Title

Thermal evolution of the Superior craton: Accessory phase U-Pb thermochronometry constraints on a diamond-forming event younger than 1.1 Ga near Attawapiskat, Ontario

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THERMAL EVOLUTION OF THE SUPERIOR CRATON: ACCESSORY PHASE U-PB THERMOCHRONOMETRY CONSTRAINTS ON A DIAMOND-FORMING EVENT YOUNGER THAN 1.1 GA NEAR ATTAWAPISKAT, ONTARIO

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by

Samuel Landis

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Thermal evolution of the Superior craton: Accessory phase U-Pb thermochronometry constraints on a diamond-forming event younger than 1.1 Ga near Attawapiskat, Ontario

by

Samuel Landis

Abstract

The Superior craton represents the largest exposure of Archean crust on Earth and can offer insights into the long-term evolution of cratonic geotherms. U-Pb geo- and thermochronology of zircon and rutile from lower and mid-crustal xenoliths record lower crustal crystallization, metamorphism, and long-term cooling prior to surface exhumation within a Jurassic-aged kimberlite. Archean igneous zircon LA-ICPMS ages range from 2751 ± 21 to 2341 ± 41 Ma, indicating a prolonged record of zircon crystallization. Preliminary U-Pb LA-ICPMS rutile data has a far wider range of ages from 2660 ± 140 to 324 ± 17 Ma and are interpreted to record the long-term relaxation of the cratonic geotherm. Nitrogen aggregation data collected from diamonds exhumed within the Jurassic and a nearby older Proterozoic (~1.1 Ga) kimberlite pipe suggest at least two major diamond-forming events after craton formation. These data require that a thermal pulse caused by the Keweenawan Midcontinent Rift at ~1.1 Ga reverted the mostly high N-aggregated IaB diamonds to graphite but did not perturb the deep crust (~50 km depth). As the geotherm relaxed after the thermal pulse, the diamond stability window was reoccupied and a second diamond-forming event took place before later Jurassic eruption of poorly nitrogen-aggregated type IaA diamonds near Attawapiskat, Ontario. Maintenance of Archean

rutile U-Pb cooling ages in middle-crustal amphibolite limits the magnitude of reheating.

Introduction

The Superior craton (Fig. 1) is the amalgamation of several continental nuclei with accreted magmatic arcs, plutonic complexes, and greenstone belts that combine to form the Earth's largest exposure of Archean crust (Kusky and Polat, 1999). With the bulk of the craton assembled ~2.7 billion years ago during a series of distinct orogenic events collectively termed the Kenoran orogeny (Langford and Morin, 1976), the Superior craton experienced two subsequent major thermo-tectonic events. At 1.8 Ga the Trans-Hudson orogeny saw the amalgamated Superior craton collide with the Western Churchill province in a Himalayan-style orogeny (Eaton and Darbyshire, 2010) and at ~1.1 Ga a large rifting event impacted the southern margin of the Superior craton (Smit et al., 2014).

The Attawapiskat kimberlite field is comprised of 21 known kimberlite pipes that occur roughly 100 km from the Attawapiskat community in northern Ontario. The kimberlites erupted between 180 and 156 Ma (Rb-Sr phlogopite; U-Pb perovskite; Kong et al., 1999; Heaman and Kjarsgaard, 2000) and were discovered in 2005 by Metalex Ventures (Smit et al., 2014). The samples for this study all come from the Victor North kimberlite pipe currently being mined by De Beers Canada.

Smit et al. (2014) investigated the aggregation states between two neighboring suites of diamonds from Attawapiskat and Kyle Lake ~100 km to the southwest (Fig. 2). Nitrogen is the primary impurity found in natural diamonds and its aggregation state is used both to classify diamond types and to estimate mantle time-temperature residence paths (Evans and Qi, 1982; Taylor et al., 1990). Type I

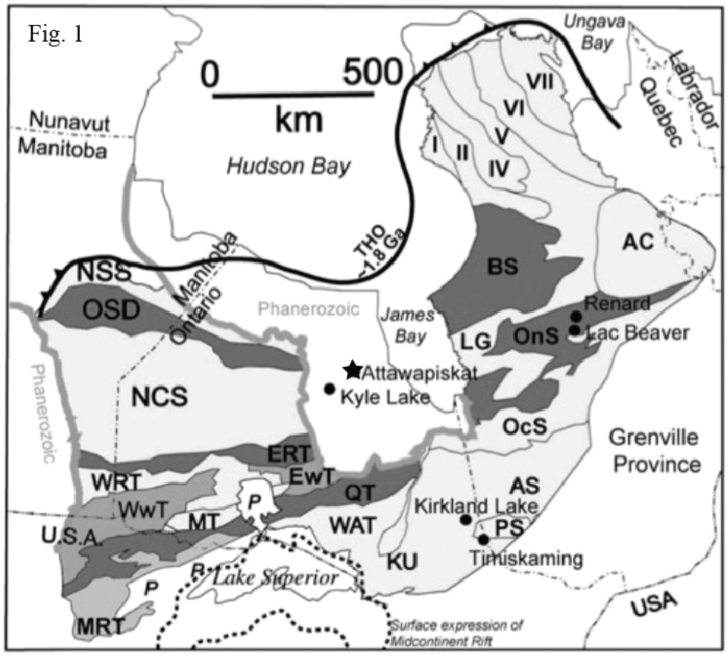


Figure 1 shows the regional setting of the Superior craton in grey with the study area of Attawapiskat (star) and Kyle Lake (dot) indicated. The northern surface expression of the Mid Continent Rift is dotted at the bottom of the figure. From Smit et al., 2014.

diamonds contain detectable amounts of nitrogen, while type II do not. Nitrogen impurities within a type I diamond mature from isolated N-N pairs (type IaA diamonds) to groups of four N atoms surrounding a vacancy (type IaB) with increased mantle residence times and/or increased thermal energy.

