A Novel Algorithm to Differential Scanning Calorimeter Temperature Control Based on Liquid Nitrogen Feedforward Technique

IOP Conf. Series: Materials Science and Engineering **569** (2019) 052021 doi:10.1088/1757-899X/569/5/052021

A Novel Algorithm to Differential Scanning Calorimeter Temperature Control Based on Liquid Nitrogen Feedforward Technique

YU Yang[1,2,3*], HU Jingtao[1,2], JIA Yang[1,2], WEI Laixing[1,2], SUN Wei[1,2]

[1]Shenyang Institute of Automation Chinese Academy of Sciences, Chinese Academy of Sciences, Shenyang, Liao Ning ,110016, China

[2]Institutes for Robotics and Intelligent Manufacturing, Chinese Academy of Sciences, Shenyang, Liao Ning, 110016, China

[3]University of the Chinese Academy of Sciences, Beijing 100049, China

[*]Corresponding author's e-mail: yuyang1@sia.cn

Abstract. Differential scanning calorimeter (DSC) is an important instrument in the thermal analysis which could be pervasively applied to property analysis of inorganic compounds, organic compounds and drugs. An effective temperature control technique could ensure DSC thermal experimental accuracy. Unlike the traditional temperature control methods which have limited temperature control ranges and change rates, this paper presents a novel algorithm which exploits the liquid nitrogen feedforward technique to control the temperature of DSC. To be specific, the proposed approach fully takes advantage of the characteristic of the liquid nitrogen that has the ability to rapidly decrease the objective temperature. Furthermore, a cold-heat synergistic feedforward-feedback control is utilized. On one hand, the feedforward controller is able to avoid the singularities for the amount of liquid nitrogen. On the other hand, the feedback controller is able to compensate for the temperature errors due to external disturbances. The proposed algorithm has been applied to the DSC. The experimental results and analysis prove that the control method is able to change the temperature accurately within a wider range.

1. Introduction

Differential scanning calorimeter (short as DSC) is a thermo-analytical instrument that is able to measure the difference of the heat amount between the sample and the reference, while the temperature is designed to be increased [1]. DSC has been widely used in the performance testing and quality control of the material and food processing industry [2]. In DSC, both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed to enable the temperature of the sample holder increases or decreases linearly as a function of time. Only when the temperature of the DSC is controlled linearly, thermo-dynamic parameters are able to be measured accurately and effectively [3]. Through this way, the heating or cooling process could be stable and non-overshoot, and the precision of the DSC temperature control could be guaranteed [4]. Therefore, it is of great significance to study the temperature control algorithm for DSC.

The temperature control is a hot topic that has been researched for many years, especially in process control. Hasegawa[5] designed a feedback temperature control system with two heaters to achieve high-precision temperature control effect for cryogenic coolers. Amine[6] proposed a control strategy for complex and nonlinear systems, based on a parallel distributed compensation and the method was applied to a small greenhouse to control its inside temperature by variation in ventilation rate inside the process. Li [7] proposed a high precision temperature control algorithm combining hierarchical control algorithm, integral separation PI algorithm, feedforward algorithm and lag compensation algorithm which was applied to immersion lithography. Du [8] proposed a PID control algorithm with constant set values in small zones for constant temperature control of the furnace and analysed the parameter selection principle under the different control conditions. Zhou [9] proposed a fuzzy PID control algorithm to achieve the constant speed temperature changing control. Wang [10] designed a Smith predictor PI controller for large time-delay temperature control. In a nutshell, above-mentioned temperature control algorithms are mainly applied to the petroleum, chemical and steel production processes. These processes have the characteristics of large inertia, large delay and strong non-linearity [11]. Basically, the cycle of these control systems is long, and it brings s great challenge for steady-state control. However, the state-of-the-art has, somehow, neglected the dynamic adjustment process, especially in the case of low-temperature dynamic control. Moreover, DSC is a precise thermal analysis instrument, and the temperature control of the DSC is supposed to have shorter control periods, faster dynamic responses and higher control precisions, in the low-temperature scenarios [12].

