EXPLORE
NUMBER 174 MARCH 2017
Newsletter for the Association of Applied Geochemists

The use of automated indicator mineral analysis in the search for mineralization – A next generation drift prospecting tool

Derek H.C. Wilton[1], Gary M. Thompson[2] and David C. Grant[3]; *[1]Department of Earth Sciences, Memorial University St. John's, Newfoundland and Labrador, Canada, A1B 3X5, [2]Office of Applied Research – College of the North Atlantic, St. John's, Newfoundland and Labrador, Canada, and [3]CREAIT Network, Memorial University St. John's, Newfoundland and Labrador, Canada.*

https://doi.org/10.70499/ROVT2038

Introduction

The natural endowment of glacial and stream sediment covering bedrock poses a significant challenge to the discovery of buried mineral deposits in Canada and elsewhere. As such, indicator mineral surveys have become a common exploration tool in till covered regions of Canada (cf. Averill 2001; 2011) with industry and research organizations, such as the Geological Survey of Canada and Geological Survey of Finland, conducting much research on the examination of fine heavy mineral fractions of till. The standard preparation techniques involve the field collection of 10-20 kg till (or other surficial sediment) samples, sieving the collected material into a range of grain sizes, then preparation of a heavy mineral concentrate (HMC) from the 0.25-2.0 mm size fractions through labour-intensive gravity techniques that ultimately involve the use of heavy liquids (McClenaghan 2011). The resultant 0.25-2.0 mm grain size samples are optically examined using a binocular microscope. Grains of specific "indicator" minerals are identified through some combination of colour, hardness, cleavage, lustre, and crystal morphology properties. They are then counted and selected grains are removed from the HMC; so-called "traditional visual analysis" or TVA (Averill & Huneault 2016). The mineral phases determined are those indicator minerals that are specific to the type of mineral deposit(s) or bedrock lithologies being explored for. The full range of mineral phases present in these samples, however, cannot be ascertained purely through visual analysis. Furthermore, positive identification of each mineral phase picked typically requires subsequent micro-analyses by scanning electron microscope or electron microprobe (e.g., McClenaghan 2011).

Over the past seven years, in collaboration with Altius Minerals Inc. and Vale Newfoundland and Labrador Ltd., we have developed a method with a FEI Scanning Electron Microscope-Mineral Liberation Analyser (SEM-MLA) to quantitatively evaluate the mineralogy of surficial sediments including till, stream sediments, soils, and gossans collected in Newfoundland and Labrador, Canada (Fig. 1). In this discussion, we will elaborate on this work with respect to indicator minerals in tills. However, the method is not restricted to use with till and stream sediments in glaciated terrain, but can be used for all types surficial sediments in all terrain types. SEM-MLA analysis is also available at commercial labs in Canada and other countries.

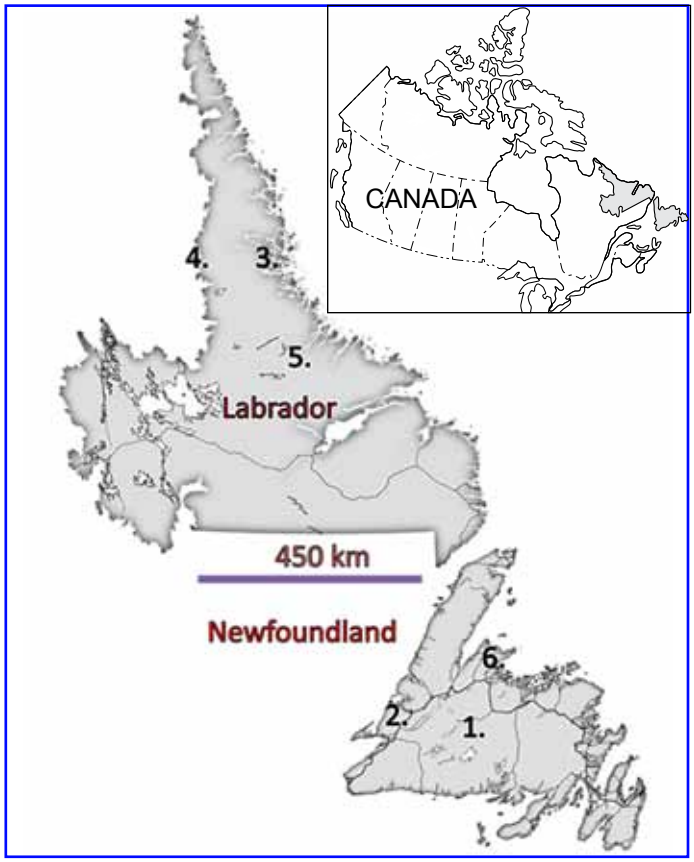

Figure 1. Map of Newfoundland and Labrador, Canada, with locations discussed in text. 1 = central Newfoundland ophiolite (Pipestone Pond); 2 = western Newfoundland ophiolites (Lewis Hills); 3 = location of Vale's Voisey's Bay Ni-Cu Mine and the village of Nain; 4 = Strange Lake REE deposit; 5 = Florence Lake PGE occurrences; and 6 = abandoned Baie Verte asbestos mine.

The basis for Mineral Liberation Analysis (MLA) studies is the energy dispersive fingerprint for each mineral or mineral association; referred to as a Species Identification Protocol or SIP[1]. The SIP matches the observed EDX

[1] SIPs is a terminology typically used by QEMSCAN users. The term also quite concisely defines our MLA library; the terms Mineral Reference Editor and MR lists or spectral libraries have also been used.

continued on page 5

The use of automated indicator mineral analysis... continued from page 1

(energy-dispersive X-ray) scanning electron (SEM) spectra of each particle against the MLA database of stored spectra (i.e., "fingerprints"). The Memorial University group (MUN) in Newfoundland, Canada, has developed a sophisticated library of SIPs that can identify almost all minerals present within a given sample, down to < 0.3% unknowns (e.g., Wilton & Winter 2012; Wilton et al. 2015), using a matching threshold of 70% in the MLA software. New minerals are added to the library as they are identified. At present we can analyse a till/stream sediment sample and accurately identify up to 99.7% of mineral particles present in the sample; a given individual sample when prepared for analysis can contain up to 20,000 particles. A typical analysis time per sample is 1-2 hours.

The SEM-MLA can define what indicator minerals are present in both ore and ore-associated rock, including alteration haloes. The latter may actually represent a much larger reservoir of indicator minerals than the deposit itself, in that the ore-associated rock and/or alteration halo will contain its own suite(s) of indicator minerals that is larger than the deposit itself. As such, the greater volume of potential indicator minerals may produce a more significant target signal for the mineral deposit system than the deposit alone.

Alteration of country rocks by hydrothermal fluids involves the development of new minerals that replace the original mineralogy (i.e., the original minerals were susceptible to alteration). Most of the potential indicator minerals of interest will be in a HMC, but lighter density, distinctive alteration minerals, such as micas, can also be present in a till sample. The HMCs produced for SEM-MLA work will typically include some lighter density phases, in particular as intergrowths with denser HMC minerals. In our SEM-MLA techniques, we analyse some of the non-HMC (i.e., light mineral concentrate) mineral separates to make sure that no potential indicator minerals are missed. A further advantage to using MLA is that it collects a spectra of all grains that are distinctive in BSE and these data can be revisited and queried offline at a later date if a new indicator mineral relevant to mineral exploration is discovered. Traditional visual analysis, or TVA, (cf. Averill & Huneault 2016) in which HMC are observed with a binocular microscope, would not allow such querying of a data set. Presumably if an important new indicator mineral has an apparently distinct visual appearance, then the operator can go back through the HMC and pick out the new phase of interest. Otherwise, all phases with similar visual aspects would have to be picked and subsequently analysed by EMP to determine their presence.

The SEM-MLA can define a greater range of indicator minerals for each deposit variety, along with mineral intergrowth textures, such that a matrix or assemblage of indicator minerals can be defined that might be specific to each deposit type. The ultimate aim would be to use the automated quantitative mineralogy capabilities of the MLA to parse through a surficial sediment sample HMC and define whether, and what, indicator minerals of any mineral deposit type are present.

