Minerals Engineering 24 (2011) 1229–1237

Contents lists available at ScienceDirect

Minerals Engineering

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

Modern Process Mineralogy: An integrated multi-disciplined approach to flowsheeting ☆

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ARTICLE INFO

Article history:
Available online 27 March 2011

Keywords:
Ore mineralogy
Sampling
Mineral processing

ABSTRACT

The history of development of Process Mineralogy is reviewed, and updated. Specifically, the approach taken by Xstrata Process Support is discussed. It is shown that across the current generation of practice, a multi-disciplinary approach of sampling, geology, quantitative and qualitative mineralogy, applied statistics and mineral processing hybridises well into a synergistic arrangement that delivers better flowsheeting for the concentrator. The development of modern mineralogical and mineral processing equipment and methodology has contributed in great measure to an overall ability to correctly assess the processing needs of an orebody. This leads to a more accurate understanding of the flowsheet requirements, thus the flotation testing becomes more focussed and delivers a more viable flowsheet at an earlier stage of the project. The role of applied statistics in this practice additionally contributes relevant quality controls to the sampling and laboratory quality controls, as well as to the all-important plant scale trial, which must be able to demonstrate the expected performance gains. Some suggestions for the path forward from here onward are made.

1. Introduction

1.1. History of Process Mineralogy

Historically, the design of concentrators treating sulphide ores was treated with the tools available at the time – for example, pioneer work by Bond (1952) for work index and mill sizing, and the various empirical sampling and testing of drill core and other geological samples or specimens, to arrive at an expectation of concentrator performance after commissioning. Others such as Restarick (1976) pioneered a series of plant survey or sampling structures that would produce a diagnosis of the flowsheet limitations. As these tools were advanced, and newer inventions and methods developed, this endeavour reached a breakthrough point in the early 1890s when two developments occurred. One was the realisation that the integrated approach using mineralogy and mineral processing would produce a synergy; the other, the development of a quantitative automated mineralogical measurement platform with Quantitative Evaluation of Minerals by Scanning Electron Microscope (QEM ∗ SEM), and later, the Mineral Liberation Analyser (MLA).

For the operating concentrator, the question had always arisen as to what further gains could be made by way of recovery, selectivity or concentrate grade whilst sustaining or increasing throughput. At the time, such a concentrator had at best a platform of monthly composites of feed, concentrate and tailings which would be sized, weighed and assayed, producing size-by-size recovery models, sometimes supported by optical microscopy, informing the grinding and classification circuit of possible improvements. Some investigators saw the link between the type of size distribution produced by the grinding circuit, and the limitations or opportunities that this presented to the flotation circuit (McIvor et al., 1990; McIvor and Finch, 1991). In a well-documented series of endeavours at Mount Isa Mines, Queensland, the ultrafine sulphide grain size issue and associated incomplete liberation and falling metal recoveries was studied by Johnson and co-workers, leading to the development and successful commissioning of the IsaMill (Johnson, 1987; Young et al., 1997, for example). Often, a programme of flotation tests would support these activities. Others, such as Trahar (1981) showed the value of describing flotation performance on a size class basis. It was common to see a series of publications of these endeavours across the life-of-mine, often concluding their best result in the last 5 years of the operation before shut-down after 30 or so years of production (Shannon et al., 1993, for example). The impact of these practices was reviewed by Mackey and Nesset (2003) showing that using the McNulty models for start-up, projects could be ranked by order of the maturity of the technology to be used; by the actual delivery of saleable metal against design in the first few years of operation; and how this impacted the nett present value delivery (NPV) of the project.

The connection between mineralogy and metallurgical performance in a plant was recognised long ago (Gaudin, 1939; Petruk,

☆ Keynote Lecture Presented to "Process Mineralogy 10", MEI, Cape Town, November 2010. Now Submitted to Minerals Engineering with Revisions February 2011.

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doi:10.1016/j.mineng.2011.03.004

1976; Petruk and Hughson, 1977; Cabri, 1981; Petruk and Schnaar, 1981; Peyerl, 1983, for example) as was the need to provide diagnostic sampling techniques of a plant (Restarick, 1976) and to improve the statistical reliability of mineralogical and process measurements (Henley, 1983; Lotter, 1995, 2005). Gaudin's first liberation model of 1939 presented a penetrating analysis of the problem. His work was followed for decades by geometrical probability models, for example Bodziony (1965) who showed that the techniques of integral geometry could accommodate the problems associated with the indeterminate nature of the geometrical mineralogical structure. Mathematical liberation models were written in the 1970s and 1980s as a lead into the definition of the grinding requirements of an ore for flotation (King, 1979, 1989, for example). The development of QEM $*$ SEM (and the second generation QEMSCAN) (Grant et al., 1976; Barbery et al., 1979; Sutherland, 1993; Gottlieb et al., 2000), and the later development of the MLA (Gu, 2003; Fandrich et al., 2007) formed the breakthrough platforms into what is now known as Modern Process Mineralogy. At Falconbridge Limited, for example, this vision was taken into a project to develop the opportunity and deliver value into operations using this new integrated approach, in which an internal rate of return of 92% p.a. was shown for the investment in the laboratory equipment, sampling, and cost of plant modifications (Lotter et al., 2002). In this case, the Process Mineralogy platform was designed using geology, sampling, mineralogy and mineral processing. The later addition of applied statistics to the interpretation of flotation tests and plant scale trials further enhanced this development. Numerous examples of equivalent development and applications have since been published, for example Lotter et al. (2003), Fragomeni et al. (2005), Charland et al. (2006), Dai et al. (2008), McKay et al. (2007), and Triffett et al. (2008).

Whereas particle measurement was itself an important part of this programme, so was the condition of the particle surfaces. In this series of endeavours, significant new understanding of activation and collection mechanisms developed, covering both sulphide and silicate species. Early electrochemical studies catalysed a series of studies in galvanic interactions between sulphide minerals, such as reported by Finch and his co-workers (Rao and Finch, 1988). The later development of TOF-SIMS in the 1990s as a tool for characterising adsorbed species on sulphide and silicate surfaces was a significant breakthrough in understanding some of the key mechanisms of flotation. Some of the earlier work by Nagaraj and Brinen (1996, 1997) additionally using X-ray Photon Spectroscopy (XPS), identified copper cementation on ferro-magnesian silicates as the activation mechanism whereby these minerals took on positive flotation characteristics. The reported mechanism was the initial adsorption of Cu^{++} on orthopyroxene followed by reduction to Cu^{+} when the collector was adsorbed. Other work by Stowe et al. (1995) identified the cementation of lead on sphalerite as the mechanism whereby unwanted sphalerite was floating to the copper and lead concentrates. Other significant work was performed in Australia (Smart et al., 1996). This new platform, accompanied by microflotation testwork, led to significant breakthroughs in understanding silicate dilution in platinum flotation concentrates. After the copper activation mechanism was first reported for the ferro-magnesian silicates by Nagaraj and Brinen (1996, 1997) a significant series of related discoveries followed in South African Platinum based at the Amplats Research Centre with work by Malaysiak and Shackleton, and with the University of Cape Town (UCT), with work by Parolis, Harris, Wiese, Bradshaw, O'Connor, Becker and others. Similar responses were also identified and reported for quartz, feldspar and chromite by Martinovic et al. (2005). Apart from the installation and successful commissioning of a TOF-SIMS at Amplats, the establishment of a practical microflotation test rig at UCT empowered several postgraduate studies to advance this understanding. Malysiak et al. (2004) and O'Connor et al. (2006) characterised the floatability of the pentlandite–pyroxenite system. A detailed study to improve the selectivity of separation of pentlandite from pyroxene using zeta-potential and TOF-SIMS measurements of flotation test products from defined conditions was reported by Shackleton et al. (2003a,b). This work was performed as a mixed mineral system, i.e., the pyroxene and pentlandite were together in the microflotation test setup. It was found that copper adsorption was non-selective. Subsequent flotation tests demonstrated that significant advances in selectivity of pentlandite over pyroxene could be gained by using DETA and EDA. Subsequently some of this work was reviewed by Lotter et al. (2008) as a discussion.

