

A laboratory-based tender X-ray spectrometer for X-ray absorption fine structure measurements

Authors: Haisheng Yu, Zhu, Jianqiu, Wu, Mr. Jiaxing, Mr. Yaotian Guo, Mr. Song Li, Mr. Yan Zhang, Qian, Miss Xiaoxu, Wang, Jian-Qiang, Zhang, Dr. Linjuan (Nuclear energy), Yu, Haisheng, Zhu Jianqiu, Wu, Mr. Jiaxing, Guo, Mr. Yaotian, Li, Mr. Song, Zhang, Mr. Yan, Xiaoxu Qian, Dr. Jian-Qiang Wang

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Abstract

This paper describes the design and performance of a laboratory-based tender X-ray spectrometer for X-ray absorption spectroscopy. The system enables effective absorption spectra measurement within the 2.0-9.0 keV range using Rowland circle geometry: it covers the K edge of 3d transition metals, the L edge of lanthanides and the M edge of actinides. The spectrometer is configured with a 500 mm diameter Rowland circle and integrates a 250 W liquid metal-jet X-ray source, a spherical bent crystal analyzer, and an energy-resolving silicon drift detector. The X-ray source is installed outside the vacuum chamber and remains fixed, while the analyzer crystals and detector are adjusted to change the Bragg angle while maintaining the Rowland condition. The energy resolution is 0.36 - 1.30 eV at 2.0 - 9.0 keV, and the monochromatic flux is approximately 5×10^5 counts/s at 7040 eV. This paper highlights the main characteristics of the spectrometer and demonstrates its capabilities through selected experimental examples. The successful development of this spectrometer especially facilitates research on actinide elements, which are often constrained in synchrotron radiation experiments due to their radioactivity, thus fostering advancements in related nuclear energy fields.

Full Text

Preamble

A Laboratory-Based Tender X-ray Spectrometer for X-ray Absorption Fine Structure Measurements

Haisheng Yu,¹ Jianqiu Zhu,^{1,2} Jiaying Wu,¹ Yaotian Guo,¹ Song Li,¹ Yan Zhang,¹ Xiaoxu Qian,¹ Jian-Qiang Wang,^{1,2} and Linjuan Zhang^{1,2,†}

¹Key Laboratory of Interfacial Physics and Technology, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201800, China

²University of Chinese Academy of Sciences, Beijing 100049, China

This paper describes the design and performance of a laboratory-based tender X-ray spectrometer for X-ray absorption spectroscopy. The system enables effective absorption spectra measurement within the 2.0–9.0 keV range using Rowland circle geometry, covering the K edge of 3d transition metals, the L edge of lanthanides, and the M edge of actinides. The spectrometer is configured with a 500 mm diameter Rowland circle and integrates a 250 W liquid metal-jet X-ray source, a spherical bent crystal analyzer, and an energy-resolving silicon drift detector. The X-ray source is installed outside the vacuum chamber and remains fixed, while the analyzer crystals and detector are adjusted to change the Bragg angle while maintaining the Rowland condition. The energy resolution is 0.36–1.30 eV at 2.0–9.0 keV, and the monochromatic flux is approximately 5×10^5 counts/s at 7040 eV. This paper highlights the main characteristics of the spectrometer and demonstrates its capabilities through selected experimental examples. The successful development of this spectrometer especially facilitates research on actinide elements, which are often constrained in synchrotron radiation experiments due to their radioactivity, thus fostering advancements in related nuclear energy fields.

Keywords: Laboratory X-ray spectroscopy, XANES, EXAFS, Tender X-ray

Introduction

X-ray Absorption Fine Structure (XAFS) spectroscopy provides local structural information, making it widely used in materials science research [?, ?], life sciences [?], environmental studies [4–7], and other fields. The advent of synchrotron radiation in the 1970s significantly advanced XAFS technology, allowing it to evolve into a distinct experimental technique integrated with synchrotron facilities [?, ?]. However, synchrotron beamtime operations are time-consuming, which limits the ability to perform rapid screening and iterative experiments critical for understanding the chemistry and local structure of new materials. Additionally, transporting radioactive samples for synchrotron radiation in situ XAFS experiments is extremely complicated. Therefore, there is an urgent need to develop laboratory-based X-ray absorption spectrometers that can replicate XAFS experimental conditions.

