3D ANALYSIS OF PARTICULATES IN MINERAL PROCESSING SYSTEMS BY CONE BEAM X-RAY MICROTOMOGRAPHY

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ABSTRACT

In general, x-ray computed tomographic (CT) techniques are able to provide 3D images of the internal structure of opaque materials in a nondestructive manner. The unique cone beam geometry allows acquisition of all 2D projections with only one rotation of the sample thus providing for fast data acquisition and better x-ray utilization, as a complete 2-D detector array receives the cone-shaped flux of rays. Thus, an isotropic 3D volume can be reconstructed without the mechanical translation and the stacking of sequential slices as is the case for more conventional CT scanners. In this regard, a state-of-the-art, custom designed x-ray microtomography facility to provide very detailed 3D spatial analysis of multiphase particles has been installed and is in operation at the University of Utah. The reconstructed 3D tomographic volume allows for an adjustable spatial resolution from 5 to 20 microns for sample dimensions from 10 to 40 mm in diameter, respectively. Utilization of this custom designed cone-beam x-ray microtomography system is discussed. Applications described include: coal washability analysis for the design and operation of coal preparation plants, liberation analysis for evaluation of grinding practice and separation efficiency, mineral exposure analysis for prediction of the ultimate recovery from heap leaching operations, and analysis of the pore structure network of packed particle beds for simulation of flow through such porous structures as encountered in filtration and heap leaching.

Keywords: X-ray microtomography, liberation, heap leaching, exposure analysis, coal washability.

INTRODUCTION

The analysis of multiphase particles and particle beds is of considerable importance for mineral processing/extractive metallurgy applications. Applications include coal washability analysis, mineral liberation analysis, mineral exposure analysis and analysis of pore structure network of packed particle beds. In each case, it is desired to obtain detailed information regarding size, shape, composition, texture,...etc.

For liberation analysis, generally, measurements are made on polished sections of carefully mounted particle samples. From the polished section, a portion of the internal structure of the particles is exposed for textural characterization and the determination of mineral liberation. The spatial interpretation of this one- and two-dimensional information extracted from such cross-sections can be accomplished by means of a variety of stereological procedures that have been developed in recent years (Miller and Lin, 1988; Schneider et al., 1991; Barbery, 1991; King and Schneider, 1993). The polished section analysis with stereological correction is a time consuming process. Furthermore, assumptions must be made either based on textural information or based on geometrical probability in order to provide the stereological correction. Finally, extension of the stereological correction for more than 2 phases is limited.

The question of ultimate recovery in heap leaching operations is always of particular concern with respect to economic considerations. Of course the particle size distribution is a critical factor which determines ultimate recovery and which must be established based on a balance between the extent of mineral exposure and transport phenomena.

In the copper heap leaching process, inclusions of the desirable valuable minerals (copper bearing minerals) are to be extracted from ore particles.

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The copper bearing minerals have some unknown grain size distribution, texture/exposure, and spatial distribution in the ore particles. The procedure is to crush the ore so that the valuable mineral grains are exposed and can be extracted during the heap leaching process. If the relationship between mineral exposure and particle size can be established for different ore types, then the ultimate recovery in the heap leaching process can be predicted for a specific particle size distribution. It is therefore extremely important to determine the percentage of exposed valuable mineral grains in the ore as a function of particle size. However, the percentage of exposed valuable grains in the ore can not be determined using conventional polished section analysis such as typically practice in the mining industry.

Filtration of fine particles involves filter cake formation and removal of surface moisture by drawing air through the porous particle bed structure. Accurate assessment of the transport properties of porous media (in this case, filter cake) is of major importance in the development of improved filtration processes. Implications from these studies are important in the design and operation of filtration equipment in order to enhance the efficiency of this important solid-liquid separation process. The microstructure and connectivity of pore space are important features necessary to describe detailed fluid flow phenomena in filter cake during fine particle filtration. In the same way, flow phenomena in heap leaching systems can be described. It is evident that 3D characterization of pore structure is most useful to describe the 3D-multiphase flow through packed particle beds.

The ability to obtain accurate 3D imaging and geometrical and textural information for a bed of multiphase, irregularly shaped particles is an important tool that can provide information to describe the performance of various processes mentioned previously in the mineral processing and extractive metallurgy industries. At the University of Utah we have been using a new technique, cone beam x-ray microtomography, to non-destructively produce accurate 3D images of packed particle beds. X-ray microtomography is not subject to the same limitations as the polished section technique. In this paper, we present information regarding the use of this new facility and review potential applications for this advanced analytical system.

METHODS

High Resolution Cone-Beam X-ray Microtomography System

In general, high-resolution x-ray microtomographic systems use penetrating x-ray radiation to image an opaque object and determine its internal features. The custom designed micro-CT system recently installed at the University of Utah (Lin and Miller, 2001), uses cone geometry to obtain data over an entire object volume. This enormously speeds up the process of imaging complete objects and provides data with the same spatial resolution in all directions

The essential feature of x-ray tomographic imaging is the determination of material density (more accurately attenuation coefficient) of a small region of three-dimensional space called a voxel. Tomography can determine the density of all voxels in the three-dimensional region of the scan. Of course, the position of each voxel is known precisely. It is desired to determine the geometric characteristics of any region of space that is subject to variations in density. In particular it is possible to determine precisely the shape, and therefore volume, as well as the mass of each individual phase within the target volume.

