

LOOKING INTO THE ORE



Recent years have seen enormous advances in the automation of key mineral identification and quantification technologies

The interest in this subject can be gauged from the *Automated Mineralogy 06* – the inaugural Minerals Engineering International (MEI) specialty meeting on the subject. That meeting attracted a wide spectrum of attendees including mineralogists, geologists, microscopists, process and mining engineers, analysts, business managers, and consultants. The depth and breadth of technologies showcased were evident from the wide variety of topics discussed, which included sessions on reviews of existing automated techniques, electron-beam technologies, new technologies and process mineralogy case studies.

Mark Pownceby, CSIRO Minerals, Australia says the presentations made it clear that mineralogists are either now, or will in the very near future, be able to draw on a wide range of techniques to allow the automated analysis of earth resource materials. These include optical, near infrared, X-ray beam, laser beam, electron beam (SEM, EPMA), and proton beam techniques, to name a few.

A number of presentations showed the progress of key SEM-based instruments such as

QEMSCAN and the Mineral Liberation Analyzer (MLA) and how the rapid uptake of both technologies has greatly assisted in planning, monitoring and controlling mining operations. The role of automated mineralogical identification methods now forms an integral part of many projects and the two techniques are intimately involved in mineralogical investigations in industries as diverse as mineral sands, coal, diamond, platinum, gold, nickel, copper and aluminium (bauxite).

Other highlights included exciting developments in non SEM-based characterization methods such as Laser Induced Fluorescence (LIF), detection of bulk mineralogy using radio frequency (RF) techniques, on-line mid-infrared reflectance characterisation, on-line X-ray diffraction (XRD) and the use of robotic instrumentation to fully automate sample analysis procedures. Although many of these techniques have not yet reached the same level of ripeness as the SEM-based (or optical) methods they will, in the very near future, complement the current automated techniques, further adding to the available arsenal of mineralogical tools.

A Mintek mineralogist in South Africa uses the MLA to do platinum searches.

It was interesting to note that at the 2006 event, the traditional mineralogical (e.g. XRD) and emerging techniques were considerably under-represented compared to existing SEM-based methods. Perhaps this reflects the maturity of the newer automated techniques plus their already comparatively wide exposure and use within the mining community. Pownceby noted: "If possible, in future *Automated Mineralogy* symposia, it would perhaps be desirable to have greater input from other mineralogical characterization techniques.

"A further point for consideration is directed to those of us with conventional geological/mineralogical backgrounds. We shouldn't be constrained to think of mineralogy as only pertaining to problems associated with hard rock mining and process metallurgy. Mineral identification is also important in oil exploration, forensics, industrial minerals processing, environmental studies, etc.

One of the key figures in this technology is Paul Gottlieb, who added the 2007 ATSE Clunies Ross Award to the list of honours won in recognition of his work developing and commercializing revolutionary mineral technology. The award recognizes top science and technology achievements and the citation read: "With unflinching dedication, Paul managed his design team, installed and promoted QEM-SEM (the precursor to QEMSCAN) around the world and organized user workshops and training sessions for its use." Gottlieb is Chief Technology Officer for Intellection, based in Brisbane.

The technology, QEMSCAN, is the world's first commercial quantitative automated mineralogical analysis system that is many orders of magnitude faster than conventional systems and is used by major mining companies Rio Tinto, BHP Billiton and CVRD. QEMSCAN is based on CSIRO research.

Today QEMSCAN conducts 12,000 mineral analyses per minute with such accuracy that base and precious metal users have reported dramatic process improvement benefits. Platinum and gold producers are able to measure minerals present in ores as low as 1 ppm.

"We are now further developing the systems to enable automated sample preparation, analyzing samples five times faster and using liquid nitrogen-free detectors that are easy for laboratory technicians to use at isolated sites," Gottlieb reports.

QEMSCAN uses an electron microscope and X-ray detectors to rapidly scan a mineralogical sample, and software to create an image data file that can be processed by a desktop computer. The next generation systems Gottlieb is currently creating will be portable, lower cost, faster, easier to use and will bring further efficiencies to processing plants, mine sites and projects.

The latest news is the successful integration of the world leading Quantax Esprit X-ray microanalyser software from Bruker AXS with the QEMSCAN automated mineralogy system, to break new ground in advanced mineral analysis.