2. Our position in 2010

2.1. Modern Process Mineralogy

As it stands at the time of writing, Modern Process Mineralogy has been advanced to a more powerful, integrated practice as a hybrid discipline. The salient features of its structure follow. This description specifically describes the current practice at Xstrata Process Support, Sudbury, Ontario:

2.1.1. Multi-disciplined approach

First and foremost, the work dynamics of a Modern Process Mineralogy team must be recognised as one of the most powerful enabling features of best practice. No single team member is able to perform an entire project in this hybrid discipline alone. It is the interaction between the individuals in the team, and their sharing of and contribution from their specific discipline, that pulls the hybrid practice together. It is obvious from the recent publications by the more advanced groups that their co-authorship approach reflects this interaction.

2.1.2. Representative sampling

Representative sampling is a key component to any testwork programme. Although historically bulk samples from one location within a deposit were deemed appropriate for process diagnosis and flowsheet development, it is now recognised that a set of unoxidised drill core samples, representative of the resource in terms of space, grade, grade distribution and lithology will better quantify process performance. In cases where random sampling methodologies are required, Gy's sampling models (Gy, 1979) are applied in conjunction with an understanding of preliminary geometallurgical unit definitions to help streamline minimum sample mass requirements. This approach stratifies the compound distribution (Cochran, 1946) and reduces the minimum sample mass required for a given fundamental variance (Lotter, 2010).

Samples collected for the metallurgical testwork programme are crushed and screened in two stages to minimise the generation of fines and blended using an odds and evens blending (Middleditch and Lotter, 2008) or equivalent (Middleditch et al., 2009) protocol to produce individual charges for flotation or other mineral separation testing. A sample of blended charge is subsampled by spinning riffler to produce replicate subsamples with minimal variance for mineralogical assessment and external reference distribution which defines a robust estimate of the sample mean grade (Lotter and Fragomeni, 2010). This respects Gy's safety line of 1979, whereby subsamples may be taken from the lot in a relationship between primary sample and subsample mass and particle topsize (Bartlett and Hawkins, 1989).

2.1.3. Geometallurgical units

Geometallurgical units (Lotter et al., 2003; Fragomeni et al. 2005) can be defined as an ore type or group of ore types that possess a unique set of textural and compositional properties from which it can be predicted they will have similar metallurgical performance. Sampling of an orebody based on geometallurgical units will define metallurgical variability and allow process engineers to design more robust flowsheet options. This variability can be muted when samples from different geometallurgical units are blended and tested as one sample. Composites are created by ensuring grade and grade distributions from a specific area defining the geometallurgical unit within a resource are maintained. The method used to divide an orebody into geometallurgical units is based on a review of geological data including host rock, alteration, grain sizes, texture, structural geology, grade, sulphide mineralogy and metal ratios with focus on characteristics which are known to affect metallurgical performance (Lotter et al., 2003). The foregoing list is, however, not complete and also uses hardness testing and the grade/recovery curve as characterising parameters (Fragomeni et al., 2005, for example). Statistical analysis is often used to help define preliminary units. In addition, it is recommended that a variability program based on smaller samples from throughout a geometallurgical unit is completed prior to finalising the divisions between geometallurgical units. This approach will quantify the range in performance that can be expected from within a unit, and provides a cross check that the geometallurgical unit definition is robust. Early predictions of likely grinding requirements of an ore using the sulphide grain size data obtained from a series of polished thin sections measured by QEMSCAN were proposed by Fragomeni et al. (2005). Earlier, equivalent work at Mount Isa Mines, Queensland, identified ranges of textures and associated grain sizes, leading to the concept of staged grinding and flotation (Bojcevski et al., 1998). Recently, an initiative to model geometallurgical units in terms of texture, predicted grind size and liberation behaviour from drill core using scanning electron microscopy was reported by Bonnici et al. (2009). Recently, this practice was advanced to a position whereby geometallurgical units may be populated with estimated recovery values of paymetals (Evans, 2010).

2.1.4. Strategy for mineralogical measurement

Scoping or pre-scoping level studies are often labelled Ore Characterisation programs. Corresponding mineralogical work is usually done on coarse samples so that in situ textures and mineral grain sizes can be defined prior to grinding. Once flotation testing has been completed on an ore, mineralogical characterisation of milled feeds, concentrate and tailings provides additional detail which can further define opportunities for improved metallurgical performance. These measurements can take place at either the scoping or pre-feasibility stage of the programme and when completed will provide additional information to help optimise the flowsheet.

Similar strategies are followed when diagnosing plant problems. Samples representing specific areas of a mine can be sampled to better understand the reasons for inferior metallurgical performance, or plant streams can be sampled to measure the mineralogical characteristics relating to a specific metallurgical result.

2.1.5. Quantitative mineralogy

Mineralogical data are collected using QEMSCAN/MLA and EPMA. At Xstrata Process Support, QEMSCAN is installed and used as an automated system that produces mineral maps (colour coded by mineral), through collection of rapidly acquired energy dispersive X-rays and/or by identification based on backscattered electron images. The mineral maps describe the texture and mineral associations in each of the samples. In addition to the coloured map, the output of the QEMSCAN measurement includes a quantitative measure of modal mineralogy, mineral grain size, mineral liberation and element deportment by mineral. A key component of the mineralogical result is that in addition to all economic minerals of interest, each gangue mineral is also identified and quantified.

In any automated mineralogical system, compositional information for every mineral is a required input to allow elemental deportment calculations. Although textbook compositions will provide a basis for these calculations, elements that occur as low level solid solution values are not captured when using textbook compositions. In addition, variations in composition can occur for the same mineral type from different deposits. Accurate deportment assessments require quantitative compositional analyses.