The University of Utah micro-CT system can achieve 2048 x 2048 pixel reconstructions over a 10-mm diameter object, while also allowing for the imaging of objects 40-mm in size. When operated at highest resolution, the smallest voxel is 5 microns, which corresponds to quantitative spatial resolution of about 12-microns. The dimensional accuracy achieved with this system is about 1 micron over 10 mm. This system is capable of imaging through high-density minerals, even minerals having a density as high as 8 g/cc. The resulting images are digitized with 16 bit-depth and this provides a large dynamic range that is required to image microsystems that are composed of a variety of minerals, such as coal macerals and pyrite.

Sample Preparation

The effectiveness of the 3-D x-ray cone-beam micro-CT for characterization and measurement of mineral liberation, mineral exposure, and the pore network microstructure of a packed particle beds has been demonstrated for various samples. Typically the particle sample is simply packed in a cylindrical plastic container of diameter from 3 to 40 mm and mounted on the micro-CT stage. Special sample preparation procedures are not necessary.

RESULTS AND DISCUSSION

Coal Washability Analysis

Application for the characterization and washability analysis of fine coal are reviewed below. The reconstructed cone-beam CT image consists of a 3-D array of x-ray attenuation coefficients, each associated with a finite-volume cube (voxel) of the sample. The attenuation coefficients are a function of the average density and composition of the material in any given voxel (Lin and Miller, 2001). A plot of the attenuation coefficient histogram is a measure of the density variation throughout the sample. Usually, the attenuation coefficient histogram obtained from known densities of a coal particle bed (such as the each density fraction obtained from sink-float tests) can provide the underlying component densities (Lin et al. 2001). By way of example, Figure 1 shows the attenuation coefficient histogram corresponding to the reconstructed 3-D microtomography image of the packed bed of coal particles. The attenuation coefficient histogram consists of an overlapping bivariate distribution as shown in Figure 1. The peak with the higher attenuation coefficient value (~ 0.053) is associated with the coal phase having a density between 1.50 and 1.60 g/cm^3. The peak corresponding to the lower attenuation coefficient value (~ 0) is associated with the external air space surrounding the sample.

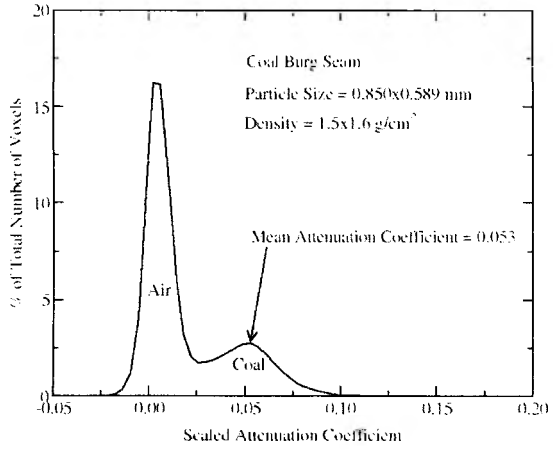

Figure 1. The x-ray attenuation coefficient histogram for the 3D x-ray microtomography analysis of a gravity fractionated sample (single size/single density) from the Coal Burg seam.

Cone-beam x-ray microtomography can be used to measure and quantitatively characterize fine coal particles directly. High spatial resolution and the direct processing of raw volumetric data are the two important benefits offered by this new method. To illustrate the ability of the high-resolution x-ray microtomography technique for quantitative characterization and analysis of fine coal particles, coal samples from Pittsburgh No. 8 seam were selected. Details of the liberation characteristics of pyrite and other ash forming minerals from these samples have been investigated using an SEM system equipped with automatic image analysis (King, 1999).

The high-resolution x-ray microtomography technique used for this study is capable of imaging through high-density material and the resulting 3D digital images are quite suitable for the characterization of fine coal particles which have a wide density range of components from organic macerals, to mineral matter including pyrite. Figure 2 illustrates the ability of the high resolution x-ray microtomography technique for quantitative analysis to determine the 3D spatial distribution of coal particles. Consider a selected 2-D x-ray tomographic slice from the reconstructed 3-D digital mapped x-ray attenuation coefficient of the packed bed of Pittsburgh No. 8 coal particles (0.500x0.355 mm, specific gravity > 1.6) as shown in Figure 2. In this image the pyrite phase has highest x-ray attenuation coefficient and is shown as white, other ash-forming minerals (most of them are silicate-based minerals) are light gray, and coal constituents are dark gray. The background (air) is black. Here the gray scale levels of the images are based on the relative x-ray attenuation coefficient and are indicative of different mineral phases present in the sample.

