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Targeted Geoscience Initiative 4: Contributions to the Understanding of Precambrian Lode Gold Deposits and Implications for Exploration

Insights into the timing of mineralization and metamorphism in the North Caribou greenstone belt, western Superior Province

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Insights into the timing of mineralization and metamorphism in the North Caribou greenstone belt, western Superior Province

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ABSTRACT

Secondary ion mass spectrometry (SIMS) U-Pb depth profiles of unpolished detrital zircon were used in an attempt to resolve the timing of metamorphism and mineralization in the North Caribou greenstone belt of northern Ontario, which hosts the Musselwhite gold mine. Eleven samples located 10–75 km from the mine site were collected from metasedimentary rocks adjacent to structures that may have promoted hydrothermal fluid flow. Zircon rim material is commonly characterized by decreased Th/U concentrations (up to an order of magnitude), as well as generally possessing concordant ages. Rim ages obtained via this technique range from 2788–2703 Ma, up to 250 m.y. younger than zircon cores. The timing of zircon rim recrystallization overlaps with the timing of potassic alteration in the surrounding plutons, as well as regional gold mineralization in the North Caribou Superterrane. These zircon rims may represent the timing and potential distribution of Au-bearing fluids within and regional to the Musselwhite deposit.

INTRODUCTION

The timing and nature of metamorphic and hydrothermal events is poorly understood at the Musselwhite gold mine, as well as elsewhere within in the North Caribou Greenstone Belt (NCGB) of the Western Superior Province. Peak metamorphic assemblages across the NCGB are apparently spatially correlated with positive magnetic highs, intrusive bodies, and areas of high strain (Kelly et al., 2013). Despite the relatively high-metamorphic grade of the NCGB, it has been a challenge to identify a suitable chronometer to date metamorphism and mineralization. Results of geochronology proximal to the mine site have resulted in ages that are >200 m.y. younger than major tectonic events in the region (Biczok et al., 2012; Kalbfleisch, 2012; Van Lankvelt, 2013). Biczok et al. (2012) reported a Sm-Nd garnet-whole rock age of 2.72–2.67 Ga for the ore-bearing unit at the Musselwhite mine, suggesting that this broadly resolves the timing of mineralization. In our study, we add to the growing geochronological framework of the Western Superior region by conducting U-Pb depth profiling of zircon from samples across the NCGB to determine the timing of thermal events. A coupled dissolution-reprecipitation of zircon by hydrothermal fluids has been shown to occur at the sub-micrometre to micrometer scale, resulting in rim material that has a unique geochemical and isotopic signature. Commonly, the rim possesses elevated U, Fe, and Al concentrations (e.g. Geisler et al., 2007), and the U-Pb isotopes record the timing of the event (Grove and Harrison, 1999; Carson et al., 2002; Mojzsis and Harrison, 2002; Schneider et al., 2012; Steely et al., 2014). Secondary ion mass spectrometry (SIMS) analysis has a lateral spatial resolution of <30 µm and depth resolution of 100 nm (Breeding et al., 2004; Kelly et al., 2014). By sputtering the primary beam normal to the fluid reaction front on unpolished zircon, SIMS U-Pb depth profiling is capable of detecting sub-micrometre isotopic changes within zircon grains that avoids significant domain mixing and produces geologically meaningful data, which has traditionally been overlooked. We hope to gain insight into the tectonothermal history of the NCGB with these data within the framework of existing geochronology of the Western Superior Province.

RESULTS AND DATA ANALYSIS

Sample Selection and Preparation

Eleven metasedimentary samples were collected across the NCGB (Fig. 1) to provide a broad spatial understanding of the thermal activity within the belt. Samples were specifically targeted adjacent to structures that may have fostered fluid activity, such as shear zones and intrusions. These targets were identified using a combination of metamorphic pattern observations (Breaks et al., 2001; Kelly et al., 2013), as well as the regional aeromagnetic anomalies. Metamorphic rocks that were sampled include siliciclastic sedimentary units ranging from mudstone to conglomerate, with metamorphic grades of greenschist

