Ti-in-zircon thermometry applied to contrasting Archean metamorphic and igneous systems

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Ti-in-zircon thermometry applied to contrasting Archean metamorphic and igneous systems

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Abstract

Ti-in-zircon thermometry with SHRIMP II multi-collector has been applied to two well-documented Archean igneous and metamorphic samples from southern West Greenland. Zircons from 2.71 Ga partial melt segregation G03/38 formed in a small (<1 m$^3$), closed system within a mafic rock under high pressure granulite facies conditions. Results of 14 Ti analyses present a mean apparent zircon crystallization temperature of 679±11°C, underestimating independent garnet-clinopyroxene thermometry by 20-50°C but consistent with reduced $a_{\text{TiO}_2}$ in this system. 36 spot analysis on 15 zircons from 3.81 Ga meta-tonalite G97/18, with an estimated magmatic temperature >1000°C, yield a low-temperature focused normal distribution with a mean of 683±32°C, further demonstrated by high resolution Ti mapping of two individual grains. This distribution is interpreted to represent the temperature of the residual magma at zircon saturation, late in the crystallization history of the tonalite. Hypothetically, Ti-in-zircon thermometry on Eoarchaean detrital zircons sourced from such a high temperature tonalite would present a low-temperature biased image of the host magma, which could be misconstrued as being a minimum melt granite. Multiple analyses from individual zircons can yield complex Ti distributions and associated apparent temperature patterns, reflecting cooling history and local chemical environments in large magma chambers. In addition to inclusions and crystal imperfections, which can yield apparent high temperature anomalies, zircon surfaces can also record extreme (>1000°C) apparent Ti temperatures. In our studies these were traced to $^{49}$Ti (or a molecular isobaric interference) contamination derived from the double sided adhesive tape used in sample preparation, and should not be assigned geological significance.

Keywords: Ti thermometry; zircon; Archean; Greenland; Isua; tonalite
1. Introduction

Incorporation of titanium into crystallizing zircon is seemingly primarily controlled by temperature and $a_{\text{TiO}_2}$ (Watson et al., 2006), and once within the zircon structure has a very low diffusivity under geologic conditions (Cherniak and Watson, 2007). This thermometer then, has the potential to yield valuable information on the thermal evolution of metamorphic and igneous rocks. Initial application of the Ti-in-zircon thermometer was to 3.91-4.35 Ga detrital zircons from the Jack Hills, Western Australia. The low temperature distributions obtained were used to propose the existence of prograde, wet minimum melting conditions during formation of the granitic protolith to the Hadean zircons (Watson and Harrison, 2005, 2006; Harrison and Schmitt, 2007; Harrison et al. 2007). This hypothesis carries far reaching implications for the dynamics of the early earth and has since stimulated ongoing debate (Nutman, 2006; Valley et al., 2006; Coogan and Hinton, 2006). In order to further explore the validity of applying the experimentally determined Ti-in-zircon thermometer to natural systems, we have examined zircons separated from Archean rocks formed in different conditions. This has the advantage that the chemistry and geologic setting of the host rocks are well known and provides for a more detailed understanding of what Ti signatures from Archean and Hadean detrital zircon populations may reveal about early Earth environments.

The new Ti thermometry results are from zircons from two contrasting Archean samples from southern West Greenland. The first, a 3.81 Ga meta-tonalite represents some of the best preserved Eoarchean felsic crust yet discovered. Such tonalites typically crystallize as large volume magma chambers from melting initiating above 1000°C (Rapp and Watson, 1995). The second sample is from a local, trondhjemitic, minimum melt segregation associated with high pressure granulite facies metamorphism. Each sample has previously determined U/Pb zircon geochronology, petrogenetic constraints and information relevant to the thermal evolution of the rocks (Nutman et al., 1989, 1999; Nutman and Friend, 2007), allowing assessment of under what situations Ti-in-zircon thermometry is best applied.
2. Samples

2.1. 3.81 Ga meta-tonalite sample G97/18
G97/18 is part of the exceptionally well-preserved suite of 3.8 Ga meta-plutonic rocks located between the Isua supracrustal belt and 65°N, initially reported by Nutman et al. (1999). The sample is a homogeneous biotite ± hornblende meta-tonalite with relict igneous texture and very weak biotite foliation (Figure 2 in Nutman et al. (1999)). The trace element characteristics of this sample are typical of Archean tonalite-trondhjemite-granodiorite (TTG) suites, with for example light rare earth element (REE) enrichment, heavy REE depletion, and high Sr/Y. The bulk chemistry of this sample can be modeled by ca. 30% melting of a mafic rock under sufficient pressure to stabilize residual garnet and clinopyroxene (eclogite), with little or no plagioclase fractionation superimposed during emplacement into the crust (Nutman et al., 1999). Extensive experimental data shows that such compositions require temperatures above 1000°C to form (Rapp and Watson, 1995), and thereby would be classified as “hot” granites.

