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An Introduction to ZEISS Mineralogic Mining and the correlation of light microscopy with automated mineralogy: a case study using BMS and PGM analysis of samples from a PGE-bearing chromitite prospect.

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Abstract

The release of ZEISS Mineralogic Mining has provided an extra dimension to the world of Automated Mineralogy (AM) through the utilization of a fully quantitative Energy Dispersive Spectroscopy (EDS) mineral classification system and advanced image analysis capabilities. A key point of difference for Mineralogic Mining is the way mineralogy is classified by using the wt% contribution of the elements present, and thus, the minerals stoichiometry. This method provides the operator a more accurate, flexible and efficient method for generating mineral classifications, as well as providing a direct measurement of assay and chemical composition. In combination, ZEISS have developed the unique capability to correlate Light Microscope (LM), Scanning Electron Microscope (SEM) and AM coloured mineral maps for more efficient and seamless analysis between these techniques in order to make the most of the information each technique provides. This paper describes the ZEISS Mineralogic Mining capabilities and workflow, and provides an example of correlating these techniques for locating and identifying Base Metal Sulphides (BMS) and Platinum-Group Minerals (PGM) from a PGE-bearing chromitite prospect.

Keywords: ZEISS Mineralogic Mining, automated mineralogy, light microscopy, correlation.

Introduction

Platinum-Group Element (PGE) deposits pose a number of challenges for mineralogists who aim to characterise the PGM, their associated mineralogy and their textural associations (i.e. size, liberation and distribution etc.). PGM are typically in the 1 – 20 μm size range and relatively rare, which makes locating and accurate identification difficult and time consuming when using reflected light techniques (e.g. Henley, 1992). Furthermore, data gathered using LM techniques is often subjective and descriptive in nature (Schouwstra and Smit, 2011), rather than being repeatable and quantitative. Despite these limitations, LM techniques are still able to provide more than a useful first impression of the sample in terms of mineralogy and textural features (Schouwstra and Smit, 2011).

The development of SEM and AM techniques have been widely employed to find and classify the PGM, their size, associated sulphide-silicate phases and liberation. The development of the 1st generation of AM techniques revolutionised mineralogical studies greatly improving data quantity, quality and speed of these studies (e.g. Goodall and Scales, 2007). Recent studies have begun to quantify the value these solutions can provide in terms of providing data that allows informed decisions to be made (Gu *et al.* 2014; Rule and Schouwstra, 2011; MacDonald et al, 2011; Lotter et al 2011). However, despite these developments the rarity of PGM in some sample streams, means a large number of samples must be analysed to gain a representative data set of the PGM (e.g. Bushell, 2011).

Techniques such as LM, SEM and AM are often viewed in isolation by manufacturers; as a result, there has been limited functional development on the correlation and application of these techniques within process mineralogy. It is possible to (using an automated LM stage) take tiled images of polished blocks and thin sections, and create large area montaged images. It is possible to produce a similar montage view using high resolution SEM images (using say backscatter electrons). It is also possible to produce digital mineral maps of the sample using automated mineralogy techniques. What has been missing is the ability to:

- Correlate stage coordinates and areas of interest between instruments
- Correlate images into data layers to benefit from the unique data available from each, and to
- Have an optimised workflow for data collection and analysis

ZEISS is able to undertake such correlation, and is developing a workflow with the potential to increase sample throughput by using LM to efficiently identify areas or particles of interest, or to screen for target samples, ready for more detailed characterisation using SEM and Mineralogic. One particular application for this would be to search for and characterise PGM's in a quicker and/or more cost effective manner.

This paper aims to present the first description of ZEISS Mineralogic Mining and provide a case study of the capabilities of ZEISS Mineralogic Mining and the LM correlation with the SEM-AM workflows.