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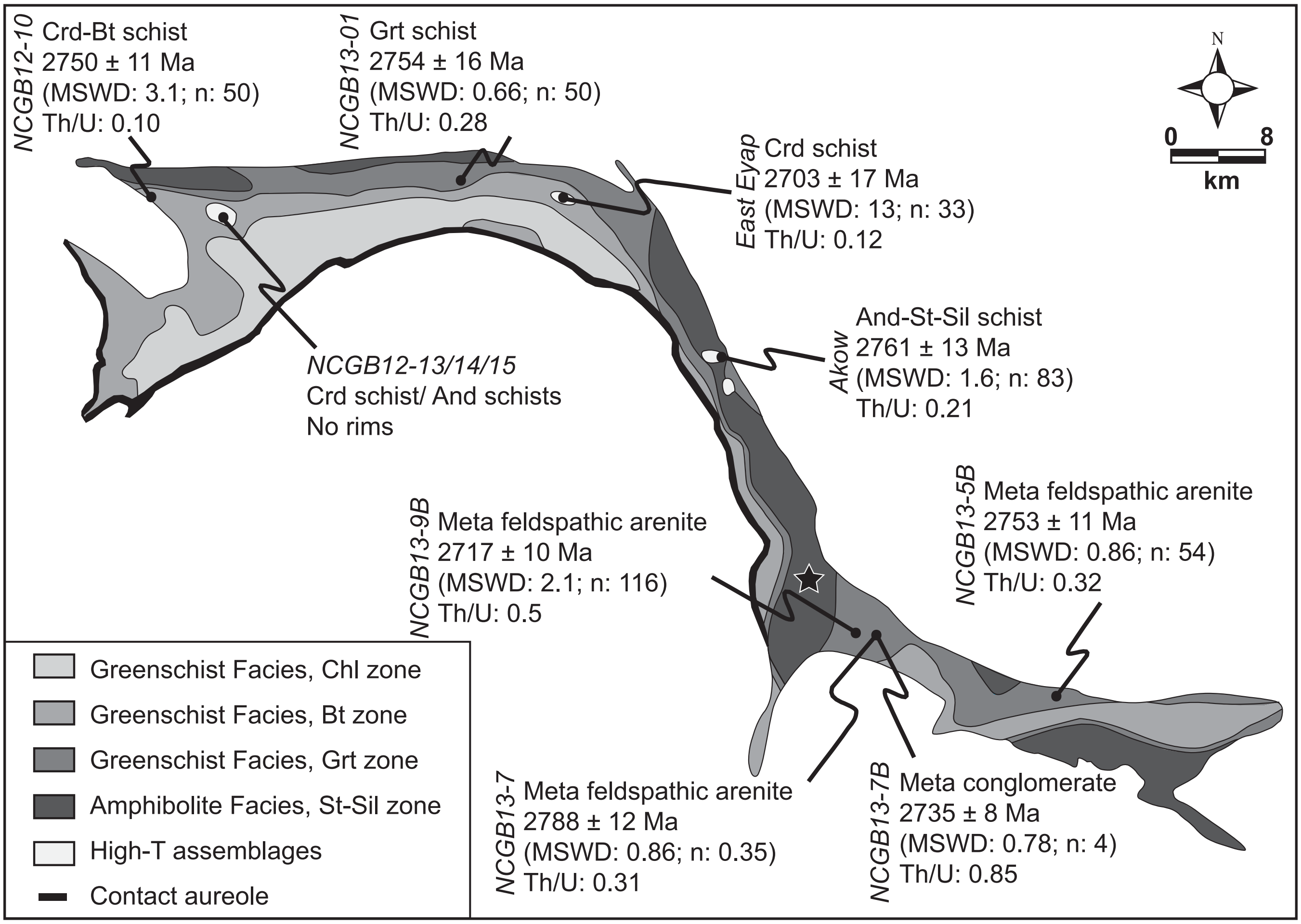