Zircons in the sample are euhedral to subhedral prisms, with generally well-preserved oscillatory igneous zonation. U/Pb geochronology provides a weighted mean $^{207}\text{Pb} / ^{206}\text{Pb}$ age of 3808 ± 4 Ma (n=26, MSWD = 1.9) for oscillatory zoned zircon (Nutman et al., 1999). Crowley (2003) replicated these ages in a U/Pb zircon study using ID-TIMS of tonalites from the same outcrop. There are rare low Th/U replacement domains on the edges of grains as old as 3790 Ma, and some other rare recrystallisation domains have yielded ages as young as 3600 Ma (Nutman et al., 1999). Petrographic studies indicate some zircons contain ilmenite inclusions (Figure 1), which are interpreted to be the predominant Ti-rich phase in equilibrium with the growing igneous zircon. Zircons from this rock, owing to their antiquity, narrow range of ages, and general absence of younger overgrowths have been used in number of geochemical studies including determining the xenon isotopic composition of the Earth at 3.81 Ga. (Honda et al., 2003).

2.2. 2.71 Ga partial melt segregation G03/38
G03/38 collected from Qilanngaarsuit Island, Nuuk district, southern West Greenland was initially reported by Nutman and Friend (2007). The island is dominated by outcrop of the Eoarchaean Itsaq Gneiss Complex (Nutman et al., 1996; Nutman and Friend, 2007) in mylonitic contact with a 2840 Ma tectonic supracrustal slice (Chadwick and Nutman, 1979). On the southern end of the island a ≤200m thick unit of amphibolite and paragneiss preserves small domains of high pressure granulite assemblages. G03/38 is a small (<1m$^3$), coarse grained, trondhjemitic segregation (Figure 3c. in Nutman and Friend, 2007) in equilibrium with garnet, clinopyroxene and ilmenite from the rock’s high pressure granulite assemblage. Mixture modeling using MIX of M (Sambridge and Compston, 1994) on 40 analyses from 29 grains indicated a major zircon age population representing metamorphism at 2714±4 Ma and a minor group representing recrystallisation at 2690±6 Ma (Nutman and Friend, 2007).

Zircons bear rare plagioclase and clinopyroxene inclusions, with only moderate HREE enrichment and negative Eu anomalies, indicating the 2714 Ma zircons should have formed in equilibrium with garnet, clinopyroxene and plagioclase (Nutman and Friend, 2007). The high pressure granulites are commonly partially retrogressed to lower pressure amphibolite facies assemblages, with the replacement of clinopyroxene by hornblendic amphibole and garnet by symplectites containing plagioclase + hornblende. However, locally, such as in sample G03/38, the higher-pressure assemblage is well preserved, with minimal retrogression and many equilibrium boundaries preserved between garnet + clinopyroxene + plagioclase + quartz (Figure 2). This suggests the small trondhjemitic melt segregations developed in the field where dehydration melting of amphibolites starts to occur, at pressures where garnet is stable (Wyllie and Wolf, 1993).

### 3. Analytical methods

Zircon separates previously extracted from rock samples by conventional crushing, heavy liquid and magnetic techniques were used in this study. Approximately 60 grains from
each sample were transferred onto double sided adhesive tape with a fine-tipped needle under a binocular microscope and flattened onto their c-axis. The grains were shielded with the adhesive tape’s paper spacer before being set in epoxy with zircon reference materials SL13 (Claué-Long et al., 1995), FC1 (Paces and Miller, 1993) and Temora-2 (Black et al., 2003). For the initial analytical session a second mount containing SL13, FC1, Temora-2 zircon reference materials; and NIST 610, 612 and 615 (Pearce et al., 1997) glass reference materials was made. Mounts were polished to expose crystal mid-sections with a rotary polisher and 1µm diamond paste. All grains were imaged with reflected light, transmitted light and cathodoluminescence spectroscopy to identify cracks, inclusions and 2-dimensional growth structure to guide analysis. Prior to each analytical session mounts were sequentially cleaned in an ultrasonic bath with petroleum spirits, ethanol, diluted laboratory detergent, 1M HCl solution, and deionized H$_2$O before being thoroughly dried in a 60°C oven. A 100Å Au coat was finally applied to the analytical surface and checked to ensure uniform and adequate conductivity before loading into the instrument.

Zircon Ti concentrations were determined using the SHRIMP II multi-collector ion microprobe at the Australian National University over five analytical sessions. Data acquisition and reduction protocols are presented in detail by Aikman (2007) and summarized here. Isotopic ratios were produced by simultaneous measurement of $^{49}$Ti$^+$ with a gain checked, Sjuts continuous dynode electron multiplier at the high mass detector; and $^{28}$Si$^{16}$O$^+$ by Faraday cup at the low mass detector using count rates $>10^6$ cps. SiO background counts (~0.1% of total SiO counts) were subtracted during setup configuration, $^{49}$Ti background counts (2-4 cps) were subtracted during data reduction. Mass resolution was 5,000 at 1% peak height. The low abundance $^{49}$Ti isotope (5.41%) was chosen to avoid interference with $^{96}$Zr$^{2+}$ or $^{48}$Ca$^+$ (from apatite inclusions) on the major $^{48}$Ti peak (73.72%) as counting statistics were not a limiting factor for precision.

