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Targeted Geoscience Initiative 4: Canadian Nickel-Copper-Platinum Group Elements-Chromium Ore Systems — Fertility, Pathfinders, New and Revised Models

Trace elements in Fe-oxide minerals from fertile and barren igneous complexes: Investigating their use as a vectoring tool for Ni-Cu-PGE sulphide mineralization

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ABSTRACT

The aim of this study was to develop a new technique to determine the fertility of mafic intrusions for Ni-Cu-PGE sulphide mineralization using the mineral chemistry of Fe oxides in the silicate host rocks. A suite of 25 trace elements was determined in magnetite and ilmenite, by laser ablation ICP-MS at LabMaTer (UQAC), from a variety of barren and fertile igneous complexes. Two of Canada's largest Ni deposits, the 1.85 Ga Sudbury Igneous Complex and its vast Ni-Cu-PGE mineral district (Ontario) and the 1.34 Ga Eastern Deeps Intrusion-hosting Ni-Cu-Co sulphide mineralization at Voisey's Bay (Newfoundland), were selected for study. Samples chosen from igneous complexes that are barren of significant Ni sulphide mineralization comprise layered mafic intrusions (Bushveld Complex, South Africa and Sept Iles, Quebec) and anorthosite suites (Saguenay-Lac-St.-Jean, Quebec) that host Fe-Ti-V-P oxide deposits, some of which contain trace amounts of Ni-Cu-PGE sulphides. Mafic rocks of the 1.33 Ga Newark Island layered intrusion (Labrador) were also studied as they are similar in composition and setting to Voisey's Bay but devoid of significant Ni sulphide mineralization.

In sulphide-undersaturated magmas, Cu, Sn, Mo, and Zn are incompatible during fractionation and thus increase in concentration in late-crystallizing magnetite and ilmenite. Upon sulphide saturation and the formation of a trace amount of sulphide, only Cu is depleted in the silicate magma relative to the incompatible elements. Copper depletion, as recorded by Fe oxides, is a sensitive indicator of sulphide saturation and can be diagnostic of whether a Ni-bearing sulphide deposit will have formed if the Cu depletion occurred early during fractionation. In contrast, Ni and Co are compatible during fractionation, partitioning into olivine, orthopyroxene, and, where present, sulphide, and their concentrations steadily decrease in the Fe oxides, together with Cr, as crystallization proceeds. Iron oxides from barren igneous complexes plot on a single Ni-Cr trend but Fe oxides from fertile complexes (those hosting Ni sulphide deposits) plot on a parallel Ni-Cr trend displaced to lower Ni concentration. Nickel depletion is therefore recorded in Fe oxides and has the potential to identify intrusions with buried Ni-sulphide mineralization. The advantages of using Fe oxides as an exploration tool include their resistance to post-magmatic processes, such as alteration, and their preservation and easy recovery from glacial till and heavy mineral separates.

INTRODUCTION

Magmatic Ni-Cu-PGE sulphide deposits are hosted towards the base of ultramafic and mafic igneous complexes and formed by efficient accumulation of immiscible sulphide liquids that scavenged chalcophile metals from the host silicate magma (Naldrett, 2004). Exploration work undertaken to identify Ni-Cu-PGE sulphide mineralization benefits from an understanding of whether the intrusions are anomalously depleted or enriched in highly chalcophile metals (PGE, Cu, and Ni). At present, this comprises bulk rock geochemical ratios, such as Ni/Ni* (Lightfoot et al., 2001, 2011; Darling et al., 2010: where Ni* is the expected Ni concentration for a given MgO content of a S-undersaturated within-plate basalt. Thus a low Ni/Ni* value indicates Ni depletion due to S-saturation and segregation), Cu/Zr (Lightfoot and Hawkesworth, 1997; Li et al., 2000; Darling et al., 2010) and Cu/Pd (Keays, 1995; Barnes and Lightfoot, 2005), and Ni depletion in olivine (Li et al., 2000). Magmatic Fe oxides (magnetite and/or ilmenite) commonly crystallize in the middle to upper parts of mafic-ultramafic intrusions,

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which may or may not host Ni or PGE mineralization at their bases. Recent studies on the trace element composition of Fe oxides in massive sulphide from Ni-Cu-PGE deposits (Dare et al., 2012; Boutroy et al., 2014) and massive oxides from Ti-V-P deposits (Dare et al., 2014b) demonstrate that Fe-oxide trace element chemistry is sensitive to fractionation of sulphide and silicate liquids and to the presence of sulphide minerals. The purpose of this mineralogical study was to investigate whether 1) the composition of Fe oxides, found in mafic intrusions that host world-class Ni deposits, records sulphide saturation and segregation via chalcophile depletion, and 2) whether this signature can be used as a fertility indicator to distinguish fertile from barren intrusions and help vector towards a Ni sulphide deposit at the base or in the footwall of prospective intrusions.