Figure 1. Metamorphic map of the North Caribou greenstone belt (modified from Breaks et al., 2001; Kelly et al., 2013) showing the locations of rock samples from this study (sample numbers NCGB12-10, NCGB12-13, NCGB12-14, NCGB12-15, NCGB13-01, East Eyap, Akow Lake, NCB13-9B, NCB13-7, NCB13-7B, NCB13-5B). Samples that exhibit U-Pb rim ages are reported as $^{207}Pb/^{206}Pb$ ages with the average Th/U values as described in the text. Metamorphic facies are variable within the belt, ranging from lower greenschist to upper amphibolite facies and apparent late high-temperature assemblages proximal to intrusions. Black star represents location of Musselwhite mine. Abbreviations: And = andalusite; Bt = biotite; Chl = chlorite; Crd = cordierite; Grt = garnet; MSWD = mean square weighted deviation; Sil = sillimanite; St = staurolite; T = temperature. Sample numbers are shown in italics.

to upper amphibolite facies, as well as localized high-temperature assemblages. Many of the samples possess well developed foliation, and at the metre-scale, folds, faults, and boudinage can be observed in the outcrops. Zircons were separated from the rocks, processed, and mounted as described by Kelly et al. (2014). The zircons were imaged before and after analysis with a JEOL6610LV scanning electron microscope (SEM; University of Ottawa, Ottawa, Canada) using secondary electron imaging in a high vacuum with a 1.7 kV beam to characterize the crystal structure, as well as to ensure that the analyzed volume was free of any fractured crystal faces or notable outgrowths. After SIMS analysis, a MicroXAM surface profilometer (University of California, Los Angeles, USA) was used to determine the pit dimensions of the material excavated from the zircon rims. We use the term rim in reference to an originally stable zircon that comes in contact with a fluid phase under fundamentally different pressure and temperature conditions. The zircon then begins to undergo coupled dissolution-reprecipitation, resulting in a recrystallized zircon rim that has re-equilibrated under these new conditions. Recrystallization commonly results in increased U concentrations, elevated light rare earth elements (LREE), and a reset isotopic age (Breeding et al., 2004; Geisler et al., 2007; Schneider et al., 2012; Kelly et al., 2014).

Analytical Methods and Data Reduction

SIMS U-Pb measurements were made using a CAMECA ims1270 ion microprobe (University of California, Los Angeles, USA) according to the methods published by Kelly et al. (2014). Our analyses were operated at beam intensities of 12 nA conducted over ~50 mass cycles resulting in ~30° µm diameter pits that are <5 µm in depth through a sputtering rate of ~100