ZEISS Mineralogic Mining

ZEISS Mineralogic Mining is an automated mineralogy and petrological analyser designed to provide a technological and methodological advance over traditional AM techniques (Tonzetic, 2014; Graham *et al.,* 2014a and references herein). Amongst the advances, Mineralogic Mining offers an advanced image processing capability and fully quantified EDS classification methodology. The solution operates on a recipes-based protocol whereby, once an aspect of the analysis is created and the suitable parameters (SEM operating conditions, calibrations, mineral classifications, assay and target mineral phases) are saved for each sample or application they can be recalled, edited, copied and reassigned to other applications/samples. As a result, routine analysis can be conducted without the need of a skilled mineralogist, and settings and mineral databases can be shared directly and simply across multiple Mineralogic Mining systems.

Analysis Modes

Five analysis modes are present, each of which are fully user-configurable; mapping, spot centroid, feature scan, line scan and Back Scatter Electron (BSE) only. These modes, and the settings adjustable within each, allow the user to find the optimal balance between the information required from a sample, and the time taken to gather that information;

- The mapping analysis allows the user to set an EDS pixel size (step size) and magnification for the analysis and the software draws the analysis grid over the sample. This method gives a full analysis of the sample, with the time taken to measure controlled by parameters such as step size, EDS dwell time, number of analysis points and the number of particles (sample surface area) to be measured.

- Spot centroid analysis segments the BSE image to find each individual mineral grains. The geometric centre of each grain is then calculated, from which a single EDS analysis is collected. The composition of this one analysis spot is then given to the grain. This is an extremely fast mode of measurement due to fewer individual EDS points being analysed, but is forced to rely on the minerals of interest having a discrete BSE value and will not report changes of elemental deportment within grains (as only one point on each grain is analysed rather than many).

- Feature scan uses the same BSE function to identify mineral grains. It then rasters the beam across the surface of the grain and sums the x-rays gathered together to give the average composition of the grain. Sitting between the spot centroid and full mapping modes in terms of analysis speed, the mode gives good average composition of mineral grains.

- Line scan, is an analysis mode designed to provide a fast and statistically valid bulk mineralogy analysis. EDS analysis are collected at pre-determined points across a single line through the centre of each particle. Line scan is another extremely fast measurement mode designed to very rapidly characterise samples for modal mineralogy and to provide an indication of relative texture (size, liberation, association) and is useful in instances where non-unique BSE values for minerals limit the use of spot-centroid.

- The Back Scatter Electron (BSE) only mode uses the BSE electron grey scales (0-255) to classify the mineral phases present. BSE-only mode is the fastest mode of analysis as it does not use any EDS analysis. This mode relies on the unique BSE value of each mineral grain to identify the mineral present.

The counts per EDS pixel can be adjusted by altering the dwell time of the beam on that particular pixel. Analysis of the impact of low counts on element detection and mineral classification indicates that typically $c.2,500$ counts or greater is advised for the accurate detection of elemental compositions and classification of minerals (Graham *et al.* 2014b).

These analysis modes can be combined with statistical stop criteria, whereby once the statistical variation in the area percentage of minerals classified falls below a user defined amount, the analysis will stop as it is deemed a statistically valid data set has been acquired. This is made possible as mineral identification occurs online during the measurement, which has the added benefit of reducing manual post-analysis data processing.

Mineral Classifications and Assay

The mineral classifications are done using a combination of the SEM image (typically BSE, although cathodoluminescence, secondary electron or other SEM image input can be used for Mineralogic), and the fully quantitative EDS, where the spectrum is quantified to provide the wt% contributions of the elements. By way of example, the PGE mineral sperrylite ($PtAs_2$) has an ideal stoichiometric composition of 57% Pt and 43% As. The Mineralogic classification would therefore be set to contain minimum and maximum percentages of Pt and As (Fig. 1), and providing the quantified EDS pixel data falls within these amounts, the pixel will be classified as sperrylite. As well as wt% compositions, elemental ratios can also be applied to distinguish phases which have a solid solution. Sobolevskite (Pd(Bi, Te)) and kotulskite (Pd(Te, Bi)) are suitable examples where a Bi/Te >1 and a Te/Bi >1 set of ratios can be used to distinguish sobolevskite and kotulskite respectively.

