



## KIMBERLITE-DERIVED HARZBUGITIC DIAMONDS FROM A >2.7 GA SOUTHERN SUPERIOR PROTOCRATON

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### Introduction

Current models of the Superior craton growth invoke its assembly from off-cratonic continental and oceanic terranes and the formation of a cold diamondiferous root soon after the 2.7 Ga orogeny that consolidated the craton. We show that the Superior craton included an older cratonic nucleus, which had developed the deep, cold, harzburgitic diamondiferous root prior to 2.7 Ga. We also provide evidence for a >2.7 Ga Southern Superior kimberlite that was eroded within 3 Ma. Comparison of thicknesses and thermal states of the diamondiferous cratonic root as it evolved through time demonstrates how the root vanished by the Proterozoic.

### Diamondiferous metaconglomerate

We studied diamondiferous metconglomerate in a successor basin in the Michipicoten Greenstone Belt (MGB) of the Wawa-Abitibi Terrane (S. Superior Craton). The metaconglomerate (~170 m thick) is matrix- or clast-supported and poorly sorted, with clast sizes from <1 mm to boulders. The greenschist metamorphic assemblage completely replaced the original mineralogy. The matrix that makes up 20-70% of the metaconglomerate is composed of a fine-grained foliated aggregate of quartz, albite, carbonate, chlorite, muscovite, apatite, biotite, epidote, sphene, spinel, rutile and Fe sulfide. The secondary mineralogy, however, reflects the original bulk composition and is distinct in various metaconglomerate beds and lenses. Petrographic observations on outcrop and drill-core metaconglomerate samples classified lithic clasts into several types, i. e. mafic to felsic volcanic clasts with various textures, mafic to felsic hypabyssal clasts, granitoid clasts and unidentified mafic or ultramafic rocks replaced by chlorite and serpentine. The metaconglomerate includes diamonds and paragenetic diamond indicator minerals. It is interpreted to form in an alluvial fan – delta succession in a shallow marine-delta environment (Wendland, 2009).

Four samples of metaconglomerate were dated using the U-Pb TIMS technique on zircons. U-Pb ages tightly constrain the timing of the metaconglomerate formation. Granite and gabbro clasts in the metaconglomerate are

dated at  $2700.4 \pm 1.1$  and  $2701.0 \pm 1.2$  Ma, respectively. The minimum limit on the metaconglomerate age is that of the cross-cutting dyke,  $2697.2 \pm 1.8$  Ma. The diamondiferous conglomerate thus formed between 2700 and 2697 Ma, being contemporaneous and intercalated with volcanic Cycle 3 of MGB (~2.7 Ga). The barren metaconglomerate 10 km to the west, however, is younger as it contains granite clasts with ages of  $2693.7 \pm 1.5$  Ma.