X-ray energies and instruments are typically divided into soft, tender, and hard X-ray ranges [?, ?]. Currently, most laboratory spectrometers operate in the hard X-ray regime. These instruments require crystal monochromators and can operate with samples in air, but X-ray attenuation becomes problematic at lower energies. However, the absorption and scattering of X-rays by air decreases significantly as the energy increases. For example, in actinide research,

although the M-absorption edge (3.3–4 keV) exhibits smaller energy broadening than the L-absorption edge and is more sensitive to valence state [?], the X-ray absorption by air in this energy range is substantial, preventing the acquisition of satisfactory XAFS data. This energy range requires a fully in-vacuum, focusing crystal spectrometer.

Laboratory XAFS spectrometers commonly employ either dispersive or scanning geometries. The spectrometer geometry and diffraction characteristics of the analyzer crystal influence the selection and detection of photon energies. Dispersive spectrometers using the Von Hamos design perform X-ray detection simultaneously across a spectrum of energies, where different X-ray energies diffract at distinct locations on the crystal analyzer surface and are directed toward a spatially resolving detector. Scanning instruments utilizing Rowland circle geometry offer improved signal-to-noise ratios but necessitate more intricate mechanical designs [?]. To maintain the Rowland condition, both the analyzer crystals and detector must be adjusted to change the Bragg angle. Silicon or germanium are commonly used as analyzer crystal materials, with multiple crystal reflections required to cover absorption edges for various elements across the X-ray wavelength range. Prior to this work, numerous theoretical calculations on energy resolution were conducted [?]. Therefore, this paper focuses on the introduction and application of a tender energy spectrometer.

This paper introduces the world's first laboratory spectrometer capable of operating in the tender energy range. The instrument is specifically designed to facilitate XAFS research within the 2.0–9.0 keV energy range. The spectrometer design is based on Rowland circle geometry, featuring a polychromatic micro-focus X-ray source, a Johann-type spherically bent crystal analyzer, and a silicon drift detector (SDD). The remainder of this paper is organized as follows: Section II outlines the spectrometer design and its setup for conducting XAFS measurements. Section III details the experimental arrangement employed. Section IV introduces the performance of the spectrometer. Section V presents and analyzes results, accompanied by considerations that advocate for the extensive utilization and further advancement of laboratory-based approaches. Finally, Section VI provides a concise summary.

II. Spectrometer Design

The spectrometer is equipped with a laboratory X-ray source, bent Johann-type spherical crystal monochromators, an SDD, and a vacuum chamber, with all devices controlled by LabVIEW. Figure 1: see original paper illustrates a schematic diagram of the main components.

A. X-ray Source

Initial X-ray spectroscopy experiments were carried out using hermetically sealed X-ray tubes [?]. Excillum Inc. [?] has developed Metal-Jet X-ray tubes, which are conventional micro-focus tubes that utilize a liquid metal jet instead

of a solid-metal anode [?]. The metal-jet supports higher electron beam power, thereby generating higher X-ray flux. Therefore, the D2 model from Excillum Inc. was selected for laboratory XAFS measurements. The X-ray source employs a high-purity liquid gallium jet anode with a focal spot size of $20\text{ }\mu\text{m} \times 80\text{ }\mu\text{m}$, which transforms to a point source size of $20\text{ }\mu\text{m} \times 20\text{ }\mu\text{m}$ at a 14.5° take-off angle. The apex angle (opening angle) of the X-ray cone is approximately 8° , covering a circular area of 70 mm diameter on the crystal. The maximum accelerating potential is 70 kV, and the maximum current is 3.57 mA. The X-ray source window is a 50 μm thick beryllium window that transmits only half of 2 keV X-rays, but more than 82% of X-rays above 3 keV.

B. Crystal

Johann-type Spherically Bent Crystal Analysers (SBCAs) are used for monochromatizing and focusing polychromatic X-ray “bremsstrahlung” energy. To minimize the impact of strain fields resulting from spherical bending on energy resolution, crystal wafers are sliced into strips measuring 15 mm in width prior to being bonded with the glass concave substrate. These strips achieve energy resolution similar to diced-bent crystals while maintaining higher efficiency. All crystals are purchased from XRS LLC Inc. [?]. The SBCAs possess a bending radius of 500 mm and exhibit a surface diameter of 100 mm. To cover as wide a working range as possible within the 2.0–9.0 keV energy range and the spectrometer’s angular scanning range (55° – 80°), we are gradually expanding our collection of crystal analyzers. Currently, the available analyzer crystals include: Si (111), Si (220), Si (311), Si (400), Si (331), Si (422), Si (533), Ge (110), and Ge (620). Table 1 provides an overview of the spectrometer crystals currently accessible, including their respective coverage within the first order of reflection across the working range and theoretical energy Darwin width corresponding to those reflections. There is an energy gap between 2.4–3.2 keV because Si or Ge do not have crystal planes suitable for this intermediate energy range. Other crystalline materials, such as quartz, can cover this energy range; however, processing quartz spherical crystals remains a challenge.