SAMPLING AND METHODOLOGY

Samples

The study focused on two well known fertile igneous complexes that contain some of Canada's largest Ni-deposits: the 1.85 Ga Sudbury Igneous Complex, Ontario (total of 69 samples, 450 Fe-oxide grains), and the 1.34 Ga Voisey's Bay intrusion of Eastern Deeps (total of 19 samples, 192 Fe-oxide grains) within the Nain Plutonic Suite of Labrador. The 1.33 Ga Newark Island layered intrusion (total of 18 samples, 76 Fe-oxide grains), also within the Nain Plutonic Suite, was chosen to represent a 'barren' mafic intrusion of similar age, composition, and setting to Voisey's Bay but without any significant Ni-mineralization. Details of the geological background, sample locations, and geochemical databases (both mineral and whole rock) for these 3 intrusions are presented in Dare et al. (2014a). This mineral database was compared to a background data set of Fe oxides (total of 54 samples, ~340 Fe-oxide grains) from Ti-V-P deposits (Méric, 2011; Néron, 2012; Dare et al., 2014b) hosted in igneous complexes that are barren of significant nickel sulphide mineralization. These comprise samples of massive Fe oxides from layered mafic intrusions, such as the Bushveld Complex (South Africa), Sept Iles (Canada), and Rio Jacaré (Brazil), as well as from the anorthosite massif of Saguenay-Lac-St.-Jean (Quebec, Canada). Although these igneous complexes are barren of massive Ni-rich sulphide mineralization, most had some history of sulphide saturation; trace amounts of magmatic sulphides, some of which are enriched in PGE, formed either in the lower parts of some of the complexes, such as the world-class Merensky Reef of the Bushveld Complex, and in the upper part with the massive oxide layers in the Rio Jacaré Complex (Sá et al., 2005).

Methodology

In situ determination of trace elements in the magnetite and ilmenite were carried out on 107 polished thin sections using 1) electron microprobe (EMP) at Université Laval, Quebec City, following normal protocol (see details in Dare et al. (2012)); and 2) laser ablation-ICP-MS at LabMaTer, Université du Québec à Chicoutimi (UQAC) for Mg, Al, Si, P, Ca, S, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, Y, Zr, Nb, Mo, Ru, Pd, Sn, La, Sm, Yb, Hf, Ta, W, Re, Ir, Pt, Au, Pb, and Bi. The laser system at UQAC comprises a RESOlution M-50 (Excimer 193 nm) coupled to an Agilent 7700x ICP-MS. Beam size varied from 33 to 75 µm with a laser frequency of 10 to 15 Hz. ^{57}Fe was used as the internal standard. USGS synthetic basaltic glass, GSE-1g, was used to calibrate and GSD-1g and an in-house standard (Bushveld magnetite) were used to monitor the results. Analytical protocol and full data sets are given in Dare et al. (2014a, b). Detection limits are typically sub-ppm, e.g., <0.1 ppm for Cu and Mo (see Dare et al., 2014a). Sulphur, Re, Ir, Ru, Pt, Pd, and Au were below detection in the Fe oxides and were thus not quantified using GSE-1g. The relatively large spot size used during LA-ICP-MS analysis homogenizes the magnetite and any subsolidus exsolutions, such as ilmenite or spinel. Thus the data represent the initial composition of the magmatic magnetite that crystallized from the silicate melt before subsolidus processes took place. The raw data signals were screened to avoid inclusions of silicates and sulphides in analysis.

RESULTS AND DISCUSSION

Data analysis: Barren Intrusions

Chalcophile elements commonly present at concentrations above detection limits in igneous Fe oxides are Ni, Cu, Co, Mo, Zn, Sn, and Pb whereas the PGE are typically below detection limits (Dare et al., 2012, 2014b). However, of these elements that can be measured in Fe oxides, only Cu and Ni record chalcophile depletion of the silicate magma upon S-saturation, due to their high partition coefficients (D) into sulphide melt (D ~1000 and ~500, respectively, see review in Barnes and Lightfoot (2005)). Iron-oxide data from mafic intrusions that are barren of significant Ni sulphide mineralization demonstrate the behaviour of chalcophile elements during fractionation of mafic magmas (Fig. 1a). Nickel and Co are compatible and thus decrease during fractionation, as they readily partition into silicate minerals (e.g. olivine and pyroxene). This evolution of silicate melt composition is recorded by both magnetite and ilmenite: the Ni, Co, Cr, and V contents of Fe oxides systematically decrease up sequence in layered intrusions with highest values in Fe oxides from the lowermost (Ti-V deposits) magnetite layers and lowest values in those from the upper-

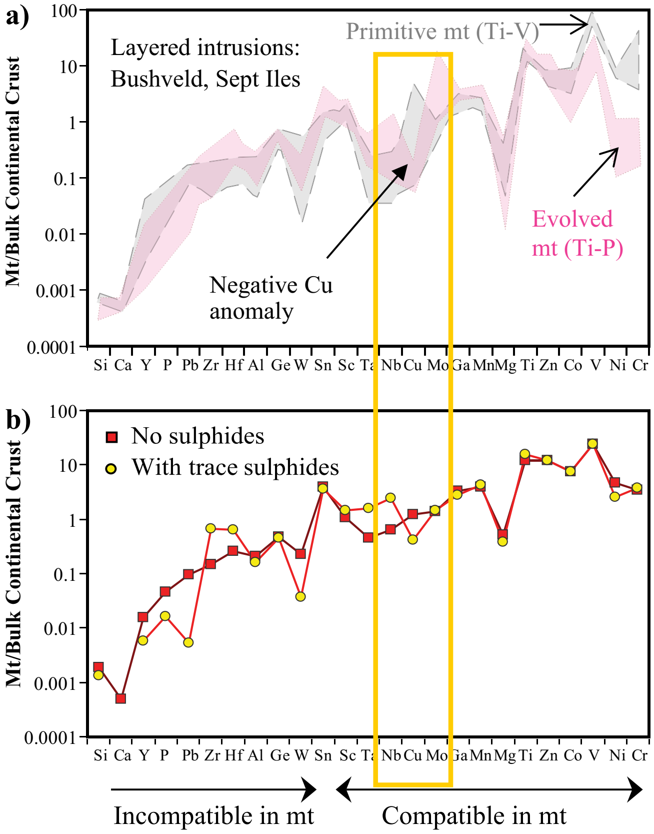

Figure 1. Multi-element diagrams for magnetite, normalized to bulk continental crust, from **(a)** layered intrusions barren of Ni deposits and **(b)** andesite lavas. Modified from Dare et al. (2014b).

