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**SCANNING ELECTRON BEAM-BASED AUTOMATED MINERALOGY - OUTLINE
OF TECHNOLOGY AND SELECTED APPLICATIONS IN THE NATURAL
RESOURCES INDUSTRY**

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1. ABSTRACT

This paper provides an up to date review of developments and applications of scanning electron beam-based automated mineralogy, with particular reference to the natural resources industry. Two main commercial products dominate the market, namely MLA (Mineral Liberation Analyzer) and QEMSCAN[®] (Quantitative Evaluation of Minerals by Scanning Electron Microscopy). Their basic operating principles are compared and contrasted with reference to system design, data acquisition, X-ray analysis, and phase identification. Examples are provided for the use of automated mineralogy for ore characterization and precious metal searching within the mining industry, and analysis of cuttings from oil and gas wells from the petroleum industry. A final example from the industrial minerals market is provided, covering the application of automated mineralogy to cement characterisation.

2. INTRODUCTION

In recent years, scanning electron beam-based quantitative mineralogy tools have advanced rapidly with improved SEM and X-ray detector hardware, and the development of sophisticated and automated image analysis methods. Automated mineralogy has now established itself as an essential enabling technology for the reliable acquisition of statistically valid mineralogical data from particulate samples, sections of rock and drill core. This has had a revolutionary effect on the industrial use of such data in the study of geology, mining and mineral processing. Previously, using manual methods, it was not feasible to attempt this work because the large data sets required could not be assembled in a realistic timescale. The speed, reliability, and repeatability of the modern automated measurements have now made this type of analysis routine.

More than 120 automated mineral analysers have been installed around the world. The installed base exclusively consists of QEMSCAN[®] and MLA. Both have an equally sized customer base and perceived to be strong in distinct areas of measurement and applications. The QEMSCAN[®] technology is part of FEI since December 2008 and will play a key role in further development of Automated Mineral Systems at FEI. The Australian mineral technology specialist JKTech and FEI are partners in MLA. The automation software, designed by JKTech, controls the SEM hardware developed and manufactured by FEI. MLA products are based on the FEI Quanta series and include configurations with tungsten filament and field emission sources, as well as with various sizes of sample stages.

Automated mineral analysers in the natural resources industry were originally developed as diagnostic metallurgical tools in mining to improve mineral processing plants using samples drawn from plant surveys and pilot-scale tests. They were used mainly for the assessment and auditing of size fractions of mill products, such as concentrates and tailings from base and

precious metal ores [1]. Subsequently, they began to be used for concentrator design and optimisation where ore characterisation was used to help understand the relationship between the feed ore and its subsequent behaviour in the plant (Fig. 1).

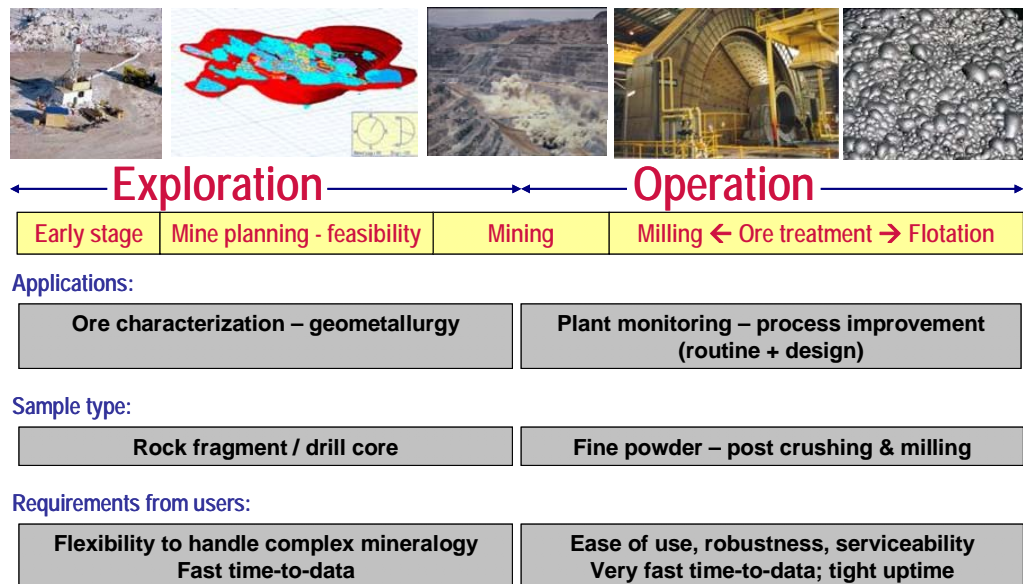


Figure 1. Range of main applications of automated mineralogy in the mining industry, from early exploration to ore characterisation to plant monitoring during ore treatment.

They are now widely used to measure exploration cores and drill chips from drilling programs to predict eventual metallurgical performance. Ore samples can be classified into metallurgical, as well as geological types, by combining mineralogical features in the ores with metallurgical properties related to grade control, separation, comminution, flotation, and leaching behaviour. Three-dimensional block models of ore deposits are now being created based on processing response, in addition to geological and mining properties.

The application of automated mineralogy in oil and gas exploration and production is a new area, and centres on the underlying core capability of SEM-based automated mineral analysers being able to quantify rock type and texture from microscopic examination of materials returned from drilling activities. Other applications include coal, cement, industrial minerals and planetary materials.

This paper describes the technology behind automated mineral analysers. It further provides case studies on the following applications of automated mineralogy in natural resources: (1) ore characterisation of base metal ores; (2) concentrator plant management of precious metal ores; (3) oil and gas; and (4) cement.

3. BASIC PRINCIPLES OF AUTOMATED MINERALOGY

3.1. System configuration

An automated mineral analyser typically consists of a scanning electron microscope (SEM) equipped with multiple energy-dispersive X-ray (EDS) detectors. The analyzer is typically used as a complementary technique to X-ray diffraction and the electron probe microanalyzer. An electron backscatter diffraction (EBSD) detector and a wavelength-dispersive X-ray spectrometer (WDS) may be added for specific requirements. Automation software controls the SEM hardware to quantitatively analyse mineral and material samples. The system has the ability to measure up to 16 sample blocks overnight without the need for operator assistance. Automated stage control and image acquisition allows for BSE (backscattered electron) imaging and subsequent X-ray analysis of several thousand particles within the time span of around one hour, depending on sample-type and mineral texture.

3.2. System design approaches

Different approaches to mineral identification have been used in electron microscope based systems, ranging from BSE based systems to X-ray dominated systems [2-4]. Although both MLA and QEMSCAN[®] (formerly QEM*SEM) technologies have uniquely evolved from rather distinct philosophies, the principles behind them are essentially the same in that they use backscattered electron image analysis and X-ray mineral identification to provide automated quantitative mineral characterisation. The MLA exemplifies a BSE based system where the information obtained from the acquisition of a backscattered electron image is fundamental to the nature of the follow-up X-ray analysis [5]. The QEMSCAN[®] is optimized for X-ray throughput and proves to be beneficial mainly on samples that require full X-ray mapping [6]; also see Fig. 2 for the process of data acquisition.

3.3. Automated mineral analysers – data acquisition technologies

Automated mineral analysis involves the setting of particles into a mould (typically 30 mm diameter) with epoxy resin to form a hardened block. Typical particle sizes range from 5 μm to 3 mm and should preferably be of a defined narrow size fraction. The block is then ground down to expose a representative cross-section of particles which is subsequently polished, and then coated with carbon before being presented to the SEM.

Various factors come into play when deciding on the measurement parameters for a particular analytical run using an automated mineral analyser. In virtually all cases, fast time-to-data requirements have to be counterbalanced against the need for high image resolution from BSE acquisition, as well as energy resolution from X-ray collection. The effect of pixel resolution to mineral classification results are shown in Fig. 3. The choice of image and pixel X-ray resolution is user-defined and depends on application requirements.

