用于在线成分分析的 X 射线荧光光谱仪设计与原型制作

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Abstract

The X-ray fluorescence (XRF) system is designed and developed to enable faster and more convenient material composition detection and analysis. The system includes a Si-PIN detector, readout electronics, and a host computer. An algorithm is designed so that the detector can be used without vacuum and the device can achieve online composition analysis and data transmission. Testing was conducted using an iron ore sample, and the results demonstrate that the device achieves an energy resolution of 7.35% @6.4 keV. The measured result shows an 8% error compared to chemical methods. The device is capable of rapid composition analysis, with the potential for further applications in fields such as nuclear physics and mineral detection for industrial use.

Full Text

Preamble

Design and Prototyping of an X-ray Fluorescence Spectrometer for Online Component Analysis

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Abstract. An X-ray fluorescence (XRF) system has been designed and developed to enable faster and more convenient material composition detection and analysis. The system includes a Si-PIN detector, readout electronics, and a host computer. A dedicated algorithm allows the detector to operate without vacuum and enables the device to perform online composition analysis and data transmission. Testing with an iron ore sample demonstrates that the device achieves an energy resolution of 7.35% at 6.4 keV, with measured results showing an 8% error compared to chemical analysis methods. The device is capable of rapid composition analysis and shows potential for further applications in fields such as nuclear physics and mineral detection for industrial use.

Keywords: X-ray fluorescence (XRF), Si-PIN detector, readout electronics, elemental analysis, real-time measurement, non-destructive testing

Introduction

Since Wilhelm Conrad Röntgen discovered X-rays in 1895 [**?**], their exceptional penetrability has led to widespread use in medical imaging. X-rays can pass through the human body [**?**], creating images by measuring absorption and scattering [**?**], enabling non-invasive diagnostics. This ability to provide detailed internal images without surgery revolutionized modern medicine. The discovery of X-rays also sparked a transformation in modern physics. Later, Max von Laue demonstrated that X-rays could diffract through crystals, a breakthrough that earned him the Nobel Prize in Physics [**?**]. Building on this, William and Lawrence Bragg extended Laue’ s theory and developed the principles of X-ray crystallography [**?**], laying the foundation for significant advancements in the study of atomic and molecular structures.

In 1913, Henry Moseley [**?**] discovered the relationship between the characteristic X-ray energy of elements and their atomic numbers, becoming the first to use X-rays for composition analysis. This work played a crucial role in promoting atomic theory and enabling X-ray-based techniques for elemental analysis. In 1923, Coster and Hevesy discovered the element hafnium, the first element to be identified using X-ray spectral analysis [**?**, **?**]. In 1928, Glocker and Schreiber [**?**] performed the first quantitative analysis of materials using X-ray fluorescence (XRF). XRF is an analytical technique based on fluorescence generated when substances are excited by X-rays. When a sample is irradiated with primary X-rays, the elements in the sample become excited and emit characteristic X-rays with specific energies. By detecting these energies and intensities, the types and contents of elements in the sample can be determined, enabling both qualitative and quantitative analysis of material composition [**?**, **?**].

In 1948, Herbert Friedman and Laverne Stanfield Birks developed the world’ s first commercial wavelength-dispersive X-ray fluorescence (WDXRF) spectrometer [**?**]. In 1965, the introduction of the Si(Li) detector for X-ray detection revolutionized the field. This detector was soon integrated into energy-dispersive X-ray fluorescence (EDXRF) spectrometers, becoming a core component in X-ray detection systems [?]. The development of XRF technology has been reviewed extensively in the literature [?].

With advancements in X-ray detection technology, applications have expanded to various fields, including geology [?, ?], archaeology [?], medicine [?], food science [?], cultural heritage analysis [?], and nuclear physics experiments [?, ?]. In nuclear physics experiments, for example, the purity of target materials is a critical parameter that significantly impacts experimental results related to nuclear properties [?] and the synthesis of new isotopes [?]. The application of X-rays in these diverse fields highlights the potential of X-ray-based techniques for material analysis, which naturally led to the development of XRF.

However, several challenges remain that have driven continued enhancement of XRF technology, enabling more convenient and versatile applications across a wide range of scientific and industrial domains. Currently, XRF devices are primarily used in laboratories, requiring complex sample preparation and operation under vacuum conditions, which limits measurement timeliness. Additionally, the lack of online transmission functionality leads to inconvenience in use and makes them unsuitable for direct operation in complex environments like production lines. Therefore, an XRF device that enables faster measurement with online transmission and greater convenience has become an important research focus.

