# OPTIMIZATION OF TEMPERATURE CONTROL IN LIQUID FLOW CRYOSTATS

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In order to optimize the temperature control in cryosystems based on liquid flow cryostats, the thermal properties of the substance, of which the structural elements of the work chamber of cryostat are made, have been taken into account. The results of the experimental cryostat control in the temperature range 4.2–350 K are used to demonstrate that the developed device enhances the accuracy and the efficiency of cryosystems.

### 1. Introduction

In modern cryosystems, the temperature of a specimen under investigation, which is located in the work chamber of a cryostat together with a temperature sensor, is controlled by maintaining the "cold-heat" balance in it [1]. There are a number of methods for thermostatting which include, in particular, the radiant heat exchange with the environment, heat exchange with a cooled surface through a mechanical contact, heat-exchange gas, and immediate heat transfer to a refrigerant. Consequently, there are several methods of temperature control and stabilization in cryostats which affect their design. Namely, these are:

1. The Swenson method [2], in which the temperature is varied in a wide range by changing the mass rate of a refrigerant flow that moves in the work chamber. For the temperature control of an investigated object to be more exact, an electric heater which is located either on the external wall of the chamber or immediately in the refrigerant vapor flow is used.

2. The method of forced blow-off of an object with a gas flow characterized by a given temperature. The method was first developed by Knox and Cathbert [3] and then elaborated by the authors [4]. Here, the temperature of a cooled object changes owing to a variation of the refrigerant temperature rather than the refrigerant mass flow.

There are also cryostats of other types, in which the object to study is located immediately in the refrigerant or in vacuum on a coldfinger that has a direct contact with the refrigerant. Since the wide-range temperature control is not effective in such cryostats, they are not considered below.

It is worth noting that, when a cryostat construction is being designed, the thermal characteristics of materials the cryostat is to be made of are taken into account. For instance, the most widespread material for manufacturing the work chamber of a cryostat is copper, because its heat conductivity is among the highest in comparison with those for other metals. In flow systems with the temperature control carried out by the Knox–Cathbert method (leading western firms are mainly oriented to fabricate temperature-control systems of this type), due to the turbulence of the gas flow and the temperature gradient at heating, the achieved temperature stability amounts to  $\pm 0.1$  K. Such cryosystems reveal a large refrigerant consumption, as well as a temperature overcontrol after the given value has been attained, which is also a considerable drawback in the case where a precision cryostatting is needed, in particular, when studying the phase transitions in substances.

Cryostats belonging to the Unified Thermoregulated Cryogenic Systems (UTRECS) are based on the Swenson method developed further by the authors [5–8]. They include cryostats of the liquid flow type, the design of which allows the laminar flow of a refrigerant through the temperature-controlled chamber to be obtained. A heat exchanger and a heater are placed on the external side of the chamber, which provides the temperature equalizing between refrigerant vapors and the chamber.

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In the chamber, there is a special frame with a specimen and a temperature sensor. In this geometry, the refrigerant flows through the cryostat work chamber owing to an excess pressure which is created above the liquid mirror in the tank with a liquid refrigerant. The "cold-heat" balance, which is needed for maintaining a given temperature, is provided making use of a temperature regulator, by vanishing a mismatch between the temperature sensor signal, which corresponds to an actual temperature of the studied specimen, and the given temperature. However, since the thermal properties of substances the cryostat work chamber is made of depend on the temperature, the wide-range temperature control is accompanied by the temperature overcontrol and temperature oscillations. The control will be more effective, if the amount of heat that is supplied into the thermostat chamber is put in agreement with the temperature dependence of the heat capacity of a work chamber material. For a number of materials – including copper which is widely applied for the fabrication of heat exchanging chambers – their temperature dependences of heat capacity are known [9–12]. For today, we do not know such cryosystem constructions, where this dependence is taken into account.

This work aimed at developing a new approach in cryostatting which would make allowance for the temperature dependence of the heat capacity of a material the work chamber of the cryostat is made of, in order to increase the accuracy of the temperature fixation, avoid the temperature overcontrol, and increase the cryostat efficiency.