For the time being, there is little research on the temperature control algorithm in the low-temperature zone of DSC. The existing DSC mostly exploits heating wire as the means of temperature control, and the heating process responds quickly, steadily and accurately. However, for the cooling process, it is difficult to achieve a rapid and stable temperature decrease by reducing the heating amount from the heating wire. The mere use of heating wire is incapable of achieving the high-precision control effects in the low-temperature zone. To resolve the above issues, this paper presents a novel temperature control algorithm that is suitable for DSC wide-range temperature control, including the low-temperature zone. The main contributions of this paper are as follows:

- The heat transmission model has taken the liquid nitrogen rapid cooling property into consideration and incorporated the balance-point optimization mechanism.
- The compound control (both feedforward control and feedback control) for DSC is designed. The liquid nitrogen feedforward controller is designed to provide precise control for the cooling process. The feedback controller is designed to correct the control effect of the liquid nitrogen and to compensate the deviation caused by disturbance or other factors.
- The control algorithm is applied to the temperature control of DSC. The experimental results show that the proposed temperature control algorithm can meet the requirements of a wide range temperature change and high control precision.

2. The Control Algorithm base on the Liquid Nitrogen Feedforward

Considering the high cost of liquid nitrogen and the low control precision of liquid nitrogen solenoid valve, the feedforward controller of liquid nitrogen is designed based on the energy needed for constant rate cooling, and the open-loop control of liquid nitrogen is carried out.[13] Moreover, the closed-loop control of heating wire is introduced to realize the precise control through the quickly adjusting the voltage of the heating wire, which could be helpful for compensating the errors caused by disturbance and other factors. A compound control system which consists of the feedforward controller as the main controller and the feedback controller as the supplementary controller is formed to reduce the consumption of electric energy and liquid nitrogen cost on the basis of ensuring the control accuracy. The structure of the control algorithm is shown in Figure 1. The control output of the heating wire controller is u_h. It indicates the voltage value of heating wire. The control output of the liquid nitrogen controller is u_c. It indicates liquid nitrogen flow.

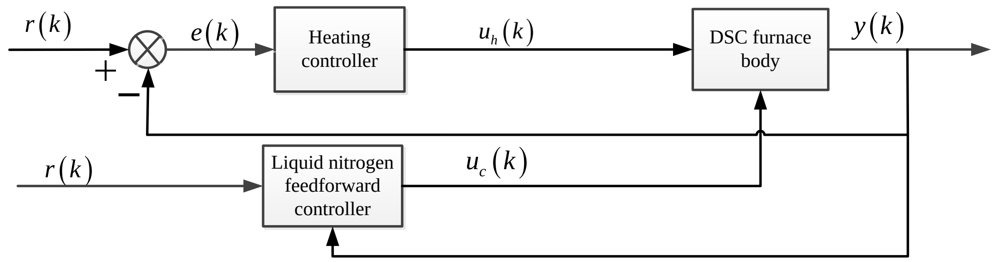

Figure.1 The structure of the synergistic temperature control algorithm of DSC combined with liquid nitrogen feedforward control

2.1. Liquid nitrogen feedforward controller

In order to calculate the relationship between the flow rate of liquid nitrogen and the temperature change in the furnace, the energy conservation law is used to obtain the following results:

$$Q_{o1} + Q_{o2} = Q_{p1} + Q_{p2} + Q_{p3} \qquad (1)$$

where Q_{o1} is the internal heat discharged by liquid nitrogen; Q_{o2} is the energy dissipation between the furnace and the environment; Q_{p1} is the internal heat storage in furnace body; Q_{p2} is the internal heat storage of the gas in the furnace; Q_{p3} is the internal heat discharged by the gas in the furnace.

$$Q_{o1} = \frac{q \cdot \delta \cdot \rho}{M} r \cdot h \qquad (2)$$

$$Q_{o2} = K \cdot A_{out} \left(T_{melt} - T_a\right) \qquad (3)$$

$$Q_{p1} = c_{p,s} \cdot m_s \left(T_s - T_0\right) \qquad (4)$$

$$Q_{p2} = c_{v,g} \cdot m_g \left(T_g - T_0\right) \qquad (5)$$

$$Q_{p3} = F_{out,g} c_{V,g} T_{out,g} \qquad (6)$$

where q is the mass of liquid nitrogen discharged from liquid nitrogen solenoid valve in unit time; δ is the opening time for liquid nitrogen solenoid valve; ρ is the liquid nitrogen density; M is the molar mass coefficient of liquid nitrogen; r is the vaporization heat coefficient of liquid nitrogen; and h is the vaporization proportionality coefficient of liquid nitrogen; A_{out} is the surface area of the insulation wall; T_{melt} is the furnace temperature; T_a is the ambient temperature; K is the comprehensive heat transfer coefficient; m_s is the mass of furnace body; $c_{p,s}$ is the specific heat capacity of the furnace body; T_s is the temperature of the furnace body; T_0 is the initial temperature of the furnace body; m_g the mass of the accumulated gas in the furnace; $c_{v,g}$ is the specific heat capacity of the gas in the furnace; T_g is the temperature of the gas in the furnace; $F_{out;g}$ is the mass of the discharged gas; $c_{v,g}$ is the specific heat capacity of the gas in the furnace; $T_{out,g}$ is the temperature of the discharged gas.