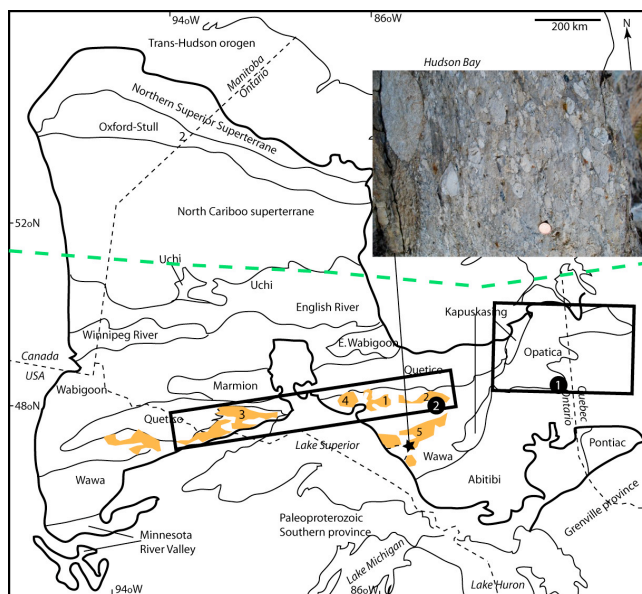


Fig. 1. Regional map of tectonic terranes in the Superior craton (Percival et al., 2006). Shaded yellow regions correspond to greenstone belts in the Wawa-Abitibi terrane. Rectangles delineate areas identified as possible sources for the metaconglomerate detritus. Thick dashed line shows the southern border of the high-velocity cratonic root in the diamond stability field (Faure et al., 2011). Solid dots are locations of post-Archean kimberlites in the vicinity of Wawa: 1) Kirkland Lake (Vicker, 1997), 2) Wawa (Kaminsky et al., 2002). The inset shows a typical outcrop of the metaconglomerate.

### Other Archean diamondiferous rocks in Wawa

To uncover primary sources for diamonds in the metaconglomerate, one should map primary diamondiferous



volcanics in the area. Nine kilometers to the north-northwest from the metaconglomerate there are outcrops of metamorphosed diamondiferous polymict volcanoclastic breccia and associated dykes of calc-alkaline lamprophyric composition. These 2724 – 2620 Ma breccias occur in 60–110 m conformable beds traceable along strike for more than 4 km and occupying several stratigraphic levels (Lefebvre et al. 2005). The lamprophyric metabreccias are invariably rich in actinolite, which is very rare in metaconglomerate thin sections and among heavy minerals. This attests to an insignificant contribution of the lamprophyric detritus and to the less mafic total composition of detrital metaconglomerate components. Importantly, lamprophyres are absent among lithic clasts of the diamond-bearing metaconglomerate.

## Diamonds

We studied 353 diamonds ranging in size from 0.8 to 2 mm (macrodiamonds) and 31 stones smaller than 0.8 mm (microdiamonds) extracted from metaconglomerate. The diamonds are octahedral or resorbed octahedral (80%), cubic (14%) and octahedral with a cubic overgrowth (6%). A considerable portion of the diamonds (43%) are unresorbed, while the remainder shows varying degrees of resorption. Ninety three per cent of the diamonds are single crystals, and the remaining are polycrystalline aggregates and twins. Among the macrodiamonds, white crystals are predominant (64%), followed by crystals with a yellow tint (26%) and grey, green, brown or black cuboids. The diamonds were found to contain < 820 ppm N and widely varying contents of totally aggregated N. Carbon isotope ratios of the diamonds vary from -4.0 to -2.5‰  $\delta^{13}\text{C}$ . The diamonds have an unusual green-yellow and red luminescence possibly related to  $\beta$ -radiation followed by metamorphic annealing (Bruce et al. 2011).

The characteristics of the metaconglomerate diamonds can be compared to diamonds (80 crystals 0.1–2 mm) in Wawa metabreccia (De Stefano et al. 2006). The metabreccia diamonds are heavily dominated by microstones whereas metaconglomerate diamonds are coarser. The metaconglomerate diamonds are significantly less resorbed and contain less aggregated N, with 42% of the suite being Type IaA stones. Metaconglomerate diamonds also had a wider variety of colours that are not seen in the metabreccia diamonds, including green and pink. The two suites of Wawa diamonds, based on size distribution, morphology and nitrogen studies, are deemed to be different. We therefore suggest that the diamonds in the metaconglomerate may not be sourced from the diamondiferous lamprophyres that brought up metabreccia diamonds.

## Heavy minerals in metaconglomerate

The mineralogy of the heavy mineral concentrates was studied for 6 samples panned from outcrops of the metaconglomerate and for 5–70 ton samples of metaconglomerate processed in commercial labs. The extracted heavy minerals include olivine, clinopyroxene,

chromite, corundum (ruby and sapphire), magnetite, pyrope, ilmenite, rutile, almandine, sapphirine, sphene, quartz, anorthite, hornblende, actinolite, epidote and chlorite. The minerals occur in very low abundances (several grains per 6–80 ton samples). In thin sections, only relics of chromite and ilmenite were found; they are partly replaced by thick fine-grained mantles of metamorphic minerals. The mantle on pyrope looks fibrous, radially-grown, and may be a metamorphosed kelyphitic rim. The majority of ilmenite and pyrope mineral grains are not abraded, matching the absence of mechanical wear on the metaconglomerate diamonds. Below we determine the provenance of heavy minerals based on their compositions.

