Cone beam X-ray microtomography — a new facility for three-dimensional analysis of multiphase materials

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Abstract

As the techniques and resolution for three-dimensional spatial analysis have advanced in the last decade, it is now possible to map in detail the microstructure of multiphase materials in three-dimensional digital space. In this regard, three-dimensional X-ray microtomography offers a unique imaging capability. Spatial resolution on the order of 10 μm can be achieved with the use of microfocus X-ray generators. Recently, a state-of-the-art cone beam X-ray microtomography system was installed at the University of Utah for the quantitative analysis of multiphase materials in three dimensions. This facility was designed to obtain 2,048 by 2,048-pixel reconstruction over a 10-mm diameter, while also allowing for the imaging of somewhat larger (40-mm) objects. The system is capable of handling high-density materials, even materials having a density as high as 8.0 g/cm^3. This unique, one-of-a-kind instrument can be used to obtain three-dimensional spatial reconstruction for many applications, such as three-dimensional liberation analysis, pore structure analysis of particle beds during filtration and the air-void system of concrete structures. The utilization of X-ray microtomography not only allows for the quantitative analysis of multiphase systems but also allows for the textural characterization and determination of phase continuity. This paper presents information regarding the current use of this new facility and a review of potential applications for the advanced analytical system.

Key words: Cone beam X-ray microtomography, Three-dimensional analysis, Material characterization

Introduction

In the mineral processing and extractive metallurgy industries, the successful concentration of mineral values depends, in part, on the degree of mineral liberation. Quantitative mineralogy can be used to provide this important information. 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 the 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; Barbery, 1991; Schneider et al., 1991; King and Schneider, 1993; King, 1994). Such a polished section analysis with stereological correction is a time-consuming process. Furthermore, assumptions must be made based on either textural information or geometrical probability to provide the stereological correction. Finally, extension of the stereological correction for more than two phases is limited.

New techniques for improving productivity and efficiency are the key to success in today's highly competitive market. For continued technological progress in the field of mineral processing and extractive metallurgy, the need for quantitative information regarding mineral liberation has increased significantly. Such quantitative information must be accurate enough so that the measured values can be used as parameters for simulation models, process design procedures and control strategies. In this regard, X-ray microtomography offers a unique capability to image multiphase systems in three dimensions. Spatial resolution on the order of 10 μm can be achieved with the use of microfocus X-ray generators. High-resolution three-dimensional X-ray microtomography can be used for the direct determination of the liberation spectrum of multiphase particles several 100 μm in size from mineral processing streams.

X-ray tomography background. The first discovery of X-rays by Roentgen in 1895 provided a new imaging technique. The primary advantage of X-rays is the capability to penetrate thick, intact and wet samples with natural contrast. At present, radiography is used for the inspection of materials in many applications. It is noted that overlaying structures are ambiguous in their physical location using conventional radiography. This problem can be solved by the use of tomographic techniques.

Image reconstruction from projections using computed tomography (CT) produces a noninvasive measure of structure from external measurements. In X-ray CT, radiation is passed along straight lines through the object to a detector. The specimen is located between the source and detector. The

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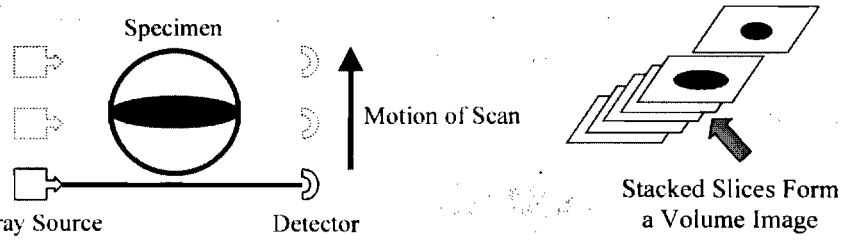

Figure 1 —Volume reconstruction from traditional "slice-by-slice" tomography.

source and detector are coupled as the specimen is rotated and stopped at predetermined locations to acquire projections through the specimen. These projections are proportional to the amount of radiation reaching the detector. These measurements can be considered line integrals of the X-ray attenuation coefficients of the specimen. Knowledge of all line integrals allows the reconstruction of the interior structure (Kak and Slaney, 1987). X-ray tomographic reconstruction produces a two-dimensional map of X-ray attenuation coefficients of the irradiated cross section of the specimen. Differentiation of features within the sample is possible because linear attenuation coefficient (μ) at each point depends directly on the electron density, the effective atomic number (Z) of the material comprising the sample and the energy of the X-ray beam (E). A simplified equation that illustrates the approximate relationship between these quantities is

$$\mu = \rho\left(a + \frac{bZ^{3.8}}{E^{3.2}}\right) \qquad (1)$$

where

ρ is the density of the phase,

a is a quantity with a relatively small energy dependence and

b is a constant (McCullough, 1975; Wellington and Vinegar, 1987).

