Development and Application of a New Type of X-ray Fluorescence Online Grade Analyzer Based on STM32

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Abstract. In order to achieve more accurate measurement of the pulp element grade in the beneficiation process, a new generation of nuclear-free X-ray fluorescence online grade analyzer was developed based on the previous generation of nuclear online grade analyzer. This new X-ray fluorescence grade analyzer is developed based on the STM32F407 control chip. It adopts a measurement structure that combines X high voltage light tube and semiconductor detector. Its technical structure and analysis principle are: X-ray tube and semiconductor detector constitute a measurement unit. The X-rays excited by the X-ray tube are irradiated to the surface of the slurry to be measured, and the semiconductor detector receives X-characteristic fluorescence excited by the element to be measured in the slurry. The main control unit receives and processes the data signals uploaded by the semiconductor detector. Send the measured element count rate to the upper computer of the industrial computer through serial communication. Finally, the higher-level software calls Matlab mathematical analysis tool while using BP neural network to perform data modeling analysis and obtain the grade value. According to a field industrial test conducted at a ore dressing plant in Shaoxing, compared with the previous generation nuclear grade analyzer, the measurement accuracy and operating stability of the new X-ray online grade analyzer have been significantly improved, and various parameters All met the design requirements for development.

1. Introduction

In the beneficiation industry, pulp grade is one of the criteria for considering the effect of beneficiation process and an important indicator to guide beneficiation production. At the same time, the measurement of pulp grade has always been the research focus and technical difficulty of the beneficiation industry. The accuracy of online measurement of pulp grade directly affects the effect of automatic control of the beneficiation process. Therefore, the development of an online grade analysis system with strong real-time performance, high accuracy, good security, and high cost performance is urgently needed in the mineral processing industry.

At present, the international method of measuring element quality online mainly uses energy dispersion. At the same time, many domestic universities and research institutions have also conducted in-depth research on elemental analysis methods of energy dispersion and related theories and methods of X-ray energy spectrum analysis. For example, Xiong Wei from Nanjing University of
Aeronautics and Astronautics designed an X-ray fluorescence spectrometer for the requirements of RoHS detection using the working principle of energy dispersion spectroscopy analysis[1]; Dai Zhiyong and others from Southeast University designed and developed an X-ray fluorescence analyzer composed of X-ray tube, high voltage power system, light path system and signal processing system using the principle of energy dispersion[2]; Li Qiushi from Chengdu University of Technology through research to solve the problem of light element characteristic X-ray absorption in air dispersion X-ray fluorescence analysis[3]; Li Jie and others from Beijing Mining and Metallurgy Technology Group Co., Ltd. developed a set of analysis software for fluorescence analyzer on the Visual C ++ 6.0 development platform by studying the energy dispersive X-ray fluorescence analyzer, which is friendly and easy to use[4]. At present, the pulp grade measurement products on the market mainly include Cloux 5X / 6X SL analyzer from Outotec Finland, WDPF online grade analyzer from Sinosteel Ma'anshan Mining Research Institute Co., Ltd., and BOBA online grade from Beijing Mining and Metallurgy Technology Group Co., Ltd., etc. Among them, the Courrier 5X / 6X SL analyzer and the BOXA online grade analyzer are composed of a primary sampler, multiplexer, analyzer control unit, analyzer probe, and analyzer management station. They use a multi-channel sampling and centralized analysis method[5,6,7]. At the same time, the automatic film change of the measurement window can be completed[8]. Due to the complexity of the environment equipment of the beneficiation industry, according to the field investigation, in this beneficiation workshop, there may be problems that the sampling pipeline is easily clogged and the measurement cycle is too long. Therefore, there is still much room for improvement.

The online pulp grade analyzer is one of the mine-specific testing instruments that has been promoted and applied by our hospital for decades. It uses the method of direct sampling and measurement at the required measurement points, which achieves real-time measurement of grade data and greatly reduces the later maintenance of the meter workload.