For spot analysis during the first four analytical sessions a 2.0 to 4.5 nA O$_2^-$ primary beam was focused to a 20µm diameter spot, producing a sensitivity of $>19$ cps $^{49}$Ti/ppm Ti/nA O$_2^-$. Prior to the start of data acquisition a 120µm surface raster was programmed
for 120 seconds to clean the analysis area and avoid Ti contamination from within the
gold coat. Data acquisitions consisted of 10-20, 10 second integrations.

Tabulated results from zircon and glass reference materials and the operating conditions
specific to each instrument session is presented in Background Dataset 1. SiO/$^{49}$Ti ratios
from zircon and glass reference materials are consistent for concentrations at the ppm
level. Internal precision from counting statistics of reference materials is better than 0.2%
with external precision matching internal precision during periods of high performance.
The standard error from SiO/$^{49}$Ti ratios on SL13 was <0.7% (n=64) over all five sessions.

Temora-2 zircon, FC1 zircon, NIST 610, 612 and 615 glasses were analysed as secondary
reference materials to monitor instrument performance, but not directly used to calculate
zircon Ti abundances or temperatures.

As part of this study, the Ti concentrations of 23 grains of SL13, FC1 and Temora-2
(previously analysed by SHRIMP) were independently determined by LA-ICPMS.
Analysis was performed on the RSES Aligent 7500 ICPMS equipped with a Lamda
Physik LPX 1201 UV ArF eximer laser and Ar-He flushed sample cell (Eggins et al.,
1998). The laser operated at 22Kv with 100mJ energy per pulse at 5Hz. The 54µm
diameter spot was placed directly over the ~1µm deep SHRIMP pits. Each acquisition
consisted of a 20 second background followed by a 20 second collection period. Blocks
of 10 unknowns were bracketed by analyses of NIST 610 and 612 glass reference
material. Raw counts were converted to concentrations using “LABRAT 0.93” written for
Lab VIEW by Antti Kallio. Corrections for mass bias drift in unknowns were made using
NIST 612. Zircon Ti abundances (Background Dataset 1) were normalized to
stoichiometric SiO$_2$. The mean (2σ) Ti concentration of SL13 was found to be relatively
homogeneous (6.3±0.3 ppm, n=10), while Temora-2 (9.1±0.6 ppm, n=6) and FC1
(21.9±1.6 ppm, n=7) are subtly more heterogeneous.

Coinciding SHRIMP (SiO/$^{49}$Ti) and LA-ICPMS [Ti] zircon analyses vary approximately
linearly when plotted in (SiO/$^{49}$Ti)$^{-1}$ vs [Ti] space. This array defines a matrix sensitive
regression through the mean value of SL13 and the origin (Figure 3A). This point and
zero calibration line was used to reference the Ti concentration of unknowns from measured SiO/^{49}Ti ratios with well characterized SL13 (Figures 3B and 3C). Ti concentrations for SL13, analysed routinely (typically once every three unknowns during spot analyses) is presented in Figure 3D. This calibration approach is currently limited by reference material heterogeneity and the complex error magnification envelope for unknowns with greater titanium content than the SL13 reference material. Relative errors of <5% are commonly achievable for most geologically useful titanium contents (1-40 ppm).

Command-line software “TiZer” (Aikman, 2007) has been written to process raw SHRIMP multi-collector output files to Ti concentrations and temperatures. SiO/^{49}Ti ratios for single analyses are calculated from scan medians and corrected for drift through the session by robust regression. Ratios are reduced to Ti concentrations using the calibration line described above, and crystallization temperatures using the empirical calibration defined by Watson and Harrison (2005) and Watson et al. (2006). Analytical errors are a product of counting statistics, dispersion in measured SiO/^{49}Ti ratios of reference materials, systematic errors in the Ti content of zircon reference materials (measured by LA-ICPMS) and the uncertainty from the thermometer calibration. Each component of uncertainty is independent. Ti concentration and temperature absolute uncertainties (2σ level) are derived by multiplying uncertainties from each term by their partial derivatives and summing the results in quadrature.