X-ray microanalysis is an analytical technique for precise determination of the chemical composition of solid samples, thin layers or particles under investigation in electron

microscopes. As part of normal operation, QEMSCAN may detect compounds that require further analysis, often by an electron probe. Such analysis can cause delays and additional costs to the process of mineral analysis.

By integrating the liquid nitrogen free energy dispersive X-ray spectrometers (EDS) that are a standard part of QEMSCAN and the Quantax Esprit software option it is possible to detect and analyze all elements down to boron simultaneously on the one QEMSCAN instrument. With true standardless analysis; proven standard related quantification; or even a combination of both methods, obtaining elemental information from a sample volume of only a few microns and providing relative detection limits in the order of a tenth of mass percent makes QEMSCAN based Esprit X-ray microanalysis one of the most sensitive analysis methods available today.

Quantax Esprit is available immediately as an option for all QEMSCAN systems using the Intellection X275HR Silicon Drift X-ray detectors.

Advanced SEM techniques

FEI Co and Australian-based JKTech are teamed to combine their innovative software and scanning electron microscope (SEM) technologies, delivering innovative applications that greatly enhance minerals processing and improve evaluation of exploration targets. The combined solution features FEI's Quanta™ SEM and JKTech's Mineral Liberation Analyser (MLA) software and is available now with liquid-nitrogen-free, high throughput silicon drift EDS technology.

The MLA, capable of analysing up to 16 different samples overnight without the need of an operator, is a high-throughput and highly-automated mineral analysis system. It rapidly identifies minerals in polished sections of drill core, particulate or lump

minerals in polished sections of drill core, particulate or lump



materials and quantifies a wide range of mineral characteristics such as abundance, grain size and liberation distributions. The automated stage control and image acquisition of the Quanta SEM enable imaging and subsequent X-ray analysis of at least 5,000 individual particles for concentrated samples and 50,000 or more particles for tailings or low-grade materials. The geometallurgical and ore characterization data enables users to optimize plant feed quality by avoiding metallurgically poor ore stocks or facilitating effective ore blending.

There are already more than a dozen leading international resource companies on four continents using the MLA package to improve processing efficiencies for copper, nickel, lead, zinc, manganese, iron ore, mineral sands and precious metals such as platinum, palladium, silver and gold. Several are using multiple tools for 24/7 analysis. Universities and geological institutions are also finding the MLA a valuable tool that contributes significantly to their research outcomes.

"The MLA package represents a complete mineral analysis solution that can be tailored to suit high-throughput production applications and research applications," said Bruno Janssens, vice president of FEI's NanoResearch and Industry division. "Our customers are seeing a rapid return on the investment from the MLA system founded upon the complementary combined expertise and support of FEI and JKTech."

FEI is a global leader in providing innovative instruments for nanoscale imaging, analysis and prototyping. It has global alliances with a number of companies besides JKTech. One is with Malvern Instruments; a joint development and marketing programme for advanced nanoparticle analysis using Malvern's particle image analysis software on FEI's line of Quanta scanning electron microscopes (SEMs). The combination delivers a powerful particle analysis solution that extends current analysis technologies for nano-sized particles.

At *Automated Mineralogy 07* Dr. Paul Miranda and Dr. Corby Anderson from The Center for Advanced Mineral and

Metallurgical Processing (CAMP) at Montana Tech of the University of Montana discuss CAMP's MLA, which it has used "for several

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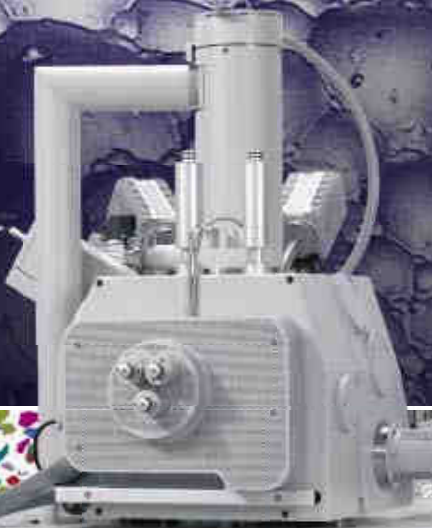
With the Mineral Liberation Analyzer from FEI Company and JKTech, you'll know the answer to this crucial question faster and easier than ever before. Combining a sophisticated scanning electron microscope, liquid nitrogen free X-ray microanalyzer, and specially designed mineral liberation analysis software, the MLA automatically analyzes thousands of particles in ores, concentrates, tailings, and PMG products without operator intervention, providing fast, accurate measurements of mineral abundance, elemental distribution, particle and grain size and shape, mineral associations, liberation, locking, and much more.

