The vacuum control system design in ultracentrifuge chamber

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Abstract

The ultracentrifuge rotor usually works at 30,000 \text{r/min} or more, so centrifuge chamber must maintain a high vacuum to reduce friction of rotor and air. The paper described the vacuum control system design based on MSP430 microcontroller. The system included the MCU, sensors, measurement and conditioning circuitry, vacuum control circuit, power circuit components and so on. Tests proved that the system is stable and reliable performance to meet the ultracentrifuge vacuum measurement and control targets.

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1. Introduction

Work principle of the centrifuge is to achieve to separate, precipitate and concentrate the different substances, in the solution of the test tube placed in the rotor, by high-speed rotating rotor, to extract the necessary ingredients [1]. The centrifuges not only can be used as analysis devices, but also can be used as production equipment, to be applied in many fields. In the process of researching and making the ultracentrifuge whose maximum speed up to 80000r/min, in order to reduce the friction between the rotor and the air, to increase the speed of the centrifuge, there is necessary to control the vacuum and temperature of centrifuge chamber, to meet the speed rotation state high vacuum and low temperature requirements. According to the working mechanism of the ultracentrifuge, the paper designed the vacuum control system based on MSP430 MCU for the vacuum signal sampling, conversion, data processing and display output, and control the vacuum pump, to achieve to control vacuum centrifuge.

2. Vacuum measurement principle

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The system uses a hot cathode ionization vacuum gauge to measure the vacuum. Ionization gauge is made based on the principle that gas ionization is proportional to the gas density. In the fixed structure and fixed-voltage conditions, the emission current $I_e$, the collected current $I_i$ and the pressure $P$ in the vacuum system have the following relationship:

$$I_i = K I_e P$$

In the formula, $K$ is the dimensionless scale factor, which is resolved by the electrode material, shape, relative position and relative potential and other factors constant, and can be simply understood as the ionization efficiency. In this paper, the ionization gauge $K$-value $(0.15 \pm 15\%) \text{ Pa}^{-1}$. Hot-wire power was supplied by the constant voltage, emitting electrons flow $I_e$ was controlled at a fixed value. Obviously, as long as we can measurement $I_i$, the pressure $P$ would be tested.

If the electron flow is too large, while density of gas molecules is lower, the saturation will occur, ion and electron flow is no longer (1) a linear relationship; and the heating current of filament (hot cathode) need to be improved, which will seriously affect the life of the filament [2]. Therefore, in the actual using, the electron flow is generally 0.1mA~10mA. Because the low vacuum pressure $P$ is too small, the value of ion current is in the pA level. In order to improve the sensitivity of the measuring, it has a high requirement to circuit performance.

3. System hardware design

Vacuum control system consists of the ionization vacuum gauge, measuring circuit, the signal conditioning circuit, the core processing unit, vacuum control circuit, data storage circuit, man-machine interface circuit and power supply circuits, etc., and the structure diagram shown in Figure 1. The key issue of ionization vacuum gauge vacuum measurement is to stabilize the emission current of the ionization vacuum gauge, making the collector ion current changes reflect changes in the vacuum.

![Figure 1 System hardware block diagram](image)

3.1 Central control unit

The system used MSP430F247 as a core processor, which is 16-bit reduced instruction set (RISC) microcontrollers, high integration, peripheral-rich, ultra-low power consumption, etc.. So the MCU has been widely used in portable instrumentation, utility detection equipment, smart sensors, motor control and other fields. The operating voltage range is between 1.8V ~ 3.6V. in low-power mode the minimum operating current is only 1μA. MSP430F247 has 2KB RAM and 60KB Flash memory, supporting for partition to use, with 12 internal A/D converter, which can meet the accuracy requirements.
3.2 Measurement and conditioning circuit

Because the ionization gauge collector current is very small, micro-current measurement is usually achieved by the current / voltage conversion method consist of high input impedance op amp [3]. In this paper, with I/V conversion principle, we carried out micro-current measurements, the micro-current measurement system shown in Figure 2.

![Figure 2 Micro-current measurement block diagram](image)

In practice, the general I / V conversion circuit can not meet the small current measurement, so it need to be improved. The op-amp in I/V conversion circuit must meet the following conditions: Input impedance is much greater than the feedback impedance; Bias current is far less than the current measured current; Input offset voltage is smaller; Gain $A$ is high as far as possible; High common mode rejection ratio, and low noise. In view of this, we choose LMC6082 as the op-amp measurement circuit.

Taking into account the practical influence of op-amp bias current $I_B$ to the detection of signals, we designed a current compensation circuit to eliminate the effect, the improved micro-current detection circuit shown in Figure 3 [4]. Compensation current is $i$, op-amp bias current is $I_B$, ion current is $I_i$, the circuit output voltage is:

$$V_{ol} = -(1 + \frac{R_f}{R_2})R_f(I_i + i - I_B)$$

(2)

From the above equation, when $i = I_B$, they can eliminate the impact of the bias current to the ion current measurement, so as to achieve ideal measurement results. Then, the circuit output is:

$$V_{ol} = -(1 + \frac{R_f}{R_2})R_f \cdot I_i$$

(3)

Visibly, $R_f$ do not have to get great that it can get current conversion and amplification effect by selecting the ratio of $R_f/R_2$. As the resistor $R_6$ has diverted the measured current, in order to ensure the accuracy of measurement, usually we take $R_6 \geq (10 \sim 100) \cdot R_f$. With the P-channel FET output characteristics, on-state, changes of $V_{ds}$ will not make the drain current change, the voltage on $R_{W1}$ tap is essentially the same, which will not affect the compensating current.