For quantitative analysis, the attenuation coefficient of each individual voxel of the 3D digital map can be used to process and to classify each particle and the different mineral grains inside each particle. This section is taken from the three-dimensional image; the image elements in the reconstruction are cubic (resolution 10 microns).

Pyrite has different crystalline forms (vein, nodule, and cluster) as shown in Figure 2. In fact, the 3D morphology and grains size distribution of pyrite provides value information for processing considerations. A volume rendered image from a subset of Pittsburgh No. 8 sample (256x256x256) is shown in Figure 3A. The width of the cube is 2.56 mm. Figure 3B shows sections of the particle bed with one half of the volume removed. The corresponding 3D view of the subset is shown in Figure 3C and reveals details of the distribution of ash-forming minerals (in gray) in a packed bed of coal particles (host coal constituents is set as transparent green). With the use of 3D image processing techniques, we can remove all the host coal phase and reveal only the ash-forming mineral grains as shown in Figure 3D. Distinct 3D morphology of different crystalline forms of pyrites is clearly established from Figure 3D. It is evident that the grain size distribution of the ash-forming minerals can be determined. In summary, it is concluded that the utilization of x-ray microtomography not only allows for quantitative analysis of multiphase systems but also allows for textural characterization and the determination of phase continuity.

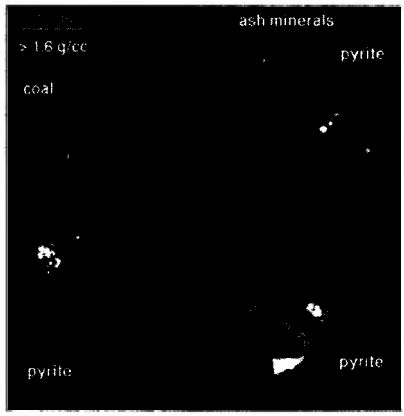

Figure 2. Coal constituents, pyrite and other ash-forming mineral phases identified from selected cross sectional image of the 3D reconstruction of multiphase coal particles (Pittsburgh No.8, 0.500x0.355 mm, specific gravity > 1.6).

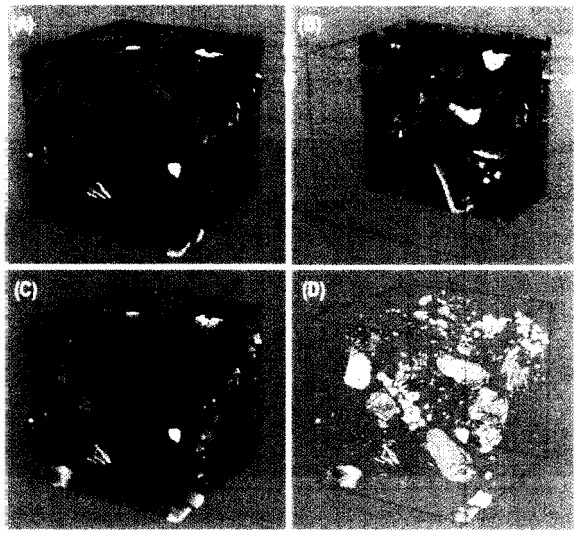

Figure 3. (A) Volume rendering image from a subset (256x256x256) of the packed bed of coal particle (Pittsburgh N0.8, 0.500x0.355 mm, specific gravity > 1.6). (B) Section views of (A) by removing half of the volume. (C) Semi-transparent volume rendering of (A). (D) Ash-forming mineral grains through the removal of coal phase.

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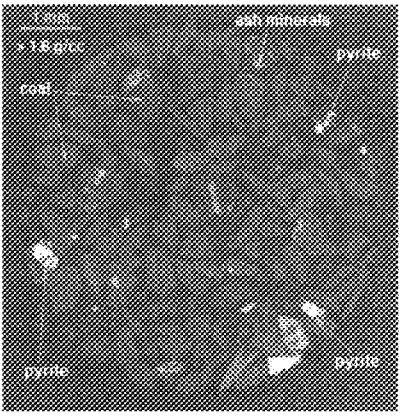

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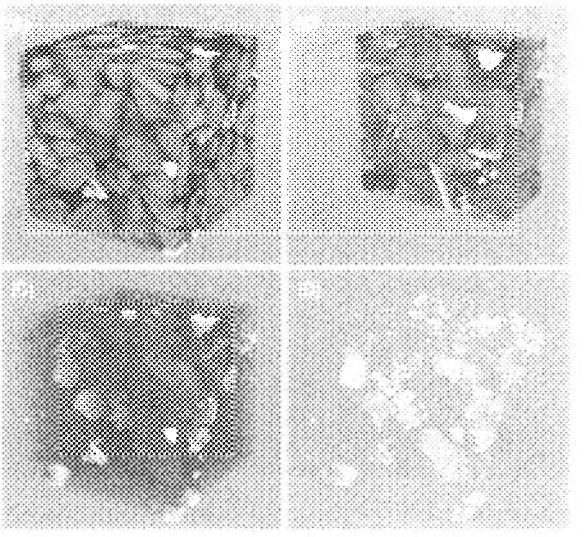

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X-ray CT scanning of single-size/single-density coal particle beds is a suitable method to obtain a statistical picture of the overall behavior of the attenuation coefficient with respected to density and size. Correlation between the density and x-ray attenuation coefficient can be obtained through the analysis of the peaks in the coal spectrum (Lin et al. 2001). Figure 4 presents a plot of attenuation coefficient versus density. It is noted that the density calibration curves fit very well for both coal samples (Coal Burg and Pittsburgh No.8 seams).

