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AN ALTERNATIVE AUTOMATED ELECTRON BEAM TECHNOLOGY FOR GOLD CHARACTERIZATION

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ABSTRACT

Automated scanning electron microscopy has been widely used in recent years for gold and other precious metals characterization. However, application of this technology can be resource intensive and in most instances needs specialized technical personnel to process the samples and information. Newmont Metallurgical Services (NMS) has recently acquired an ASPEX SEM, with the aim of realizing significant benefits/improvements in sample preparation and sample processing time. NMS and ASPEX have been working together to develop an approach utilizing this technology for the minerals processing industry and specifically for gold characterization. Results to date confirm this as a viable alternative, with potential for mine-site deployment.

INTRODUCTION

In the processing of gold-bearing ores the ability to characterize gold occurrences in various metallurgical products is an important matter. Historically, metallurgical methods such as diagnostic leaching [1] were used in support of gold deportment and association analyses. In recent years, automated mineralogical approaches such as QEMSCAN and MLA have found increased application in determining the size, speciation and association of microscopic gold in a variety of metallurgical applications. Newmont Metallurgical Services (NMS) recently acquired the ASPEX Explorer™ and has been working with ASPEX to develop the instrument for utilization in their laboratory as well as exploring additional capabilities that it may offer in the process operations environment.

The ASPEX Explorer™ is a compact, rugged, cost effective, fully integrated automated scanning electron microscope (SEM) designed for high speed particle analysis in any environment. The ASPEX Explorer™ includes a secondary electron detector (SED) and a backscattered electron (BSE) detector for imaging, integrated with a silicon drift detector (SDD) for energy dispersive elemental analysis (EDS). The system provides both high vacuum and variable pressure modes for characterization of a variety of sample types. The chamber has a stage that accommodates a choice of removable, interchangeable and customizable sample holders. A built in vibration isolation system and standard 110V operation allows for operation at the mine-site as well as in the laboratory without modification.

The ASPEX Explorer™ uses a single hardware control configuration for both the SEM and EDS components resulting in faster analysis times [3]. Unlike frame-based systems, the dynamic scan control in the ASPEX Explorer™ uses "stage fields" that are further subdivided into "mag fields". The "mag fields" are individually defined by deflection of the beam. Movement between fields is done electronically as opposed to frame-based systems that mechanically move the stage. Instead of capturing a high resolution image of the frame, the ASPEX moves the beam across the field in an array of fairly coarse steps. The steps width, or size, is dictated by the minimum particle required for the detection. The smaller the step size the slower the analysis, however by not spending as much time on background matrix the scan is more efficient and ultimately saves time for the operator [3]. particle is detected when the BSE intensity exceeds the predefined threshold. This particle-sizing sequence initiates a "rotating chord" algorithm to measure the particle. At a 2048 pixel resolution, a series of chords are drawn through the center of the particle at equal angular spacings. Particle size and shape measurements are derived from the chords. Figure 1 illustrates the dynamic scan and rotating chord algorithm sequence.[2-3].

