temperatures is thought to be from photoelectrons ejected from atoms following the absorption of X-rays. As the photoelectrons traverse the crystal, they lose energy through interactions with atoms in their path resulting in damage. If the X-ray beam is polarized, the photoelectrons are ejected preferentially along the polarization vector. Monte-Carlo simulations [Nave, C., and Hill, M. A. (2005). J Syn. Rad. 12, 299-303] suggest that, when the beam size is only a few microns, most photoelectrons escape the illuminated volume. This leads to the peculiar conclusion that the radiation damage due to photoelectrons may be significantly lower within the illuminated volume than in the volume immediately surrounding the irradiated spot. A second prediction of the calculations is that most of the photoelectron's energy is abruptly dissipated within the last few microns of its trajectory. Recently, a long focal length Fresnel zone plate was used to provide a focused beam of ~1-micron cross section at the sample position, and high quality diffraction data was obtained from protein crystals. The 15.1 keV, 1-micron beam was used to probe the geometrical distribution and extent of radiation damage in protein crystals. These data confirm that radiation damage is greater along the polarization vector than in the perpendicular direction, radiation damage is maximal 3-4 microns from the center of the beam, and radiation damage does not extend beyond 6 microns.

Keywords: radiation damage, microcrystalls, microcrystallography

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Towards protein structure determination using two-dimensional crystals and powders

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Reliable determination of the structure of integral cell membrane proteins (IMPs) is one of the most important problems in biology today. The structure of the vast majority of IMPs remains unsolved however, mainly due to difficulties associated with conventional crystallisation and the concomitant need for preservation of the active form of the protein as it is taken out of its natural environment within the cell membrane. In order to address these problems, we consider the possibility of using two-dimensional (2D) ordered micro-arrays of proteins, i.e., 2D crystals, in X-ray diffraction (XRD) experiments, instead of conventional three-dimensional crystals. In this work, we discuss the potentials and limitations of using 2D protein crystals for XRD based structure determination. We present a systematic approach to data analysis and fitting based on physical description of X-ray scattering by 2D crystals. Scattering by large assemblies of 2D crystals with random preferential orientations is also considered as a model for XRD with 2D crystal powders. We illustrate how 2D crystal powder diffraction data may be used to reconstruct a 2D projection map of the electron density in the unit cell with reference to preliminary results obtained for 2D crystals of bacteriorhodopsin.

Keywords: membrane proteins, two-dimensional protein crystals, X-ray diffraction

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8C2 high resolution powder diffraction beamline at Pohang Light Source and its recent results

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We introduce 8C2 high resolution powder diffraction (HRPD) beamline at Pohang Light Source. This beamline is designed for a powder crystallography, i.e., very high angular resolution and various sample environments. The technical characteristics of the beamline and some performance indicators are listed, such as the incoming photon flux and the angular/energy resolutions obtainable under typical experimental conditions. We present several recent results using synchrotron x-ray powder diffraction data collected from this beamline, not detected by previous powder diffraction experiments.

Keywords: powder crystallography, powder diffraction, synchrotron X-rays

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Accurate powder diffraction standards: Determination of the lattice parameter of LaB_6 SRM(660)

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We use X-ray powder diffraction and synchrotron radiation to determine the lattice parameter of the NIST standard reference material (SRM 660) LaB_6 to be 4.157580 Å with an accuracy of 12 parts per million (ppm), calibrated relative to the lattice parameter of the Si powder standard (a0 =5.430940(11) Å, Si 640b). A discrepancy is observed between the currently accepted lattice spacing of LaB6 and the measured value, of 0.00048(5) Å, or nine standard deviations from the NIST reference. Twelve different measurements of the lattice parameter are made at beam energies between 10 keV and 20 keV. The observed discrepancy in the lattice parameter is consistent for the different energies used. The absolute values of the mean difference between the measured and calculated 2 theta centroids, are highly consistent, between 0.0002o and 0.0004o for energies from 5 keV to 14 keV, and between 0.0005o and 0.0008o for energies from 15 keVto 20 keV. In order to determine the peak positions with high precision, account must be taken of observed peak asymmetry. Significant asymmetry is due to peak broadening and must be taken into account in order to determine accurate peak locations and lattice spacings. Our approach shows significant advantages over conventional analysis. Our analysis of peak broadening is compared with models used in Rietveld analysis.