Quantitative compositional analysis at XPS is performed using a Cameca SX-100 Electron Microprobe. This model represents considerable improvement over the earlier microprobes, and includes programmable measurement programmes and imaging abilities that earlier models did not offer. Other platforms such as produced by Jeol also offer this type of capability. Compared to energy dispersive spectrometry (EDS), Electron Probe Microanalysis produces higher electron beam currents and increased beam stability, coupled with higher resolution wavelength dispersive spectrometry (WDS). These features allow for improved detection limits and accuracy of the resulting analysis. Detection limits can be as low as 100 ppm, providing detailed trace element compositions within the various mineral species. Individual grains and textures as small as 2–5 μm are targeted for analysis and care is taken whenever possible to ensure proportional analysis relative to species availability and grain size. Resulting detailed compositional data are then input back into the QEMSCAN software, in order to refine the final elemental deportment calculations. Compositional data from microprobe analysis can also be used to update the Species Identification Protocol (SIP) within QEMSCAN and mineralogical measurements can be reclassified in order to update overall modal and deportment data. The composition of minerals varies from deposit to deposit, and often from geomet unit to geomet unit within a deposit. In order to produce accurate deportment information, EPMA analysis on each geomet unit within a deposit is necessary. An example of this practice was presented by Kormos et al. (2010) describing the bimodal deportment of bismuth in bornite at the Antamina mine, Perú. For minerals that require a measurement with a lower detection limit that can be provided by EPMA, Laser Ablation (LAM-ICP-MS) should be considered to ensure the reporting of the correct composition in the metal deportment calculations (Cabri et al.,2010, for example).

The older X-ray Diffraction (XRD) technology may still be recognised for its place in quantitative mineral measurement. This technology was developed by W.L. and W.H. Bragg from 1912 on, and resulted in them being awarded the Nobel Prize in Physics in 1915. This platform has undergone steady modernisation and automation since then, including development of the Rietveld refinement, which uses a least squares approach to a best fit to the instrument's output, and dealt to some extent with the issue of overlapping reflections (Rietveld, 1969). Whereas XPS do have and use a modern automated XRD unit with Rietveld refinement software, this instrument is not integrally used in their flowsheeting work because of its higher detection limits and inability to measure mineral grain size because of the signal, i.e. diffraction, that is used (the samples presented to an XRD also have to be micronised). Rather, it is separately used for semi-quantitative measurement of the major mineral species, such as silicates, present in ore samples. This provides a useful introductory idea of the whole rock analysis. A good example was cited by Lotter et al. (2002, 2010) in the identification and semi-quantitative measurement of talc in a serpentinised section of the Raglan orebody, leading to a flotation testing programme to scope out gangue depressants for the flotation process at that site.

2.1.6. Mineral surface analysis – TOF-SIMS

In cases where it is desirable to understand the activation of mineral species and/or the identification of which collector has adsorbed on certain minerals in the flotation system being studied, TOF-SIMS has a definite place in the best practice of Modern Process Mineralogy. Various commercial laboratories now offer this service, thus it is not necessary to purchase the instrument and invest in the long learning and training curve involved, since the sample preparation methods and data interpretation are very specialist (Shackleton, 2007). However, the clear identification of dixanthogen on the TOF-SIMS platform is an important capability. Shackleton has reported progress with this objective in 2010 (Shackleton, 2010). As others develop the mixed collector platform and use dixanthogen for specific purposes in flotation (Chryssoulis et al., 2003; Lotter and Bradshaw, 2009; Lotter et al., 2011), this would be a key piece of future work. Dixanthogen has become recognised as a key species in the flotation of certain sulphides (chalcopyrite, discrete PGM, native gold, pyrrhotite, pyrite for example). Thus, in collector suites using only xanthate, if these minerals are the ones to be floated, dixanthogen will have to be produced, either by acidic pH, by oxidation with copper sulphate, or by dithiocarbamate catalysis (Bradshaw, 1997).

2.1.7. Metallurgical testing

In addition to the tools described to measure mineralogical characteristics, XPS uses several mineral processing procedures in an attempt to correlate mineralogy with the metallurgical performance. These procedures use replicate flotation as described in high confidence flotation testing (Lotter, 1995; Lotter and Fragomeni, 2010) and factorial design of experiments to quantify and minimise fundamental error and allow for distinguishing real differences in metallurgical performance between the geometallurgical units described.

2.1.7.1. High confidence flotation testing. High confidence flotation is based on two principles: one, to ensure that the ore sample is representative and has been well-blended and representatively subsampled; two, to perform a sufficient number of flotation tests with appropriate quality controls, so as to improve the reproducibility of the test data and reduce the error level. The use of representative samples, as previously discussed, is a necessary prerequisite for these high confidence tests (Lotter, 1995).

The crushed mill feed sample is blended using a spinning riffler with an odds and evens procedure as referenced in the earlier discussion. After blending, a total of 10–12 subsamples are taken from the blended 2 kg charges. The 10–12 pulverised samples are pooled to form an external reference distribution, which estimates the sample mean grade. It is a requirement that the relative standard deviation of these measurements does not exceed a set criterion.

A minimum number of replicate flotation tests is required for each high confidence flotation test observation. This is so as to engage the powerful averaging effects of the Central Limit Theorem, which in this context reduces the random errors and leaves residual error normally-distributed (Grant and Leavenworth, 1988). It is an additional requirement that the first rougher concentrate masses agree within a prescribed relative standard deviation. As the procedure became applied to a wider range of base metal ores, this rule was rewritten so as to determine this point of measurement was made according to the cumulative recovery, and not the cumulative flotation time (Fragomeni et al., 2002). The sets of reconciled head grades resulting from the mass and value balances of the flotation tests are pooled to form the internal reference distribution. The sample mean and standard deviation are calculated. The external reference distribution and the internal reference distribution means are compared as a percentage ratio called the call factor. This call factor must lie within a range of 96.73–103.27%. If not, the metal balance lies outside the 95% confidence limits and the flotation test is rejected and repeated.

2.1.7.2. Factorial design of experiments. Factorial design or design or experiments (DOE) is documented elsewhere in detail (Box et al., 1978a). The use of DOE is significant when used in conjunction with quantitative mineralogy as mineralogical features can be manipulated using a combination of mineral processing treatments such as grind size or reagent type and quantity. Frequently, XPS uses DOE laboratory scale testing in a three variable, two level DOE as a suitable format. The factorial cube involves eight test points, plus midpoint replication.

The sequence of these tests is randomised so as to avoid any operator preference. Data are interpreted in terms of main effects and interactions on key performance measurements such as tailings grade, concentrate grade or recovery. This analysis is done using Mini-Tab or Stat-Ease software, however the same results can be calculated by hand.