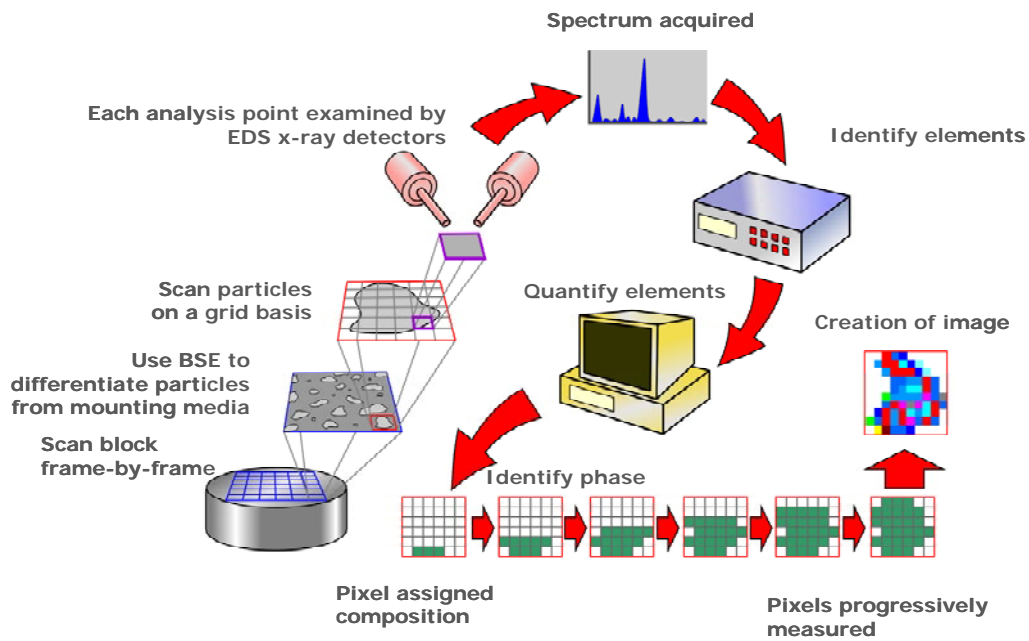


Figure 2. Flow diagram of data acquisition using QEMSCAN[®] technology.

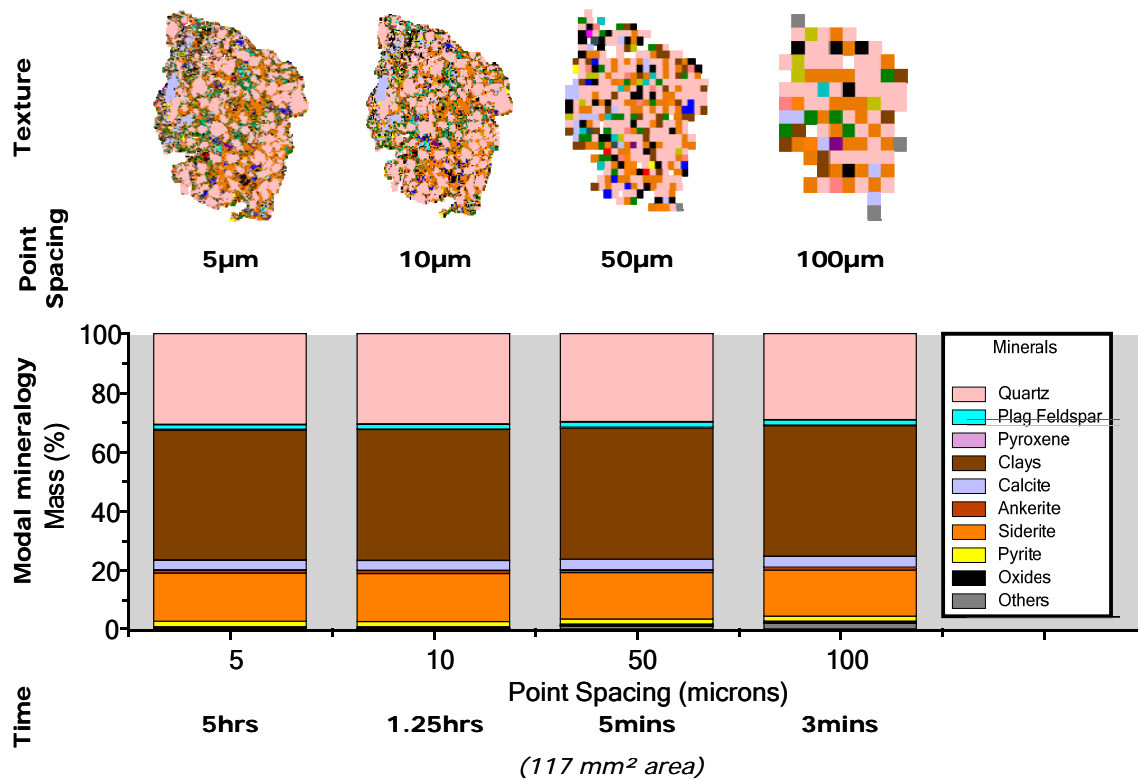


Figure 3. Effect of pixel resolution to mineral classification.

Apart from resolution settings, fast image acquisition can be obtained from short dwell times and low magnification so that fewer frames are needed to cover the surface of the sample

block. Fast X-ray acquisition is also achieved by higher sample current from higher KV, larger spot sizes and other optimisation of settings on both the SEM tool and EDS detector. Fast X-ray acquisition times of a 1000 to 2000 photon count spectrum in 1 - 2 ms per analysis point can be achieved and still yield sufficient counts for reliable mineral classification (Fig. 4). Applications are presented in this abstract where both requirements for analytical throughput and image resolution are taken into account.

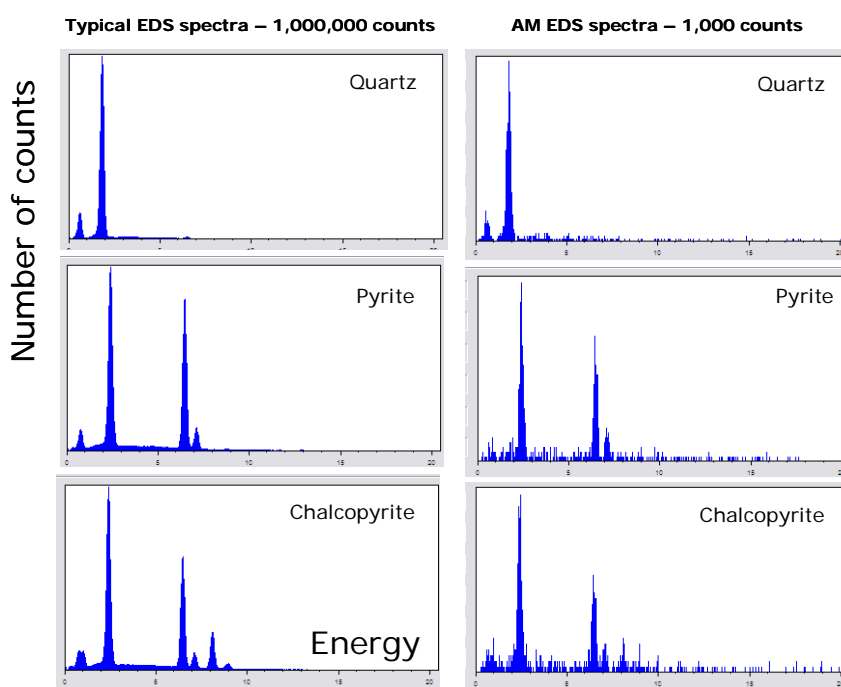


Figure 4. EDS spectra from automated mineral analysers (typically 1000 - 2000 counts; right) compared to typical EDS spectra (1 million counts; left) used for quantification.