**Clinopyroxene** varies from Cr-diopside to jadeite- and tschermakite-rich clinopyroxene. A considerable part of metaconglomerate clinopyroxene is low-Mg, low-Al clinopyroxenes from mafic rocks. The rest is chromian clinopyroxene identical to clinopyroxene from garnet-free Archean mafic-ultramafic complexes. The latter occur in both 2.75 Ga and 2.7 Ga MGB assemblages and in adjacent greenstone belts of the Wawa-Abitibi Terrane (Williams et al. 1991)

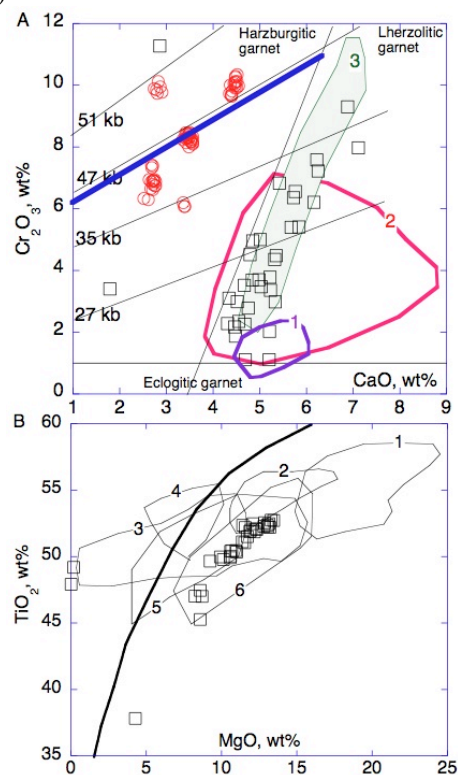


Fig. 2. Compositions of pyrope (A) and picroilmenite (B) in heavy mineral concentrates of Wawa metaconglomerate (open squares). Also shown on A: pyrope in inclusions in the metaconglomerate diamond (red open circles); isobars for a 41 mW/m<sup>2</sup> geotherm and the Graphite-Diamond constraint (thick blue line) of Grutter et al. (2006); garnet in the Phanerozoic mantle (field 1), peridotitic garnet in Wawa kimberlite (field 2; Kaminsky et al., 2002), garnet from peridotite xenoliths in Kirkland Lake kimberlite (Vicker, 1997). Also shown in B: the threshold between kimberlitic and non-kimberlitic ilmenite (thick black line) (Wyatt et al. 2004), fields for ilmenite in kimberlites (1, 2, 6) and ultramafic lamprophyres (3–5) (references in Kopylova et al., in print).



**Pyrope** is lherzolitic, with the Cr<sub>2</sub>O<sub>3</sub> content up to 11 wt% (Fig. 2A). Such high-Cr pyrope occurs only in Proterozoic or Archean continental mantle, i.e. under cratons. Lherzolitic pyrope demonstrates decreasing Cr content with decreasing crustal age because of the “secular and irreversible changes in the composition of the newly-created lithospheric mantle throughout the Earth’s history” (Griffin et al. 1998). Pyrope of the Phanerozoic mantle, i.e. in garnet-bearing mantle xenoliths emplaced by volcanics into the Phanerozoic crust, commonly has no more than 2.6 wt% Cr<sub>2</sub>O<sub>3</sub> (Fig. 2A).