To address these issues, the present work has designed an XRF prototype, covering both hardware design and algorithm development. This system overcomes the limitations of traditional XRF devices to some extent, enabling efficient elemental analysis across a wide range of practical applications.

This paper describes the design and performance of the XRF system. Section 2 describes the system design, while Sections 3, 4, and 5 cover the hardware design, software design, and performance test results, respectively. Section 6 provides a summary.

II. Design of the System

The XRF system is an elemental analysis device based on the principle of X-ray excitation fluorescence, primarily used for analyzing material composition. Its physical foundation is based on Moseley's law [?] in atomic physics, which states that the energy of characteristic X-rays emitted by an atom is proportional to the square of its atomic number. The concentration of target elements can be calculated from the intensity of characteristic X-rays, enabling both qualitative and quantitative analysis [?]. Figure 1 [Figure 1: see original paper] shows the full view of the device. The external dimensions are 600 mm × 450 mm × 300 mm, with a total weight of 20 kg. The body is made of aluminum alloy and equipped with 2 mm thick lead (Pb) shielding to block excess radiation. The main parameters of the X-ray generator and detector are shown in Table 1 and Table 2 .

The workflow diagram of the XRF system is presented in Fig. 2 [Figure 2: see original paper]. The working process begins with the X-ray generator, which produces primary X-rays under excitation from a high-voltage power supply. The sample is placed in a sample holder that rotates at constant speed to eliminate effects of uneven sample distribution. The X-rays pass through a collimator and irradiate the sample, which becomes excited and produces characteristic X-rays. These characteristic X-rays are detected by a semiconductor detector. The signal is then amplified by a preamplifier and sent to readout electronics for processing. The results are transmitted to the host computer via a data interface for further processing, display, and analysis. Through this complete signal processing and transmission flow, the XRF system performs both qualitative and quantitative analysis of elements in the sample, providing an efficient and reliable technical means for material composition detection.

III. Hardware Design of the Readout Electronics

The prototyping requirements for readout electronics (PRE) for precision measurement differ from those for on-site rough measurement. Due to the need for fast and accurate measurement, the front-end electronics require high speed and low noise. Readout electronics for XRF are significant to produce, making it necessary to first design prototypes and validate their functionality.

The PRE uses an FPGA to achieve high reliability, speed, and low noise levels. For the PRE, two AD9248 chips [?] were used to realize 4-channel detector signal readout. Figure 3 [Figure 3: see original paper] shows the block diagram and photograph of the PRE, which includes a filter circuit, power management circuit, ADC (Analog-to-Digital Converter) circuit, field-programmable gate array (FPGA) circuit, communication interface circuit, and detector signal interface circuit. The PRE uses a Xilinx Kintex-7 FPGA for control and data transmission.

The charge pulse signal generated by the detector is connected to the PRE via a LEMO connector or FPC connector. The charge signal is integrated and shaped in the filter circuit before being sent to the ADC circuit for digitization. The FPGA receives data from the ADC circuit, processes and packages it, and transmits it to the host computer through a Gigabit Ethernet link. Additionally, the PRE can receive control commands through a universal asynchronous receiver/transmitter (UART) interface or universal serial bus (USB) 3.0, and transmit parameter information to the host.

A. Filter Circuit

The filter circuit plays a crucial role in the system, with its primary function being to eliminate noise and unwanted frequency components from the input signal, thereby ensuring signal stability and accuracy. Capacitors placed near the power supply voltage regulator chip filter out high-frequency noise and fluctuations, ensuring stable DC power output and preventing power fluctuations from affecting normal operation of amplifiers and other components. A filtering network is implemented at the input and output terminals of each operational amplifier. The input stage high-pass filter, formed by a combination of capacitors and resistors, eliminates the DC component or low-frequency noise from the input signal, allowing the operational amplifier to process only high-frequency AC signals. This design effectively prevents DC offset from affecting amplifier stability. The output stage low-pass filter, placed at the output terminal of the operational amplifier, suppresses high-frequency noise and prevents interference signals from entering subsequent circuits or the data acquisition system.