Particulation involves background removal and particle de-agglomeration whilst segmentation defines regions of homogeneous backscatter brightness within each individual particle. Both processes normally precede the X-ray analysis technique. Both systems use various X-ray analysis techniques to identify mineral species: point X-ray, linear X-ray and full X-ray mapping [5-7].

3.3.1. Point X-ray analysis

Point X-rays can be performed for each grey level region identified within a segmented particle. The spectrum is collected at the centre of a phase to avoid contamination from bordering phases and hence acquire the “cleanest” spectrum possible. This spectrum is linked to its corresponding particle and grain in the segmented image to generate an X-ray image (Fig. 5). Both the MLA and QEMSCAN® offer the option of collecting a single “average”

spectrum over the entire area of a segmented grain as opposed to the spot analysis above. Alternatively, an X-ray map may be performed when there is the possibility of two or more associated minerals having the same Average Atomic Number and therefore BSE grey level (see 2.3.3).

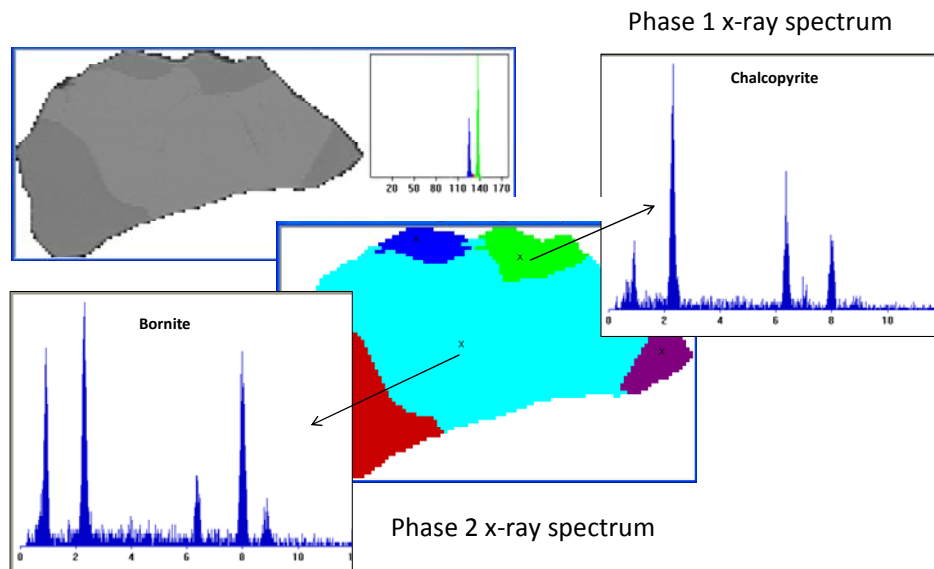


Figure 5. Segmentation of a composite particle. A spot X-ray analysis is performed on the centre of homogeneous grey levels.

Another variety of point X-ray analysis involves the searching of BSE images for phases of interest by using a grey level or X-ray standard trigger to filter out sparse phases (usually value minerals such as copper sulphides) or rare bright phases containing precious metals (e.g., gold, platinum, silver and uranium). This measurement technique subsequently performs single X-ray analyses in the centre of each unique grey level on both the phase of interest as well as its surroundings with the aim to establish mineral liberation and associations (Fig. 6).

Point X-ray analysis can also be performed on a grid of widely spaced points on widely spaced lines (Fig. 7). This mode uses BSE imaging to discriminate particle matter from background and then collects one X-ray spectrum from each grid point across the particle. This very rapid technique provides mineralogical data for each phase identified and produces volume percentages of the mineral components of the sample.

3.3.2. Linear X-ray analysis

Minerals are identified at closely spaced points on widely spaced lines. This technique provides all modal mineralogy information obtained in a point scan, but also indicative particle and grain size distributions as well as mineral associations at contact areas (Fig. 8).

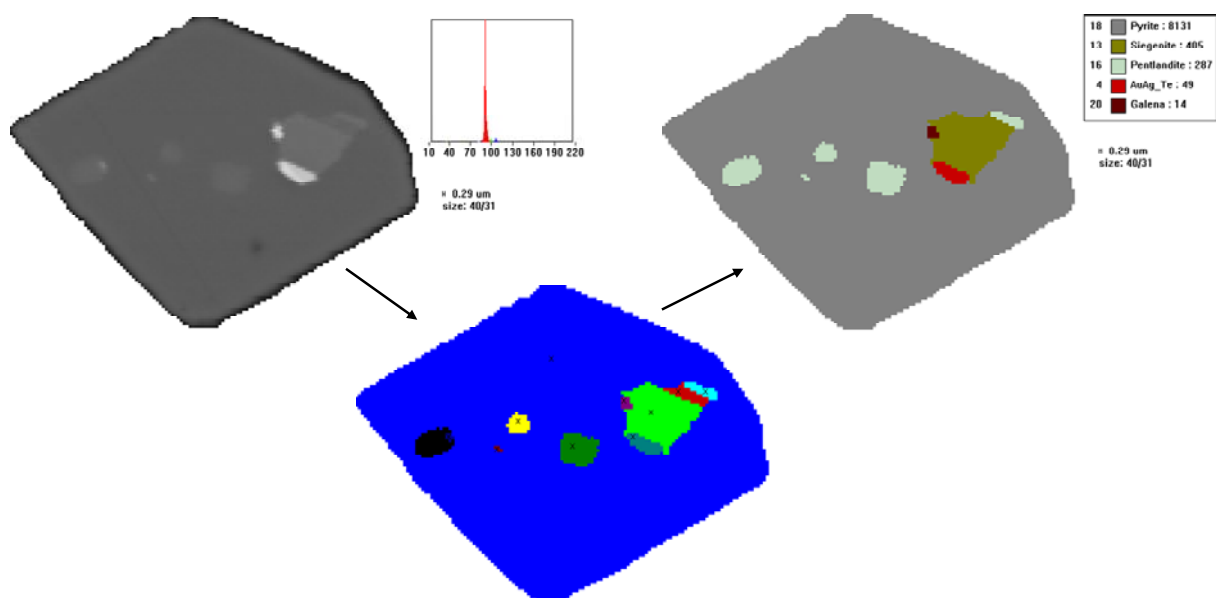


Figure 6. After bright phase search, a single X-ray analysis is performed on the gold phase and its surroundings.

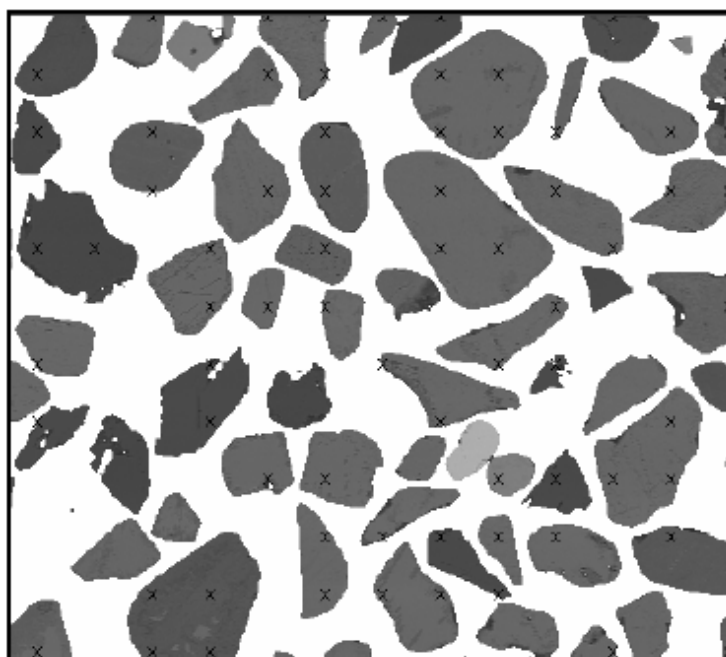


Figure 7. Point X-ray analysis on a grid of widely spaced points on widely spaced lines. BSE imaging is used to discriminate particle matter from background and then collects one X-ray spectrum from each grid point across the particle.