**Picroilmenite** compositions form a tight trend on the “kimberlite” side of the line that separates “non-kimberlitic” from “kimberlitic” ilmenites (Fig. 2B). Picroilmenite in various types of ultramafic lamprophyres (e.g. aillikite and alnoite) on the MgO-TiO<sub>2</sub> plot (Fig. 2B) shows the minimal MgO contents of 4 wt%. Manganese-rich ilmenite, like the one in Greenland aillikites, demonstrates an even lower MgO content (field 3 on Fig. 2B). In contrast, all fields for picroilmenite in kimberlites are shifted to higher MgO contents, with the minimal MgO equal or higher than 8 wt%. The high MgO concentrations in the metaconglomerate picroilmenite and the absence of grains with MgO content between 4 and 8 wt% suggest that the picroilmenite was sourced from a kimberlite. The Wawa metaconglomerate, like the Witwatersrand conglomerate, thus record indirect evidence for an Archean kimberlite.

**Olivine and chromite.** Forsterite (Fo<sub>91-93</sub>, 0.36-0.43 wt% NiO) and chromite (0.4-2.7 wt% TiO<sub>2</sub>, 42-62% wt% Cr<sub>2</sub>O<sub>3</sub>) were extracted from the metaconglomerate. These minerals cannot be kimberlite indicators. Magnesian olivine occurs in all ultramafic rocks. Likewise, Cr- and Mg-rich chromites with relatively low Ti content (< 2 wt% TiO<sub>2</sub>) are common in all primitive mantle magmas (Barnes and Roeder, 2001). Most importantly, these non-kimberlitic chromites from ultramafic massifs can be found as large grains identical in size to kimberlite macrocrysts and therefore potentially extracted as indicators (Afanasyev et al. 2010). Numerous occurrences of Archean mafic and ultramafic rocks that may be suitable hosts for the metaconglomerate olivine and chromite are mapped in the Wawa and Abitibi terranes (Williams et al. 1991).

**Almandine.** Pink low-Ca almandine with 1-4 wt% MnO, as typical of metamorphic garnets, is found in the metaconglomerate. The almandine compositions are broadly comparable with those from metapelites and felsic-intermediate igneous rocks metamorphosed in the granulite and amphibolite facies. Among these, amphibolite facies source rocks are favoured by the presence of hornblende in the heavy mineral concentrates. Mafic granulites and amphibolites are rejected as a viable host rocks as their almandine is more calcic. The most likely parent rock for the metaconglomerate almandine is therefore quartzofeldspathic, metamorphosed in the amphibolite facies.

**Anorthite.** Plagioclase in the heavy concentrate of the metaconglomerate is An<sub>90-100</sub>. Such Ca-rich plagioclase cannot be metamorphic as the latter is more sodic

(references in Kopylova et al., in print). Anorthite cannot be sourced from peridotite (An<sub><90</sub>) or massif-type and oceanic anorthosites (An<sub>50-70</sub>). Pure anorthite can be found only in certain types of Archean anorthosites, so-called basement-type anorthosites and megacrystic and layered mafic intrusion-associated anorthosites (Ashwal, 2010). Multiple Archean basement-type anorthosites are mapped in greenstone belts of the Wawa-Abitibi terrane (Williams et al. 1991).

### Provenance of sedimentary material in metaconglomerate

Three major sources contributed detritus to the diamondiferous metaconglomerate. The first, vastly predominant, is the ~2.7 Ga igneous rocks of the Cycle 3 MGB assemblage. They comprise all lithic clasts, i.e. 30-80% of the metaconglomerate and abundant quartz and plagioclase in fine-grained matrix. Clinopyroxene, olivine, chromite, anorthite and possibly corundum are likely to have also been sourced from Wawa-Abitibi terrane’s mafic layered intrusive complexes that include peridotite, gabbro and anorthosite. The second, very minor (<< 1%) component of the metaconglomerate detritus is medium grade metamorphic minerals. It is not possible to see them in thin sections, but panning of about 10 kg of conglomerate yields metamorphic hornblende and almandine. The third, the least volumetrically significant component is kimberlitic, including diamond, picroilmenite, pyrope and possibly corundum. The most abundant of these minerals is diamond, as it was panned from 10 kg samples. The rest of the minerals have never been found in panned concentrates or thin sections, and only processing of ~ 5-80 ton samples yields 0-10 grains of the minerals. This component was also sourced outside of the MGB, but shapes and the assemblage of kimberlite indicator minerals cannot constrain their transport distance within the maximum limit. The degree of mechanical abrasion of the indicators is mainly controlled by the dynamic conditions of transport and deposition and is not a function of the source remoteness (Afanasyev et al. 2010). Alluvial transport for hundreds of kilometers in a platform environment does not abrade indicators.