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A Blue Cube scanner head installed in a slurry stream

projects with excellent results. The MLA is able to both analyze and quantify mineral phases. Therefore, the MLA is becoming the Sherlock Holmes of the mining and metallurgical industry."

One project is a silver project where initial flotation scoping tests were performed by attempting to separate silver from gangue material. Because of the poor recovery results (less than 5%), MLA was used to determine possible scenarios for increasing silver recovery. The second project involved a tungsten mine in Nevada. Previous mining practices had yielded poor tungsten results and recoveries. By using the MLA, tungsten mineral phases, along with possible contaminants, were verified and results used to increase overall tungsten recoveries. The third project involved gold evaluation in tailings. The fourth project evaluated a molybdenate/chalcopyrite concentrate with surprising results. Finally, Stillwater Mining is currently upgrading its plant. By using the MLA, flotation mineral phases and liberation were identified completely.

Another paper, from Anglo Research notes that data gathered by automated mineralogical instruments offer essential information for plant optimization that cannot be provided by routine chemical analysis. The mineralogical data generated includes modal mineralogy, liberated grain sizes and mineral associations.

Quality management of the technology is imperative, as incorrect data could result in incorrect decision-making. Internal risks to the validity of data produced by automated mineralogical instruments are changes to hardware, software, techniques, operators and key-files. Anglo Research has implemented various working procedures to address these risks.

Anglo Research tests on MLAs and QEMSCANS proved that both techniques produce reliable data. PGM analyses on various MLA instruments and base metal sulphide analyses on all QEMSCANS confirmed reproducibility. Data from these instruments are verified by comparing chemical data with chemical compositions calculated from bulk modal mineralogical data. In the Anglo Research presentation it was demonstrated how quality control has been applied to develop a methodology to track changes in mineralogy during the progressive leaching of base metal sulphides in a pilot plant trial.

Rogério Kwitko-Ribeiro of CVRD's Center for Mineral Development reports that three samples of marginal, originally sub-economic high reactive-silica bauxite ores, where characterized by QEMSCAN. "Kaolinite was focused as the unique source of reactive-silica grades. Bulk liberation data was compared to mean exposed area in the three samples, showing an equivalence between both modes of measurement, as a useful tool

for surface processes prediction. Recovery charts confirmed the potential of reactive-silica reduction by kaolinite reverse flotation, with manageable gibbsite losses."

D. De Nooy explains that SGS's Advanced Mineralogy Network provides comprehensive, state-of-the-art, automated process mineralogy services at its sites in Canada, South Africa, Australia and Chile. The Network uses a range of instruments and technologies, in conjunction with unique in-house methods, to provide "fit-for-purpose" data to clients in the minerals industry.

SGS has over six years of experience using QEMSCAN and has completed hundreds of projects integrating QEMSCAN with mineral exploration, geometallurgical mapping, process design, plant optimization and tailings disposal.

The type of mineral deposit to which this technology has been applied is equally varied, with selected examples including copper porphyry ores, complex massive sulphide ores, heavy mineral sands, low-grade laterites and rare earth elements ores.

Intellection personnel note that the "identification of trace mineral phases is an important aspect of the QEMSCAN system and has been subject to significant development over the last few years. The core concept behind trace phase identification is to use fast backscatter electron scanning to identify 'bright' phases for subsequent X-Ray mapping. This method is subject to bottlenecks due to identification of 'false positive' readings and the current study investigated the application of new solutions to reduce bottlenecks and enhance overall analysis speed.

The improvements achieved using new software solutions have been investigated and compared to historic data"

Process optimization

At Mintek, South Africa's national minerals Research & Development organization, mineralogical investigations are used extensively when developing processing routes for new mining projects, as well as for process optimization studies on existing operations, throughout Africa and further afield.