C. Detector

The spectrometer is equipped with an Amptek Fast SDD for transmission XAFS measurements, effectively capturing the intensity of the diffraction signal throughout the data collection process. The Amptek Fast SDD is a vacuum-compatible detector with excellent energy resolution that suppresses higher-order harmonics in the diffraction signal while minimizing background noise. It features a 500 μm thick silicon sensor layer bonded to the top of the electronic layer, providing an effective area of 50 mm^2 . The SDD can achieve a counting rate of up to 1×10^6 counts/s with a peaking time constant of 0.2 μs . Its energy resolution is 123 eV at 5.9 keV. Integrated within the SDD is a two-stage Peltier cooling system designed to prevent heat buildup and ensure

efficient operation.

D. Motors and Movement

The X-ray source remains stationary outside the vacuum chamber. The crystal analyzer is mounted on a motorized module that provides vertical and pitch angular adjustments for alignment, as well as horizontal and rotational adjustments for energy scanning. The detector is held by a goniometer positioned on two linear stages called Dx and Dy in Fig. 1(b). The energy scanning mechanism of the spectrometer follows the Bragg angle equation and trigonometric formulas, which define the geometric requirements for the Rowland circle.

Fig. 1[Figure 1: see original paper] (a) Design schematic of laboratory X-ray absorption spectrometer based on Rowland circle geometry, (b) Schematic representation of the relative translations (δA_x , δD_x , δD_y , δ) of motors Ax, Dx, Dy, for two different photon energies; the crystal is maintained on the exact Rowland circle (shown with dotted lines), the center of the coordinate system is the X-ray source.

The X-ray source, sample, and bent crystal analyzer are arranged on a Rowland circle with diameter R, with the detector rigidly connected to the sample, which is always directed at the center of the spherical curved crystal by mechanical linkage. For testing, the sample is placed in front of the detector. Whenever there is a change in the Bragg angle or energy, it becomes necessary to shift the position of the detector on the Rowland circle. Simultaneously, adjustments must be made to both the angle and distance of the bent crystal analyzer. Taking the position of the X-ray source as the origin of the coordinate system, the X-axis is defined as the direction from the source to the crystal, and the Y-axis is defined as the direction from the source to the detector. Notably, the bent crystal moves exclusively along the X-axis. The spectrometer's motor positions (Ax, Dx and Dy) for a specific photon energy E (measured in eV) are given by:

$$2d_{hkl} \sin \theta = \frac{hc}{E} \quad (1)$$

$$A_x = R \sin \theta \quad (2)$$

$$D_x = 2R \sin \theta \cos^2 \theta \quad (3)$$

$$D_y = 2R \sin^2 \theta \cos \theta \quad (4)$$

where h is Planck's constant, c is the speed of light, R is the radius of the spherical bent crystal analyzer (Rowland circle diameter), θ is the Bragg angle, $d_{\{hkl\}}$ is the d-spacing for the given crystal type used, A_x is the x-coordinate of the bent crystal, and D_x and D_y are the x- and y-coordinates of the detector.

E. Vacuum Chamber

The tender X-ray monochromator employs a custom vacuum chamber with a pressure of 10^{-6} mbar, achieved using a 700 L/s vacuum pump. To prevent mechanical deformation from pressure differences, all mechanical components are mounted on a separate sturdy steel plate within the vacuum chamber, ensuring the alignment of the diffraction plane. Due to the volume limitations of individual components, achieving proximity during operation proved challenging, resulting in vacuum chamber dimensions of $1\text{ m} \times 0.8\text{ m} \times 0.6\text{ m}$ and Bragg angles ranging from 55 to 80 degrees.

III. Experimental Setup

XAFS measurements are conducted in transmission mode. The emission power and spot position of the Metal-Jet X-ray tubes are highly stable, allowing separate measurements of transmitted and direct beams using the same detector. All tests were conducted using the laboratory tender XAFS spectrometer at the Shanghai Institute of Applied Physics, Chinese Academy of Sciences.

