JAERI Material Science II (BL11XU)

1. Introduction

The third beamline of JAERI, BL11XU, was constructed to study material sciences using synchrotron radiation from an undulator source at the end of 1998.

In BL11XU, one optics hutch and three experimental hutches are prepared for any kind of scientific programs. There are two "multipurpose spaces" for installing mirrors, differential pumping systems, and other experimental apparatuses for use in future developments in the optics hutch. We planned four mainly scientific programs of inelastic scattering and microscopic spectroscopy by means of the nuclear resonant scattering, high-pressure high-temperature studies with a cubic type multi-anvil press, that is called "SMAP180", inelastic scattering experiments with a high energy resolution diffractometer, and in-situ studies on the growing surfaces of III-V group semiconductors with the (4+2) type diffractometer connected to the MBE system.

The commissioning of the beamline including detailed monochromator adjustment was finished by February 1999. Then, we began the commissioning of experimental apparatuses for nuclear resonant scattering from the spring of the same year. Now we are studying scientific programs. Details of the beamline characteristics and beamline commissioning have been reported elsewhere [1-4].

2. Diamond Monochromator

Until the end of July 1999, silicon crystals which was a standard pin-post type double crystal monochromator for the undulator source, was employed as a monochromatization device, and provided us with good performance. But we decided to change the X-ray optical device from silicon crystal to diamond crystal. Because spatial divergence is smaller than silicon, and it is easy to treat a monochromator. Moreover, diamond transmits most X-rays without contributing diffraction. Then the heat load on the crystal surface is small and it is possible to use an indirect water-cooling system.

We installed the diamond double-crystal monochromator in Bragg geometry at BL11XU. This is the first use of Bragg geometry of diamond crystals at SPring-8. The size of the diamond used is 8.6 mm $\times$ 3.5 mm $\times$ 0.35 mm (as a first crystal) and 10 mm $\times$ 4.7 mm $\times$ 0.39 mm (as a second crystal). Figure 1 shows the picture of the water-cooled copper block as a crystal holder. The crystal is mounted on a copper block with Ga/In by touching the both sides of the diamond crystal with thin copper plate. Figure 2 shows the double-crystal rocking curve FWHM (full width at half maximum) of diamond (111) with respect to the photon energy. The calculation is by $\sqrt{2}$ times the width of the diamond (111) intrinsic width. As shown in Fig. 2, the measured widths are larger than the calculation width. This discrepancy might be due to the distortion and the mosaic spread of the diamond crystals.

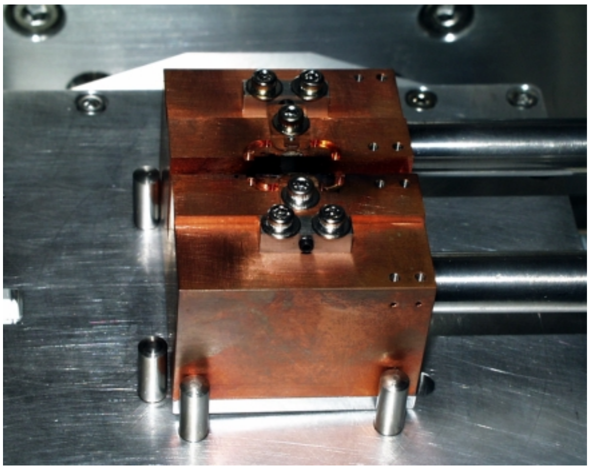

Fig. 1. A picture of the water-cooled copper block.