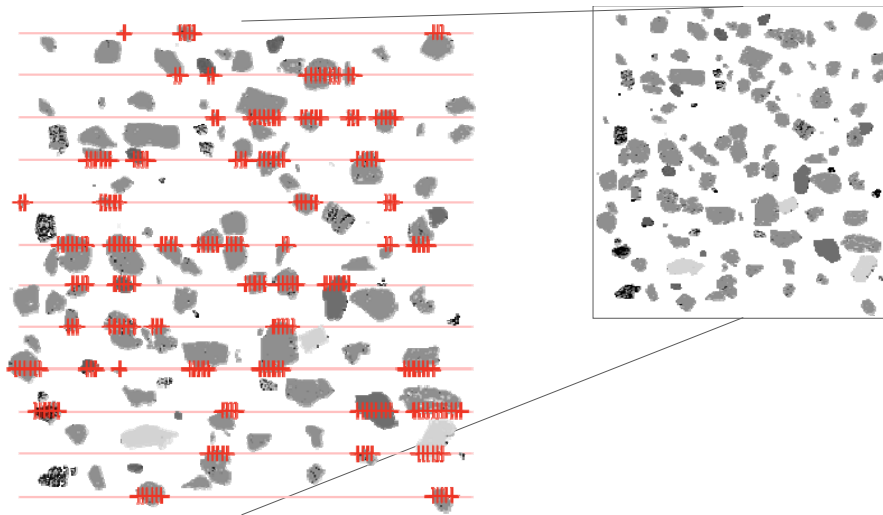


Figure 8. Linear X-ray analysis where mineral phases are identified at closely spaced points on widely spaced lines.

3.3.3. X-ray mapping

This technique provides full textural information such as degree of mineral liberation and phase associations. X-ray mapping imposes a grid over an entire particle image, or specific grains thereof, and collects X-ray data at each grid point to determine the mineral identity. This can either be done on every particle in the sample regardless of grey level (Fig. 9) or on selected particles of interest where segmentation cannot be achieved through grey scale discrimination (Fig. 10). Mineral identification using mapping requires significantly more time than point X-ray analysis as many more spectra are collected to generate a comprehensive mineral map.

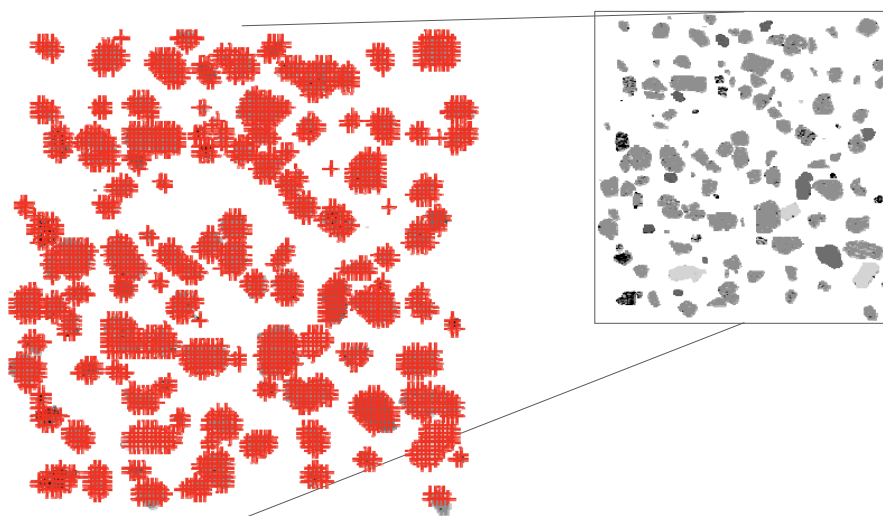


Figure 9. X-ray mapping with full particle scan.



Figure 10. Particle with quartz (dark grey) and sulphides (light grey). An X-ray map is performed on the sulphides only to distinguish chalcopyrite (blue) from pentlandite (green).

3.3.4. Frame scan

A frame or field scan usually takes place on a polished section of drill core or other rock types. This technique allows for full textural in-situ information, especially after stitching individual frames or fields into one broad-scale map.

3.3.5. Mineral identification from X-ray analysis

Mineral classification techniques usually compare sample information with a library of mineral standards. For MLA, the stored spectra from the various types of X-ray acquisition are compared with a predefined list or library of standard mineral spectra, using a pattern matching algorithm, to complete the identification procedure and produce a classified image (Fig. 11). This library is usually constructed before an automated run and involves the collection of a high quality X-ray spectrum for each mineral in the sample. The building of a standards library directly from the sample ensures that measurement conditions are reflected in the standards, such as beam energy (i.e., keV), and it also provides for an elemental department that better reflects the chemistry of the sample. Phase boundaries in the classified images are usually delineated by backscattered electron and X-ray information.

QEMSCAN[®] classifies minerals and phases based on their characteristic chemical composition. This list, known as the mineral library, comprises over 500 mineral species, as well as mixed compositional and chemical groupings. The mixed compositional groupings allow for grain boundary effects (i.e., where an analysis point lies on the boundary between two different mineral phases) and fine grain mixtures of minerals (e.g., intermixed clays). The resulting data are typically too detailed to be directly interpreted, so iDiscover[™] (FEI's proprietary image analysis software) allows for the simplification of these mineral species into a manageable format, by the creation of mineral and compositional groupings. Phase boundaries in the classified images are usually delineated by using X-ray information per pixel (Fig. 12).

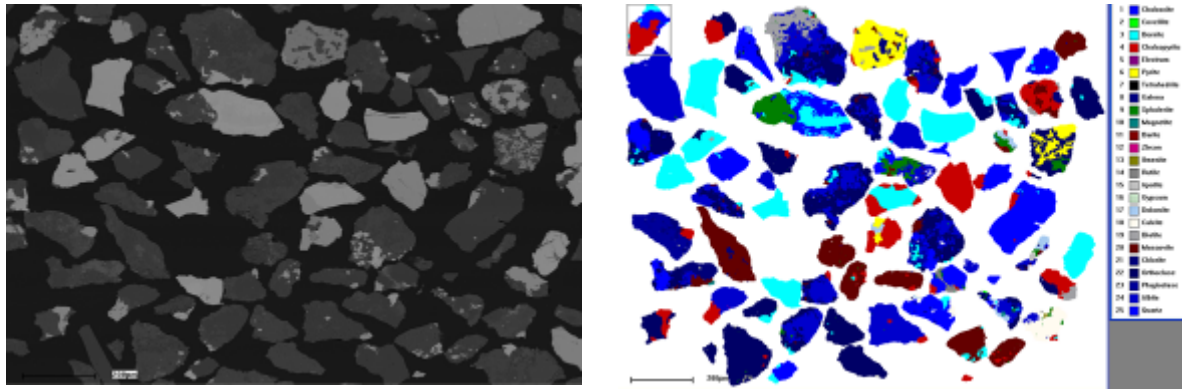


Figure 11. Mineral classification with MLA.

