

# The use of QEMSCAN techniques to characterize entrained matte in platinum furnace slags

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**Abstract:** Dried concentrates produced by Anglo Americans Platinum business from their mining operations in South Africa are processed in three smelters. The matte produced during primary melting contains most of the value metals, which are further concentrated by converting and refining. Tapped furnace slag, which is granulated in water contains value metals in entrained matte phases and as trace components in slag silicate. Accurate knowledge of the size and composition of the matte sulphides contained in granulated slag, therefore, is of primary importance for the assessment of slag cleaning possibilities and process adjustment.

The QEMSCAN, an automated scanning electron microscope with multi-feature software which is traditionally employed for the furnace feed or concentrate characterization, can also be used for granulated slag investigations. Possible determinations include compositional, modal and size analyses of the entrained matte, as well as phase liberation if the slag is to be cleaned by flotation.

A method has now been developed to calculate overall matte composition and to relate this to matte size. The new technique was set up by inputting manual scanning electron microscope analysis results into the QEMSCAN SIP (Species Identification Program) files, and tailoring these to overcome the effects of the microscopic matte phase segregation which occurs during slag granulation. The automation of this process allows many matte inclusions to be analysed, thus improving the statistics involved in data interpretation. The new technique can also be adapted for use on slag cleaning furnace and converter slags.

**Keywords:** Slag losses, Platinum slags, QEMSCAN

## 1. Introduction

Over eighty per cent of worldwide platinum group element reserves are found in South Africa [1,2]. These metals are mined from the Bushveld Complex, where they are found in the Merensky reef, the Platreef and the UG-2 chromitite reef in the North West and Limpopo Provinces. The ores are processed at three smelters operated by Anglo American's Platinum business (hereafter referred to as Platinum), and the base metals (nickel, copper and cobalt) are recovered as part of the platinum-producing process. After smelting, the sulphide matte produced is converted and value metals are recovered at the refineries in Rustenburg [3].

At Platinum's Waterval Smelter, near Rustenburg, blended ore concentrate is dried in the flash driers, and then fed into the furnaces. Matte from the six-in-line furnaces is tapped into ladles and granulated in water, after which it is stored prior to being used as converter feed. Furnace slag is tapped continuously and granulated in water. These processes, as well as others at the smelter, have been described in detail in various publications [4,5]. The furnace slag

is dewatered and stored before treatment in the Slag Plant, where a portion of the slag is milled and floated, and the concentrate returned to the furnaces. The nature of matte losses experienced in the Slag Plant has previously been documented [6].

This paper describes the QEMSCAN characterization of entrained matte in furnace slag. Such information can be combined with electron probe micro-analysis (EPMA) of trace metals dissolved in the slag silicate and oxide phases to complete the picture of base metal distribution in the slag. In turn this data can be used to model furnace equilibrium parameters [7].

## **2. Experimental**

The procedures used for sampling, sample preparation and analysis are shown below.

### **2.1 Sampling procedures**

After granulation, the tapped furnace slag is dewatered and deposited onto a moving conveyor belt. Daily samples were taken as a cut across the belt for a period of one month; these were later composited to form one monthly composite sample.

### **2.2 Sample preparation**

The slag samples were dried and split. One split was milled and submitted for chemical analysis, and the other was mounted in resin and polished sections for QEMSCAN analysis were prepared and then carbon coated.

### **2.3 Chemical analysis**

The methods used for chemical analysis were base metal fusion and ICP-MS, except for sulphur, which was analysed by LECO (combustion) and 4E (Pt+Pd+Au+Rh) by NIMAC – nickel sulphide collection and ICP-MS.

### **2.4 QEMSCAN measurement techniques**

The QEMSCAN is an automated scanning electron microscope with multi-feature software which was developed predominantly for the examination of geological samples and ore concentrates [8]. The instrument is used on a routine basis to characterize feed to Platinum's furnaces [9]. A photograph of a QEMSCAN of the type used at Anglo American is shown in Figure 1.



Fig.1 QEMSCAN – an automated SEM

QEMSCAN measurement modes used in the slag study were bulk mineralogical analysis (BMA) and trace mineral search (TMS).

*BMA*

This is a line scan-based measurement where the instrument scans over sample in one direction. The spacing between such lines is adjusted to achieve maximum coverage of the phases in the sample and depends on the size of the phases of interest. The technique supplies a modal analysis in area per cent and this is converted to weight per cent using the relative density of the phases analysed. BMA acquisition is shown diagrammatically in Figure 2.

