BL practice

BL17SU : Site-selective Observations of the Electronic States for Solid and Liquid Phase Molecules by Means of Soft X-ray Emission Spectroscopy

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1. Introduction

Knowledge of an electronic structure is very important for understanding the properties of materials. Occupied states in valence band of the materials have been commonly investigated by means of x-ray or soft x-ray photoemission spectroscopy (PES). An ordinary PES is, however, unsuited for studying the electronic structure of materials in liquid phase. As an alternative method to study the electronic structure of valence band of liquid sample, the x-ray emission (XE) spectroscopy in soft x-ray region has been developed in this decade in order to promote the spectroscopic study for liquids [1-3].

In the course, the participants will learn a principle of the XE spectrometer as well as the liquid flow-cell, and gain experience in soft x-ray absorption and emission spectroscopies for the typical samples, such as acetate in aqueous solution and in solid salt.

2. Outline of the soft x-ray undulator beamline BL17SU and the experimental apparatus

Soft x-ray beamline BL17SU [4] is constructed to advance the spectroscopic studies for mainly solid state physics and materials science using high brilliant soft x-ray undulator radiation. Photoabsorption, photoemission and XE spectroscopies in soft x-ray region are adopted to investigate the electronic structure of various kinds of materials. A novel insertion device ID17, called a multi- polarization-mode undulator, has been developed for BL17SU [5], and ID17 can be operated as a helical, elliptical, pseudo-linear or pseudo-vertical undulator. The intense soft x-ray beam of circularly / linearly polarized light or the mixed polarization state of the beam can thus be obtained by changing the operational mode of ID17. The BL has branched beamlines (branch-a and -b) which can be switched by the pre-focusing mirrors and used alternatively. Each branch has high-resolution and highly stabilized monochromator [4,6] and several end-stations. The available energy is ranged between 300 and 1800 eV. The resolving power $E/\Delta E$ of the monochromator is achievable to be higher than 10,000 and the photon flux is of the order of 10^{11} photons/s.

At A3 station of the branch-a, High Efficiency Photon “energy” Analyzer (HEPA) [7], which is aimed at advancing site-selective observations of the electronic states for liquid phase molecules by means of the XE spectroscopy in soft x-ray region [3,8-12], is installed and we will mainly utilize this instruments for the XE spectroscopy in this course. The apparatus consists of the XE spectrometer and the main chamber equipped with the compact flange-mount liquid flow-cell.

3. Experimental procedure

3.1 Preparation of the powdered sample

In the beginning of the course, the participants will be asked to prepare the powdered sample of acetate in solid salt. The sample will be supported by the carbon tape sticked on the sample holder.

3.2 Explanation of the beamline and the SXE spectroscopy

During the evacuation of the chamber, the whole of the beamline as well as the principles of the XE spectrometer will be explained to the participants.

3.3 Calibration of the beamline monochromator

After explanation, calibration of the beamline monochromator will be performed by using a hemispherical electron energy analyzer (SES-2002, VG-SCIENTA) installed at the different station. The kinetic energy spectrum of Au 4f electrons will be measured as a function of the exciting photon energy in order to establish the relationship between the wavelength of the exciting photons and the setup of the beamline monochromator.

3.4 X-ray absorption (XA) and XE measurements for the powdered sample

An XA spectrum of the powdered sample in near O 1s-edge region will be measured by means of the total photon yield (TPY) method. After the XA spectrum measurement, calibration of the spectrometer will be carried out by measuring the elastically scattered photons from the sample. Resonant photon-in－photon-out measurements will be performed at some energy points where the XA spectrum shows characteristic features.

3.5 Change the setup of sample chamber for the measurement of liquid sample

When the XE measurements for the powdered sample is finished, the participants will be asked to change the setup of sample chamber for the liquid sample measurement. During the evacuation of the chamber, a preparation of the liquid sample will be carried out.

3.6 XA and XE measurements for the liquid sample

The spectrometer will be adjusted for the measurements of the liquid sample prior to the sample measurement. When the spectrometer is ready for the measurement, an XA spectrum of the liquid sample in near O 1s-edge region will be performed by means of the TPY method. Resonant photon-in－photon-out measurements will be performed at some energy points as in the case of the powdered sample.

3.7 Data analysis and discussions

When all the measurements are finished, the spectrum for each sample will be analyzed and compared. The participants will be able to compare the results of the XE measurements for solid and liquid samples.

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