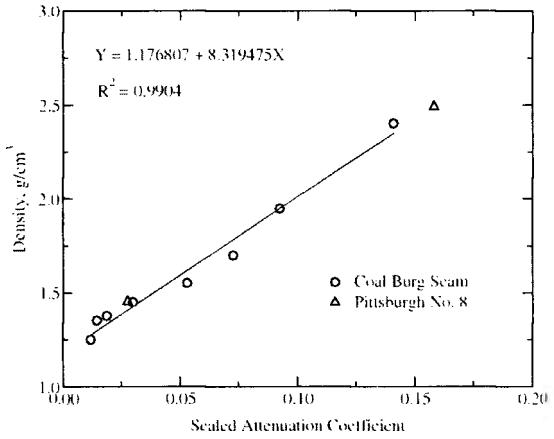

Figure 4. 3-D x-ray microtomography density calibration for the single-size/single-density coal particles (packed bed).

A fine coal sample (0.850x0.589 mm, multiple density fractions, Coal Burg seam) was prepared for washability analysis using 3-D x-ray microtomographic technique. Under carefully controlled laboratory conditions, coal particles from each density fraction (out of eight density classes) were collected and mixed. Using a plastic container (9 mm inside diameter and 20 mm in length), this mixed coal sample was scanned by 3-D x-ray microtomography. The 3D reconstructed image set contains 512x512x912 voxels (voxel resolution = 20 μm). The washability curve (yield/density) for this carefully prepared sample is shown in Figure 5 based on sink-float data. Previous study with coarse coal samples (Lin et al. 2001) indicated that such washability curve can be constructed based on x-ray CT analysis using a successive subtraction process. Based on this procedure, the washability curve can be established with the used of scale attenuation coefficient histogram and density calibration curve (Figure 4). Figure 5 illustrates the constructed yield/density curves based on the sink-float data and on the 3-D x-ray CT data.

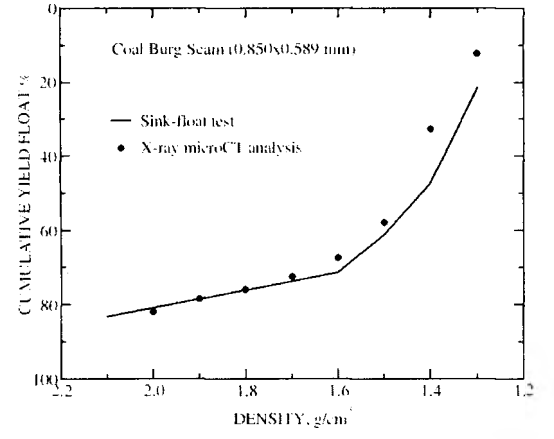

Figure 5. Comparison of the yield/density curve for mixed coal particles from 8 different density fractions (Coal Burg Seam, 0.850x0.589 mm) as determined by sink-float data with the results from 3-D x-ray microtomography analysis.

Mineral Liberation Analysis

For detailed liberation analysis, the volumetric grade distribution of multiphase mineral particles can be measured directly by cone-beam x-ray microtomography as described in a preliminary study (Lin and Miller, 1996). High spatial resolution and the direct processing of raw volumetric data are the two important benefits offered by this new method. Figure 6 illustrates the ability of the high resolution x-ray microtomography system for quantitative analysis to determine the 3D spatial distribution of mineral phases in multiphase particles. Four cross sections (from a total of 300 sections) along the Z-direction are shown as established from the three dimensional reconstruction of a copper ore sample (2.00x1.18 mm, specific gravity 3.5x3.1). It should be noted that these sections are taken from the three dimensional image. The image elements in the reconstruction are cubic, so the spacing between planes equals the resolution which in this case corresponds to 20 μm. Here the gray scale levels of the images indicate the relative attenuation coefficient present in the bulk of the sample. Based on x-ray attenuation coefficient, differentiation of mineral phases within the sample is possible as indicated in Figure 6 for pyrite and chalcopyrite.