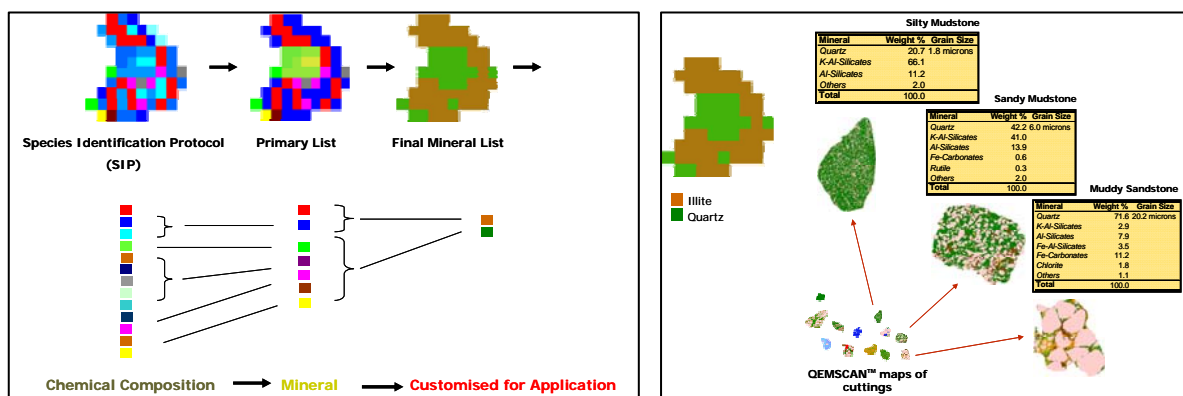


Figure 12. Mineral classification with QEMSCAN®.

4. EXAMPLES OF THE USE OF AUTOMATED MINERALOGY IN THE NATURAL RESOURCES INDUSTRY

4.1. Ore characterisation of base metal ores (MLA)

The mineral and textural variability of an ore deposit, and hence the feed to a mineral processing plant, has implications for concentrate grades and recoveries produced by that plant. The department of deleterious and precious elements in that feed is also important for minimization of environmental impacts and the maximization of profits. Quantitatively capturing the variations of important ore characteristics is the aim of ore characterisation and can be effectively achieved with using automated mineralogy.

Gu and Burrows [8] describe an ore characterisation measurement technique using the MLA known as coarse particle analysis. Here the key to effective ore characterisation is the selection of a particle size for analysis that preserves the *in-situ* textures of the sampled material. Fig. 13 illustrates the coarse particle analysis at a particle size around 600 microns. The purpose of the measurement is to characterize the ore using values such as, modal mineralogy, elemental

department, phase size and association of the ore samples. The collection of phase liberation data is not an aim of this measurement as this data is generally obtained on a size by size basis. Verification that the particle size being analysed is representative of the entire ore sample is essential and can be done by comparing chemical assay data with the chemistry calculated by the coarse particle analysis.

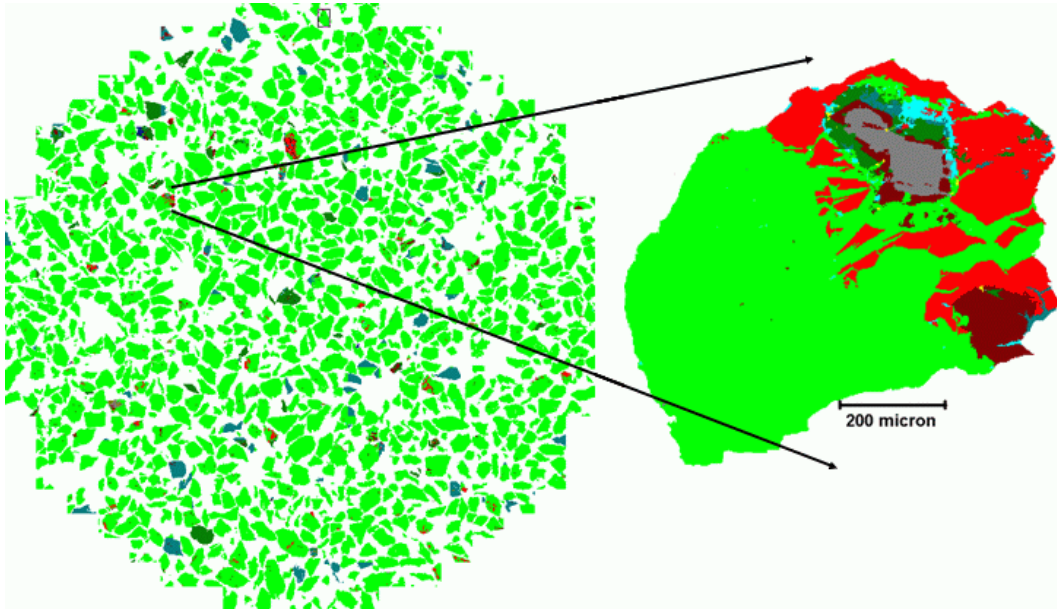


Figure 13. Coarse particle analysis using the MLA for ore characterisation.

4.2. Precious metal search (MLA)

Mineralogical examinations of precious metal ores can simply have the aim of characterising only the native grains of these metals. Searching for very few bright grains within a very large population of dark particles is ideal task for automated mineral analysers such as the MLA.

The Rare Phase Search (RPS) analysis mode employed by the MLA searches the BSE images for bright phases of interest (e.g., PGM, Ag, Au, U) using a BSE or spectral trigger and collects a corresponding characteristic X-ray spectrum. For each grain found, the system saves the image of the particle containing the grain, the stage location and its X-ray spectrum. The operator can subsequently move to the SEM stage location where the grain was located and manually investigate it and its surroundings further. RPS is designed to efficiently locate very fine (sub-micron) components in large particle populations, such as gold in tailings and deliver data such as grain size and associated minerals (see Fig 14). The ability to classify off-line allows the operator to automatically eliminate other bright phases, such as galena, from the phases of interest.

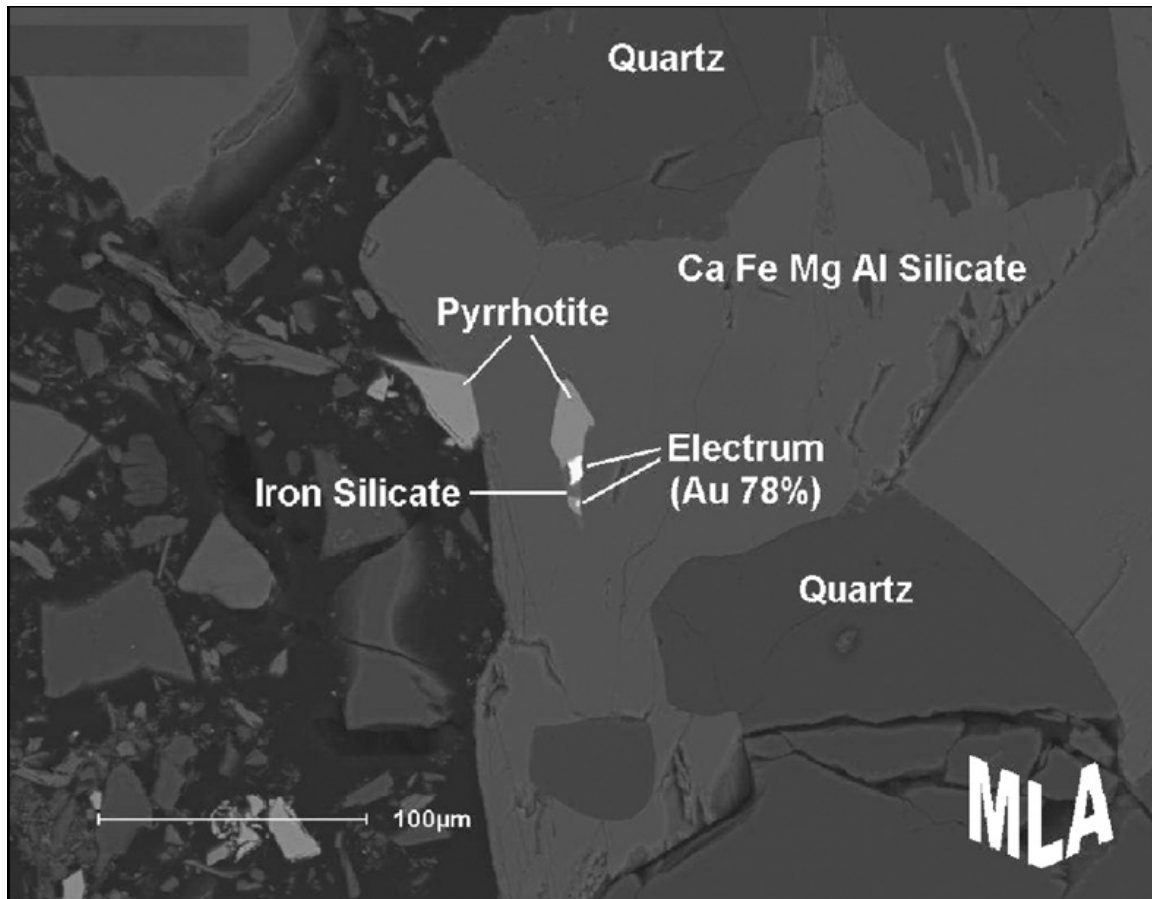


Figure 14. Gold found by the RPS mode.