When a mixture of atomic species is present, Z (the effective atomic number) is defined by

$$Z^{3.8} = \sum_i \left(f_i [Z_i]^{3.8}\right) \qquad (2)$$

where

f_i is the fraction of the total number of electrons contributed by element i with atomic number Z_i.

For the case of transmission tomography such as X-ray CT, the modern CT scanners are capable of discriminating between values of μ that differ by as little as 0.1%. Different mineral phases that may be contained inside a host ore body can be discriminated easily using the X-ray CT technique. In practice, density measurement from X-ray tomographic data can be done either by calibrating the CT machines with objects of known density and obtaining a correlation equation that relates density with attenuation coefficients or using dual energy scanning to directly determine the density of the material. In such a fashion, then, a two-dimensional density map of the object under investigation can be established.

Typically, volumetric imaging of the sample is obtained by stacking a series of two-dimensional slices of CT image data. It should be noted that the quality and utility of the CT data ultimately depends on the resolution of the machine employed. Medical X-ray CT systems have a beam width of a few millimeters and an energy source of about 130 keV X-rays. Generally, such a system would not be adequate for liberation analysis of particles several hundred micrometers in size, which would be of interest to mineral processing operations.

High-resolution three-dimensional X-ray microtomography. The application of the principles of CT at the microscale level, or microtomography, allows quantitative investigation of objects in three dimensions. Only recently were practical microtomography systems developed. Early microtomography systems produced two-dimensional images. Some current systems, including the one described here, produces true three-dimensional image data. In addition to electron-impact X-ray sources, X-rays from synchrotrons were also used in microtomography. Spatial resolution of the order of 1 to 15 μm can be achieved with the use of synchrotron radiation (Grodzins, 1983; Flannery et al., 1987; Kinney et al., 1988) and conventional microfocus X-ray generators (Feldkamp and Jesion, 1986; Dunsmuir et al., 1991; Kinney et al., 1991).

As the resolution and the techniques for three-dimensional geometric analysis have advanced in the last decade, it is now possible to map in detail the mineralogical texture of ore particles in three-dimensional digital space. It is expected that the mineral phases inside particles will be described in three dimensions with micrometer resolution. In this regard, three-dimensional X-ray microtomography offers a unique imaging capability. Spatial resolution on the order of 10 μm can be achieved with the use of microfocus X-ray generators.

For many years, the standard method of acquiring a volumetric CT scan was by scanning a sample one slice at a time. In this method, a linear detector array and a X-ray point source are mounted opposite each other. A fan-shaped X-ray beam traverses the sample and the attenuated fan is stored as a one-dimensional linear image. By rotating the source/detector pair around the sample, a series of linear images are obtained that are subsequently used for the two-dimensional slice reconstruction. A volumetric representation is obtained by advancing the table on which the sample rests after each rotation in order to acquire a stack of such slices. Figure 1 shows the approach of traditional "slice-by-slice" tomography.

The problem with this approach is:

- Projection data covering sample regions between the thin slice sections is not available, which may account for missing small object information if the small object is located between two slices. For this reason, the related spacing factor cannot be determine accurately.
- The stop-and-go table movement may cause sample displacement between sequential scans, leading to slice misalignment in the volume reconstruction.
- Better lateral resolution is achieved when compared to axial resolution. The problem in image reconstruction is that the volume elements have different lateral and axial extents. This makes the reconstruction complicated and significantly inaccurate if the three dimensional reconstruction is to be used for quantitative purposes.

On the other hand, for cone-beam CT, a whole three-dimensional data set is acquired within only one rotation of the

sample. This provides for fast data acquisition and better X-ray utilization, as a complete two-dimensional detector array receives the cone-shaped flux of rays. In the cone-beam design, all data in a two-dimensional image now originates from the same instance in time and enables easy gated imaging. In addition, the cone-beam geometry provides an opportunity to use image-based methods for three-dimensional reconstruction. This reconstruction algorithm is a generalization in three dimensions of the widely used convolution-back projection method. Figure 2 shows a schematic diagram for the cone-beam geometry microtomography. Clearly, cone-bean CT has the best prospects for true three-dimensional liberation analysis and should be able to provide the necessary accuracy to quantitatively determine the three dimensional distribution of mineral phases in multiphase particles.

