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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.
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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.
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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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X-ray focusing technique is essential for probing microscopic samples at high pressures. Compound refractive lens (CRL) is one of the most versatile devices that are capable of focusing and collimating x-rays in the high-energy range of 20－60 keV [1, 2]. The CRL offers many advantages: good efficiency for focusing, compactness, robustness, and easy alignment (in-line focusing element). At high-pressure x-ray diffraction beamline BL10XU of SPring-8, the x-ray focusing optics consisting two different types of CRL devices have been installed in tandem. The first CRL (16 m focal distance) made from glassy carbon (GC), which is situated at a distance of 42 m from the source, has been used incidentally to collimate the x-ray beam. The aperture of this CRL is about 1 mm, whose size matches the beam size of the undulator. Because of difficulty in focusing the x-ray beam size down to 10μm through the first CRL, a second focusing CRL device was placed at 0.5 m upstream to the sample. The second CRL is sets of planar cross-lenses with a quasi-parabolic profile for focusing in two directions, and were fabricated from SU-8 polymer by deep x-ray lithography at the ANKA in Germany [3]. The placement of the SU-8 CRL after the GC-CRL produces an x-ray beam with a spot size of less than 7 μm at 30 keV. The photon flux density at the focal point is approximately 10^{15} photons/sec/mm^2. This x-ray optics allow us to collect high-quality x-ray diffraction data on materials subjected to extreme pressures of up to 400 GPa, which exceeds the condition found at the Earth's center.

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Keywords: high-pressure X-ray diffraction, X-ray focusing optics, synchrotron radiation optics

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Total reflection and multilayer optics for synchrotrons and free-electron lasers

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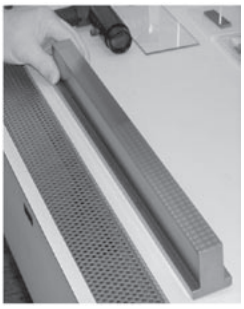
We will be presenting selected aspects of simulation, preparation and characterization of total reflection and multilayer X-ray optics. The optimum coating is found by calculating the optical properties. Sophisticated improvements in manufacturing technology allow the precise realization of the specified parameters. Our capabilities for the production of multi stripe optics as well as for very long mirrors will be shown. Two applications demonstrate our possibilities: First of all we will be showing results of the development of carbon coatings as total reflection mirrors for Free-Electron Lasers. Over the past years, we have developed optimized optics for the XUV range up to 200 eV. The investigations have shown that carbon coatings offer high reflectivity >95%, high radiation stability and good uniformity both in thickness and roughness. Secondly, we will be presenting some results of the production of multi stripe optics for the use at different energies. One example shows a three-stripe optics for a tomography beamline. A Ru/C multilayer was choosen for energies of 10-22 keV, a W/Si multilayer for energies of 22-45 keV and one stripe of the (111)-Si substrate remained uncoated.



Keywords: synchrotron optics, multilayer optics, total Reflection Optics

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Center of mass grain maps in 3D

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We would like to visualise the microstructure of crystalline materials. Diffraction based mapping techniques [1] offer a way to determine the position, orientation, size and strain state of multiple crystallites within a sample. Mixtures of different crystalline phases can also be treated, as each crystallite is indexed and fitted separately. Multiple crystallites can be indexed from a single dataset to give an orientation matrix for each one. Any small misalignment of each crystallite with respect to the diffractometer axis gives systematic shifts in the Bragg peak positions during rotation. The area detector geometry and axis direction can be fitted together with the positions of the crystallites using the ImageD11 software package [2]. The position, orientation and lattice parameters of each indexed crystallite are normally refined. Peak intensities give the volume of each grain that has been illuminated by the beam. We will show several examples of maps produced using high energy x-ray diffraction data collected on an area detector at beamline ID11 at the ESRF. Spatial resolutions for the center of mass grain position are typically somewhat better than the detector pixel size (50 micron) and cover an area as large as the beam size (up to 2 mm). Maps containing several hundred grains have been produced.

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Keywords: instrumentation and software, area detectors, crystallites

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Grazing-angle incidence hard X-ray nanoscope

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We consider in presented theoretical paper a possibility to overcome the limitations of the x-ray microscope and increase its resolution from tenth a micrometer to a nanometre or less. Proposed nanoscope would allow studies of the nanoscale structures. We foresee the