Our geochronological data attest to the tight localization of the diamondiferous metaconglomerate in time (2701-2697 Ma) and space. The younger, <2694 Ma metaconglomerate 10 km to the west and sandstones at the diamondiferous metaconglomerate location on higher stratigraphic levels are barren. We explain it by a quick (~3 Ma) erosion of the kimberlite body. This process contributes to the scarcity of Archean kimberlite outcrops.

### A pre-2.7 Ga Superior protocraton

Detrital grains of high-Cr pyrope and diamonds in the metaconglomerate suggests the presence of diamondiferous cratonic root under one of the pre-2.7 Ga continental blocks that later amalgamated to form the Superior. The protocraton’s location can be inferred from the provenance of the metaconglomerate detrital material. The older





protocraton nucleus should meet several criteria. Firstly, it must contain amphibolite facies rocks with ages  $>2.7$  Ga, as inferred from the presence of detrital almandine and hornblende. Secondly, the protocraton cannot be too distal from MGB ( $< 1000$  km) or be separated from MGB by ocean to ensure the preservation of possible kelyphitic rims on detrital pyrope. Thirdly, the protocraton should be situated north of MGB on the same continent to match general southerly transport direction of detritus as inferred from independent provenance studies. The two terranes that remain viable candidates for the source of metamorphic lithic clasts are the Opatca terrane (500 km to the northeast) or the Hemlo - Manitouwadge areas of Wawa-Abitibi (150 km to the north) (Fig. 1).

## Mineral inclusions in diamonds

Analysis of 173 mineral inclusions in 46 diamonds from the metaconglomerate has yielded four main mineral phases, pyrope garnet, Mg-chromite, olivine ( $\text{Fo}_{93}$ ), and orthopyroxene ( $\text{En}_{93-95}$ ). Chromite is the most abundant inclusion, followed by the chromite+olivine assemblage. Garnet is classified as harzburgitic pyrope (Fig. 2A). Chromite contains between 60.4 and 69.0 wt%  $\text{Cr}_2\text{O}_3$  and medium to high MgO (12.8-15.3 wt%), placing it within the diamond inclusion field. Even though the majority of studied diamond inclusions (DIs) are not in contact with each other, the phases are well equilibrated. This is evidenced by 1) the higher Mg# of orthopyroxene than that of olivine typical of peridotite minerals in equilibrium (Brey and Kohler, 1990); 2) low Al content of orthopyroxene typical of garnet peridotite (Boyd et al., 1997); 3) high Fe content of chromite characteristic of this phase in garnet peridotite (Boyd et al., 1997). These facts suggest that all minerals found as metaconglomerate DIs originated in the garnet-bearing facies of harzburgite, i.e. in the spinel-garnet and garnet-only peridotite. Garnets from spinel-free garnet harzburgites on the  $\text{Cr}_2\text{O}_3$ -CaO diagram (Fig. 2A) plot below the Graphite-Diamond Constraint indicating the underestimated pressure due to lack of equilibrium with spinel.

Temperature estimates for mineral pairs coexisting in single diamonds were calculated using the Fe-Mg exchange between garnet and olivine (O'Neill and Wood, 1979, Fig. 3). The requirement that the olivine-garnet temperatures fall within the diamond stability field constrains the highest possible heat flow at  $41 \text{ mW/m}^2$  (Fig. 3). The diamonds occur at a depth interval from 47 kb,  $1020^\circ\text{C}$  to 62 kb,  $1260^\circ\text{C}$ , as implied by the intersection of this model geotherm with univariant garnet-olivine P-T lines. The maximal pressure and temperatures correspond to the minimum depth of the lithosphere-asthenosphere boundary at approximately 190 km (Fig. 3). The highest viable geotherm of  $41 \text{ mW/m}^2$  is also matched by invariant estimates for pressures and temperatures that were done for a diamond containing orthopyroxene, garnet and olivine. Two points computed for two garnet compositions inside a single diamond fall on this geotherm (Fig. 3).