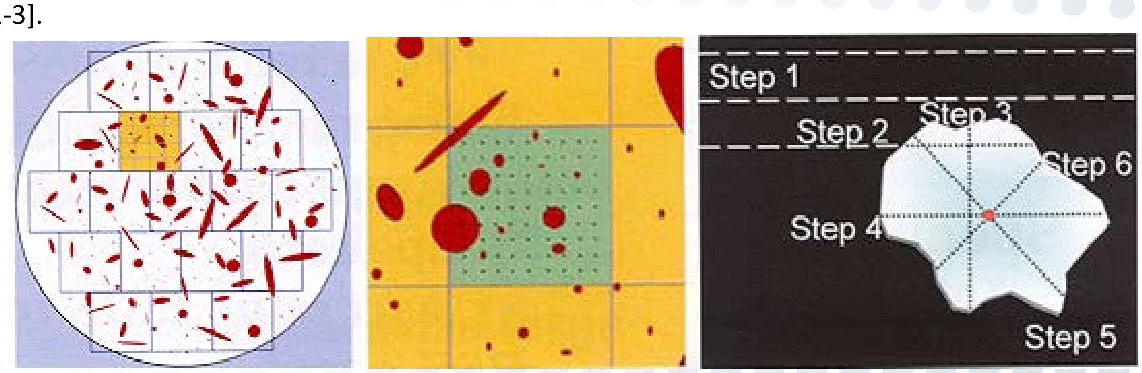

Figure 1A- Automated feature analysis showing mag fields in a stage field (left), sampling point grid (center) and the rotating chords (right)

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After the particle is detected and measured, an energy dispersive X-ray spectrum is acquired in the center of the particle (for this study) or over the area of the feature. Once the particle is characterized (size, shape and elemental composition) user-defined rules placed them into a "class". If needed, the particles can be relocated and further examined by the operator. The Metals Quality Analyzer™ (MQA™), a customizable reporting tool, then automatically generates reports of analyses. A database stores all results of analyses for monitoring long term processing trends.

This paper describes the first application of the ASPEX Explorer™ for gold characterization at NMS and includes a discussion of appropriate and recommended sample preparation methods.

EXPERIMENTAL

Sample Preparation Methods

Several metallurgical products were submitted to the NMS mineralogy laboratory for gold characterization. The samples were mounted on double sticky carbon tape as well as in polished epoxy mounts. Each sample preparation method has certain advantages as well as limitations. Polished epoxy mounts are routinely used at NMS for examinations by MLA because locking occurrences and mineral associations are more defined and easier to discriminate. By contrast, mounting loose particles on double-sided sticky tape (also known as a sprinkle-mount) is a quick and inexpensive method for gold characterization. If the purpose of an examination is to detect the presence of gold and its size distributions (i.e. is gold free and/or recoverable, yes/no) for investigating reasons for lower than anticipated physical gold recoveries achieved by gravity or flotation, then sprinkle-mount results may provide enough information for mineral processing determinations. When more detail is required, characterization of gold in polished epoxy mounts could then be made to better understand the gold association (encapsulated or exposed). Improved understanding of the gold association supports decisions on metallurgical processing routes to incorporate for gold extraction (changes in cyanidation conditions, ultra-fine grinding, and oxidation pre-treatments such as roasting, pressure oxidation or bio-oxidation).[1]

Minimal sample preparation time and equipment is required for sprinkle-mounts. Less than one gram of sample was pressed onto a mount containing a removable, double-sided sticky carbon-tape and then blown with pressurized air to remove non-adhering particles. For this study, the samples were dried prior to mounting, carbon-coated and examined in high vacuum mode in the ASPEX Explorer™. This preparation method allows for quicker turn-around time for measurement and minimal technical expertise.

Samples mounted in epoxy require relatively extensive sample preparation time and a somewhat higher level of technical expertise. Preparation of polished mounts involves epoxy resin/hardener products, polishing units, grinding and polishing cloths, and lubricants, and hence greater cost and lead-time to analysis Turn-around times are on the order of hours to days, compared to minutes for sprinkle mounts. For this investigation, the polished epoxy mounts were also carbon-coated and examined in high vacuum mode in the ASPEX Explorer™.

Automated Feature Analysis (AFA)

ASPEX and NMS personnel created an "Au-AFA" method composed of pre-defined parameters for the detection and characterization of Au particles in both loose particle and polished epoxy mounts. Once a particle is fully characterized, an EDS spectrum and a magnified snapshot of the particle is taken. ASPEX and NMS also created a user-defined rule file ("Au Search Rule") that automatically sorts characterized particles into meaningful classes based on chemical composition. For gold searches at NMS, the final report includes the size and class along with magnified images for all individual grains along with their respective EDS spectra. This automation was instituted to eliminate the need for manual characterization and the experienced staff usually required, and integrate this functionality with the data processing and presentation steps.