#### 2. Theoretical Analysis

The constructional elements of a work chamber of the UTRECS cryostat system are fabricated of copper. The temperature dependence of its heat capacity  $C_{\rm Cu}$  is exhibited in Fig. 1 [6]. It is known [6] that  $C_{\rm Cu}$  is proportional to the squared temperature at low temperatures (20 - 70 K) and weakly depends on it at high temperatures (above 200 K). The temperature point  $T_{\rm lim} = 200$  K can be selected as a threshold value which separates the ranges with drastically different thermal properties of copper.

In the stationary regime and when the refrigerant flow is laminar, an equilibrium state is attained in the thermostatting chamber of a cryostat. In this case, the amount of heat brought into the chamber,  $Q_c$ , is equal to the difference between the heats that is obtained due





Fig. 1. Temperature dependence of the heat capacity of copper

to heating,  $Q_h$ , and extracted at cooling,  $Q_{fr}$ ,

$$Q_c = Q_h - Q_{fr}.\tag{1}$$

The fulfillment of Eq. (1) is provided by controlling the parameters of executive devices which include the voltage across the heater  $U_h$  and the magnitude of refrigerant vapor flow, the latter being governed by the cooling control voltage  $U_{fr}$ .

If the chamber temperature  $T_c$  varies by  $dT_c$  within the time interval dt, the thermal balance equation (1) can be rewritten as

$$C_{\rm Cu}m_c\frac{dT_c}{dt} = P_h - P_{fr},\tag{2}$$

where  $m_c$  is the work chamber mass, and  $P_h$  and  $P_{fr}$  are the heating and cooling powers, respectively.

# 3. Experimental Technique

Since the power supplied to the chamber is proportional to the squared heating voltage  $U_h$ , it is necessary to design such a working mechanism for executive devices that the control heating voltage should depend linearly on the chamber temperature, in order to simulate the square-law temperature dependence of the heat capacity. Let us introduce the control parameter  $\lambda$  which approximately corresponds to the temperature dependence of the chamber material heat capacity:  $\lambda = \frac{T_c}{T_{\rm lim}}$  at  $T_c \leq 200$  K, and  $\lambda = 1$  at  $T_c > 200$  K. Then, the maximal voltage across



Fig. 2. Block diagram of the control device

a heater  $U_{hmax} = \lambda U_{ref}$ , where  $U_{ref}$  is the reference voltage for a heater, and the maximal capacity produced by it is

$$P_{h\max} = \frac{U_{h\max}^2}{R_h} = \lambda^2 \frac{U_{\text{ref}}^2}{R_h},\tag{3}$$

where  $R_h$  is the heater resistance.

To provide the maximal heat exchange between refrigerant vapors and the work chamber, the former pass through a capillary-heat exchanger system which is a component of the chamber. As a result, the gas gets a temperature that is practically equal to that of the chamber, and the cooling power can be expressed in terms of the heat which is extracted by the gas blown through the chamber within a time unit. Taking the Clapeyron-Mendeleev equation into account at  $T_c > T_0$ , where  $T_0$ is the initial gas temperature ( $T_0 = 4.2$  K for helium), we obtain

$$P_{fr} = \frac{C_{\rm He}\mu p}{R} \frac{dV}{dt},\tag{4}$$

where p,  $C_{\text{He}}$ , and  $\mu$  are, respectively, the pressure, heat capacity, and molar mass of the refrigerant;  $\frac{dV}{dt}$  is the rate of refrigerant vapor blowing through the cryostat chimney, and R is the gas constant.

To provide the relation  $P_{fr} \sim T_c^2$ , we must realize the dependence  $\frac{dV}{dt} \sim T_c^2$ . The quadratic dependence of the cryostat column-in blowing rate is ensured by the squarelaw dependence of the relative pulse duration for pulses, which are used to control the gas flow by means of an electrodynamic valve, on the voltage  $U_{fr}$  which is used to control a pulse-width modulator. If the maximal cooling voltage  $U_{fr \max} = \lambda U_{ref}$ , then the maximal cooling pulse ratio is determined from the expression

$$\frac{\tau}{\tau_0} = \frac{U_{fr}^2}{U_{\text{ref}}^2} = \lambda^2,\tag{5}$$

where  $\tau$  and  $\tau_0$  are the duration and the repetition period of cooling pulses, respectively. At the pulse ratio (5), the rate of column-in blowing with refrigerant vapors is

$$\frac{dV}{dt} = \frac{dV_0}{dt}\frac{\tau}{\tau_0} = \frac{dV_0}{dt}\lambda^2,\tag{6}$$

where  $\frac{dV_0}{dt}$  is the maximal rate of column-in blowing at a completely open valve.