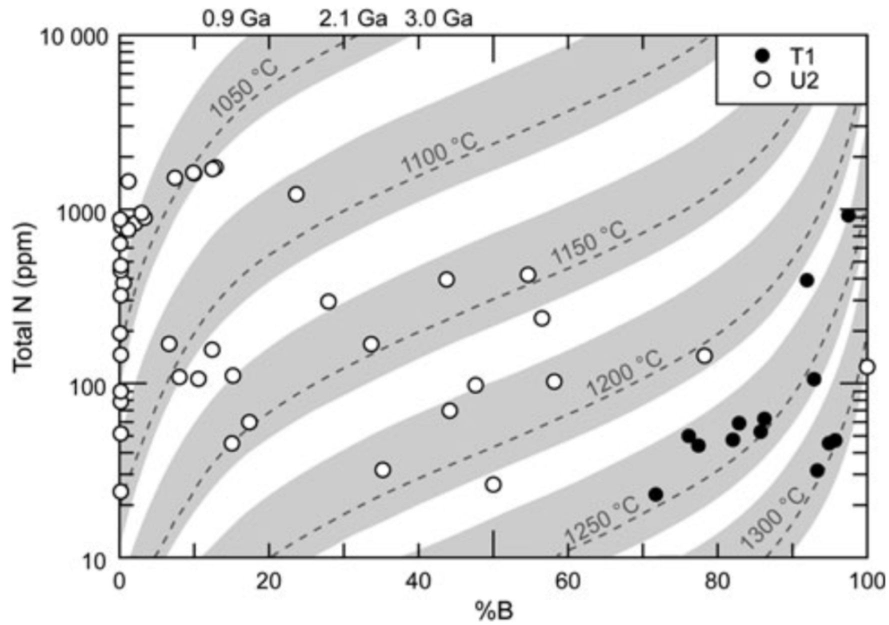


Figure 2 shows the nitrogen aggregation state of the two suites of diamonds examined by Smit et al. (2014). The white circles correspond to immature type IaA (low %B along the A to B transition) diamonds erupted at Attawapiskat in the Late Jurassic while the black circles are the highly aggregated type IaB (high %B) diamonds erupted earlier at ~1.1 Ga at Kyle Lake. From Smit et al., 2014.

Smit et al. (2014) inferred the older, mature, high-B aggregation state Kyle Lake diamonds were erupted before the Keweenawan Rift thermal pulse destroyed the remaining diamonds, thereby resetting the diamonds to graphite until the deep mantle keel cooled back to the diamond stability window where new, immature A-state aggregation types were formed and later erupted in Attawapiskat. The general lack of highly aggregated type IaB diamonds in Attawapiskat is interpreted by Smit et al. (2014) to be strong evidence for a thermal pulse destroying nearly all highly-aggregated diamonds at ~1.1 Ga.

The Midcontinent Rift System, or Keweenawan Rift, is one of the world's major continental rifts and extended approximately 2,300 km through central North

America roughly 1.1 billion years ago (Fig. 1) (Perry et al., 2004). The Keweenewan Rift ultimately failed before significant crustal separation occurred but deposited expansive basalts along the shores of Lake Superior and may have thermally impacted much farther north to the Attawapiskat area (Heaman et al., 2004; Smit et al., 2014).

The diamondiferous kimberlite pipes of Attawapiskat, Ontario offer a unique opportunity for analyzing time-temperature geothermal evolution of an Archean craton. Recent advances in U-Pb accessory phase thermochronometry of apatite, rutile, and titanite allow for the estimation of rock cooling histories as they pass from high to moderate temperatures and corresponding depths of 20-50km (Schmitz and Bowring, 2003). Accessory minerals from deep crustal xenoliths can provide insights into the thermal relaxation of ancient cratonic lithosphere as rocks cool from high temperature igneous crystallization events to lower crustal steady state geotherms (Blackburn et al., 2011). These enhanced understandings of geothermal evolution can help elucidate the evolution of diamond stability windows beneath Archean cratons and may be able to help dispel the dogma that all diamonds must be billions of years old. To this end, the purpose of this study was to juxtapose high precision U-Pb thermochronology analysis with previous nitrogen aggregation diamond data reported by Smit et al. (2014).

Methods

Of a total 22 samples cut into thin sections, 6 samples were rutile-bearing and 5 were zircon-bearing (2 samples had both rutile and zircon). Lithological compositions ranged from deep crustal eclogites, to moderate depth mafic granulites, to shallower amphibolite samples.

The duel decay of ^{238}U and ^{235}U to ^{206}Pb and ^{207}Pb, respectively, makes accessory minerals robust geochronometers and thermochronometers that can reliably record cratonic geothermal evolution (Schmitz and Bowring, 2003). Zircon U-Pb dates record high temperature igneous or metamorphic zircon growth. If the suspected thermal pulse at ~1.1 Ga resulted in pervasive magmatism throughout the lithosphere near Attawapiskat, zircon U-Pb apparent ages could reflect the event or at least reflect it in younger metamorphic rims.

