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SEM-based quantitative mineralogical analysis of peridotite, kimberlite, and concentrate

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Quantitative automated mineralogy is the acquisition of mineralogical and textural data by scanning electron microscopy-based energy dispersive analytical methods. The technique is used in the metals and energy resource extraction industries to provide accurate mineral characterization information over large data sets. We have employed this method to three projects related to diamonds: mantle peridotite, kimberlite, and garnet concentrate. The first example assesses the metasomatic clinopyroxene-phlogopite modal mineralogy in peridotite xenoliths from Premier/Cullinan (South Africa). Understanding mantle mineral variability can be coupled with measureable mineral properties to develop mantle geophysical and geochemical models. The second example compares kimberlites from Letšeng Satellite Pipe (Lesotho) and Ngamiland (Botswana), to assess the variability of kimberlite mineralogy. Kimberlite domains can be identified on a micro scale with potential to understand parameters such as hardness and grindability, or to identify kimberlite clusters with discrete mineral assemblages. The third example applies mineral compositional variability in garnet concentrate samples as a possible tool for evaluating exploration projects. Application of garnet definitions to concentrates through digitally grouping grains into discrete populations results in improved understanding of large sample populations and hence diamond prospectivity. Quantitative mineralogy applied to diamondrelated projects utilizes the principles of geometallurgy in evaluating large data sets for greater understanding of the variability of mantle materials.

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1. Introduction

Quantitative mineralogy based on scanning electron microscopy (SEM) has been in development for over 20 years, primarily as a tool to understand mineral liberation and element deportment in metal mining projects. The method is used to understand mineralogy, texture, mineral associations, the presence of gangue minerals and deleterious elements that may potentially interfere with mineral processing and planning, and the overall impact of mineralogy on grinding and flotation processes [\(Gottlieb et al., 2000; Kendrick et al.,](#page--1-0) [2003](#page--1-0)). SEM-based quantitative mineralogy also is a key component of geometallurgy (geomet) studies, wherein full geological and mineralogical characterization of materials is linked to metallurgical response to optimize the value of resource projects [\(Hoal et al.,](#page--1-0) [2006; Grguric and Riley, 2006; Hoal, 2008](#page--1-0)). Other applications include the oil and gas industry where porosity and fracture distribution related to fine-grained clay mineralogy influence reservoir characterization for oil shales and carbonate reservoirs [\(Butcher et al., 2000](#page--1-0)), the coal, cement, and fly ash industries ([Liu et al., 2005; Ho-Tun, 2001](#page--1-0)), environmental, wastewater, dust, soil and precipitate studies [\(Camm](#page--1-0)

[et al., 2005](#page--1-0)), planetary materials [\(Rickman et al., 2008; Appleby et al.,](#page--1-0) [2008\)](#page--1-0), and viticulture and forensics ([Pirrie et al., 2004\)](#page--1-0). In each of these fields, the ability to quantify the mineralogy and texture of materials results in improved understanding of the geological processes of formation and development of key processing technologies.

For diamond deposits, geomet refers to identifying the important parameters in a deposit, such as diamond grade, recovery, kimberlite hardness, grindability, and mineral abundances. The distribution and alteration of megacrysts, the degree of silicification or serpentinization, and the presence or absence of clays, for example, can directly impact the processing and breakage characteristics of the various volcanogenic phases of a kimberlite and the final recovery of diamonds. The degree to which these variables can be predicted will impact the final risk and cost effectiveness of a project ([Hoal, 2008](#page--1-0)). This application was described by [Benvie \(2007\)](#page--1-0), who discussed the effectiveness of quantitative mineral characterization techniques at 15 μm pixel resolution for kimberlite from the Venetia mine, South Africa. The present paper expands on [Benvie's \(2007\)](#page--1-0) contribution by presentation and discussion of three further applications. By paying particular attention to characterization and statistical modeling of mineral assemblages at micron-scale spatial resolution, current SEMbased mineralogy techniques enable false-color imagery and quantification of the comparatively fine-grained and diverse mineralogical

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components encountered in projects concerned with kimberlites, diamonds and related mantle materials. The examples chosen for characterization in this investigation include metasomatized mantle peridotite xenoliths from Premier, the groundmass mineralogy of kimberlites from Lesotho and Botswana, and garnet xenocrysts from South African kimberlites and Botswana exploration samples.

