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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.

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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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