Rutile has a much lower U-Pb closure temperature of 450-550° C and records the final stage of geothermal relaxation to temperatures predicted for a steady state geotherm in the lower crust (Blackburn et al., 2011). Together rutile and zircon offer long-term insights into cratonic geothermal evolution and relaxation.

Accessory minerals were separated from the kimberlite xenoliths using standard crushing, magnetic, and heavy liquid techniques with final separates sorted based on grain size, crystal morphology, and color. Zircons were then mounted on an epoxy disk, polished, and carbon coated before being imaged with a cathodoluminescence (CL) scanning electron microscope at Stanford University to identify zonation and potential targets for laser ablation. Rutiles were also mounted and polished on epoxy disks but not CL imaged. Both disks were then loaded for laser ablation inductively-coupled mass spectrometry (LA-ICPMS) at the University of California, Santa Cruz on a PhotonMachines Analyte 193H with a 193nm ArF excimer laser system, following the methods of Steely et al. (2014). Samples were standardized against FC-1 as primary standard and Temora as a secondary standard. The average $^{207}Pb/^{206}Pb$ age for secondary standard Temora was 406.7 ± 8.3 Ma. Raw data were reduced, downhole fractionation corrected, and filtered with 2σ outlier rejection in Iolite v. 2.5 run within IgorPro v. 6.3.

In order to draw meaningful conclusions about temperatures indicated by U-Pb dates, it is necessary to quantitatively evaluate Pb diffusion behaviors within an accessory mineral. Diffusion kinetics dictate concentrations of an element (in this case Pb) as a function of time, temperature, a production term which balances radiogenic decay of parent and daughter nuclides, and the mineral's radius (Blackburn et al., 2011). Further complicating the matter is the presence of two distinct parent and two daughter masses with variable parent and daughter retention rates with different temperatures. Open system behavior occurs at higher temperatures where diffusion occurs fast enough to liberate daughter nuclides as soon as they form. Closed system behavior occurs at lower temperatures where diffusion is slow enough that essentially all daughter product is retained. The time-temperature space between open and closed systems is represented by the Partial Retention Zone (PRZ) (Blackburn et al., 2011).

This project utilized advanced quantitative modeling of the above Pb diffusion kinetics to constrain the time-temperature evolution of the cratonic geotherm through a wide range of different time-temperature paths in a separate thermal evolution model. The modeled Pb diffusion parameters were used to help constrain the thermal evolution models used to bracket and interpret measured U-Pb ratios and dates to better understand the complex evolutionary history of the Superior craton and its diamond stability field and nitrogen aggregation states. The implementation of thermal models to predict a geothermal evolution that can then be used to compare and bracket analytic LA-ICPMS or TIMS data has been shown to be a robust method for thermochronological analyses (Blackburn et al., 2012).

Results

U-Pb Zircon

Five zircon-bearing deep crustal xenolith samples from the Victor North kimberlite pipe were analyzed using LA-ICPMS techniques. The ages were consistently clustered around Superior craton accretion ages. Although 6 zircon grains featured metamorphic rims (Figs. 3, 4 below), the average $^{207}Pb/^{206}Pb$ rim ages were just marginally younger (average of 2563 ± 23 Ma) than their core ages (average of 2657 ± 23 Ma). Individual sample $^{207}Pb/^{206}Pb$ ages and results are summarized in Figure 4.

Interestingly, of the 10 youngest ages of all zircons (from 2341 ± 41 Ma to 2509 ± 32 Ma), only one was a metamorphic rim. The other 9 youngest samples were all igneous cores, indicating a prolonged zircon growth window. The lone young metamorphic rim age (2434 ± 40 Ma in 14-VK-06) could be related to isobaric granulite-facies metamorphism as suggested by Moser and Heaman (1997), though Attawapiskat may be too far north to be related to the same Matachewan ocean opening event. The fact that metamorphic rim ages all fall well within the range of igneous growths, even within the same samples, suggests that the young Superior craton featured a prolonged window of suitable conditions for zircon growth and that observed metamorphic rims may be the result of ongoing igneous/orogenic events.

