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Stereological correction of perimeter based estimates of exposed grain surface area

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ABSTRACT

Data necessary for liberation and/or exposure analysis is frequently obtained from two-dimensional (2D) polished section images of mineral particles of specified size. This data overestimates the extent of liberation and/or exposure, and in the past an empirical correction (locking) factor has been used to give a better estimate of the extent of dispersed phase liberation and/or exposure. With such 2D data, exposed grain surface area is estimated from perimeter analysis of sectioned particles. Although many researchers have reported 2D mineral liberation analysis in terms of area grade and/or perimeter based grain exposure, comparison of 2D perimeter based liberation with three-dimensional (3D) mineral liberation analysis has not been reported.

In this current research, perimeter based estimates of exposed grain surface areas from 2D polished section analysis of feed and products from HydroFloat (HF) flotation of a copper porphyry ore are evaluated from corresponding 3D high resolution X-ray microtomography (HRXMT) data. Results show that for mean dispersed phase grades (sulfide mineral content) greater than 10%, perimeter based polished section analysis provides a good approximation of the exposed grain surface area determined by HRXMT. For mean grades less than 10%, stereological correction of the 2D data is required for satisfactory estimation of the exposed grain surface area. The most significant stereological correction with respect to exposed grain surface area is for the 0% grade class ("liberated" gangue minerals). For example, in the case of the 417 × 212 µm size class, 2D perimeter analysis indicates that about 96% of the particles have 0% grain area exposure whereas 3D analysis shows that in reality about 77% of the particles have 0% grain area exposure.

1. Introduction

Optimization of mineral processing operations requires knowledge of the spatial distribution of valuable minerals in the multiphase ore particles. Accurate mineral liberation analysis enables us to avoid overgrinding, which reduces the cost and energy required in the comminution process. In addition, accurate mineral liberation analysis also gives assessment of mineral processing products and helps to suggest action to improve the separation efficiency.

Traditionally, in order to characterize and analyze the spatial characteristics of multiphase particulate systems, such as in mineral liberation analysis, two-dimensional (2D) polished section images are obtained and analyzed to acquire useful information. SEM-based automated image analysis systems, such as quantitative evaluation of minerals by scanning electron microscopy, QEMSCAN (Gottlieb et al., 2000), MLA (Fandrich et al., 2007) and TESCAN (TESCAN, 2012), have been used for 2D mineral liberation analysis, although the polished section analysis procedures are time consuming compared to the procedure for tomographic analysis. SEM techniques provide information regarding chemical analysis used to identify mineral types, however, the liberation analysis of polished sections generally overestimates the extent of liberation because sectioned locked particles can appear to be fully liberated in the polished section. In order to estimate the volumetric liberation from polished section analysis, stereological models have to be applied. However, the stereological models are usually not satisfactory because model parameters depend on ore texture, which is unique for each ore type. Finally, extension of the stereological correction for more than 2 phases is limited (Gaudin, 1939; Petruk, 1978; Miller and Lin, 1988; King and Schneider, 1998; Gay and Morrison, 2006; Lätti and Adair, 2001; Spencer and Sutherland, 2000).

In the analysis of flotation separations, the exposed grain surface area distribution is the most appropriate characteristic to describe flotation response rather than the liberation spectrum (Wang et al., 2017).

Although many researchers have reported 2D mineral liberation analysis in terms of area grade and/or perimeter based grain exposure, comparison of 2D perimeter based liberation with 3D mineral liberation

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analysis has not been reported in the literature. In this research, using recently developed software and X-ray microtomography (micro-CT) scans for 3D grain liberation/exposure, 2D perimeter based grain liberation/exposure was analyzed and directly compared with 3D grain liberation/exposure.

Micro-CT has emerged as a nondestructive technique for 3D characterization of multiphase particulate systems in packed particle beds. With the advantages of three-dimensional visualization of the internal structures of opaque materials, simple and fast sample preparation, and short scanning and analysis time, micro-CT has been used to analyze mineral liberation (Miller and Lin, 1986; Miller et al., 2009; Lin and Miller, 2002; Miller et al., 1990) and exposed grain surface area of multiphase particles in three dimensions. The algorithm for exposed grain surface area analysis has been published and used successfully to evaluate the flotation separation efficiency (Wang et al., 2017; Wang, 2016).

In this paper, high resolution X-ray microtomography (HRXMT) scans were used for acquisition of 3D image data for multiphase particles. From the 3D image data, volume grade and exposed grain surface area were determined, whereas the 2D image data were used to determine area grade and exposed grain perimeter. Two dimensional sectional slices from 3D tomographic data were used to determine two dimensional mineral liberation, in addition to data from the MLA system. The comparative analysis results indicate that 2D mineral liberation generally overestimates the extent of mineral liberation compared to 3D mineral liberation analysis. It is noted that exposed grain perimeter is much more appropriate to evaluate 2D mineral liberation compared to area grade analysis (Lastra, 2002).