Formula (2) - (6) are introduced into formula (1):

$$\frac{q \cdot \delta \cdot \rho}{M} r \cdot h + K \cdot A_{out} \left(T_{melt} - T_a\right) = c_{p,s} \cdot m_s \left(T_s - T_0\right) + c_{v,g} \cdot m_g \left(T_g - T_0\right) + F_{out,g} c_{V,g} T_{out,g} \qquad (7)$$

The formula (7) can also be converted to:

$$u_c = q \cdot \delta = \frac{M}{\rho \cdot r \cdot h}\left[c_{p,s} \cdot m_s \left(T_s - T_0\right) + c_{v,g} \cdot m_g \left(T_g - T_0\right) + F_{out,g} c_{V,g} T_{out,g} - K \cdot A_{out} \left(T_{melt} - T_a\right)\right] \qquad (8)$$

According to formula (8), there is a corresponding relationship between the liquid nitrogen flow rate and the furnace temperature. The feedforward control volume is the liquid nitrogen flow value corresponding to the temperature setting value set at the cooling rate.

2.2. Feedback controller

In the temperature control system of DSC, the feedforward control of liquid nitrogen is used as the main controller to provide the main energy needed for cooling, and the feedback controller of the heating wire is used as the auxiliary controller. The function of the feedback controller is to compensate the control precision of the solenoid valve through the quickly adjusting the voltage of the heating wire. At the same time, the deviation caused by disturbance or other factors is compensated by feedback. The heating wire feedback controller adopts PID controller. [14]

Control increment of heating wire feedback controller:

$$\Delta u_1 = k_p\left(e(k)-e(k-1)\right)+k_i e(k)+k_d\left(e(k)-2e(k-1)+e(k-2)\right) \quad (9)$$

Control amount of heating wire feedback controller:

$$u_h(k)=\Delta u_1(k)+u_h(k-1) \quad (10)$$

where $\Delta u_1(k)$ is the control increment of heating wire controller at time k; $u_h(k)$ is the control variable of the heating wire controller at time k; $e(k)$ is the deviation between the set-point and the actual value of temperature at time k; $e(k-1)$ is the deviation between the set-value and the actual value of temperature at time k-1; $e(k-2)$ is the deviation between the set-value and the actual value of temperature at time k-2; k_p is the scale factor of the PID controller; k_i is the integration factor of the PID controller; k_d is the differential factor of the PID controller.

3. Experimental Result

3.1. The temperature control system of DSC

The liquid nitrogen cooling method is applied to the DSC (ZF-DSC-D2H) shown in Figure 2. The hardware structure of the temperature control system is shown in Figure 3.

Figure. 2 the DSC of version ZF-DSC-D2H

The temperature control system of the DSC consists of three parts: signal acquisition and processing subsystem, microprocessor subsystem and signal output subsystem. The signal acquisition part includes a temperature sensor, voltage conversion circuit and A/D conversion module. The platinum resistance is chosen as the temperature sensor due to its high precision. Temperature signal of the heating furnace is collected by a platinum resistance. Then the temperature is converted to the voltage ranging between 0V and 5V by using the voltage conversion amplifying circuit. Afterwards, the voltage signal is transmitted to the signal microprocessor by the SPI interface through the A/D conversion module. Due to the limitation of volume and function, the DSC uses the single-chip microcomputer (R5F21346) as the micro-processing unit. The temperature falling rate is calculated according to the requirements of the thermal analysis experiment. And the real-time control amount of the heating controller and the real-time control quantity of the cooling controller are obtained, and the real-time control quantity is outputted in the form of PWM wave. The signal output system includes a phase modulation control module and relay control module. The phase modulation control module includes thyristor and phase-shift control circuit, and the relay control module includes a relay and a solenoid valve. Through the phase modulation control module, the control quantity of the heating controller is converted to the voltage value of the 0-5V. The voltage of the trigger circuit is phase-shifting and the heating power of the heating wire is changed to realize the control of the temperature. Through the relay control module, the control quantity of the cooling controller is converted to the opening degree of the solenoid valve, and the flow rate of liquid nitrogen is changed to control the temperature.