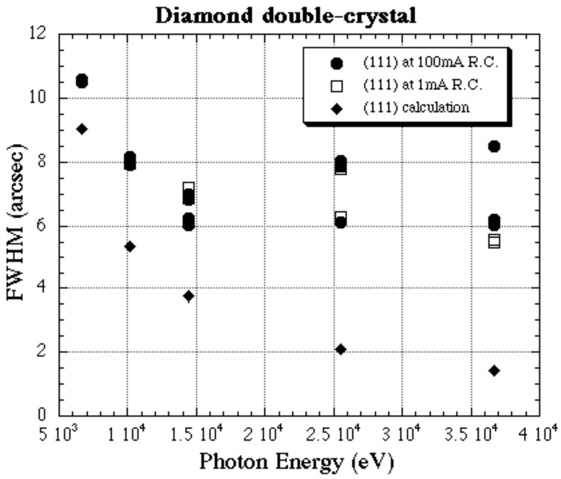

Fig. 2. FWHM of the diamond (111) double-crystal rocking curve as a function of energy.

3. Current Status of Experimental Hutches

3.1 Mössbauer Spectroscopy

In the experimental hutch 1, the photon and the electronic state of various materials has been investigated using synchrotron Mössbauer spectroscopy. From our recent researches, the existence of the local vibration mode of the dilute Fe atom in the Al and Cu matrixes was proved clearly [5]. The speed up effects of the time spectrum were studied in relation to the magnetic phase transition, too [6]. In addition, we looked for the possibility of the high-energy (over 25 keV) synchrotron Mössbauer spectroscopy. This type experiments become possible with the high energy X-ray of the SPring-8. As an example, the energy spectrum of the nuclear excitation of ^{40}K is shown in Fig. 3 [7]. The K atom is one of the most important elements in material and biological sciences. Usually, the measurement of the Mössbauer spectrum of ^{40}K is very difficult, because this atom can not be populated by any radioactive parent nuclide. But, Fig. 3 indicates the possibility of easy measurement of the Mössbauer spectrum of by using the third generation synchrotron radiation. In BL11XU, we have already been successful in the nuclear excitation of some atom [8].

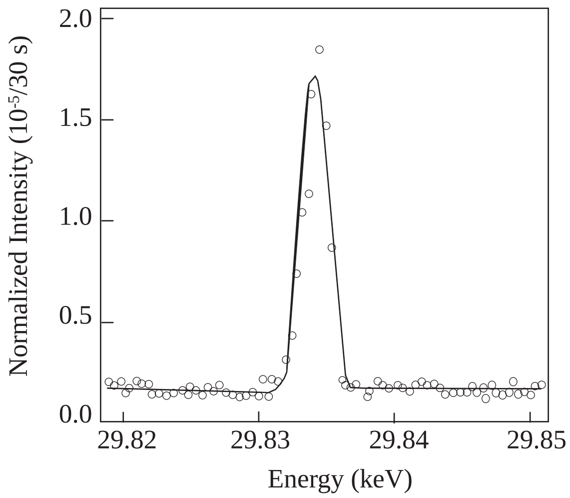

Fig. 3. Eenrgy spectrum of delayed emission from ^{40}K in KCl.

3.2 High-pressure High-temperature Study

For high-pressure high-temperature studies, a cubic-type multi-anvil press, SMAP180, has been moved from BL14B1 to the experimental hutch 1 of BL11XU. The accessible pressure and temperature ranges are up to 15 GPa and 1500 K, respectively. With this apparatus, we started two kinds of experiments: one is angle-dispersive X-ray diffraction (ADX) measurement and the other is density measurement.

In X-ray diffraction measurements using a MAP, a sharp collimator is necessary to eliminate diffraction from the materials surrounding the sample. An energy-dispersive X-ray diffraction (EDX) method is conventionally used in this situation, because it is highly efficient in data acquisition. With EDX, however, it needs some corrections to obtain accurate diffraction intensity. These corrections cannot be determined theoretically and empirical methods introduce uncertainty. The ADX method is desirable for accurate intensity measurement. The problem in ADX is its long data acquisition time with a single collimator: the typical measuring time is about 100 times longer than the EDX method. Bright X-rays from an undulator source greatly reduce measuring time and make the ADX method practical. In addition, we installed a radial slits system to reduce the measuring time. It consists of multiple channels of collimators in a radial alignment [9]. We developed a method to align the system and tested its performance. The result was satisfactory.