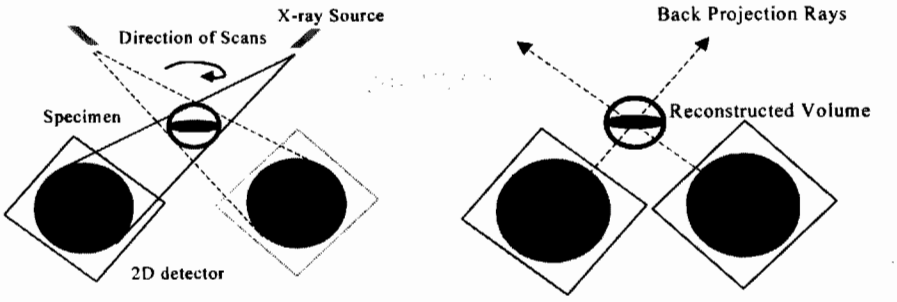

Figure 2 — Volume reconstruction from cone-beam tomography.

X-ray microtomography system

The state-of-the-art X-ray micro-CT machine, Konoscope 40-130 (cone beam geometry from ARACOR), was custom-designed and installed in September 2000. The unit is now operating in a dedicated laboratory at the University of Utah. The Konoscope system was designed based on the following considerations:

- a system geometry optimized to obtain high-resolution two- and three-dimensional CT images of small samples but with the flexibility to examine larger objects as required;
- a detector with the resolution, efficiency and dynamic range required to obtain high-quality data from a broad spectrum of samples;
- a reliable X-ray source that will allow one to focus on research rather than maintenance; and
- a high-accuracy positioning system required to maintain the spatial resolution provided by the X-ray imaging component.

In this regard, the Konoscope system was designed and assembled to obtain 2,048 x 2,048-pixel reconstruction over a 10-mm diameter, while allowing for the imaging of somewhat larger (40-mm) objects. Specifically, the specimen-positioning stage system can be manually mounted at one of three locations, providing system magnifications of 5, 2.5 or 1.25 and spheres of reconstruction with respective diameters of 10, 20 or 40 mm. In addition, the system was designed to be capable of handling high-density materials, even materials having a density as high as 8.0 g/cm^3.

The Konoscope system consists of a microfocus X-ray source, a specimen positioning stage and a digital X-ray detection camera. All these hardware components are integrated inside an interlocked radiation-safety enclosure. This container is mounted on a vibration-isolated stand that will also supports the power-supplies, drivers and controllers for the X-ray source, CCD camera and positioning system. Figure 3 provides a perspective view of the system. A photograph of the cone beam X-ray microtomography system is shown in Fig. 4. A Macintosh Power PC scan-control computer (with

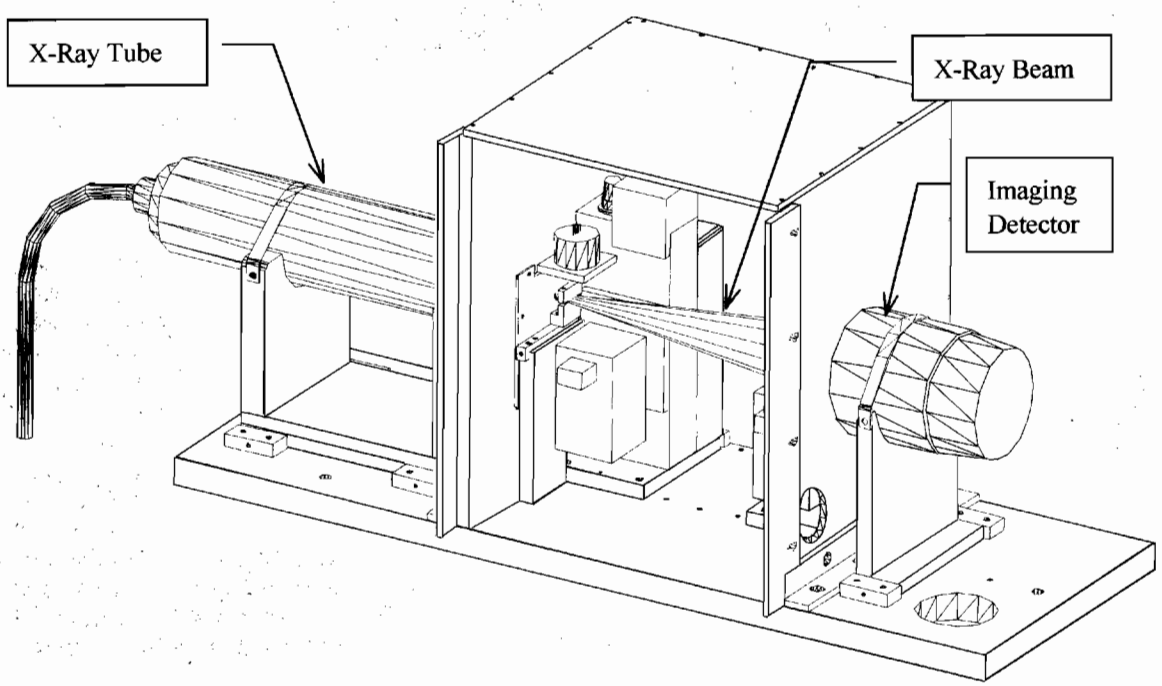

Figure 3 — Schematic diagram of X-ray microtomography system (Konoscope 40-130).

