

AUTOMATED SCANNING ELECTRON MICROSCOPY BY IMAGE ANALYSIS SYSTEM

Henrique Kahn, henrkahn@usp.br. Mining Eng. Dept., Polytechnic School, São Paulo Univ.
Lilia M. Sant'Agostino, Economic Geology Dept., Geoscience Inst., São Paulo Univ.
Eliana Satiko Mano, Mining Eng. Dept., Polytechnic School, São Paulo Univ.
Maria Manuela Tassinari, Mining Eng. Dept., Polytechnic School, São Paulo Univ.

INTRODUCTION

Scanning electron microscopy is nowadays a widespread technique on materials characterization. Some routine works, as analysis of fire gun residues, wear debris monitoring, steel, phase quantification, mineral liberation and others can be performed by a specific analysis tools supplied by some manufactures. Many of these analysis tools are very close to a black box system, presenting low flexibility and designed to perform very specific tasks.

Image analysis systems have drastically extended the microscopy data information from qualitative to a quantitative basis. Digital images SEM became a standard for almost all new SEM's, off-line image analysis is an ordinarily tool applied by a large number of programs. By other hand, on line image analysis or SEM automation is still a narrowspread technique, despite their enormous potential on materials characterization and the limitations of automated optical microscopy.

Only a few image analysis systems available on the market, current the state of the art, can properly automated a SEM. Some basic requirements for these systems are summarized bellow:

- Allow to process a large number of images as well as number of features (up to 10,000);
- Allow a large number of specific feature measurements (up to 10,000 or more);
- Interface to SEM, as well as optical microscopes and video inputs (desirable);
- Stage control (X, Y and Z at least);
- Electron bean control;
- Input of auxiliary data to phase discrimination, such as EDS or WDS data;
- Allow free programming;
- Allow DDE interaction with spreadsheet software (Excell, Qpro, etc.).

The SEM automation - image, electron bean and stage control - is performed by dedicated hardware or external DDE control.

METHODOLOGY

Desirable situation is to perform the SEM automated work for a large number of samples with a minimum image processing requirements and with a lower possible time.

Special care must be taken regarding sample preparation and beam control. Samples usually must be very well polished, or at least flat for some specific analysis, as an example of tracing for one specific phase. If volumetric proportions are required the phase distribution in the sample must be truly random, no mater if the measurements are made by point, line or area.

Since BSE signals are used for phase discrimination, it is essential that the beam must be exceptionally stable. Particular filament adjustment in the Wehnelt cup as well as filament heating procedure and beam control is required for an automated routine work.

Materials characterization often deals with particulate material, which would require particular routine to separate touching particles. This separation is easily done when there is a narrow neck between the particles. First an eroding operation is performed until the particles are separated, then a restoring routine is required to bring the individual particles to the initial size keeping them separated.

The most complex task is to detecting, identifying and segmenting the phases presented in a sample. The most simple and fast case is when phases can be discriminated by their average atomic number using a single grey level BSE image. In more complex situations phase discrimination may require processing of two grey images, each one acquired with different brightness and contrast values, complemented by binary logical operations. Other procedure consist of processing a grey scale image plus external inputs, such as X-ray EDS or WDS counts from selected elements windows. Phase discrimination by X-ray is timing consuming, and ideally it must be avoided whenever is possible. In extreme complex situations, as touching phases with the same BSE contrast, multi elemental X-ray mapping images or point by point X-ray counts from selected areas (QEM*SEM concept) would also be used.

At the end of detection and segmentation procedures each phase of interest will be represented by a binary plane. The required information can be obtained by field or feature measurements and the results are directly presented, stored in a file, or transferred by DDE control to another system for on line processing.

APPLICATION ROUTINES ON PROCESS MINERALOGY

In this work some routines applied for characterization of a massive cryolite ore and cassiterite tailings are presented. The SEM used is a Stereoscan 440, Leo, coupled to an Isis EDS System, Oxford, both remotely controlled by a Quantimet 600, Leica, image analysis system. Image size is 1024 by 768 pixels, operational systems could be Win3.11 or Win95 and communications between the tree systems are made under 100MB/s local network. Samples studied are represented by thin/polished sections of closely screened fractions embebed in an Araldite resin.

Particle density per field is one factor that direct affects the processing time. If the particle density is very high there will be a large number of touching particles and will be very difficult or even impossible to individualize them. Although if it is too small probably there will be no touching particles, but a great number of fields will be required, increasing the measurement time. An ideal compromise is required to optimize the measurement. A routine for separation of touching particles is presented on figure 1.

An example of phase detection and segmentation is presented on figure 2 considering two grey images acquired with different brightness and contrast values.