most (Ti-P deposits) magnetite layers (Méric, 2011; Dare et al., 2014b). In contrast, elements incompatible during fractionation, such as Sn, Mo, and Zn, increase in Fe oxides, together with Nb, Ta, and Mn, even when trace amounts of immiscible sulphides formed (Méric, 2011; Dare et al., 2014b). This is because their partition coefficients into sulphide melt are not high enough (D~10–20) to deplete the silicate magma upon the crystallization of a small amount of sulphides. In the absence of sulphides, Cu is also incompatible and increases in the silicate melt during fractionation, whereas upon sulphide-saturation Cu becomes compatible and is depleted (Li et al., 2000). Recent experimental studies indicate that Cu should partition into Ti-rich magnetite (Simon et al., 2008). Analysis of Ti-rich magnetite in andesite lavas from Chile confirms that Cu can be present in solid solution in magnetite (30–100 ppm: Dare et al., 2014b). These andesite samples are sulphide-undersaturated due to the oxidized nature of magmas developed in a continental arc setting, which was calculated as +2 QFM (quartz-fayalite-magnetite buffer) for these samples (Dare et al., 2014b). On the multi-element diagram (Fig. 1b), Cu is positioned between Nb and Mo because these three elements have similar partition coefficients into magnetite from silicate melt (Dare et al., 2014b). For magnetite that crystallized from sulphide-undersaturated andesitic magma, the Cu, Nb, and Mo content of magnetite on the multi-element diagram define a flat line, i.e., there is no Cu anomaly. However, crystallization of magnetite may locally reduce andesitic magma and trigger formation of small amounts of sulphide droplets (Chiaradia et al., 2011). In this study, one of the andesite samples has small Cu sulphide inclusions in magnetite and as a result the magnetite has a small negative Cu anomaly (Fig. 1b). This is because the sulphide droplet has preferentially taken Cu from the silicate melt and thus depleted the magnetite in Cu (11 ppm). A large Cu anomaly is present in magnetite from the uppermost layers of the Sept Iles and Bushveld layered intrusions (Fig. 1a), which supports the observation that small amounts of immiscible sulphide formed in the lower parts of these intrusions. Thus a negative Cu anomaly is indicative of the formation of a small amount (cotectic proportions: 0.3 wt% S) of sulphide liquid in barren intrusions.

Ni and Cu Content of Fe-Oxide Minerals as Indicators of Fertility of Mafic Magmas

This study proposes that the most useful elements/element ratios to monitor the amount and timing of chalcophile depletion of mafic magmas, from which the history of sulphide saturation can be obtained, are the Cu anomaly ($Cu/Cu^*_N = 2Cu_N/(Nb_N + Mo_N)$) and Ni content of Fe oxides (both magnetite and ilmenite) plotted against Cr, which is used to monitor the degree of fractionation (Fig. 2). Fe oxides from all igneous complexes that are barren of significant Ni sulphide mineralization, but contain small amounts of sulphides, are plotted on these diagrams to define the field of barren intrusions (Fig. 2). During fractionation, the Cr content of magnetite and ilmenite decreases 4 orders of magnitude. However, there is a slight tendency for the negative Cu anomaly to increase, i.e., the value gets smaller during fractionation (Fig. 2a, b). This is most evident for magnetite from magnetite-rich intrusions (Fig. 2a). This is because Cu is depleted in the melt (and in the Fe oxides) upon sulphide saturation and therefore does not increase during fractionation, unlike Nb and Mo. Both Ni and Cr in magnetite and ilmenite decrease with fractionation with or without a small amount of sulphide removal (Fig. 2c, d).

The effect of the crystallization of different amounts of sulphide on the behaviour of chalcophile elements during fractionation is modelled in Figure 3. The cotectic proportion (0.3%) of sulphide was chosen to represent the maximum amount of sulphides forming in igneous complexes that are barren of significant Ni sulphide mineralization. A larger amount of sulphides (1%) was chosen for fertile igneous complexes with