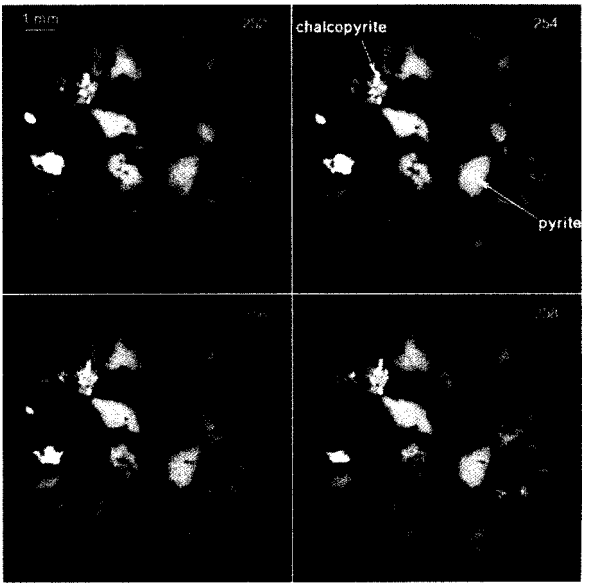

Figure 6. Cross sectional images from the three dimensional x-ray microtomography reconstruction of multiphase copper ore particles (2.00x1.18 mm, specific gravity 3.5x3.1). The dark colored grains distinguish the pyrite, chalcopyrite mineral phases in a silicate matrix.

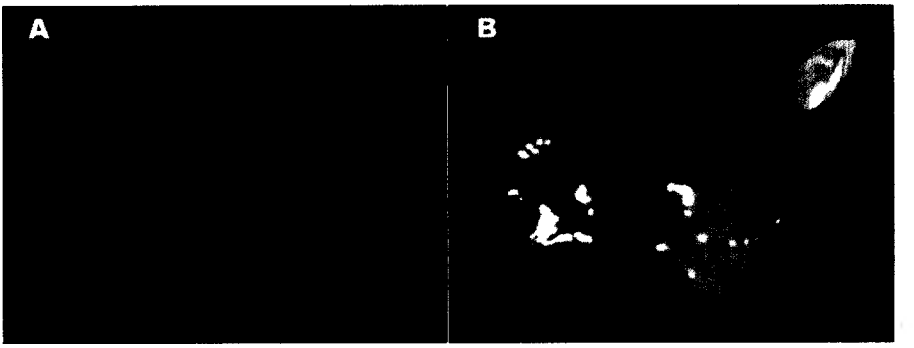

Figure 7. (A) Volume rendering image from sphalerite/dolomite particles, sphalerite in red and dolomite in green. (B) Sectional view of (A) by removing portion of the volume.

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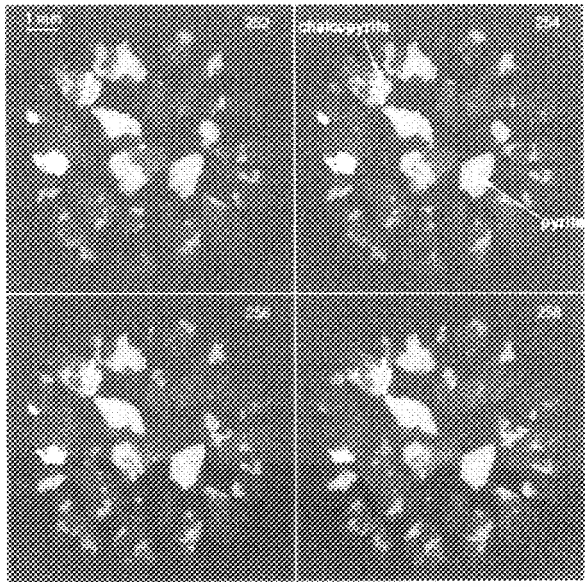

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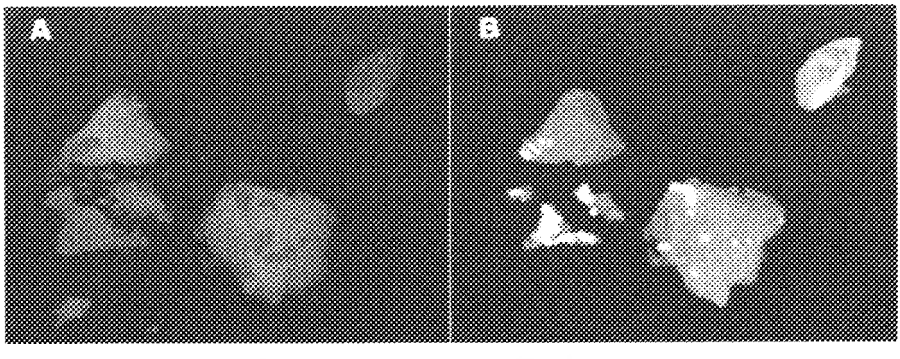

Figure 7. (A) Volume rendering image from sphalerite/dolomite particles, sphalerite in red and dolomite in green. (B) Sectional view of (A) by removing portion of the volume.