The metaconglomerate DIs may have formed at a thermal regime colder than  $41 \text{ mW/m}^2$ . The temperatures obtained for Wawa DIs may be overestimated by  $\sim 100^\circ\text{C}$ , since we based thermobarometric calculations on analyses of non-touching DI pairs (Phillips et al., 2004). If a different orthopyroxene-garnet barometer (Nickel and Green, 1985) is used for estimates of the P-T invariant points, they would fall on the  $39\text{-}40 \text{ mW/m}^2$  model geotherm. Moreover, the lower thermal regime would better match the abundance of DI chromite. A possible  $39\text{-}40 \text{ mW/m}^2$  regime would ensure that numerous chromite-bearing diamonds also plot in the diamond stability field.

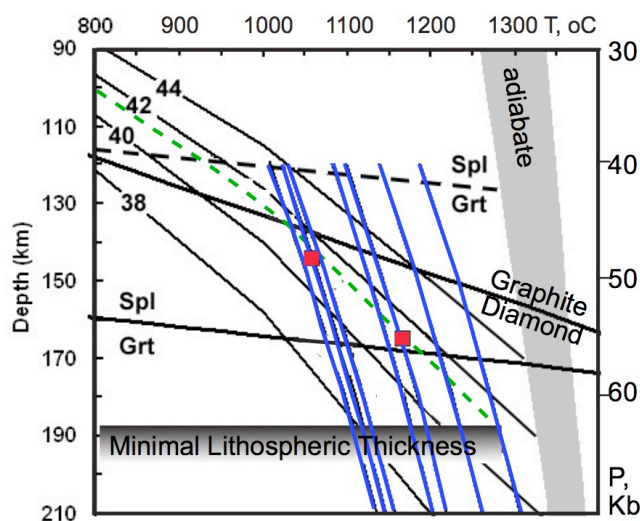


Fig. 3. Pressure-temperature diagram for metaconglomerate diamond inclusions. Graphite-diamond transition is from Kennedy and Kennedy (1976); model geotherms labeled with heat flow values in  $\text{mW/m}^2$  are from Pollack and Chapman (1977). Thick blue lines represent univariant P-T lines (O'Neill and Wood, 1979) for garnet-olivine DI pairs. Dashed line is the highest possible model geotherm that would plot diamonds with olivine-garnet inclusions in the diamond stability field. Grey field represents a range of mantle adiabates from Rudnick et al. (1998). The spinel-garnet transition line (Girnis and Brey, 1999) is calculated using average  $\text{Cr}\#$  for DI chromite and garnet, extrapolated to lower and higher temperatures using the pressure-temperature gradient of O'Neill (1981). A dashed line of spinel to garnet transition is computed based on barometer of O'Neill (1981). Solid squares are P-T estimates for a single diamond containing Opx+Ol+Chr using O'Neill and Wood's (1979) thermometer and Brey and Kohler's (1990) Al-in-orthopyroxene barometer.

## Temporal evolution of the thermal regime and the cratonic root below Southern Superior

The mineral chemistry, thermobarometry of DI and mantle values of  $\delta^{13}\text{C}$  in the diamond carbon in Wawa metaconglomerate is evidence for the harzburgitic deep cratonic root below a Southern Superior protocraton before 2.7 Ga. It might be possible that eclogites and eclogitic diamonds did not exist in the Southern Superior mantle at the time, possibly because subduction in the Wawa and Opatca terranes didn't begin until  $\sim 2.7$  Ga (Williams et al., 1991).

