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GSECARS

Sector 13

Materials Science

Microtomography: Polymer Blends, Environmental Waste Remediation, and Extremely Wide Field-of-View Imaging

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Introduction

Prior tomography runs on environmental waste samples, polymer blends, and the structure of eyes were provocative but inconclusive. Therefore, samples and data acquisition methods were reconfigured to optimize the yield of reliable data from the tomography experiment. The techniques include multiple X-ray energies for chemical distributions and multiple CCD frames for extremely large and high-resolution tomography.

Methods and Materials

Flame Retardants: Flame retardants in polymer blends provide both a interest testbed for 3D chemical analysis as well as insight into a relevant problem for the industry. In prior work, we developed the data acquisition and analysis procedure for determining the chemical distribution of a brominated aromatic flame retardant and its synergist.[1,2] In the current run, the sample complexity was increased with the addition of a more complicated flame retardant and multiple synergists. Images at seven X-ray energies were acquired for two samples with varying amounts of flame retardants. The data analysis methods developed earlier are being applied to generate compositional images. These samples are more complicated than the prior work because of the greater number of components.

Environmental Samples: Some hazardous wastes are encapsulated in cement, then buried in landfills. In prior work, the chemistry of phenols and brominated phenols was studied with solid-state NMR. The NMR data suggested a sequence of precipitation events within the cement matrix. Preliminary tomography at NSLS in 1998 suggested a distinct layer structure on the inner surfaces of waste-filled vescicles entrapped within the cement matrix. Unfortunately, those images were compromised by low resolution. In the APS work, fresh samples were imaged at 3.26 micron resolution. The tomography analysis will be compared with the NMR data to ascertain the order of precipitation and waste stabilization events.

Internal Structure of Eyes: Glaucoma is all too common, especially in an obese population such as found in Louisiana. The LSU Eye Medical Center maintains a large research project on eye diseases, including a monkey population prone to induced glaucoma. One area of glaucoma research is the relationship between internal eye pressure and distortion and distension of the sclera shell, the main structural component of the eye. With finite element methods and the known stress-strain of sclera, they hope to model the hypertensive eye. However, the model requires accurate sclera thicknesses which are known to vary from 100 to 400 microns throughout the sclera.

A hemispheric sclera shell of monkey eye was mounted on a polystyrene sphere and immersed in saline to avoid slumping and shrinkage. A continuing problem with x-ray imaging of eyes and eye structures is the small range of x-ray absorption. The sclera is barely visible relative to the polystyrene holder and the saline. A series of 2D images from 18 to 30 keV showed best contrast at the higher energies. A second problem with the eye is the wide range in size scales. The sclera diameter is 25 mm and needed thickness resolution about 10 microns. Therefore, multiple CCD frames were acquired and assembled into a large sinogram set. At 30 keV and 8.6 micron resolution, six frames were acquired with 100 pixel overlap to yield a field of view of 3700 x 1440 pixels, corresponding to 31.8 x 12.4 mm. At 24 keV and 17.2 micron resolution, three frames were acquired with 40 pixel overlap to yield a field of view of 1870 x 390 pixels, corresponding to 31.2 x 6.7 mm.

Results

Figure 1 shows histogram of voxel linear attenuation coefficients of one of flame retardants.

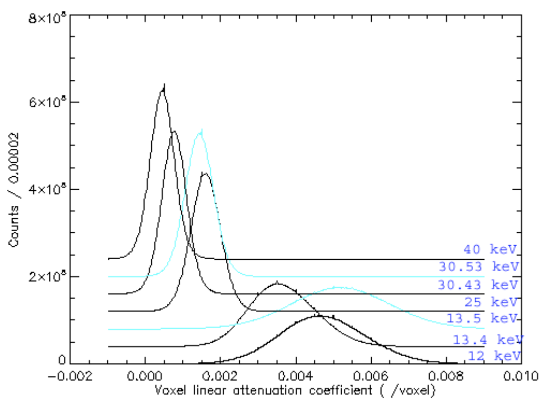

Fig1. Histogram of linear attenuation coefficients of a flame retardant at 7 different x-ray energy

Clearly shows the Br and Sb existence. 3 dimensional composition concenturation will be determined by computational fitting[1,2]. Data analysis was interrupted by a death in the family of the lead investigator. Analysis will resume in the near future.

Discussion

The flame retardant/polymer blend images will provide a critical test of the algorithm developed [1,2] to extract 3D chemical distributions. We foresee a broad range of applications for 3D chemical analysis, including time-resolved imaging (over months) to assess diffusional processes that affect materials properties but are otherwise extremely difficult to characterize. The environmental sample builds upon past work with tomography [3]. The eye sample expands methods for acquiring large field of view at high resolution, though the data processing steps are not yet completely resolved.

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References

[1] “Three-Dimensional Chemical Analysis: Synchrotron Tomography at Multiple X-ray Energies of Brominated Aromatic and Antimony Oxide Additives in Polystyrene.” Ham, K.; Jin, H.; Al-Raoush, R.; Xie, X.; Willson, C. S.; Byerly, G. R.; Simeral, L. S.; Rivers, M. L.; Kurtz, R. L.; Butler, L. G. *Chemistry of Materials.*, **2004**, *16*, 4032-42.

[2] "Algorithms for three-dimensional chemical analysis with multi-energy tomography data", K. Ham; C. S. Willson; M. L. Rivers; R. L. Kurtz; L. G. Butler, SPIE "Developments in X-Ray Tomography IV", **2004**, *5535*, 286-292.

[3] " Synchrotron X-ray microtomography, electron probe microanalysis, and NMR of toluene waste in cement", L. G. Butler; J. W. Owens; F. K. Cartledge; R. L. Kurtz; G. R. Byerly; A. J. Wales; P. L. Bryant; E. F. Emery; B. Dowd; X. G. Xie, *Environmental Science & Technology,* **2000**, *34*, 3269-3275.