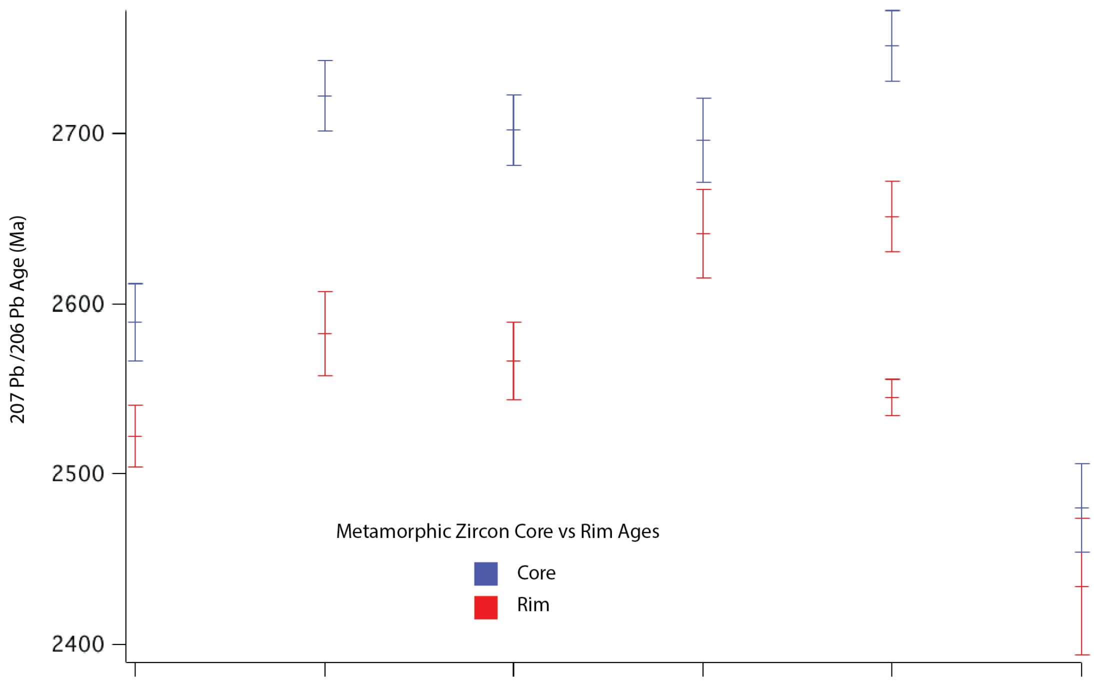


Figure 3 shows a comparison of metamorphic rim to core ages from the 6 individual zircons that were observed to have metamorphic rims by CL imaging (CL images can be found in Fig.4). Metamorphic rims were not the youngest samples analyzed.

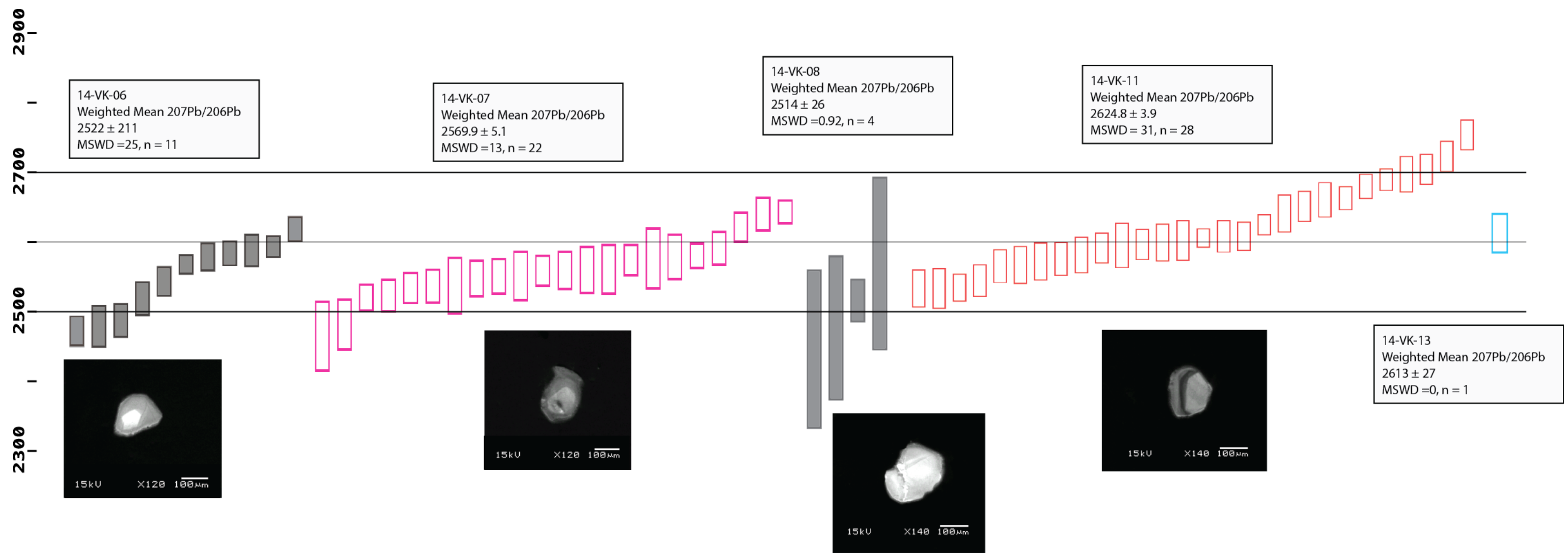


Figure 4 shows the combined 207 Pb / 206Pb ages of all 5 zircon-bearing samples as a function of MWSD. Grey samples are granulites, white boxes denote amphibolites. CL images show representative metamorphic rim/core combinations that are plotted in Fig. 3. Only 6 zircon grains displayed metamorphic textures.

U-Pb Rutile

The six rutile-bearing samples comprised 13 acceptable LA-ICPMS data points that span a far greater range of apparent ages than zircon. The oldest apparent rutile $^{206}Pb/^{238}U$ ages are consistent with zircon U-Pb crystallization ages at ~2660 Ma. These oldest dates are observed in granulites and reflect rapid cooling through rutile Pb-closure. Additional samples record a continuous range of dates, from 2.6 Ga to the ages that overlap within uncertainty of the kimberlite eruption ~320 Ma recorded in deep crustal eclogites. These data are tabulated below in Table 1 and schematically illustrated in Figure 5.