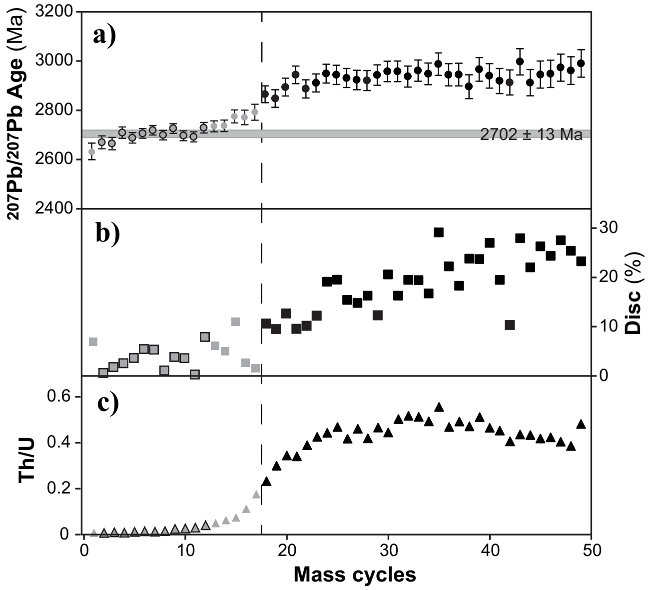

Figure 2. An example of a SIMS U-Pb zircon depth profile analysis from sample NCGB12-10, grain 8. **a)** $^{207}Pb/^{206}Pb$ age plotted against the number of mass cycles, which is a proxy for depth into the zircon crystal. Errors bars are 1σ and the vertical dashed line represents the cutoff used as the boundary between interior and rim material. Black symbols represent interior data; grey symbols represent younger (rim) data. Grey symbols with a black outline meet the criteria developed in the text as rim material and are therefore included in the rim calculation; the data points without the outline were ignored. In this example, the rim is 2702 ± 13 Ma (MSWD: 0.84; n: 11), consisting of 11 mass cycles and is ~1 µm thick. The interior of the crystal has an age of 2930 ± 15. Panel **(b)** illustrates the relationship between discordance and the mass cycle analyses. In this example, the rim is clearly more concordant. Panel **(c)** shows the changes of Th/U value with depth into the crystal. In this example, the rim material has an average Th/U ratio of 0.02, compared to the interior that has an average value of 0.44.

nm/mass cycle. The zircon reference material AS3 ($^{206}Pb/^{238}U$ age: 1099 ± 1 Ma; Paces and Miller, 1993) was analyzed after every five unknowns, following the procedure of Schmitt et al. (2003). U-Pb ages were calculated from the measured ion intensities using UCLA's in-house ZIPS v3.0.4 software written by C.D. Coath and ISOPLOT v3.75 software (Ludwig, 2012). Uncertainties of individual mass cycles are reported at 1σ and integrated ages are reported at 2σ. By examining the $^{207}Pb/^{206}Pb$ ages (younger in rims), Th/U concentrations (often lower in rims), and percent discordance (often lower in rims) from all mass cycles as a function of depth into the crystal (Fig. 2), we are able to resolve sub-micron variations in the zircon rims. For internal consistency in the identification of rim material $^{207}Pb/^{206}Pb$ age and % discordance was plotted as a function of depth, allowing for core ages to be identified and eliminated. Rim ages were calculated through the interrogation of mass cycles identified as younger than the core. To be defined as fully re-equilibrated rim material, three criteria were used: (1) containing three or more contiguous mass cycles comprising more than 30% of the total cycles that are younger than the core material; (2) the probability of fit of the weighted mean of the rims is >5%; and (3) the ages of the outermost two mass cycles for either side of the identified rim must not be significantly different (at 1.8σ) than the weighted mean rim age (six or more cycles only). In addition, any mass cycles that have a discordance of >15% were not included in the age calculation (cf. Cottle et al., 2009; 2012). Rim ages reported here (with 2σ uncertainties) were assigned to an entire rock sample (comprising many zircon analyses) through a weighted average calculation inclusive of all mass cycles identified as rim material. Rims, when present, can range in thickness from 3 to 30 mass cycles (<3 µm).

Results

Below we report the results of 172 single zircon analyses from eleven samples (as summarized in Table 1), reporting only the $^{207}Pb/^{206}Pb$ rim ages in the text. Recrystallized rim material is commonly 0.1 to 1.5 µm thick, as determined by the Th/U values and young ages. Zircon interior or core $^{207}Pb/^{206}Pb$ ages range from ca. 2800 to 3045 Ma for all eleven samples. The full SIMS U-Pb data for each sample will be published in a forthcoming journal article authored by the Kelly and Schneider; we discuss the results of the rim analyses of each sample below.

A total of 34 zircon grains were analyzed from a cordierite-bearing biotite schist (sample NCGB12-10) collected on the northeastern shore of Miskeesik Lake. The sample is a metapelite with a mineral assemblage of quartz, plagioclase, orthoclase, and biotite, with foliation defined by biotite. The sample location is within mapped within upper greenschist rocks and the sample itself also contains cordierite relics, which are now chloritized (Kelly et al., 2013). Zircons in the sample are prismatic to subrounded. The interpreted rim age calculated from ten grains is 2750 ± 11 Ma (mean square weighted deviation (MSWD): 3.1; n: 50). The Th/U value for the rim material ranges from 0.01 to 0.46, with an average value of 0.1.

Three samples were collected at the western end of Eyapimikima Lake, within a high-temperature metamorphic aureole (cordierite facies; Kelly et al., 2013). The three samples, an andalusite schist, and two cordierite schists (samples NCGB12-13, NCGB12-14, and NCGB12-15, respectively) show no evidence of zircon rim material, despite evidence of alteration and mineralization within the rock (e.g. chlorite, sericite, pyrite). Zircons in the sample are prismatic to rounded.

