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APPLICATION OF QXAFS IN THE MEDIUM-ENERGY X-RAY ABSORPTION SPECTROSCOPY

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Abstract

A quick scanning X-ray absorption fine structure spectroscopy (QXAFS) system has just been installed in 4B7A, a general medium-energy X-ray beamline at Beijing Synchrotron Radiation Facility (BSRF). This system is independent so that the QXAFS system can be employed by other beamlines equipped with a double-crystal monochromator (DCM) to achieve quick scanning and data acquisition. Continuous scanning is available in this system to satisfy the time scale from a few seconds to several minutes, depending on the energy range to be scanned. In this case, our QXAFS system applied to medium-energy X-ray beamlines will broaden the application of time-resolved measurement to a greater range of elements, thereby benefiting a wider user community.

INTRODUCTION

Time-resolved X-ray absorption fine structure (XAFS) measurements play a crucial role in studying in situ dynamic processes. Numerous techniques have already been applied to many synchrotron radiation beamlines to shorten the acquisition time of a XAFS spectrum down to a few seconds or even milliseconds. Among these, quick scanning XAFS is one of the successful modes.

QXAFS maintains a full compatibility with the step-by-step mode, based on the double-crystal monochromator, that is commonly used for general XAFS experiments. In QXAFS, one key difference from traditional XAFS methods is that the crystal monochromator continuously and rapidly rotates, significantly reducing the collection time of the spectrum. More importantly, it is easily compatible with various sample conditions. Therefore, with the development of photon sources and mechanization, QXAFS has the potential to become a primary method in the future.

Up to now, QXAFS has mainly been applied to hard X-ray beamlines, with limited applications in the lower energy range. However, in the medium-energy X-ray regime, there is a pressing need for a time-resolved XAFS experimental technique to investigate dynamic processes occurring within short time frames, especially elements like sulfur that are active in the field of electrochemistry.

In this paper, we will introduce the QXAFS system built at 4B7A, where can conduct medium-energy XAFS experiments at BSRF. The newly equipped QXAFS will be applied in the total electron yield experimental mode, providing a new and reliable experimental platform for various in-situ experiments in the future [1].

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BEAMLINE OVERVIEW

The 4B7A beamline, completed in 2005 at BSRF, is dedicated to experiments in the medium-energy X-ray range [2]. BSRF is the first-generation synchrotron radiation facility of China, with its storage ring supporting high-energy physics experiments (Beijing Electron Positron Collider) and synchrotron radiation research. After an upgrade project in 2008, BSRF now operates in 2.5 GeV full-energy injection and top-up mode with 250 mA beam current in dedicated synchrotron radiation mode. Beamline's source is the No. 7 bending magnet in region 4 of storage ring. The bending magnet generates a magnetic field of 0.808 T and has a critical energy of 3358.6 eV. The source size is approximately 1.5 mm (H) × 0.4 mm (V). At the critical energy, the vertical divergence of the source is around 0.28 mrad. The maximum horizontal acceptance angle is 5 mrad, defined by the apertures in the front-end section.

As shown in Fig. 1, this beamline is equipped with a fixed-exit DCM, and usually used crystals are Si(111) and InSb(111). The corresponding energy range is from 1.75 to 3.5 keV while using InSb(111), and from 2.1 to 6.0 keV for Si(111), the useful Bragg angle is about from 19° to 71°, only one pair of crystals can be used at the same time. The energy resolution power (E/ΔE) was higher than 5000 at 3206 eV and 1800 at 5465 eV. The measured flux at the sample is higher than 3×10^{10} photons/s/250 mA in the energy region of 1.75–6.0 keV. The measured beam size at the sample position is about 5 mm (H) × 1.5 mm (V). Finally, due to diffraction forbiddance, Si(111) cannot emit even-order harmonics of X-rays. This will allow the experimental station to obtain high-purity monochromatic light.

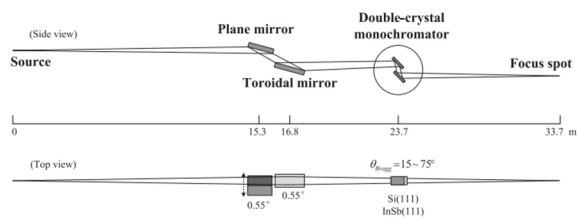

Figure 1: Schematic layout of beamline 4B7A.

QXAFS SYSTEM AND PERFORMANCE

For time-resolved XAFS experiments, QXAFS converts the motion mechanism of the monochromator and improves the data acquisition system. The 4B7A beamline has been equipped with a fixed-exit Si(111) DCM. The angle position is rotated through a stepper motor and recorded by

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an encoder. Ionization chamber (IC) is used to measure the incident X-ray intensity while an ammeter measures the photocurrent signal generated by the incident light excitation in total electron yield mode. By utilizing these two detectors, we aim to determine how fast does the QXAFS system can operate.

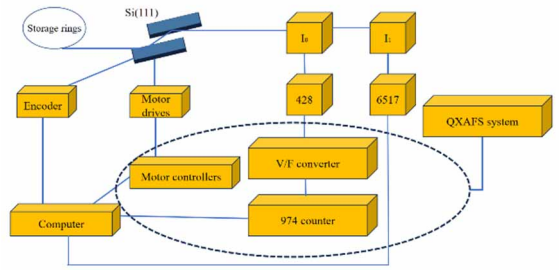

Figure 2: Schematic diagram of QXAFS.