The key advantage of this testwork layout is that each main effect is calculated from eight observations. Therefore, the standard error of that main effect is much lower than with conventional singleton or duplicate tests. It is thus possible to test for small differences in recovery and establish, for example, the best combination or grind size and reagent addition as suggested by the mineralogy. Typically, optimised conditions are tested using the high confidence testing protocols to further increase confidence in results. Modelling of the response surface within the cube forms the final step of the data interpretation. This often finds an internal (untested) point in the cube which offers superior performance (Deng et al., 2010). In certain cases, a partial factorial in the form of a Latin Square may be performed where prior information has already been developed.

2.1.7.3. Flotation mini pilot plant. Typically, high confidence flotation testing and DOE procedures are performed on a batch basis in either rougher or open circuit cleaner formats. Following batch testing and correlation between quantitative mineralogy and metallurgical testing, the geometallurgical units and flotation conditions are confirmed. Once confirmed, these geometallurgical units can be tested in a locked cycle test or in continuous mode using a Mini Flotation Pilot Plant (MPP) (Fragomeni et al., 2006).

The inherent advantages of the MPP over conventional piloting include smaller sample size, reduced time to steady state and ability to test samples on a geometallurgical unit basis. This smaller sample size is made possible by stratified sampling. The MPP requires significantly lower sample mass than a conventional pilot plant, and can operate a continuous flowsheet test including regrinding and column cleaning at feed rates as low as 8 kg/h and a total campaign ore sample requirement of 600 kg. Typically, this sample mass is available from pre-feasibility level exploration drilling programs, enabling the creation of a representative composite rather than using a bulk sample from one location within a resource. Testing of individual geometallurgical units or specific production periods are possible. MPP testing has been used for evaluation of reagent changes, (DiFeo, 2006) testing of various flowsheet concepts on a continuous scale, (Yu and Fragomeni, 2006) or to define design basis parameters for scale up and design (Ouellet and Fragomeni, 2007).

2.1.7.4. Statistical Benchmark Surveying – a sampling strategy. At plant operations scale, a procedure was developed and validated whereby a representative suite of flowsheet samples could be taken from an operating concentrator for mass and value balancing, followed by QEMSCAN measurement across a series of closed size fractions. This became known as Statistical Benchmark Surveying, and made the connection between the operating plant and the microscopic-scale measurements of the minerals. The key in this system is to tie the tests of representativity to measurements of ore grade, so that the accepted set of flowsheet samples correspond to treatment of ore of similar grade to that which is typically milled at that operation in the 3-month period surrounding the survey. Ultimately two forms of this survey were developed. One, the standard benchmark form, or "Statistical Benchmark Survey", described the flowsheet behaviours under typical mining and milling conditions (Lotter, 2005; Lotter and Laplante, 2007a). This is used to update a concentrator's performance across several years, identifying the flowsheet opportunities for improved performance in stages. The other, called the "Campaign Survey", was specifically designed to capture a suite of flowsheet samples for a known ore type that had been specifically mined and stockpiled for a demonstration run that would last between a week and 10 days (Lotter and Laplante, 2007b). Both systems operate at the 95% confidence level.

2.1.7.5. Successful plant trials – a measurement strategy. The role of applied statistics has long presented an opportunity in mineral processing, both at laboratory scale in flotation testing, and at plant scale with trials such as new reagents (Lotter, 1995; Napier-Munn, 1995, 1998; Napier-Munn and Meyer, 1999; Xiao et al., 2009; Lotter et al., 2010, for example). The problem of noisy autocorrelated data in the plant measurements of feed, concentrate and tailings challenges our endeavours to prove small improvements in grade and/or recovery that we have worked hard at laboratory scale to achieve. These small gains are worth significant financial revenues to the mining company concerned, yet are commonly delegated to the realm of the unprovable by the operations staff, leaving several small opportunities that could collectively amount to a large sum of extra operating revenue if all successfully implemented and proven. Rather, larger performance targets have been sought and pursued; however, in this case there are fewer opportunities.

By recognising the autocorrelation problem and rather designing the plant trial with this problem in mind, it is possible to break the autocorrelation by using a replicated blocking technique. This results in a plant trial that operates in an on–off format with variable block lengths of anywhere from 1 to 2 weeks. In one version, such as Lotter et al. (2010) the reference distribution is written in two dimensions (grade and recovery) and is analysed by the Analysis of Variance (ANOVA) for significance testing. Otherwise, use of the more conventional moving average reference distribution suitably deals with a reagent trial (Xiao et al., 2009) In the case where it is not possible to revert to the "off" position, such as the irreversible installation of a regrind mill or a different cyclone nest, the approach takes a slightly different form using the moving average version of reference distributions (Box et al., 1978b).

In all, this interface between applied statistics and successful plant trials has contributed to a recent history of more successful proofs of these small performance opportunities, providing a key technology transfer tool for Process Mineralogy.

3. A case study – Montcalm operations

The Montcalm Ni/Cu Ore was processed at the Kidd Creek concentrator located near Timmins, in Northern Ontario, Canada between 2004 and 2009. It is hosted in a norite and gabbro intrusive complex with minor peridotite lenses and mafic and granodiorite dykes. Shear zones and faults are locally encountered and host chloritic alteration products including talc (Charland et al., 2006). The following summary was made from this publication as a case study using Modern Process Mineralogy, to demonstrate the diagnostic power and accurate scale-up characteristics of this integrated modern practice.

The Montcalm ore reserves occurred as three distinct lenses, referred to as the East, West and Deep Zones. The mineralogical assemblage is locally variable, with changing ratios of the main sulphides: pyrrhotite, pyrite, pentlandite and chalcopyrite. The silicate gangue is primarily composed of plagioclase and amphibole, exhibiting variable degrees of sericitization or chloritization. Calcite, magnetite, zoisite (a Ca–Al silicate), quartz, biotite and talc occur as minor accessory gangue minerals.

Table 1 lists the reserve tonnages and average grades for each zone as estimated by Falconbridge (September 6, 2002). It was initially a short life-of-mine project of seven years, thus the importance of early metallurgical optimisation was highlighted. The operation was, however, shut-down in 2009 due to the development of certain technical problems in the mining operation.

At the time Falconbridge Limited acquired full ownership of the project, an extensive body of metallurgical work existed from the prior Outokumpu ownership. It was recognised, however, that further work would be necessary to align this project with Falconbridge Project Guidelines. Falconbridge's mandate was to evaluate the project's economic viability and validate the proposed flowsheet (shown in Fig. 1). In order to mitigate risk with respect to the metallurgical performance of the ore, several phases of mineralogical and metallurgical work were initiated. This extra work would make an accurate prediction of the expected grade and recovery to be obtained from the inherited flowsheet.

In Fig. 1, the flowsheet shows that soda ash is added to the primary mill (A) to modify the pH to 9.5. At the mill discharge, where a designed grind size of 87% passing 45 μm was specified by the earlier project history, additional soda ash is added to complete the pH control. This will depress most of the pyrite. Xanthate PIBX and frother MIBC are added to the float feed (B). A bulk rougher and scavenger concentrate is collected and presented to a two-stage cleaner section. The first cleaner tailings recycle to the rougher float feed (B), and the second cleaner tailings, to the first cleaner feed. Lime, dextrin and frother MIBC are added at D. The testwork was focused on making statements about the grade and recovery at the point of release of the final bulk concentrate from the second cleaner.