Figure 4 — The cone-beam X-ray microtomography system.

LabView/LVCam software) is used for control of image acquisition and specimen movement. At present, three image-acquisition modes (radiograph, fan-beam and cone-beam geometry) can be performed depending on the application. In the future, the system will be easily adapted with suitable scan-control and reconstruction software to perform the spiral or helical scan-geometry tomography. Three-dimensional tomographic images are reconstructed using SGI Octane workstation (2 MIPS R12000 processors, 2Gigabyte of RAM).

High-resolution X-ray microtomographic applications

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, the volume, as well as the mass of each individual phase within the target volume. Examples of three applications are reviewed below.

Particle composition distribution (three-dimensional liberation analysis). As mentioned previously, polished section techniques for the examination of the internal structures of particles are destructive and require lengthy experimental procedures. Recently, the application of X-ray CT in mineral processing technology was reviewed (Miller et al., 1990; Lin et al., 1992). For quantitative analysis of particulate systems such as coal washability analysis, a recent study (Lin et al., 2000) indicated that a conventional medical X-ray CT scanner could provide sufficient information to construct the washability curve within minutes of sample collection. In fact, it now seems possible to design an on-line washability system for the control of coarse-coal cleaning circuits.

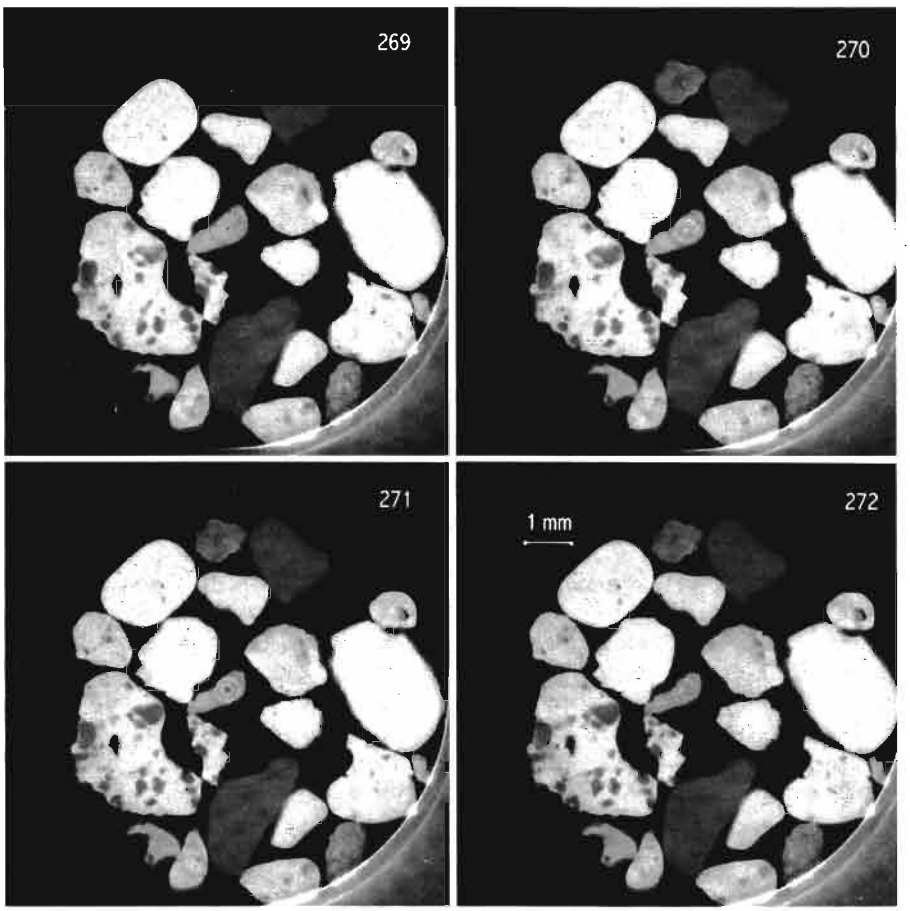

Figure 5 — Cross-sectional images from the three-dimensional X-ray microtomography reconstruction of multiphase phosphate.