Each mineral classification has a specific gravity (Fig. 1) entered that is used within a range of calculations including modal mineralogy (for example to calculate wt%), and the measured assay value for the sample. Using sperrylite as an example again, where Pt is the target element, the measured amount of Pt combined with the specific gravity is used to calculate the mass, and in turn then used to produce the Pt assay value of the sample. The assay data outputs are also complimented with measured elemental deportment data, measured average mineral composition, association data, liberation, partial particle perimeter and the standard bulk mineralogy output.
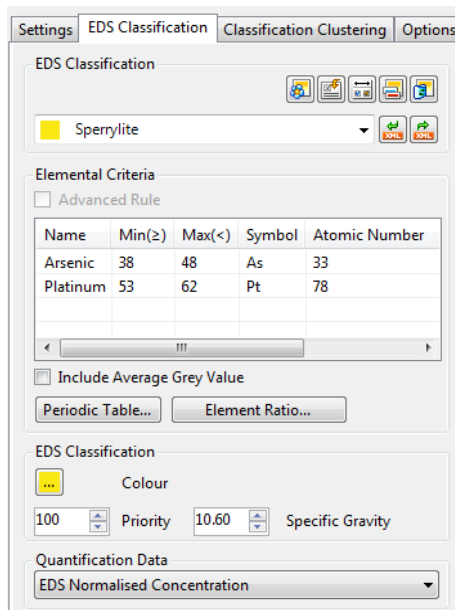
Figure 1. An example of a Mineralogic Mining mineral classification for sperrylite.

Image Processing and BSE Images

The analysed particles and fields are accompanied with a high resolution BSE image which is automatically collected and stored in the final dataset. All the physical measurements requested (area, elongation, ferret maximum diameter etc.) are carried out on the high resolution BSE image, and not on the final digital mineral map. The input image is used to isolate the BSE grey scales by selecting particles for analysis and removing resin or purely focussing on bright phases (Fig. 2). Saving of the high resolution BSE image (or any other detector input i.e. CL) results in improved physical measurement of the particles, improved touching particle separation and storing of an additional visual data set of the sample. Combined with an advanced image processing capability, numerous image processing functions can be applied to further segment, improve and tailor the AM analysis.
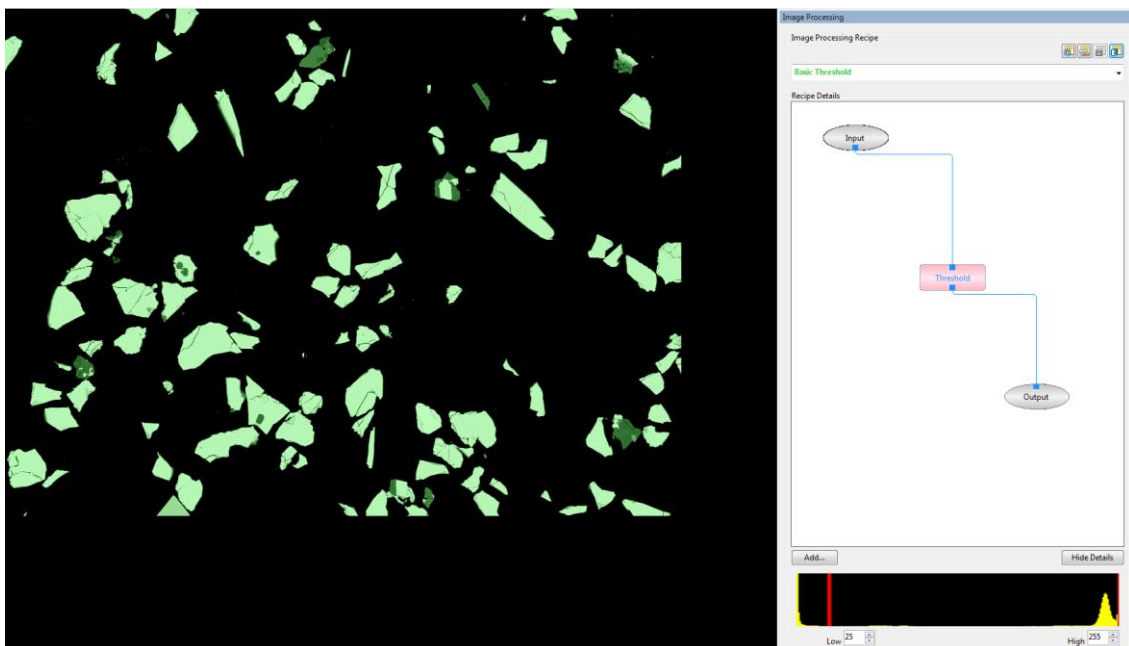