Sample	Lithology	206/238 Age	2 SE
14_VK_06_1	Granulite	2660	140
14_VK_06_2	Granulite	1029	57
14_VK_06_3	Granulite	748	88
14_VK_08_3	Granulite	1330	470
14_VK_11_1	Amphibolite	1634	62
14_VK_11_2	Amphibolite	1798	55
14_VK_11_3	Amphibolite	1786	56
14_VK_17_1	Shallow Granulite	1660	160
14_VK_17_2	Shallow Granulite	1490	120
14_VK_17_3	Shallow Granulite	1650	77
14_VK_22_1	Eclogite	324	17
14_VK_22_2	Eclogite	334	19
14_VK_22_3	Eclogite	452	43

Table 1 lists each rutile sample with corresponding lithology, $^{206}Pb/^{238}U$ age, and uncertainty.

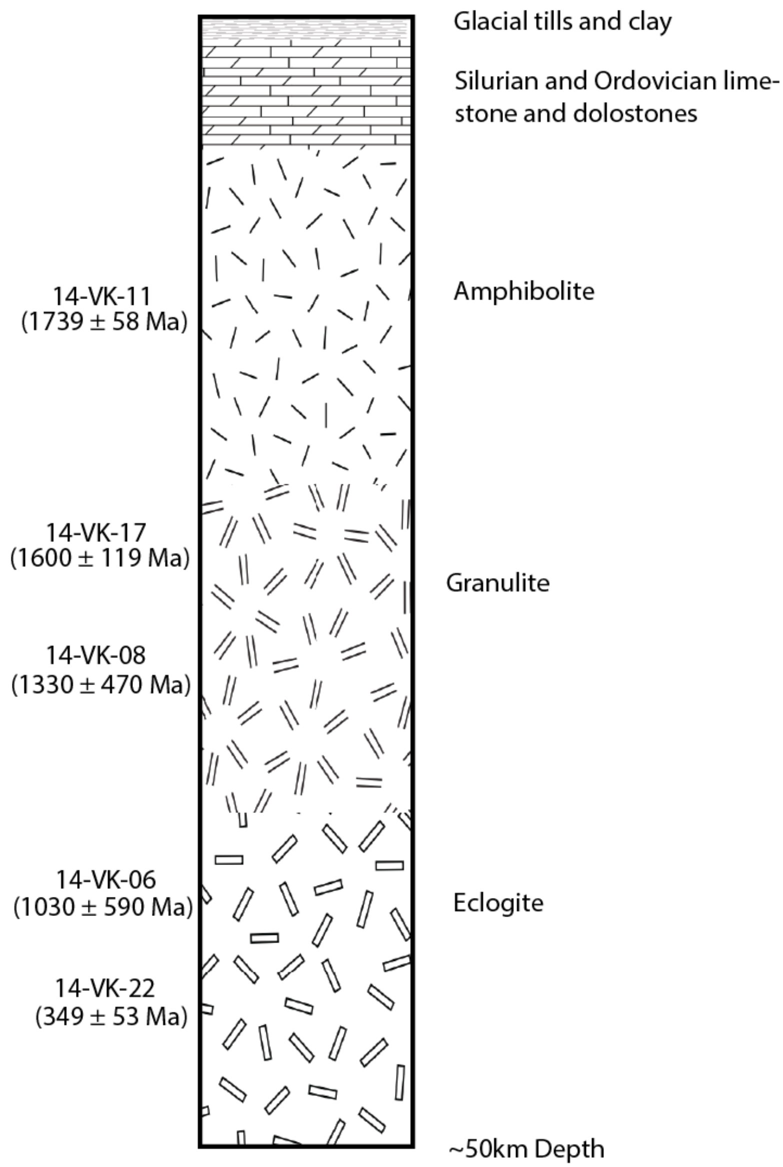


Figure 5 shows a stylized (not to scale) representation of the Superior craton crust as indicated by average rutile U-Pb dates and lithology. Individual ages can be found in Table 1 above.

Unlike the zircon samples, the rutile data is largely affected by high uncertainties and extreme discordance. Future TIMS dating work should help resolve these high uncertainties and allow for higher resolution analyses and model bracketing.

Thermal and Pb Diffusion Models

The thermal evolution model predicts long-term geothermal evolution through conductive cooling. Figure 6 shows different stages of modeled geothermal time-temperature relaxation from a hot, shallow, orogenic geotherm (e.g. Superior craton at ~2.7 Ga) down to the lower, steep modern cratonic geotherm indicated by seismic velocity studies (surface heat flux) (Percival et al., 2006; Yuan and Romanowicz, 2010). The model can predict the geothermal response to a potential heating event (Fig. 7) and subsequent relaxation at any number of input parameters.

Thermal evolution and diamond nitrogen aggregation models show that temperature is the key factor in IaA to IaB transition through time. Though extended residence time certainly can play a role in maturation to IaB, a +15° C change in temperature can achieve the same effect as 1.2 billion years' additional mantle residence time (Fig. 2). This high sensitivity to slight changes in temperature can be observed at all modeled temperatures and initial nitrogen concentrations. The model also indicates that the deep crustal geotherm can easily rise above the diamond stability field domain with a mantle intrusion event (~1350° C) at 1.1 Ga and then conductively cool back to diamond stability temperatures before potential kimberlite eruption in the Jurassic. The few diamonds that could survive the thermal pulse on the upper limit of the diamond stability field temperatures would then be highly aggregated IaB type.