Figure 2 is a schematic diagram of the QXAFS data acquisition process. QXAFS system is an independent system that uses a field programmable gate array (FPGA) module as a key logic unit to control the movement of the Bragg motor and data acquisition, thus it is more convenient and portable compared with previous complex acquisition systems for QXAFS [3]. The original data acquisition process for 4B7A is, the weak current detected by the IC is amplified by a 428 current amplifier to convert it into voltage. Then, the voltage signal is input into the compute through voltage-to-frequency converter (VFC) and analog-to-digital converter (ADC). The electronic signal from I_1 is directly read by a 6517B picoammeter and input into the computer. The computer controls the monochromator rotation and collects the signals from both channels, it then calculates the absorption coefficient. In the QXAFS system, integrating FPGA programming functionality does bring some advantages. By using FPGA programming, fast communication between hardware components and perfect time synchronization can be achieved without waiting for responses from other devices. This can save time and improve system efficiency. Additionally, FPGA offers flexibility and customization, allowing programming and configuration based on specific requirements to meet different application scenarios and experimental needs. In summary, integrating FPGA programming into the QXAFS system can provide better performance and faster data acquisition speed. Considering the signal-to-noise ratio (SNR) of the data and the finite response time of the detectors, the collection time for each data point is set to be greater than 1 ms.

By continuously scanning the motor at maximum speed, the acquisition time can be reduced greatly. The XANES spectrum can be obtained in approximately ten seconds, while the EXAFS spectrum requires about one minute. In practice, the speed limitation of QXAFS mainly comes from the inability of the monochromator to rotate too quickly, as it cannot exceed 0.28 degrees per second.

After setting up the apparatus, we first compared the differences between QXAFS and the conventional step-scan mode by using a potassium sulfate standard sample to ensure the reliability of the system.

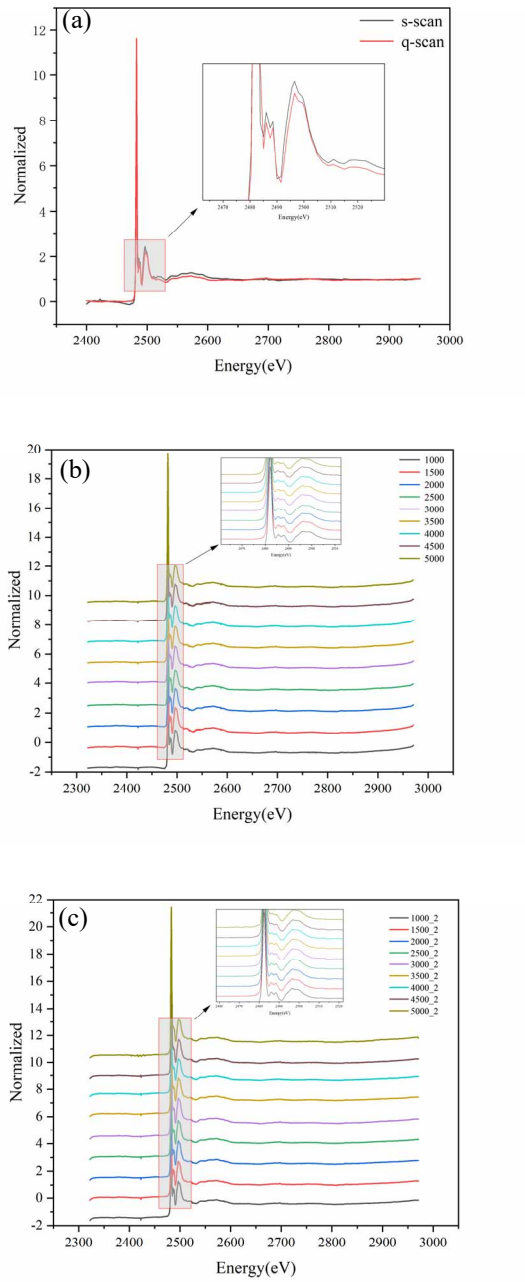

Figure 3: (a) Comparison of s-scan and q-scan for potassium sulfate; absorption spectra obtained from monochromator (b) forward rotation and (c) reverse rotation.

For the potassium sulfate standard sample, the data collection range was 2322~2972 eV at the monochromator speed was 0.17 degrees per second, here the step-scan took over half an hour, while the quick-scan only took 100 seconds. Figure 3 (a) shows that both QXAFS and the conventional mode exhibit similar oscillation features in the near-edge region. Additionally, we analyzed the effect of monochromator speed on spectrum quality. As shown in Fig. 3 (b) and (c), we tested spectra under different pulse speeds

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ranging from 1000 pulses per second (pps) to 5000 pulses per second (the monochromator rotates one degree every 18000 pulses) [4]. The obtained spectra represent the positive and negative spectra during one complete rotation of the monochromator. It can be observed that for spectra in the same direction, the near-edge oscillation parts are similar for different speeds, and the amplitude of the oscillations decreases as the speed increases, which means we can adjust the testing speed according to experimental needs with minimal sacrifice.

CONCLUSION

We have developed a QXAFS system at medium-energy X-ray absorption spectroscopy beamline and conducted preliminary performance testing, which can improve the function of XAFS beamlines and extend their capabilities to a wider user community. The results indicate that the spectra obtained from QXAFS are essentially consistent with those from conventional step-scanning, while significantly reducing the acquisition time. This provides a viable approach for elements in the medium-energy range that require time-resolved XAFS experiments. Furthermore, we will continue to develop QXAFS methods related to fluorescence yield mode and transmission mode to expand the application range of this system.

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