Figure 2. A basic example of the image processing whereby the particles (highlighted in green) are isolated for the analysis.

Mineralogic Mining Analysis Workflow

The following section describes in brief a typical automated analysis workflow using Mineralogic Mining. Each application or sample can be viewed as a modularised analysis where recipes are created for the SEM Set-Up, Image Analysis, EDS Analysis and the Data Output (Fig. 3). The first step is to produce a suitable SEM image (correct brightness, contrast etc.), and identify the sample block for analysis.
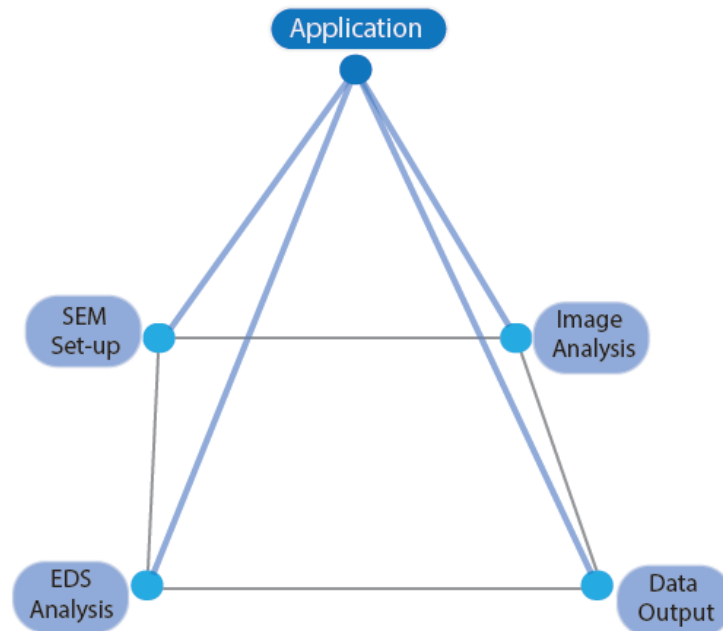

Figure 3. A visual representation of the modularised nature of Mineralogic Mining whereby each application (or sample analysis) is segmented into 4 separate aspects; SEM Set-up, Image Analysis, EDS analysis and Data Output.

Image Analysis: Image processing is undertaken to segment the image by using a BSE threshold to ensure only target particles or grains are measured (e.g. not the mounting resin). The physical measurement parameters required can then be selected and are carried out during the analysis on the high resolution BSE image.

EDS Analysis: The EDS analysis mode (or BSE) can be selected and the correct mineral classifications entered or selected from other mineral classification lists. The EDS dwell time can be adjusted to ensure that sufficient number of counts per EDS pixel are generated for the nature of the analysis.