Importantly, the model also predicts the U-Pb concordia evolution for any given thermal history. Here we propose two end member events to constrain the nitrogen aggregation states observed by Smit et al. (2014): one in which the the

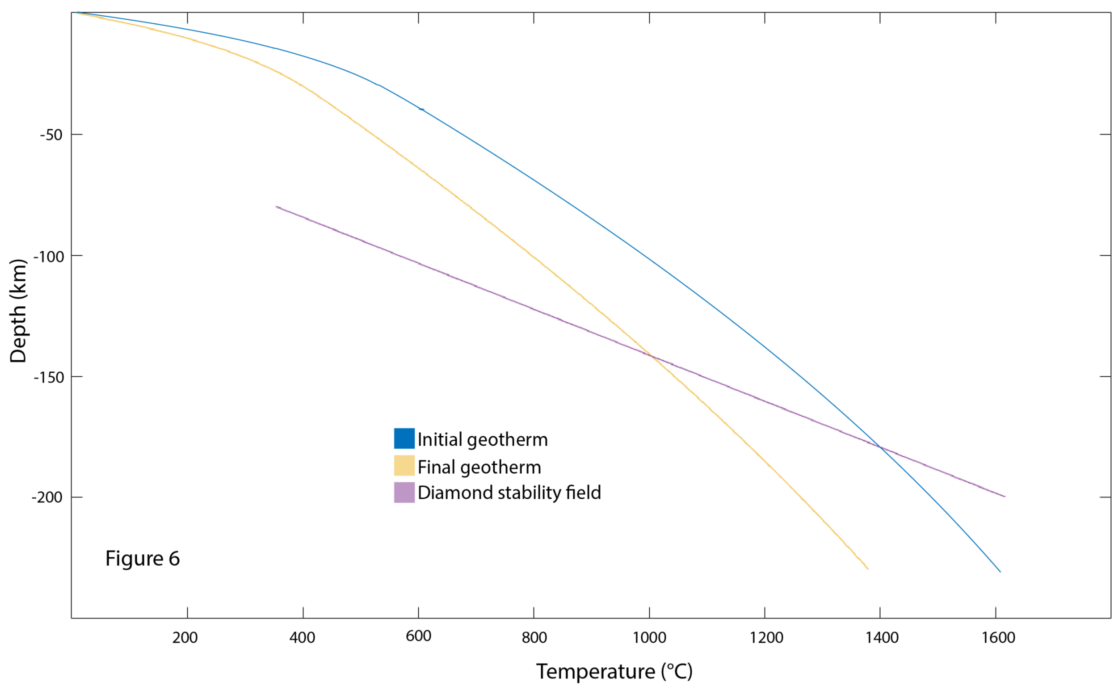


Figure 6 above shows examples of a modeled geotherm relaxing through time from hot, orogenic (blue) to the cool, modern cratonic geotherm (yellow) all in relation to the diamond stability field (purple).

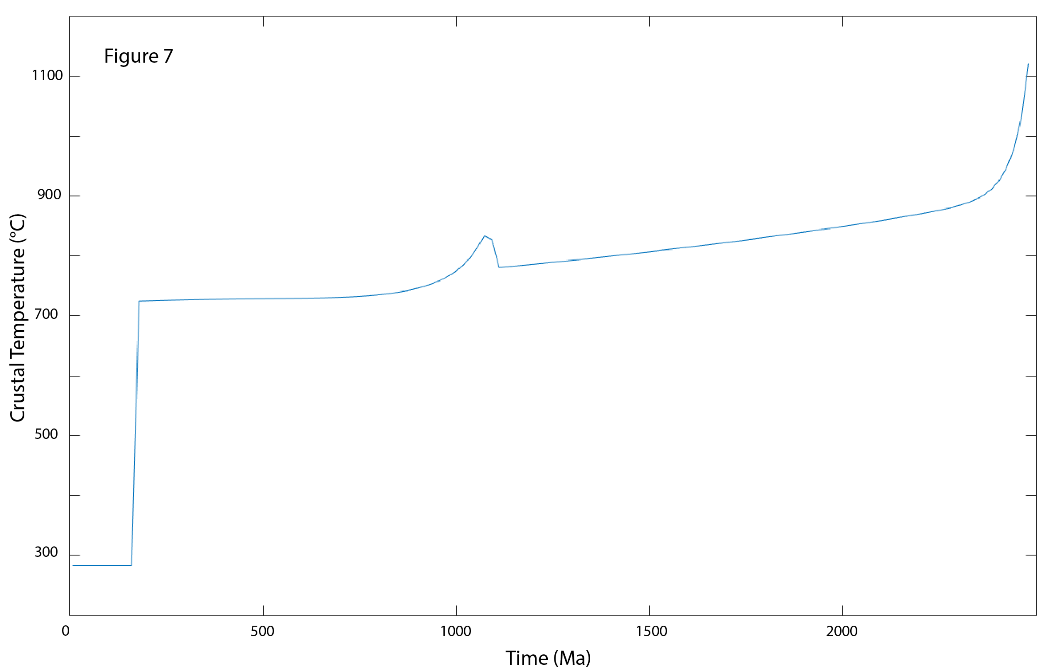
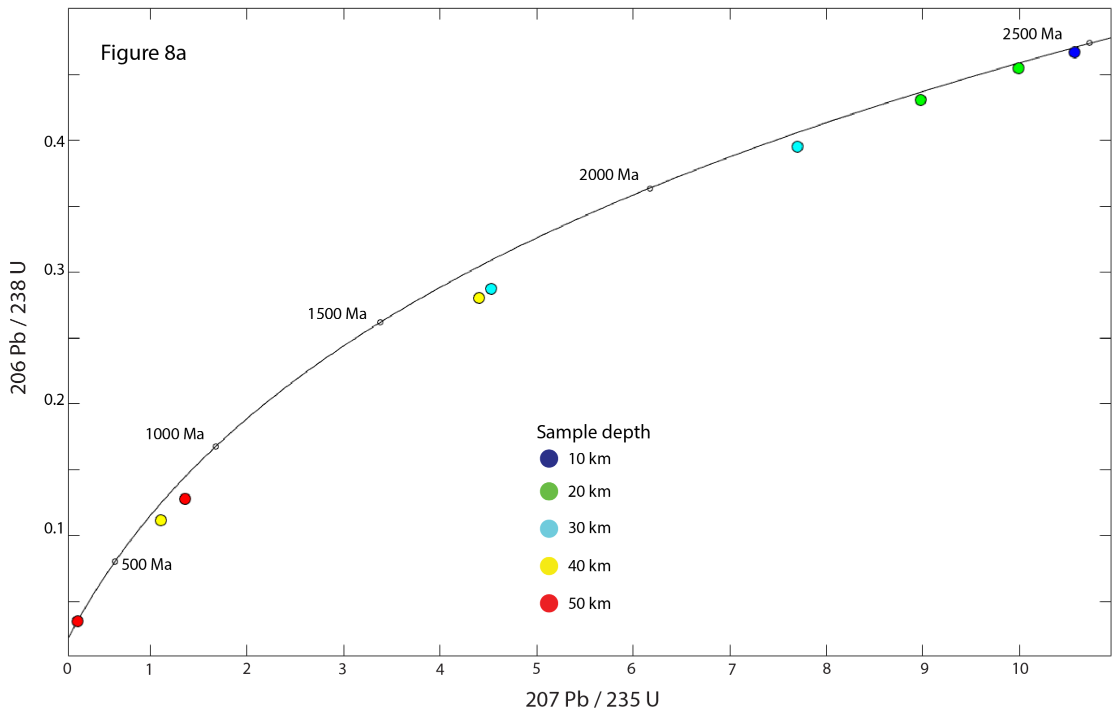