Sample Preparation

The initial till sample as collected in the field consists of about 10 kg of material. The till is air-dried after which we typically sieve into nine size fractions: < 63 μm, 63-125 μm, 125-180 μm, 18-250 μm, 250-500 μm, 500 μm – 1 mm, 1 - 2 mm, 2-4 mm, >4 mm; although only one fraction is actually used for the SEM-MLA analysis, the other fractions are retained in case additional information is required. To determine the optimum grain size of till HMC minerals for SEM-MLA analysis, a series of different size fractions were analyzed and it was found that the 125-180 µm fraction provided the optimal results (Wilton & Winter 2012). If the grain size was smaller than 0.125 mm, the analyses took longer (30-50%) and the minerals were more difficult to definitively identify as the X-ray beam is statistically more likely to miss a grain centroid and instead land on a grain boundary or crack/pit producing an unwanted result. If the grain size was larger than 0.180 mm, the number of particles that could be analyzed in a given grain mount decreased by 25 to 75% and the range of observable intergrowth textures decreased. The 0.125-0.180 mm grain size was the easiest to evaluate for contamination, as, in general, any airborne contaminant grains in a processing (sieving) facility, or contaminant grains introduced to the mount during polishing, would be demonstrably finer grained.

The 125-180 μm size fraction is processed using a shaking (Wilfley) table to separate lighter from denser minerals; our specific gravity threshold between heavy and light minerals using the shaking table is about 2.7-2.8. In general, we would only use one pass over the table, as we wished to analyse minerals of intermediate density and/or dense-light intergrowths to fully understand the till material. The table concentrate, of course, can be repeatedly processed over the table using different splitting parameters, tilt, etc. to produce a higher density HMC. We do not use heavy liquid or magnetic separation techniques on the separates.

The resultant HMC for each sample is then partitioned using an automated micro-riffler to produce a 0.3 g separate that is poured as a 25 mm in diameter layer within a 30 mm mold in diameter (Fig. 2a). This amount of material settles as a mono-layer such that there is no gravity-induced separation of denser particles. The mould is filled with epoxy and the hardened puck is polished (Fig. 2b). We use a round

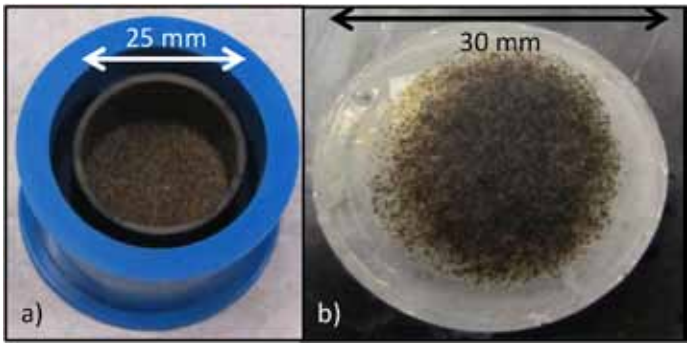

Figure 2. a) Till HMC separate (0.3 g of 125-180 µm fraction) forming monolayer within inner 25 mm diameter plastic ring within 30 mm in diameter puck (blue); **b)** polished puck containing upwards of 20,000 particles.

continued on page 6

The use of automated indicator mineral analysis... continued from page 5

mould for the till samples, but the Core Research Equipment and Instrument Training (CREAIT) network group at MUN (Grant et al. 2016) have also produced 30 x 17 mm (x 10 mm) rectangular moulds that hold up 1 g of material. Sample pucks are carbon-coated and the grains are analysed via the SEM-MLA. For the 30 mm round pucks we use an 8-Round sample holder so that eight samples are analysed per run. Figure 3 shows the SEM sample chamber and 8-Round sample holder for the 30 mm pucks.

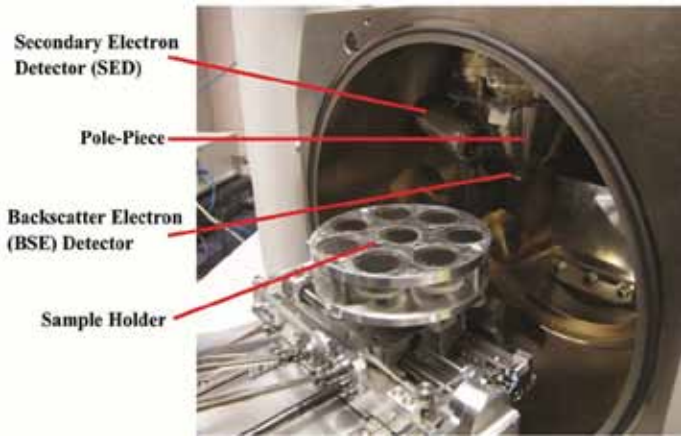

Figure 3. The SEM sample chamber with 8-round (30 mm) holder for epoxy grain mounts (from Sylvester 2012).

Samples can also be directly prepared from surficial sediment material without going through the processes of sieving and density separation. In such a case, a split is removed from the sample and mounted in the epoxy puck; obviously grains must be less than 25 mm in diameter or they won't fit in the mould.

SEM-MLA analytical techniques

Sylvester (2012) provides a detailed overview of the theory and systematics of the SEM-MLA system. The SEM portion of the SEM-MLA laboratory in the CREAIT facility at MUN consists of a FEI Quanta 400 environmental SEM equipped with a Bruker XFlash EDX Detector. The SEM electron gun uses a tungsten filament at an operating voltage of 25 kV and a beam current of 13 nA. The working distance between sample and detector is 12 mm. The SEM is equipped with Mineral Liberation Analyser (MLA) software developed at the University of Queensland (Australia) Julius Kruttschnitt Mineral Research Centre (JKTech) (e.g., Pietersen et al. 2009). The sophisticated MLA software associated with the SEM allows for quantitative evaluation of the abundance, association, size and shape of minerals in automated, systematic fashion. In other words, the MLA allows for the quantitative mapping of mineral phases in individual grain mounts and/or polished thin sections, essentially providing a digital point count of mineral species. The MLA software requires that the SEM be configured in back-scatter electron (BSE) image mode wherein minerals that contain denser elements produce brighter images. Essentially the MLA detects mineral particles in the grain mount based on variations in BSE grey scale and then analyses each particle. The particles can be inclusions, or, as in many cases several inclusions, within a larger grain.

continued on page 8

Note: This EXPLORE article has been extracted from the original EXPLORE Newsletter. Therefore, page numbers may not be continuous and any advertisement has been masked.

The use of automated indicator mineral analysis... continued from page 6

For till HMC samples, the grey-scale "threshold" was set with the epoxy as black and metallic copper as white. The dwell time (i.e., time spent acquiring data) for each X-ray analysis of a particle is 60 microseconds. Image size resolution was set at 800 dpi and minimum particle diameter detected was about 5 μm. The MLA compares the elemental spectra for any particle analyzed with a "library" of mineral spectra to identify the particular phase. If the MLA software cannot identify a mineral during analysis, it is recorded as an "unknown" and the analyst manually re-examines the spot analysed after the automated run to ascertain whether it truly is an unidentified mineral or a defect in the polished surface (i.e., crack, hole, etc.). In rare cases, the analysis is obtained on a boundary between two minerals in a particle containing more than one mineral species and is therefore a mix of two mineral spectra.

During the post-processing of the MLA run, the operator can also query the location and/or form of particular particles. The instrument can be engaged to return to specific particles of interest and the operator can examine the particles in detail up to, and including, the conducting of real-time EDX analyses of the mineral and intergrown phases. The operator can also obtain higher resolution BSE images at this stage. During the analytical run, spatial registration points can be taken for the grain mount, such that all particles can be precisely located if future analyses, such as Laser Ablation ICP-MS, or EPMA, are to be performed on the grains of any particular mineral phase.

The MLA maps each grain mount in terms of frames per mount, typically using a horizontal field width (HFW) of 1-2 mm. About 100-120 frames are measured with approximately 100 particles/frame, this is sufficient for a typical MLA analysis as >100 particles will saturate the X-ray detector, thus missing X-rays and leading to inadequate results; each frame represents a portion of the mount. The particles in each frame are analyzed and then their relative and absolute abundances can be calculated. The EDX spectrum collected by the MLA simply indicates what elements are present in the mineral and their relative concentrations; it does not determine the crystallographic nature of the particle (e.g., gypsum cannot be readily distinguished from anhydrite). Furthermore, as the MLA analyses are spectrum-based, the EDX component cannot identify minor (trace) elements within individual minerals. Thus, any elements identified in a mineral are significant (>1%) components of the given mineral phase.