B. Power Supply Circuit

To ensure consistent operation of the PRE, a stable power supply is necessary. The circuit system is supported by an external 6V power supply that powers the FPGA and other chips, meeting the power-up sequence requirements of the Xilinx FPGA. The power supply circuit provides stable voltage outputs of 1.8 V, 2.5 V, 1.5 V, and 3.3 V, meeting the operational requirements of different modules. By adjusting components and feedback loops, the system can precisely maintain the stability of each voltage rail, preventing voltage fluctuations from affecting system operation. The power supply circuit utilizes the LTM4644 voltage regulator chip, which converts the input 6V voltage into the different required voltages for each module. Additionally, by adjusting the output voltage of each rail, the system ensures that all components receive precise and appropriate voltage values.

C. ADC Circuit

The ADC circuit is centered around the AD9248 dual-channel 14-bit analog-to-digital converter, whose primary function is to convert the input analog signal into a digital signal for output to the FPGA. To ensure ADC conversion accuracy and stability, the circuit design covers multiple aspects, including power supply, analog input, reference voltage, digital interfaces, and signal conditioning. The ADC power section is supplied with +3.3V power, and filtering capacitors effectively suppress high-frequency noise in the power supply, ensuring stable chip operation. The analog input channels are configured with a series of filtering components before the input signal to remove high-frequency noise and improve signal quality. The reference voltage is provided by the precision reference voltage source MCP1501T-10E, ensuring that the AD9248 has a stable voltage reference during analog-to-digital conversion, thereby enhancing conversion precision. The AD9248 outputs a 14-bit digital signal through a parallel interface, with the sampling rate controlled by a clock signal to ensure reliable data sampling. Control signals are managed by the FPGA or other controllers, allowing flexible adjustment of the ADC’ s operational state. To further optimize signal quality, an anti-aliasing filter is added at the ADC input to reduce the impact of high-frequency noise and aliasing on sampling fidelity.

D. FPGA Circuit

The FPGA used in this system is the Xilinx Kintex-7 XC7K70T-2FBG676I [?], which executes control and data transmission via an optical link. Figure 4 [Figure 4: see original paper] illustrates the FPGA-based architecture design. The process begins with the detector capturing X-rays and converting them into current signals. These signals are initially processed by the front-end electronics, which include filters to remove noise, ensuring the signal stability and accuracy necessary for precise measurements.

Once processed, the analog signals are converted into digital signals by the AD9248, which are then sent to the FPGA for further digital signal processing. The FPGA uses high-frequency clock signals provided by an external crystal oscillator, performing tasks such as data preprocessing and packetization. The processed data is ultimately transmitted to a computer for further analysis via a Gigabit Ethernet interface. The system' s power module ensures that each functional unit receives stable voltage, thereby guaranteeing the reliability and efficiency of the entire system. This design is particularly well-suited for applications requiring high-precision data acquisition and processing, ensuring both accuracy and robustness in complex measurement environments.

E. Interface Circuit

The Si-PIN detector offers good energy resolution and a low detection threshold. For the PRE implementation, a LEMO connector or FPC connector is adopted for signal transmission. Additionally, to test the electronics performance, four standard LEMO connectors and four standard FPC connectors can be used interchangeably to extract the four input signals from the Si-PIN detector for real-time monitoring.

A highly reliable communication interface connection is required between the PRE and the host computer. The PRE' s configuration commands downstream and status parameters upstream are transmitted through a UART interface. The scientific data transfer interface is designed to use a small form-factor pluggable (SFP) connector. Data transmission to the host is carried out via Gigabit Ethernet, and for compatibility with different environments, a USB 3.0 interface is also provided.

IV. Software Design of the Readout Electronics

The algorithm section outlines the framework used for signal processing and elemental analysis within the system. The process begins by acquiring original signals from the detector, followed by a series of signal conditioning and processing steps aimed at noise rejection, peak identification, and concentration calculation of target elements. Each step is carefully designed to address challenges such as signal noise, background interference, and X-ray attenuation, which can all impact measurement precision. Specific methods employed in signal processing include Moving Average Filtering (MAF) [?], Moving Window Deconvolution (MWD) [?], and background subtraction, all of which collectively enhance system accuracy.

A. FPGA Internal Algorithms

Signal amplitude and arrival time are critical parameters for physical studies, with amplitude directly reflecting particle energy information. During signal acquisition from the detector, the signal is first amplified by a charge-sensitive preamplifier and then sampled by an ADC. However, due to environmental background noise and hardware noise within the electronic system, the signal is inevitably affected during acquisition, which impacts the detector' s energy resolution and reduces measurement accuracy. Excessive noise may even cause false triggering, generating invalid data. Therefore, filtering [?] must be applied before readout to minimize noise impact while preserving signal characteristics and improving the signal-to-noise ratio (SNR).