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 previous 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 5 illustrates the ability of the high-resolution X-ray microtomography (Konoscope) system for quantitative analysis to determine the three-dimensional spatial distribution of mineral phases in multiphase particles. Four consecutive cross sections (from a total of 500 sections) along the Z-direction are shown as established from the three-dimensional reconstruction of a phosphate

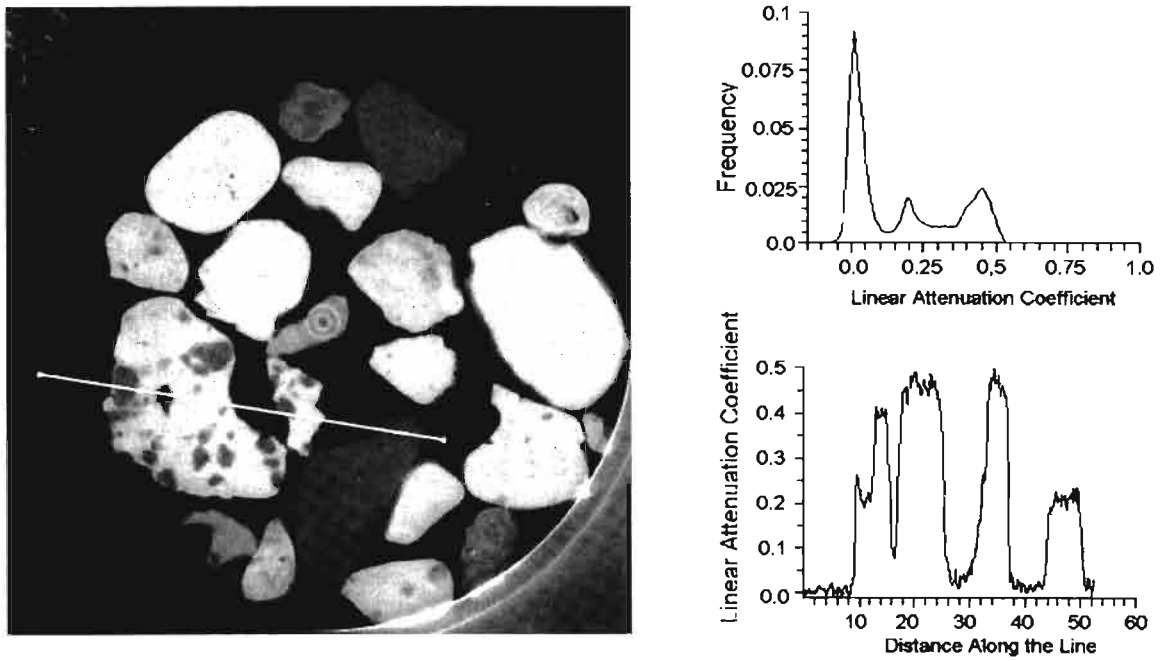

Figure 6 — X-ray attenuation coefficient histogram and individual voxels along the line from the reconstructed image using X-ray microtomography.

rock sample. It should be noted that these sections are taken from the three-dimensional image and not from the "slice-by-slice" approach mentioned above. The image elements in the reconstruction are cubic, so the spacing between planes equals the resolution in which the Z-direction corresponds to 20 μm. Here, the grayscale levels of the images indicate the relative attenuation coefficient present in the bulk of the sample. The X-ray attenuation coefficient histogram of the left-hand side image in Fig. 6 is shown in the upper right-hand side. X-ray attenuation coefficients of individual voxels along the line are shown in the lower right-hand side of Fig. 6.

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 three-dimensional spatial distribution of mineral phases in each particle is possible, including grain size distribution, interfacial area, shape features and textural information.

Pore structure analysis of particle beds during filtration. 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 medium (in our 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 to enhance the efficiency of this important solid-liquid separation process. 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 that 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 based on 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 three-dimensional 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. To illustrate the nature of an interconnected porous structure, a two-dimensional image slice of a packed bed of irregularly shaped particles was taken from three-dimensional CT data obtained from high-resolution cone-beam X-ray CT and is shown in Fig. 7 (Lin and Miller, 2000). As shown in Fig. 7, the three-dimensional CT data set was preprocessed to facilitate the pore geometry analysis. Although the binary image of the pore space shown in Fig. 7 is not connected, analysis from three-dimensional connection of components indicates that more than 99% of the pore space belongs to a well-connected pore system. Surface rendering of a subset (64 x 64 x 64) of this well-connected pore network is shown in Fig. 8.

The voxel size for the previous pore geometry study of a packed bed is 17 μm. To evaluate the effectiveness of the newly installed high-resolution three-dimensional X-ray microtomography measurement of complex filter cake pore structure, a packed bed of iron ore particles (180 x 106 μm) was prepared for preliminary CT analysis. The sample was scanned using the Konoscope microtomography system. Figure 9 shows one slice from the volume data set for the packed bed of iron ore particles. The voxel size for this sample is 10 μm. From the image in Fig. 9, it evident that the resolution of the newly installed X-ray microtomography system is better than the system used for the previous study.