Fourteen zircons were dated from a garnet schist (sample NCGB13-001) in a 1 km^2 sliver of sedimentary rocks to the east of Macgruer Lake. This foliated

Table 1. Summary of locations, descriptions, and SIMS U-Pb depth profile data of samples collected throughout the North Caribou greenstone belt.

Sample number	Location (NAD 83)	Rock name	Zircon with rims (# of zircons analyzed)	Zircon morphologies	Zircon rim data†	
					$^{207}Pb/^{206}Pb$ age (Ma)	Th/U
NCGB12-10	627540E, 5872755N (zone 15)	Cordierite-bearing biotite schist	10/(34)	Prismatic/subrounded	2750±11 (MSWD: 3.1; n: 50)	0.01–0.46 (0.1)
NCGB12-13	627909E, 5868746N (zone 15)	Andalucite schist	0/(4)	Prismatic/rounded	No Rims	
NCGB12-14	626394E, 5869259N (zone 15)	Cordierite schist	0/(18)	Prismatic		
NCGB12-15	627540E, 5869191N (zone 15)	Cordierite schist	0/(5)	Prismatic/subrounded/ rounded		
NCGB13-01	648708E, 5871865N (zone 15)	Garnet schist	5/(14)	Prismatic/subrounded	2754±16 (MSWD: 0.66; n: 50)	0.05–0.64 (0.28)
East eyap	657715E, 5868570N (zone 15)	Cordierite schist	7/(20)	Prismatic/subrounded	2703±17 (MSWD: 13; n: 33)	0.05–0.27 (0.12)
Akow Lake	670288E, 5850306N (zone 15)	Andalucite-bearing siliminite-staurolite schist	8/(27)	Subrounded/rounded	2761±13 (MSWD: 1.6; n: 83)	0.11–0.42 (0.21)
NCB13-9B	688250E, 5825109N (zone 15)	Meta-feldspathic aranite	9/(20)	Prismatic/subrounded/ rounded	2717±10 (MSWD: 2.1; n: 116)	0.16–1.22 (0.5)
NCB13-7	689548E, 5825474N (zone 15)	Meta-feldspathic aranite	3/(14)	Prismatic/subrounded	2788±12 (MSWD: 0.86; n: 35)	0.11–1.85 (0.31)
NCB13-7B	689548E, 5825474N (zone 15)	Meta-clast supported polymictic conglomerate	2/(14)	Prismatic/subrounded	2735±8 (MSWD: 0.78; n: 4)	0.5–1.37 (0.85)
NCB13-5B	300330E, 5820880N (zone 16)	Meta-feldspathic aranite	3/(17)	Prismatic/subrounded	2753±11 (MSWD: 0.86; n: 54)	0.16–0.64 (0.32)

† ages calculated from integrated $^{207}Pb/^{206}Pb$ ages of SIMS mass cycles (n) with low Th/U values, shown as ranges (and averages)

metapelite is dominated by quartz and feldspar with biotite defining, and garnet occurring along, the foliation. The location sample is within a mapped a garnet-zone greenschist-facies assemblage (Kelly et al. 2013). Zircons in the sample are prismatic to subrounded and have a rim age of 2754 ± 16 Ma (MSWD: 0.66; n: 50). Th/U values from the young rims range from 0.05 to 0.64, with an average value of 0.28.

A total of twenty zircon grains were analyzed from a cordierite-schist collected from the eastern shore of Eyapimikima Lake (East Eyap) within a high-temperature aureole (cordierite facies; Kelly et al., 2013). The metapelite is a weakly foliated rock characterized by a mineral assemblage of quartz, plagioclase, and biotite with centimetre-scale porphyroblasts of cordierite. Zircons in the sample are prismatic to subrounded and yield a rim age of 2705 ± 17 Ma (MSWD: 13; n: 33), with Th/U values from the rims of 0.04 to 0.27, with an average value of 0.12.