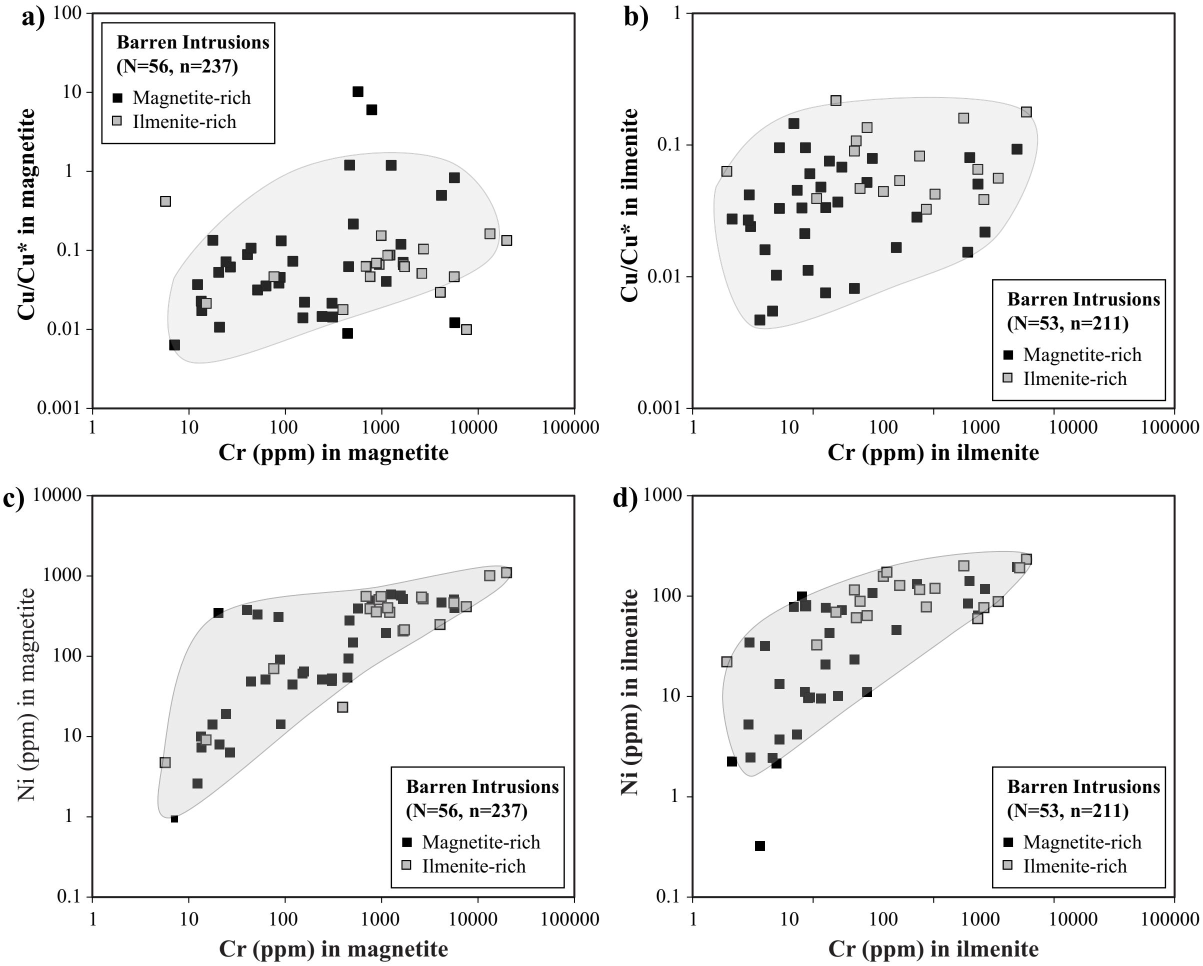

Figure 2. Fe-oxide data for Cu, Ni, and Cr in intrusions that are barren of significant Ni sulphide mineralization: the Cu anomaly (Cu/Cu*) versus Cr content of **(a)** magnetite and **(b)** ilmenite. Nickel versus Cr content of **(c)** magnetite and **(d)** ilmenite. Sources of data: Méric (2011), Néron (2012), Dare et al. (2014b), and Dare (unpublished).

significant Ni sulphide mineralization. Figure 3 indicates that the size and timing of Ni and Cu depletion should be different for barren and fertile magmas. As indicated above, the size and rate of metal depletion depends on the partition coefficient between sulphide and silicate magmas. In both models, the PGEs (D ≈ 10000) are the most rapidly depleted, followed by Cu (D ≈ 1000), then Ni (D ≈ 500). The remaining chalcophile elements (D ≈ 10 for Mo, Co, Pb, Sn, and Zn) show no significant depletion in the melt due to sulphide crystallization. As the crystallization of Fe oxides typically occurs late, after 40 to 70% of fractionation, they are ideal minerals to record significant depletion of Ni and Cu in the magma. Only a mineral that crystallizes early, such as chromite, is able to record depletion of PGE upon sulphide saturation (Locmelis et al., 2013; Pagé et al., 2015). Figure 3b illustrates that for fertile magmas, which either crystallized (or interacted with) a large proportion of sulphide, the depletion of Ni and Cu occurs earlier than in barren intrusions. Fe oxides that crystallize from a fertile magma after the same degree (40–70%) of fractionation (i.e. same Cr value) should therefore display significant depletion in Ni and Cu. Magnetite and ilmenite from fertile igneous complexes at Voisey's Bay and Sudbury were plotted on these new Fe-oxide diagrams to test this hypothesis (Figs. 4, 5).