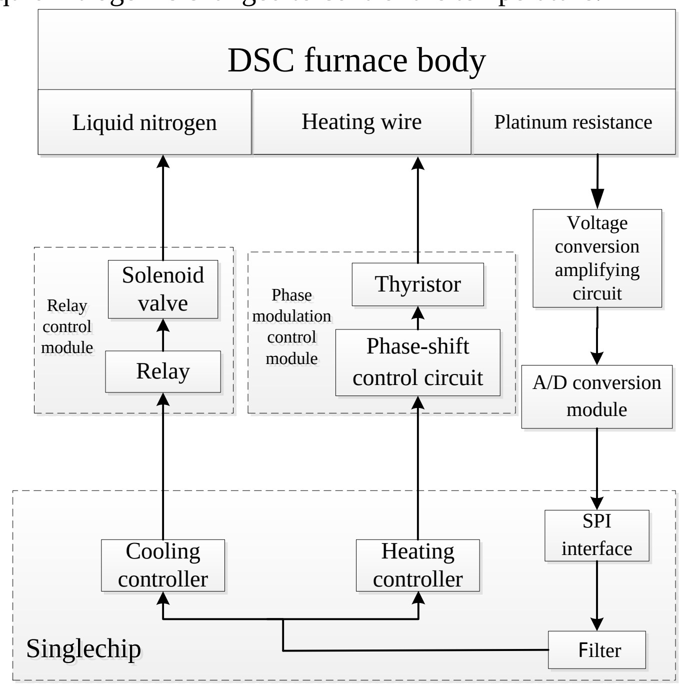

Figure. 3 The hardware structure of the temperature control system

3.2. Calculation of the feedforward control amount

In the experiments, the amount of the liquid nitrogen feedforward control is obtained by the off-line look-up table [15], because the mechanism of heat transfer process in DSC furnace is complex and the thermodynamic parameters are difficult to identify. Firstly, the DSC furnace body is heated to the maximum temperature of 600℃. Under different liquid nitrogen control functions, the cooling curve is obtained by collecting the temperature of the furnace body. Secondly, the temperature range of DSC is divided into 16 sub-temperature zones with a 50℃ interval. Finally, the cooling rate at the boundary of the sub-temperature zones is obtained by differential operations of the cooling curve. The liquid nitrogen feedforward control amount, the cooling rate at the boundary of the sub-temperature zones and the temperature at the boundary of the sub-temperature zones are shown in Table 1. In the process of temperature control, the amount of the liquid nitrogen feedforward control is calculated by the formula (11).

$$u_c = \frac{PV - Tem\_l_i}{Tem\_h_i - Tem\_l_i} \cdot u_{ci} \quad (11)$$

where PV is the current temperature value; PV is in the temperature range of $[Tem\_l_i, Tem\_h_i]$; u_{ci} is the liquid nitrogen feedforward control amount of the Tem_l_i.

The starting temperature of feedforward control is the temperature that the natural cooling rate is equal to the predefined cooling rate. The natural cooling rate is calculated offline. The DSC furnace body is heated to the maximum temperature; then the heating amount is gradually reduced. The slope at each temperature point is obtained by the differentiation of the natural cooling curve at each temperature point. When the cooling rate is lower than the natural cooling rate, liquid nitrogen feedforward control is added.

Table 1.The liquid nitrogen feedforward control variable under the different cooling rates

100℃/min		50℃/min		10℃/min	
Temperature	liquid nitrogen control variable	Temperature	liquid nitrogen control variable	Temperature	liquid nitrogen control variable
-200	90	-200	75	-200	80
-150	90	-150	65	-150	65
-100	85	-100	55	-100	55
-50	80	-50	45	-50	45
0	80	0	35	0	35
50	80	50	30	50	25
100	75	100	25	100	20
150	75	150	20	150	17
200	70	200	15	200	14
250	70	250	13	250	12
300	65	300	10	300	10
350	60	350	8	350	5
400	55	400	5	400	0
450	50	450	0	450	0
500	45	500	0	500	0
550	40	550	0	550	0
600	35	600	0	600	0

3.3. Experimental results of DSC temperature control

The temperature rate is set at 10 ℃/min. Two group of constant speed cooling experiment are carried out by the combined PID algorithm [16] and the control algorithm proposed in this paper. The control effect is shown in Figure 4 and Figure 5. The ordinate is the temperature (℃) and the abscissa is time (min).