For quantitative determination of the volumetric grade distribution, suitable volumetric image analysis algorithms such as 3D segmentation, and classification, will be necessary to treat the three-dimensional array. In this regard, a volume rendered image from a sphalerite/dolomite sample is shown in Figure 7A. Figure 7B shows sections of the particles with a portion of the volume removed. The sphalerite (in white) and dolomite (grey) phases are clearly distinguished from this image. Table 1 shows the results for particles with a larger volume.

Table 1. Size and grade by volume for selected locked dolomite/sphalerite particles (Figure 11) as measured by three dimensional x-ray microtomography.

Particle No.	Volume, $mm^3 \times 10^6$	Equivalent Size, μm	Volumetric Grade, %
1	36.15.3	826.5	4.60
2	3562.6	708.9	4.79
3	1252.4	386.3	49.89
4	723.3	416.6	66.41
5	558.6	382.3	18.21
6	166.1	255.2	25.68

Three dimensional liberation analysis by microtomography provides an excellent opportunity to overcome many of the limitations of currently used polished section techniques. With the x-ray microtomography system, complete accounting of the 3D spatial distribution of mineral phases in each particle is possible, including grain size distribution, interfacial area, shape features and textural information.

Mineral Exposure Analysis for Heap Leach Operation

To further illustrates the ability of the high resolution XMT system for the quantitative mineral exposure/liberation analysis. One slice of cross sectional image from a total of 512 slices of the 3D data set for the original CT image is extracted and shown in Figure 8. Using a 3D image analysis algorithm, the overall copper bearing grains and internal/exposed grains can be identified and shown in the bottom of Figure 8 for comparison. In this case the percent of copper bearing mineral exposed was found to be 78 %. It must be emphasized that mineral exposure analysis can only be determined from the complete 3D data set.

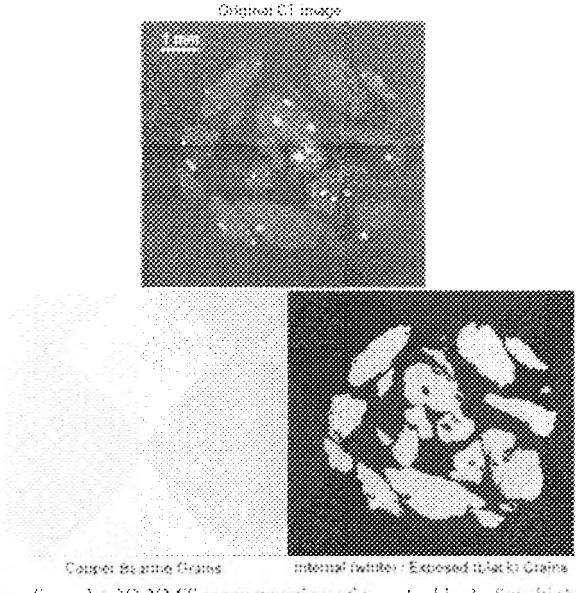

Figure 8. Cross sectional images from the 3D XMT reconstruction of a packed bed of multiphase copper ore particles. The overall copper bearing grains and internal/exposed grains obtained from 3D image analysis are shown in the bottom for comparison.

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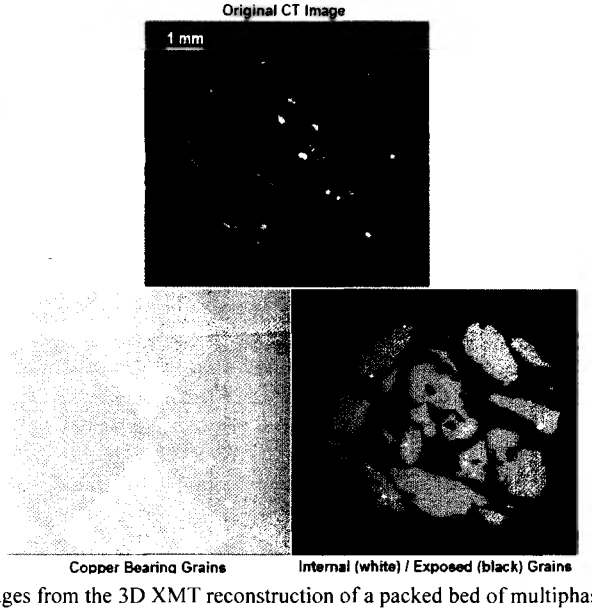

Figure 8. Cross sectional images from the 3D XMT reconstruction of a packed bed of multiphase copper ore particles. The overall copper bearing grains and internal/exposed grains obtained from 3D image analysis are shown in the bottom for comparison.

The ultimate recovery in the heap leaching process can be predicted for a specific particle size distribution, if we can determine the relationship between the percentage of the exposed valuable mineral with respect to particle size for different ore types. It is therefore extremely important to characterize the percentage of the exposed valuable mineral grains in the ore as a function of particle size. In this regard, three types of copper ore from different parts of the deposit were collected, sampled and sized to ten size intervals following the procedures mentioned previously in the sample preparation section.

