thesis all highlight substantial reworking within Palaeo- to Meso-Proterozoic orogenic systems.

Nevertheless, juvenile crust form as well, and is even periodically dominant when these orogenic systems are dominated by e.g. accretionary orogenic phases and arc retreat. Juvenile rocks dominate the Birimian terrane in the West African craton, and in the Fennoscandian Shield, juvenile material plays a major role in the emplacement of both the Eastern Segment and the Idefjorden terrane. The Grenvillian rocks of subsurface Ohio may be dominated by reworked material, but mantle derivation of the now reworked material occurred during the Proterozoic.

The conclusions regarding these regions, presented in paper I–IV, are all based on coupled zircon U–Pb–Lu–Hf(O) isotope data and show a great variety between different orogenic systems, and also between different orogens of the same tectonic “type” (e.g. the Eastern Segment, the Idefjorden terrane and the Birimian terrane). This highlights the complexity in this kind of data, and if treated collectively, within a larger data set many of the aspects discussed in paper I–IV would most likely be lost.

Is combined zircon U–Pb–Lu–Hf(O) isotopes the best suited system of choice when approaching these questions? I argue that in regional studies where crucial parameters, such as mantle composition, composition of possible contaminants and genetic relationships between different rock suites, can be addressed, coupled zircon U–Pb–Lu–Hf(O) isotope data is well suited. As shown by DeCelles et al. (2009) and Kemp et al. (2009) even the regional and temporal dynamics of a single orogen can be interpreted using isotopes of zircon from igneous rocks. These studies conclude that generation of juvenile continental crust mainly occur in a retreating orogen where slab retreat lead to trench ward movement of the magmatic activity, and an increased influx of mantle derived melts.

Convergent margins are considered the primary site of crustal growth (e.g. Reymer and Schubert, 1984; Clift et al. 2009), and as shown by e.g. Nebel et al. (2011), Dhuime et al. (2011b) and Iizuka et al. (2013) a generic depleted MORB-mantle as proposed by e.g. Griffin et al. (2000) is not suitable in studies of convergent margins. I argue that subducted material returned to the crust through subduction related magmatism should be regarded as juvenile crust (c.f. isotope variation in MORB due to recycled crust; Chauvel et al., 2008). For this reason, mantle enrichment caused by different rates of subduction renders generic depleted mantle curves inapplicable. I propose that regional coupled U–Pb–Lu–Hf(O) studies on zircon from syn-orogenic primitive intrusions (e.g. gabbros) would provide better constraints for the regional mantle at any given time, and for mantle extraction times of granite precursors. This might provide a basis for future work within regional studies. However, as this approach is inapplicable to the large detrital zircon data archives, and because pre-Proterozoic zircon is clearly underrepresented in these archives (c.f. Fig. 13), the question remains whether zircon Hf-isotopes is suitable when trying to generate crustal growth curves through time. Detrital zircon data is certainly applicable and useful, but perhaps not a “silver bullet” that lack shortcomings. One must just be aware of what these are and treat the data accordingly.

Finally, the work included in this thesis agrees with an increased amount of reworking during continental arc magmatism and anatexis during the continent-continent collisional orogenic stage, while mantle derived juvenile contribution increases during slab retreat and is recorded mainly within island arcs and back arc settings.

## Svensk sammanfattning

För att förstå tillväxten av den kontinentala jordskorpan är det nödvändigt att kunna uppskatta balansen mellan juvenilt bidrag från manteln, infra-krustal omarbetning av jordskorpa och transport av krustalt material tillbaka till manteln. Sedan början av seklet har användandet av in situ U–Pb, Lu–Hf och O-isotop analyser av mineralet zirkon som ett verktyg för att adressera dessa frågor ökat exponentiellt. Många sammanställningar av ständigt växande databaser, som använder kombinerade U–Pb–Lu–Hf och/eller U–Pb–Lu–Hf–O isotop-data, har presenterats tillsammans med modeller som påvisar globala konsekvenser för den kontinentala jordskorpan tillväxt. Många av dessa modeller, lider dock av ett antal antaganden, bland annat inkluderandet av en homogen mantelreservoar som utarmats linjärt sedan jordens skapelse.

Användandet av framför allt detritala zirkoner tagna ur sitt geologiska sammanhang, och tillämpningen av dess modell-åldrar, försvagar trovärdigheten hos dessa modeller. För att ta itu med frågan om den kontinentala jordskorpan tillväxt, med hjälp av kombinerad zirkon U–Pb–Lu–Hf (–O) isotop-data, måste man ha kontextuell kontroll och minimera osäkerheterna i de tillämpade modellerna.

I denna avhandling har ett sådant tillvägagångssätt använts på tre olika Palaeo- och Meso-Proterozoiska (2500–1000 miljoner år gamla) orogena bälten; i den Fennoskandiska skölden, i nordamerikanska Grenville provinsen och i den Birimiska terrängen på den västafrikanska kratonen.

Den östra delen av den Svekonorvegiska provinsen (belägen i de sydvästra delarna av den Fennoskandiska skölden) skapades genom sekventiell omarbetning av en reservoar som bildades genom blandning mellan en 2.1–1.9 miljarder år gammal juvenil komponent samt Arkeisk skorpa. Från 1.7 till och med 1.4 miljarder år sedan genererades den kontinentala jordskorpan med liten eller ingen tillförsel av juvenilt material.

Längre västerut, i Idefjordsterrängen av Svekonorvegiska provinsen, återfinns 1.65–1.33 miljarder år gammal berggrund med isotopsignaturer som indikerar omarbetning av äldre kontinental jordskorpa och sediment. På det hela taget indikerar dock isotopsignaturerna från berggrunden i Idefjordsterrängen en ökning av juvenilt material med tiden. Detta är i linje med en retirerande öbåge, samtida med avsättning av den lokala sedimentära bassängen, Stora Le-Marstrand.

Trender i isotopdata från den Grenvilliska orogenen under Ohio föreslår juvenilt bidrag från manteln för ca. 1.65 miljarder år sedan, följt av sekventiell omarbetning av denna reservoar med litet eller inget ytterligare bidrag från manteln.

Den ca. 2.31–2.06 miljarder år gamla Birimiska terrängen i Ghana, är känd för att till hög grad uppvisa juvenila signaturer. Men vi kan visa att omarbetad Arkeisk skorpa bidragit i mycket större utsträckning än tidigare känt, vilket återigen betonar vikten av infrakrustal omarbetning under bildandet av kontinental jordskorpa. Vi kan även påvisa att denna terräng, som normalt beskrivs som bildad via initialt bidrag från en mantelplym, har granitiska bergarter som är äldre än de mafiska leden. Detta påvisar istället en subduktionsrelaterad tillväxt för den kontinentala krustan i denna terräng.

Tillsammans understryker dessa studier vikten av infrakrustal omarbetning i Palaeo- och Meso-Proterozoiska ackretionsorogener. Dessa studier ger också goda exempel på tillämpning av kombinerade zirkon U–Pb–Lu–Hf (–O) isotop-analyser av berggrund med känd affinitet, där valet av använda modeller kan styrkas.





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