The confidence level for the spectral match is set during the initial analysis; typically set at about 90% probability. The software scales the probability between 0% and 100%, with 100% as a perfect match (probability of 1) whereas 50% has a probability match of 1.00E-80 which termed an "average match" (Mateo 2010). With post-processing and identification of unknowns, the confidence level can be reset to another level. A higher threshold would require a closer match between observed and stored spectra increas-

continued on page 11

The use of automated indicator mineral analysis... continued from page 8

ing the precision on the results. Generally during the initial processing stage the level is adjusted to 90% ensuring all atypical mineral spectra are examined in detail. During this stage it may be possible to identify multiple variants of the same mineral, i.e., magnetite with high or low Ti contents. The frame is the fundamental organizational template for the MLA technology. The software can produce a BSE map of all particles in a frame (Fig. 4a) and a false-color map (Fig. 4b) of particles based on an assigned color legend for each particle.

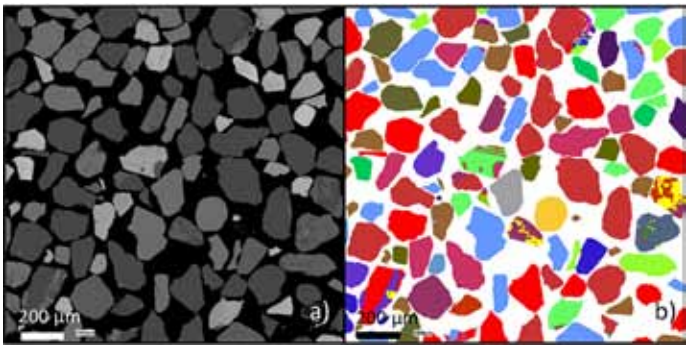

Figure 4. a) BSE map of single frame; **b)** MLA false colour map of the same frame (note scale bars are 200 µm long); plagioclase grains are red and magnetite grains are green on the MLA map.

The frames can be stitched together to provide a map of the entire grain mount analyzed (Fig. 5). More detailed queries can provide information on a particle, and thus mineral grain size and shape.

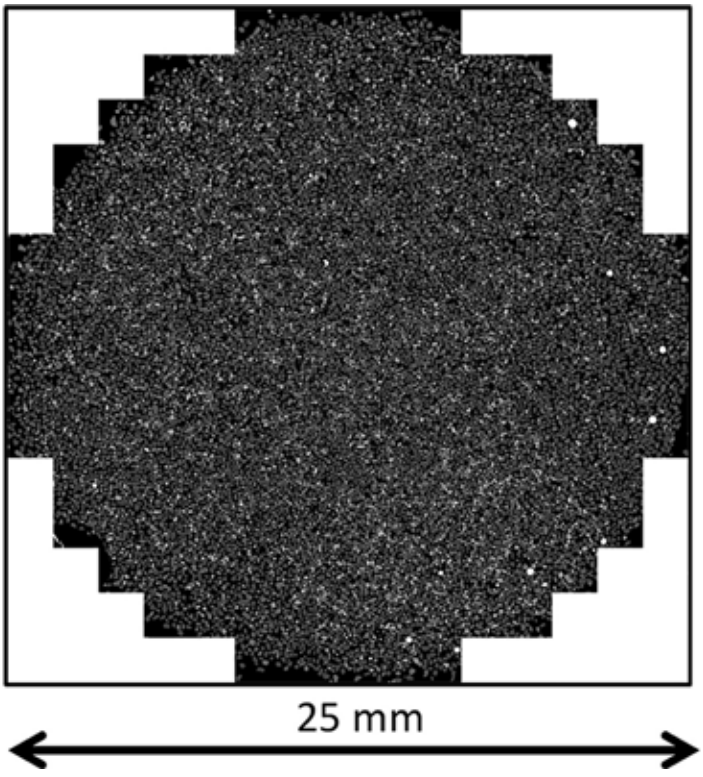

Figure 5. BSE map of entire sample made from stitching all frames together; sample contains upwards of 20,000 particles.

Our SEM-MLA routinely analyzes and identifies 10,000 to 20,000 particles in an individual 0.3 g grain mount; "particles" include larger mineral grains as well as inclusions within a larger grain. Following standardization of the amount of material mounted in an epoxy puck and also the analytical parameters, the range of particles typically identified in a grain mount is 10,000 to 17,000. The analytical area is set as close to 25 mm in diameter as the grain mount allows.

Using the mineral library, a typical analysis of a grain mount would be completed in 1 to 2 hours with post-processing of 1 to 1.5 hours for a total analytical time per grain mount (or till HMC separate) of 2 to 3.5 hours. The initial analytical times, prior to derivation of a more complete SIP library, were on the order of 4 to 6 hours per sample. Using the MUN spectral library, unknown particles in a grain mount generally make up less than 0.25%, a remarkably low proportion of the more than 10,000 particles identified in a sample (i.e., generally less than 25 particles). Many of these "unknown" particles are actually misidentified phases. If a truly "new" (i.e., previously unseen) mineral phase is identified, a clean spectrum is collected and added to the spectral library.

Data Output

Table 1 illustrates the type of digital data produced from the MLA analysis of each mineral in a till sample; for the sake of brevity, the ten most abundant phases in the sample are listed. Each mineral present in a sample is mapped in terms of weight % (based on the proportion of the mineral in sample compared to all minerals in the sample from the ideal molecular weight of the mineral), area % that the mineral represents compared to all other minerals in the sample, the actual area of the mineral in the sample as mapped by the MLA (in μm), the number of particles of the mineral mapped in the sample, and the number of grains of the mineral in the sample. The number of grains is always greater than the number of particles, as the MLA definitions can have a particle that is composed of 2 or more minerals, such as a quartz inclusion in a magnetite

Table 1. Digital output for sample T-001 - ten most abundant minerals mapped using SEM-MLA.

Mineral	Wt%	Area%	Area (micron)	Particle Count	Grain Count
Total	100.00	100.00	161347556.25	13579	22959
Ilmenite	29.45	23.95	38640787.50	3588	3920
Magnetite-Ti	14.04	10.74	17329350.00	1804	2095
Plag-An50	7.50	10.97	17696937.50	1672	1762
Almandine	9.72	9.05	14608650.00	1350	1397
Hypersthene	7.16	7.87	12701712.50	1206	1286
Plag-An25	4.59	6.76	10902475.00	1249	1477
Quartz	3.36	4.95	7990918.75	939	1063
Magnetite	5.40	4.06	6544631.25	644	677
Grunerite	2.60	2.95	4754506.25	516	575
Cpx_Diop	2.08	2.39	3851768.75	443	489

continued on page 12

The use of automated indicator mineral analysis... continued from page 11

grain. Because many of the potentially important indicator minerals may represent less than 0.01% of the sample, the mineral's area in microns is the best parameter to use for comparing mineral contents between samples. Note that the SEM-MLA can detect variations in magnetite composition and identify magnetite grains that contain significant Ti; traditional visual analyses would not be able to detect such variations. The MLA software has tools for advanced classification, including 1) using EDX spectral windows to characterize percentage of elements that must be present to match a particular species ID, or 2) the ability to place thresholds of minimum and maximum elemental percentages on classification scripts that can be used, for example, to further distinguish a pure magnetite from titano-magnetite.

Examples of the use of SEM-MLA in indicator mineral search

Awaruite in Newfoundland

The Altius-MUN SEM-MLA surficial sediment indicator mineral work was initiated to evaluate whether the mineral awaruite was present in Newfoundland ophiolites (Wilton & Winter 2012). Awaruite (Ni_3Fe) is a magnetic, dense, nickel-iron alloy formed during the serpentinization of ultramafic, particularly olivine-bearing, rocks (Filippidis 1985). In 2009, First Point Minerals Corporation began an exploration and economic evaluation program on the Decar Property in northern British Columbia, Canada. The target units on the property were serpentinized ultramafic rocks (Verley 2011). Nickel assay values of up to 0.1 to 0.15% in the ultramafic rocks suggested that the property might represent a new class of potentially commercial nickel deposits, consisting of disseminated awaruite *(op cit)*.

Using the First Point concept of disseminated awaruite as a potentially commercial source of nickel, and based on the large number of ophiolitic ultramafic complexes in western and central Newfoundland, Canada, Altius Resources Inc. initiated an exploration program for awaruite in Newfoundland with a joint venture partner Cliffs Natural Resources Inc. But exploration for awaruite in the ophiolites was problematic because awaruite had not been previously reported, and the ophiolites, themselves, represented two contrasting targets. On the one hand, western Newfoundland ophiolites (Fig. 1 location 2) comprise huge massifs of barren outcrop in which it would have been extremely difficult to define prospective awaruite-bearing horizons without intense, detailed mapping and petrographic examination. In contrast, ophiolites in central Newfoundland (Fig. 1 location 1) are very poorly exposed as widely separated patches of bedrock outcrop extensively covered by till.