Accurate knowledge of $a_{\text{TiO}_2}$ and $a_{\text{SiO}_2}$ at the time of zircon crystallization remains fundamental to the accuracy of the thermometer. Both samples indicate simultaneous crystallization of quartz, zircon and ilmenite suggesting $a_{\text{SiO}_2} = 1$ and $a_{\text{TiO}_2} < 1$. All temperatures are uncorrected for $a_{\text{TiO}_2} < 1$, which in these systems we conservatively estimate to be ~0.6 based on the presence of ilmenite and calculated $a_{\text{TiO}_2}$ for similar phase assemblages (Ghent and Stout, 1984). This would lead to underestimations of zircon crystallization temperatures by <50°C (Watson and Harrison, 2005).
Following spot analyses, the mount was lightly polished to remove pits from Ti work, then re-cleaned and re-coated with Au for U/Pb geochronology on SHRIMP RG at RSES. The methods used are described in detail by Stern (1998) and Williams (1998) and are summarized here. A 2.5nA primary beam was focused to a 20μm spot diameter and positioned over sites of Ti analysis. A 120μm raster was programmed for 120 sec to clean the mount surface prior to data acquisition. FC1 zircon reference material was analysed once every 3 unknowns. Data reduction was preformed using the Excel™ macro SQUID (Ludwig, 2001). Zircon reference materials SL13 (U = 238 ppm) and FC1 ($^{206}\text{Pb}^{238}\text{U}$ age = 1099.0±0.5 Ma) were used for U abundance and $^{206}\text{Pb}^{238}\text{U}$ calibrations respectively.

The fifth Ti analytical session involved Ti imaging of selected grains. An ion microprobe is the best tool suited to measure low ppm Ti levels in zircon, with maximum spatial resolution. Imaging allows for determination of the origin of Ti variation as either geologically significant temperature differences, or due to localized features and imperfections such as edge effects, inclusions or cracks in the crystal structure.

Prior to imaging, the mounts were again lightly polished, re-cleaned and re-coated with Au. Zircon grain maps were segmented into blocks to minimize the analytical time spent off the grain and on surrounding epoxy. Each block was setup to run though a matrix with automatic stage drive, primary and secondary tuning. Horizontal and vertical stage movements were programmed to match the spot size so grain coverage was uniform and maximized, without pit overlap or associated geometric effects. Instrumental setup was identical to that described for spot analyses, except a 1.6nA O$_2$ primary beam was focused to a ~5μm spot, providing maximum spatial resolution without compromising sensitivity. Prior to the start of data acquisition, a 120 second, 10μm pre-sputter burn-in was programmed directly over the analysis area to clean it of Ti contamination, with minimal disturbance to adjacent analytical sites, or to the sample’s potential field defined by the conductive coating. Data acquisitions for each spot consisted of 4, 10 second integrations.
No detectable impact to primary beam focusing or secondary ion yields was observed with progressive removal of the Au layer, or due to the formation of topography on the mount surface from material build-up from adjacent analytical pits. Ti concentrations and temperatures were calculated with TiZer using SL13 zircon reference material analyses separating blocks of unknowns. Concentration values were converted first to 2D matrices and then to unsmoothed Ti intensity grain maps using “Intensity and 3D plot” written for Lab VIEW by Peter Lanc.

New independent temperature estimates for sample G03/38 were determined by WDS mineral analysis on a Cameca SX-51 electron microprobe at the Institute of Geology, the Chinese Academy of Geological Sciences. The vicinity of analysed mineral assemblages was imaged by backscattered electrons (BSE). Garnet-clinopyroxene Fe-Mg exchange thermometry was calculated via the Ellis and Green (1979) calibration using the Excel™ spreadsheet PX-NOM of Sturm (2003).

4. Results

4.1. Spot Analyses

A summary of Ti thermometry and U-Pb dating results for G97/18 and G03/38 spot analyses are presented in Table 1. Composite CL images with analysis locations, crystallization temperatures and $^{207}\text{Pb}/^{206}\text{Pb}$ ages corrected for very small amounts of common Pb (based on measured $^{204}\text{Pb}$) are presented in Figures 4 and 5.

4.1.1. G97/18

Zircons from G97/18 are typically ~200µm but upto 300µm in length and prismatic in habit. Oscillatory zonation is fine, running parallel to grain boundaries and locally cut by domains of recrystallization. Fourteen $^{207}\text{Pb}/^{206}\text{Pb}$ ages range from 3741 to 3833 Ma. Ten analyses on oscillatory zoned zircon indicate ages of >3800 Ma, while three of the four analyses <3800 Ma are recrystallized domains. 36 Ti spot analyses from 15 grains yielded a range of crystallization temperatures from 631°C to 777°C. The normal
distribution is skewed towards its low temperature end, peaking from 660°C to 690°C and with a mean of 683±32°C, 1σ (Figure 6A). Based on growth history identified by oscillatory zonation the dataset consists of 9 grain core analyses (mean = 699±44°C, 1σ), 21 mid grain analyses (mean = 682±25°C, 1σ) and 6 grain edge analyses (mean = 662±19°C, 1σ). Five of the nine core analyses were made on recrystallized areas (mean = 677±33°C, 1σ) but typically show no indication of disturbance to Ti concentrations when compared with adjacent analyses on the same crystal. Six grains received at least three analyses and four grains were analysed twice. From these ten grains with multiple spots, seven show expected systematic trends from apparently high temperature cores that become cooler towards the grain edge; two grains show reverse patterns with lower temperatures towards the grain interior, while one shows an oscillating temperature profile from core to edge. Uncertainties from individual spot temperatures might be broad enough to incorporate at least some of these apparent differences. Rim overgrowth domains were deliberately avoided during these analyses. No systematic relationship exists between Ti temperature and trace element variation as revealed by dark and light CL domains. However, oscillatory zonation in these grains is particularly fine (<5µm), and analysis by the 20µm beam would potentially average micrometer scale compositional variations. Similarly no association can be made between temperature and recrystallised domains, U concentration, Th concentration, U/Th ratio, $^{204}$Pb corrected $^{207}$Pb/$^{206}$Pb age or discordance.

