

Physico-Chemical Analysis Beamline

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1. Introduction

Low emittance storage ring such as SPring-8 is excellent in both brilliance and polarization property. We aim for making practical use of the advantages with linearly or circularly polarized x-rays. For the purpose a combination of linear undulator and phase retarder is suitable because of the high-brilliant and high-rate polarized x-rays. Such a specification will be realized at XU4 beamline in the photon energy range from 5 to 20 keV. It is difficult to make this achievement without the 3rd generation synchrotron radiation.

The XU4 beamline is constructed in collaboration among the subgroups of "X-ray magnetic absorption and scattering", "Spectrochemical analysis", and "Medical applications". This beam-line will offer much opportunities to advance physical and chemical researches in material and biological sciences. In particular the correlation between structure and function is a basic issue in this field. For magnetic materials the well-defined polarization states are crucial to make both diffractometry and spectroscopy. High-spatial resolution is also indispensable for x-ray fluorescence analysis with microprobe. Combination of these techniques will open up new applications to the material and biological sciences. The layout of XU4 beamline is shown in Fig. 1. The beamline is mainly composed of a linear undulator, a standard rotated-inclined double-

crystal monochromator, a Pt-coated plane mirror, and a phase retarder. The dimensions of the experimental hutch are 4 m in width, 6 m in length and 4 m in height. A phase retardation system is placed at the most up-stream in the hutch.

2. Insertion Device and Optics

For the XU4 beamline an in-vacuum linear undulator (U032V) is installed at the section of BL-39IN. Its design has been reported in detail in ref.1. Major parameters of the device are $\lambda_u = 3.2$ cm, $N_{\text{period}} = 140$, and power density 470 kW/mrad² at 5 keV. The calculated brilliance spectrum [2] of the radiation is shown in Fig. 2. The peak brilliance exceeding 1×10^{19} phs/sec/mrad²/mm²/0.1%b.w. at 100 mA can be achieved up to 30 keV with higher harmonics of the undulator radiation. The x-rays with degree of linear polarization $P_l \sim 1$ on-axis are available not only direct uses but also conversion into circular polarization.

Monochromatized x-ray is led to a quarter wave plate following the rotation of its polarization plane from horizontal to 45° inclined. Phase retardation system using a single crystal of synthetic diamond can efficiently convert a linearly polarized incident x-ray into a circularly polarized one. This has fine advantages of high- degree circular polarization ($P_c \sim 1$), fast switching of helicity (<100 Hz), and conversion into vertical linear polarization using a half wave plate [3,4]. These features are very practical for spectroscopy and diffractometry in polarization modulation mode because of an improvement of data quality and flexibility according to the experimental conditions. In particular the alternate polarization, *i.e.* between horizontal and vertical linear or left and