The overall analytical technique for precious metal search has been described by Gu [5] for MLA, and by Gottlieb *et al.* for QEMSCAN[®] [5, 6]. The detection of bright particles with the MLA is performed with a rapid search for the phase of interest on backscattered electron images at low resolution and repeated with an image capture at higher resolution once the particular phase is found. Both tungsten filament and FEG SEM platforms of the MLA are suitable for this application. Comparison between the two systems at 25 KV and 10 mm working distance shows that the high brightness of the field emission source produces an improved BSE image resolution due to smaller probe sizes (Fig. 15; [9]). For a given probe current, the probe size on the FEG MLA is found to be at least 10 times smaller than on the tungsten MLA (Fig. 16). The ability of the FEG to run at higher probe currents with a smaller probe size generally results in much higher input count rates and therefore improved EDS throughput, whilst maintaining or even improving imaging resolution. The FEG MLA is therefore an ideal solution for e.g., precious metal searches with fast throughput requirements in combination with high image resolution.

Fast time-to-data has always been one of the main requirements from customers in the mining industry. More recently, mining giant and automated mineralogy pioneer Anglo Platinum from

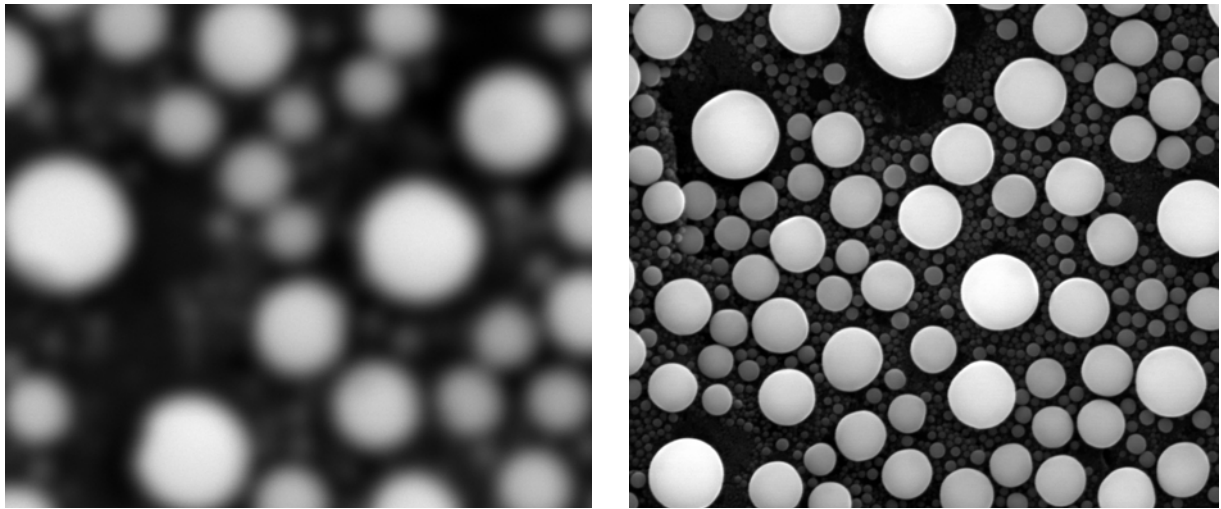


Figure 15. Comparison of BSE image of a sample with tin microspheres. Tungsten MLA on the left, FEG MLA on the right. Magnification = 50000x.

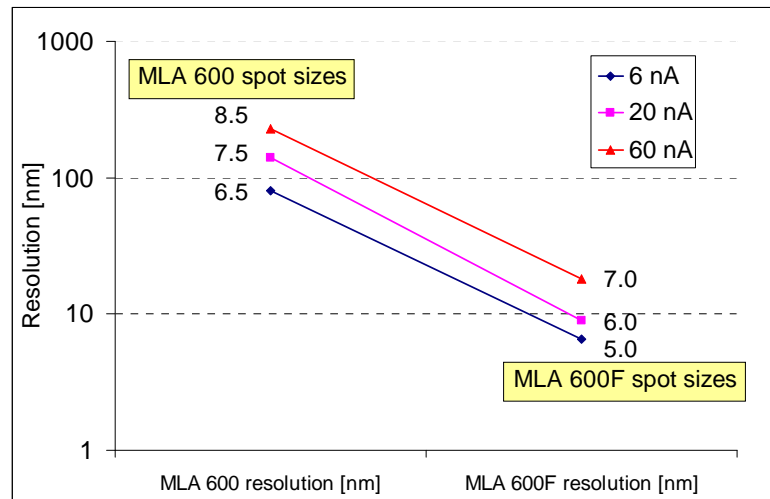


Figure 16. Comparison of probe sizes between tungsten MLA (600) and FEG MLA (600F) at given probe currents.

South Africa, was the first to submit even faster but well contextualized time-to-data requirements for automated mineralogy instruments at their operational sites for concentrator process management applications [9]. The central laboratory at Anglo routinely monitors plant performance of various operations and rapidly assists in the detection of potential inefficiencies. There is much time-to-data benefit if this work could be performed at the operational mining site.

Acquired data at Anglo Platinum shows that the mode of platinum group mineral occurrence varies with geological setting and reef type. Knowledge of the complex mineralogy and liberation characteristics of the plant feed makes it possible to operate the plant more

efficiently. The faster the analytical turnaround, the quicker the response time to changes in feed mineralogy and thereby improvement of overall recovery. Frequent analysis of the feed as part of concentrator process management may also help to better resolve the “background noise” of the plant performance from actual variations in mineralogy.

For operational mining site requirements, fast time-to-data results from automated mineral analyzers make it possible to set targets for the optimisation of processes at the concentrator plant within the time span of a single 8-hour shift on the mining operations.

4.3. Oil and gas (QEMSCAN®)

Sample blocks of drill cuttings and core for oil and gas applications are analysed by the FieldScan technique. This is a two dimensional analysis methodology on the QEMSCAN®, in which an operator defined area of every sample block is scanned on a field-by-field basis and measured. Each field is divided into a virtual grid, the size of which is determined by the vertical and horizontal spacing of analysis points designated by the system operator. For example, in the case of 10 µm spacing, a backscatter electron (BSE) brightness reading and, if above a ‘background’ threshold, a 1000 count X-ray spectrum is taken at a point every 10 µm across the grid, in lines spaced 10 µm apart. Conversely, if the BSE value is below the threshold, no X-ray spectrum is acquired and only the BSE value is recorded. Pixel spacing is assigned to best resolve the modal and textural information of the sample cuttings from all particle size ranges.

The FieldScan images are stitched together and then ‘particulated’ a process whereby the individual cuttings particles are extracted from the image (Fig. 17). Once particulated, any touching particles are electronically separated. QEMSCAN® further classifies minerals and phases based on their characteristic chemical composition. A selection of samples are typically also analysed by whole rock XRD and clay fraction XRD in order to assist in the classification of chemically similar, or very finely (< 5 µm) inter-grown minerals, and also in the calibration of QEMSCAN® with respect to clay minerals.