4.1.2. G03/38

Zircons from G03/38 are dominantly ~100µm but upto 200µm in length and typically oval to equant in shape. Most display turbid or sector zonation while smaller components are bright and structureless, or show oscillatory zonation. Eleven $^{207}$Pb/$^{206}$Pb ages range from 2666 to 2756 Ma, eight analyses are associated with the recognized 2714±4 Ma peak and three with the minor 2690±6 Ma peak. Fourteen Ti analyses from eleven grains yielded a narrow range of crystallization temperatures from 660°C to 697°C, with a mean of 679°C±11°C, 1σ (Figure 6B). Again no correlation between Ti concentration or temperature and CL, degree of recrystallisation, U, Th, U/Th, $^{204}$Pb corrected $^{207}$Pb/$^{206}$Pb or discordance exists.
Results from G03/38 garnet-clinopyroxene mineral analyses are presented in Table 2. Garnet + clinopyroxene + plagioclase + quartz domains display minor alteration by retrogressive reactions, and ilmenite is the main Ti-bearing phase (Figure 2). Within the garnets are well preserved clinopyroxene inclusions, whose edges are unaffected by retrogressive amphibole-forming reactions. Along the edges of garnets are coronas of hornblende + plagioclase, locally as narrow as 100μm. The main garnet + clinopyroxene + plagioclase (An41-42) + quartz assemblage is interpreted to be coeval with the felsic segregation observed in outcrop. This segregation was the source of the zircon used for Ti-in-zircon thermometry.

Garnet-clinopyroxene pairs C and D from the grain interiors (Figure 2) yield temperatures of 700-730°C (between 6 and 10 kbar). Garnet-clinopyroxene pairs A (at the margin of unaltered clinopyroxene within garnet) and B (separated by a 100 μm hornblende + An45-45 plagioclase corona) give lower temperatures of 590-630°C (between 6 and 10 kbar).

4.2. Grain Matrix Mapping
Titanium intensity grain maps were produced on grains G97/18-2, G97/18-11 and FC1-7 in order to investigate the details of Ti distribution within single oscillatory zoned grains. Grains from G97/18 were targeted as they emerged with significant Ti variability during spot analyses. FC1-7 was selected to look for an association between large, distinct CL brightness domains and Ti concentrations. The tabulated results are presented in Background Dataset 2.

4.2.1. G97/18-2
Four spot analyses on grain G97/18-2 (Figure 4, Table 1) presented the largest variation of Ti concentration (2.5 to 14.9 ppm) and crystallization temperature (631 to 777°C). The distribution of spot concentrations indicates temperatures are highest at the core and
become lower towards both tips. Hence this grain may provide the best thermal representation of G97/18 zircon growth in the history of one single crystal.

The Ti intensity map for G97/18-2 is presented in Figure 7A with associated reflected light, transmitted light and CL images. The grain appears pristine, free of any cracks or inclusions and to have experienced a smooth, uninhibited growth. Its concentration profile demonstrates a systematic decline in Ti from core to rim with mutually consistent values where spot analyses would overlap. This provides a valuable internal check on the validity of measurements made under different analytical approaches during different sessions. The unsmoothed grain image is constructed of 431 pixels, excluding the 0.0 ppm background. Each pixel represents an independent SHRIMP analysis which yielded concentrations ranging from to 2.0 ppm (617°C) to 572.5 ppm (1289°C). Of these 431 analyses, 59 recorded concentrations >15.0 ppm (16 of these >100.0 ppm) and are irregularly concentrated around the grain’s extreme rim. Rim overgrowth domains were avoided during spot analyses, subsequently these high and variable concentrations feature as an unexpected result in the grain matrix map. The figure was constructed with the Ti concentration range capped at 15.0 ppm to highlight the grain’s internal variability from core to mid regions rather than extreme concentrations on the grain edge. This limit corresponds to the maximum concentrations measured in the grain’s core and represents a crystallization temperature of approximately 778°C.

Ti crystallization temperatures derived from G97/18-2 are presented in Figure 6C. The profile consists of 372 analyses with concentrations from 2.0 ppm (617°C) to 15.0 ppm (778°C). The normal distribution is relatively smooth, uninterrupted, and skewed towards lower temperatures, with a mean of 676±40°C (1σ). Again temperatures associated with rim concentrations >15.0 ppm were filtered from the image. Figure 8 has been added to represent a revised version of the Ti intensity map if >15.0 ppm pixels are excluded. Here the grain appears with a more uniform edge and better resembling morphology from reflected, transmitted and CL imaging.