Keywords: lattice parameters, powder and single crystal diffraction, synchrotron radiation experimental

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A halogen lamp furnace to synthesize nanoparticles: *In situ* X-ray absorption spectroscopy

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A compact new furnace, based on a halogen lamp as heater element, and a sample-cell were designed and constructed initially for in situ X-ray absorption spectroscopy (XAS) experiment in conventional and dispersive mode (transmission and fluorescence geometries). The main application of the apparatus is the thermal treatment studies under controlled conditions for dynamical processes up to 1000 K, with and without gas flux. As example, we have utilized the sol-gel (gelatin) method to synthesize NiO nanoparticles [1,2]. During this heating process, *in situ* (Ni K-edge) X-ray absorption near edge structure (XANES) measurements provided evidence of the evolution of Ni environment until the complete NiO nanoparticle crystallization. These results revealed a sequence of phase transformation during the heat treatment, starting from amorphous to NiO crystalline phase, and confirm the capability of XAS techniques to follow the early stage of crystallization of NiO nanoparticles. Also, we have noticed that the particle growth is dependent of carbon matrix concentration, which is produced during the calcinations and act as barrier. All apparatus were developed for experiments at the D06A-DXAS and D04B-XAFS beamlines of the Laboratorio Nacional de Luz Sincrotron (LNLS), Campinas, Brazil.
Acknowledgments: CNPq and LNLS.
References:
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Keywords: furnaces, X-ray and synchrotron radiation instrumentation, X-ray absorption spectroscopy

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Vacuum-ultraviolet circular dichroism of amino acid films by polarizing-undulator based system

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We have developed the beamline for vacuum ultraviolet circular dichroism (VUV-CD) measurement in the storage ring TERAS BL-5 at AIST, Tsukuba, Japan using a the four-period Onuki-type crossed undulator as an insertion device. CD measurement has been widely used for analyzing protein structures and obtaining the structural information of chiral molecules. Extension of wavelength coverage of CD measurement to VUV region gives much structural information. Since natural CD is known to be weak signal, high-sensitive AC modulation spectroscopic method is required for accurate measurement. Our undulator can modulate the circular and linear polarization relatively high frequencies, up to 5 Hz, sufficient for AC modulation spectroscopy. Using this undulator as a polarization light source, we have succeeded in measuring the VUV-CD spectra of alanine films down to 120 nm [1]. In this work, we will present the recent status of our VUV-CD measurement system and results of amino acid films. CD spectra of major aliphatic amino acid films in UV region exhibit only one peak and show the almost same spectral feature. In contrast, VUV-CD spectra of these films show clear difference corresponding to the variation of side chain. These spectra also show the difference in comparison with those of aqueous solutions. This difference implies that the molecular structures of amino acids are strongly dependent on their states. The results of theoretical calculation with TD-DFT method are reasonably consistent with those of experiment. It is therefore reasonable that the change of molecular structure can be predicted from the CD study.
[1] K. Yagi-Watanabe, et al., Rev. Sci. Instrum. 78, 123106 (2007).

Keywords: circular dichroism measurement methods, amino acids, vacuum ultraviolet

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Bringing the power of synchrotron crystallography to the chemical community

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Single-crystal diffraction with synchrotron radiation greatly extends the capabilities of this technique to very small crystals and other weak X-ray scatterers, but many potential beneficiaries are prevented from taking advantage of it by the special expertise it seems to demand, in addition to formidable administrative and organisational barriers and the long time between proposals and experiments at most facilities. The facilities at Daresbury SRS, first established over 10 years ago in station 9.8 and substantially developed since then, were designed with the needs of mainstream chemical users in mind, and similar considerations have been involved in the planning for replacement facilities at Diamond (beamline I19), due for first operation this year. One important development has been the introduction of a synchrotron component of the UK National Crystallography Service, available free of charge to academic users after peer review of proposed chemistry research projects. Since 2001 this has provided convenient, efficient and rapid access for chemists without crystallographic training, through screening of samples with a high-intensity rotating-anode source and then data collection by a dedicated expert team, providing either datasets for processing by the end-users or full structure determination. Over 1000 datasets have been measured, leading to many excellent results and publications. The service moves to Diamond in 2008, and provides a model for other central facilities, and possibly for other experimental techniques. Service management and operation will be discussed, together with a selection of key results.

Keywords: synchrotron radiation applications, chemical crystallography, service crystallography

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X-ray focusing by using compound refractive lens optimized for high-pressure XRD at SPring-8

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