Representative samples of particles from different size intervals were taken and put into a cylindrical container for XMT analysis. Figure 9 shows the relationship between the percent of copper exposed with respect to particle size for composite 4. It is noted that more than 98% of the copper mineral grains are exposed for particle sizes less than 0.425 micron (40 mesh). Many large almost completely liberated copper mineral grains (clusters of grains) were found in particle size classes of 3.18x1.7 mm (1/8"x10 mesh), and 1.7x0.425 mm (10x40 mesh).

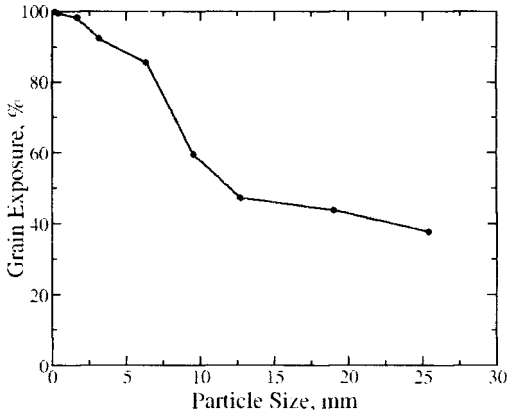

Figure 9. Relationship between the exposed valuable minerals with respected to particle size for composite 4.

The relationship between the percent of copper exposed and particle size, provides a basis for the prediction of copper recovery for specified particle size distributions. In this regard, three different particle size distributions were selected and 1.5- meter column tests were run for about 70 days. Figure 10 shows the predicted copper recovery based on the exposure analysis and actual copper recoveries from these column tests with different particle size distributions. As expected, good agreement between the predicted and actual recovery is obtained

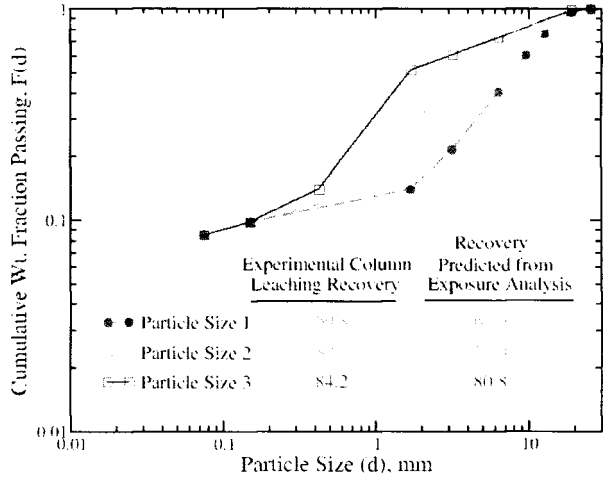

Figure 10 Comparison of experimental column recovery with that predicted from mineral exposure analysis by XMT for three different particle size distributions.

As expected the smaller the particle size, the higher the percentage of copper mineral exposed. However, in addition to exposure analysis, the fluid flow phenomena inside the packed bed of particles is an important issue that needs to be investigated. It is known that the smaller the particle size the lower the permeability of the packed particle bed. Once these issues (such as how particle size distribution influences both mineral exposure/liberation and flow behavior) are resolved optimal chemical conditions can be established and the chemistry designed for the optimum utilization of the various reactants. In any event, it is evident that ultimate recovery for any specific particle size distribution can be estimated from mineral exposure analysis by XMT.

Pore Structure Analysis of Particle Beds

Continuous filtration of fine particles involves filter cake formation and removal of surface moisture by drawing air through the porous structure. Accurate assessment of the transport properties of porous media (in our case filter cake) is of major importance in the development of improved filtration processes. The microstructure and the connectivity of the pore space are important to describe fluid flow in filter cake during fine particle filtration. In this regard, characterization of pore structure based on parameters permitting inferences on the fluid balance is of particular interest. The pore structure has to be described by parameters which are of special relevance for the interpretation of fluid transport phenomena. These parameters should be based on directly measured variables of the pore system and not indirect variables (such as those determined empirically from transport processes) valid only for a particular pore structure. In this way fundamental relationships between pore structure and fluid transport at the microstructure level can be described. Thus, it is desired to be able to directly measure the three-dimensional interconnected pore structure of filter cake.

Most present methods to characterize the pore microstructure and its completed interconnected network rely on the microscopic observation of a series of thin or polished sections of the porous media. These data sets are then used to reconstruct and to display the three-dimensional image of the porous system with the help of advanced computer graphic techniques. Complete analysis of the 3-D porous system from serial sections is a tedious and time-consuming process. In addition, for a completely interconnected porous system, pore size distribution is not a well-defined parameter (Lin and Miller, 2000).

A packed bed of iron ore particles (180x106 μm) was prepared for the study of complex filter cake pore structure using high-resolution 3-D x-ray microtomography. Figure 11A shows one slice from the volume data set for the packed bed of iron ore particles. A volume rendering image from a subset of this sample (256x256x128) is shown in Figure 11B. The voxel size for this sample is 10 μm. Connectivity is an important concept when flow problems are considered. Fluid flow can occur between two points only when the pore space is connected. In this regard, overall pore structure is extracted from Figure 11A and the surface rendering image is shown in the Figure 12A. Results from a 3-D connection of components analysis indicates that the overall pore structure is composed with several independent major networks (Figure 12B) and some isolated pores. The fundamental relationship between pore microstructure and effective transport coefficients can be established based on the flow simulation through the porous media and has been discussed in a separated paper (Lin and Miller, 2002).