2. Measurement Analysis Principle

The main difference between the measurement analysis principle of the new X-ray online grade analyzer and previous generations is that the new online grade analyzer uses X-ray tubes to excite X-ray fluorescence of the elements. At the same time, the imported component semiconductor detector is used to replace the original proportional counting tube. The semiconductor detector uses a Si-Pin semiconductor X-ray probe and a two-stage thermoelectric cooling mode. The measurement accuracy is higher, and the safety and stability have been greatly improved. As shown in Figure 1, the X-rays emitted by the new online grade analyzer X-ray tube interact with each element atom in the pulp to be measured in the sampling window. When the incident photon energy is greater than the binding energy of the electrons in the inner layer of the element atom, Layer electrons escape to generate holes, and the outer layer electrons will undergo energy level transitions to replenish the holes and emit characteristic X-ray fluorescence at a certain energy. A semiconductor detector is used to receive X fluorescence of all different energies. The X fluorescence is converted into an electrical pulse signal by the detector. After pre-amplification, the multi-channel pulse height analyzer is used to perform signal processing to obtain X-ray intensity distribution spectra of different energy. This spectrum is called the energy dispersion spectrum and also called the X-ray fluorescence spectrum. Finally, the element can be analyzed qualitatively through the track of energy pulses in the energy spectrum. At the same time, the count rate of the pulses in the energy spectrum is proportional to the content of the element, and the element can be analyzed quantitatively.
3. Instrument Structure and Design Principle

The overall structure of the new X-ray fluorescence grade analyzer is shown in Figure 2. Including the main control unit, measurement unit, calibration unit, collimation unit, temperature control unit and power supply unit, the pulp sampling equipment is installed nearby in the mixing tank, and the high-level software for data analysis and processing runs in the PC-side industrial computer; The main control unit uses STM32F407ZET6 series as the microprocessor; The core measurement unit consists of an X-ray tube, a high-voltage power supply, and a semiconductor detector; The calibration unit consists of a detection actuator and a stepper motor control module; The collimation unit includes a collimator and a filter; the temperature control unit is composed of a semiconductor refrigeration sheet and a microcomputer temperature controller; The power supply unit provides 3.3 V and 5.5 V DC power for the main control chip and 24 V DC power for the X-ray tube high voltage power supply; There are two serial communication chips in the main control unit, which are MAX3232 and MAX3485. The main control unit is connected to the X-ray tube in the measurement unit and its supporting high-voltage power supply detector and temperature control unit through the RS-232 communication module, and is connected to the data analysis and processing higher-level software through the RS-485 communication module.

Figure 1. Schematic diagram of grade measurement and analysis of a new on-line grade meter

Figure 2. Overall Structure of Grade Analyzer
4. Master Control Unit Design

4.1. Main Control Unit Hardware Structure

The hardware structure of the main control unit is shown in Figure 3. Select STM32F407ZET6 series as the microprocessor of this embedded system. The system chip includes the STM32F407 minimum system, JTAG debug interface, power supply module, RS-232 and RS-485 communication module, stepper motor control module for standard sample detection actuator, temperature control module, X-ray tube high voltage power control module, and X fluorescence detector control module. The main control unit mainly completes the control of each measurement unit of the chassis, the adjustment of the chassis temperature, and the communication with the host computer.

![Figure 3. Hardware structure diagram of main control unit](image)

The STM32F407 minimum system consists of a microprocessor STM32F407ZET6, external SRAM, EEPROM memory, and a clock crystal. The STM32F407ZET6 is a 32-bit high-performance chip based on the Cortex-M4 core, with a main frequency of 168 MHz. Faster data processing capabilities and rich peripheral interfaces can meet the data processing speed and multi-class requirements of online grade analyzers\cite{13}; External SRAM uses ISSI memory IS62WV51216BLL-55TLI, its memory capacity is 8Mbit, and its memory configuration is 512K * 16; The EEPROM memory is ATMLH751, which can be rewritten multiple times by electronic means; STM32F407 minimum system clock crystal adopts external crystal mode. This circuit connects the crystal to both ends of an inverting amplifier. There are two capacitors connected to the two ends of the crystal while the other capacitor is grounded. The left end of the circuit is connected to the clock crystal IO pin of STM32F407ZET6. The principle of the smallest unit circuit of the microprocessor is shown in Figure 4.
Because the external high-voltage power supply requires a main control unit for +24 V power supply, while microprocessors, semiconductor detectors and stepper motors require 3.3 V and 5 V power supply, Therefore, the power supply module of the main control unit is composed of a switching voltage regulator LM2596 and a forward low-dropout regulator AMS1117. Among them, LM2596 is a step-down power management monolithic integrated circuit, AMS1117 is a forward low-dropout voltage regulator, and the power supply chip provides stable power of 3.3 V and 5 V for each module of the main control unit. The circuit principle of the power supply unit is shown in Figure 5.
The stepper motor control module uses stepper motor driver A4984SLPT, which can convert electrical pulses into angular displacement. When the stepping driver receives a pulse signal, it drives the stepping motor to rotate a fixed angle according to the set direction, and its rotation runs step by step at a fixed angle. The driver controls the rotation of the stepping motor to promote the forward and backward movement of the pulp standard sample block. At the same time, the angular displacement can be controlled by controlling the number of pulses, so as to achieve the purpose of accurate positioning and accurate measurement. The STM32F407 minimum system uses the RS-232 communication chip MAX3232 to communicate with the X-ray tube high-voltage power supply, semiconductor detector and temperature controller, it realizes the conversion from the equipment RS232 data to the main control microcontroller TTL data, and at the same time guarantees the zero-delay automatic transmission and reception conversion and the baud rate adaptation.

