Probing atomic vibration using nuclear resonant inelastic scattering
BL09XU: Nuclear Resonant Scattering Beamline

1. Introduction

BL09XU is an X-ray undulator beamline constructed for the research using nuclear resonant scattering (NRS), which is caused by the nuclear level as shown in Fig. 1. Usually X-rays are scattered by electrons and nuclear Thomson scattering is negligible. But when the energy of the incident X-ray coincides with that of the nuclear level, its scattering cross-section gets larger. The nuclear levels of various isotopes are different from each other and its energy width is extremely narrow, typically 10^{-6} to 10^{-9} eV. For example, the resonant energy of ^{57}Fe is 14.4 keV and the energy width is 4.7 x 10^{-9} eV. The half lifetime of the ^{57}Fe excited state is 98 nsec, which follows the Heisenberg uncertainty principle.

NRS of synchrotron radiation has specific features compared with conventional Mössbauer spectroscopy. Synchrotron radiation can produce a well-collimated and small beam, which allows the study of materials under extreme conditions and under diffraction conditions including grazing incident geometry. Another important technique is nuclear resonant inelastic scattering (NRIS) to study the localized vibrational density of states of the Mössbauer isotopes. Quantized vibrational motion is called a phonon, which is investigated by several spectroscopic methods such as Raman spectroscopy, inelastic X-ray scattering and neutron scattering. Its typical energy range is ~100 meV. Every isotope has different nuclear level, so NRIS has distinguished feature of just probing a specific isotope. Recently biochemical materials such as an enzyme have been intensively studied by NRIS to focus on its active center in the complicated system.

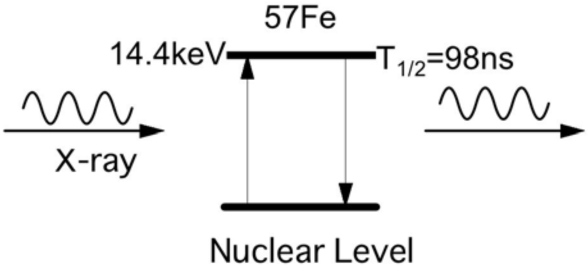

Fig. 1 Nuclear level scheme of ^{57}Fe and nuclear resonant scattering

2. BL09XU

BL09XU is a standard X-ray beamline in SPring-8 with a 32mm period linear undulator and a cryogenic cooling high-heat load monochromator. We have two experimental hutches as shown in Fig. 2 after one optics hutch where the high-heat load monochromator is located. High-resolution monochromators are usually arranged in Experimental Hutch 1. Samples are set with temperature control in Experimental Hutch 2.

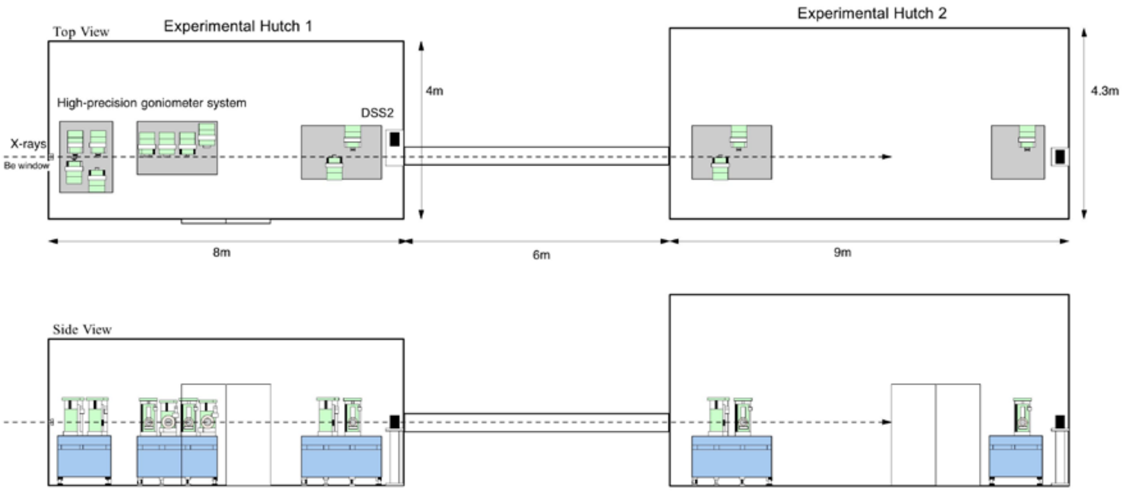

Fig. 2 schematic view of experimental hutches at BL09XU

2.1 High-resolution monochromator

A high-resolution monochromator (HRM) is one of the most important tools for the NRS study using synchrotron radiation. It is used to change the energy of the incident X-rays in case of the study of the vibrational states of materials using NRIS. The resolution of the obtained energy spectrum is determined by that of HRM. The 3 bounce-type HRM for ^{57}Fe with 0.8 meV resolution is shown in Fig. 3. It is composed of one germanium crystal and two silicon crystals.