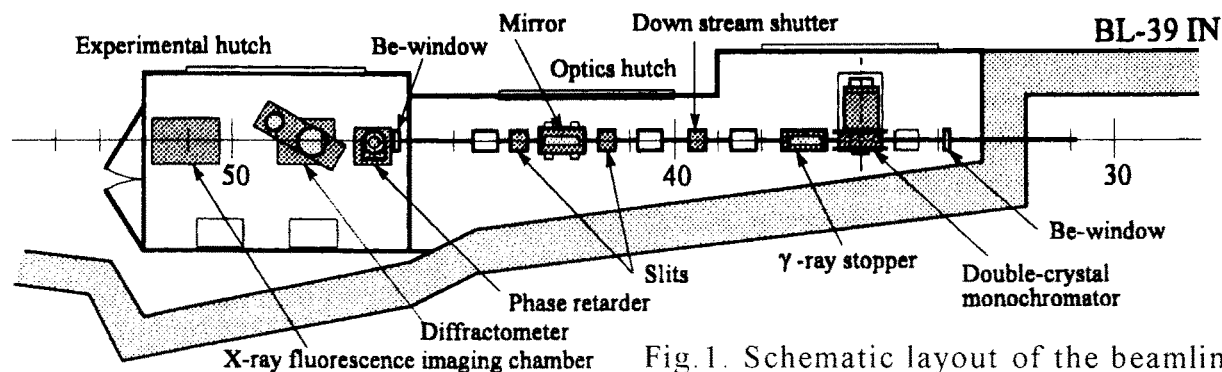


Fig.1. Schematic layout of the beamline

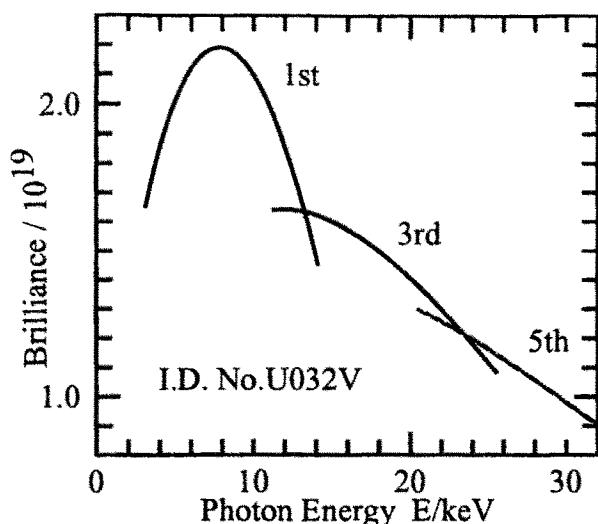


Fig.2. Calculated brilliance spectrum of the radiation emitted from the undulator.

right circular polarizations, stimulates interest in new applications by spectroscopy. A fine energy resolution as well as the stability in polarization is essential to our researches.

3. Experimental Station

We present here several experimental instruments proposed by the subgroups. The apparatus is relocatable and can be adjusted to the beam position. The operation will be made by time-sharing among the collaborating subgroups.

3-1. X-ray magnetic diffractometry and spectroscopy

Diffractometry has been preferred to spectroscopy because of the instrumental complication of movable part. A 2-circle diffractometer, which is equipped with another 4-circle diffractometer as an analyzer on the 2θ -arm made of Al-honeycomb, will be installed in the end station. The diffractometer was designed for experiments under the magnetic fields generated by an electromagnet or a superconducting magnet in future. The configuration of goniometer axes is schematically shown in Fig.3. An additional ω -axis is used for a fluorescent detector or the sample to rotate independently of the magnet. The scattered x-rays are analyzed by decomposing into the σ -

and π -polarization components using the 4-circle diffractometer.

Some experimental accessories such as a superconducting magnet up to 12 Tesla, a diamond anvil cell up to 10 GPa for the experiments under extreme conditions, a 2-dimensional detector, *e.g.* CCD, and a photoelectron detecting device will be equipped in future.

3-2. X-ray fluorescence spectroscopy

X-ray focusing optics with total reflection mirrors is a fundamental technique for x-ray spectroscopy with spatial resolution. A paraboloidal mirror is ideal for a source of finite size whenever the divergence is negligible. Considering the beam divergence of the undulator radiation at XU4, the beam size of 1 μm in diameter is obtainable at the focal plane. The spatial resolution of less than 1 μm will be achievable with the further collimation of incident x-ray beam.

Figure 4 shows an x-ray fluorescence imaging system. A sample is mounted on an xyz and rotational stage inside a chamber. Various kinds of x-ray image will be obtained simultaneously by collecting transmitted and fluorescent x-rays, diffracted x-rays, and optical luminescence. A wavelength dispersive spectrometer is also designed, which is composed of a flat analyzer crystal to disperse the fluorescent x-rays and a position sensitive detector (PSD). The chamber and the optical path can be evacuated (or He filled) to be accessible to the lower energy region down to 3 keV.