Voisey's Bay

Ilmenite is the dominant Fe oxide in the troctolite and olivine gabbro units of the Eastern Deep Intrusion, which is host to part of the Voisey's Bay Ni sulphide deposit. Some samples are weakly mineralized but the majority are devoid of sulphide mineralization. Massive sulphide mineralization occurs towards the base of the intrusion and in the feeder dyke (Lightfoot et al., 2012). Ilmenite occurs as an intercumulus mineral in these primitive rocks and is thus Cr-rich (2000–100 ppm); however, magnetite is rare. Ilmenite displays an early Cu depletion (at high Cr values) and most of the data plot outside the barren field on the Cu/Cu* versus Cr plot (Fig. 4a), indicating a large and early depletion of Cu in the magma due to the formation of significant amounts of sulphide mineralization

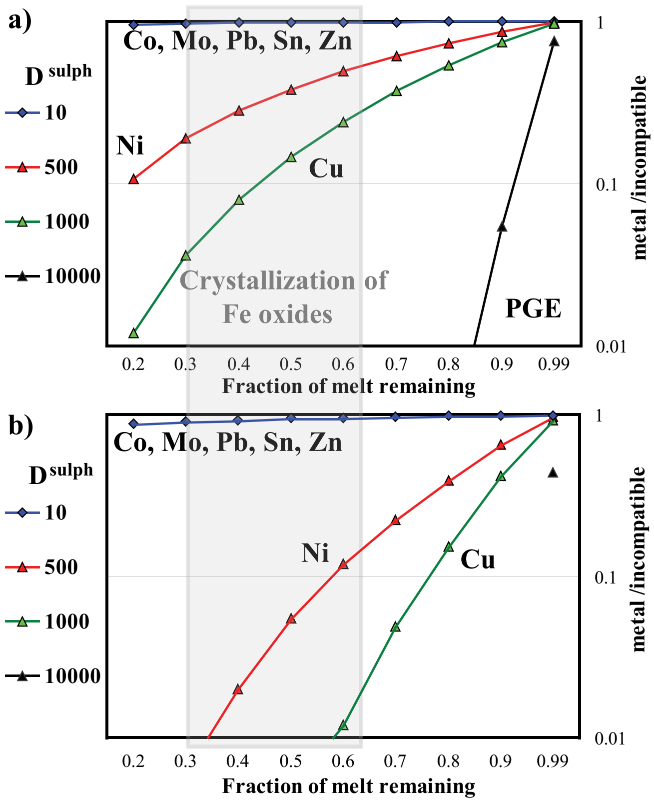

Figure 3. Element modelling of chalcophile depletion during fractionation of mafic magma with different amounts of sulphides. **a)** Crystallization of cotectic proportion of sulphide (0.3%), common in intrusions barren of Ni deposits. **b)** Crystallization of larger amounts of sulphide (1%), to simulate chalcophile depletion in fertile intrusions. The grey box indicates the typical 'window' of crystallization of cumulus Fe oxides (40–70% fractionation).

at depth. This result is supportive of the whole rock Cu/Zr geochemistry results of Li et al. (2000). On the Ni-Cr diagram, however, most of the ilmenite data plots within the barren field, with only a few plotting at lower Ni values, indicating that there was no significant Ni depletion (Fig. 4b) of the magma. This is also in agreement with the whole rock data (Lightfoot et al., 2012). For primitive rocks, that crystallize after 15 to 20% fractionation (Li et al., 2000), the model in Figure 4c indicates that there will be significant Cu depletion (1 order of magnitude difference) but less significant Ni depletion (only half order of magnitude difference).

Sudbury Igneous Complex

Both ilmenite and magnetite are present as cumulus and intercumulus minerals in the oxide-rich gabbro and underlying norite, respectively, of the Sudbury Igneous Complex. Samples were selected both above and away from known massive sulphide mineralization. Darling et al. (2010) showed that samples above mineralization are the most depleted in Ni and Cu (~65%) whereas samples away from mineralization are still significantly depleted (~45%). Both magnetite and ilmenite