The application of automated mineralogy in oil and gas exploration and production (E & P) is a new area, and centres around the underlying core capability of SEM-based automated mineral analysers being able to quantify rock type and texture from microscopic examination of materials returned from drilling activities (cuttings and cores) [10-12].

Most rock-forming silicates, carbonates, oxides and phosphates are well characterized by automated systems using X-ray analysis and present little challenge. Grain size is also an estimated value readily extracted from images, and therefore the ability to micro-lithotype cuttings and cores is possible. Porosity, especially in core, is another feature that can be quantified, and has found significant application. By using a combination of modal and

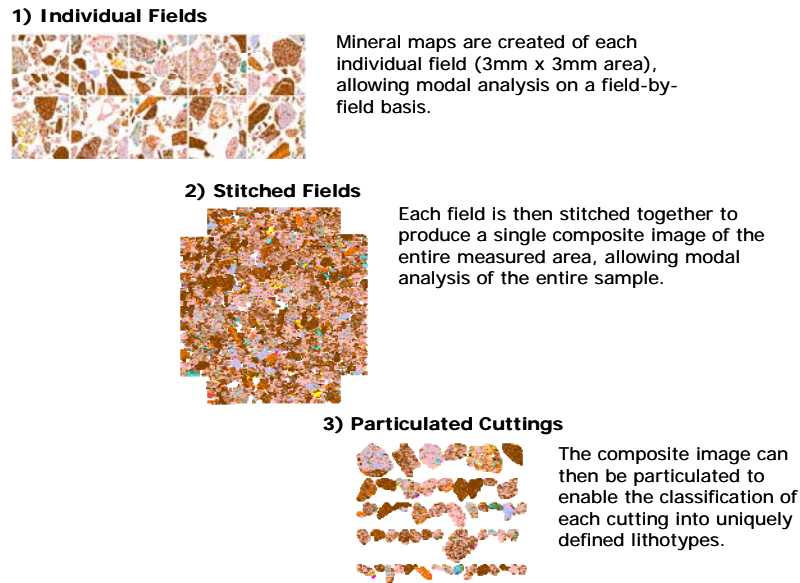


Figure 17. Analytical technique in the analysis of fields and particles for oil and gas applications.

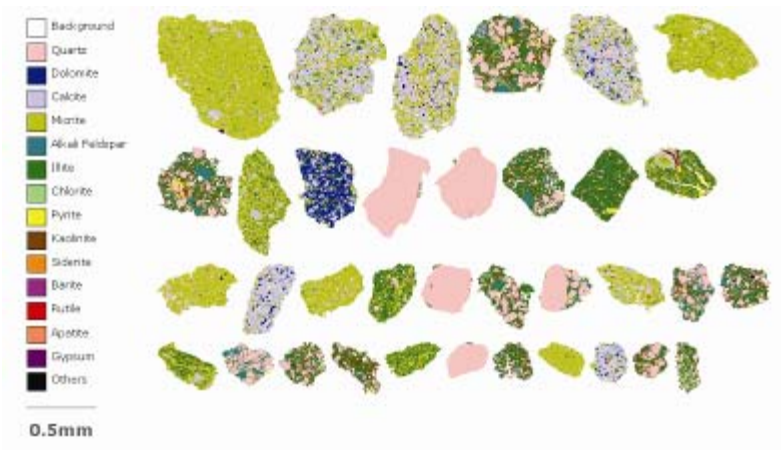
textural data, it is possible to classify each cutting analysed from each sampling interval (typically over 1000), in terms of user-defined micro-lithotype (Fig. 18), and then a plot can be generated which illustrates variation with depth, thus allowing recreation of the stratigraphy through which the drilling has taken place.

Outstanding issues remain, including the reliable identification of certain clay group minerals; estimation of meaningful porosity in cuttings; robust discrimination of detrital phases from overgrowths where both happen to be the same composition (e.g., quartz or calcite; telling lithics from mineral fragments; identification of polymorphs such as rutile from anatase, marcasite from pyrite). Many of these can be worked around if the geological context is known. Others will have to await further development.

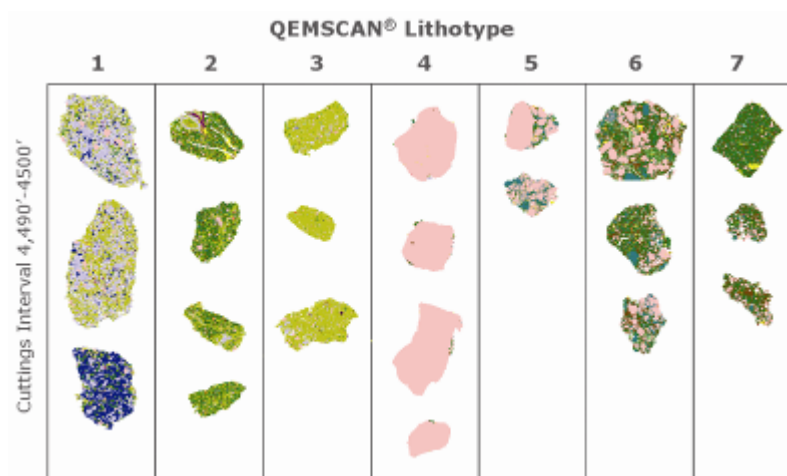
A case study concerning full well correlation analysis in the Basker Field, Bass Strait, Australia, undertaken by Anzon Australia (now Roc Oil), showed quite clearly that major lithotypes can be identified within the cuttings populations examined, and changes in these lithotypes can be used to locate and confirm litho-stratigraphic boundaries, which in turn can be used to undertake cross-well correlation [13]. Other recent studies have demonstrated the potential of automated mineralogy to make significant contributions to the field of fundamental sedimentology [14].

4.4. Cement (QEMSCAN®)

The challenge in cement characterisation is remarkably similar to ore grade characterisation in base and precious metal mining. Different phases need to be classified and the morphology of



(A)



(B)

Figure 18. Automated classification of cuttings based digital images created by QEMSCAN® which allows the mineralogy and textures to be quantified on a cutting-by-cutting basis (A), and then on lithotype basis (B).

mineral grains determined for further processing (grinding efficiency). Association data are crucial since this is exactly what will determine reactivity and ultimately the final properties of cement and concrete. High throughput is needed to make the transition from central lab technology to a QC tool in production.

Portland cement is the most common type of cement and a basic ingredient of concrete, mortar and stucco. It is a fine powder produced by grinding Portland cement clinker (more than 90 %), a limited amount of gypsum and up to 5 % minor constituents (as allowed by various national or international standards). As defined by the European Standard EN197.1, "Portland cement clinker is a hydraulic material which shall consist of at least two-thirds by mass of calcium silicates, the remainder consisting of aluminium- and iron-containing clinker phases and other compounds". Alite (tricalcium silicate) and belite (dicalcium silicate) are the two

most important silicates and their abundance and morphology determines Portland cement properties. Alite reacts rapidly and is important for the early strength of cement while belite reacts more slowly and affects the long term strength.

Quality control in the modern cement plant takes place by XRF and Rietveld analysis. Both techniques are highly automated and especially Rietveld analysis gives very accurate measurements of the crystalline phases present. Morphology and association data however are not obtained. Some cement plants complement this by optical microscopy. Chemical attack allows differentiation between the alite and belite phases and hence morphology and abundance can be measured manually. This approach is slow and requires a well-trained operator, both for sample preparation and microscopy analysis. It must be stated however that an experienced microscopist can do wonders in fine-tuning the process parameters and raw feed based on observations in the optical microscope. The Ono method for cement quality control based on optical microscopy has long been a standard practice in the cement plant [15].