In order to evaluate the two different ophiolite areas (massifs with 100% outcrop vs. till-covered outcrop remnants), the Altius-MUN group decided to sample stream sediments from the massifs and till from the till-covered units and then analyse derived HMC separates via SEM-MLA for awaruite and any associated minerals. Awaruite was detected in both the tills and stream sediments (Fig. 6) along with the rare pair of hydrated Ni-bearing minerals, asbolane ($(Ni,Co)_2$-$xMn^{+4}(O,OH)_4 - H_2O$)) and pecoraite ($Ni_3(Si_2O_5)(OH)_4$). The SEM-MLA technique proved successful and identified areas in both ophiolite regions in which awaruite is present in outcrop (Wilton & Winter 2012).

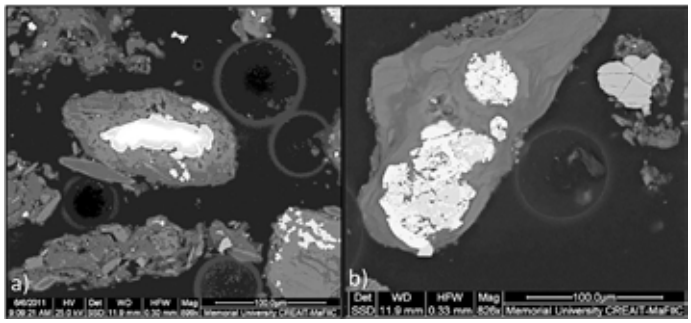

Figure 6. BSE images of awaruite grains mapped by MLA: **a)** awaruite (white) in serpentine grain (darker grey) with Cr-bearing awaruite rim (medium grey) from a till sample collected in central Newfoundland (location 1 on Figure 1); **b)** awaruite (white) intergrown with magnetite (medium grey) within serpentine grain (dark grey) from a stream sediment sample collected in western Newfoundland (location 2 in Figure 1).

Awaruite is a silvery-white mineral that can be mistaken for chromite, magnetite or some other metallic phases in traditional visual analysis (Fig. 7). It was the EDX component of the SEM-MLA system that actually defined its presence in the surficial material. The mineral would be difficult to identify using TVA.

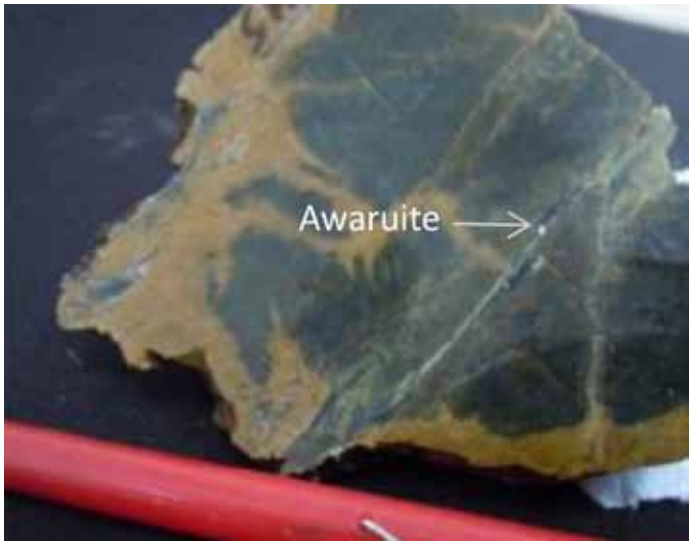

Figure 7. Awaruite grain in serpentinized ultramafic rock, Pipestone Pond Ophiolite Complex, central Newfoundland (location 1 in Figure 1) from Piller (2012). The outcrop was discovered in follow-up mapping after the identification of awaruite in till using SEM-MLA.

Sulphides in surficial sediments around Voisey's Bay

In a 2009 study (unpublished Vale report), we examined indicator minerals in till and stream sediments around the Voisey's Bay region of northern Labrador (Fig. 1 location 3), Canada, using the SEM-MLA. We subsequently conducted a more detailed SEM-MLA analysis of surficial sedi-

continued on page 13

The use of automated indicator mineral analysis... continued from page 12

ments in the vicinity of the Voisey's Bay orthomagmatic Ni-Cu-Co sulphide deposit (Evans et al. 2000; Lightfoot et al. 2012) to ascertain whether there were detectable sulphide minerals present and whether they might be a vector towards Ni-Cu sulphide mineralization. The surveys included a cross-section from bedrock gossan through overlying till in the Ovoid mine pit wall, and the sampling of till in a sonic drill hole 7 km north of the Ovoid open pit and five split spoon till cores surrounding the Ovoid open pit (Wilton at el. 2014, 2015). The split spoon core was collected by hammering a 10 cm in diameter plastic pipe through the till to bedrock.

In the cross-section study, data from the BSE and MLA were used to identify a previously unrecognized regolith developed on the massive sulphide bedrock beneath overlying till (Fig. 8). The regolith material contained angular to acicular grains composed mainly of oxidized Fe-sulphides (Fig. 8a), whereas the till was composed of rounded dominantly silicate phases with Fe-oxide rims (Fig. 8b). The age of this regolith remains undetermined but it could be as old as Cretaceous (cf. Conliffe 2015). The recognition of this regolith by SEM-MLA provides an important clue for future exploration.

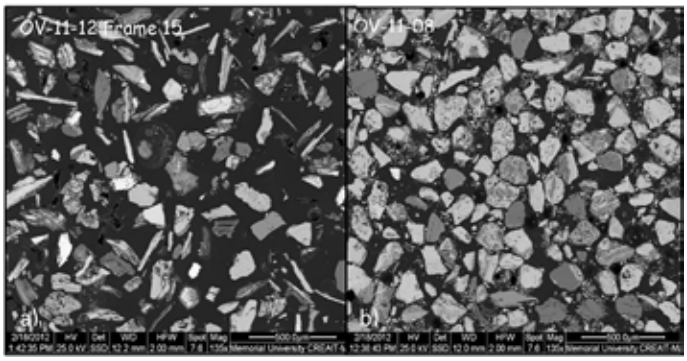

Figure 8. BSE images of grains recovered from **a)** regolith, and **b)** till samples in cross section from bedrock through regolith to till in the Ovoid Open Pit, Voisey's Bay Mine, NL, (location 3 in Figure 1) as determined by SEM-MLA (from Wilton et al. 2015).

Core from the sonic drill hole was sampled at five different intervals over its 26 m length; the sample material was not sieved, nor tabled and was simply mounted in epoxy. The mineralogy of the till material in all five samples as analysed by the SEM-MLA was basically the same with the dominant minerals being quartz, clay minerals, feldspars and hornblende. Diopside was prominent in the lowermost two till samples. Similarly, there is more hedenbergite and ilmenite in till samples further down the hole. Epidote and biotite are more abundant in the upper three samples. Thus it appears that the two deepest samples, from just above bedrock, have greater concentrations of minerals that would be considered to be derived from the local mafic igneous rocks. Other minerals, such as hercynite and magnetite, associated with Voisey's Bay mineralization do not have any downhole variation. Sulphide grains, very predominantly iron sulphides (gossan) and/or oxidized iron sulphides were detected in the till, but are only abundant (43 grains) in the bottom 1.2 m of the hole. Chalcopyrite (four grains) and sphalerite (1 grain) were detected in till at the bottom of the hole.

The five split spoon holes were all drilled around the Ovoid mine. Seventeen till samples (four each from two holes, and three each from the others) were analysed by SEM-MLA. These samples were not sieved, nor tabled, and represent a complete sample of the material, excepting larger pebbles and cobbles. All 17 samples were poorly sorted diamicton (i.e., typical till). Largest grains were up to 600 μm in diameter and fine fraction material was clay-sized (<5 μm). Some samples were more equigranular and clay-rich than others. Locally, portions of the samples were clast-supported. The dominant modal mineralogy, all samples contain variations of quartz, feldspars, clay minerals, hornblende, micas and scapolite.

The split spoon holes contained variable amounts of sulphide and the SEM-MLA was able to identify pyrrhotite (up to 25 grains in a single sample), oxidized Fe-sulphide (gossan – up to 65 grains), pentlandite (up to 20 grains), chalcopyrite (up to 14 grains) and tetrahedrite (up to 3 grains). Pentlandite grains are variably (little to complete) oxidized. Some pyrrhotite grains have exsolved pentlandite and chalcopyrite (Fig. 9).