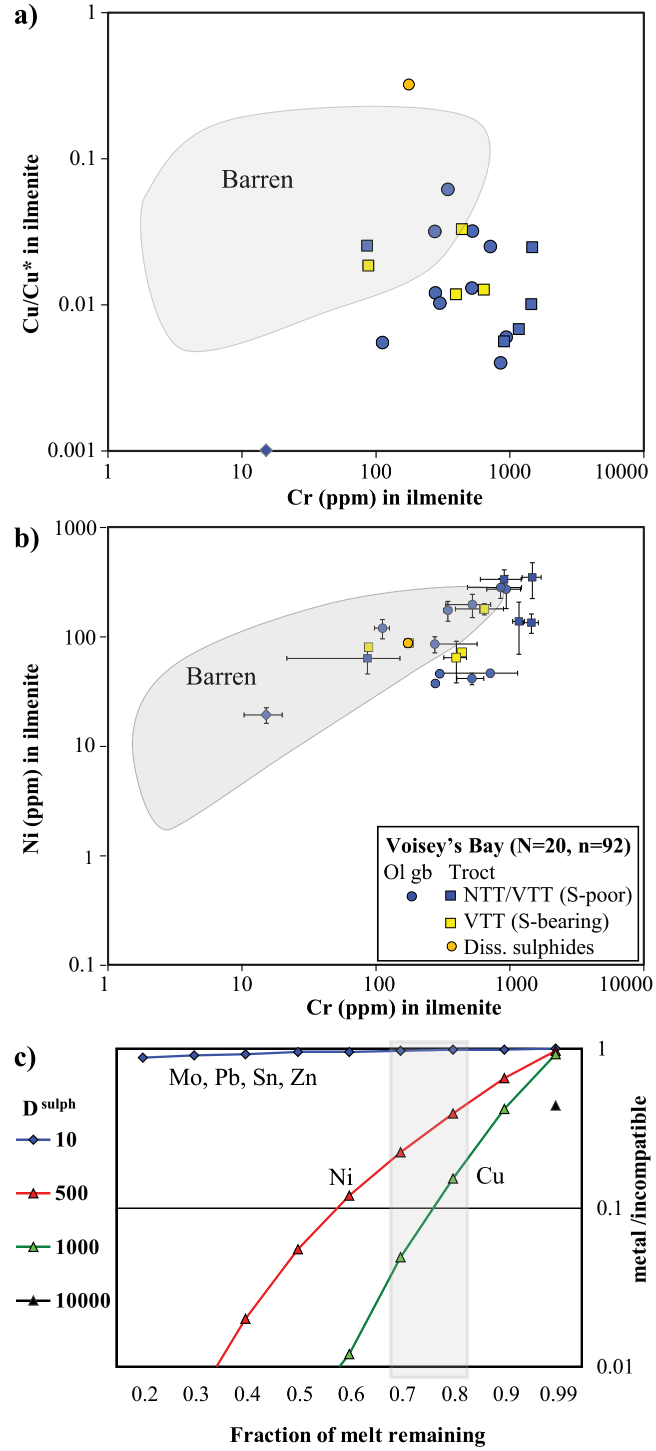

Figure 4. Results for ilmenite in troctolite (troct) and olivine gabbro (ol gb) from the fertile intrusion of Voisey's Bay Ni-Co deposit: **a)** Cu anomaly (Cu/Cu*) versus Cr; **b)** Ni versus Cr; **c)** model of the behaviour of the chalcophile depletion for fertile intrusion (same as in Fig. 3b). Legend for the rock types are given in (b), where NTT = normal-textured troctolite; VTT = variable-textured troctolite; and diss. sulphides = disseminate sulphides. The grey box in (c) indicates that the low degree of fractionation (15–20%) of the samples may explain the lack of Ni depletion recorded by ilmenite. N = number of samples; n = number of grains analysed.