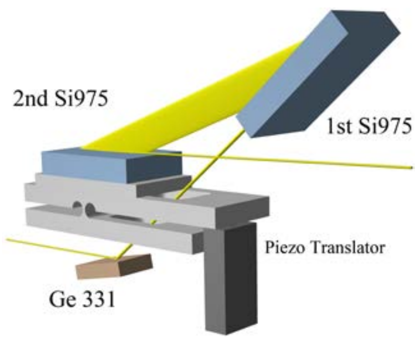

Fig. 3. Schematic view of HRM arrangements with resolution of 0.8 meV

2.2 Fast timing discrimination

The time structure of the incident X-ray is quite important for the time-resolved experiments such as a nuclear resonant scattering. The storage ring of SPring-8 has a harmonic number of 2436 and an R.F. frequency of 508.58 MHz. So the bunch interval is 1.966 nsec and it takes 4.8 □sec for electrons to turn around. The scheduled bunch mode at the beamline practical is F-mode, which is a hybrid bunch mode with 342.1 nsec interval. Its profile is shown in the following web site.

http://www.spring8.or.jp/en/users/operation_status/schedule/bunch_modes/1-14fill_12bunch

An avalanche photo diode (APD) detector with 1 nsec resolution and fast electric circuits are used to discriminate the prompt electronic scattering noise from the delayed signal, which originates from the nuclear resonant scattering.

2.3 Cryostat

A Continuous flow cryostat for X-ray diffraction, Oxford CF1104 is used for the temperature control of samples. Liquid nitrogen or liquid helium can be selected as flowing gas according to the controlled temperature of the sample. An outer vacuum chamber was specially designed for nuclear inelastic scattering experiments to make the distance between the sample and a detector as short as possible, because delayed signals are scattered into the whole solid angle after the nuclear inelastic absorption. The sample is mounted on the sample holder attached to the cryohead so as not to touch the top Be window as shown in Fig. 4.

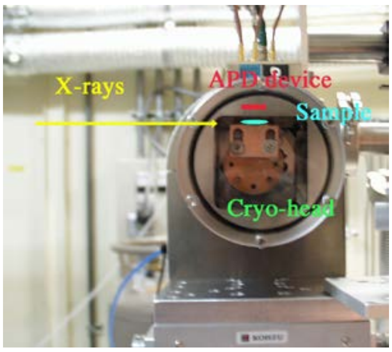

Fig. 4 Cryostat designed for nuclear resonant inelastic scattering experiments.

3. Practical

The following practical will be done after the introduction of the beamline and NRS.

1) HRM setting to get the beam with narrow energy width
   Adjust the undulator gap and high heat-load monochromator to get the beam with ~ 2eV resolution before HRM at Experimental Hutch 1. Tune the high heat-load monochromator to get the beam with ~ 2.5 meV through the HRM.

2) Fast electronics adjustment to get the delayed signal
   Adjust the fast electric circuits after setting the ^{57}Fe foil and APD detector to discriminate the prompt electronic scattering noise from the delayed signal.

3) Energy tuning to find the resonance of ^{57}Fe nuclear level
   Tune the X-ray energy from the HRM in to the energy of the ^{57}Fe nuclear level. The X-ray energy is changed by means of rotating the one of the crystals of the HRM.

4) NRIS measurement at room temperature
   Mount the model sample $[FeCl_4]^{-1}$ in the cryostat and start the NRIS measurement at room temperature counting the delayed signal with the finite energy step.

5) NRIS measurement at low temperature
   Start the flow of liquid He and mount the model sample again at low temperature. Start the NRIS measurement at low temperature. Compare the data between at room temperature and at low temperature.

References

1) "Nuclear Resonant Scattering Beamline at SPring-8", Y. Yoda et. al., Nucl. Instrum. Methods A, 467-468 (2001) 715-718.

2)"Observation of Nuclear Resonant Scattering accompanied by Phonon Excitation using Synchrotron Radiation", M. Seto et. al., Phys. Rev. Lett., **74** (1995) 3828