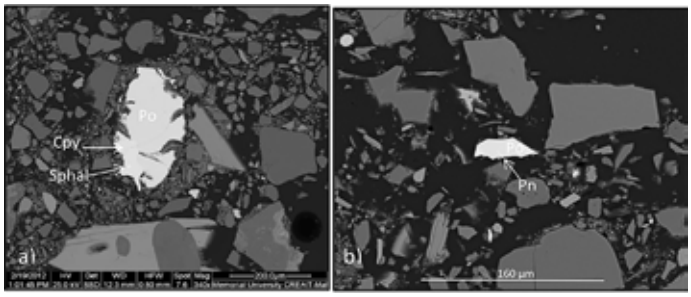

Figure 9. BSE images of till from split spoon drill holes near Ovoid Open Pit Mine, Voisey's Bay, NL as mapped by SEM-MLA: **a)** ragged pyrrhotite grain (Po) intergrown with chalcopyrite (Cpy) and sphalerite (Sphal) to lower left; **b)** pyrrhotite (Po) exsolved pentlandite lamellae (Pn).

The SEM-MLA analyses proved successful in identifying the dominant modal mineralogy of each split spoon sample and essentially classified the mineral composition as being typical till. No systematic spatial variations between holes of silicate (e.g., hercynite) or oxide (magnetite) indicator minerals typical of Voisey's Bay mineralization were observed. In terms of identifying trace sulphide minerals, the MLA analyses likewise proved quite useful. Altogether, the amounts of pyrrhotite-pentlandite intergrowths, abundance of pentlandite and chalcopyrite particles, and presence of metal-rich sulphide grains suggest that at least some of the minerals in the split spoon till samples were derived from a nickeliferous source, likely the nearby Voisey's Bay mineralization.

One final point to note is that, although the SEM-MLA analyses indicated that sulphide minerals could be detected in the till samples, they were few in number (< 200 particles

continued on page 14

The use of automated indicator mineral analysis... continued from page 13

in a sample with >10,000 particles). Also, the particles/grains were minute, rarely > 100 μm (i.e., 0.1 mm) in diameter; the largest sulphide grain detected was only 1.25 mm long. These observations from the SEM-MLA work are at odds with those of Averill (2001) who stated that heavy mineral concentrates derived from 10 kg surficial sediment samples from the Voisey's Bay area contained chalcopyrite and an "overabundance of garnet" (p.74). The survey referred to by Averill (op cit.) was of bulk river sediment samples (Chislett 1994) from a ca. 7-km grid spacing. SEM-MLA analysis of till around the mine site did not indicate the presence of appreciable garnet. The split spoon and sonic samples contained < 0.65% (mainly <0.40%) and 0.41%, respectively, garnet. Chislett (op cit.), in fact, did not actually report the presence of any chalcopyrite in the river sediment samples.

Sourcing till origins

One of the most significant observations from our work on tills with the SEM-MLA is its ability to define source regions for till. In one spectacular case, the SEM-MLA was able to identify gittinsite ($CaZrSi_2O_7$) grains (Fig. 10) in till samples collected across a very limited region near Nain (Fig. 1 location 3), which is within the glacial ice stream extending from the Strange Lake REE deposit over 100 km to the east.

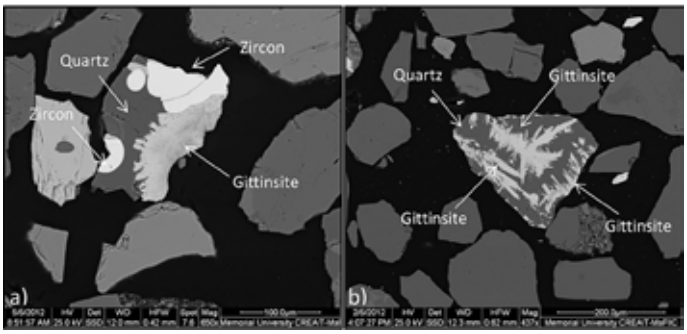

Figure 10. BSE images of till from near Nain NL, as mapped by SEM-MLA: **a)** gittinsite intergrown with zircon and quartz; **b)** feathery-shaped gittinsite intergrown with quartz (dark grey).

The Strange Lake deposit (Miller 1990; Gysi et al. 2016) is a 6 km wide peralkaline granite complex (Fig. 1 location 4), that hosts REE, Zr, Y, and Nb mineralization in granite, aplite, and pegmatite phases along with associated quartz veins. Zirconium is dominantly contained within gittinsite, a common phase in the complex (Kerr 2013). Due to the point-source nature of the granite and associated REE mineralization and well-developed surficial geochemical signatures, the deposit has become a classic example of a glacial dispersal train (e.g., Batterson 1989a; Batterson & Taylor 2009; McClenaghan 2007). McConnell and Batterson (1987) determined that the Strange Lake REE mineralization signal could be detected in till, stream sediment, stream waters, lake sediment and lake waters along a northeastern trend from the deposit. Batterson (1989b) reported that a ribbon-shaped, northeast-trending, geochemical dispersal train extended for up to 40 km from the deposit, and he concluded (p. 27) "there is evidence of considerable transport distances. Within the study area, maximum transport is at least 40 km, with expectation that a clearly defined dispersal train extends for at least 55 km and probably farther".

The SEM-MLA study of mineral grains in till samples proved Batterson's (1989) supposition that the Strange Lake dispersal train extended much further to the northeast. The SEM-MLA data allows for the rapid and precise identification of unique minerals that can define the source, or at least a portion, of the till material at distances (i.e., >100 km), well past the decay rate (rate of elemental concentration decrease with distance down ice) for till geochemistry.

Zircon is a common component in any surficial sediment HMC and we use the abundance, size and shape of zircons in each sample as monitors of a variety of parameters in our surveys, including bedrock types, sample quality, etc. The ability to of the SEM-MLA to examine zircon interiors, allows for the derivation of fundamental information on probable sources of the till material. For instance, internal zoning within a zircon grain with a very well-developed core overgrown by an equally well-defined rim (Fig. 11a) suggests origin from a high-temperature granitic source (Cox 2003). In other examples, altered zircon grains are observed to contain uraninite-galena or thorite-huttonite inclusions (Figs 11b and c). Such zircons were derived from a U or Th rich source, but because of their metamict nature and inherent fragility, these zircons could not have been transported any significant distance (Cox 2003). Finally in some other zircon grains that we have mapped in till samples, the MLA analysis revealed xenotime overgrowths on zircon (Fig. 11d). These types of overgrowths occur during diagenetic reactions in sediment (e.g., Prost et al. 2013)

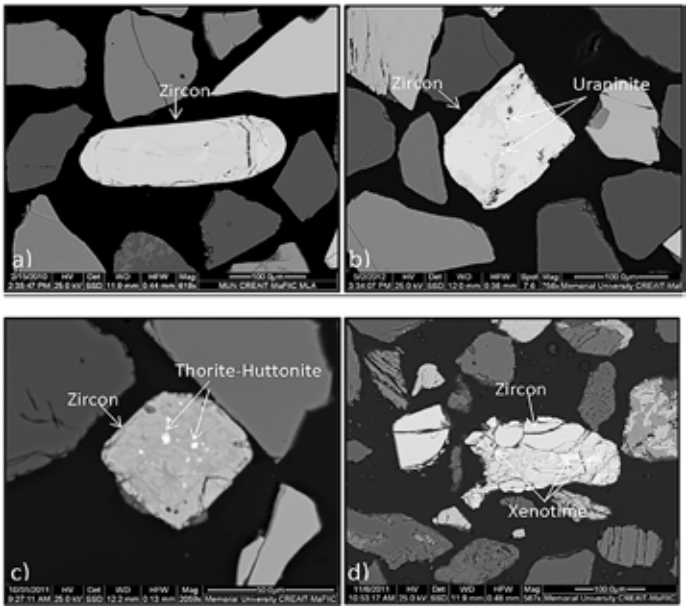

Figure 11. BSE images of zircon grains in till from northern Labrador, as mapped by SEM-MLA: **a)** zircon with well-defined core and rim; **b)** altered zircon with uraninite inclusions inclusions; **c)** altered zircon with thorite and huttonite inclusions inclusions; **d)** altered zircon overgrown by xenotime.

continued on page 15

The use of automated indicator mineral analysis... continued from page 14

indicating that the till was derived in part from a potentially low-grade sedimentary bedrock unit.

Inclusions and minerals that should not exist in tills

Traditional visual analysis (TVA) cannot see inside mineral grains unless they are removed from the concentrate, mounted and subsequently polished. Furthermore TVA is predicated on the fact that all representative minerals will be of a common size and generally monomineralic. This is not the case in nature, minerals are intimately mixed with each other from full-fledged intergrowths to inclusions of one mineral within another.