![Figure 3 Micro-current detection circuit](image)
As the output voltage of I/V conversion is relatively weak, and susceptible to noise interference, so it choose three op amp structure to amplifying voltage shown in Figure 4 [5]. Operational amplifiers A1 and A2 form the first stage differential amplifier, A3 form the second stage differential amplifier circuit, the front output $V_{o1}$ added to the A1-phase side, $R_{11}$, $R_{12}$, $R_{13}$ and $R_{17}$ form the feedback network, which is the voltage series negative feedback. In order to improve the circuit's ability of resisting to common mode interference and suppress effects of drift, according to the principles of symmetry up and down, take $R_{12} = R_{13}$, $R_{14} = R_{15}$, $R_{16} = R_{17}$, the output voltage is:

$$V_{o2} = (V_i - V_1) \frac{R_{34}}{R_{13}} = -\left(1 + \frac{2R_{34}}{R_{13}}\right) \frac{R_{34}}{R_{13}} V_{o1}$$

(4)

To eliminate interference to ion exchange signals, after the voltage amplified linearly, it increases a second-order active low-pass filter, and the filter circuit shown in Figure 5. To simplify the calculation, to take $R_{20} = R_{21} = R_{22} = R_{23} = R = 3.57\,k\Omega$, $C_7 = C_8 = C = 4.7\,\mu F$, the filter cutoff frequency is 10Hz, which can filter out AC interference. Through a conditioning circuit, the signal of low-pass filter converts to the signal range of 0 ~ 3.3V, input A / D interface of MCU.

3.3 Vacuum control module

Vacuum control module mainly controls a compression pump and a diffusion pump through relays, to achieves two-step vacuum control. When the indoor centrifugal vacuum is lower, the module controls large displacement pump to exhaust air, the process is defined as roughing; When the centrifuge reached to 0.1Pa indoor vacuum, the compressed air pump can not achieve satisfactory results, we must change with the proliferation of small displacement pumps to extract air, the process is defined as fashioning. In course of the centrifuge working, to ensure the indoor centrifugal vacuum device is down due to other factors, we must keep fashioning process work. The relay driver control circuit shown in Figure 6.

4. System software design

System software design adopt modular programming approach, which consist of the main program and several subroutines software modules. Each module can control a particular mandate to achieve a specific
function. The system includes A/D sampling subroutine, data processing subroutine, the vacuum control subroutine, the data storage module, display procedures etc.

4.1 A/D sample procedure

Sample procedure is achieved by ADC MSP430F247 microcontroller chip. The ADC provides four kinds of conversion modes: single-channel single conversion, serial-channel single conversion, single-channel multiple conversions, and sequence multiple-channel conversions [6]. Single-channel multiple conversions mode is selected here. After each conversion, the conversion results will automatically convert into the corresponding storage register ADC12MEMx, and the end of conversion is marked by the corresponding interrupt flag. Because the system requires the user to set the sampling frequency, so a fixed time interval data extraction methods is adopted to achieve sampling. The conversion results of A/D converter are updated in real time, but to take the data in the conversion array is accomplished through the timer interrupt.

4.2 Data processing subroutine

Ion current output is very weak, very vulnerable to external interference. Therefore, the acquisition value of the measurement voltage signal pulse using anti-interference filter average filtering method. Specific process is: continuous sampling n data, removing a maximum and a minimum value, then calculate the arithmetic mean of the n-2 data (n=10), the arithmetic mean is voltage measurement value. This method can effectively overcome the glitches caused by accidental factors, and eliminate the interference caused by the sampling pulse deviation.

5. Experimental results and analysis

Table 1 Compare standard vacuum with measured value

<table>
<thead>
<tr>
<th>Standard value /×10⁻⁵Pa</th>
<th>Measured value /×10⁻⁵Pa</th>
<th>Relative error</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 1.3</td>
<td>1.1</td>
<td>15.4%</td>
</tr>
<tr>
<td>2 8.2</td>
<td>6.6</td>
<td>19.5%</td>
</tr>
<tr>
<td>3 15.3</td>
<td>18.2</td>
<td>-19.0%</td>
</tr>
<tr>
<td>4 63.2</td>
<td>51.0</td>
<td>19.3%</td>
</tr>
<tr>
<td>5 120.4</td>
<td>100.3</td>
<td>16.7%</td>
</tr>
<tr>
<td>6 456.7</td>
<td>378.1</td>
<td>17.2%</td>
</tr>
<tr>
<td>7 1450.6</td>
<td>1282.3</td>
<td>11.6%</td>
</tr>
<tr>
<td>8 3371.1</td>
<td>2956.5</td>
<td>12.3%</td>
</tr>
<tr>
<td>9 5735.6</td>
<td>5076.0</td>
<td>11.5%</td>
</tr>
<tr>
<td>10 8765.7</td>
<td>7862.8</td>
<td>10.3%</td>
</tr>
</tbody>
</table>

In order to study the performance of the system, we install a magnetic rotor gauge in a vacuum calibration chamber as a reference standard. The magnetic rotor gauge measuring range is 1.0×10⁻¹\text{Pa} ~
1.0×10^{-5}\text{Pa}. The results show that the system measurements and magnetic measurements rotor rules are basically the same, the error range of ± 20% or less, it reach to system design specifications, the measurement results compared in Table 1 and Figure 7.

6. Conclusion

The vacuum control system is an important part of the ultracentrifuge, which provide the rotor a high-vacuum environment to reduce the friction between the rotor and the gas, in order to achieve the required speed requirements. What is studied in this paper has been carefully vacuum control system hardware and software design, and the online test shown that it can meet the speed centrifuge vacuum measurement and control targets, the effective range of vacuum measurement and control is 1.0×10^{-1}\text{Pa}~\text{~}1.0×10^{-5}\text{Pa}; and the successful development of the ultracentrifuge fill the domestic ultracentrifuge technology blank.

References