## 4. Experimental Part

To implement the thermostatting by means of the method described above, we developed a device for the temperature control, the block diagram of which is shown in Fig. 2.

A required temperature  $T_{\rm set}$  can be set by means of either a control keyboard or an external computer. The microprocessor provides a control over the process of temperature setting in the chamber,  $T_c$ , at the level  $T_{\rm set}$ . The duration of every program cycle is  $\Delta t = 100$  ms, and it includes the following operations which are executed in sequence:

- an analog-to-digital converter is used to measure the voltage across the temperature sensor,  $U(T_{c_i})$  (the subscript *i* corresponds to the measurement time moment  $t_i$ );

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Fig. 3. Relative heating and cooling powers and their sum

– the measured voltage  $U(T_{c_i})$  is recalculated into the actual temperature code,  $T_{c_i}$ , in accordance with the data obtained at the temperature sensor calibration and stored in the nonvolatile memory of the processor;

- the difference  $\Delta T_i = T_{\text{set}} - T_{c_i}$  between the codes of the setted,  $T_{\text{set}}$ , and actual,  $T_c$ , temperatures is calculated; - the rate of actual temperature change is calculated,  $\frac{dT_i}{dt} \approx \frac{\Delta T_{i-1} - \Delta T_i}{\Delta t}$ , where the subscript i - 1 corresponds to the previous measurement;

- the values obtained for  $\Delta T_i$  and  $\frac{dT_i}{dt}$  are used to calculate the control code  $n_i$  which is used to form the  $U_h$ and  $U_{fr}$ -signals.

The control code is calculated by the expression

$$n_i = n_{i-1} + K_1 \Delta T_i + K_2 \frac{dT_i}{dt},\tag{7}$$

where  $K_1$  and  $K_2$  are the weight coefficients for the proportional and differential components, respectively; and  $n_{i-1}$  is the previous value of control code at the moment  $t_{i-1}$ .

To form the signal  $U_h$ , the code of  $\lambda n_i$  is written into the registers of a digital-to-analog converter (DAC). In this case, the heating voltage is

$$U_h = U_{\rm ref} \lambda \frac{n_i}{n_0},\tag{8}$$

where  $n_0$  is the maximal value of control code which is defined by the register capacity of DAC. Then, the

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Fig. 4. Time diagrams for the final stage of reaching the the preset value of temperature in the old (1) and new (2) cryostat systems. The initial temperature of the chamber is 300 K, the final one is 5 K

heating power is

$$P_h = \frac{\lambda^2 \left( U_{\text{ref}} \frac{n_i}{n_0} \right)^2}{R_h}.$$
(9)

To form  $U_{fr}$ , the code of  $\lambda (n_0 - n_i)$  is written into the DAC registers. Then, we have

$$U_{fr} = U_{\text{ref}} \lambda \frac{n_0 - n_i}{n_0}.$$
(10)

After the squaring by means of an amplifier-multiplier, the signal  $U_{fr}$  is applied to a pulse-width modulator, and the cooling power is

$$P_{fr} = \frac{C_{\rm He}\mu p}{R\Delta t} \lambda^2 \left( U_{\rm ref} \frac{n_0 - n_i}{n_0} \right)^2.$$
(11)

#### 5. Results and Their Discussion

The described approach to thermostatting was put into the basis of a new device developed by us for the temperature control in the range from 4.2 to 350 K. In Fig. 3, the dependences of the relative heating and cooling powers, as well as their sum, on the relative value of control

353



Fig. 5. Temperature dependences of the overcontrol for the old (1) and new (2) cryostat systems



Fig. 6. Temperature dependences of the stability for the old (1) and new (2) cryostat systems

code are depicted. One can see that, at the quadratic character of control influences, the sum of the relative heating and cooling powers is linear in the relative value of control code.

In Fig. 4, the typical experimental plots showing how the temperature approaches a given value are shown: when a temperature control device is used, but the thermal properties of a substance the chamber was fabricated of are not taken into consideration, and when the new approach was applied. It is evident that the new control device allows the temperature to reach a preset value



Fig. 7. Temperature dependences of the refrigerant consumption for the old (1) and new (2) cryostat systems

without overcontrol and faster in comparison with the old method.