Aside from the ability to quantify and identify all mineral phases in samples, the most significant aspect of the SEM-MLA till analysis is the ability to define inclusions within other mineral grains and the ability to see through alteration rims to detect the true nature of a grain. In many cases resistant minerals mantling less resistant minerals allows for the detection of minerals in a till that should not be there because of their inability to withstand physical or chemical weathering. Figure 12 contains images of sulphide minerals that can be mapped as inclusions by the SEM-MLA, including galena, stibnite, sphalerite and arsenopyrite. Without the capabilities of the SEM-MLA, these inclusions would not have been detected by TVA.

The detail provided by the SEM-MLA can also include the identification and quantification of exsolution textures (Fig.

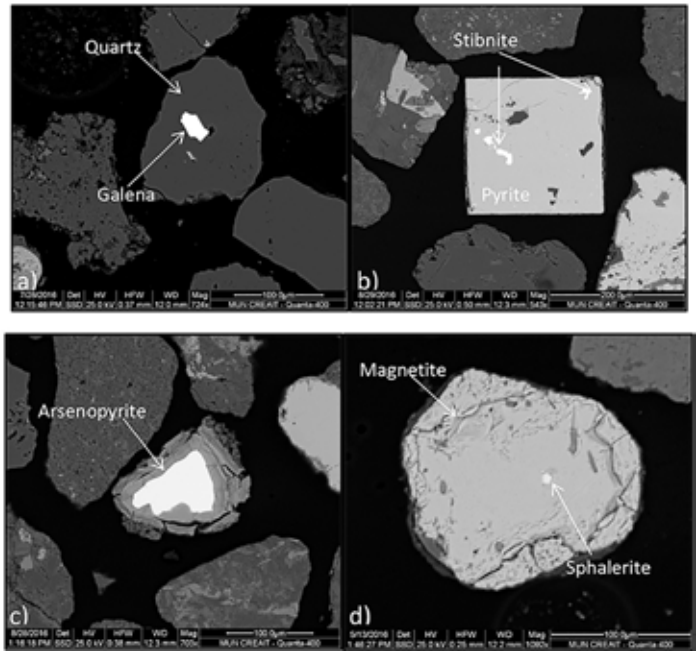

Figure 12. BSE images of mineral inclusions in other minerals from tills in NL, as mapped by SEM-MLA. **a)** galena in quartz; **b)** stibnite in pyrite cube; **c)** arsenopyrite surrounded by Fe-oxide alteration rim; **d)** sphalerite in magnetite.

13) such as pentlandite or troilite from pyrrhotite, bornite from chalcopyrite and Ir-Rh alloys from cobaltite minerals. The grains in Figure 13d were actually derived from crushed host rock to the Florence Lake PGE occurrence in northern Labrador (Fig. 1 location 5), Canada, in a study to

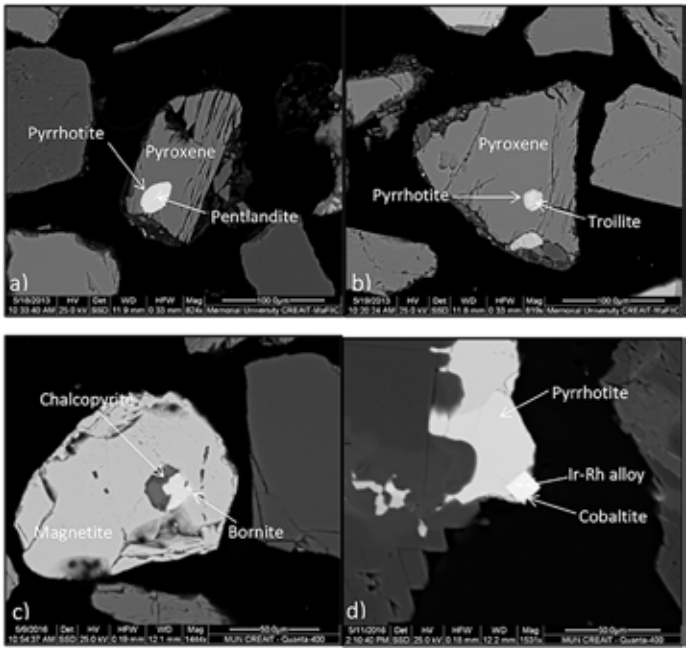

Figure 13. BSE images of sulphide mineral exsolution in inclusions from tills in NL, as mapped by SEM-MLA. **a)** pentlandite exsolved from pyrrhotite in pyroxene; **b)** troilite exsolved from pyrrhotite in pyroxene; **c)** -bornite (white) exsolved from chalcopyrite in magnetite; **d)** Ir-Rh alloy exsolved from cobaltite (cube) intergrown with pyrrhotite (note this is from a broken rock sample not till).

produce "anthropogenic" till (Wilton et al. 2016). As unmixing (exsolution) of sulphide phases is such a common and intrinsic feature of orthomagmatic sulphide deposits (e.g., Naldrett 2013), the observation of these types of textures in mineral grains from a till would strongly suggest the presence of such mineralization in the source area of the till. Such fine and delicate features would not be observable via TVA.

Precious metals and proxies

Because of inherent densities, and hence extreme brightness in BSE image mode, gold and silver are readily identified by the SEM-MLA (Fig. 14). Not only can the SEM-MLA locate and define these minerals, the innate capability to identify precious metal inclusions means that the mineralogical context or association of precious metals can be defined. The EDX component of the SEM-MLA can also distinguish different Au-Ag contents in electrum grains (Fig. 14b – upper right), aiding in definition of source and mineralization type (e.g., Chang et al. 2011).

The SEM-MLA examination of mineral grains in till (and other surficial sediments) can determine the presence of robust proxy minerals for gold that would have greater abundances that gold itself (Fig. 15). For instance, tourmaline can be a common accessory in orogenic gold deposits (e.g., Beaudoin & Chiaradia 2016). Cinnabar is likewise associated with many epithermal-type auriferous deposits (e.g., Sillitoe 1994).

Evans (1995) reported that leucoxene is a common

continued on page 16

The use of automated indicator mineral analysis... continued from page 15

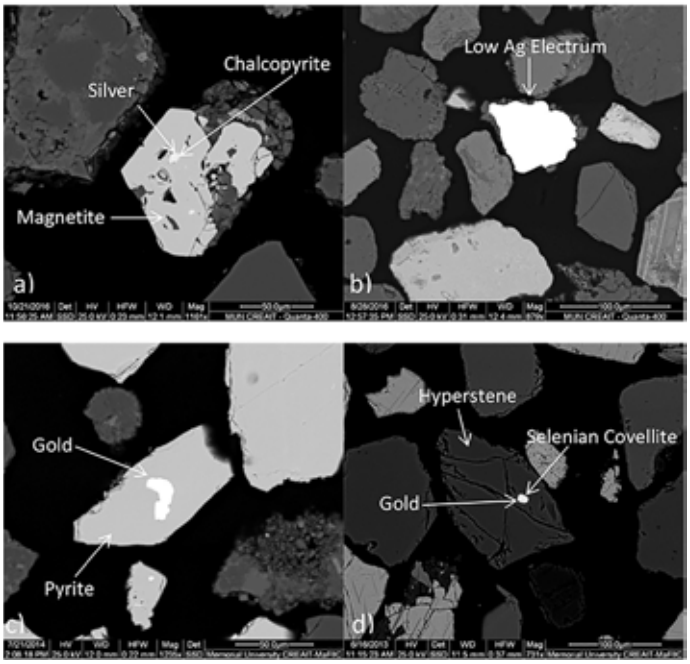

Figure 14. BSE images of precious metals in grains from tills in NL, as mapped by SEM-MLA. **a)** silver intergrown with chalcopyrite in magnetite; **b)** low-silver electrum; **c)** gold in pyrite; **d)** inute gold grains intergrown with selenian covellite in hypersthene.