Figure 7 is an example of a mid-crust response to a reheating event at 1.1 Ga.



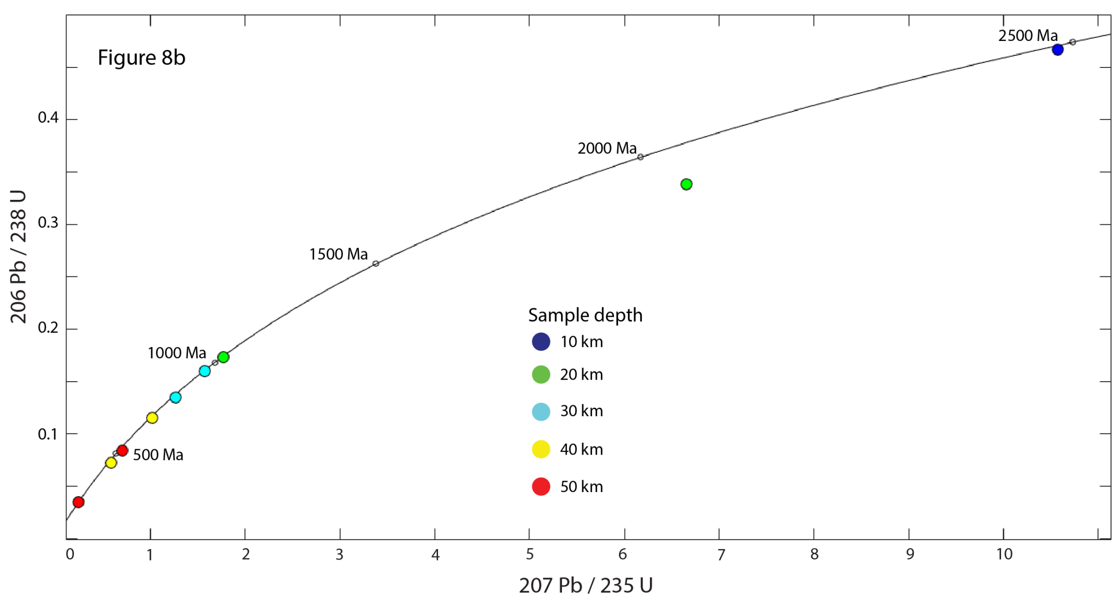


Figure 8 Rutile U-Pb thermochronologic results predicted for: (A) a continuous cooling history following 2500 Ma crystallization (Blackburn et al., 2012) and (B) cooling followed by reheating at 1.1 Ga.

geotherm is conductively cooled, and one in which the lithosphere is affected by a reheating event at 1.1 Ga and otherwise allowed to conductively cool afterwards. Figure 8a shows rutile U-Pb apparent ages predicted by a scenario in which no reheating occurs. A pervasive reheating event (Fig. 8b) predicts Pb loss (open system) in the deeper samples while shallower, faster-cooling samples retain some information as to their original cooling history. The deepest samples only record cooling after the thermal event at 1.1 Ga. Subsequent conductive cooling after the thermal pulse event eventually closes the Pb loss from the deeper samples, resulting in a new cooling path that slowly moves down concordia until intersecting the age of kimberlite eruption which closes Pb loss in even the deepest samples.