SEM Set-up: The SEM operating conditions are saved (kV, beam parameters, working distance etc.) so that consistent analytical conditions are maintained per analysis. Numerous macros can be saved to account for the variations in SEM operating conditions required for the various samples presented to the SEM. Calibration locations for the BSE brightness, I Probe (only for W and LaB_6 systems) and the Cu Kα EDS calibration are all selected at suitable locations on the standard, and also the duration between each standard calibration is set.

Data Output: Bulk data (mineralogy by area and weight percent, average grain size and grain size standard deviation and average composition) are provided as standard data outputs. Elements can be selected for assay

measurement and elemental deportment outputs. Target mineral phases can also be selected for the association measurements of the sample. Liberation outputs are flexible where the definition of liberated, middling and locked can be adjusted. The partial particle perimeter reports on the exposed free surface of mineral grains within particles.

Whilst undertaking the analysis, the data is classified using the fully quantified EDS analysis on-line and therefore when the analysis is finished, the results are ready. There is no need to process data offline, if the data is of sufficient quality to use, i.e. correct mineral classifications selected.

With all the results gathered, off-line data reprocessing can be carried out, if required. For example, additional mineral classifications, target mineral classifications (associations) and elements for assay can be added or existing ones can be edited and will produce updated data outputs after offline data reanalysis is carried out. In addition, because the BSE image is saved, additional physical measurements can be carried out and new target minerals and elements for assay added.

Methodology

Analysis of the sample was carried out at the ZEISS Natural Resources Laboratory in Cambridge. The sample used for this investigation was derived from a PGE-rich Cliff chromitite locality from the Unst ophiolite in Shetland. Whilst not large, the Cliff chromitite prospect contains highly anomalous PGE grades, the sample investigated was CF2 which recently reported an estimated total PGE grade of 50 ppm (Brough *et al.* 2015).

The light microscope analysis was carried out in reflected light and using a ZEISS Axio Imager. Z2m equipped with transmitted and reflected light capability (Fig. 4). The LM was equipped with a halogen light source and a scanning stage 130x85 STEP (D) allowing automated image collection. Images were acquired using a high resolution AxioCamMR3, 24 bit RGB colour with the EC Epiplan-Neofluar 10x/0.25 HD DIC M27 objective providing a 100x magnification (1.36 μm pixel size).

Figure 4. Image showing the Axio Imager. Z2M and the Mineralogic SIGMA VP at the ZEISS Natural Resources Laboratory, Cambridge.

Before the LM imaging, 3 fiducial marks where placed on the polished block surface that were used to correlate the SEM stage with the LM image. Following the LM analysis, the samples were carbon coated using a Quorum Q150T ES high resolution and vacuum sputter coater. The samples were then mounted on a ZEISS SEM sample holder and placed in a Mineralogic Mining SIGMA VP (Field Emission; Fig. 1) SEM equipped with 2 x Bruker 6│30 EDS detectors. On completion of the registration, navigation in the SEM could be carried out by selecting areas of interest on the correlated reflected light image.

Using the correlated LM and SEM stage, it was possible to select points of interest on the LM image which results in the movement of the SEM stage to that location. EDS point and ID analysis was carried out to confirm the presence of PGMs.

The AM analysis was carried out at 20 kV with a measured I probe of 10.6 nA. Image acquisition was done using the ZEISS HDBSD detector with the grey levels calibrated to a predetermined grey level every 60 minutes. Standardless calibration of the EDS detectors was also carried out every 60 mins, by calibrating on a Cu standard the Cu Kα peak. The Bright Phase Search (BPS) image processing function was used to rapidly search for and measure only those fields containing minerals with a high z-value, which equates to a high BSE value (namely PGM's). In this case, a BSE value greater than 250 was used. Mapping parameters included as 3 μm EDS pixel size with *c.* 4,000 counts per EDS pixel.

