Contents lists available at ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

The application of automated electron beam mapping techniques to the characterisation of low grade, fine-grained mineralisation; potential problems and recommendations

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ABSTRACT

Automated electron beam techniques using instruments such as the scanning electron microscope (SEM) and the electron probe micro analyser (EPMA) are commonly used by the mineral and metallurgical processing industries to characterise ore and feed materials. The correct choice of instrument and operating conditions is essential, yet often overlooked, and depends on parameters such as composition, particle size, heterogeneity and physical competency of the sample being examined, as well as the type of data sought.

In this study, a thorough understanding of the analytical capabilities and the operating advantages and limitations of each instrument are discussed to evaluate and establish the application suitability. A case study is presented comparing FEG-EPMA and QEMSCAN® technologies using a sample of fine-grained (micron scale), disseminated, calcrete-hosted uranium mineralisation. The paper demonstrates how differences between results from the two instruments are principally the consequence of the operating setup and data processing, rather than instrument capability. Each instrument has a different best practice application and when used correctly (independently or together) can provide information of unparalleled quality. Both are excellent choice tools for characterisation.

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1. Introduction

As the discovery of new, large, high-grade ore deposits declines, it is becoming increasingly important to maximise ore extraction from existing reserves in order to meet global resource demands. There is also a growing need to establish economically viable methods to extract ore from presently sub-economic resources. Thus, the development of processing methods to extract, low-grade, fine-grained mineralisation is paramount. In doing so ore cut-off grade thresholds will be lowered and the loss of fines to tailings will be reduced; this will increase the amount of ore extracted and the economic return of a deposit, and reduce the potentially adverse environmental effects of metal accumulation in waste tailings.

Low grade, fine-grained ore mineralisation (<5 μm) is particularly challenging to characterise. Commonly used characterisation techniques (e.g. optical microscopy, X-ray Diffraction [XRD], X-ray Absorption Spectroscopy [XAS], X-ray Fluorescence [XRF] and Scanning Electron Microscopy Energy Dispersive Spectroscopy [SEM-EDS]) can be ineffective when concentration levels are low, and may fail to report quantitative composition data or are unable to provide adequately high resolution image results to clearly distinguish ore textures and relationships. Furthermore, many of the analytical techniques available to study ore mineralogy are often manually operated. For research studies that apply a "search, find and identify" approach to mineralogy, such techniques may be suitable. However, for studies that require large numbers of samples to be analysed rapidly and repeatedly in order to generate statistically rigorous datasets, manually operated instruments are not appropriate and automated mineralogy techniques are preferred. By comparison, automated sample analysis offers several advantages, principally, improved efficiency when acquiring data and the elimination of potential operator bias, error and/or fatigue (Fandrich et al., 2007; Pirrie and Rollinson, 2011).

Automated mineralogy techniques that quantitatively report phase compositions, modal proportions, textures and fabric relationships between mineral grains have been available since the mid 1970s (Grant et al., 1976; Grant and Reid, 1981). The systems were initially developed based on a scanning electron microscope

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(SEM) platform (Huggins et al., 1982; Reid et al., 1984; Pignolet-Brendom and Reid, 1988; King and Schneider, 1993; Robinson, 1998; Robinson et al., 2000; Gottlieb et al., 2000; Gu, 2003) but have since been expanded to include electron probe micro-analytical (EPMA) instruments (Bernard et al., 1986; Mainwaring and Petruk, 1986; Harrowfield et al., 1993). Both are capable of simultaneous, high resolution imaging and composition analysis. However, the ability of either automated technique to characterise fine scale mineralisation varies depending on the physico-chemical properties of the sample mineralogy and the features, capabilities and operating limitations of the instrument (Rowlands, 1985).

This paper evaluates the use of two automated electron-beam based mineral mapping techniques; quantitative evaluation of minerals by scanning electron microscopy – QEMSCAN® (Gottlieb et al., 2000) and automated electron probe micro-analysis – EPMA mapping (Harrowfield et al., 1993; Pownceby et al., 2007). From the array of electron beam microscopes that have been developed there is a spectrum of features and capabilities. Every instrument is different, able to deliver unique benefits. These two instruments contrast well together; in fact, it might be considered that they fall at opposite ends of the spectrum. A compare and contrast approach that analyses the same samples using each technique highlights the differences and identifies the similarities between these geochemical analytical tools.

An overview of the operating parameters of each instrument is presented followed by a case study to demonstrate the similarities, differences and limitations of both techniques. The case study addresses the characterisation of micro-scale mineralisation in a calcrete-hosted uranium (CHU) ore using a field emission gun electron probe micro-analyser (FEG-EPMA) and QEMSCAN®. The results have been evaluated based on the instrument capabilities with recommendations suggested for operating condition set-up, data acquisition procedure and technique application suitability.