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DISCUSSION

Dry Powder and Polished Epoxy Mount Analyses

The “Au-AFA” method used in this study had a step size of 0.33 microns and a minimum size of 0.3 microns. The ASPEX Explorer™ successfully detected gold grains in the sprinkle-mounts. Figure 2 shows three Au grains detected by the “Au-AFA” method in a loose particle mount. However, the smaller Au grains shown in Figure 2 were not detected due to the step size and minimum size parameters chosen for this investigation.

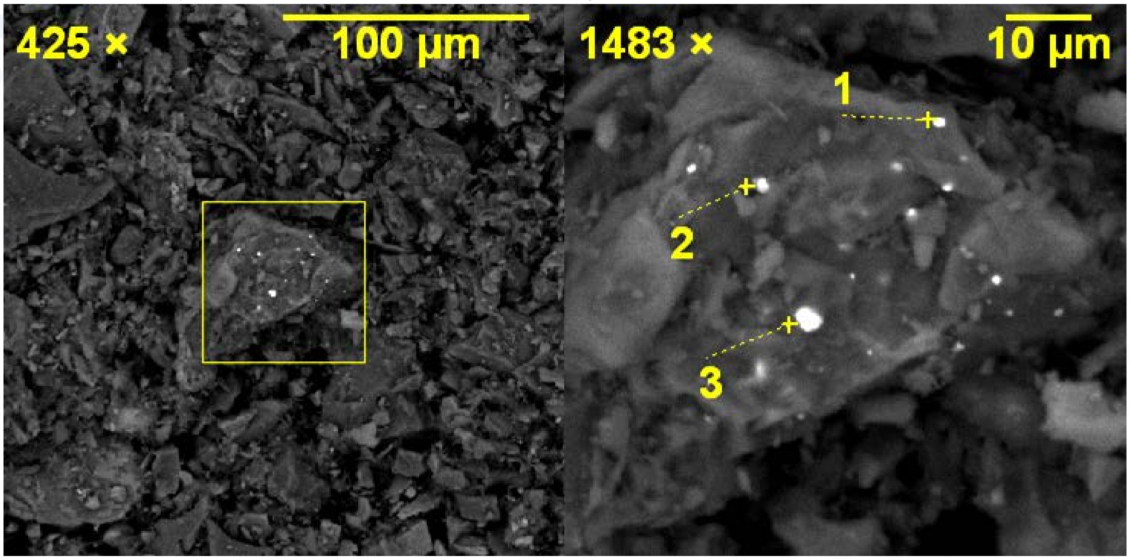

Figure 2 - Backscattered electron images from loose particles mounted on double sticky tape. The image on the right is a magnified view of the boxed-in region from the left-side image. White grains are Au associated with dark grey gangue. The three detected Au grains are numbered in the image on the right. The smaller white grains were not detected by the “Au-AFA” method but were found to be Au by manual examination.

A limitation of the sprinkle-mount method results from the obstruction and shadowing effect caused by the analysis of topographically rough surfaces. Figure 3 shows four Au grains detected by the ASPEX Explorer™, however grain 4 was rejected by the rule file because the EDS included overlying/surrounding gangue. Misclassification of gold grains occurs due to the signals generated from - and absorbed by -the surrounding gangue material resulting in a mixed EDS spectrum containing peaks from Au and gangue. Masking effects – and hence misclassifications - can result from BSE signals being reduced (Figure 3C) and/or gold peaks in EDS spectra being ‘swamped’ and masked by the gangue peaks. The benefit of the AFA is that the rule-sets can be improved, parameters changed, and no additional time is wasted as would be expended in a manual search with an experienced operator.