Results

Following the LM analysis, the resulting tiled reflected micrograph is (shown in Fig. 5a) was used as the basis for the SEM stage registration using a three point calibration on the fiducial marks. From the LM analysis, it was noted that in numerous locations high reflectance minerals were identified and these were used to navigate the SEM stage to confirm the presence of PGM using an EDS point and ID analysis.
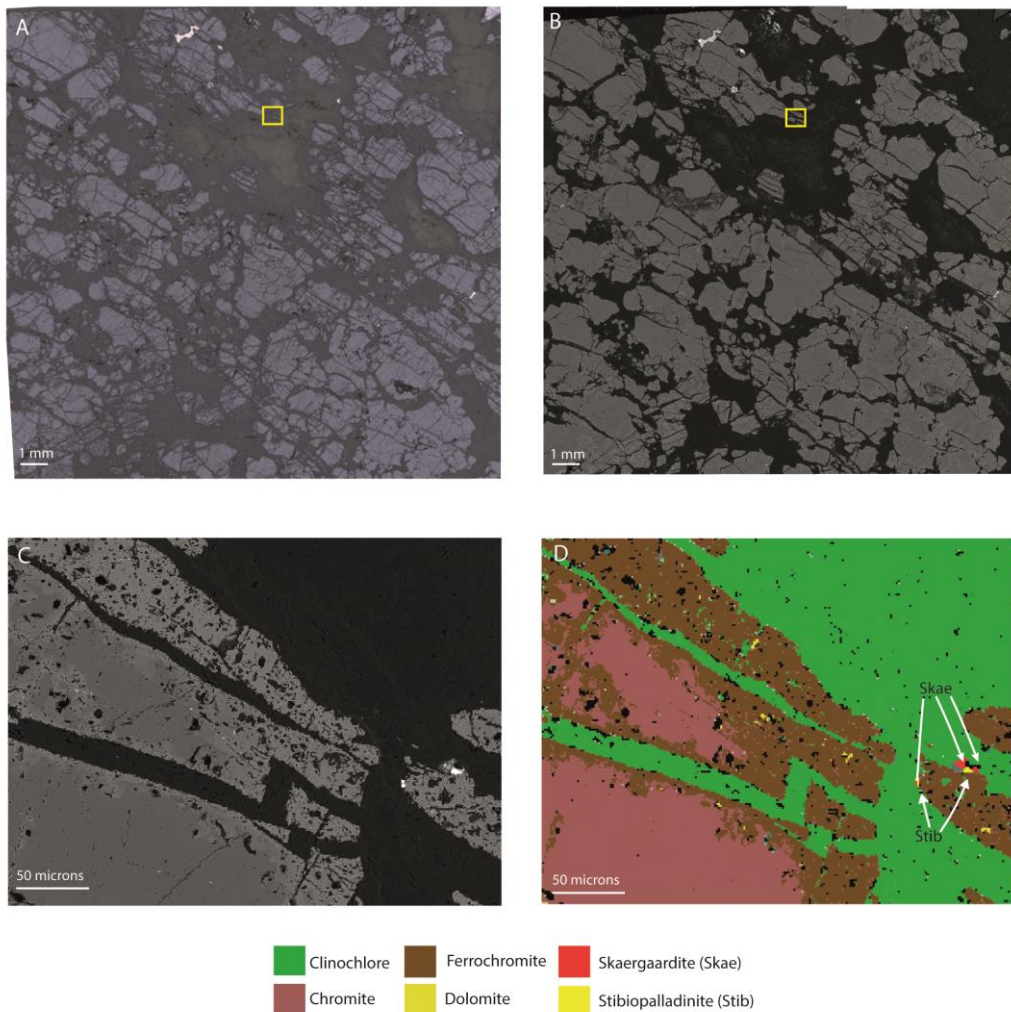
Figure 5. Photomicrographs outlining the images of the sample required and the location of an area mapped due to the presence of a bright phase (PGM); (a) A stitched reflected light image from the initial LM image mapping (b) BSE image from the AM analysis which is also stitched (c) individual tile showing the presence of bright phases (PGM) (c) AM mineralogy map showing the occurrence of skaergaardite and stibiopalladinite. The black features on the Mineralogic image are a result of the holes removed in the image processing.