The communication between the STM32F407 minimum system and the host PC software uses RS-485 communication chip MAX3485. The circuit principle of the 485 communication module is shown in Figure 6. The "PC11" pin of STM32F407ZET6 is connected to the "TX" of MAX485, and "PC10" is connected to "RX". These two ports complete data transmission in the form of half duplex. At the same time, there are two pins of “EN1” and “EN2” in the MAX485 chip, which are used to control the working status of the 485 chip. When the “EN1” pin is low, the 485 chip is in the receiving state. When the pin is high, the chip is in the transmitting state. The "PD6" interface of the STM32F407ZET6 used in the main control unit is directly connected to these two pins, and the RS-485 transmission and reception status can be controlled by controlling the output level of "PD6". The AB interfaces of MAX485 are connected to the PC terminal through terminal blocks on the board, and finally the measurement data is uploaded and the switch instructions are issued.

4.2. Main Control Unit Software Program Programming

The programming of the main control unit STM32F407 software program is mainly completed in Keil5, and the currently more mature firmware library programming method is used. The programming content includes the STM32F407 kernel assembly register definition, STM32F407 system initialization and system clock configuration functions, interrupt related service functions and interrupt firmware library, STM32F407 on-chip peripheral firmware library, main control chip board-level support package functions and program main functions.

The programming of the main control unit software program requires the support of the STM32F407 on-chip peripheral firmware library. The required on-chip peripheral firmware library is packaged into the Driver folder so that the board supports the calling of package functions and main functions. The interrupt service function is mainly used to set the corresponding interrupt information for program operation, to ensure the timely processing and feedback of interrupt information by the STM32F407ZET6, and to improve the system's operating efficiency and stability. The main control chip board-level support package function is mainly used to set the corresponding interface on the main control unit PCB board, so that the peripheral module interface on the PCB board corresponds to the STM32F407ZET6 pin definition. The block diagram is as follows:
Figure 7. Embedded program block diagram

The embedded main program mainly includes the standard sample and ore sample detection program, the program to query the status of each module of the main control unit, the X-ray tube high-voltage power supply setting program, and the energy spectrum data transmission program to the host computer. The logical relationship between the programs is shown in Figure 26.

The main functions of the program code include:
1. Temperature control module, X-ray tube high voltage power supply control module and X-fluorescence detector control module data receiving and switching control function;
2. Data upload and download functions of VB upper software and lower measurement instruments;
3. Standard sample detection actuator's stepper motor running time and distance control function;
4. Temperature control module temperature data acquisition and thermostat switch control function.

5. Design of Data Modeling for Host Software
For the measurement of pulp grade, instrumental analysis is a relative analysis method, chemical analysis should be used as a benchmark before formal use. The calibration of the detection point of the
online grade analyzer is to make the displayed value of the measured element content at that point move closer to and closer to the test value.

The specific calibration method is that the analyzer records the fluorescence spectrum information of the online pulp flow at the detection point during a complete measurement period. At the same time, during the same detection cycle, the measured pulp flow is intercepted manually at the detection point. The pulp sample is filtered, dried, and cable-separated and then sent to the laboratory for analysis. The above operation results in a single sample. The grade range and concentration range of the measured elements of the sample should cover the use interval and be normally distributed throughout the interval. In order to obtain a calibration sample representative of production, to ensure that the sample can cover most of the pulp grade in the beneficiation process, According to the work statistics of the on-site laboratory team of the ore dressing plant, about 30 single samples need to be collected for each detection point to form a sample. At the same time, the time span for obtaining samples based on the periodic changes of the ore slurry in the beneficiation process should be as long as possible. For this industrial field test, by analyzing the ore pulp test value data accumulated by the workshop test team, a time node that can cover various grade data of the process flow was delineated, and a time span of 15 days was determined. At the same time, in order to meet the needs of the BP neural network training set capacity, 8 sets of effective data of counting rate, laboratory value, and pulp concentration were collected each day at a uniform time, and a total of 120 sets of valid data were collected. Finally, the test value of each single element in the sample is placed in the computer. Set reasonable track parameter files according to the measured elements, and the computer automatically establishes the best mathematical model and calculates the measurement accuracy results of each measured element in the calibration sample according to the appropriate modeling method. The computer automatically detects the grade value of each element in the pulp stream with unknown content according to the established mathematical model to guide the beneficiation production.