Table 1
Salient features of the Montcalm ore reserve as at 2002.

Geomet unit	East	West	Deep	Total
Parameter				
Tonnes	1,910,000	3,780,000	1,690,000	7,380,000
Grade% Ni	1.24	1.45	1.41	1.39
Grade% Cu	0.56	0.73	0.67	0.67

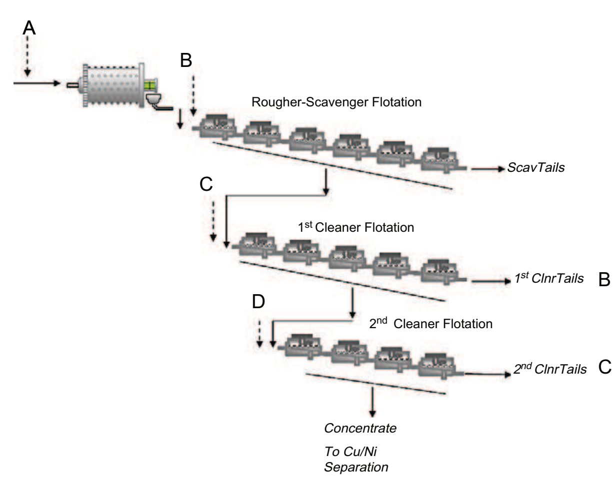

Fig. 1. Schematic of the Montcalm flowsheet.

3.1. Process Mineralogy study

It was important to understand the variability that the concentrator would probably encounter. Use of the geomet unit approach (described earlier in this paper) found three geomet units. These were massive sulphides, net-textured sulphides and disseminated sulphides. Sample material taken from drill core allowed a whole rock thin section investigation, and size-by-size liberation measurements of laboratory scale simulated rougher float feed at the designed grind. High-Confidence Flotation Testing (HCFT) was then used to simulate the specified flowsheet and to characterise the probable metallurgical response.

Images of polished thin sections prepared from these geomet units are shown in Fig. 2. The Process Mineralogy study at Falconbridge prior to commissioning identified two key processing implications for the frozen flowsheet. One, the designed grind was finer than necessary for the pentlandite, and would not favour optimal grade and recovery; and two, that pyrite was present in the ore at significant and variable levels. Given the well-known positive flotation characteristics of this mineral (Bradshaw, 1997), the flowsheet would have to devise a pyrite depression strategy so as to minimise dilution of the bulk concentrate. An example of the massive pyrite is shown in Fig. 3. A coarsening of the grind from a d80 size of 39–53 μm was recommended after commissioning.

A Life-of-Mine drill core composite was prepared for flotation testing to simulate the flowsheet as set out in Fig. 1. The approach of HCFT was used. Ultimately the work led to a locked cycle test to simulate the production of the final bulk concentrate. The results are summarised, together with commissioned plant data, in Table 2. Although HCFT is an integral part of Process Mineralogy practiced at XPS, the specific contribution to the scale-up success in this project is discussed in detail by Lotter and Fragomeni (2010).

Further, a McNulty start-up model was constructed after the commissioning to benchmark the start-up in terms of type. The format of the model is discussed in detail by McNulty (1998). The result is shown in Fig. 4.

The Montcalm start-up met or exceeded Type 1, the very best outcome desired. This is directly a result of the Process Mineralogy approach. Thereafter, a Statistical Benchmark Survey of the concentrator operations was commissioned so as to identify and quantify the next flowsheet opportunities (Fragomeni et al., 2009).

Since the life-of-mine was estimated at 7 years, concentrator optimisation endeavours had to be fast-tracked. These joint endeavours between the Kidd Creek Metallurgical Site staff and Xstrata Process Support were focussed on increasing ore treatment capacity and metallurgical performance. This survey and associated work reviews these endeavours. A flotation circuit survey, together with appropriate Process Mineralogy measurements, identified recovery opportunities in both locked and overground pentlandite in final tailings, confirming what had already been identified in the earlier pre-commissioning project study. The bypassing of first rougher concentrate directly to final concentrate was also recommended, given the measured composition of that stream. A subsequent grinding circuit survey identified a low circulating load due to poor classification performance. Modelling of these data suggested a change to the cyclone nest and pumping arrangements in the grinding circuit. Implementation of these recommendations has led to a 25% gain in milling circuit capacity as well as improved flotation circuit performance. These improvements amount to nickel and copper recovery gains of 2.57% and 1.83% respectively (Fragomeni et al., 2009).

These outcomes confirm the ability of Process Mineralogy to retrofit improvements to a non-optimal design that has been commissioned into operation, as well as the key ability to scale-up accurately from laboratory testwork to production operations.

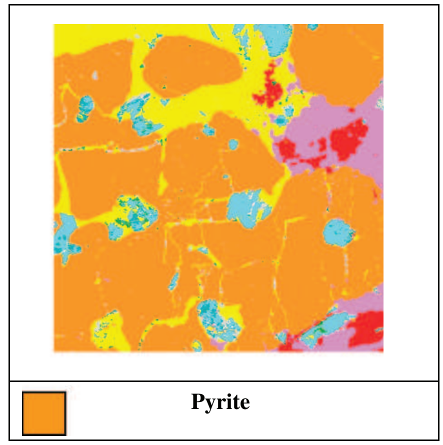

Fig. 3. Polished thin section showing massive pyrite grains.

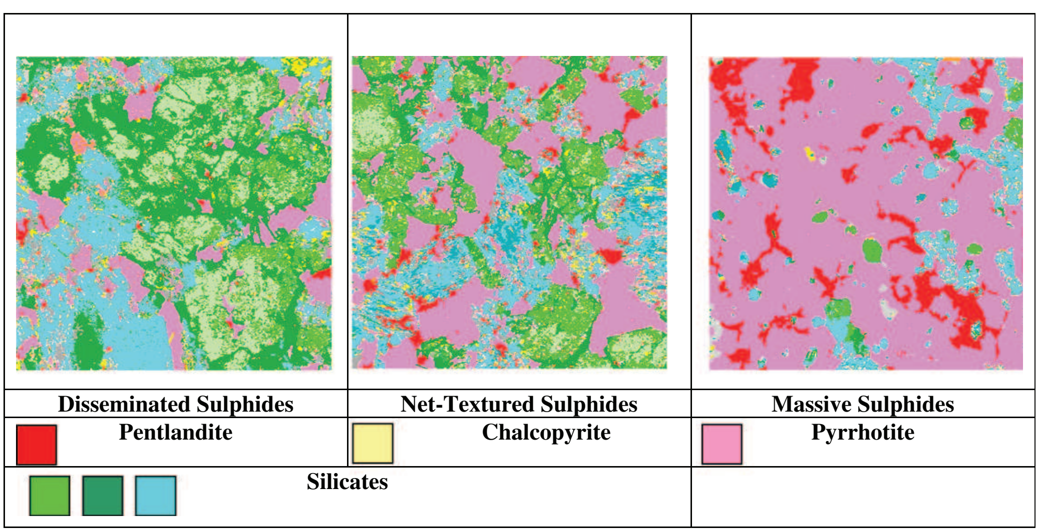

Fig. 2. Polished thin section images of Montcalm geomet units.