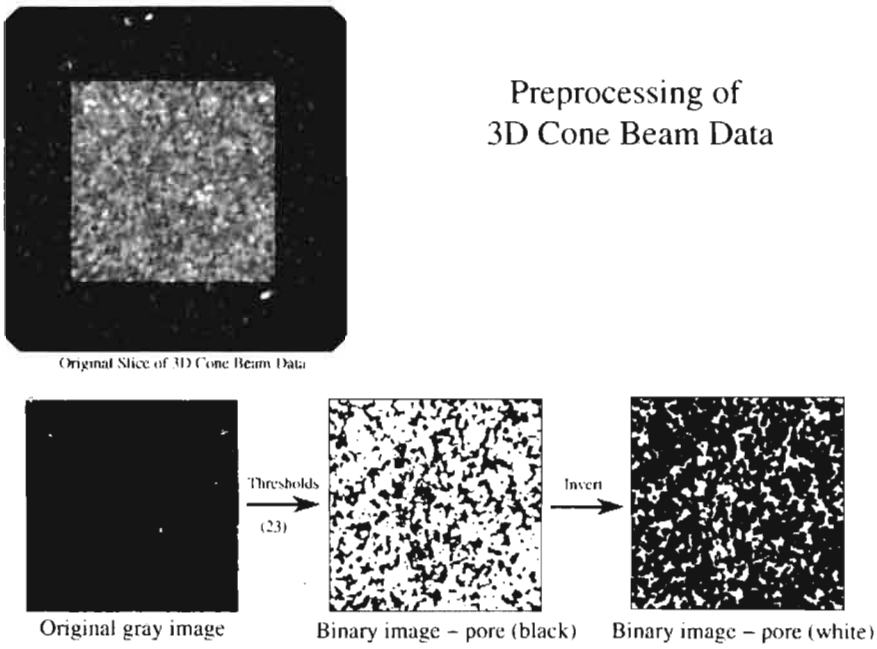

Figure 7 — Completed interconnected pore structure of packed bed of irregularly shape particles.

Air-void system of concrete structures. Entraining air in concrete imparts enhanced freeze-thaw resistance, better resistance to damage from deicers and salts, improved resistance to sulfate attack and reduced destruction due to alkali-silicate reactivity. Air entrainment is usually achieved using anionic air-entraining surfactants. These air-entraining reagents adsorb at the air/water interface and act to stabilize the air bubbles and to prevent their coalescence. The air bubbles formed are dispersed in the cement during the mechanical mixing of the material. Air bubbles entrained in this way are generally between 10 and 1000 μm in diameter. Larger air bubbles (>1,000 μm in diameter) are also present in all concrete due to entrapment of air during mechanical mixing. In any event, it is evident that the air-void size distribution is of particular interest. To achieve the maximum positive effect of air-entrainment, the spacing between the air bubbles is also a very important parameter and should be considered. Correct determination of the air-void system properties is of paramount importance to establish the durability of concrete particularly with respect to freeze-thaw damage.

In contrast to the traditional ASTM procedure, use of X-ray microtomography offers the opportunity for the determination of the true three-dimensional air-void size distribution. Figure 10 illustrates the ability of the high-resolution X-ray microtomography (Konoscope) system for three-dimensional quantitative analysis of air-voids inside a concrete structure. Four consecutive cross sections along the Z-direction are shown in Fig. 10, as established from the three-dimensional reconstruction of the concrete specimen.

Conclusions

Recently, a state-of-the-art cone beam X-ray microtomography system (Konoscope) was installed at the University of Utah for the quantitative analysis of multiphase materials in three dimensions. This facility was designed to obtain 2,048 x 2,048-pixel reconstruction over a 10-mm diameter, while allowing for the imaging of somewhat larger (40-mm) objects. The system is capable of handling high-density materials, even materials having densities as high as 8.0 g/cm^3. Details of the cone-beam X-ray microtomography system were presented. This unique, one-of-a-kind, instrument is used to obtain three-dimensional spatial reconstruction

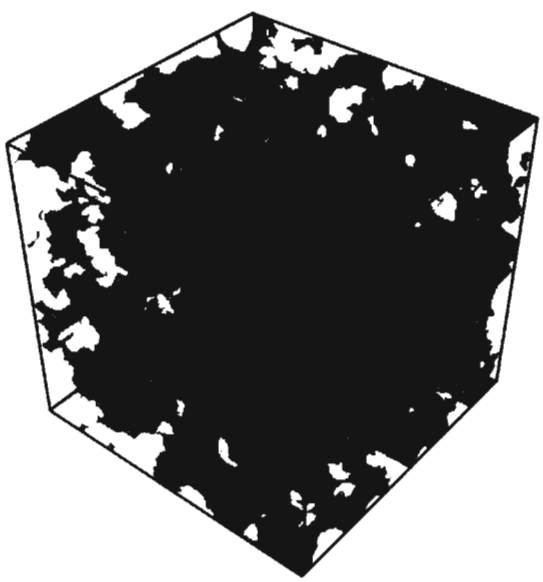

Figure 8 — Surface rendered image of the well-connected pore network from subset (64 x 64 x 64) of three-dimensional data set obtained from X-ray microtomography.