2. Analytical methodology

The development of SEM-based automated quantitative mineralogy had its roots in the 1980s at Australia's Commonwealth Scientific and Industrial Research Organisation (CSIRO). Two systems were developed: one is the QEM-SEM system that developed into the QEMSCAN, produced by Intellection until the end of 2008 and now by FEI Corporation. The other is the Mineral Liberation Analyzer (MLA), developed by JKTech at the University of Queensland. Both systems were developed to provide statistically valuable mineralogical data sets for the metals mining industry, for the purpose of optimizing metallurgical and mining operations. The ability to quantify mineral distributions, textural relationships, and paragenetic associations in a variety of materials (cores, blast hole products, concentrates, plant feed, leach residues, tails) enabled improved monitoring and prediction of metallurgical recoveries, grinding behavior, and operating costs. Several generations of instruments have been used over the past three decades. The present study employed the QEMSCAN® system housed at the Advanced Mineralogy Research Center at Colorado School of Mines. The system combines a fully automated Carl Zeiss EVO5O scanning electron microscope platform with four Bruker silicon-drift energy dispersive (EDS) X-ray detectors, an energy resolution of 133 eV (Mn $K\alpha$), a four-quadrant solid-state backscatter electron detector, a secondary electron detector, and 1000-count combined Xray counts per determination. Standard analytical operating conditions are Peltier cooling (no liquid nitrogen), an accelerating voltage of 25 kV, a specimen current of 5 nA on the Faraday Cup, and a working distance of ~24 mm. The beam diameter is typically 0.25–0.5 μm. The four EDS-detector array allows for fast acquisition of data (~500 kcps combined on Au) and enables the automated analysis of large sample populations to deliver statistically reliable data sets. iMeasure software automates the stepping of the electron beam across samples at a userdefined pixel resolution. The resolution of the system is limited by the beam diameter of about 0.5 μm, and analyses are typically made at 2– 20 μm. At each pixel location, the system collects a backscattered electron (BSE) signal and EDS spectrum and correlates them with Xray and BSE count-based mineral definitions developed for the project. The system is not a wavelength-dispersive technique, and thus is not as useful as electron microprobe analysis for discriminating trace element abundances in minerals. Minor elements can be used to define differences in mineral or phase composition if standards are employed or if appropriately long count rates (e.g. 1,000,000 counts) and resolution (e.g. 2 μm) are used, depending on the material and project purpose. The system should be viewed as a mineral mapping and quantification tool, providing modal mineralogy and textural information in quantifiable data sets. As such, it should be used to complement other data sets such as procured by electron microprobe or X-ray diffraction. The mineral definitions and correlations on the basis of Xray counts are routinely assessed through duplicate sample analysis and quality assurance and quality control (QA/QC) steps to ensure data quality and accurate representation of the materials. The product is a false-colored image of the specimen with a large data set of digital information that can be queried and displayed depending on project requirements.

3. Mantle peridotites

The mantle peridotite study was aimed at portraying the distribution of mineral phases in metasomatised mantle xenoliths. Discrete peridotite samples were collected from the Premier (Cullinan) kimberlite mine, South Africa, from 1990 to 1992. Previous studies [\(Olson and Erlank, 1993; Hoal, 2003](#page--1-0)) documented the metasomatic assemblage of Fe-phlogopite, phlogopite-diopside, and in few occurrences phlogopite-pargasitic amphibole [\(Hoal, 2003\)](#page--1-0). This assemblage differs from the metasomatic mineral assemblages typically attributed to other southern African metasomatised peridotites, which are more commonly characterized by Mg-phlogopite and K-richteritic amphibole ([Erlank et al., 1987](#page--1-0)). The Fe–Al signature of the Premier suite has been attributed to mantle metasomatism accompanying emplacement of the Bushveld Igneous Complex at about 2.05 Ga ([Hoal, 2003\)](#page--1-0), as underscored by recent Re–Os isotopic evidence [\(Richardson et al., this](#page--1-0) [issue\)](#page--1-0). The goal of this study was to image and quantify the distribution of phlogopite and phlogopite-diopside metasomatic assemblages, and to determine the modal mineralogy. Samples analyzed were circularcut thin sections previously gold coated and analyzed by ion microprobe. The QEMSCAN analyses were made using a 5-micron pixel resolution.

Fig. 1 shows false-colored images, area percent calculations, the mineral list employed, and modal proportions for a spinel lherzolite and a phlogopite garnet lherzolite from Premier. These two samples illustrate different textural relationships of clinopyroxene and clinopyroxene-phlogopite with olivine, and serpentine-veined olivine. In addition to having the advantage of the false-colored images and mineral maps, we have calculated the area percent of each mineral phases (volume and mass percent also can be calculated), as well as displaying the modal abundances as a single bar chart. This example demonstrates the spatial distribution of clinopyroxene and phlogopite over a defined area of a thin section, and provides quantitative modal abundance data. Information gained from this study augments mineralogical results obtained by optical microscopy and by electron microprobe, and provides a quantification of the mineralogy for application to, for example, geophysical models, that would be difficult to do on

Fig. 1. False-colored image of a spinel lherzolite (above) and phlogopite garnet lherzolite (below) from Premier. A thin section photomicrograph of the phlogopite garnet lherzolite sample is shown for comparison. The color-coded legends show the mineral lists used for the samples. The mineral abundance determined (by area) is listed and illustrated by a horizontal bar chart. Note the textural variability illustrated in the two samples, reflecting different degrees of modal metasomatism. Clinopyroxene occurs interstitial to olivine in the upper image, whereas in the lower image it occurs both as discrete grains and locally as intergrowths with phlogopite.

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