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Evolution of synchrotron-radiation-based Mössbauer absorption spectroscopy for various isotopes

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Abstract Synchrotron-radiation-based Mössbauer spectroscopy that yields absorption type Mössbauer spectra has been applied to various isotopes. This method enables the advanced measurement by using the excellent features of synchrotron radiation, such as Mössbauer spectroscopic measurement under high-pressures. Furthermore, energy selectivity of synchrotron radiation allows us to measure ^{40}K Mössbauer spectra, of which observation is impossible by using ordinary radioactive sources because the first excited state of ^{40}K is not populated by any radioactive parent nuclides. Moreover, this method has flexibility of the experimental setup that the measured sample can be used as a transmitter or a scatterer, depending on the sample conditions. To enhance the measurement efficiency of the spectroscopy, we developed a detection system in which a windowless avalanche photodiode (APD) detector is combined with a vacuum cryostat to detect internal conversion electrons adding to X-rays accompanied by nuclear de-excitation. In particular, by selecting the emission from the scatterer sample, depth selective synchrotron-radiation-based Mössbauer spectroscopy is possible. Furthermore, limitation of the time window in the delayed components enables us to obtain narrow linewidth in Mössbauer spectra. Measurement system

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that records velocity dependent time spectra and energy information simultaneously realizes the depth selective and narrow linewidth measurement.

Keywords Synchrotron-radiation-based Mössbauer absorption spectroscopy · Depth selective measurement · Simultaneous acquisition system for timing and energy information

1 Introduction

Mössbauer spectroscopy has been used extensively and has given lots of successful scientific results. Mössbauer spectroscopy gives us element specific information on the surrounding electronic states, which is one of the most superior features and is required for modern precise materials science and complex systems such as biological substances. The Mössbauer effects can be observed for about 100 nuclear transitions of nearly 50 elements. However, not all of the Mössbauer elements are used for actual exploitation. It is mainly due to difficulty of the preparation and retaining of the radioactive Mössbauer γ-ray sources with finite life times. In addition, in the case of nuclide ^{40}K whose excited state is not populated by any radioactive parent nuclides, the observation of the Mössbauer effect is impossible by using ordinary radioactive sources. In that case, in-beam methods with accelerators or reactors should be used. On the other hand, the energy tunable, highly-bright, well-collimated, pulsed and polarized synchrotron radiation is distinct and advantageous. The use of synchrotron radiation as an alternative source was first proposed in 1974 [1], and the first confirmed result of nuclear resonant Bragg scattering of synchrotron radiation was observed in 1984 [2] after pioneering experiments [3]. The energy tunablity of synchrotron radiation allowed us to observe nuclear resonance excitation of the ^{40}K nuclide [4]. Since the bandwidth of incident synchrotron radiation is much broader than the line-width of the nuclear levels and even hyperfine splitting, synchrotron radiation cannot be used as a simple alternative Mössbauer γ-ray source. The use of nuclear Bragg scattering offers a synchrotron radiation Mössbauer source with a single-line energy spectrum [5, 6]. However, the synchrotron radiation Mössbauer source has been made only for ^{57}Fe owing to difficulty to make perfect single crystals with pure nuclear reflections for other elements. The standard approach using synchrotron radiation is nuclear resonant forward scattering (NFS) measurement in time domain [7], which is closely related to Mössbauer spectroscopy and one of the most important methods. A nuclear light house effect is used to measure NFS in space domain [8], and this method is effective for high energy and short life time excited states. As another detection method of NFS, a heterodyne (stroboscopic) method has been developed [9]. In 2009, we have developed synchrotron-radiation-based Mössbauer spectroscopy yielding absorption spectra that are similar to Mössbauer spectra measured with radioactive sources [10]; using this method, we could measure Mössbauer spectra for the third excited state of ^{73}Ge.

In this article, recent results obtained using this method are shown with emphasis on the applicability to various Mössbauer isotopes. Moreover, we discuss the possibility of the depth selective Mössbauer measurement with the experimental and simulated results.