4.2.2. G97/18-11
Five spot analyses on grain G97/18-11 revealed complex and unsystematic distributions of Ti concentrations (range of 3.5 to 13.6 ppm) and crystallization temperatures (range of 656 to 768°C) that undulate on a transect from the grain core to tip, following oscillatory growth domains identified in CL imaging. Absolute uncertainties on analytical spots provide inadequate overlap to explain these differences.

The Ti intensity map for G97/18-11 is presented in Figure 7B. Orientation has been rotated clockwise ~45° with respect to the grain as presented in Figure 4. Reflected and transmitted light imaging reveal an inclusion and cracks that meet the polished surface. CL imaging makes clear the large recrystallised core and small opaque inclusion within large oscillatory zoned domains. The image is made of 583 pixels of concentrations ranging from 2.0 ppm (616°C) to 68125.4 ppm (4044°C). Of the 583 analyses, 100 recorded concentrations >15.0 ppm with 25 >100.0 ppm. Ti concentrations are again consistent with values from spot analyses where overlapping. The figure concentration range was similarly capped at 15.0 ppm to provide clarity to the image. Highest values are again irregularly distributed around the grain’s extreme rim and also penetrate the grain edge with anomalously large excursions occurring near the cracks and inclusion. The distribution of Ti is complex but shows no indication of disturbance in the large recrystallized core that appears as a uniform thermo-chemical domain. In the vicinity of the earlier spot analysis transect, Ti concentrations from pristine zircon appear to display a transient oscillation between approximately 3.0–8.0 ppm.

Crystallization temperatures from G97/18-11 are presented in Figure 6D from analyses of concentrations ranging from 2.0 ppm (616°C) to 14.6 ppm (775°C). The distribution is normal with a locus at the mean of 691±26°C (1σ). Temperatures associated with concentrations >15.0 ppm were again removed from the profile.

### 4.2.3. FC1-7

No spot analysis was previously performed on grain FC1-7. It was analysed to firstly look for relationship between Ti distribution and spatially resolvable CL domains, and secondly to test if high Ti rims were a unique feature to grains from sample G97/18. The
Ti intensity map for FC1-7 is presented in Figure 7C. The crystal appears pristine, free of any cracks or inclusions. Its concentration profile demonstrates a gradient in Ti from one side to the other but can not be clearly associated with the tone of CL domains. The image comprises 156 pixels with concentrations ranging from 7.9 ppm to 240.7 ppm. 19 analyses recorded concentrations >15.0 ppm, with 11 >100.0 ppm. The highest Ti concentrations are irregularly distributed along the length of the higher concentration side of the crystal. The figure concentration range was capped at 50.0 ppm for clarity.

4.3. Origin of high Ti rims

To further pursue the origin of the very high Ti rims observed on G97/18-2, G97/18-11 and FC1-7, whole zircons from G97/18, G03/38, SL13, FC1 and Temora-2 were depth profiled using LA-ICPMS. Crystals were placed onto a glass slide with double sided adhesive tape and flattened onto their c-axis. Grains were again protected from airborne contamination by the adhesive tape’s paper spacer when not inside the LA-ICPMS sample cell or being photographed. Analytical methods were identical to those described above for the reference material calibration except that acquisitions consisted of collection periods of upto 360 seconds to try and penetrate the grains entire thickness. Drilling started from the top surface, and generally passed through the entire grain, the bottom surface, adhesive tape and into the glass slide. Results are presented in Table 3. Grains from every aliquot recorded a marked increase in Ti concentrations on most top and bottom surfaces (Figure 9A). Of the 77 surfaces analysed from 51 grains of G97/18, 86% showed anomalously high Ti concentrations, 90% of the 10 surfaces on 5 grains of G03/38 showed excursions, 53% of the 32 surfaces on 15 grains of SL13 showed excursions, 68% of the 53 surfaces on 32 grains of FC1 showed excursions, 85% of the 27 surfaces on 17 grains of Temora-2 showed excursions. Anomalous Ti values could often be detected within the crystal structure during laser drilling but these probably represent analysis of ilmenite inclusions.

SL13 crystals are broken fragments from a larger zircon megacryst. As such their surfaces are modern features. This suggests that the apparent high Ti rims are an artifact of sample processing rather intrinsic features. To test this, further aliquots of the Temora-
2 reference material were variously subjected to either: (1) air abrasion with pyrite crystals to remove the grain exteriors; (2) HF partial dissolution with 1:1 HF-HCl in a sealed teflon beaker for 24 hours on a 120°C hotplate; (3) repeated acetone, or (4) ethanol rinses with an ultrasonic bath followed by decanting. The aim of this exercise was to remove any surface contamination originating from magmatic chemical disequilibrium, minor phases, residues from sample crushing or milling, heavy liquids or other aspect of sample processing that may resulted in high Ti. Results are presented in Table 3. High Ti rims were still widely observed (on 83% to 100% of analysed surfaces) from all treated aliquots suggesting none of these contamination processes are the cause of this effect.