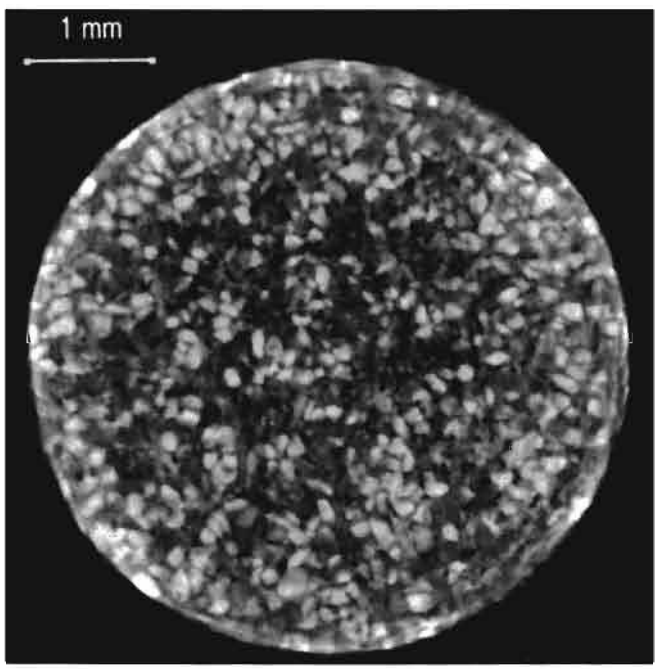

Figure 9 — Selected cross-sectional images of packed bed samples of iron ore particles (180 x 106 mm).

multiphase materials of irregular shape. The utilization of X-ray microtomography will allow for not only quantitative analysis of multiphase systems, but it will allow for textural characterization and the determination of phase continuity. Potential applications for the advanced analytical system were reviewed and include particle composition distribution (liberation analysis), pore structure analysis of packed beds and the air-void system of concrete. Many other applications of X-ray microtomography analysis are expected, including microelectromechanical system (MEMS), fault mechanics and fluid-transport studies, bone architecture and multilayered ceramic composites for structural and electronic applications.

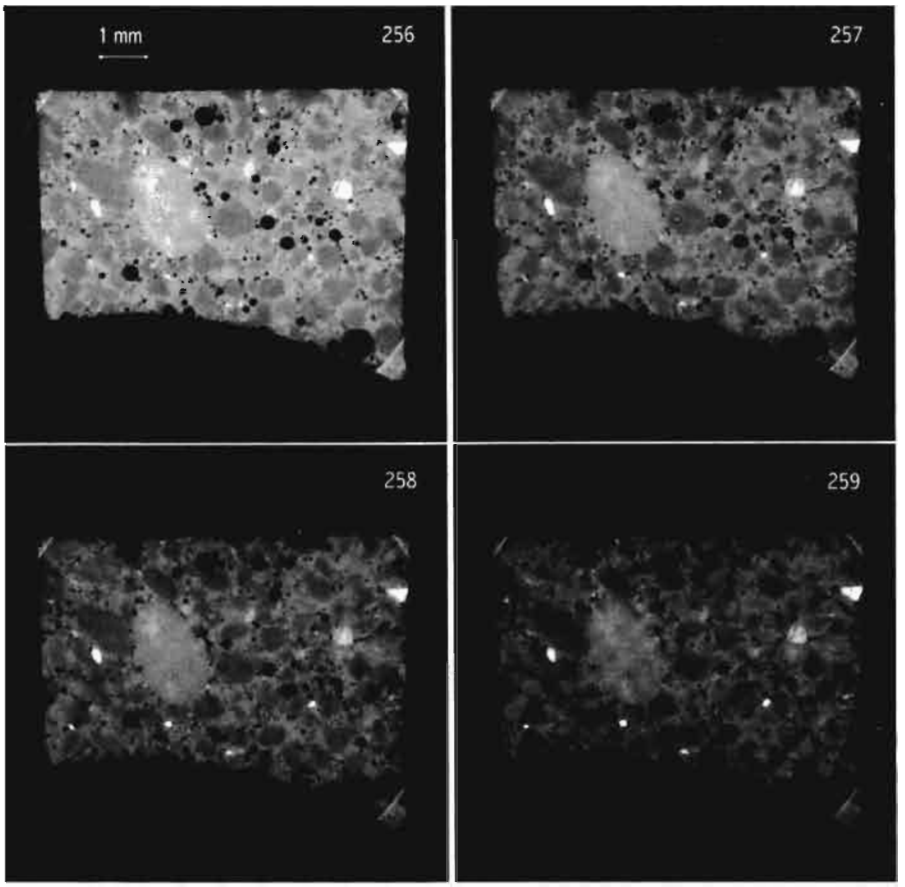

Figure 10 — Cross-sectional images from the three-dimensional X-ray microtomography reconstruction of a cement concrete specimen. The space between these sequential cross-sectional images is 20 μm.