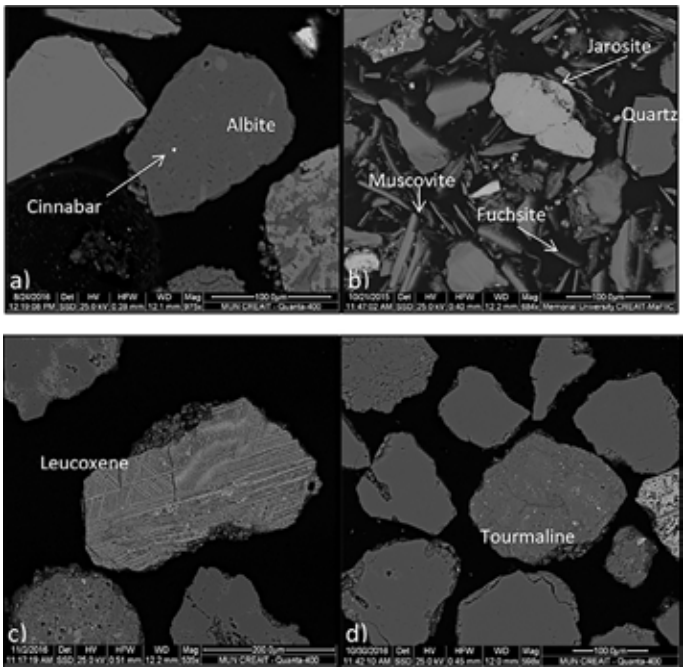

Figure 15. BSE images of proxy minerals for auriferous mineralization from tills in NL, as mapped by SEM-MLA: **a)** cinnabar in albite; **b)** jarosite grains with muscovite, fuchsite and quartz, note this is a regolith developed on the Klondyke Schist; **c)** a grain of leucoxene (altered rutile) with rutile lamellae (white); **d)** tourmaline (spongey gray).

alteration mineral surrounding gabbro-hosted gold deposits in Newfoundland. Leucoxene forms as fine-grained alteration of primary titanium minerals, primarily ilmenite and/or Ti-rich magnetite, and as Ti and its constituent minerals are widely viewed (eg., Govett 2013) as being immobile to chemical transport, the presence of leucoxene in a surficial sediment would suggest that the sediment has sampled a region of hydrothermal alteration that might be associated with an auriferous system. Deysel (2007) demonstrated that the SEM-MLA can reliably distinguish between leucoxene and ilmenite, something that would be difficult by TVA.

Kelley et al. (2011) reported on a detailed examination of indicator minerals in till down ice of the Pebble porphyry Cu-Au-Mo deposit of Alaska, USA. Minerals of particular interest include gold, jarosite, cinnabar, Mn-epidote, andradite, apatite and pyrite. The study indicated that the different mineral phases were each important, though they may have reflected a different component of the ore-forming system. Indicator mineral identification in the study (op cit.) was based on TVA techniques, but selected grains had to be analysed by electron microprobe to confirm their identity. In contrast, the SEM-MLA can readily identify and quantify these same minerals immediately, including mineral phases such as jarosite (Fig. 15b) which can easily be mistaken for limonite in simple visual observation.

Mine remediation

A project with the Centre for Applied Health Research, Memorial University, involved examining surface sediment samples around the tailings piles at the Baie Verte Asbestos Mine (Fig. 1 location 6) in Baie Verte NL by SEM-MLA (Wilton 2013). The aim of the study was to evaluate the possible presence of asbestos minerals and, if present, the extent of their distribution. A secondary aim was to determine whether any asbestos present was solely chrysotile, or was the more toxic grunerite variety present as well. The SEM-MLA mapping identified and quantitatively defined chrysotile contents (Fig. 16) and no grunerite was detected

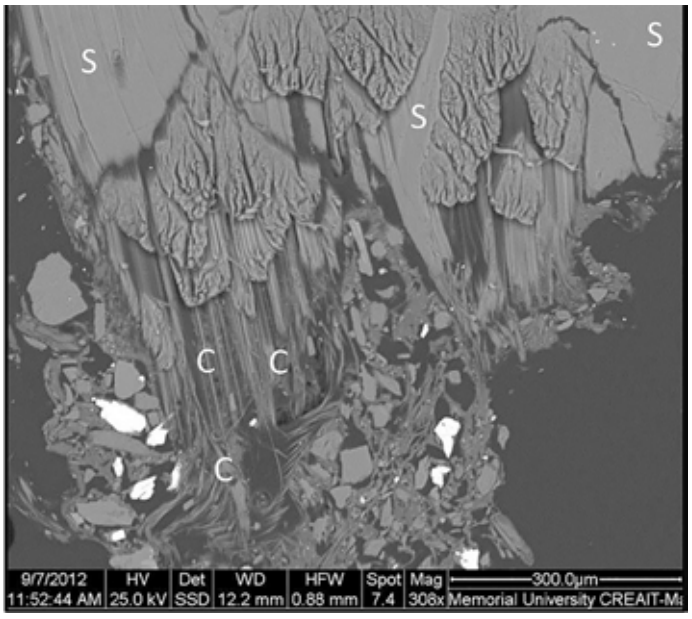

Figure 16. BSE image of serpentine (S) terminating into chrysotile (C) asbestos along the bottom; from surface sample at the abandoned Baie Verte asbestos mine tailings dump, NL (location 6 in Figure1).

continued on page 17

The use of automated indicator mineral analysis... continued from page 16

in the surface sediment around the tailings. This type of environmental monitoring can be readily conducted for other mining operations and could even be used to evaluate air-borne mineral contaminants.

Conclusions

The SEM-MLA is an important new tool that quantifies indicator mineral species in the fine fraction (<0.25 mm) in surficial sediment surveys. The data provided by SEM-MLA will not only provide the information that helps to vector towards the mineralization for a wide variety of mineral deposit types, but also to define source of the sediment itself. The SEM-MLA provides information that helps to define alteration haloes around ore bodies, and thus allows for the confirmation of till transport directions based on bedrock geology. The SEM-MLA can identify a range of minerals as inclusions in larger mineral grains that would not usually survive weathering, such that the inclusions are also indicator minerals.

Acknowledgements

This research was initiated with Altius Resources Inc. at the suggestion of Dr. Lawrence Winter and also with Rod Smith at that time with Vale NL. Much of the work described was conducted with Altius Resources Inc. and their crew of Jeff Morgan, Carol Seymour, Lloyd King and Bob Patey. Altius Resources Inc. also graciously permitted the use of many of the BSE images herein. Some of this research was funded by the Newfoundland Research and Development Corporation. The actual SEM-MLA technique initially drew on the talented Michael Shaffer (MUN CREAIT Network) and his vast knowledge of EDX. Laterally, Dylan Goudie of the MUN CREAIT labs was of considerable assistance. Beth McClenaghan and Daniel Layton-Matthews provided insightful reviews, we thank Beth McClenaghan for her encouragement.

References

AVERILL, S.A. 2001. The application of heavy indicator mineralogy in mineral exploration with emphasis on base metal indicators in glaciated metamorphic and plutonic terrains. *In:* MCCLENAGHAN, M.B., BOBROWSKY, P.T., HALL, G.E.M. & COOK, S.J. (eds.) *Drift Exploration in Glaciated Terrains.* Geological Society, London, Special Publication, **185** 69–81.

AVERILL, S.A. 2011. Viable indicator minerals in surficial sediments for two major base metal deposit types: Ni-Cu-PGE and porphyry Cu. *Geochemistry: Exploration, Environment, Analysis*, **11**, 279–291.

AVERILL, S.A. & HUNEAULT, R.G. 2016. Basic indicator mineral math: Why visual analysis of the entire heavy mineral fraction of large sediment samples is required on indicator mineral exploration programs in glaciated terrains. *Explore (Newsletter for the Association of Applied Geochemists),* Number **172** - September 2016.

BATTERSON, M.J. 1989. Glacial dispersal from the Strange Lake alkali complex, northern Labrador. *In:* R.N.W. DILABIO and W.B. COKER (Eds.) *Drift Prospecting.* Geological Survey of Canada, Paper **89-20**, 31-40.

BATTERSON, M.J. 1989b. Quaternary geology and glacial dispersal in the Strange Lake area, Labrador. *Newfoundland Department of Mines and Energy Report* **89-03**.

BATTERSON, M.J. & TAYLOR, D.M. 2009. Geochemical reanalysis of till samples from the Strange Lake area, Labrador (NTS Map Sheets 14D/5 and 24A/8). Government of Newfoundland and Labrador, Department of Natural Resources, Geological Survey, *Open File* **LAB/1479**.

BEAUDOIN, G., and CHIARADIA, M. 2016. Fluid mixing in orogenic gold deposits: Evidence from the H-O-Sr isotope composition of the Val-d'Or vein field (Abitibi, Canada). *Chemical Geology*, **437**, 7–18.

CHANG,Z., HEDENQUIST, J.W., WHITE, N.C., COOKE, D.R., ROACH, M., DEYELL, C.L., GARCIA JR,J., GEMMELL, J.B., MCKNIGHT, S. & CUISON, A.L. 2011. Exploration Tools for Linked Porphyry and Epithermal Deposits: Example from the Mankayan Intrusion-Centered Cu-Au District, Luzon, Philippines. *Economic Geology* **106**, 1365-1398.

