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Transient measurement apparatus for material thermal conductivity at ultra-low temperature

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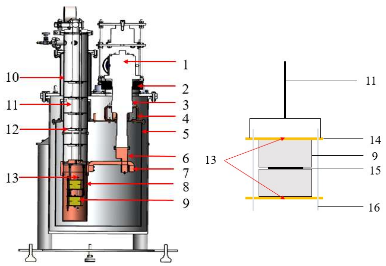
Abstract

This study was the first attempt to apply the transient plane heat source method to the thermal conductivity measurement at low temperature. First of all, a detachable cryogenic thermostat was designed, which used a GM cryocooler to obtain the cooling capacity. The chamber was filled with cryogenic helium gas, where the sample was mounted. The precooling process, heat leakage and temperature field division were simulated by finite volume method, and the results verified the rationality of the structural design. Secondly, the measurement probe of the transient plane heat source method was recalibrated at low temperature. Finally, the thermal conductivities of 304 stainless steel and polyurethane at low temperature were measured, and the values were compared with those in the literature, so as to verify the reliability of the measuring device.

Cryogenic thermostat structure design

- The cryostat is cooled by a GM chiller model **35W@50K and 1.5W@4.2K**.
- The cryostat is vacuumed to below **10Pa** at room temperature.
- **Cold shield** is set up to minimize radiant heat leakage.
- The cold quantity is transferred between the secondary cold head and the copper test chamber through **a copper cold chain**.
- **Cryogenic helium gas** fills the chamber to cool the sample placed inside.
- A number of **baffles** are distributed on the test rod to **prevent the flow of helium** in the test chamber.
- The entire test rod can be **easily disassembled** for convenient sample replacement.
- The **probe** is tightened by two samples, which are clamped by two splints. The splints are fastened with **springs and nuts**.
- The purpose of using springs is to **prevent poor contact** between the probe and the specimen caused by **cold shrinkage** at the low temperature.



Section view of cryogenic thermostat apparatus and sample support diagram. 1- GM cryocooler, 2- Buffer threaded pipe, 3- 1st stage of GM cryocooler, 4- 1st stage copper cold chain, 5-Cold shield, 6-2nd stage of GM cryocooler, 7-2nd stage copper cold chain, 8-Copper test chamber, 9- Test sample, 10-Stainless steel test chamber, 11-Test rod, 12-Separation plate, 13-DT670 temperature sensor, 14- splint, 15-measuring probe, 16-Fixed spring/nut.

Numerical simulation of cryogenic thermostat

Physical models and mathematical models

Geometric model:

- Delete: bolts and nuts, seals
- Simplify: holes, chamfers, etc

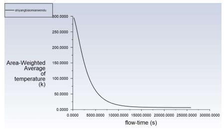
Mathematic model:

- Heat conduction model
- Thermal radiation DO model

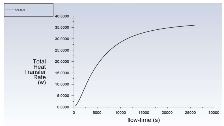
Boundary conditions and physical parameters

Materials	Thermostat chamber: 304 stainless steel; Cold shield: Copper, emissivity 0.04 Sample: Rigid polyurethane foam, emissivity 0.9
Boundary conditions	Outer wall surface: temperature300K, convective heat transfer coefficient 10W/(m²·K), emissivity 0.8 Inner wall face: emissivity 0.04(Vacuum multilayer insulation material wrapped) 1st stage cold head: temperature 50K 2nd stage cold head: temperature 4.2K

Transient simulation results

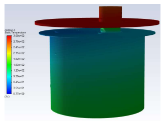


Variation of sample mean temperature with time

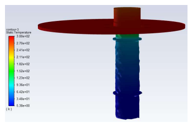


The heat flow variation on the outside surface of thermostat with time

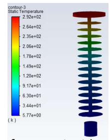
Temperature field distribution at the end of the transient simulation



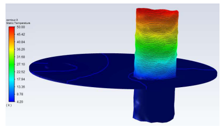
cold shield



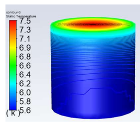
Test chamber



reference test bar



Test chamber surface



test sample

Principle of transient plane heat source method and probe calibration

Principle of transient method

- The partial differential equation of heat conduction problem:

$$\kappa \nabla^2 T + \frac{Q}{\rho c} = \frac{\partial T}{\partial t}$$

- The temperature rise model obtained by Green's function method:

$$\Delta T(\tau) = A + \frac{P_0}{\pi^{\frac{3}{2}} a \kappa} D(\tau)$$

- where D (τ) is the dimensionless time function:

$$D(\tau) = \frac{1}{m^2(m+1)^2} \int_0^{\tau} \frac{d\sigma}{\sigma^2} \sum_{k=1}^{m} k \sum_{l=1}^{m} l \exp\left(-\frac{k^2+l^2}{4\sigma^2 m^2}\right) I_0\left(\frac{kl}{2m^2\sigma^2}\right)$$

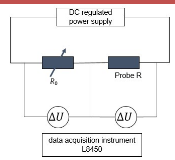
- The temperature rise of the probe is calculated by the following formula:

$$R(t) = R_0\left[1 + \alpha \cdot \Delta T(t)\right]$$

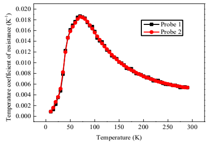
Double spiral heating zone

$$x = \left(\frac{s}{c_1 \pi}\right) \sin(s) \text{ and } y = \left(\frac{s}{c_2 \pi}\right) \cos(s)$$

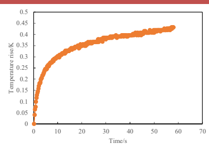
Electrical measurement scheme



Calibration of nickel probes at low temperature



Probe temperature rise curve for stainless steel at 70K

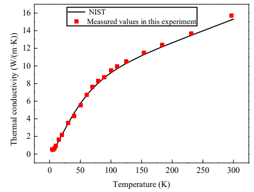


Temperature control effect test of thermostat

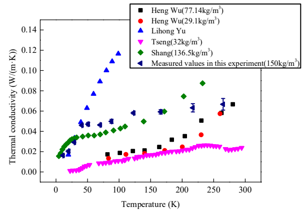
Temperature/K	Upper and lower surface temperature difference	temperature stability
4K	46mK	2.5mK
10K	37mK	5mK
20K	70mK	10mK
50K	302mK	6mK
70K	550mK	25mK

- At temperatures below 70K, the temperature difference between the upper and lower surfaces of the sample is less than 0.55K.
- At temperatures below 70K, the temperature stability of the test chamber is less than 0.025K.

Material thermal conductivity test



Thermal conductivity of 304 stainless



Thermal conductivity of polyurethane rigid foam

- The 304 stainless thermal conductivity deviation between the measured values and the Nist values above 20K was less than 8%.
- The measured value of thermal conductivity of polyurethane rigid foam is closest to that in Shang with a density of 136.5kg/m³.

Conclusion

- A cryogenic thermostat suitable for the transient plane heat source method was built;
- The temperature distribution of cryogenic thermostat was simulated and verified by experiments;
- The temperature coefficient of resistance at low temperature was recalibrated for the nickel probe.
- The thermal conductivities of 304 stainless steel and rigid polyurethane foam at low temperature were measured, and the results were compared with those in the literature, which confirmed the reliability of the experimental device.