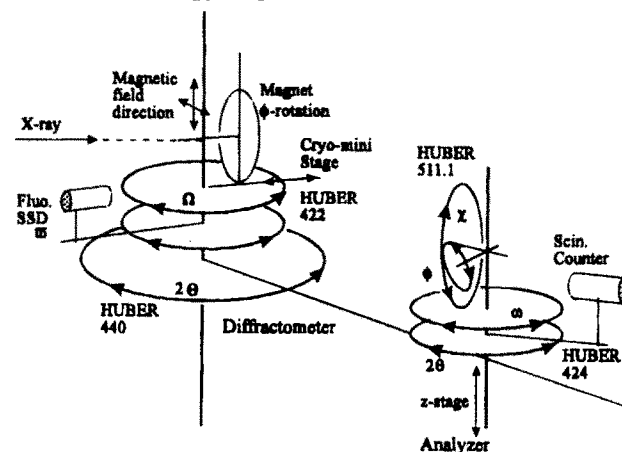


Fig.3. Goniometer axes configuration for magnetic diffractometry and spectroscopy.

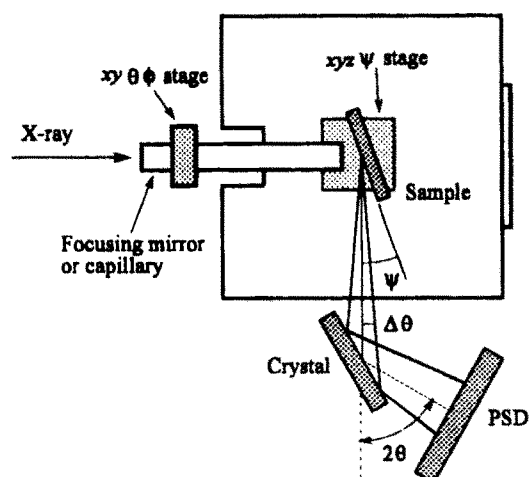


Fig.4. X-ray fluorescence imaging chamber with a wavelength dispersive spectrometer.

4. Research Subjects

4-1. X-ray magnetic absorption and scattering

Our research subjects are classified into the three categories: (1) non-resonant scattering, e.g. x-ray magnetic Bragg scattering, and x-ray magnetic diffraction, (2) resonant scattering, and (3) magnetic absorption, e.g. magnetic circular x-ray dichroism, magnetic Raman scattering, and spectroscopy under standing wave condition. The well-defined polarization states are prerequisite for both the diffraction and the spectroscopy, so that the phase retardation system plays an essential role for our subjects. Moreover, the polarization analysis of the scattered x-rays is crucial to decomposition into orbital and spin components. Such experiments under extreme conditions are promoted for studying modification in magnetic structure and magnetic states.

4-2. Spectrochemical analysis

Ultra trace elemental analysis is made using the wavelength dispersive spectrometer system within the minimum detection less than 100 ppb, which corresponds to 1 fg or 10^7 atoms in absolute amount. Threshold x-ray fluorescent spectroscopy around absorption edge is used for analyzing chemical states through peak shifts and profile changes. The brilliant undulator radiation of XU4 is attractive for measuring the spectral changes. Mul-

tiple ionization effect and resonant phenomena will be experimentally derived. Trace chemical characterization of liquid drop is also made using x-ray fluorescence measurement under total reflection condition with grazing incidence technique. This can provide the information on chemical states of liquid less than 10 microliter in volume.

4-3. Medical application

We will establish a new technique in cytochemistry by using a combination of SR x-ray fluorescence imaging and histopathological staining. Trace element distributions in tissues and cells will be clarified by nondestructive x-ray microbeam analysis. Fluorescence XAFS analysis of trace mercury will be used to clarify the mechanism of interaction between mercury and metalloprotein. Total reflection x-ray fluorescence technique is suitable for ultra trace element analysis of biological samples in extremely small amount. The high brilliant x-rays of XU4 are prerequisite for our research objectives.

References

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