The pulp in the beneficiation industry site often contains multiple elements, and the content of each element also affects each other. Therefore, the grade value of one element and the X-ray fluorescence information of various elements in the pulp (the number of element pulses received by the host software) Correlation, the proportion of the grade as the content of ore elements in the pulp and the pulp concentration are also related. At the same time, the copper element grade value has a strong correlation with the copper pulse count rate and the pulp concentration, and a weak correlation with the iron pulse count rate; Similarly, the iron grade value has a strong correlation with the iron pulse count rate and pulp concentration, and a weak correlation with the copper pulse count rate. Therefore, the use of black box data modeling is suitable for the processing of grade data, and the correlation between the data is found through the training of big data. In the field of intelligent algorithms, BP neural network algorithm is one of the most effective multilayer neural network learning methods, its main characteristic is that the signal is transmitted forward, and the error is propagated backward. By continuously adjusting the network weight value, the final output of the network is as close as possible to the expected output to achieve the purpose of training modeling. At the same time, its characteristics of multiple inputs and multiple outputs are also suitable for modeling and analysis of element pulse number and grade value.

Therefore, the BP neural network algorithm is considered for the modeling and analysis of high-level software grade data for the above reasons, adopt Matlab programming method, use Matlab to model BP neural network, generate '.m' executable file 'MCRInstaller' and run it on PC. At the same time, the dynamic link library file 'myOneRegress_1_0.dll' is installed in the VB software to realize the connection between the VB host software and the Matlab data processing library.

![Figure 8. BP neural network schematic diagram for grade data modeling and analysis](image-url)
Data processing adopts BP neural network-based data modeling to improve the accuracy of data modeling[14]. As shown in Figure 6, the typical lead-zinc ore grade data modeling analysis BP neural network consists of an input layer, a hidden layer, and an output layer. The input layer consists of the pulse number of copper and iron elements and the real-time concentration of the pulp. They are measured by the semiconductor detector's X-ray fluorescence pulse number of the pulp element and transmitted to the host software through the STM32F407 main control chip. The hidden layer is the middle layer of the BP neural network. It requires a certain number of hidden nodes. The choice of hidden nodes in the hidden layer is based on the empirical formula:

\[ h = \sqrt{m + n + a} \]  

(1)

Where \( h \) is the number of nodes in the hidden layer, \( m \) is the number of nodes in the input layer, \( n \) is the number of nodes in the output layer, and \( a \) is an adjustment constant between 1 and 10. According to BP neural network modeling experience, the value of \( a \) is selected to 2, so the hidden layer is set to 4 neurons.

The output layer outputs the iron and copper grade values, and the output value is displayed in real time by the upper-level software. By measuring the typical value of the element pulse number and the exact value of the test grade over a period of time, self-learning of the BP neural network in Matlab to modify the weight of each neuron, this self-learning uses the neural network toolbox in MATLAB for network training[15]. The training set of the neural network uses the 120 sets of data collected at the copper ore dressing flotation cell site within 15 days as described above, 20 sets of data are used as verification data after training, and 100 sets are used as training data, each set of data is the pulse count rate of copper and iron, the concentration of pulp, and the corresponding elemental grade test value. The specific implementation steps of the prediction model are as follows: The 100 sets of training data of the training set are normalized and input to the network. The hidden and output layers of the network are set to tansig and logsig functions, the network training function is traingdx, the network performance function is mse, and the number of hidden neurons is set to 4, set the network parameters, the number of network iterations epochs is 5000, the expected error goal is 0.00000001. After setting the parameters, start training the network. After the network has reached the desired error through repeated learning, it finishes learning. The training minimum error convergence curve is shown in fig.9, and the regression convergence of the training data, the measurement network generalization data, and the test data is shown in fig. 10.
In practical use, the regular grade chemical inspection value can be input into the upper software of the analyzer. Relying on a large number of grade chemical inspection values, pulp concentration and corresponding element pulses, the BP neural network data model can continuously self-learn to correct the weight of each neuron, improve the accuracy of the online grade analyzer measurement and Adaptability of sudden changes in pulp grade values.

6. Field Industrial Test Effect
In March 2019, the new X-ray on-line grade analyzer was tested on-site at a mining plant in Shaoxing to test the stability of the instrument and the accuracy of the on-site grade measurement.