Combinations of analytical and mineralogical techniques such as chemical analysis, optical microscopy, automated mineralogy, and X-ray diffraction are used to identify and quantify the minerals present in the ore samples and in the mineral separates that are derived from these ore samples.

Every mineral has a unique combination of chemical and physical properties, which are used not only for identification (as in traditional



Sample being inserted on the turntable of the Blue Cube MQi Universal Spot Mineral Quantifier

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A laser-induced fluorescence (LIF) analyzer.

mineralogy), but frequently form the basis for selecting and optimizing the techniques used to separate and recover the component minerals in the ore. A comprehensive mineralogical investigation will allow the prediction of factors such as the grind size for effective liberation, the likely efficiency of various separation processes, the maximum grade attainable, and the probable extent of tailings losses, as well as problems that may arise during processing.

Mintek explains that the information that can be obtained encompasses:

- Mineral/phase identification and quantification using XRD, optical microscopy and scanning electron microscopy coupled with EDS (QEMSCAN and MLA systems)
- Inorganic mineral/phase-chemical compositions using electron microprobes with light element detection capabilities down to boron
- Modal analysis and quantitative liberation characteristics of minerals/phases in ores, feeds, products and tailings samples using automated mineralogical techniques
- Petrography of ores using optical microscopy supported by XRD, bulk chemical, and mineral chemical data.

Recent investigations by Mintek have included the mode of occurrence of sulphide minerals in the dense-media separation products from a nickel ore, as input to the design of a pre-concentration flow sheet. In another project on the mode of occurrence, liberation characteristics, and composition of PGM particles in flotation tailings, poorly floating PGM-bearing phases were identified, and modifications were recommended for the proposed plant configuration and flotation conditions.

Liberation analysis, composition, and mode of occurrence of gold-bearing particles in feed and cyanide leach residue samples can be used to identify reasons for erratic gold recoveries and high cyanide and oxygen consumption. Also, mineral compositions of uranium-bearing phases and gangue minerals in various uranium ores have been used to identify the most cost effective processing route (acid versus alkaline leaching).

Blue Cube Systems specialises in providing hi-tech solutions for mineral processing plants. Its Blue Cube MQi in-line mineral quantifiers allow process operators in mineral separation plants to access minute-by-minute mineral

composition information. The data provided by the in-line mineral scanners assist operators to optimize recoveries by providing immediate feedback. The latest model, the MQi Spot Mineral Quantifier, addresses the need for a versatile unit, able to accept batch samples from different streams.

It is available in two versions. The MQi Spot Pour Through model is suitable for dry, free flowing ores and has been successfully tested in the heavy minerals industries in South Africa. The MQi Spot Universal is suited to both dry samples and wet filter cake. It has already proved its worth in a base minerals plant and on furnace slags. Positive tests have also been concluded on chromite and silica.

The MQi Spot system relies on the proven technology of Blue Cube's MQi in-line mineral quantifier in terms of embedded software, spectrometer and data processor components. Sample presentation has been changed from in-line to batch presentation. Sample preparation is uncomplicated and fast. A graphical user interface (GUI) has been added to provide a suitable operator interface. Output data is channelled directly to a client's database for immediate use by operators. The technology provides consistent and accurate information on actual mineral composition information, not only the elemental components.

The MQi technology can be installed at relatively low capital cost and imposes almost zero operating costs. It provides instant information in a format understood by plant operators and can thereby enhance their ability for rapid stabilization and tuning of mineral separation.

All installations of the Blue Cube MQi technology are linked to the company's facilities in Stellenbosch by a data link. This enables remote operational supervision and calibration. Blue Cube Managing Director Francois du Plessis "It is also possible to link

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the output of the MQI directly to the control circuitry of some types of separation equipment. This was proven in our pilot plant and is already being implemented in the separation plant of one of our customers.

Automatic control of mineral separation processes has become a reality."

Oswaldo Maldonado of SAX Chile (which sells Bruker-AXS and Herzog machines in South America) reports on *Online mineralogical analysis for copper slurries with DRX: latest advances*. He notes that the online X-ray diffraction system is a fully automated instrument capable of carrying out quantitative mineralogical analysis of the main mineralogical phases directly on copper slurries. "The use of parallel optics combined with TOPAS provides a powerful tool for process control in copper flotation. The measurements are performed directly in the slurry flows and the prompt information can be used to optimize additive dosing, among other applications." He presented the latest advances that have made the measurements faster and more accurate.