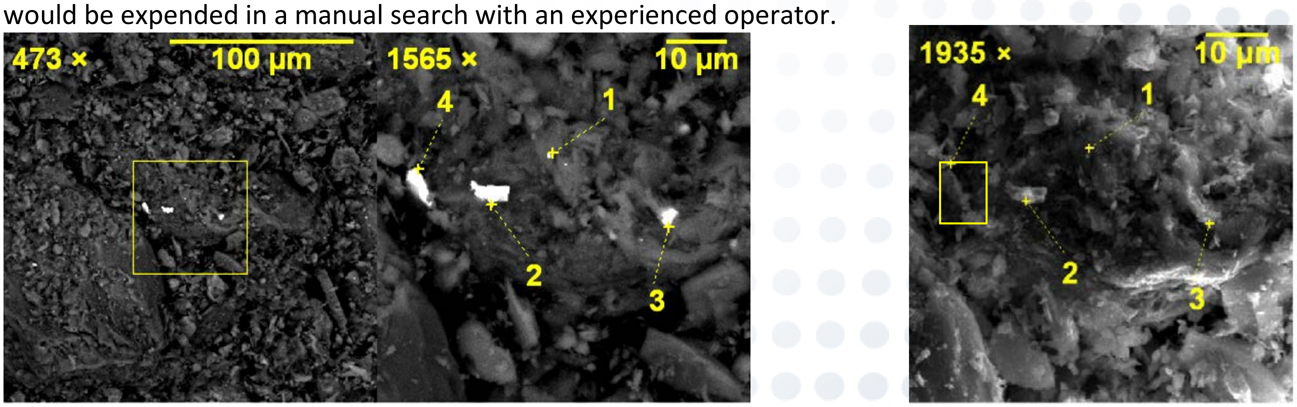

Figure 3 - Two backscattered electron images (left and center images) and a secondary electron image (right image) from a loose particle mount. Center and right-side images are a magnified view of the boxed-in region from the left-side image. White grains are Au associated with dark grey finely intermixed gangue. The secondary electron image on the far right (Figure 3C) shows how Au grain # 4 (boxed in) is masked by the surrounding topographically higher gangue material. The grain was rejected based on the resulting misclassification from ‘swamped’ EDS spectra.

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Figures 2 and 3 also show how locking occurrences are more difficult to determine in sprinkle mounts.   A degree of uncertainty arises from the inability to determine if the Au grain is locked with the gangue (i.e merely exposed) or resting on top. Experienced technical personnel are needed to assess the uncertainties in liberation and mineral association or a request for polished epoxy mounts could then be made for more detailed analyses.

Improved resolution provided by analyses of polished sectioned particle surfaces is essential in evaluating locking and association characteristics. A flat scanning surface eliminates the artifacts seen in the topographically rough surfaces in sprinkle mounts. Figure 3 shows how locking occurrences and mineral associations are more defined and easier to determine, which is a critical benefit when this type of data is required. The images illustrate encapsulation (center image), gold exposure (right image), and potentially leachable gold found in a fracture partially filled-in with iron oxide (left image). Documenting these characteristics aids in determining opportunities to improve gold recovery.