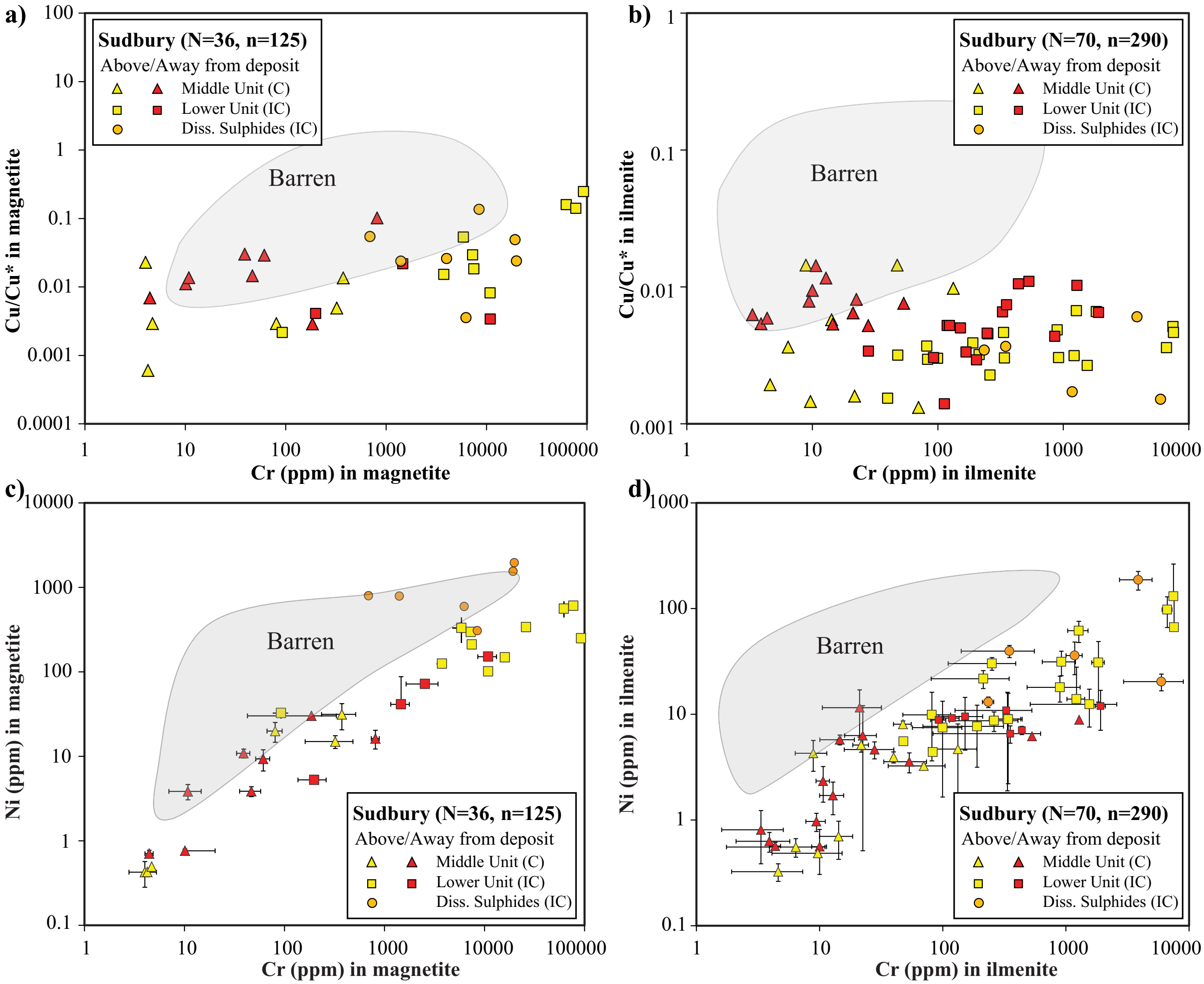

Figure 5. Results for Fe oxides from the Sudbury Igneous Complex, which is the world's largest Ni mining camp: **(a and b)** Cu anomaly versus Cr content of magnetite and ilmenite and **(c and d)** Ni versus Cr content of magnetite and ilmenite. Note that magnetite from disseminated sulphide samples have unusually high Ni contents, probably due to subsolidus re-equilibration with Ni-rich sulphide. N = number of samples; n = number of grains analysed.

have large negative Cu anomalies and as such most plot at lower values of Cu/Cu*, outside of the barren field (Fig. 5a, b), with the exception of a few more evolved samples collected away from known deposits. This indicates that the Fe oxides in the Sudbury Igneous Complex record a large and extensive Cu depletion of the magma that was present at the start of fractionation. Compared to ilmenite from Voisey's Bay, the depletion in Cu at Sudbury is an order of magnitude larger. On the Ni-Cr diagrams (Fig. 5c, d), all ilmenite data, and most of the magnetite data plot off the barren trend, at much lower Ni values. Thus the widespread Cu and Ni depletion recorded by the Fe oxides is in agreement with the whole rock geochemical data (Cu/Zr and Ni/Ni*: Lightfoot et al., 2001; Darling et al., 2010) and indicates that a large volume of sulphides efficiently collected the chalcophile metals from the overlying silicate melt. However, there is no significant difference in the chemistry of the Fe oxides in samples from directly above or away from mineralization.

Newark Island Layered Intrusion: Barren or Fertile?

This intrusion is part of the same Nain Plutonic Suite as Voisey's Bay; it was considered on a whole to be barren of significant Ni mineralization, based on whole rock data and past exploration (Lightfoot et al., 2012). Mafic samples were selected from the Newark Island intrusion specifically for this study to compare with those from the Eastern Deeps Intrusion at Voisey's Bay. They range in composition from olivine gabbro to gabbro but are slightly more evolved than the mafic-ultramafic samples from Voisey's Bay. As such, the Fe oxides are mainly cumulus phases: ilmenite is domi-