Liberation analysis is an essential information for mineral processing; it gives the relative amount of free valuable particles available to be concentrated. Two basic concepts are applied for liberation analysis. The liberated-locked technique gives a single number of the ratio between liberated particles and total amount of the valuable mineral calculated by cords (linear intercepts) or area. Area-Volume technique (Figure 3) and its variations determines the area fraction of each particle that contents the valuable mineral; particles are classified by fraction area and results are expressed as cumulative curves (liberation or potential recovery curves) or in incremental steps of 5 or 10%. The area-volume procedure is not only the more suitable for the establishment of flexible and adequate criteria of free particle, but also gives important information regarding the distribution of locked particles. Correction the liberation data from 2D to 3D could also be applied, but it will never give a truly stereological correction.

FIGURE 1 - SEPARATION OF TOUCHING PARTICLES

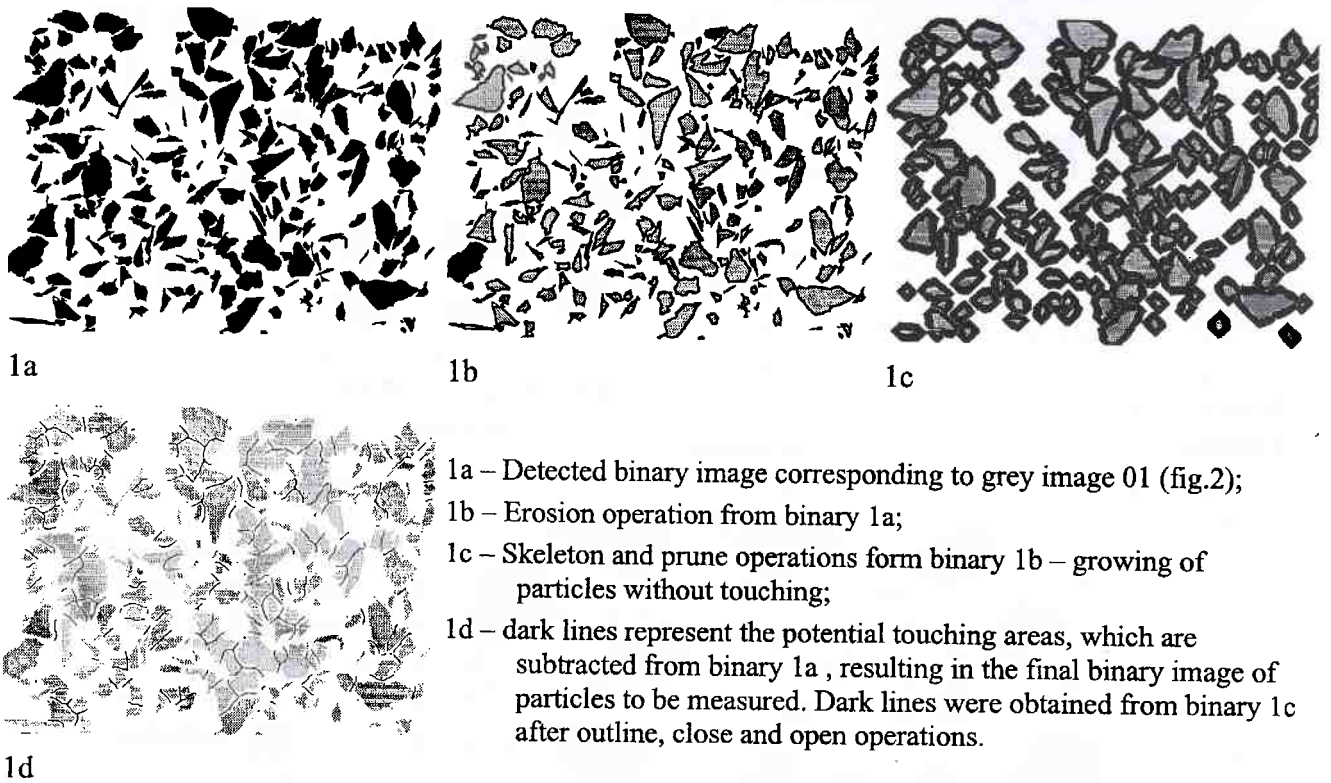


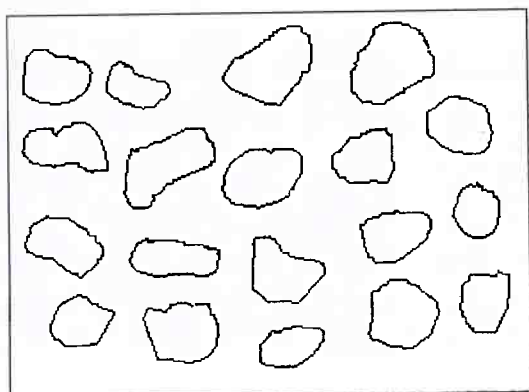
FIGURE 2 – PHASE DETECTING, IDENTIFYING AND SEGMENTING