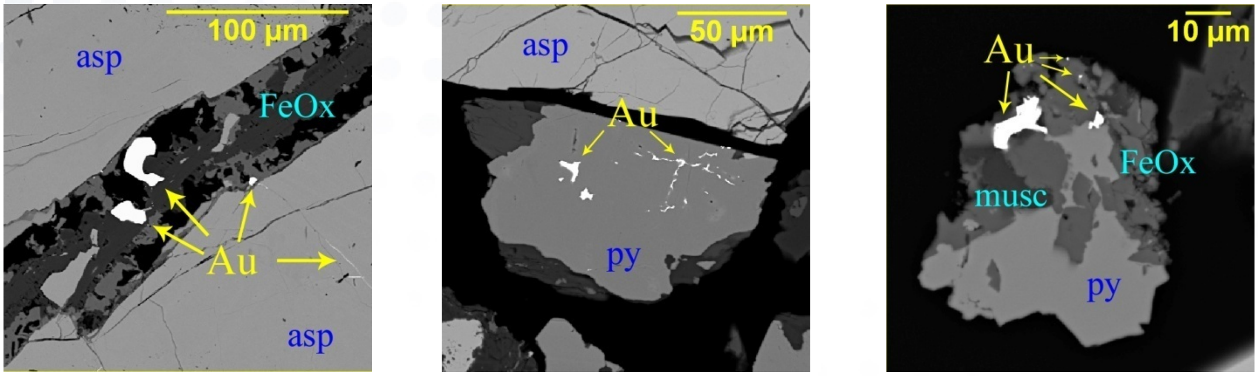

Figure 4 - Backscattered electron images from a polished epoxy mount measured by the “Au-AFA method”. White grains are Au associated with iron oxide (FeOx), arsenopyrite (asp), pyrite (py), and muscovite (musc).

The information obtained using the ASPEX Explorer™ for gold characterization was validated using conventional automated mineralogy. Good comparisons were achieved in terms of the number of gold occurrences confirmed, corroborating that the use of this method/instrument for these applications is appropriate and will produce reliable gold characterization data.

Automatic Reporting – Analysis Summary Report

Once sample analyses are complete, the results are available in a spreadsheet containing spatial coordinates, sizes, shape, compositions, and classifications for all detected particles. The Au Analysis Summary Report customized for gold characterizations at NMS by the ASPEX Explorer™ is shown in Figure 5. Project and sample background information shown at the top of the report are automatically extrapolated from information entered in the “Au-AFA” measurement. Details about the analysis are also included, such as start time, duration, and completion. A table is created below these entries that show total particles detected from each class. The bottom portion of the report includes the magnified snapshots and EDS spectrum for each particle detected together with the parameters selected by the user. The automation of this data for each particle saved a significant amount of time in data gathering, processing and reporting. By conventional SEM, the operator manually captures the images and EDS spectra, and compiles the data for every particle of interest. The automated Au Analysis Summary Report allowed the reader to view each particle in conjunction with relevant data, without the expense of the operator’s time.