To demonstrate the capabilities of the instrument, seven samples were tested as shown in Table 2. Due to the presence of spherical aberration, there is a noticeable difference in the focal lengths along the vertical and horizontal directions at the detector. This discrepancy varies depending on factors such as the Bragg angle and crystal plane. The measurements indicate an approximate range of 4–6 mm for the vertical focal length and 1–2 mm for the horizontal focal length. For XAFS testing in transmission mode, high sample homogeneity is crucial in both laboratory and synchrotron radiation setups [?]. By placing a slit in front of the sample, the spot size can be adjusted accordingly. Additionally, a uniform 10 mm sample can be obtained by pressing, ensuring uniformity over the spot scale.

The test conditions for the samples are summarized in Table 2. All metal foils were purchased from Exafs Materials Inc. [?], and TiO_2 was purchased from Aladdin Inc. [?]. The beam intensity was measured without the sample (I_0) and with the sample (I), with each scan consisting of 350 energy points, a counting time of 5 s per point, and a motor delay time of 1 s per point. The minimum energy step was set to 0.1 eV. The dead time of all the detectors was less than 25%.

IV. Spectrometer Performance

A. Monochromatic Flux

The monochromatic flux was tested with Ge (620) crystals in the energy position of 7040–7050 eV (Fig. 2Figure 2: see original paper). When the voltage was held constant and the current was reduced, the counting rate decreased proportionally, with similar rates corresponding to the same power. When the current was held constant and the voltage was increased, the counting rate increased,

reaching a maximum of approximately 5×10^5 counts/s at 60 kV, 3.5 mA, and 210 W.

B. Energy Resolution

It is difficult to directly evaluate the energy resolution of laboratory source systems, as two factors contribute to the total energy resolution: the energy broadening corresponding to the core-hole lifetime and the intrinsic energy resolution of the spectrometer. To characterize the spectrometer's energy resolution, we used the characteristic peaks of the X-ray source as a sample to provide a strong signal. The X-ray source operated at 120 W (40 kV, 3 mA) and used a Si553 crystal monochromator. Fig. 2(b) shows the test results: peaks A and B are fluorescence peaks for gallium $K\alpha_2$ (9223.8 eV, Bragg angle 71.905°) and $K\alpha_1$ (9250.6 eV, Bragg angle 71.403°). The full width at half maximum (FWHM) of peaks A and B are 2.96 eV and 2.89 eV, respectively. The energy broadening corresponding to the core-hole lifetime of the Ga $K\alpha_2$ and $K\alpha_1$ peaks are 2.66 eV and 2.59 eV, respectively [?]. The final energy resolution of the instrument (ΔE) is 1.298 eV at 9223.8 eV and 1.28 eV at 9250.6 eV.

Additionally, we have theoretically derived the instrument resolution for different energy ranges (Eq. 6). First, we obtained the ΔE_1 of the crystal based on the tested Δ and the crystal energy at 71.905° . ΔE_1 represents the difference between the energy resolution of the spectrometer and the Energy Darwinian Width of the crystal. In the laboratory spectrometer system, different energy ranges correspond to different crystal diffraction planes. The Darwin width correction for each diffraction plane is applied to obtain the final instrument resolution (the Darwin width of each crystal at Bragg angle 71.905° is obtained from XAS X-ray data for the elements). As shown in Table 3, the energy resolution ranges from 0.36 to 1.30 eV at 2.0 to 9.0 keV, covering the instrument's entire energy range.

$$\Delta E = \Delta \theta \cdot \cot \theta$$

where θ is the Bragg angle, ΔE is the intrinsic energy resolution, and E is the energy. (Δ is 0.00043 rad, the Bragg angle is 71.403° for Si553 at 9250.6 eV and 71.905° for others.)

V. Results and Discussion

A. XANES

Figures 3(a) and 3(b) show the normalized K-edge X-ray absorption near edge structure (XANES) of titanium foil and anatase TiO_2 alongside synchrotron data from BL14W1 of the Shanghai Synchrotron Radiation Facility (SSRF). All XAFS data were background-subtracted and normalized using the software Athena [?]. The Bragg angle, denoted as $\theta = 66.865^\circ$, was determined based on the first peak observed in the derivative spectrum of Ti foil and assigned a

value of 4966 eV. The consistency of the spectra shown in Fig. 3 (Figure 3: see original paper suggests that the energy resolution of the laboratory monochromator is similar to that (1.0 eV) achieved by using a double crystal Si(111) monochromator at a synchrotron. Nevertheless, it is crucial to recognize the inherent challenge associated with directly assessing energy resolution in laboratory source systems, encompassing both the measurement of FWHM and comprehensive characterization of the entire energy spectrum. The FWHM of the pre-edge peak of the Ti foil is about 1.2 eV. All features can be reproduced, demonstrating that the lab device is suitable for a wide range of applications.