2 Conceptualization of developed method

In this method, to realize Mössbauer spectrum measurement using synchrotron radiation having much broad energy bandwidth compared with that of usual Mössbauer linewidth,

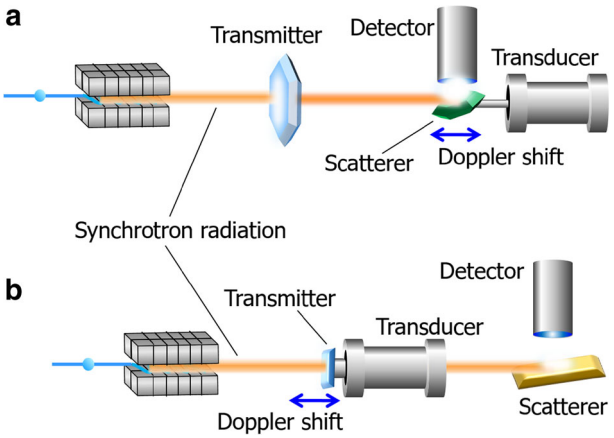

Fig. 1 Schematic drawings of the experimental setup of synchrotron-radiation-based Mössbauer absorption spectroscopy. **a** Sample is measured as a transmitter. **b** Sample is measured as a scatterer

we use a reference sample that corresponds to the Mössbauer radioactive source. In this method, radioactivity is not needed for this reference sample and may get into disturbance for the required signals. The schematic of this method is shown in Fig. 1. The measurement is done by irradiating a transmitter with synchrotron radiation and detecting the emission from a scatterer placed behind the transmitter and irradiated by the transmitted radiation. However, the scattered emission contains not only the emission due to nuclear resonant process but that caused by the prompt electronic processes. Since the electronic emission obscures the weak nuclear resonant emission, we use only delayed emission due to the finite life time originated from the nuclear resonant excitation. To realize this, we usually use a fast Si avalanche photodiode (APD) detector. In this measurement, either the transmitter or the scatterer is moved by a velocity transducer, as in the usual Mössbauer spectroscopy using radioactive sources. When the relative resonance energy of the scatterer coincides with the resonance energy of the transmitter via Doppler motion, the intensity of nuclear resonant scattering from the scatterer is reduced; this is due to nuclear resonance absorption in the transmitter. When the resonance energy of the scatterer differs from that of the transmitter, except for electronic processes, such as the photoelectric effect, that are independent of the Doppler velocity, there are no reduction of scattering in the scatterer. Therefore, one can observe a local minimum in the intensity spectrum of scattering at the same energy of the scatterer as that of the transmitter. This velocity-dependent scattering intensity yields a Mössbauer absorption-type spectrum in the energy domain. The theoretical framework of this method has been described previously [10, 11].

One of the advantageous features of this method is flexibility of the experimental setup, whereby the measured sample can be used either as a transmitter or as a scatterer. The transmission experiments are suitable for high-pressure measurement, and measurement of small samples. In contrast, scattering experiments are suitable for surface measurement, measurement of films on a substrate, and measurement of very thick samples. Note that this method accepts the detection of not only γ-rays but also fluorescent X-rays and internal conversion electrons, which are emitted during de-excitation. Fluorescent X-rays are easier to detect than γ-rays with a Si APD detector. It is because the energies of fluorescent X-rays are lower than those of γ-rays and, therefore, easy to be detected by thin Si diodes that

are required to achieve the fast time resolution. Moreover, electron detection is not difficult with APD detectors.

3 Measurement performed using various Mössbauer isotopes

After the development of this method, it has been applied to the Mössbauer spectroscopic measurement of many isotopes. In the first experiment, we could measure a ^{73}Ge Mössbauer spectrum of 68.752 keV excited state, which is difficult to measure with a radioactive source [10]. Moreover, in the paper, ^{57}Fe Mössbauer spectrum was also measured. For ^{151}Eu Mössbauer spectroscopy, time-window effect was studied [11]. Taking advantage of synchrotron radiation, Mössbauer spectroscopic measurement under high-pressures was performed for europium hydrides, and the valence change accompanied with the application of the pressures was clearly observed [12]. In this method, measurement in scattering geometry is possible, and ^{57}Fe and ^{119}Sn Mössbauer spectra of thin films were measured [13]. One of the most advantageous features of the use of the synchrotron radiation is the energy selectivity and this allowed the measurement of ^{40}K Mössbauer spectra [14]. Adding to these, ^{189}Os Mössbauer spectra [15] and ^{125}Te Mössbauer spectra [16] have been observed.