Arnt Kern of Bruker-AXS explains that X-ray powder diffraction (XRPD) combined with quantitative Rietveld analysis is an essential tool for the determination of relative phase amounts in crystalline materials, including both powdered solids and slurries; analysis times are normally less than 10 minutes per sample. Quantitative phase analysis by XRPD allows users to characterize and optimize the whole process from exploration, processing, through to waste stockpiling. "Currently, XRPD finds exponentially growing interest in important application areas such as in leaching and flotation (optimization of acid consumption, pH control, etc.) and in environmental mineralogy (prediction and control of acid rock drainage, verification and quantification of carbon dioxide sequestration, etc.).

Rock doctoring

Exploration has many uses for automated mineralogy. Dr Gavin Hunt, Technical Director of Spectra-Map explains that his company developed a rugged, non-contact, imaging IR spectrometer, the SpecCam, to allow high-throughput, automated identification and quantification of minerals from a variety of rock types. The camera can automatically highlight and/or provide accurate modal % values for a range of minerals, including clays, phyllosilicates and carbonates.

The modular and high-speed, spectrally flexible design means that the SpecCam can be easily installed on different equipment, each one suited to a different stage and specific application in the mining process. For example the SpecCam can be used on an automated portable logging frame to provide detailed mineralogical log data from core, blastholes, RC chips or hand samples.

Dr.-Ing. Heribert F. Broicher of Rock ID Consulting notes that non-contact and non-destructive analyzers operating on-line over belt conveyors, LHDs, trucks or trains and providing real-time information on the type and quality of the respective mineral bulk solids have been wanted and found wanting for many years.

"Development and field testing of analyzers based on laser-induced fluorescence (LIF) has come a long way. In the late 1990s early prototypes were tested at LKAB's Kiruna mine in Sweden, in 2001/02 work continued with different lasers installed in the analyzers, operations from the industrial minerals industries (shown in the picture) were included in the test work, in 2004 a new generation of prototypes based on diode-pumped lasers were operated at Kiruna and in 2005 at Rio Tinto's Argyle diamond mine in Australia. Also in 2005 a very detailed investigation at Phelps Dodge's operations in the US proved the applicability of LIF in the porphyry copper environment.

"LIF Analyzers use pulsed high-energy lasers to stimulate fluorescence in minerals. With a pulse power to the order of megawatts almost all minerals fluoresce. A very short pulse duration of a few nanoseconds allows for measuring short fluorescence decay curves. Highly sensitive

and very fast photomultipliers pick up the analogue fluorescence signals in selected spectral bands. High-speed digitizers operating at GHz sampling rates facilitate computerized evaluation of the decay curves.

"The main areas of application are in dilution control, assessment of ore types and quality control. An especially favourable environment for dilution control exists in diamond mining, where the fluorescence of diamondiferous kimberlites or lamproites differs significantly from that of sterile country rock. Different ore types can be identified in gold or porphyry copper or molybdenum deposits due to the mineral composition and/or the degree of alteration or oxidation. The fluorescence signals can be correlated with grinding index, acid consumption or the degree of alteration or oxidation. Quality control has been realized in dolomite limestone and also in phosphorous iron ore of the Kiruna type. At Kiruna, loaded trains passed underneath the LIF analyzer at a speed of 20 km/h and a split second later the analyzer gives a correct estimate of the average phosphorus content of the train load.

"The weakness of pre-2006 LIF analyzers was the laser, with respect to rigidity, emitted wavelength, pulse energy and pulse rate as well as service intervals and maintenance needs only a very few suitable products were available. Now, in 2007, a new generation of lasers without the shortcomings of previous generations is on the market, and with all the experience gained during the many years of research and development LIF analyzers can be offered with confidence of meeting the expectations regarding availability and accuracy of results."

Siebtechnik and ROCK ID Consulting started a co-operative situation in early 2007. Siebtechnik has expertise in processing of mineral bulk materials as well as solids/liquids separation and for more than 80 years has built screens and sorting machines, centrifuges and automatic samplers. Rock ID Consulting has been involved in LIF research and application since 1982. *IM*

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