CHISLETT, A. 1994. First year report of field work in Labrador July 8, to Sept. 16, 1993 Unpublished Assessment report, Archean Resources Ltd., 48 p. with appendices (available at http://gis.geosurv.gov.nl.ca/geofilePDFS/Batch06/PDF/LAB_1027.pdf)

CONLIFFE, J. 2015. Geological setting and genesis of high-grade iron-ore deposits in the eastern Labrador Trough. *In:* Newfoundland and Labrador Department of Natural Resources Geological Survey, *Current Research Report* **15-1**, 1-25.

COX, R.A. 2003. Morphological, chemical, and geochronological techniques for characterizing detrital zircon. *In:* LENTZ, D.R., (ed.) *Geochemistry of Sediments and Sedimentary Rocks: Evolutionary Considerations to Mineral Deposit-Forming Environment.* Geological Association of Canada GEOtext **4**, 105-120.

DEYSEL, K. 2007. Leucoxene study: a mineral liberation analysis (MLA) investigation. The South African Institute of Mining and Metallurgy *6th International Heavy Minerals Conference 'Back to Basics'*, 167-172.

DROST, K., WIRTH, R., KOŠLER, J., JØRGENSEN, H.F. & NTAFLOS, T. 2013. Chemical and structural relations of epitaxial xenotime and zircon substratum in sedimentary and hydrothermal environments: a TEM study. *Contributions Mineralogy and Petrology,* **165**, 737–756.

EVANS-LAMSWOOD, D.M., BUTT, D.P., JACKSON, R.S., LEE, D.V., MUGGRIDGE, M.G., WHEELER, R.I. & WILTON, D.H.C. 2000. Physical controls associated with the distribution of sulfides in the Voisey's Bay Ni-Cu-Co deposit, Labrador. *Economic Geology*, **95**, 749-769.

EVANS, D. T. W. 1995. Epigenetic gold occurrences eastern Dunnage Zone, Newfoundland. Newfoundland Department of Mines and Energy Mineral *Resource Report* **9**.

FILIPPIDIS, A. 1985. Formation of awaruite in the system Ni-Fe-Mg-Si-O-H-S and olivine hydration with NaOH solution, an experimental study. *Economic Geology,* **80**, 1974-1980.

GOVETT, G.J.S. 2013. Rock Geochemistry in Mineral Exploration. *Elsevier*.

GRANT, D.C., GOUDIE, D.J., SHAFFER, M. & SYLVESTER, P. 2016. A single-step trans-vertical epoxy prepa-

continued on page 18

The use of automated indicator mineral analysis... continued from page 17

ration method for maximising throughput of iron-ore samples via SEM-MLA analysis. Applied Earth Science: Transactions of the Institutions of Mining and Metallurgy, Section B: Applied Earth Science **125**, 57-62.

GYSI, A.P., WILLIAMS-JONES, A.E. & COLLINS, P. 2016. Lithogeochemical Vectors for Hydrothermal Processes in the Strange Lake Peralkaline Granitic REE-Zr-Nb Deposit. *Economic Geology*, **111**, 1241–1276.

KELLEY, K.D., EPPINGER, R.G., LANG, J, SMITH, S.M. & FEY, D.L. 2011. Porphyry Cu indicator minerals in till as an exploration tool: example from the giant Pebble porphyry Cu-Au-Mo deposit, Alaska, USA. *Geochemistry: Exploration, Environment, Analysis,* **11**, 321–334.

KERR, A. 2013. Rare-Earth-Element (REE) behaviour in the Strange Lake Intrusion, Labrador: Resource estimation using predictive methods. *In:* Newfoundland and Labrador Department of Natural Resources Geological Survey, *Current Research Report* **13-1**, 117-136.

LIGHTFOOT, P.C., KEAYS, R.R., EVANS-LAMSWOOD, D. & WHEELER, R. 2012. S saturation history of Nain Plutonic Suite mafic intrusions: origin of the Voisey's Bay Ni–Cu–Co sulfide deposit, Labrador, Canada. *Mineralium Deposita,* **47**, 23–50.

MATEO, S. 2010. MLA v 3.0 Regional Training Manual 2010. *Unpublished Report FEI Ltd*.

McCLENAGHAN, M.B. 2007. Till Geochemistry and Indicator Mineral Methods for Exploration in Glaciated Terrain. Exploration 07 Workshop 2 Exploration Geochemistry: *Basic Principles and Concepts* (available at https://www.appliedgeochemists.org/images/stories/Exploration_07/B_McClenaghantill.pdf)

McCLENAGHAN, M.B, 2011. Overview of common processing methods for recovery of indicator minerals from sediment and bedrock in mineral exploration. *Geochemistry: Exploration, Environment, Analysis,* **11**, 265–278.

McCONNELL, J.W. & BATTERSON, M.J. 1987. The Strange Lake Zr-Y-Nb-Be-REE deposit Labrador; A geochemical profile in till, lake and stream sediment and water. *Journal of Geochemical Exploration*, **29**, 105-127.

MILLER, R. 1990, The Strange Lake pegmatite-aplite–hosted rare-metal deposit, Labrador: *In:* Newfoundland Department of Mines and Energy, *Current Research, Report* **90-1**, 171–182.

NALDRETT, A.J. 2013. Magmatic Sulfide Deposits: Geology, Geochemistry and Exploration. *Springer.*

PIETERSEN, K. PHAN, V.H., CHEN, W., LAM, A., DONALDSON A. & LASKER, R. 2009. Dataview 2009 Beta 2": Release notes for Build 2. *Unpublished report JKTech Ltd.*

PILLER, M.P. 2012. An examination of awaruite (Ni_3Fe) formation during serpentinization of the Pipestone Pond ophiolitic complex in the Atlantic Lake area, central Newfoundland. *Unpublished BSc. (Hons.) dissertation, Memorial University of Newfoundland.*

SILLITOE, R.H. 1994. Epithermal gold-silver-mercury deposits at Paradise Peak, Nevada; ore controls, porphyry gold association, detachment faulting, and supergene oxidation. *Economic Geology,* **89**, 1128-1248.

SYLVESTER, P.J. 2012. Use of the mineral liberation analyzer (MLA) for mineralogical studies of sediments and sedimentary rocks. In: SYLVESTER, P.J. (ed.) *Quantitative Mineralogy and Microanalysis of Sediments and Sedimentary Rocks.* Mineralogical Association of Canada Short Course **42**, St. John's, 1-16.

VERLEY, C.G. 2011. Report on the Decar Nickel Property Trembleur Lake Area (NTS 93K/13, 14), Omineca Mining Division, British Columbia, Canada. *Unpublished Report for First PointMinerals Corporation (available on SEDAR http://www.sedar.com/).*

WILTON, D. H.C., 2013. Report on MLA-SEM analyses of surface soil samples from the former Baie Verte Asbestos Mine, Baie Verte, NL. *Unpublished Report for Centre for Applied Health Research, Memorial University of Newfoundland.*

WILTON, D.H.C., THOMPSON, G.M. & EVANS-LAMSWOOD, D. 2014. MLA-SEM examination of sulphide mineral breakdown (and preservation) in till, Voisey's Bay Ni-Cu-Co deposit, Labrador – The distribution and quantitative mineralogy of weathered sulphide phases. *Fredericton 2014 GAC/MAC program with abstracts,* 290-291.

WILTON, D.H.C., THOMPSON, G.M. & EVANS-LAMSWOOD, D. 2015. MLA-SEM examination of sulphide mineral breakdown and preservation in till, Voisey's Bay Ni-Cu-Co deposit, Labrador - The distribution and quantitative mineralogy of weathered sulphide phases in a transect from massive sulphide through gossanous regolith to till cover. *In: Environmental and Economic Significance of Gossans,* (ed.) M.-C. Williamson; Geological Survey of Canada, Open File **7718**, 31-41.

WILTON, D.H.C. & WINTER, L.S.,2012. SEM-MLA (Scanning Electron Microprobe – Mineral Liberation Analyser) research on indicator minerals in glacial till and stream sediments – An example from the exploration for awarauite in Newfoundland and Labrador. In: SYLVESTER, P.J. (ed.) *Quantitative Mineralogy and Microanalysis of Sediments and Sedimentary Rocks.* Mineralogical Association of Canada Short Course **42**, St. John's, 265-283.

WILTON, D., WINTER, L., PATEY, B., MORGAN, J. & SEYMOUR, C. 2016. Indicator mineral fingerprints for base metals and gold: Developing a next generation drift prospecting tool. Prospectors and Developers Association of Canada. *Exploration Insights 2016.*