We have developed a method for density measurements under high-temperature and high-pressure by means of X-ray absorption [10]. Brilliant X-ray beams are necessary, because the accuracy of the density data depends on how accurately one can measure the absorption profile of a small sample, typically 1 mm in diameter. The preliminary results indicated that the measurements were accurate enough to discuss density change upon liquid-liquid transition.

3.3 Inelastic X-ray Scattering

The experimental hutch 2 is dedicated to inelastic X-ray scattering measurements. The typical energy resolution is 0.1 ~ 1 eV, thus the main target is electron excitations in solids, especially using resonant scattering technique. A Rowland X-ray spectrometer with a radius of 2 m including a bent Ge crystal analyzer is to be installed at the beginning of 2000.

3.4 Surface Science

The third experimental station is designed for in-situ studies on the growing surfaces of III-V group semiconductors. An X-ray diffractometer connected with an MBE system has been installed on this station since 1999. Machine commissioning using X-rays is planned for 2000.

The diffractometer is based on the (4+2) type, which has four axes for orientating the sample, and two axes for positioning the detector. In addition, this diffractometer has the axis for rotating the receiving slit about the normal of the slit plane[11]. The sample is connected to the ϕ-circle via an XYZ-stage, by which the sample surface can be positioned exactly at the center of the rotations. The whole setup is mounted on a table, which is capable of two translations and a tilting motion for the alignment with respect to the incident X-ray beam.

The MBE chamber is equipped with five Knudsen cells, a RHEED system and a sample-loading chamber. X-rays enter and leave the chamber through two cylindrical Be windows. An exchangeable graphite sheet is placed along the inside of the Be windows to protect the Be window from being coated with evaporated materials. This sheet can be heated up to 250ºC by supplying electric current directly to prevent the evaporated materials from depositing onto the sheet itself.

4. Conclusions

The preparation of the experimental apparatus is proceeding satisfactory, and we started to users' experiments in the experimental hutch 1. Some new outcome, therefore, have been published. Then, we aim chiefly at commissioning two diffractometers in the experimental hutch 2 and hutch 3 in its early stage. Further, we have a plan to start XAFS study using high energy X-ray (over 35 keV) from the undulator. The driving system of the diamond double crystal monochromator connected to the undulator [12,13] will be installed in the near future.

Finally, the contact persons (who are authors of this manuscript) for Bl11XU are as follows; H. Shiwaku for all over the beamline and XAFS, M. Marushita for the monochromator, T. Mitsui and M. Seto for nuclear resonance scattering including Mössbauer spectroscopy, Y. Katayama for high-pressure high-temperature science, T. Inami for inelastic scattering, and M. Takahashi for surface science.

References

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Light Source	
Type of Insertion Device	In-vacuum undulator
Undulator Period, λu	32 mm
Number of Periods, N	140
Tunable Range	6 ~ 70 keV

X-rays at Sample	
Photon Flux	1.22×10^{13} photons/sec (14.4 keV, 48 m far from a source point)
Beam Size	0.5 mm(V)×1.8 mm(H)

Facilities in Experimental Station

Hutch 1 (Mössbauer Spectroscopy)
- experimental tables (vibration proof table, air pad type)
- precision goniometers
- versatile goniometers
- XY table and XZ table
- 4-jaw slit
- vacuum pumping system
- cryostat
- room temperature controller
- detectors
  - APD, PIN, scintillation counter, ionization chamber...

Hutch 1 (high-pressure science)
- cubic-type multi-anvil press, SMAP180
- IP reader

Hutch 2 (inelastic scattering)
- inelastic X-ray spectrometer
- spherical analyzer
- 8T super conducting magnet
- 2-stage closed-cycle He refrigerator

Hutch 3 (surface science)
- 4+2 type four axes X-ray diffractometer
- MBE chamber
- RHEED