Both models predict that mantle temperatures were likely substantial enough to highly aggregate (high %B) type IaB diamonds by 1.1 Ga (as observed in Kyle Lake) and beyond, which is not observed in Victor.

The modeled heating event was further subdivided into two scenarios: one in which the thermal pulse was just strong enough to heat the mantle to the edge of diamond stability and one in which the crust is also heated. Heating the mantle geotherm to the edge of diamond stability does not result in U-Pb apparent ages significantly different than the unperturbed geotherm model.

Discussion

Coupled together, the diamond nitrogen aggregation, zircon U-Pb, and rutile U-Pb data offer a unique insight into the thermal evolution of the Superior craton geotherm within both maximum and minimum constraints. The old U-Pb ages given by the zircon data indicate that at ~2.7-2.5 Ga the Superior craton at Attawapiskat experienced a prolonged period of igneous crystallization and/or overprinting by rifting (Moser and Heaman, 1997). The relatively few metamorphic textures observed in the zircon samples (and the few metamorphic rims that were observed are all >2.4 Ga) (see Figs. 3, 4) suggest that any potential thermal event (at 1.1 Ga or any other time) was not responsible for expansive magmatism throughout the crust. The fact that not a single significantly younger rim was observed further suggests that the deep crust (~50 km) did not experience magmatism or fluid infiltration at 1.1 Ga.

The presence of Proterozoic diamonds at Kyle Lake (erupted at roughly 1.1 Ga) indicates that the hot orogenic geotherm cooled enough from ~2.5 Ga to 1.1 Ga to enter the diamond stability field. The high concentration of thermally mature IaB diamonds suggests that the geotherm was within the diamond stability field and further, that these diamonds formed at high temperatures at greater than 150 km depths within the lithosphere. A low geotherm prior to 1.1 Ga rifting is supported by rutile U-Pb apparent ages as old as 2.6 Ga. These data record cooling at least to the onset of Pb retention in rutile (~650° C) at mid crustal depths (~20-30 km) between 2.5 Ga and 1.1 Ga.

Evidence for a thermal pulse and corresponding elevated geotherm around 1.1 Ga is supported by the diamond nitrogen aggregation states of Kyle Lake and Attawapiskat. The close proximity of Kyle Lake to Attawapiskat (~100 km) suggests the same or a very proximal source of diamond. The highly aggregated IaB type diamonds of Kyle Lake would indicate that all diamonds in the mantle keel near the study area were highly aggregated by 1.1 Ga. In order to erupt non-aggregated type

IaA diamonds at Attawapiskat in the Jurassic, the vast majority of diamonds in the mantle keel would have to be converted to graphite to reset the aggregation state. As drastic changes in mantle depth/pressure are unlikely, this must be achieved by a geotherm elevated to the very upper limit of the diamond stability window, but not too far out of it because at least two highly aggregated diamond samples survived.

Although there was indeed a diamond-destroying thermal pulse at ~1.1 Ga, the heating event was evidently insufficient in magnitude to drive total Pb loss from rutiles within shallow, fast cooling samples. The retention of Archean rutile U-Pb apparent ages, and telling lack of concentrated ages at or younger than 1.1 Ga, indicates the the thermal pulse was not long-lived.

After the thermal pulse around 1.1 Ga, the geotherm then relaxed back into the diamond stability window where the Attawapiskat diamonds revert back to diamond from graphite in a major Neoproterozoic diamond-forming event. This post-1.1 Ga relaxation is evidenced by the low aggregation state of the mostly type IaA diamonds at Attawapiskat, indicating lower temperatures and/or short residence time. Proterozoic to Mesozoic rutile ages further support continued cooling and geotherm relaxation after the thermal event. Cooling then continued through to kimberlite eruption and beyond, as supported by modern surface heat flux data and seismic velocity models that indicate the cool cratonic geotherm of present.

Together the data tell a story of a geotherm that starts off hot and shallow at the time of Superior craton accretion and amalgamation at 2.7-2.5 Ga that then cools and relaxes into the diamond stability field before 1.1 Ga, is then heated to the upper temperature limit of diamond stability around 1.1 Ga, and then cools back through the diamond stability window to the modern cool, deep geotherm observed today.

Conclusions

The Attawapiskat area of the Superior craton experienced conductive cooling from ~2.5 Ga to ~1.1 Ga that led to a major diamond forming event as the geotherm relaxed into the diamond stability field. Diamond nitrogen aggregation state differences between the highly aggregated (mature) IaB-type Proterozoic Kyle Lake kimberlites and the Jurassic IaA (immature) Attawapiskat kimberlites suggest a large-scale diamond-destroying thermal pulse penetrated the deep lithosphere at ~1.1 Ga, converting nearly all diamonds to graphite. Zircon and rutile U-Pb data show that this thermal pulse did not cause magmatism or high-temperature metamorphism in the crust. Indeed, heating was insufficient to fully reset the pre-rifting cooling history of shallow crustal amphibolites as recorded by rutile U-Pb data. Continued cooling and geothermal relaxation led to a second major diamond forming event in the Neoproterozoic. These poorly aggregated (type IaA) diamonds were then erupted in the Late Jurassic at Attawapiskat, where the geotherm continued cooling to its present cool cratonic state observed today.

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