Figure 5 [Figure 5: see original paper] illustrates the overall algorithm flow used in the system. To obtain the final energy spectrum, we employ the Moving Average Filter (MAF) [?] algorithm. This algorithm smooths the signal waveform to filter out noise influence while minimizing changes to the overall signal shape, ensuring integrity of signal characteristics. Here, the leading-edge discrimination (LED) [?] method is employed as a time-triggering mechanism, providing support for subsequent data processing.

B. Data Processing Algorithms

The main functions of the host computer include receiving and storing data processed by the FPGA, and displaying system test results and waveforms in a visualized manner in real-time. The host software interface provides an intuitive view of processed data and supports real-time transmission of results to a cloud platform, facilitating subsequent data analysis and processing.

Figure 6 [Figure 6: see original paper] presents the complete data processing flow. During data acquisition, two sets of data are recorded sequentially. Subsequently, the host computer reads these files along with several data processing parameters, including the characteristic X-ray energy table, background from the silver element in the X-ray generator, the attenuation factor of X-rays in air, and the efficiency of the Si-PIN detector. To eliminate background interference, we collect the background signal under the same conditions as the actual test but without placing the sample. The collected data is then subjected to Gaussian fitting, and the fitted results are subtracted from subsequent data to achieve correction.

Attenuation correction of X-rays in air is also considered in the current work, enabling the device to be used in a non-vacuum environment. We simulated the proportion of X-rays passing through 10 mm of air (the distance between detector and sample) at different energies, covering X-ray energies from 0.5 keV to 50 keV. Using Geant4 [?, ?] software, a sensitive detector with a square shape and 800 mm side length was set with air as the material. X-rays were emitted at a 2π angle from a point 10 mm away from the air, and the number of X-rays remaining after passing through 10 mm of air was recorded. The simulation results show the relationship between X-ray energy and remaining X-ray proportions after passing through air, as depicted in Fig. 7 [Figure 7: see original paper]. The x-axis represents X-ray energy, while the y-axis shows the X-ray attenuation factor, which is the penetration ratio remaining after passing through 10.0 mm of air, denoted as (cid:15)0.

In addition, detector efficiency is corrected. Based on detector data provided by Amptek [?], we performed a polynomial fit to model the efficiency of the Si-PIN detector. To simplify the fitting process, we fit the curve at three segment endpoints for different energy ranges: the red line represents 0-1.8 keV, blue line represents 1.8-15 keV, and green line represents 15-40 keV. The results are presented in Fig. 8 [Figure 8: see original paper]. At this stage, the detector efficiency is denoted as (cid:15)1. The initial X-ray counts are denoted as y0, and the actual X-ray counts y should be written as: (cid:15)0 × (cid:15)1.

Peak shape identification is performed after reading file parameters and correcting counts. For each detected peak, a Gaussian function is used to fit the data, determining the peak' s center position, shape, and height. The energy of the characteristic peak is then compared with known X-ray data tables to identify the corresponding element. To obtain element concentration, we calculate the peak area based on the fitted curve using the left and right boundaries of the peak. The integral method of the histogram [?] is employed, and the background integral is subtracted. The concentration calculation process is shown in equation (2), where P represents the proportion of a specific element and Ai represents the intensity of characteristic X-rays of specific elements. Through this process, the relative concentration of the element in the detected sample can be determined. To reduce systematic error, results from the two files are averaged and presented as the final result.

In the process of signal amplitude extraction, issues such as ballistic loss, noise, and signal pile-up in real-world systems may affect accuracy when directly obtaining the maximum value, thereby reducing the detector' s energy resolution. To address this, this paper adopts the Moving Window Deconvolution (MWD) [?] trapezoidal shaping algorithm. The MWD algorithm is a hardware-friendly and computationally efficient trapezoidal shaping method that works by convolving the slowly decaying exponential signal, transforming the long tail into a rectangular pulse where the pulse duration equals the width of the moving window. This algorithm achieves the trapezoidal shaping effect with minimal computational effort and significantly enhances system performance. Through this processing method, signal amplitude can be extracted more accurately, improving energy resolution and reducing the impact of noise and pile-up effects.