In a last attempt to understand the origin of these signatures direct analysis of the glass slide, epoxy and adhesive tape used to hold the grains was made. While glass and epoxy produced no anomalies, high Ti excursions from the adhesive tape provided identical enrichment profiles to the grain surfaces (Figure 9B), making this contact medium the favored cause for the excursions. It appears $^{49}$Ti (or a molecular isobar) can unevenly mix from the adhesive tape into the adjacent epoxy during mount casting, and also be transferred to the grain’s top surface by contact with the adhesive tape’s paper spacer, which is commonly placed on top of the zircons to protect them at various times during mount preparation. The Ti signature of the tape may be an organic molecule that is unresolved from $^{49}$Ti in the SHRIMP even at the ~5000 mass resolution used during analysis. However, it is noted that similar features have been seen on zircon exteriors by other workers when measuring $^{48}$Ti (Harrison and Schmitt, 2007). These features have in some cases been attributed to extreme high temperature geologic events (Trail et al., 2006).

5. Discussion

5.1. Temperatures from zircons of metamorphic rock G03/38

The G03/38 Ti-in-zircon temperatures of $679\pm11^\circ$C (1σ) are based on a calibration that assumes rutile as the Ti-buffering phase. Because the main Ti-bearing phase in G03/38 is
ilmenite the calibration may underestimate the calculated G03/38 zircon temperatures by up to 50°C. With this consideration there is a reasonable agreement between the Ellis and Green (1979) garnet-clinopyroxene Fe-Mg exchange thermometer (700-730°C) and the Ti-in-zircon thermometer for this sample. Garnet-clinopyroxene pairs A and B give lower temperatures (590-630°C) probably reflecting conditions during superimposed hydration reactions. These results indicate that well equilibrated metamorphic zircon, which forms over a relatively narrow time interval is capable of yielding precise Ti crystallization temperatures that are relatively consistent with established thermometers, although this will require accurate knowledge of $a_{\text{TiO}_2}$ for the given system. It suggests the method could be a useful tool for determining (possibly multiple) metamorphic conditions in complicated rocks.

5.2. Ti thermometry of 3.81 Ga meta-tonalite G97/18

Magmatic temperatures expected during formation of tonalitic melt compositions have been constrained by extensive experimental data at above 1000°C (Rapp and Watson, 1995), classifying such lithologies as “hot” granites. Sample G97/18 is free of inherited zircon and has a Zr abundance of 121 ppm (Nutman et al., 1999). Using the zirconium saturation in melts relationship of Watson and Harrison (1983) as a guide, G97/18 should become saturated in zircon at ~729°C, hence zircon should crystallize as a late phase from a residual melt at temperatures significantly less than that of the parental liquidus (cf. Nutman, 2006). Modeling has been used to predict the expected distribution of zircon formation as a function of temperature during cooling of a similar intermediate plutonic system (Harrison and Watson, 2006; Figure 3 in Harrison et al. 2007). These calculations predict the distribution would exist as a broad spectrum, with zircon growth declining away from its high temperature end.

The maximum Ti-in-zircon temperatures from G97/18 tonalite (reaching 777±14°C) are notably higher than those expected from zircon saturation thermometry. However the majority of temperatures (mean of 683±32°C, 1σ), reinforced by detailed mapping of grains G97/18-2 (676±40°C) and G97/18-11 (691±26°C) are significantly lower. These
temperatures and distributions are identical, within errors, to those associated with wet, minimum melt, low temperature granites (Watson and Harrison, 2005). The form of these temperature distributions may reflect real gradients for natural zircon formation in similar plutonic systems over protracted retrograde intervals.

The dataset is not biased by lack of high temperature analyses from any specific crystal domains (such as cores) as we have analyzed entire grain mid-sections. Given a plausible $a_{\text{TiO}_2}$ for this igneous system of ~0.6, corrections for subunity $a_{\text{TiO}_2}$ would lead to upward temperature adjustments of <50°C, however, the $a_{\text{TiO}_2}$ of individual detrital zircons can not be precisely known. Subsequently, analysis of Eoarchaean detrital zircons derived from a tonalite such as G97/18 would present a low temperature biased image for their magma source and perhaps lead to the erroneous interpretation of their origin in a minimum melt, low temperature granite.

In some grains the distribution of Ti can be correlated to core and rim domains identified by grain imaging. Trends of Ti variation are consistent with processes of magma cooling (such as in grain G97/18-2) and less frequently as magma recharge or grain convection (in grain G97/18-11). However, during a recharge scenario the introduction of hot, Zr undersaturated melt in the presence of late-crystallizing zircons would be likely to partially or entirely dissolve existing grains (Watson, 1996; Nutman et al., 1999; Mojzsis and Harrison, 2002). No indication of dissolution is found on any crystal. Hence a favored alternative interpretation to the oscillating Ti variations seen in G97/18-11 relates to local disequilibrium in trace element partitioning at the zircon/melt interface (Hoskin, 2000).