These introduced results were performed using an 8-element APD detector with a Be window shading the APD from optical photons [10]. However, this Be window blocked emitted electrons from the scatterer. Therefore, we have developed a windowless detection system that enables us to measure electrons adding to X-rays and γ-rays emitted from scattrerer [17]. In the developed system, a windowless APD detector was combined with a vacuum cryostat and, therefore, electron detection was realized. Moreover, the windowless detection system made the distance between the scatterer and the APD shorter than that of previous system; the covered solid angle was increased. As a feasibility study, this system was applied to Mössbauer spectroscopic measurement for ^{174}Yb with high resonance energy of 76.5 keV and it was confirmed that the counting rate is five times higher than that of the previous system. By using this system, Mössbauer spectroscopic studies have been performed for ^{61}Ni, which has the high excitation resonance energy of 67.4 keV. Nickel-based nanoparticles with a hexagonal structure that were synthesized by a chemical reduction method without enrichment were studied using this system [18], and nickel coordination complexes and metalloproteins samples were measured to demonstrate the potential of this approach [19]. Lithium-ion rechargeable batteries are now very actively studied materials, and ^{61}Ni Mössbauer measurement using this method was applied to study the change of the electronic states between the charge and discharge states [20]. Even in the low energy nuclides such as ^{149}Sm (22.507 keV), measurement time is much reduced [21].

As described above, this method has been applied to various isotopes so far, and it is applicable to other isotopes. In Table 1, the isotopes expected to be observed using the synchrotron-radiation-based Mössbauer absorption spectroscopy are shown [22–27].

4 Depth selective Mössbauer spectroscopy

Our developed windowless detecting system enables us to measure electrons and X-rays adding to γ-rays. Therefore, discrimination of them allows the measurement of the depth selective Mössbauer spectrum by using the difference of the escape ranges. Depth selective

Table 1 The isotopes expected to be observed using synchrotron-radiation-based Mössbauer absorption spectroscopy [22]

Nuclides	Natural abundace (%)	Energy (keV)	Half life (ns)	Internal conversion coefficient
^{40}K	0.0117	29.8299	4.24	0.2982
^{57}Fe	2.2	14.412497 [23]	98.3	8.56
^{61}Ni	1.14	67.413	5.34	0.139
^{73}Ge	7.73	68.752	1.74	0.227
^{83}Kr	11.5	9.4035 [24]	147	17.09
^{99}Ru	12.7	89.68	20.5	1.498
^{119}Sn	8.59	23.8795 [25]	18.03	5.22
^{121}Sb	57.36	37.133	3.46	11.11
^{125}Te	7.139	35.4922	1.48	14
^{127}I	100	57.608	1.95	3.77
^{129}Xe	26.4	39.578	0.97	12.31
^{133}Cs	100	80.9974	6.28	1.72
^{145}Nd	8.3	72.5	0.72	3.64
^{149}Sm	13.8	22.507	7.12	29.2
^{151}Eu	47.8	21.54149 [26]	9.6	28
^{158}Gd	24.84	79.51	2.52	6.02
^{161}Dy	18.9	25.6515	29.1	2.35
^{168}Er	26.8	79.804	1.88	7.14
^{169}Tm	100	8.4103	4.08	285
^{174}Yb	31.8	76.471	1.79	9.43
^{176}Hf	5.206	88.351	1.43	5.86
^{181}Ta	99.988	6.214 [27]	6050	70.5
^{183}W	14.3	46.4839	0.188	8.63
^{189}Os	16.1	36.202	1.62	21
^{193}Ir	62.7	73.044	6.09	6.24
^{197}Au	100	77.351	1.91	4.36
^{201}Hg	13.18	32.138	0.1	41.9
^{238}U	99.2745	44.91	0.203	618

conversion Mössbauer spectroscopy (DCEMS) with radioactive sources are well known [28].