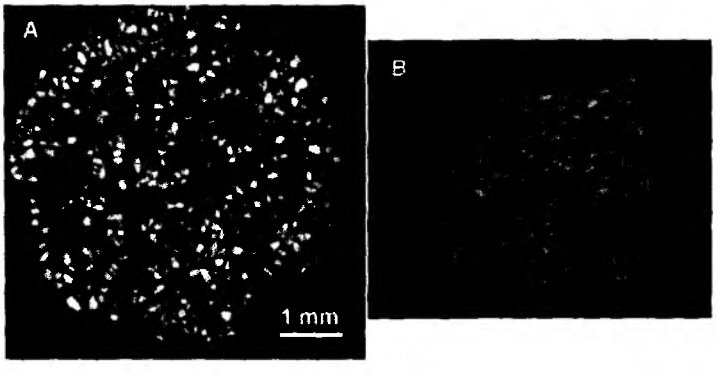

Figure 11. (A) Selected cross sectional image from a packed bed of iron ore particles (180x106 μm). (B) Volume rendering image from a subset (256x256x128) of the packed bed of iron ore particles.

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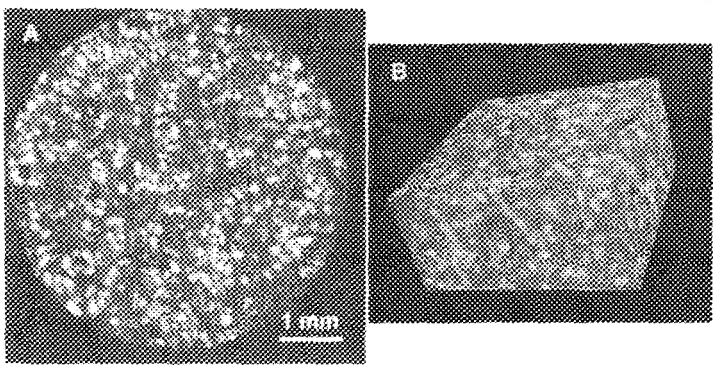

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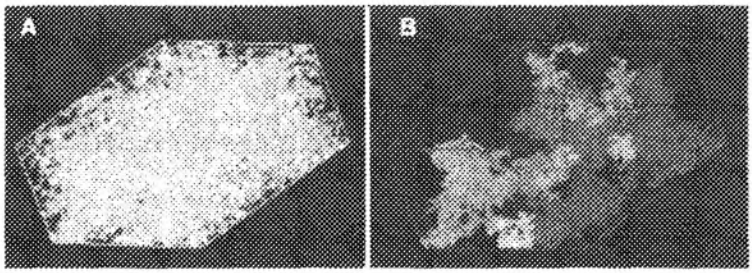

Figure 12. (A) Surface rendered image of the pore structure, from a subset (256x256x128) of the 3-D Micro-CT data set (Figure 11B). (B) Overall pore structure is composed with several independent major networks.

SUMMARY AND CONCLUSIONS

The ability to obtain accurate 3D imaging and geometrical/textural information of multiphase, irregularly shaped particles is an important tool that can provide information to evaluate the performance of various particulate processes encountered in mineral processing and hydrometallurgy. In this regard, the utilization of x-ray microtomography not only will allow for quantitative analysis of multiphase particulate systems but also allow for textural characterization and the determination of phase continuity. Potential applications for this advanced analytical system were reviewed and include particle composition (coal washability/3D liberation analysis), mineral exposure analysis for heap leaching operations and pore structure analysis of packed beds to describe filtration. It is noted that 3D mineral liberation analysis using x-ray microtomography can be achieved within one hour. In this regard, it now seems possible to design an on-line system for the control of plant operations.

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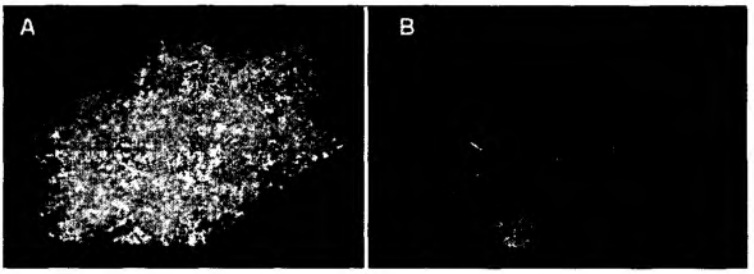

Figure 12. (A) Surface rendered image of the pore structure from a subset (256x256x128) of the 3-D Micro-CT data set (Figure 11B). (B) Overall pore structure is composed with several independent major networks.

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