*Acta Cryst.* (2002). A**58** (Supplement), C69

## PARALLEL BEAM POWDER DIFFRACTION IN THE LABORATORY FROM MULTILAYER AND CRYSTAL OPTICS

S.T. Misture M.R. Haluska S. Zdzieszynski
Alfred University Ceramic Engineering And Materials Science 2 Pines St. ALFRED NY 14802 USA

An empirical evaluation of data obtained using parallel beam geometry powder diffraction from laboratory sources is presented based on signal to noise ratios, count times, and Rietveld refinement of data from NIST standards. Two distinct geometries were used, Debye-Scherrer and reflection, as well as point detectors and a linear position-sensitive detector. The diffraction geometries used include: 1. Parabolic multilayer optic coupled into an asymmetric Si (220) channel-cut two-bounce crystal with a similar crystal on the receiving side. 2. Parabolic multilayer optic on the incident beam side and soller collimator on the receiving side. 3. Parabolic multilayer optic on the incident beam side and flat multilayer optic on the receiving side. 4. Parabolic multilayer optic on the incident side with a linear position-sensitive detector on the receiving side.
With respect to conventional Bragg-Brentano geometry, the parallel beam systems provide accurate lattice parameters and highly reliable peak locations. The peak widths as a function of angle can be modeled using Cagliotti parameters and show no significant problems. However, it is apparent from Rietveld refinement that the systems that incorporate multilayer optics without crystal optics provide data that is slightly different than that obtained using parafocusing systems. In particular, the peak tails and location of the α two components are not correctly modeled when using traditional approaches.

**Keywords: POWDER DIFFRACTION MULTILAYER OPTIC RIETVELD**

*Acta Cryst.* (2002). A**58** (Supplement), C69

## CHARACTERIZATION OF PHOTO-CATALYST BY LABORATORY XAFS

T. Taguchi[1] K. Tohji[2] K. Shinoda[2]
[1]RIGAKU Corporation XRD Division 3-9-12 Matsubara-Cho AKISHIMA TOKYO 196-8666 JAPAN [2]TOHOKU University

X-ray Absorption Fine Structure (XAFS) spectroscopy is one of the most powerful tool to determine the valence state of local atoms in various material. XAFS spectroscopy has an advantage in providing the structural information of amorphous, liquid or catalyst, which are difficult to characterize by the ordinary powder diffraction technique. Results from XAFS spectroscopy are commonly reported using synchrotron radiation facilities, but result obtained using a laboratory XAFS apparatus has rarely been reported. The shortage of data obtained from a laboratory XAFS apparatus has been mainly due to its complicated mechanism. We have recently developed a new laboratory XAFS apparatus, which is compact in size and easy to use.
The new laboratory XAFS apparatus features a newly developed compact 'demountable' X-ray source, dedicated vertical goniometer and easy control software. The vertical goniometer design allows an easy access to optical components and the sample, and each component is placed at equal distances from the operator. In the newly developed system, the sample position is fixed, while the X-ray source and the monochromator moves around. So the position and the direction in which monochromatic X-rays is fixed. Therefore experimenter can put an *in-situ* cell without concerning about wiring and plumbing. We have characterized a photo-catalyst (very fine ZnS nanoparticle) using this apparatus.

**Keywords**: **XAFS IN-LABORATORY PHOTO-CATALYST**

*Acta Cryst.* (2002). A**58** (Supplement), C69

## ADVANCES IN X-RAY SOURCE TECHNOLOGY FOR DIFFRACTION APPLICATIONS

T. A. Thorson[1] R. D. Durst[1] S. I. Foundling[1] G. J. Kerpershoek[2] A. B. Storm[2]
[1]Bruker AXS Inc [2]Bruker Nonius B. V.

Advances in X-ray source technology provide additional capabilities to the laboratory crystallographer. The use of shorter acquisition times, larger area detectors, data from fainter reflections or from diffuse scattering can all be enhanced with brighter sources. In addition, the lifetime of the source (limited by filament or tube life) is also critical to high-throughput, unattended systems and can disrupt the data collection schedule for the laboratory. Limitations of present laboratory X-ray source brightness are due to constraints of the power deposition on the anode or from the electron emission characteristics of the cathode. For example, heating of the anode leads to localized melting or evaporation of the anode material, and the resulting roughening of the anode surface degrades the X-ray output performance, especially at low takeoff angles. Limitations of filament life are strongly related to the cathode design, vacuum conditions, and the power at which the source is operated. We review some of these limitations and discuss recent developments that improve these aspects and better utilize the brightness of the source. For example, by improving the uniformity of the electron distribution on the anode, a higher brightness can be achieved due to a larger amount of allowed power deposition on the anode. The resulting increase in performance from these advances will be compared against presently available laboratory sources, including sealed tubes, rotating anodes, and microsources.

**Keywords**: **X-RAY SOURCE, ROTATING ANODE GENERATOR, SEALED TUBE GENERATOR**

*Acta Cryst.* (2002). A**58** (Supplement), C69

## A NIGMS EAST COAST STRUCTURAL BIOLOGY FACILITY

M. Allaire L.E. Berman E.D. Johnson D.P. Siddons Z. Yin V. Stojanoff
Brookhaven National Laboratory National Synchrotron Light Source Bldg 725D UPTON NEW YORK 11786 USA

A new bio-molecular crystallography facility has been built at the national synchrotron light source (NSLS) to provide structural biologists an easy access to structure determination. The facility will serve expert and non-expert crystallographers from protein purification to atomic coordinates. This facility consists of beamline x6a and an associated laboratory. X6a was designed to facilitate multi-wavelength diffraction data acquisition and processing. A Si(111) monochromator NSLS design and an Oxford Danfysik focusing mirror are part of the basic optical elements. The end station includes a Crystallogic κ-diffractometer, an ADSC 210 ccd detector and an Oxford Cryosystem series 700 nitrogen jet. Open source software is available to users for data analysis. In the laboratory users are able to refine their purification protocols and crystallization parameters. A pilot program to facilitate access to the research infrastructure of this facility is under implementation. This program will provide assistance for long term stays at the NSLS for training and scientific collaborations.
This work is supported by the National Institute of General Medicine and Science under contract #40-04-12-000 and the Department of Energy under contract #DE-AC02-98CH10886.

**Keywords**: **CRYSTALLOGRAPHY, SYNCHROTRON RADIATION, BIOLOGY**