We have performed feasibility study of the depth selective measurement using synchrotron-radiation-based ^{151}Eu Mössbauer spectroscopy. Experiments were performed at the nuclear resonant scattering beamline (BL09XU) of SPring-8. The storage ring was operated in the 203-bunch mode yielding a bunch distance of 23.6 ns. We used a powder sample of EuF_3 (not enriched, 40-μm thick) as the transmitter. A disk of EuF_3 powder (not enriched, 30-μm thick) was used as the scatterer. Delayed emission from the irradiated surface of the scatterer was measured using a multi-element Si-APD detector (4×2 APDs, each with a detection area of 3×5 mm^2). The experimental setup is described in ref. [11]. The scatterer sample was placed at a tilt; the angle between incident radiation and the sample surface was around 5 degrees. The measurement was done by detecting the electorons and X-rays emitted from the sample surface. The Mössbauer spectrum of europium fluoride

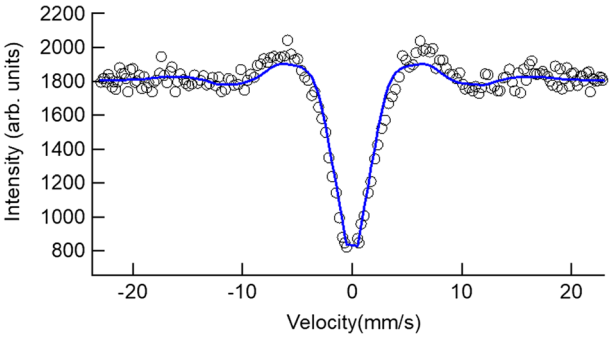

Fig. 2 The synchrotron-radiation-based ^{151}Eu Mössbauer spectrum of europium fluoride measured with the windowless detecting system

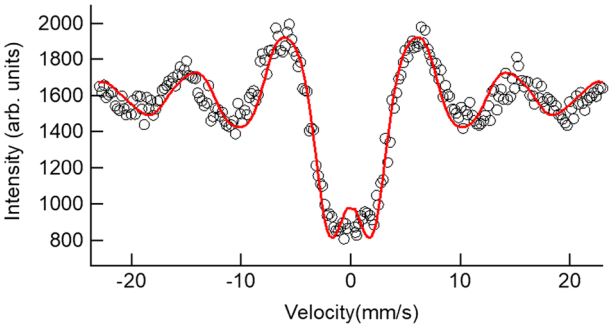

Fig. 3 The synchrotron-radiation-based ^{151}Eu Mössbauer spectrum of europium fluoride measured with a detecting system with a Be window. Moreover, the contribution from lower fluorescence X-rays was removed by using the high discrimination of the output signal from APD

measured with our windowless system is shown in Fig. 2. In this case, γ-rays, directly scattered X-rays, fluorescence X-rays and internal conversion electrons are detected by the APD detector. The typical orders of the escape ranges are 100 μm for the γ-rays (direct X-rays), 10 μm for fluorescence X-rays, and 1 μm for the electrons. Observed spectrum is well reproduced by the method discussed in [10]. In the calculation, all emitted contributions were taken into account by estimating their branching ratios, energies, absorbed ratio in the detector, and transmitted rates from the sample. Next, we removed the contribution from the electrons and the fluorescence X-rays by using the detector with a Be window and high discrimination of the output signal from APDs to cut only lower fluorescence X-rays. In this case, the apparent change is observed as show in Fig. 3. To confirm this result, we placed an aluminum plate with the thickness of 300 μm between the detector and the sample. In this case, only 21.5 keV γ (X)-rays can transmit this aluminum plate. The measured spectrum is almost the same as the previous one. Lines in the figures are the calculated spectra and reproduce the measured spectra very well. But, it should be noted that the difference does not reflect the difference of the electronic states between the surface and the area deep inside. In fact, these calculated results assumed the same electronic states; same isomer shifts with no magnetic and no quadrupole interactions were assumed. These changes of the spectra are caused by the differences of the path lengths along which