Post-Archean kimberlites in the vicinity of Wawa are key in constraining the evolution of the root thickness and the thermal regime. Thermobarometry of mantle xenoliths



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brought by the 1.1 Ga Wawa kimberlites (Kaminsky et al., 2002) demonstrates heating of the root to 46 mW/m<sup>2</sup> by the Proterozoic (Fig. 4B). Under Opatica terrane, the Jurassic

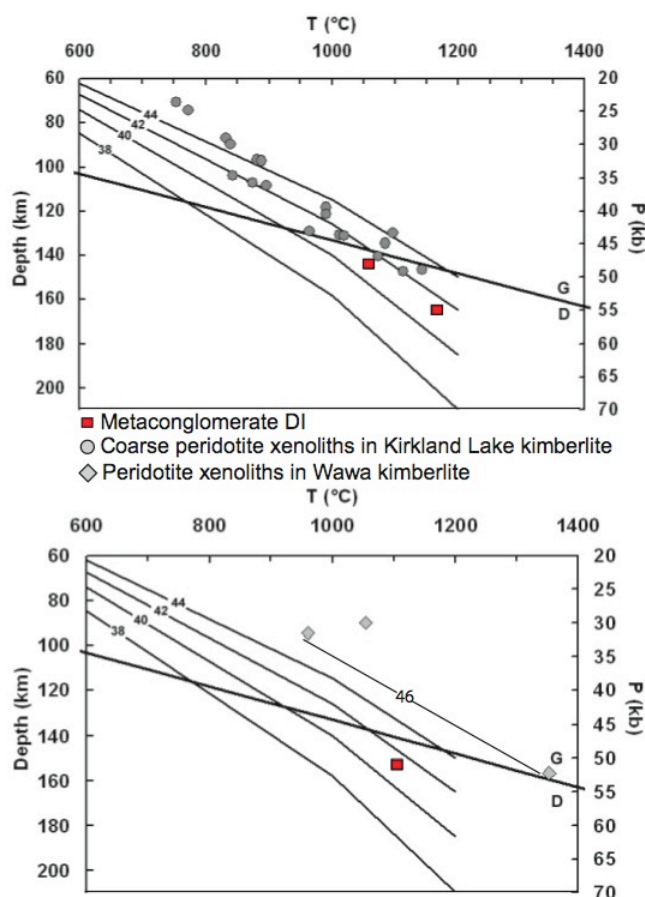


Fig. 4. Pressure-temperature diagrams for metaconglomerate DI in comparison with xenoliths in post-Archean kimberlites of the Southern Superior. A: Temperature (O'Neill and Wood, 1979) and pressure (Brey and Kohler, 1990) of coarse peridotite xenoliths of the Kirkland Lake kimberlite (Vicker, 1998). B: Temperature (Ca-in-Opx, Brey and Kohler, 1990) and pressure (Brey and Kohler, 1990) of peridotite xenoliths in the Wawa kimberlite (Kaminsky et al., 2002). Model geotherms labeled with heat flow values in mW/m<sup>2</sup> are from Pollack and Chapman (1977).

mantle was sampled by the 156 Ma Kirkland Lake kimberlites, which established its thermal state as matching 41-44 mW/m<sup>2</sup> (Fig. 4A). Both post-Archean kimberlites lack economic quantities of diamond and do not sample diamondiferous mantle (Fig. 4) suggesting destruction of the deep diamondiferous root. The heating of the mantle and apparent loss of the diamondiferous root between the Neoproterozoic and Proterozoic is further supported by the high lateral resolution seismic survey (Faure et al. 2011), which demonstrated the current absence of the cold diamondiferous root (Fig. 1). The modification of the Southern Superior root started as early as the Neoproterozoic, as Abitibi Neoproterozoic greenstone belts are visible in the mantle velocity structure patterns (Faure et al. 2011). We propose that the root was destroyed by the Neoproterozoic assembly of the Southern Superior craton in multiple

subduction zones. Our discovery of Neoproterozoic kimberlites in the Southern Superior suggest that formation of this kimberlite field may have been the first event that started the gradual destruction of the now vanished >2.7 Ga root.

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