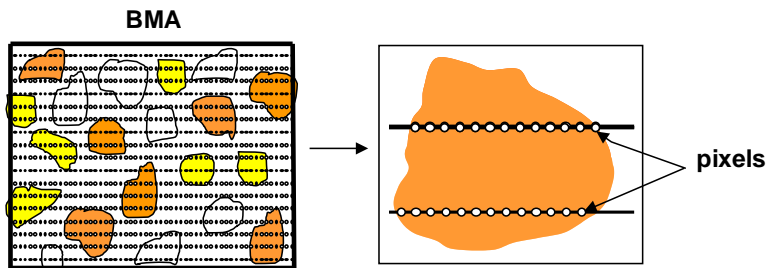


Fig. 2 Diagrammatical representation of BMA measurement on the QEMSCAN [9]

*Trace Mineral Search (TMS)*

Trace mineral searches are normally carried out where a very low concentration of the target mineral is present. The software also allows for preset back-scattered electron (BSE) ranges to be mapped during a TMS run. All minerals with a BSE value lower than the preset one will be seen as background. The instrument will scan over the sample and only minerals with a BSE intensity higher than the predetermined one will be mapped. The technique allows for automated results on entrained matte sizes and compositions.

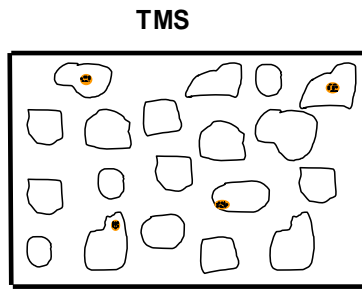


Fig. 3 Trace mineral search on the QEMSCAN

After the samples have been analysed on the QEMSCAN, the data is processed using iDiscover software. Results can be presented in tables or as false colour particle maps (see Section 3).

In order to set up new Species Identification Program (SIP) files, selected entrained matte phases were first examined on a Scanning Electron Microscope (SEM), where BSE images were acquired so that the phases could be relocated on the QEMSCAN. Area and phase analyses were acquired using energy dispersive X-ray spectroscopy (EDS) on the SEM and these compositions were used to tailor the new SIP files. Examples of entrained matte phases used for this purpose are shown in Figure 4.

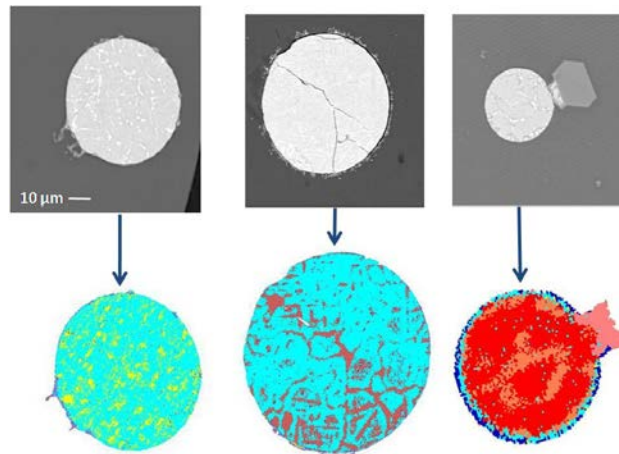


Fig. 4 BSE images (top) acquired on the SEM and false colour images (base) showing the SIP file distribution on the QEMSCAN

### 3. Results

The results of chemical and QEMSCAN analysis are presented below.

#### 3.1 Chemical analysis

The results of whole slag chemical analysis are shown in Table 1. Please note that sulfur values cannot be used directly to estimate entrained matte content because sulfur is also present in the silicate slag glass. The monthly

composite studied here proved to be relatively “clean”; *i.e.* only a small amount of entrained matte is present. This is where automated SEM techniques prove to be useful, as many sections can be scanned without operator input.

Table 1 Results of chemical analysis of the slag composite in weight %.

Mg	Al	Si	Ca	Cr	Fe	Co	Ni	Cu	S
13.24	3.26	23.54	2.72	1.26	10.20	0.012	0.074	0.032	0.13

### 3.2 QEMSCAN results

The results of the modal analysis run on the slag sample are shown in Table 2, and the size distribution of the entrained matte phases are shown in Figure 5. Note that a cut-off size of 3  $\mu\text{m}$  was set on the QEMSCAN.

Table 2 Results of QEMSCAN bulk modal analysis (BMA) on the slag sample.\*Other refers to artifacts.

Phase	Modal analysis (wt %)
Matte	0.02
Furnace Slag	99.26
Spinel	0.04
Other*	0.68
Total	100.00