Table 2
Comparison of laboratory scale and commissioned performances[a].

Scale	Laboratory: High-Confidence Flotation Testing 2002	Commissioned project: Statistical Benchmark Survey of operations 2005
Nickel		
Bulk concentrate grade% Ni	9.0	9.93
Bulk concentrate recovery%	82.9	84.0

[a] At bulk concentrate stage before copper–nickel separation.

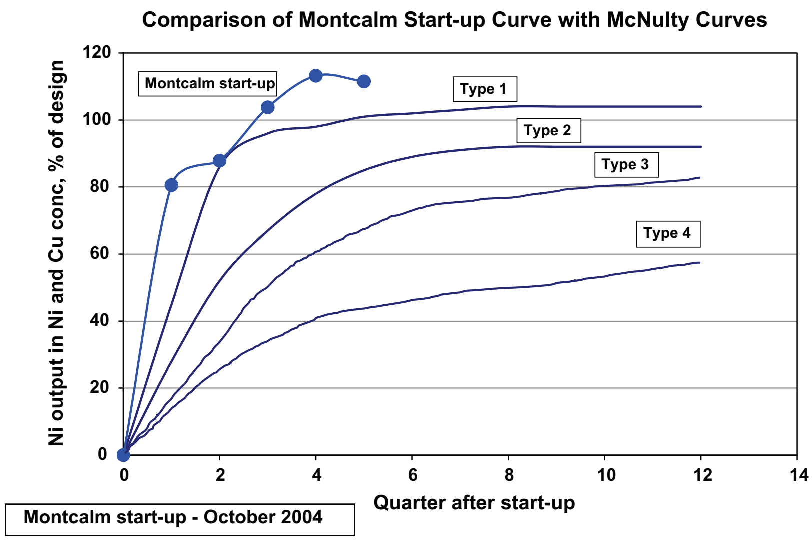

Fig. 4. McNulty model of the Montcalm start-up.

4. Concluding remarks

As a discipline, Process Mineralogy has come a long way in a short time. If we regard the milestone reference of Henley (1983) as the turning point, the current generation of the discipline as such has been under development for 27 years. In this generation, many of the empowering technologies such as QEMSCAN and the MLA, the advancement of the EPMA to a more modern platform, and the mini pilot plant, have been developed and commercialised. Other significant empowering developments have been successfully validated and entrenched in best practices. These include, but are not limited to, techniques such as connecting the sampling models of Pierre Gy to the procedures; High-Confidence Flotation Testing; the use of the conventional polished thin section in both the microprobe and QEMSCAN to lead into virtual flowsheeting, an understanding of the molecular species present on mineral surfaces via TOF-SIMS, etc.

Provided that future Process Mineralogy teams keep the ongoing practice of hybridized operations in which synergy develops, it is certain that many more significant tools and methods will develop in this field.

Acknowledgements

The author would like to gratefully thank the management of Xstrata Nickel for permission to publish this paper. They and the earlier Falconbridge Ltd. have been a founder client for this Xstrata Process Mineralogy Group, and have provided many meaningful projects and associated challenges for us to work on and solve whilst we developed the practice. Additionally I would like to sincerely thank my colleagues and co-founders of the Xstrata (and earlier Falconbridge) Process Mineralogy team for their energetic and innovative contributions to the group. Several others, such as Professor Dee Bradshaw, of the SMI, University of Queensland; Professor Cyril O'Connor, of the University of Cape Town; Dr. Michael King, retired Director of the earlier Falconbridge Metallurgical Technology Group; Paul Gottlieb, originally of CSIRO, and now of FEI; and Greg Wilkie, originally of CSIRO, and now of Rio Tinto, are gratefully acknowledged for their encouragement and support in these endeavours.

References

Barbery, G., Huet, G., Gateau, G., 1979. Liberation analysis by means of image analysers: theory and applications. In: Proc XIII IMPC, vol. 2, pp. 568–599.
Bartlett, H.E., Hawkins, D., 1989. The extractive metallurgy of gold in South Africa. In: Stanley, G., (Ed.), Process Control. SAIMM Monograph, vol. 2 (Chapter 13).
Bodziony, J., 1965. On the possibility of application of integral geometry methods in certain problems of liberation of mineral grains. Bull. Acad. Polonaise Sci. Ser. Sci. Tech. 13, 459–467.
Bojcevski, D., Vink, L., Johnson, N.W., Landmark, V., Mackenzie, J., Young, M.F., 1998. Metallurgical characterisation of George Fisher ore textures and implications for ore processing. In: AusIMM Mine to Mill Conference, Brisbane, Australia, 11–14 October 1998, pp. 29–41.
Bond, F.C., 1952. The third theory of comminution. Trans. AIME Miner. Eng. 193, 484–494.
Bonnici, N., Hunt, J., Walters, S., Berry, R., Kamenetsky, M., McMahon, C., Nguyen, K., 2009. Integrating meso- and micro-textural information into mineral processing: an example from the Ernest Henry iron–oxide copper–gold deposit. In: Proc. Canadian Mineral Processors, Ottawa, January 2009, Paper 16, pp. 259–278.
Box, G.E.P., Hunter, W.G., Hunter, J.S., 1978a. Statistics for Experimenters. Wiley, New York (Chapter 10).
Box, G.E.P., Hunter, W.G., Hunter, J.S., 1978b. Statistics for Experimenters. Wiley, New York. pp. 190–194 (Chapter 6).
Bradshaw, D.J., 1997. Synergistic Effects between Thiol Collectors Used in the Flotation of Pyrite. Ph.D. Chem. Eng. Thesis, University of Cape Town, 261 pp.
Cabri, L.J., 1981. Relationship of mineralogy for the recovery of PGE from ores. In: Cabri, L.J. (Ed.), Platinum-Group Elements: Mineralogy, Geology, Recovery, special vol. 23. Canadian Institute of Mining, Metallurgy and Petroleum, pp. 233–250.

Cabri, L.J., Choi, Y., Nelson, M., Tubrett, M., Sylvester, P., 2010. Advances in precious metal trace element analyses DOE deportment using LAM-ICP-MS. In: Proc. Canadian Mineral Processors, Ottawa, January 2010, Paper No. 12, pp. 181–196.