For the experimental testing of the efficiency of the proposed improvements for cryostat systems, we compared the metrological (the overcontrol level and the stability) and economic (the refrigerant consumption) parameters of the new and old cryostat systems. In so doing, the overcontrol level was defined as the maximal deviation of the temperature  $T_c$  from  $T_{\text{set}}$  after the time moment  $t_{\text{eq}}$ , when they became equal. The stability was defined as the maximal deviation of  $T_c$  from  $T_{\text{set}}$  within 30 min after the time  $t_{\text{eq}} + 2$  min. The temperature dependences of the overcontrol, stability of the established temperature, and refrigerant consumption are shown in Figs. 5 to 7, in that order.

It follows from Fig. 5 that the restrictions of the maximal heater power and the cooling level leads to a substantial reduction of the overcontrol in the whole temperature range under consideration. The minimal level of the overcontrol (of 0.02 K) is observed in the range 20–200 K which is characterized by a fast change of the temperature dependence of the copper heat capacity (see Fig. 1).

From Fig. 6, it follows that the consideration of the heat capacity of a thermostatting chamber material allowed us to make the stability of established temperatures as much as 1.5 to 2 times higher. In the examined temperature interval, the largest instability was observed at a temperature of 10 K and did not exceed 0.04 K.

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Figure 7 demonstrates that the refrigerant consumption in the new cryosystem decreased by 15% on the average in comparison with the old one.

## 6. Conclusion

Hence, taking the thermal properties of the substance of a work chamber of cryostatting systems into account has allowed us to substantially increase their precision characteristics, avoid the overcontrol, and reduce the refrigerant consumption, which opens new opportunities for carrying out a precision physical experiment. As a result of the practical use of the control device developed by us, the cryosystem tuning became simpler, and its reliability is enhanced.

- A.I. Belyaeva, V.I. Silaev, and Yu.E. Stetsenko, *Flow*ing Cryostats for Laboratory Researches (Kyiv, Naukova Dumka, 1987) (in Russian).
- C.A. Swenson and R.H. Staht, Rev. Sci. Instrum. 25, 608 (1954).
- W.P. Knox and J.D. Cathbert, Rev. Sci. Instrum. 39, 1181 (1968).
- A.I. Belyaeva, V.I. Silaev, Yu.N. Stelmakhov, and Yu.E. Stetsenko, Cryogenics 23, 303 (1983).
- V.S. Medvedev, V.M. Ermakov, P.V. Vodolazskii *et al.*, Authors' Certificate USSR N 436334, MKI G05d 23/30, G05d 16/06; Byull. Izobret. SSSR N 26, 126 (July 15, 1974) (in Russian).
- I.P. Zharkov, V.M. Ermakov, V.V. Safronov, and A.G. Tchmul, Visn. Khark. Derzh. Univ., Ser. Biofiz. N 3, 125 (1999).

- I.P. Zharkov, V.M. Ermakov, V.V. Safronov, and A.G. Tchmul, Proceedings of the 1-st Ukrainian Scientific Conference on Semiconductor Physics UNKN-1 (Asrtoprint, Odesa, 2002), Vol. 2, p. 279 (in Russian).
- I.P. Zharkov, V.V. Safronov, and A.G. Tchmul, Ukrainian Patent N 18778, MPK (2006) G05D23/30 (in Russian); Byul. N 11, 1 (November 15, 2006).
- 9. H. Jones, Proc. Roy. Soc. A 240, 321 (1957).
- 10. D.L. Martin, Can. J. Phys. 38, 17 (1960).
- 11. K.K. Kelley, J. Chem. Phys. 11, 16 (1943).
- W.S. Corak, M.P. Garfunkel, C.B. Satterthwaite, and A. Wexler, Phys. Rev. 98, 1699 (1955).

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### ОПТИМІЗАЦІЯ РЕГУЛЮВАННЯ ТЕМПЕРАТУРИ У РІДИННО-ПРОТОЧНИХ КРІОСТАТАХ

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Резюме

З метою оптимізації регулювання температури у кріосистемах на основі рідинно-проточних кріостатів враховано теплофізичні властивості матеріалу, із якого виготовлено елементи конструкції робочої камери кріостата. На прикладі результатів експериментального використання розробленого пристрою для управління кріостатом в температурному діапазоні 4,2–350 К показано, що застосування цього методу підвищує точність та економічність кріосистем.