the incident synchrotron radiation travels in the sample. Since the tilted sample was used in this measurement, the path length of the incident radiation from the surface to the scattering point is proportional to that of the emission from the scattering point to the exit surface. The increase of the beam path length causes the interfered forward scattering component due to the transmitter and the scatterer, and this component induces the change of the shape of the measured spectrum. These results clearly show that it is possible to discern the depth of the scattered area in the sample by selecting the scattered component although any change of the electronic states is not observed in this measurement.

As for other nuclides, the possibility of the depth selective measurement strongly depends on the resonance energy and fluorescence X-ray energy, and so on. For example, since ^{61}Ni has high resonance energy of 67.4 keV, K electrons can travel around 10 μm and surface measurement may be difficult. On the other hand, ^{174}Yb has also high resonance energy of 76.5 keV but surface within 1 μm may be studied using the K electrons.

5 Detection system under development

In the synchrotron-radiation-based Mössbauer absorption spectroscopy, it is possible to obtain narrow linewidth Mössbauer spectrum by limiting the time region of delayed scattering [11, 17]. Although it is accompanied by the wavy background, it might be useful and effective for the determination of the small change, etc. However, the narrowing is attained at the cost of the intensity. Before starting the measurement, it is a difficult decision to fix the limited timing area. However, if we are able to measure the velocity dependent time spectra containing prompt scattering, it is possible to reconstruct the narrow linewidth Mössbauer spectrum after finishing the measurement without losing anything. Furthermore, if the energy information is indexed to the timing information, depth selective spectra, which were discussed in a previous section, can be obtained. These measurement are possible by using the detection system with simultaneous measurement of timing and energy, which was developed recently [29]. In this system, an amplitude to digital convertor is used to measure the energy information, and it is recorded as timing information to tag the counted photons through a time to digital convertor; the main purpose of this system is to observe the resonance excitation of the second-excited state in ^{229}Th. The simultaneous acquisition of the energy and timing, and post analysis availability are useful for measuring the nuclear resonant scattering of radioactive nuclides. Moreover, as for the APD detector, use of very thin (about 10 μm) APDs, which is difficult to detect high energy γ (X)-rays but can detect electrons mostly, enables us to measure the spectra due to the electrons emitted from the sample surface. By placing the thin APD in front of the thick APD, we can obtain electrons by the thin APD and γ (X)-rays by the behind thick APD without counting loss.

6 Summary

We developed synchrotron-radiation-based Mössbauer spectroscopy that yields absorption type Mössbauer spectra. By the use of the excellent feature of synchrotron radiation, advantageous measurement is possible. For example, highly bright and well-collimated synchrotron radiation is very effective for high-pressure and imaging measurement. Moreover, energy selectivity of the synchrotron radiation makes it possible to measure Mössbauer effect for various isotopes. Continuous development of this method has made it an effective method that enables practical measurement for various isotopes, particularly, a windowless

detection system improved the efficiency. In this method, there are many unique features and one of them is that the obtained spectra are similar to usual Mössbauer spectra obtained with radioactive sources. This makes the analysis of the measured spectra easy and accessible. Moreover, it has flexibility of the experimental setup that the measured sample can be used as a transmitter or a scatterer, depending on the sample conditions. The transmission experiments are suitable for high-pressure measurement, measurement of small samples, etc. On the other hand, scattering experiments are suitable for surface measurement, measurement of films on a substrate, measurement of very thick samples, etc. Particularly, by discriminating the emission from the scatterer, depth selective synchrotron-radiation-based Mössbauer spectroscopy is possible. Measurement system that records velocity dependent time spectra and energy information simultaneously will realize the depth selective and narrow linewidth measurement.

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