V. Performance Measurements

To explore whether the PRE design meets application requirements, a series of tests were performed to evaluate the functionality and performance of the proposed design. In laboratory tests, a signal generator (Tektronix AFG31000) produced exponential signals that were fanned out to the PRE. The test results were analyzed to evaluate PRE performance and verify design reliability. The pulse signal output simulated the charge signal generated by the Si-PIN detector.

A. Baseline Noise Characterization

Noise characterization refers to modeling random perturbations introduced during signal transmission and processing, which is significant for interpreting experimental results, electronic model verification, and physical information extraction. Baseline noise testing quantifies system noise levels and evaluates electronic data reliability.

During testing, baseline noise analysis of the 4 channels of the PRE was carried out at a trigger frequency of 10 kHz to simulate the state of the Si-PIN detector during transmission of uncompressed raw data. Noise level was characterized through its Root Mean Square (RMS) value. Figure 9 [Figure 9: see original paper] illustrates the baseline data and corresponding RMS noise levels. The red circle represents the baseline level, with ADC codes ranging between 1250 and 1350. The black dots represent the RMS noise of the electronic device. Analysis of 100,000 data points sampled from the baseline shows that the RMS noise of the PRE is below 1.3 ADC codes.

B. Channel Linearity and Amplitude Resolution Test

To verify channel linearity, a pulse signal generator was used to generate exponential voltage pulses that were sent to the PRE for data collection. Ideally, the relationship between input and output of a readout electronics system is a linear positive proportion function, whose slope represents the system' s magnification. In the present test, measurements were first taken at 30 mV, 50 mV, and 70 mV. For subsequent measurements, data was collected at increasing intervals, with each collection occurring every 100 mV increase in voltage. After testing, all four channels exhibited similar characteristics. Figure 10 [Figure 10: see original paper] illustrates the linearity and amplitude resolution of channel 1 as an example. The channel shows good linearity with a slope of approximately 21. Amplitude resolution indicates the electronics system' s ability to resolve different input signal amplitudes and equivalently characterizes the energy resolution. After 200 mV, the amplitude resolution is 0.15%. The other three channels also exhibit similar linearities and resolutions.

C. Joint Test with Si-PIN Detector

To verify the performance of the XRF design, a joint test was conducted. The experimental platform was built as shown in Fig. 11 [Figure 11: see original paper]. With a single click on the user interface to initiate measurement, the system begins testing. The testing duration was set to the standard 3 minutes. This streamlined process saves time and enhances overall ease of use, enabling rapid, real-time analysis without the complexity of traditional methods. The test system included the PRE, a Si-PIN detector, a low-noise digital power supply, and a host computer. During testing, a piece of iron ore was used with an X-ray generator voltage of 15 kV and a current of 0.02 mA to irradiate the sample.

Figure 12 [Figure 12: see original paper] shows the energy spectrum obtained under these conditions. The red and green curves correspond to the fitted characteristic peaks of iron (Fe), while the black curve represents the overall fit of the entire peak shape. The two characteristic peaks of iron are distinctly resolved, demonstrating the system' s ability to effectively differentiate closely spaced energy levels in the lower energy range. The energy resolution of the 6.4 keV characteristic peak of iron is 7.35%.

In the test, we also obtained the iron content in the iron ore sample, yielding a result of 41% while the chemical method gave 49%. This indicates that the current XRF method achieves results close to those of chemical methods while offering fast and convenient measurement. This demonstrates that the XRF system function is fully operational and meets expected goals, confirming its effectiveness and reliability for elemental analysis.

VI. Summary

In this paper, an XRF system including a Si-PIN detector, PRE, and software algorithm has been designed. The PRE mainly consists of the power management circuit, ADC circuit, FPGA circuit, communication interface circuit, and detector signal interface circuit. Tests indicate that the PRE system performs well. The algorithm includes both the internal FPGA algorithm and the data processing algorithm on the host computer, working together to achieve qualitative and quantitative analysis results. The joint test shows results similar to those of chemical methods. The current work has designed an XRF system that allows the detector to be used in air and enables online data transmission to a cloud platform, making it suitable for non-destructive measurement in complex environments such as industrial production lines and scientific research. It is worth noting that the current algorithm does not account for correction of matrix effects. With the addition of this correction in future work, the accuracy of the system is expected to improve further.

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Note: Figure translations are in progress. See original paper for figures.