The XANES spectra of TiO_2 in anatase and rutile forms are shown in Fig. 3(c). The spectrum consists of pre-edge components A1–A3, a distinctive shoulder B, and primary peaks C1–C3. The origin of these features has been described in other reports [26–28]. In the rutile TiO_2 sample, features A1 and A2 appear at lower energies compared to the anatase sample, while features B and C appear at the same energy as in anatase.

In our previous study, we employed a tender X-ray monochromator to analyze MXene materials (a kind of 2D materials [?]), depicted in Fig. 3(d). Our primary focus was on performing near-edge structure analysis of Ti_3AlC_2 and $\text{Ti}_3\text{C}_2\text{T}$ materials (a type of MXene), particularly those pertinent to supercapacitor applications [?]. Furthermore, we thoroughly investigated structural attributes and valence changes, building upon these findings.

Unlike the actinide L-edge, the M-edge of actinide elements shows less core energy level broadening and greater sensitivity to valence states. However, the M-edges of Th (3.3 keV) and U (3.5 keV) are outside the range of hard X-rays (> 5 keV) and have not been effectively tested using laboratory X-ray absorption spectrometers. They are within the energy range suitable for tender X-ray monochromators. The Th and U M-edge absorption spectra were first experimentally examined using a laboratory light source, as depicted in Figs. 3(e) and 3(f).

Figure 3(f) shows normalized XANES spectra of UO_2 and $\text{UO}_2(\text{NO}_3)_2$ alongside synchrotron data obtained from beamline XAFCA at the Singapore Synchrotron Light Source (SSLS). The XAFS data were normalized using the software Athena [?]. Compared with the XAFCA SSLS data, the $\text{UO}_2(\text{NO}_3)_2$ and UO_2 spectra collected by the laboratory spectrometer show a high degree of agreement, indicating that the spectrometer's energy resolution is comparable to that of synchrotron radiation near 3.5 keV. All observed features are reproducible, and this level of replication is adequate for most applications.

In the nuclear energy sector's efforts to immobilize actinide waste efficiently (Fig. 3(g)), we employed conventional U- M_4 edge XANES spectra to test the valence state of uranium ions and their coordination environment in U-doped $\text{La}_2\text{Zr}_2\text{O}_7$ -MS samples [?]. In the realm of efficient catalysts (Fig. 3(h)), XANES analysis detected an upward shift in the U- M_5 XANES spectra obtained from transmission mode measurements of UCoO_4 catalysts [?], confirming the presence of

U⁶⁺ and providing critical evidence of electronic structure modulation among polymetallic sites.

B. EXAFS

The Bragg angle ranges from 55 to 80 degrees, allowing extended X-ray absorption fine structure (EXAFS) measurements to extend up to or beyond 1000 eV above the absorption edge. Ni and Co foils were selected as samples for different energy ranges. The XAFS data for these samples are presented in Fig. 4[Figure 4: see original paper]. Figures 4(c) and 4(d) show the k-space spectra for the two samples, while Figs. 4(e) and 4(f) display the R-space spectra. The EXAFS spectra obtained from the laboratory spectrometer and Beamline 14W1 at the SSRF are comparable in their k-space oscillations (2-12 Å⁻¹), as shown in Figs. 4(a-f).

In the realm of electrocatalytic CO₂ reduction (Figs. 4(g-i)), the transition metal EXAFS of MPc (M=Fe, Co, Ni) catalysts supported on carbon nanotubes were analyzed using the tender X-ray monochromator [?]. The coordination number and bond length were ascertained through the fitting results, significantly contributing to the elucidation of the catalyst's structure.

VI. Conclusions and Outlook

We have presented the design and performance of a laboratory-based XAFS spectrometer utilizing an X-ray source. Laboratory investigations play a crucial role in the preliminary characterization of materials prior to synchrotron analysis. The enhanced configuration incorporates a focusing mirror and positions the sample behind the detector, thereby enabling a broader energy range due to the utilization of high-power X-ray sources and high-count rate detectors. With accessible laboratory spectrometers, XAFS has the potential to become a standard method for sample characterization, similar to other X-ray based experimental methods.

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