Due to the complex conditions of industrial equipment and ambient temperature in the beneficiation plant, and the operation of X-ray tubes and semiconductor detectors has high temperature requirements. Therefore, in the industrial test, we need to understand the filament current, power supply temperature, power supply voltage, tube voltage, tube current of the X-ray tube high-voltage power supply when the grade meter is running, and the detector high voltage, probe temperature, and body temperature of the semiconductor detector. Fig.11 is the monitoring window of the total copper channel on the site of the selection plant in the upper software, where the dotted curve is the intensity distribution spectrum of X-fluorescence of different energy in the measured ore sample, referred to as the X-ray fluorescence spectrum. By analyzing the peak position and peak value, the types and grades of the corresponding elements in the pulp can be obtained. From the top to the bottom of the monitoring window, the real-time data of the semiconductor detector's detector high voltage, probe temperature, body temperature, and filament current, power supply temperature, power supply voltage, tube voltage, tube voltage, and tube current of the X-ray tube high voltage power supply. The data can be used to monitor the running status of the equipment in real time.

![Figure 11. on-site total copper channel monitoring window](image)

The parameters of the actual field operation for a period of time are shown in Table 1, and real-time data at 9 am every day is taken. From the data in Table 1, it can be seen that the parameters of the device are relatively stable during operation, and the parameters only change slightly with the change of ambient temperature, which meets the design requirements of the grade analyzer. The long-term stability of the instrument was verified by industrial field tests.

| Date | Detector X-123 parameters | X-ray tube high voltage power supply parameters | Ambient temperature |
|------|--------------------------|-----------------------------------------------|---------------------|
|      | Detector high voltage    | Probe temperature                             | Body temperature    | Filament current | Power temperature | Tube voltage | Tube current |
| 3.26 | 180                      | -35                                           | 40                  | 1.41545         | 28.57             | 23.21        | 9.978        | 0.0504       | 23          |
| 3.27 | 180                      | -38                                           | 34                  | 1.41533         | 24.14             | 23.16        | 9.978        | 0.0504       | 22          |
| 3.28 | 180                      | -37                                           | 39                  | 1.41245         | 27.55             | 23.16        | 9.978        | 0.0504       | 16          |
| 3.29 | 180                      | -37                                           | 39                  | 1.41245         | 27.55             | 23.16        | 9.978        | 0.0504       | 18          |
| 4.1  | 180                      | -37                                           | 39                  | 1.41533         | 27.33             | 23.11        | 9.993        | 0.0504       | 19          |
| 4.2  | 180                      | -38                                           | 38                  | 1.41245         | 26.74             | 23.10        | 9.993        | 0.0504       | 18          |
It can be known from the X-ray fluorescence spectrum that the peak position and peak value of the X-ray fluorescence measured by the semiconductor detector are the data support for data modeling, by recording the peak position and peak value of the X-ray fluorescence of the measured element over a period, the accuracy of the grade meter measurement can be checked. The peak position and peak value of the X-ray fluorescence of the copper element at the total copper point of the beneficiation shop at the site are shown in Table 2, take real-time data at 9 AM every day. From the data in Table 2, it can be seen that the peak position of the measured X-ray fluorescence has been stable around 912, with a small fluctuation range, and the corresponding peak changes proportionally with the change of the grade value, which meets the design requirements.

| Date | Peak position | Peak   |
|------|---------------|--------|
| 3.26 | 911           | 12900  |
| 3.27 | 912           | 13500  |
| 3.28 | 914           | 13900  |
| 3.29 | 914           | 13400  |
| 4.1  | 913           | 12700  |
| 4.2  | 913           | 12600  |

7. Conclusion
This new X fluorescence online grade analyzer is designed based on STM32F407, with higher integration of main control unit and lower overall power consumption. The use of X-ray high-pressure light tubes and semiconductor detectors ensures the requirements for denuclearization of the instrument. At the same time, the temperature control and the addition of a collimating filter unit improve the stability of the equipment operation. The use of BP neural network modeling improves the accuracy of data modeling and the adaptive ability of data processing. Through industrial tests, the operation stability and measurement accuracy of the new X-ray on-line grade analyzer have met the design requirements. Compared with the previous generation on-line grade analyzer with nuclear source, first of all, the safety of on-site use has been improved, eliminating the potential safety hazards caused by nuclear sources; Secondly, the measurement accuracy of concentrate grade has been improved from about 5% of the previous generation to 2%-3%, which has achieved a more accurate feedback effect on the beneficiation automation process; Finally, the improvement of the measurement count rate resolution brought by the upgrade of the main control unit has expanded the element grade measurement range of the new instrument, and the measurement capability has basically covered various ore elements in the mining industry. The successful development of this new online grade analyzer meets the needs of the beneficiation process for high-precision real-time monitoring of pulp grades, and provides more advanced technical support for the optimization of the entire process automation control of the beneficiation industry.

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