Charland, A., Kormos, L.J., Whittaker, P.J., Arrué-Canales, C.A., Fragomeni, D., Lotter, N.O., Mackey, P, Anes, J., 2006. A case study for the integrated use of automated mineralogy in plant optimisation: the Montcalm concentrator. In: Proc. Automated Mineralogy, MEI Conference, Brisbane, July 2006.

Chryssoulis, S.L., Venter, D., Dimov, S., 2003. On the floatability of gold grains. In: Proc. Canadian Mineral Processors, Paper No. 28, pp. 455–472.

Cochran, W.G., 1946. Relative accuracy of systematic and stratified random samples for a certain class of populations. J. Ann. Math. Stat. 17, 164–177.

Dai, Z., Bos, J.-A., Lee, A., Wells, P., 2008. Mass balance and mineralogical analysis of flotation plant survey samples to improve plant metallurgy. In: Minerals Engineering, "Flotation 2007". special 21 ed., pp. 826–831.

Deng, T., Yu, S., Lotter, N.O., Di Feo, A., 2010. Laboratory testwork of mixed xanthates for the raglan ore. In: Proc. Canadian Mineral Processors, Ottawa, January 2010, Paper No. 16, pp. 253–268.

DiFeo, A., 2006. Mini Pilot Plant Craig Ore – DNBX. Falconbridge Technology Centre, Internal Memo, June 2006.

Evans, C.L., 2010. Development of a Methodology to Estimate Flotation Separability from Ore Microtexture. Ph.D. Thesis, University of Queensland, Brisbane, December 2010, 279 pp.

Fandrich, R., Gu, Y., Burrows, D., Moeller, K., 2007. Modern SEM-based mineral liberation analysis. Int. J. Miner. Proc. 84 (1–4), 310–320.

Fragomeni, D., Lotter, N.O., 2002, Personal Communication. Falconbridge.

Fragomeni, D., Boyd, L.J., Charland, A., Kormos, L.J., Lotter, N.O., Potts, G., 2005. The use of end-members for grind-recovery modelling, tonnage prediction and flowsheet development at Raglan. In: Proc. Canadian Mineral Processors, Ottawa, January 2005, Paper No. 6, pp. 75–98.

Fragomeni, D., Hoffman, M., Kelly, A., Yu, S., Lotter, N.O., 2006. Flotation mini pilot plant experience at Falconbridge Limited. In: Proc. Mineral Process Modelling, Simulation and Control, Laurentian University, Sudbury, Canada, pp. 329–355.

Fragomeni, D., Yu, S., Anes, J., Edwards, T., Scott, D., 2009. Advancing asset performance at the Montcalm operation. In: Proc. Canadian Mineral Processors, Ottawa, January 2009, Paper No. 12, pp. 205–219.

Gaudin, A.M., 1939. Principles of Mineral Dressing. McGraw-Hill, New York.

Gottlieb, P., Wilkie, G., Sutherland, D.N., Ho-Tun, E., Suthers, S., Perera, K., Jenkins, B., Spencer, S., Butcher, A., Rayner, J., 2000. Using quantitative electron microscopy for process mineralogy applications. JOM 52 (4), 24–25.

Grant, E.L., Leavenworth, R.S., 1988. Statistical Quality Control. McGraw-Hill. pp. 213–215.

Grant, G., Hall, J.S., Reid, A.F., Zuiderwyk, M., 1976. Multi-compositional particle characterisation using the SEM-microprobe. In: Proc. Scanning Electron Microscopy/1976, Part III, Workshop on Techniques for Particulate Matter Studies in SEM, ITT Research Institute, pp. 401–408.

Gu, Y., 2003. Automated scanning electron microscope based mineral liberation analysis. JOM 2 (1), 33–41.

Gy, P.M., 1979. Sampling of Particulate Materials, Theory and Practice. Elsevier, Amsterdam.

Henley, K.J., 1983. Ore-dressing mineralogy: a review of techniques, applications and recent developments. Spec. Publ. Geol. Soc. South Africa 7, 175–200.

Johnson, N.W., 1987. Applications of kinetics and liberation data to analysis of an industrial flotation process. CIM Bull. 80 (899), 113–117.

King, R.P., 1979. A model for the quantitative estimation of mineral liberation by grinding. Int. J. Miner. Proc. 6 (3), 207–220.

King, R.P., 1989. Mineral Liberation and its Influence on Mineral Processing Operations. SME, Denver.

Kormos, L.J., Oliveira, J., Fragomeni, D., Whiteman, E., Carrión De la Cruz, J., 2010. Process diagnosis using quantitative mineralogy. In: Proc. Canadian Mineral Processors, Ottawa, January 2010, Paper No. 8, pp. 97–120.

Lotter, N.O., 1995. A Quality Control Model for the Development of High-Confidence Flotation Test Data. M.Sc. Chem. Eng. Thesis, University of Cape Town, June 1995.

Lotter, N.O., 2005. Statistical Benchmark Surveying of Production Concentrators. Ph.D. Met. Eng. Thesis, McGill University, Montréal.

Lotter, N.O., 2010. Stratified sampling of drill core. In: Proc. Canadian Mineral Processors, Ottawa, January 2010, Paper No 11, pp. 163–179.

Lotter, N.O., Bradshaw, D.J., 2009. The formulation and use of mixed collectors in sulfide flotation. In: Proc. MEI "Flotation '09", Cape Town, South Africa, November 2009.

Lotter, N.O., Fragomeni, D., 2010. High-confidence flotation testing at Xstrata Process Support. J. Miner. Metall. Process. 27 (1), 47–54.

Lotter, N.O., Laplante, A.R., 2007a. Statistical benchmark surveying of production concentrators. Miner. Eng. 20, 793–801.

Lotter, N.O., Laplante, A.R., 2007b. The campaign survey model – a case study at the Raglan mine, Québec. Miner. Eng. 20, 480–486.

Lotter, N.O., Whittaker, P.J., Kormos, L.J., Stickling, J.S., Wilkie, G.J., 2002. The development of process mineralogy at Falconbridge Limited, and application to the Raglan mill. CIM Bull. 95 (1066), 85–92.

Lotter, N.O., Kowal, D.L., Tuzun, M.A., Whittaker, P.J., Kormos, L.J., 2003. Sampling and flotation testing of Sudbury Basin drill core for process mineralogy modelling. Miner. Eng. 16, 857–864.

Lotter, N.O., Bradshaw, D.J., Becker, M., Parolis, L.A.S., Kormos, L.J., 2008. A discussion of the occurrence and undesirable flotation behaviour of orthopyroxene and talc in the processing of mafic deposits. Miner. Eng. 21, 905–912.

Lotter, N.O., Di Feo, A., Kormos, L.J., Fragomeni, D., Comeau, G., 2010. Design and measurement of small recovery gains – a case study at Raglan concentrator. Miner. Eng. 23, 567–577.

Lotter, N.O., Monnapula, R., Oliveira, J., Fragomeni, D., Brdshaw, D.J., 2011. Formulation and plant trial of a mixed collector suite for eland platinum. In: Proc. Canadian Mineral Processors, Ottawa, January 2011, Paper No. 10, pp.161–183.

