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A new SEM–EDS based automated mineral analysis solution for PGM-bearing ores and flotation products

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ABSTRACT

Reliable automated PGM identification is a consistent challenge on SEM–EDS based automated mineral analysis systems. The main reason for this is the size of the PGM grains, which are often smaller than 3 μm in diameter, particularly in chromitite ores of the Bushveld Complex. This leads to "mixed" X-ray (EDS) spectra, in which the relative PGM: gangue elemental contribution is variable. This is further complicated by the fact that PGM species often occur in the form of a solid solution series, and ideal chemical formulae of these minerals cannot be relied upon for automated mineral identification. These conditions make it difficult to automatically identify PGM species based on spectral matching or "windowed" elemental EDS X-ray analysis schemes.

Mintek's Mineralogy Division has developed and tested an algorithm that can identify PGM species from raw EDS spectral data, and largely overcomes the problem of mixed spectra. This PGM identification algorithm is being integrated as a plug-in into the Carl Zeiss SmartPI™ particle analysis software by the Carl Zeiss software team in Cambridge, UK, to provide a reliable automated PGM analysis system.

1. Introduction

Mintek has carried out test work for several South African platinum group mineral (PGM) producers over the past decades, primarily in the field of optimising PGM concentration by froth flotation. As a result, the Mineralogy Division has often been requested to provide mineralogical data for various streams of PGM recovery plants, on both pilot plants and full scale flotation plants at several mine sites. This has led to a substantial knowledge base of PGM ores and minerals from all areas of the Bushveld Complex.

Due to the low levels of PGM, particularly in tailing streams, providing statistically valid mineralogical PGM data is a time-consuming exercise, as a sufficient number of PGM grains needs to be located.

Applying a brightness threshold to backscattered electron (BSE) images on a scanning electron microscope (SEM) to locate potential PGM-bearing particles by means of their high BSE signal intensity became a useable technique as SEM technology advanced during the 1970s and 1980s. Computer controlled, motorized sample stages on SEMs allowed many sub-samples to be searched, field by field, in unattended runs. Stage co-ordinates of potential PGM grains were recorded by the SEM control computer, and the chemical composition and mode of occurrence of these grains were confirmed manually by a mineralogist after the run.

Further technological advances led to the development of SEM and energy dispersive spectroscopy (EDS) based systems that could automatically locate potential PGM-bearing particles by BSE thresholding, and then perform X-ray micro-analyses on the mineral grains within candidate particles. These EDS spectra were then used for mineral identification, by comparing the collected spectra with either X-ray counts in fixed elemental energy windows, or a set of previously identified standard EDS spectra.

A mineral map that describes the particle mineralogy in the form of a binary image is produced. An example of a mineral map is shown in Fig. 1. The mineral maps produced from a sample can be interrogated by processing software, which extracts useful information such as relative mineral abundance, particle and grain size and liberation characteristics.

In the case of PGM, the small size of the PGM grains can make mineral identification by the methods described above difficult and unreliable. This is particularly problematic in chromitite ores of the Bushveld Complex, where most of the PGM grains are smaller than 10 μm in diameter (Chetty et al., 2009; Penberthy et al., 2000; McLaren, 1980). During milling of the ore (generally to 80% < 75 μm), the grain size of the PGMs may be even further reduced (Penberthy, 2001). As a result, the samples submitted for mineralogical analysis often contain a significant proportion of PGM grains smaller than 3 μm in diameter. EDS analysis on a small

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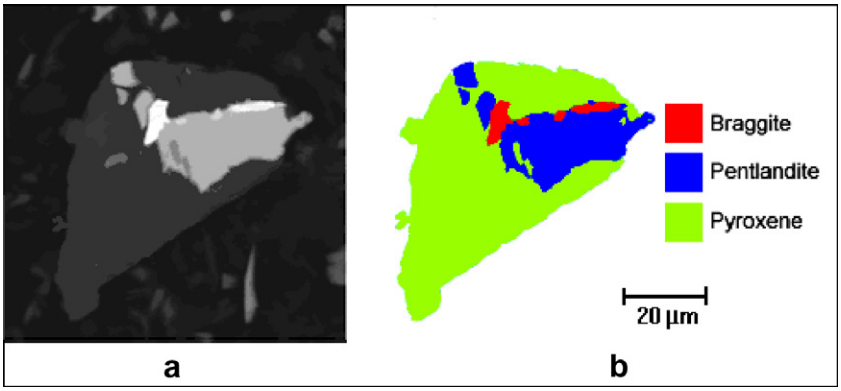