Two grey images acquired with different brightness and contrast values

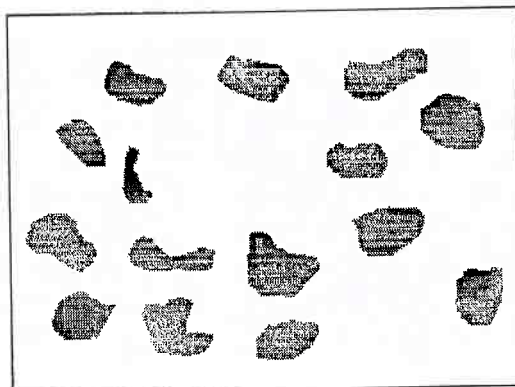


Segmented image is represented by five distinct binary planes. From darker to lighter: 1- cryolite, 2 - thomsenolite, 3 - feldspar and mica, 4 – fluorspar, 5 - heavy minerals

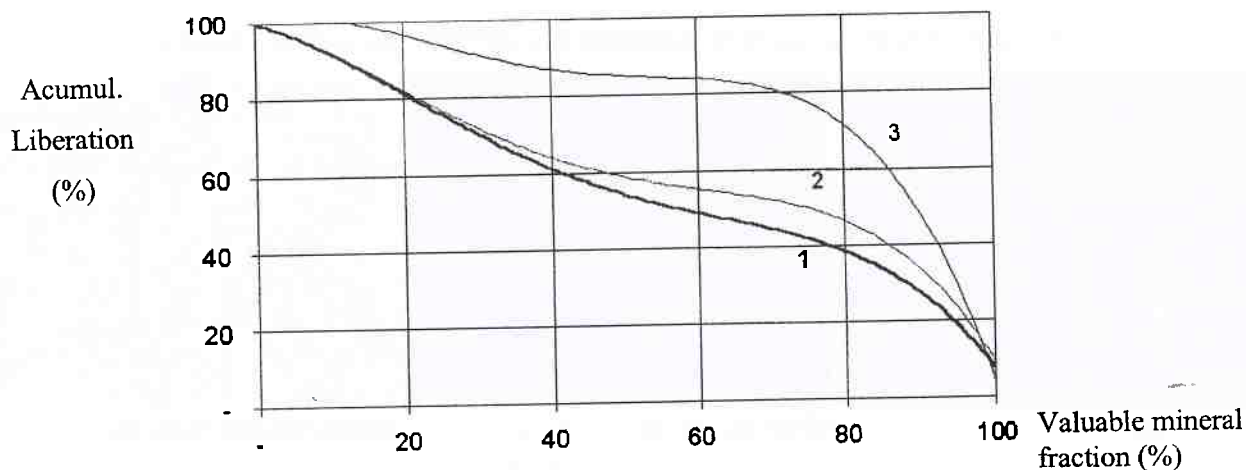
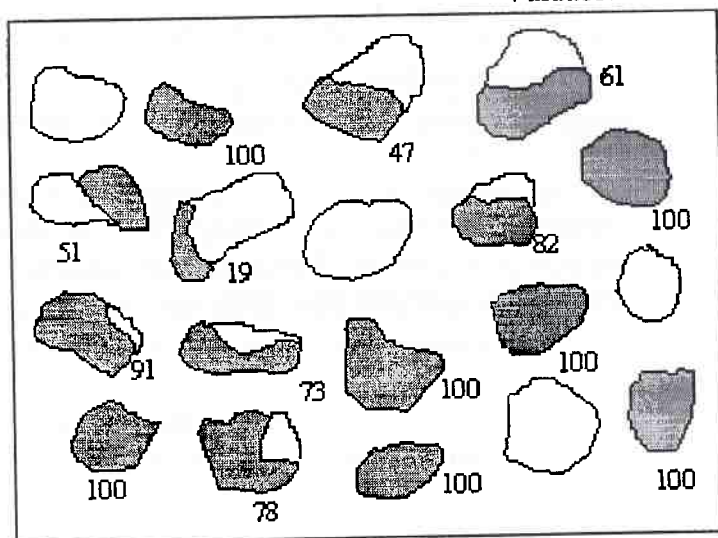
FIGURE 3 - LIBERATION ANALYSIS – Area-Volume Technique



Binary image 01 (bin01) -
Particles



Binary Image 02 (bin02) -
Valuable mineral



Liberation curves for fractions of cassiterite tailings – heavy products from mineral separations

1) – 0,42 + 0,30 mm

2) – 0,30 + 0,21 mm

3) – 0,15 + 0,074 mm