2. High-resolution X-ray microtomography (HRXMT)

Now micro-CT is used frequently as a non-destructive 3D imaging and analysis technique for the investigation of internal structures. Typically in micro-CT, the sample for investigation rotates, whilst the X-ray source and detector remain stationary. The principle used to identify different materials depends on the photon's capability to penetrate materials in varying degrees, which is mathematically formulated by Beer's law (McCullough, 1975). In this equation it assumes the X-ray source passing through the sample is monochromatic.

$$I = I_0 e^{-\int \mu(x)dx} \quad (1)$$

where I is the transmitted intensity, I_0 is the incident intensity of the beam and $\mu(x)$ is the linear attenuation coefficient for the material along the ray path x.

Micro-CT has been widely used recently in geoscience applications, such as 3D pore characterization (Brunke et al., 2010; Bhuiyan et al., 2013), 3D grain analysis (Evans et al., 2015), fracture analysis, multiscale imaging, ore analysis (Chetty et al., 2011), structure dynamic processes, fluid flow analysis and morphological characterization of fossils (Cnudde and Boone, 2013). Specifically, in the case of particle characterization, the applications have been significantly extended in recent years for particle surface characteristics (Masad et al., 2005), specific mineral quantification in stone (Dewanckele et al., 2009), classification of geological materials (Cnudde et al., 2012) and metallurgical significance for gold and related ore minerals (Kyle et al., 2008; Ghorbani et al., 2011). Mineral liberation analysis using HRXMT has been reported by Miller et al. in previous years using 3D volume grade measurements (Miller and Lin, 1986; Miller et al., 2009).

In this study, the HRXMT system (Zeiss XCT-400) with voxel resolution as low as one micron, was used for 3D exposed grain surface area analysis of packed particle beds. Polished section 2D data was acquired with an MLA system (Fandrich et al., 2007).

3. Sample preparation

The copper ore particles considered in this study contain valuable minerals such as pyrite, chalcopyrite and molybdenite, with gangue minerals such as quartz, potash feldspar, biotite and talc. The main gangue minerals are quartz and feldspar. The primary flotation separation focuses on the recovery of all sulfur-bearing minerals, although the different sulfide minerals can be distinguished by HRXMT. The feed particles were separated by flotation into concentrate and tailings products using the HydroFloat (HF) separator, which shows better performance compared to conventional bench scale flotation machines for coarse particle flotation (Kohmuench et al., 2007; Kohmuench et al., 2010; Kohmuench et al., 2013; Miller et al., 2016). Individual size classes of feed (XFU) and product, namely concentrate (HFO) and tailing (HFU), were analyzed by both MLA and high-resolution X-ray microtomography (HRXMT). In order to compare the 2D MLA perimeter based surface exposure results with the 3D HRXMT exposed grain surface area, 2D MLA perimeter based surface exposure results were calculated for the 417 × 212 µm size class, and in the case of tailing samples, also for the 212 × 150 µm size class. Particle measurements varied between 5000 and 13,000 particles per sample according to size fraction and sample type, with a pixel resolution of 7.3 µm. For HRXMT samples, the particle count was between 2000 and 5000 particles per sample, with a voxel resolution of 5.19 µm. The amount of sample analyzed was greater than that required for statistical significance (Wang et al., 2017).

Generally, after acquisition of the original 3D HRXMT data, the data were preprocessed and corrected for the partial voxel effect, as discussed in the following section. From the 3D partial voxel corrected image, the valuable minerals were clearly identified. There are a total of 994 sectional slices in the 3D partial voxel corrected data. The 2D perimeter based surface exposure distribution was analyzed in each case based on all 994 sections for each sample. In this case, about 500 particles per section were analyzed. The 3D exposed grain surface area analysis was obtained from the 3D HRXMT data.

4. Grain exposure image analysis procedure

Although micro-CT has been widely used in medical science, engineering, and earth science, there are some advantages and limitations for micro-CT. The best advantage of micro-CT is the nature of the nondestructive technique. It allows investigation of the interior structure of samples without mechanical stress, calculation of volume fractions of multiphase systems, geometrical properties (size, shape, sphericity, roundness…), surface texture, etc. However, quantitative analysis using micro-CT is usually not straightforward due to the nature of the X-ray source; there are always some artifacts for micro-CT data such as noise, partial voxel effects and beam hardening effects. Considering these issues, detailed image analysis procedures are needed to eliminate the artifacts for accurate quantitative analysis. Therefore, micro-CT analysis is operator dependent on the 3D quantitative image analysis from reconstructed data, the partial voxel effect, and possible imaging artifacts.

The original HRXMT image data can be reconstructed in both 2D and 3D. The beam hardening effect (Boas and Fleischmann, 2012) can be eliminated during reconstruction using the commercial software package provided by Zeiss for the MicroCT-400 machine. After reconstruction, specialized rendering software such as Fiji (Schindelin et al., 2012) or Vaa3d (Peng et al., 2010) allows for visual inspection of the 3D volume. However, quantitative analyses such as exposed grain surface area and volume grade measurements require specific expertise with image processing algorithms. In this regard, a custom image analysis procedure is shown in Fig. 1, which gives a comparative quantitative analysis procedure for both exposed grain surface area and volume grade measurements in 3D.

Basically, the reconstructed 3D tomographic data was first denoised