Fig. 1. (a) Backscattered electron image and (b) corresponding binary mineral map.

grain often results in a mixed spectrum, containing elemental X-ray counts from both the PGM grain and the host gangue mineral. The elemental contribution of PGM and gangue is thus variable from analysis to analysis, and a mineralogist's input is often required to interpret these spectra, and to accurately assign them to a suitable PGM species. All possible PGM/gangue spectrum combinations and elemental ratios need to be considered to use spectral matching or windowed elemental X-ray counts as a mineral identification system. To add to the problem, some of the PGM species occur in the form of a solid solution series, so variable within-PGM species compositional variance needs to be considered as well. It is also not uncommon to find even a 3 µm sized grain to be a binary PGM occurrence, a fact which complicates matters considerably.

Mintek has developed an algorithm, trademarked under the name "Identiplat™", which can identify PGM species from raw EDS spectrum data, and to a large extent resolves the problem of mixed spectra.

In order to provide analytical capability, the Mintek PGM identification algorithm is being integrated into Carl Zeiss' existing SEM/EDS based particle analysis system, SmartPI™, in the form of a software "plug-in" (www.zeiss.com). The integrated system will produce a reliable automated mineral analysis system for PGM-bearing ores.

2. The identiplat™ pgm identification system

The Identiplat™ algorithm is capable of identifying PGM species and their associated gangue minerals. To achieve this, the system relies on a combination of:

- Backscattered electron (BSE) intensity thresholding to locate candidate particles.
- Identifying unique EDS spectrum properties for each of the mineral species.
- Arranging these in a logical order so that first match principles can be used as an additional mineral identification parameter, and
- the count rate and improved energy resolution of silicon drift EDS detectors (SDD).

The unique EDS spectrum properties hold true for individual PGM species, regardless of whether the spectrum is mixed with other gangue mineral elemental peaks. These unique properties are isolated and identified in the raw EDS spectrum, and not the processed EDS spectrum that is normally displayed in EDS analysis systems. This allows the algorithm to take advantage of the full resolution of the EDS spectrum, resulting in the ability to distinguish between several of the common problem EDS peak overlaps. Peak overlaps include Pt/P/Zr, Ca/Te/Sb, and Mg/As/Pb, which affect both PGM and gangue resolution.

The ability of Identiplat™ to properly identify PGM was tested on South African Bushveld Complex PGM ores. More than 150 PGM grains, representing a variety of PGM species, were subjected to manual WDS microprobe analyses, performed by an independent international facility. The resulting quantitative WDS data were systematically identified by an acclaimed PhD level PGM mineralogist, and compared against identification by Identiplat™ on a grain-by-grain basis. The result of this comparison was a success rate of over 98% for the automated identification algorithm. The ~2% mis-identifications were caused by rare PGM species that had not been included in the Identiplat™ database at the time of the test: the addition of three PGM species to the mineral identification database resulted in better than 99% correct PGM identification.

3. Single commodity vs. Multi commodity systems – dedicated instrumentation

The Identiplat™ system is designed to cater for single commodities and ore deposits rather than being an "all in one" multi-commodity analytical system. As stated, the system presently caters for PGM ores only. The advantage of this approach is that the mineral identification system, results processing and formatting can be set up to provide only information that is relevant to PGM mining and processing, even going as far as to set up each system to provide a customized outputs as per user requirements. This approach removes the need for the user to set up the instrument – mineral identifications and result outputs will be established for the end-user during instrument commissioning However, the user will be able to access the mineral identification modules to adjust parameters if desired.

There is a worrying global shortage of skilled mineralogists who have the necessary experience and knowledge to operate conventional automated mineral analysis systems. Providing a system that will perform the necessary analyses "as commissioned" without any need for user set-up alleviates this problem to a large degree, as an experienced mineralogist will not be required to run the instrument. This allows, for example, several less-skilled operators to provide results from several instruments to a single experienced mineralogist or metallurgist for interpretation.

Should the instrument encounter a PGM grain that does not match chemical compositions in the mineral ID database, an "unidentified" result will be returned. As each EDS spectrum collected by the system is saved to disk, the unidentified spectrum can be manually evaluated, and a new entry can be easily added to the mineral ID database.