Acknowledgements

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References

Barbery, G., 1991, *Mineral Liberation Measurement, Simulation and Practical Use in Mineral Processing*, Les Editions GB, Quebec.

Dunsmuir, J.H., Ferguson, S.R., D'Amico, K.L., Flannery, B.P., and Deckman, H.W., 1991, "X-ray microtomography: Quantitative three-dimensional X-ray microscopy," *Review of Progress in Quantitative Nondestructive Evaluation*, D.O. Thompson and D.E. Chimenti, eds., Plenum Press, New York, Vol. 10A, pp. 443-449.

Feldkamp, L.A., and Jesion, G., 1986, "Three-dimensional X-ray computed tomography," *Review of Progress in Quantitative Nondestructive Evaluation*, D.O. Thompson and D.E. Chimenti, eds., Plenum Press, New York, Vol. 8A, pp. 555-566.

Flannery, B.P., Deckman, H.W., Roberg, W.G., and D'Amico, K.L., 1987, "Three-dimensional X-ray microtomography," *Science*, Vol. 237, pp. 1439-1444.

Grodzins, L., 1983, "Optimum energies for X-ray transmission tomography of small samples — applications of schnchrotron radiation to computerized tomography I," *Nucl. Instrum. Meth.*, Vol. 206, pp. 541-545.

Kak, A.C., and Slaney, M., 1987, *Principles of Computerized Tomographic Imaging*, IEEE Press, New York.

Kinney, J.H., Johnson, Q.C., and Saroyan, R.A., 1988, "Energy-modulated X-ray microtomography," *Rev. Sci. Instrum.*, 59 (1), pp. 196-197.

Kinney, J.H., Saroyan, R.A., and Massey, W.N., 1991, "X-ray tomography microscopy for nondestructive characterization of composite," *Review of Progress in Quantitative Nondestructive Evaluation*, D.O. Thompson and D.E. Chimenti, eds., Plenum Press, New York, Vol. 10A, pp. 427-433.

King, R.P., 1994, "Linear stochastic models for mineral liberation," *Powder Technology*, Vol. 81, pp. 217-234.

King, R.P., and Schneider, C.L., 1993, "An effective SEM-based image analysis for quantitative mineralogy," *KONA*, Vol. 11, pp. 165-177.

Lin, C.L., Miller, J.D., and Cortes, A.B., 1992, "Applications of X-ray computed tomography in particulate systems," *KONA*, Vol. 10, pp. 88-95.

Lin. C.L., and Miller, J.D., 1996, "Cone beam X-ray microtomography for three-dimensional liberation analysis in the 21st Century," *Int. J. Miner. Process.*, Vol. 47, pp. 61-73.

Lin, C.L., Miller, J.D., Luttrell, G.H., and Adel, G.T., 2000, "Development of an on-line coal washability analysis system using X-ray computed tomography," *Coal Preparation*, Vol. 21, pp. 383-409.

Lin, C.L., and Miller, J.D., 2000, "Network analysis of filter cake pore structure by high resolution X-ray microtomography," *Chemical Engineering Journal*, Vol. 77, pp. 79-86.

McCullough, E.C., 1975, "Photon attenuation in computed tomography," *Medical Physics*, Vol. 2, pp. 307-320.

Miller, J.D., and Lin, C.L., 1988, "Treatment of polished section data for detailed liberation analysis," *International Journal of Mineral Processing*, Vol. 22, pp. 41-58.

Miller, J.D., Lin, C.L., and Cortes, A.B., 1990, "A review of X-ray computed tomography and its applications in mineral processing," *Miner. Process. Extract. Metall. Rev.*, Vol. 7, pp. 1-18.

Schneider, C.L., Lin, C.L., King, R.P., and Miller, J.D., 1991, "Improved transformation technique for the prediction of liberation by random fracture model," *Powder Technology*, Vol. 67, pp. 103-111.

Wellington, S.L., and Vinegar, H.J., 1987, "X-ray computerized tomography," *J. of Petroleum Technology*, Vol. 8, pp. 885-898.