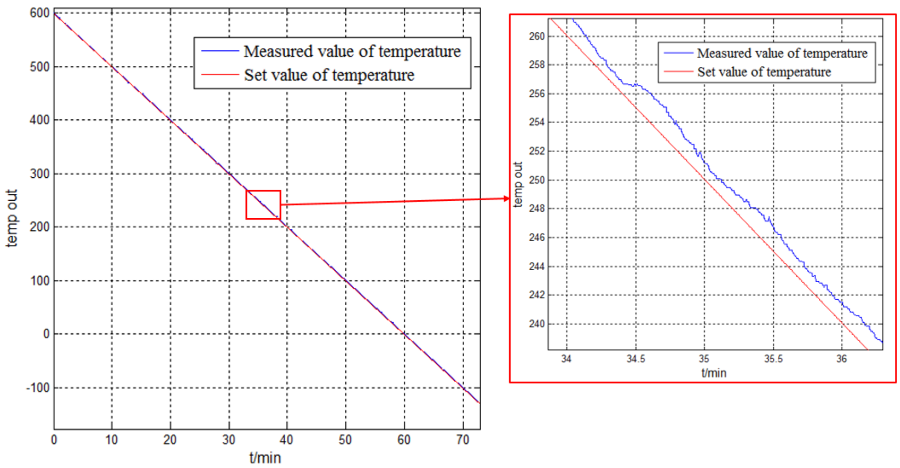

Figure.4 The control effect of the conventional combined PID algorithm

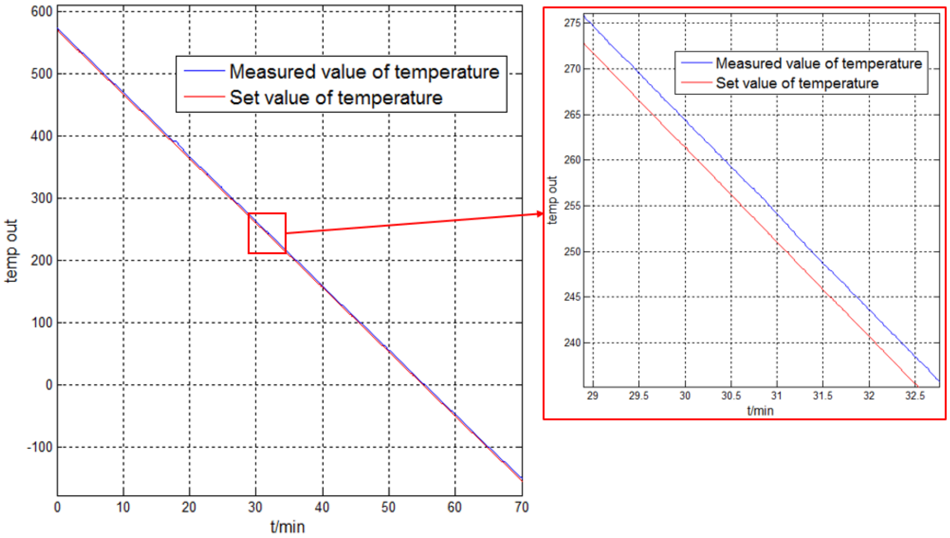

Figure.5 The control effect of the control algorithm proposed in this paper

3.4. Control effect evaluation

The temperature control effect of DSC is evaluated by the linearity of temperature curve and the accuracy of temperature control. The linearity of the temperature curve indicates the fluctuation degree of temperature, which can be evaluated by the error variance. The error variance represents the linearity of the controlled temperature, given by Eq. 12.

$$J = \frac{1}{n}\sum_{i=1}^{n}\left(e_i - \bar{e}\right)^2 \tag{12}$$

The accuracy of temperature control is evaluated by the deviation of the cooling rate.[17] The formula of the cooling rate deviation is as follows.

$$\Delta v = \left(\frac{T_0 - T_{10}}{100} - 1\right) \times 100\% \tag{13}$$

where Δv is the deviation of the cooling rate; T_0 is the temperature at the initial time; T_{10} is the temperature at the tenth minutes.

The error variance and the deviation of the cooling rate are shown in Table 2.