2. Electron-beam microscopy: SEM and EPMA instruments

The SEM and EPMA instruments are broadly similar in terms of their functional capabilities. In the simplest terms, they are microscopes that use an electron beam to excite a sample surface. The sample-beam interaction generates electrons (secondary, back scatter and Auger electrons) and photons (X-rays and cathodoluminescence) that are used to image the textures and determine the composition of different minerals within the sample. Although the capabilities of the instruments are similar, each was designed for a different purpose. The SEM was developed principally as an advanced, high resolution imaging instrument, while the EPMA was developed for improved, element specific, low-level detection, chemical sample analysis. The key functional characteristics of the two instruments are described in detail.

2.1. The scanning electron microscope

Traditionally, a SEM is used to examine three-dimensional objects providing higher magnification and greater depth of field than can be achieved under an optical microscope (Trimby and Prior, 1999). The instrument is primarily used for examining surface topography and average atomic number imaging (Goldstein and Yakowitz, 1975; Goldstein et al., 1981; Newbury et al., 1987). Using this information, phase distributions and relationships can be examined. Qualitative and quantitative X-ray analysis (Be to U) is used to identify the major and minor elements. The chemical resolution is usually greater than 0.5 wt% for the elements Na to U, while lighter elements Be, B, C, N, O and F generally need to be present at percent levels (>1 wt%) (Pownceby and

MacRae, 2011). Applications of scanning electron microscopy include: backscattered electron (BSE) compositional imaging; composition mineral analysis by X-ray spectroscopy; X-ray mapping for phase distribution; structural and compositional characterisation by cathodoluminescence (CL); and structural analysis via electron backscattered diffraction (EBSD). Each method requires the standard SEM to be fitted with specialised additional features.

A standard SEM instrument can be adapted to perform automated mineral characterisation tasks. This has led to the development of several SEM instrument-based capabilities, e.g. QEMSCAN® (Gottlieb et al., 2000), Mineral Liberation Analysis – MLA (Gu, 2003), RoqSCAN (Oliver et al., 2013), INCAMineral (OINA/INCAMinerals/0312), Zeiss Mineralogic System (www.zeiss.com/mineralogic) and the TIMA (Tescan Integrated Mineral Analyzer) (Ortolano et al., in press). Compared with a standard SEM, a QEMSCAN® (or similar instrument) is an automated capability that operates using multiple energy dispersive X-ray (EDX) detectors, which allow the X-ray signal to be acquired more rapidly. The image and X-ray data are computationally processed using sophisticated proprietary image analysis software usually supplied with the instrument.

2.2. The electron probe microanalyser

The EPMA has been engineered to provide quantitative X-ray analysis at major, minor and trace element levels (McKinley et al., 1966; Mead, 1969; Birks, 1971; Keil, 1973; Smith, 1976, 2003; Reed, 1990, 1993). It is a highly automated instrument, equipped with one or more wavelength dispersive X-ray (WDX) spectrometers. The key advantages of WDX spectrometry over EDX spectrometry include a large count throughput and superior spectrum peak resolution (a few eV for WDS compared to ~100 eV for EDS), meaning that peak overlaps are much less of a problem and peak to background ratios are improved. This leads to lower detection limits (~2 ppm) (Pownceby et al., 2007) and improved element specific analytical precision. An EPMA is built with an integrated optical microscope and may be fitted with a conventional EDX spectrometer. On more modern instruments one or more silicon drift detectors (SDD) may replace EDX detectors and a cathodoluminescence (CL) detector (MacRae and Miller, 2003). In the past, the image resolution from an EPMA has not been as good as a SEM. However, this has been addressed with the advent of the warm Field Emission Gun (FEG) equipped electron microprobe where electrons are sourced from a FEG rather than a tungsten filament. The FEG allows a finer, more precise electron beam to be generated.

The FEG-EPMA provides superior image chemical and spatial resolution (down to 50–100 nm) and operates to lower detection limits (in the range of a few hundred parts per million) compared to a conventional tungsten based SEM or EPMA (Pownceby and MacRae, 2011). The inbuilt WDS of the FEG-EPMA (a defining feature), puts the instrument at the forefront of micro electron-beam techniques that are able to analyse solid solution phases and sub-microscopic particles. The FEG-EPMA technique has also been developed for automated, high resolution sample mapping. In current systems, integrated software for image analysis is limited and users often develop their own or use off-the-shelf software to process data (e.g. Harrowfield et al., 1993; Kotula et al., 2003; Wilson and MacRae, 2005; Wilson et al., 2010).

3. Automated SEM and FEG-EPMA operating conditions

The setup for sample mapping using automated SEM (QEMSCAN®) and FEG-EPMA instruments is broadly similar; it is possible to select the same operating condition parameters on both