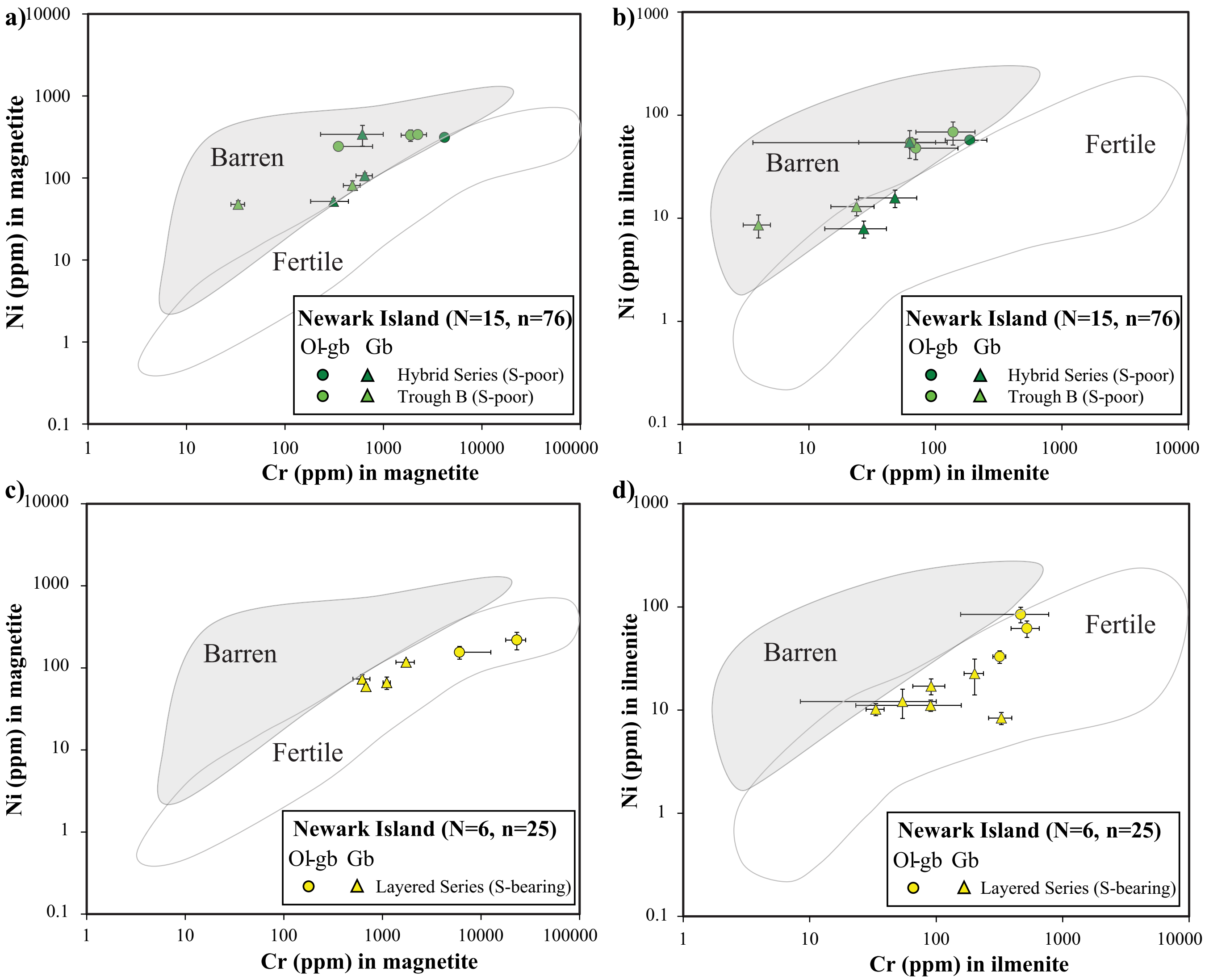

Figure 6. Results for Fe oxides from the supposedly barren Newark Island layered intrusion using Ni versus Cr diagrams. **a and b)** Magnetite and ilmenite from the upper part of the intrusion (Hybrid Series and Trough B) plot in barren field. **c and d)** Magnetite and ilmenite from the lower part of the intrusion (Layered Series) plot in the fertile field. N = number of samples; n = number of grains analysed.

nant but some magnetite crystallized shortly afterwards, followed by pyrrhotite (Wiebe and Snyder, 1993). Iron oxides from the upper part of the layered intrusion (Hybrid and Trough series) plot entirely in the barren field on the Ni-Cr diagram for magnetite and mostly in the barren field for ilmenite (Fig. 6a, b). In contrast, samples from the lower part of the intrusion (Layered Series) are relatively depleted in Ni and plot entirely in the fertile field defined by Sudbury Igneous Complex Fe oxides. A close inspection of the Ni-MgO whole rock data of Lightfoot et al. (2012) for these two series agrees with the results from the Fe oxides, showing no Ni depletion for the Hybrid Series and some Ni depletion for the Layered Series (Fig. 7). This indicates that the lower part of the intrusion is potentially fertile. However, this cannot yet be determined as the lower part of the intrusion is not exposed onshore. It is noteworthy to highlight that the fertile field for Fe oxides, although defined by Sudbury Igneous Complex samples that were formed by melting of the continental crust, can also be applied to evaluate the fertility of mantle-derived melts.

IMPLICATIONS FOR EXPLORATION

The examination of Fe-oxide geochemistry in fertile Ni-Cu-PGE environments, compared to barren environments, provides critical knowledge on the sulphide saturation and segregation history of the intrusion. We have shown that sulphide saturation is recorded by Ni and Cu depletion in Fe oxides. Significantly, fertile intrusions can be distinguished from barren intrusions by the size and timing of Ni and Cu depletion in relation to fractionation of the silicate magma. The mineral chemistry results are in agreement with whole-rock

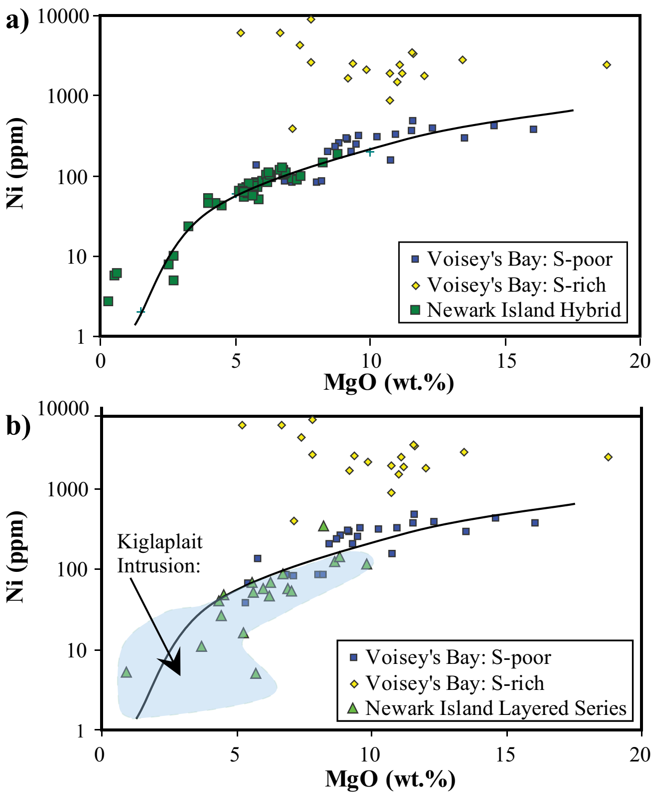

Figure 7. Whole rock Ni versus MgO plots for Nain plutonic suite rocks: a) Voisey's Bay (fertile) and Newark Island Hybrid Series (barren) both show no Ni depletion; b) Newark Island Layered Series shows similar Ni depletion to that from the nearby Kiglaplait intrusion. Data from Lightfoot et al. (2012) and Dare et al. (2014a).

data. The advantages of using Fe-oxide chemistry are that they are resistant to alteration and are commonly preserved and easily recovered from till and sediments. This new mineral technique provides a novel approach for determining the potential fertility of an intrusion for Ni-Cu-PGE deposits in support of exploration targeting.

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