An andalusite-bearing sillimanite-staurolite schist from the shores of Akow Lake was sampled. The strongly foliated metapelite comprises quartz, plagioclase, and biotite with porphyroblasts of staurolite and andalusite that possess sillimanite overgrowths. Twenty-seven zircon subrounded to rounded grains were analyzed resolving a rim age of 2761 ± 8.4 Ma (MSWD: 1.6; n: 83) with Th/U values ranging from 0.11 to 0.42, with an average of 0.21.

A total of twenty zircon grains were analyzed from a gossanous metafeldspathic arenite (sample NCGB13-9B). The rock is a non-foliated metasedimentary unit that has a mineral assemblage dominated by feldspar as well as quartz and biotite. Mineralization is evinced by the presence of late pyrite. Zircons in the sample are prismatic to rounded. The rim age for this sample is 2717 ± 10 Ma (MSWD: 2.1; n: 116) with Th/U values ranging from 0.16 to 1.22, with an average of 0.5. Our data reduction scheme overlooks a population of ca. 2450 Ma ages, which can be resolved through the interrogation of the data on an individual cycle basis.

A metasedimentary unit adjacent to a gabbroic intrusion on the shores of the Pipestone River was sampled. Two sub-samples were identified (samples NCGB13-07 and NCGB13-07b) based on variation in grain size and presence of clasts (sand-sized metafeldspathic arenite and a clast-supported polymictic metaconglomerate, respectively). The feldspathic arenite is characterized by mineralogy of plagioclase, quartz, and orthoclase with a very weak foliation defined by biotite. Alteration is evinced by sericitization of feldspar as well as secondary biotite. Zircons in the sample are prismatic to subrounded. The rim age calculated through the analysis of fourteen zircon grains is 2788 ± 12 Ma (MSWD: 0.86; n: 35) and with Th/U ratios ranging from 0.11 to 1.85, with an average of 0.31. The conglomerate is characterized by a sand-sized matrix not unlike the feldspathic arenite described previously, whereas clast-size ranges from centimetre to decimetre. Zircons in the sample are prismatic to subrounded. Fourteen zircon grains from the conglomerate yield a rim age of 2735 ± 89 (MSWD: 0.78; n: 4) with Th/U values ranging from 0.5 to 1.37, with an average of 0.85.

Seventeen zircon grains were analyzed from a weakly foliated metafeldspathic arenite (sample NCGB13-05b) sampled adjacent to a granitic intrusion. The primary mineralogy includes plagioclase, quartz, and orthoclase with a weak foliation defined by biotite and muscovite. Secondary mineral phases include sericite, chlorite, and oxides. Zircon grains in the sample range from prismatic to subrounded. A calculated rim age of 2753 ± 11 Ma (MSWD: 0.86; n: 54) is resolved from the rims of three grains. The Th/U values range from 0.16 to 0.64, with an average value of 0.32. These zircons in particular are characterized by elevated U concentrations (up to 5000 ppm for single mass cycles). This may have been outside of the calibration range of the reference material, which may explain the abundance of discordant mass cycles for this particular sample.

DISCUSSION AND MODELS

All of the samples presented in this study from across the belt have integrated zircon rim age populations between ca. 2790 and 2700 Ma. Despite the intensity of metamorphism ranging from hornfels and greenschist to upper amphibolite, the presence of recrystallized zircon rims occurring throughout the belt suggests that the rim-forming process is apparently independent of metamorphic facies. The lack of well-defined zircon rim material in western Eyapamikima Lake suggests that the occurrence of rims, and therefore fluid activity, is not pervasive over the entire belt. One sample (NCGB12-9B) from the Markop shear zone, south of Musselwhite mine, possesses a zircon rim population of ca. 2450 Ma, which has been observed in total-Pb monazite ages from NCGB (Kalbfleisch, 2012), Ar-Ar ages from the Musselwhite mine (Biczok et al., 2012), and zircon rims from the Abitibi (Schneider et al. 2012). This enigmatic event remains unresolved and we will not address this event further in the report.