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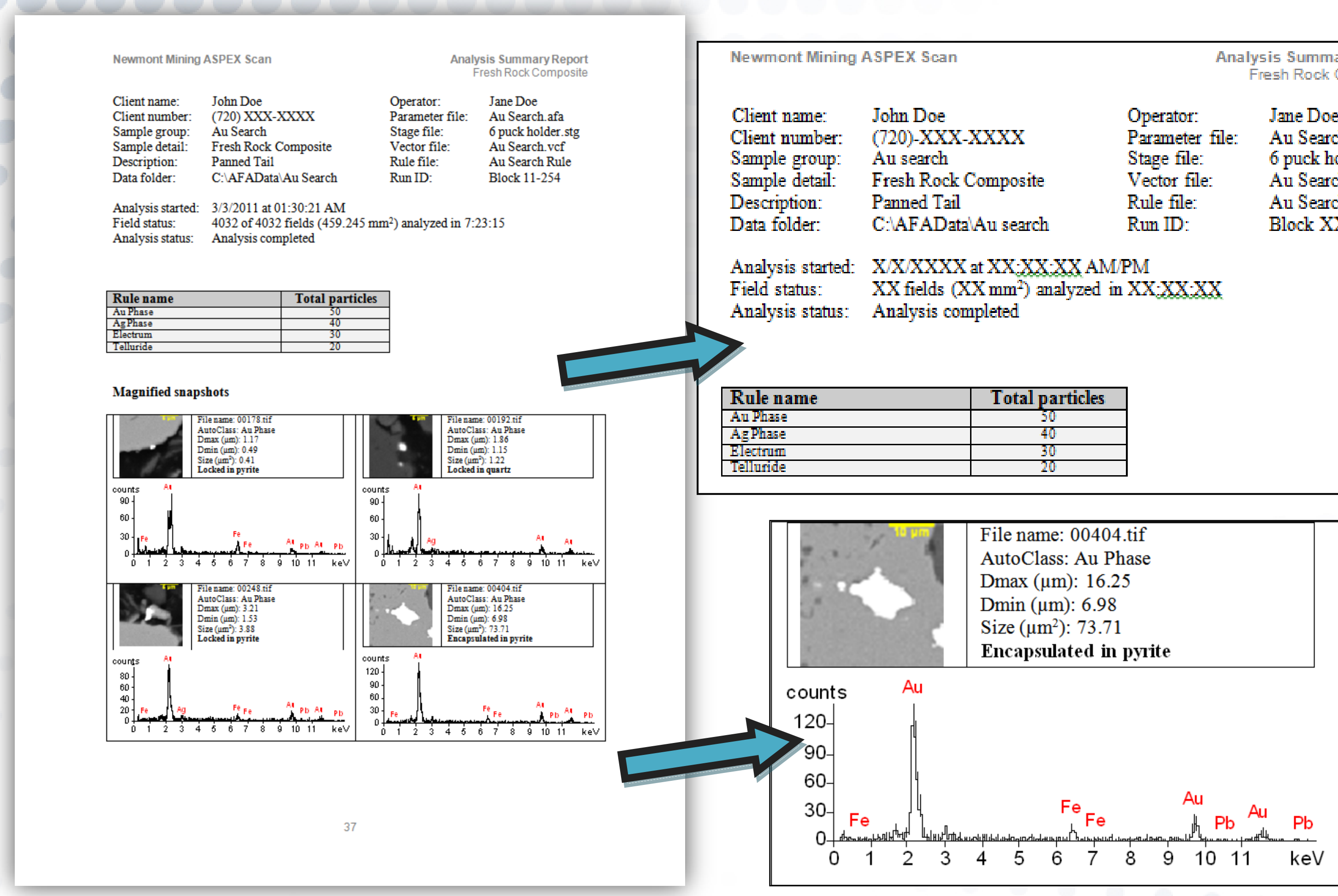

Newmont Mining ASPEX Scan

Analysis Summary Report
Fresh Rock Composite

Client name:	John Doe	Operator:	Jane Doe
Client number:	(720) XXX-XXXX	Parameter file:	Au Search.afa
Sample group:	Au Search	Stage file:	6 puck holder.stg
Sample detail:	Fresh Rock Composite	Vector file:	Au Search.vcf
Description:	Panned Tail	Rule file:	Au Search Rule
Data folder:	C:\AFAData\Au Search	Run ID:	Block 11-254

Analysis started: 3/3/2011 at 01:30:21 AM
Field status: 4032 of 4032 fields (459.245 mm^2) analyzed in 7:23:15
Analysis status: Analysis completed

Rule name	Total particles
Au Phase	50
Ag Phase	40
Electrum	30
Telluride	20

Magnified snapshots

Figure 5 – Example of the automatic AU Analysis Summery Report. Arrows point to blown-up images from the report. Top-right image displays overall background information of the sample analysis. Bottom right image displays a magnified snapshot of the detected gold accomplished by its corresponding EDS spectrum. In this area there is a space for the user to add comments about the particle (shown in bold).

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CONCLUSIONS

The ASPEX Explorer's™ unique electron beam technology and "rotating chord" algorithm proved to be accurate and fast and was successfully utilized in a gold characterization application. Time consuming image processing common in conventional frame-based automated SEM was eliminated through the use of pre-defined feature analysis templates. The "Au-AFA" measurement routine and the "Au Analysis Summary report" templates designed by NMS and ASPEX personnel requires no specific microscopy training to operate so mine-site technicians can quickly generate data for process control. With its compact, rugged and cost-effective design, it is conceivable that this instrument can be located and operated at a mine-site.

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