6. Conclusions

The Ti-in-zircon thermometer can provide precise temperature estimates in well-equilibrated high grade metamorphic systems. However the accuracy will depend on
accurate knowledge of the $a_{\text{TiO}_2}$. Large igneous systems such as those that produce
 tonalites, undersaturated in Zr and with $a_{\text{TiO}_2} < 1$, produce low apparent Ti-in-zircon
 temperatures and distributions that may accurately record the crystallization temperatures
 of zircon, but likely underestimate that of the parental melt. An inference from this study
 is that detrital zircons from a rock like tonalite G97/18 would yield low temperature
 distributions, which could lead to an erroneous interpretation of the lithology from which
 these zircons were eroded. Thus, there is an inability to distinguish with confidence,
 using only Ti thermometry, zircons grown in granitic magmas generated by “hot” (900-
 1000°C) and “cool” (650-750°C) melting. Furthermore individual zircons from large
 magma chambers can yield complex Ti temperature distributions reflecting variable
 cooling histories and local chemical environments. Apparent extreme (>1000°C) Ti-in-
 zircon temperatures derived from zircon rims of samples and reference materials are an
 artifact of sample preparation. Our study monitoring mass $^{49}\text{Ti}$ has traced the origin of
 excess Ti to a contaminant in the double sided adhesive tape on which zircons are held
 prior to casting in epoxy mounts, or on which they are held during laser ablation ICP-MS
 depth-profiling. Caution in assigning geologic meaning to temperatures from near grain
 surfaces as well as inclusions and cracks is emphasized.

Acknowledgments

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Laurence Coogan and Paul Hoskin.
References


Figure Captions

Figure 1.

Cathodoluminescence (CL) and transmitted light (trans) images of fine oscillatory zoned G97/18 grain with exposed ilmenite (ilm) inclusion.

Figure 2.

Backscattered electron image of G03/38 high pressure assemblage with well preserved garnet (gnt) + clinopyroxene (cpx) + plagioclase (plag) + quartz (qtz) equilibrium.
boundaries. Retrogression to hornblende (hbl) is minimal. A, B, C and D indicate locations of garnet and clinopyroxene WDS mineral analysis.

Figure 3.

Zircon calibration and reference material data A) SHRIMP $^{49}$Ti/SiO vs LA-ICPMS [Ti] linear relationship from SL13, Temora-2 and FC1 reference materials. Point and zero calibration line passes from the origin through the mean of SL13 values; B) SHRIMP $^{49}$Ti/SiO with TiZer calculated [Ti] for unknowns and related reference materials from sessions 1-4; C) As B) for session 5; D) Summary of Ti concentrations from 64 analyses of SL13 over all sessions. Mean = 6.14±0.02 ppm (2σ).

Figure 4.

Composite CL image for G97/18 sample zircons with analysis spots, uncorrected crystallization temperatures and $^{207}$Pb/$^{206}$Pb ages. Grains G97/18-2 and G97/18-11 reveal complex temperature profiles targeted for Ti matrix mapping.

Figure 5.

Composite CL image with analysis spots, uncorrected crystallization temperatures and $^{207}$Pb/$^{206}$Pb ages for zircon of sample G03/38. Note the narrow range of crystallization temperatures for this simple closed system.

Figure 6.

Histogram and probability density plots of uncorrected crystallization temperatures for A) spot analysis of sample G97/18, B) spot analysis of sample G03/38, C) mapping of single grain G97/18-2, D) mapping of single grain G97/18-11. Temperatures associated with Ti concentrations >15.0 ppm (>778°C) and >14.6 ppm (>775°C) filtered from figures C and D respectively.

Figure 7.

From left to right: reflected light, transmitted light, CL images and Ti concentration maps for grains A) G97/18-2, length of crystal 200µm, note highest concentrations observed in
Figure 3

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**Zircon calibration:**

**A**

Ti/Sm ratio from spot analysis during session 2 and 3, Ti (ppm) from LA-ICPMS.

- SL13 (n=10)
- Temora-2 (n=6)
- FC1 (n=7)
- SL13 Calibration

---

**Zircon calibration:**

**B**

Ti/Sm ratio from spot analysis during sessions 1-4, Ti (ppm) from TiZr

- SL13
- Temora-2
- FC1
- G97/18
- G03/38
- SL13 Calibration

---

**Zircon calibration:**

**C**

Ti/Sm ratio from grain matrix mapping during session 5, Ti (ppm) from TiZr.

- G97/18-2
- G97/18-11
- FC1-7
- SL13

---

**SL13 zircon reference material**

**D**

Ti (ppm) vs. Analysis

- Session 1
- Session 2
- Session 3
- Session 4
- Session 5
Figure 9

Click here to download high resolution image
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