Mackey, P.J., Nesset, J.E., 2003. The impact of commissioning and start-up performance on a mining/metallurgical project. In: Proc. Canadian Mineral Processors, Ottawa, January 2003, Paper No. 21, pp. 331–347.

Malysiak, V., Shackleton, N.J., O'Connor, C.T., 2004. An investigation into the floatability of a pentlandite–pyroxene system. Int. J. Miner. Process. 74, 251–262.

Martinovic, J., Bradshaw, D.J., Harris, P.J., 2005. Investigation of surface properties of gangue minerals in platinum bearing ores. J. South African Inst. Min. Metall. 105, 1–7.

McIvor, R.E., Finch, J.A., 1991. A guide to interfacing of plant grinding and flotation operations. Miner. Eng. 4 (1), 9–23.

McIvor, R., Lavallee, M., Wood, K., Blythe, P., 1990. Functional performance characteristics of ball milling. In: Proc. Canadian Mineral Processors, Ottawa, pp. 100–126.

McKay, N., Wilson, S., Lacouture, B., 2007. Ore characterisation of the Aqqaluk deposit at Red Dog. In: Proc. Canadian Mineral Processors, Ottawa, January 23–25, 2007, Paper No. 5, pp. 55–74.

McNulty, T., 1998. Developing innovative technology. Min. Eng. 50 (10), 50–55.

Middleditch, D., Lotter, N.O., 2008. Laboratory Quality Standards Manual. Xstrata Process Support Internal Document, May 13, 2008, pp. 14–16.

Middleditch, D., Carriere, P., Danyliw, J., 2009. Crushing Plant Manual. Xstrata Process Support Internal Document, October, 2009, pp. 22–26.

Nagaraj, D.R., Brinen, J.S., 1996. SIMS and XPS study of the adsorption and of sulphide collectors on pyroxene: a case for inadvertent metal ion activation. J. Colloids Surf. A 116, 241–249.

Nagaraj, D.R., Brinen, J.S., 1997. SIMS analysis of flotation collector adsorption and metal ion activation on minerals: recent studies. J. Indian Inst. Metals 50 (5), 365–376.

Napier-Munn, T.J., 1995. Detecting performance improvements in trials with time varying mineral processes – three case studies. Miner. Eng. 8 (8), 843–858.

Napier-Munn, T.J., 1998. Analysing plant trials by comparing recovery – grade regression lines. Miner. Eng. 11 (10), 949–958.

Napier-Munn, T.J., Meyer, D.H., 1999. A modified paired *t*-test for the analysis of plant trials with data autocorrelated in time. Miner. Eng. 12 (9), 1093–1100.

O'Connor, C.T., Malysiak, V., Shackleton, N.J., 2006. The interaction of xanthates and amines with pyroxene activated by copper and nickel. Miner. Eng. 19, 799–806.

Ouellet, N., Fragomeni, D., 2007. Design Basis Summary for the Kabanga Plant. XPS Final Report, November 17, 2007.

Petruk, W., 1976. The application of quantitative mineralogical analysis of ores to ore dressing. CIM Bull. 767, 146–153.

Petruk, W., Hughson, M.R., 1977. Image analysis evaluation of the effect of grinding media on selective flotation of two zinc–lead–copper ores. CIM Bull. 787, 128–135.

Petruk, W., Schnaar, J.R., 1981. An evaluation of free and unlibarated mineral grains, metals and trace elements in the concentrator of Brunswick Mining and Smelting Corporation Limited. CIM Bull. 833, 132–159.

Peyerl, W., 1983. The metallurgical implications of the mode of occurrence of platinum-group metals in the Merensky Reef and UG-2 chromitite of the Bushveld complex. Spec. Publ. Geol. Soc. South Africa 7, 295–300.

Rao, S.R., Finch, J.A., 1988. Galvanic interaction studies on sulphide minerals. Can. Metall. Quart. 27 (4), 253–259.

Restarick, C.J., 1976. Pulp sampling techniques for steady state assessment of mineral concentrators. Proc. Sampl. Symp. Aust. Inst. Min. Metall., 161–168.

Rietveld, H.M., 1969. A profile refinement method for nuclear and magnetic structures. J. Appl. Crystallogr. 2, 65–71.

Shackleton, N., 2007. Personal Communication, Johannesburg, South Africa.

Shackleton, N.J., 2010. Personal Communication, Johannesburg, South Africa.

Shackleton, N.J., Malysiak, V., O'Connor, C.T., de Vaux, D.V., 2003a. The role of complexing agents and dispersants in pentlandite–pyroxene separation by flotation. In: Proceedings of the XXII IMPC, Cape Town, pp. 743–752.

Shackleton, N.J., Malysiak, V., O' Connor, C.T., 2003b. The use of amine complexes in managing inadvertent activation of pyroxene in a pentlandite–pyroxene flotation system. Miner. Eng. 16, 849–856.

Shannon, E.R., Grant, R.J., Scott, D.W., 1993. Back to basics – the road to recovery milling practice at Brunswick. In: Proc. Canadian Mineral Processors, Ottawa, January 1993, Paper No. 2.

Smart, R.St.C., Amarantidis, J., Skinner, W., Prestidge, C.A., LaVanier, L., Grano, S., 1996. Surface analytical studies of oxidation and collector adsorption in sulphide mineral flotation. J. Scan. Microsc. 12 (4), 553–583.

Stowe, K.G., Chryssoulis, S.L., Kim, J.Y., 1995. Mapping of composition of mineral surfaces by Tof-SIMS. Miner. Eng. 8, 421–430.

Sutherland, D.N., 1993. Image analysis for off-line characterisation of mineral particles and prediction of processing properties. Particle Particle Syst. Charact. 10, 271–274.

Trahar, W.J., 1981. A rational interpretation of the role of particle size in flotation. Int. J. Miner. Process. 8, 289–327.

Triffett, B., Veloo, C., Adair, B.J.I., Bradshaw, D.J., 2008. An investigation into the recovery of molybdenite in the Kennecott Utah Copper bulk flotation circuit. In: Minerals Engineering, "Flotation 2007". special 21 ed., pp. 832–840.

Xiao, Z., Lockhart, C., Reynolds, J., 2009. Statistical applications in planning and analyzing plant flotation trials of evaluating a collector. Int. J. Miner. Proc. 92 (3–4), 196–200.

Young, M.F., Pease, J.D., Johnson, N.W., Munro, P.D., 1997. Developments in milling practice at the lead/zinc concentrator of mount is a mines limited from 1990. In: Proc. Aus. I.M.M. Sixth Mill Operators Conference, Madang, Papua New Guinea, October 1997.

Yu, S., Fragomeni, D., 2006. '06 Nickel Rim South Mini Pilot Flotation Plant Campaign Report. Falconbridge Technology Centre Internal Report, September 2006.