Synchrotron-Based X-ray Microtomography Imaging of Particles

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Introduction

Measurement of the relative quantities of minerals and the degree of liberation of an ore mineral is needed by applied mineralogists and mineral processing engineers. Such measurements are currently made on 2-D surfaces under an optical microscope or a scanning electron microscope interfaced to an image analyzer. There are several sources of error arising from measurement of 3-D features on 2-D surfaces. Correction methodologies developed so far have limitations, and there does not appear to be a satisfactory correction method.

High-intensity x-ray beams from the synchrotron make microtomographic imaging of small particles in 3-D possible at a resolution and sensitivity to detect compositional differences between particles. An exploratory microtomography experiment was carried out with the ultimate objective to evaluate the sources and magnitudes of uncertainties arising from quantitative mineralogical measurements on 2-D surfaces.

Methods and Materials

Several sets of binary mixtures made of glass and epoxy resin, and lead borate and glass particles were analyzed. The mixtures were synthetic, prepared by combining known concentrations of material, crushing and wet screening. Mixtures made of glass and epoxy were placed in polyethylene tubes measuring approximately 2 to 4 mm in diameter and 20 to 27 mm in length. The particles were freestanding in the polyethylene sample holder with air filling the interstices. The set of mixtures made of lead borate and glass were placed in a cone-shaped polyethylene container that was 2 cm high with 5 mm outer diameter at the top and 2 mm diameter on the bottom.

X-ray microtomography measurements were performed on the GSE-CARS bending magnet beamline on sector 13 of the Advanced Photon Source at Argonne National Laboratory. The data were collected using monochromatic x-rays at 15.5 and 18.6 keV. The radiographs were obtained using a 50 mm single-crystal YAG scintillator and a high-speed 12-bit CCD camera placed downstream of the sample. An objective of 5x magnification was used to cover the entire width of the samples with a final resolution of 6.66 x 6.66 µm per pixel. The samples were rotated counter-clockwise at 0.5o intervals for 180º during analysis with the exposure time at each interval of 8 or 10 seconds.

Results

Individual images were preprocessed and combined into a volume file by the use of software written in IDL. After the volume files were prepared, approximately 500 slices, perpendicular to the rotation axis, were reconstructed at 7 µm intervals. An example is shown in Fig. 1 for four slices taken at 33.5 µm and 67 µm intervals from the sample containing a mixture with 15 to 25 vol % glass.

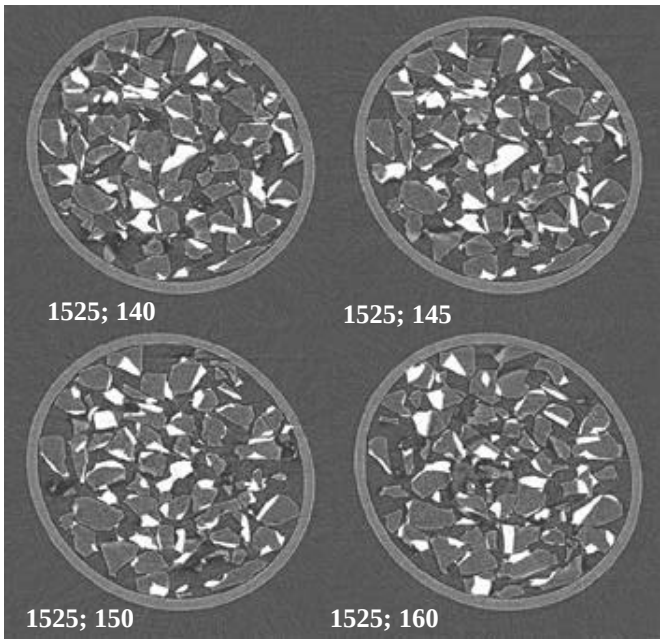

FIG. 1. X-ray images of particles taken at intervals of 33.5 µm (140, 145 and 150) and 67 µm (150 and 160). The sample is a mixture of 15 to 25% glass (bright phase) and 75 to 85% epoxy (dark phase). The outer diameter of the polyethylene tube (grey) is 3.7 mm.

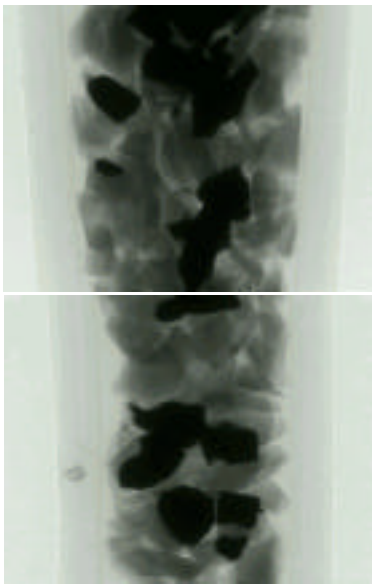

FIG. 2. Radiographs of lead borate (black) and glass (gray translucent) particles in a polyethylene tube. The width of the images is approximately 4 mm.

Radiographs from the sample containing mixtures made of lead borate and glass are shown in Fig. 2. Lead borate particles are highly absorbing at the energy used and appear black in the x-ray images.

Discussion

This study forms the first successful application of synchrotron-based microtomography to 3-D imaging of particles and to the determination of the volumetric proportion and liberation properties of particles.

Individual slices are being analyzed by an image analyzer to determine the quantities of individual phases and their liberation characteristics. The magnitude of variation resulting from image analysis of more than 500 slices will provide a statistical basis to evaluate routine liberation data and to derive a correction function.

Acknowledgments

Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38. Technical and administrative support from the staff of GeoSoilEnviroCARS (GSECARS), sector 13, is greatly appreciated.