Conventional interpretation of zircon Th/U values <0.2 suggest the neo- or recrystallized zircon material formed under metamorphic or hydrothermal conditions, likely in the presence of a crystallizing Th-bearing phase (i.e. monazite, apatite). Conversely Th/U values >0.2 occur because of primary igneous processes or in metamorphic rocks where Th-bearing phases are not present or actively crystallizing (Moeller et al., 2003). The average Th/U values in the northwestern and central portion of the NCGB average 0.175. In the southeastern portion of the belt, values are typically greater (>0.3), where monazite total-Pb ages are younger than 2600 Ma (Kalbfleisch, 2012). The elevated zircon Th/U values suggest that monazite was not present during zircon recrystallization, which is consistent with the relatively young (late) monazite ages. Additional work is required to understand the controls of bulk-rock geochemistry on the zircon rim recrystallization.

Some of the scatter in our data, represented by moderate MSWD values, is no doubt higher than expected. One explanation may be the inclusive approach to data reduction we took, and by including all of the relatively concordant data younger than the zircon core, we have sacrificed some statistical quality at the cost of a consistent data-reduction scheme. Samples with larger MSWDs can be interpreted as having thinner rim material (<300 nm) while MSWDs closer to unity are indicative of thicker rims (0.5–1.5 µm).

Regional Age Correlations

Our goal was to help resolve the timing of metamorphism and mineralization within the NCGB. Gold mineralization within the Western Superior Province occurs at 2720–2690 Ma within the Red Lake camp (Corfu and Stone, 1998; Dubé et al., 2004) and between 2730–2700 Ma at the Pickle Lake camp (Young et al., 2006). The North Caribou Greenstone Belt has a protracted history of igneous activity and thermal events. This is punctuated by three intrusive episodes at ca. >2950 Ma, 2870–2850 Ma, and 2730–2716 Ma (Biczok et al., 2012; Van Lankvelt, 2013). Within these plutons surrounding the greenstone belt, Van Lankvelt (2013) has identified a potassic alteration event at 2760–2680 Ma, as resolved from U-Pb zircon and titanite analyses. Biczok et al. (2012) suggest that gold mineralization at the Musselwhite mine may have occurred between 2720 and 2670 Ma, as determined by Sm-Nd analyses of hydrothermal garnets believed to be coeval with mineralization. Regionally within the North Caribou Superterrane (Thurston et al., 1991) and coincident with the Uchian orogeny (Percival et al., 2006), the Berens River Intrusive event affected zircon and apatite ages, resulting in 2750 to 2685 Ma U-Pb ages (Corfu and Stone, 1998). Ernst and Jowitt (2013) describe the widespread Bird River large igneous province at ca. 2735 Ma, which may have additionally acted as a driver for the hydrothermal fluid circulation. Though our results cannot explicitly resolve between the different regional tectonic episodes, they indicate that the NCGB has witnessed a metamorphic and mineralization episode broadly coeval with that of the Western Superior region.

IMPLICATIONS FOR EXPLORATION

The age of zircon rim recrystallization within the North Caribou Greenstone belt is similar to the timing of potassic alteration in the adjacent plutons and broadly correlative to mineralization within the North Caribou Superterrane. This synchronicity across a range of scales suggests that we may have determined the timing of hydrothermal events responsible for the gold mineralization within the Musselwhite mine, adding to a growing set of observations of pervasive lower temperature tectonism across the Western Superior Province. Although the zircon depth-profiling technique is likely not a primary exploration tool, it can be used as a secondary method to resolve the timing of fluid flow within Archean greenstone belts. For example, the lack of significant zircon rim material at the western end of Eyapimikima Lake probably suggests that this is a region of low economic interest, since it is an area of relatively low fluid-mineral interaction.

FUTURE WORK

The authors are currently pursuing a couple of geochronology-geochemistry methodologies, using LA-ICPMS to resolve rare earth element depth-profiles in combination with the U-Pb age depth-profiles. Moreover, we are conducting LA-ICPMS trace element mapping on the unpolished zircon grains to determine the extent of rim recrystallization, since it is apparent that not all of the zircon we examined possessed a recrystallized rim. We believe alkaline fluids are, in part, responsible for the recrystallization, and potentially the armoring effects of adjacent refractory mineral phases prohibit complete rim recrystallization.

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