Table 2.The temperature control effect evaluation

Temperature control algorithm	The error variance	The deviation of the cooling rate
The combined PID algorithm	0.3514	1.3%
Ours	0.1901	0.6%

By analyzing the control effects of the two control algorithms shown in Figure. 4 and Figure. 5, it can be found that the temperature control effect of the conventional combined PID algorithm fluctuates seriously. Through Table 2, it can be seen that the error variance of the proposed control algorithm is larger than ours. The cooling rate deviation values in Table 2 show that the cooling rate deviation of the control algorithm meets the performance requirements of Class A instruments required by the verification regulations, which indicates that the control algorithm proposed in this paper is able to satisfy the requirements of temperature control by DSC.

4. Conclusion

According to the control requirement of DSC with a wide range and high precision, a novel algorithm based on liquid nitrogen feedforward is proposed for DSC temperature control. The feedforward controller of liquid nitrogen is used as the main controller to provide the main energy needed for cooling, and the feedback controller of the heating wire is used as the auxiliary controller. The control algorithm proposed in this paper is used on DSC. The results show that the proposed temperature control algorithm can meet the requirements of the wide range temperature variation. It has a remarkable effect on improving the precision of temperature control and the control performance meets the requirements of high precision thermal analysis instruments.

References

[1] LIU ZH H,XU G H,ZHANG H L. Thermal analysis calorimeter and its application[M], Chemical Industry Press,2011.

[2] ZENG Y . The Development of Measurement and Control System of A High Accuracy Differential Scanning Calorimeter[D]. China Jiliang University,2014.

[3] Broadhurst C L, Schmidt W F, Nguyen J K, et al. Continuous gradient temperature Raman spectroscopy and differential scanning calorimetry of N-3DPA and DHA from -100 to 10°C.[J]. Chemistry & Physics of Lipids, 2017(204):94-104.

[4] Paiva F L, Calado V M A, Marchesini F H. On the use of Modulated Temperature Differential Scanning Calorimetry to assess wax crystallization in crude oils[J]. Fuel, 2017(202):216-226.

[5] Hasegawa Y , Nakamura D , Murata M , et al. High-precision temperature control and stabilization using a cryocooler[J]. Review of Scientific Instruments, 2010, 81(9):354.

[6] Amine C , Elyes F , Abdelkader M . Stabilization Using a Discrete Fuzzy PDC Control with PID Controllers and Pole Placement: Application to an Experimental Greenhouse [J]. Journal of Control Science and Engineering, 2011:1-9.

[7] Li X , Zhao Y , Lei M . High precision and stability temperature control system for the immersion liquid in immersion lithography[J]. Flow Measurement & Instrumentation, 2016, 318-325.

[8] DU J Q,GAO SH Q,LUO CH. Control method with control quantum varying in the form of arbitrary function based on PID algorithm[J].Chinese Journal of Scientific Instrument, 2011, 32(6):1317-1323.

[9] ZHOU L Y,ZHAO G S. Application of fuzzy-PID control algorithm in uniform velocity temperature control system of resistance furnace[J]. Chinese Journal of Scientific Instrument, 2008, 29(2):405-409.

[10] WANG M,ZENG Q SH,LIU CH. The Microprocessor Based Control for the Temperature Chamber in the Test Tables[J]. Journal of Chinese Inertial Technology, 1998,6(4):89-93.

[11] Yamasaki H, Morita S. Temperature dependence of isothermal curing reaction of epoxy resin studied by modulated differential scanning calorimetry and infrared spectroscopy[J]. Journal of Molecular Structure, 2016, 11(24):249-255.

[12] Zhang D, Atkinson H V, Dong H, et al. Differential Scanning Calorimetry (DSC) and Thermodynamic Prediction of Liquid Fraction vs, Temperature for Two High-Performance Alloys for Semi-Solid Processing (Al-Si-Cu-Mg (319s) and Al-Cu-Ag (201))[J]. Metallurgical & Materials Transactions A, 2017, 48(10):4701-4712.

[13] Xin L, Zhang Z, Deng D, et al. Study design of container intelligent temperature controller[J]. Electronic Measurement Technology, 2017(5):37-40.

[14] DE Seborg ,TF Edgar, DA Mellichamp ,FJD Iii. Process Dynamics and Control [M]. John Wiley & Sons, Inc, 2011.

[15] Wei H, Gong G, Li N. Improved look-up table method of computer-generated holograms[J]. Appl Opt, 2016, 55(32):9255-9264.

[16] WU B,YANG L,DUAN C T. Realization of temperature control system of differential scanning calorimeter based on PID decision fuzzy controller[J]. Machinery,2012, 50(3):75-77.

[17] Zhang M, Morris K. Sensor Choice for Minimum Error Variance Estimation[J]. IEEE Transactions on Automatic Control, 2017 (99):1-16.