Following the confirmation of the presence of PGM, the AM analysis was undertaken and a large area tiled BSE image was produced (Fig. 5b). This provided the contextual data for the setting of the PGM, including identifying the type and texture of the surrounding minerals. In each field where a PGM was identified, an AM analysis was carried out with an example of the BSE field in Fig. 5c and the Mineralogic mineral map in Fig 5d.

The identified PGM's were hongshiite (PtCu), irarsite (IrAsS), sperrylite (PtAs$_2$), skaergaardite (PdCu), stibiopalladinite (Pd$_5$Sb$_2$) and laurite (RuS$_2$). Sulphide phases identified were heazelwoodite, chalcopyrite, pentlandite, pyrite and millerite. Non-sulphide mineralogy was dominated by chromite, ferrochromite and clinochlore with low amounts of serpentine (lizardite, antigorite and népouite), nickeline and dolomite.

Discussion

The realisation of the growing complexity of ores and the falling in priority of process mineralogy highlights the need for methodological and technological development (Baum *et al*. 2014; Bradshaw, 2014). High throughput of complex and variable ores provide more strain on process mineralogists and the laboratory equipment required to characterise ores. In addition, high sample volumes are required to successfully

characterise the rare and often illusive PGM. As a result, there is a growing strain on analytical equipment and skilled mineralogists time.

The relatively low-cost light microscope techniques could be used to rapidly search for and locate potential low grade phases of interest (such as PGM's, or copper minerals in tailings) in samples, saving the location for use in a second stage of analysis using an SEM and Mineralogic Mining for full characterisation. This workflow could therefore be used to save time and money in these types of analysis. Efficiently being able to navigate to areas of interest across different analytical techniques can help optimise processes, increase sample throughput and sample to data turnaround time. Recent work by Harman *et al*. (2014) further illustrate the desire to correlate between AM solutions and other analytical techniques.

The introduction of steps towards the correlation of AM techniques with other equipment provides the potential to free-up time on AM systems by effectively pre-screening samples for PGM content. Furthermore, the recipe based protocol of Mineralogic Mining reduces the need for skilled mineralogists to run the AM system; allowing them to spend time interpreting and reporting data, and making informed decision whilst allowing a trained technician to carry out routine analyses using Mineralogic Mining. The work and methodology above shows how it is possible to;

1. Automate the pre-screening of samples using automated LM analysis.

2. Correlate with the tiled LM images and the SEM stage then make it possible to navigate the SEM stage effectively.

3. Reduce the AM/SEM utilization time for these samples

4. Reduce the time a mineralogist needs to spend running routine analysis, allowing their skills to be better utilized

Conclusion

Mineralogic Mining represents a technological advance with the introduction of the next generation of AM solutions. The development and application of the fully quantitative mineral classification methodology, advanced image processing capabilities and analysis flexibility add an extra dimension to AM solutions. The potential to correlate AM techniques with LM analysis opens up new applications for mineralogical characterisation in industry and academia. In addition to LM-SEM-AM correlation, correlation with other analytical techniques, such as Mineralogic to Laser Inductively-Coupled-Plasma Mass Spectrometry (LA-ICP-MS), is also being explored (Harman et al. 2014).

The evolution of such techniques allow for the potential of pre-screening of samples and/or purely focussing on areas of interest identified. Alternatively, AM can be used as the pre-screening analysis and correlation with other analytical techniques used to validate and add extra analytical information. The development of the ability to correlate techniques such as LA-ICP-MS, LM, X-ray microscopy and electron microprobe with ZEISS Mineralogic Mining can further boost the automated analytical capability.

Acknowledgments

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