Automated mineralogy was performed on three NIST standard reference clinkers (Fig. 19). The reference clinkers were analyzed at NIST by X-ray diffraction and optical microscopy [16]. Comparison with automated mineralogy results shows good correlation. However, automated mineralogy shows much more details. An alite-belite intermediate phase was defined for better surface reactivity analysis and morphology aspects such as grain size statistics can easily be retrieved. Phases present in very small amounts are easily overseen or even undetectable by optical microscopy but show up in the SEM analysis. These phases are particularly relevant for corrosion prediction and critical strength applications.

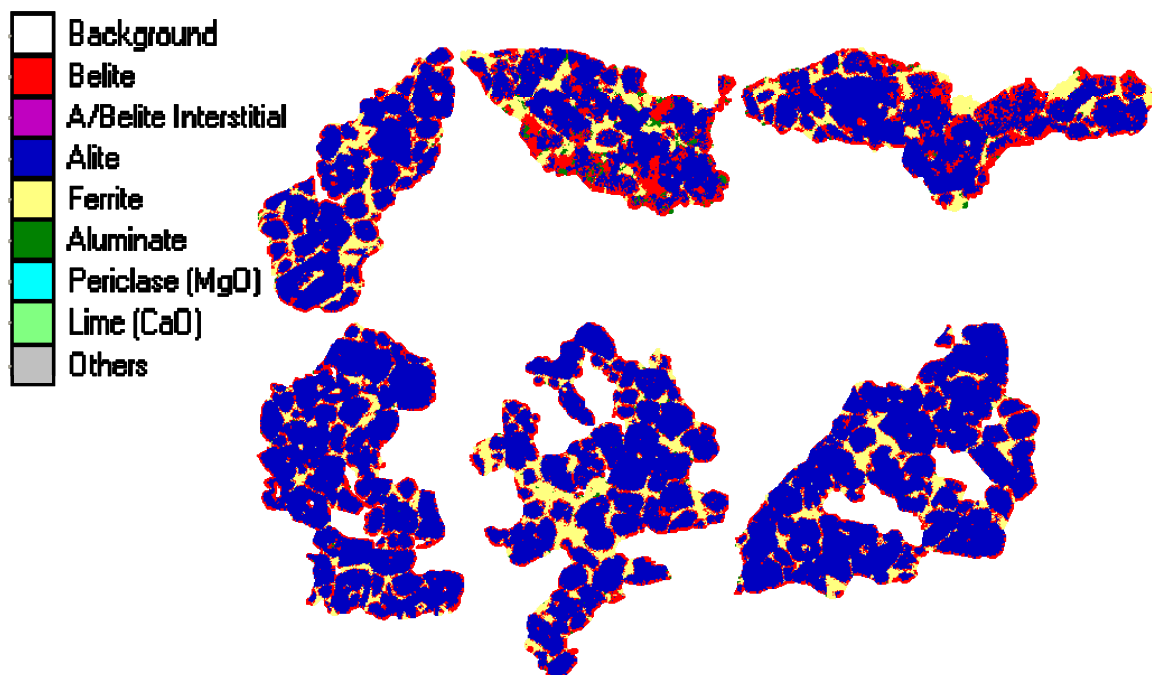


Figure 19. Phase distribution in cement. QEMSCAN®.

World production of cement in 2007 totalled 2.6 billion tonne generating 6 % of the global CO₂ emissions, mostly from the clinker manufacturing process [17]. Clinker is made by heating, in a rotating furnace (kiln), a mixture of raw materials to a sintering temperature of about 1450 °C. The major raw material for the clinker-making is usually limestone (CaCO₃) mixed with a second material containing clay as source of alumino-silicate. At the high temperatures in the kiln the limestone converts to lime (CaO) with consequent release of CO₂ into the atmosphere.

The last two decades most effort has been directed towards more energy efficient production. The highly inefficient vertical kilns are now almost completely replaced by optimized horizontal kilns with pre-calciners and other measures to lower energy losses. Little room is left for improvements in this area. A lot of research is now focused on clinker replacement materials (CRM) to reduce CO₂ emissions and production cost (mainly energy). Fly ash as by-product from coal-fired power plants and blast furnace slag from steel production are widely used as clinker replacement materials. The use of this kind of industrial waste as constituents in building material is an attractive alternative to landfill disposal. The biggest advantage however comes from replacing clinker by material that needs no calcination in a kiln anymore. Reduced energy consumption and CO₂ emission comes from reduced energy consumption (firing of the kiln) and more important from avoiding process emissions from the calcination reaction (limestone to lime).

Quality control of cements where part of the clinker is replaced by fly ash is also X-ray based. Due to the amorphous character of fly ash constituents, large uncertainties are introduced in the determination of chemical composition. Fig. 20 shows a backscattered image of a fly ash sample from an electrical power plant where the grey levels clearly demonstrate the wide range of chemical composition of the individual grains. The grain sizes also vary widely from sub micron to several tens of micrometre in diameter.

Automated mineralogy allows for the chemical and morphological characterisation of the amorphous phases in fly ash cement. This knowledge is crucial for the development of new cement types containing clinker replacement materials and for quality control of known cement mixes. Clever setup of selection criteria based on association data allows for the separate classification of hollow particles (cenospheres). A better understanding of material-property relationships in cement by automated mineralogy leads to an optimal and increased used of CRM's and hence to a considerable amount of CO₂ reduction.

5. SUMMARY

Today's SEM-based automated mineralogy analysers implement prudent use of both BSE and X-ray signals, in conjunction with advanced image and pattern recognition analysis, to

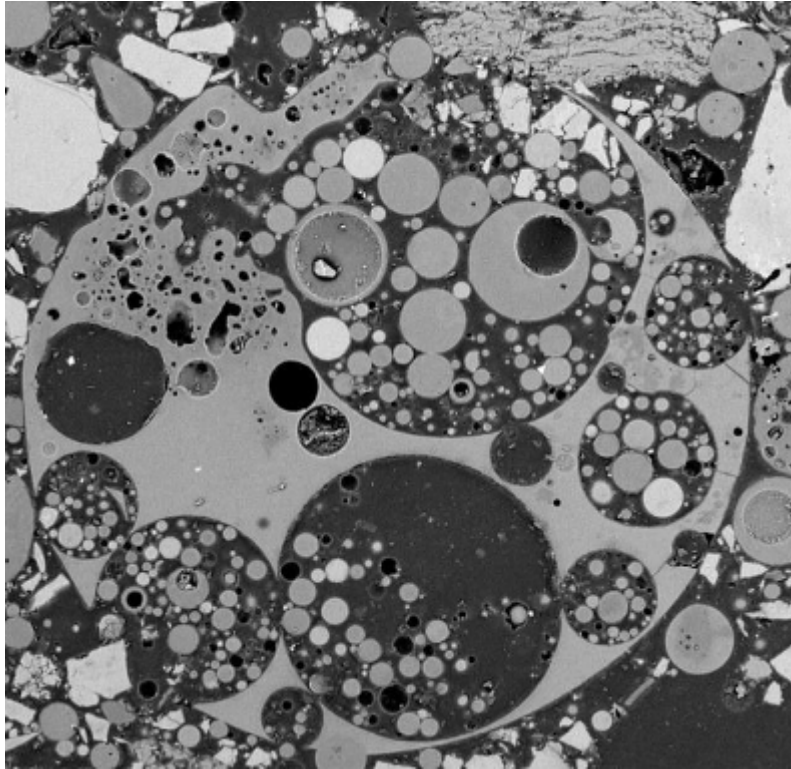


Figure 20. Backscattered image of a fly ash sample from an electrical power plant. Width of field = 60 micrometre.

successfully provide quantitative mineralogical data for a wide range applications in the natural resources sector, automatically. Historically the principal application for automated mineralogy has been in the area of mineral processing plant optimisation by examining plant streams, but today the spread of applications is much broader. Examples from ore characterisation in precious and base metals mining, the oil and gas sector and the cement industry have been presented, but other applications for automated mineralogy also exist, such as mineral matter in coal, and soil analysis from crime scenes, have also been widely used.

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