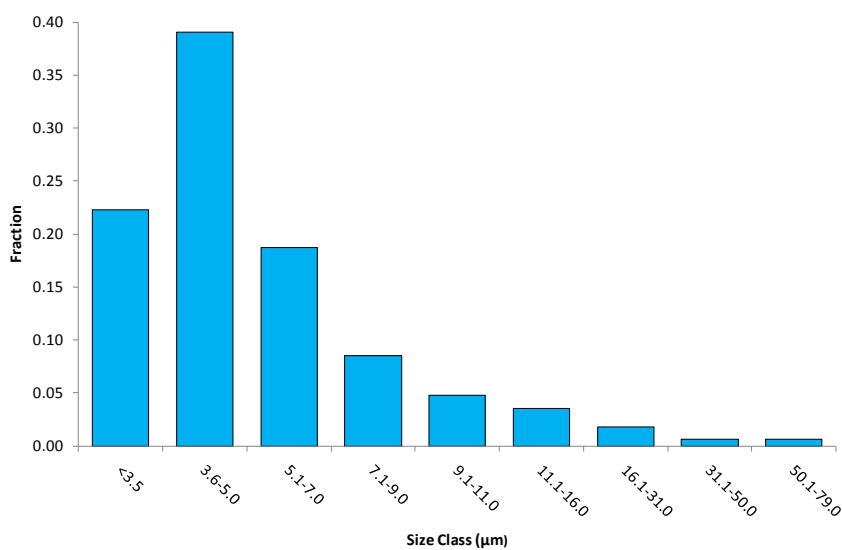


Fig. 5 Matte phase size distribution ( $\sqrt{2}$ ) shown graphically

False colour images of the entrained matte inclusions found in one polished section of the slag sample are shown in Figure 6. The presentation of the matte phases in this way allows rapid visual assessment of size and composition. Out-of-place phases can then be relocated on the QEMSCAN to check whether they are a genuine feature of the sample. This allows elimination of any problems caused by sample contamination or artifacts (caused by surface irregularities or charging).



Fig. 6 False colour representation of entrained matte found in slag in section. The largest matte phase shown is approximately 100  $\mu\text{m}$  in diameter

The base metal and sulfur levels present in the entrained matte inclusions were calculated from the number of pixels and the SIP file composition. Sections of a 2005 furnace slag characterized using semi-manual techniques previously [7] were also run as a check and the results compared favorably. Compositional data acquired by QEMSCAN in the present study, and calculated for whole matte inclusions are plotted against inclusion size in Figure 7.

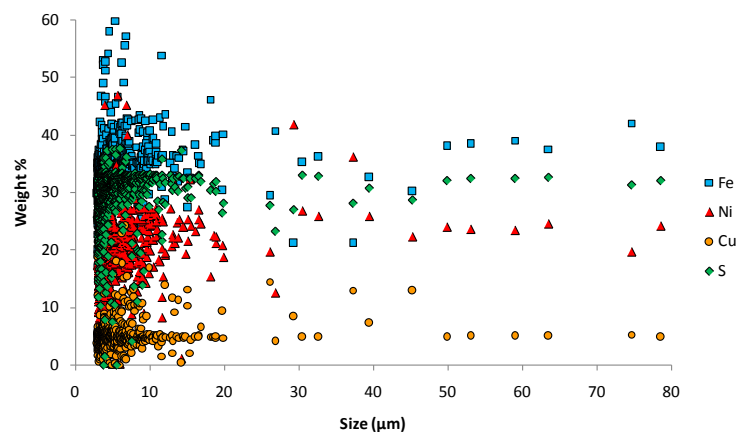


Fig. 7 Variation of nickel, copper, iron and sulfur with entrained matte size

#### 4. Discussion

The most common phases present in bulk (tapped) furnace matte after cooling or casting are synthetic pentlandite ( $(\text{Fe,Ni,Cu})_9\text{S}_8$ ), troilite/pyrrhotite ( $\text{FeS}$ ), bornite ( $\text{Cu}_5\text{FeS}_4$ ) and minor Fe-Ni-Cu alloy which may contain platinum-rich areas. Similar phases are present in the small entrained matte phases in slag, but these are very finely-intergrown and frequently not segregated due to the quenched nature of granulated slag. This necessitated the set up procedure described in Section 2.3 to define new SIP files.

Although small amounts of platinum may be seen in entrained matte on the SEM, and are occasionally detected by QEMSCAN, it is not possible to determine levels of this element in the silicate and oxide phases by EPMA as it is present below detection limit ( $<50$  ppm). The base metals, however, are easily measured by both techniques and so

these are used for the calculation of equilibrium in the slag as has been reported previously [7]. The advantage of modeling granulated slag is that the composition and structure of the molten slag inside the furnace are preserved.

Due to the low levels of matte entrainment in platinum furnace slags, QEMSCAN is an invaluable tool for the analysis of relatively large amounts of slag which are required to produce statistically viable results. Drawbacks which have been experienced with similar techniques in the past, such as increased processing time and inability to resolve same-field matte inclusions, are not experienced using QEMSCAN. This means that all the QEMSCAN techniques described in this paper can be adapted for use on slag cleaning furnace and converter slags, which generally exhibit higher matte entrainment.

## 5. Conclusions

QEMSCAN can be used successfully not only for slag modal analysis and entrained matte sizing, but for the whole-phase matte analysis required for furnace modeling.

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