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Cryogen-free apparatus for rapid thermal emissivity measurements at low temperature

FZ. Shen[1,2], D. Xu[1,2], X. Li[1, 2], HM. Liu[1], and LF. Li[1, 2]

[1]Key Laboratory of Cryogenics, Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, P. R. China.
[2]University of Chinese Academy of Sciences, Beijing 100049, P. R. China

E-mail: xudong@mail.ipc.ac.cn

Abstract: Thermal radiation is a significant way of heat transfer among insulation parts. For minimization of heat leakage, the radiative properties of materials are essential for the design of cryogenic devices. Emissivity is a key element of radiation properties of materials. However, such experimental data are always not sufficient, especially at cryogenic temperature. In order to measure the radiative properties of material surfaces, a measurement device was presented. The device is based on a two-stage Gifford-McMahon refrigerator and the dismountable sample test rod has independent vacuum environment, which allows the sample to be replaced rapidly. In emissivity measurement, the temperature of examined samples can be controlled precisely between 15.4 K to room temperature.

1. Introduction

Thermal radiation is an important mode of energy transfer, especially in the high vacuum insulation environment where radiative heat transfer dominates the requirement of thermal insulation. [1] Material data on radiation properties are required for the design of cryogenic applications to minimize parasitic heat. [2] However, reliable thermal radiative properties data of cryogenic materials are quite insufficient in published literatures.

Most apparatus for cryogenic emissivity measurement rely on liquid cryogens, which is very scarce and expensive. Therefore, experiments are limited by the supply of cryogen. In addition, for reducing consumption of cryogen, the cryogen recovery unit, which has complex contraption and huge dimension, is always needed in traditional cryogenic apparatus. Besides, the apparatus costs comparatively long time, at least ten hours, to replace the sample and reset the experimental environment. [3]

A cryogen-free apparatus for rapid thermal emissivity measurements based on a two-stage Gifford-McMahon cryocooler was presented in this paper. The experiment system consists of a helium

circulation loop, a transition vacuum chamber and a dismountable sample test rod. Samples can be cooled to 15.4 K without consumption of cryogen by the cold helium gas circulated in the cooling loop. With the introduction of the dismountable sample test rod, the replacement of samples can be easily operated and is time-saving without destruction of whole vacuum environment in the described apparatus.

2.   Measurement principle

There are two main categories of emissivity determination: the radiometric method and calorimetric method. The radiometric method detects the electromagnetic heat by a sensitive bolometer. The calorimetric method can be divided into two basic groups: the dynamic method which measure the total emissivity or absorption by throwing the sample out of thermal equilibrium. On the other side, the key point of the steady method is substituting the calibration heat for the radiation heat. [4]- [5]

   In this paper, steady calorimetric method is adapted to measure the material emissivity. The steady-state method determines thermal emissivity by measuring radiative heat flow between radiator and absorber, which are two parallel circular surfaces and equal on radiative surface area, radiator temperature and absorber temperature. With these parameters, material emissivity can be calculated by equation 1.

$$Q_R = A\sigma(\varepsilon_R T_R^4 - \varepsilon_A T_A^4) \tag{1}$$

   where σ stays Stefan-Boltzmann constant, ε_R and ε_A are respectively emissivity of radiator and absorber, T_R and T_A are respectively temperature of radiator and absorber, Q_R is radiative heat flow between radiator and absorber, and A is radiative area of the radiator and the absorber.

3.   Description of the cryogen-free system

The thermal emissivity measurements mentioned in this paper can be divided into three parts: helium circulation loop for heat transfer, dismountable sample test bar for sample testing and transition vacuum room for sample rapid replacement. The configuration of cryogen-free system is illustrated in figure 1.
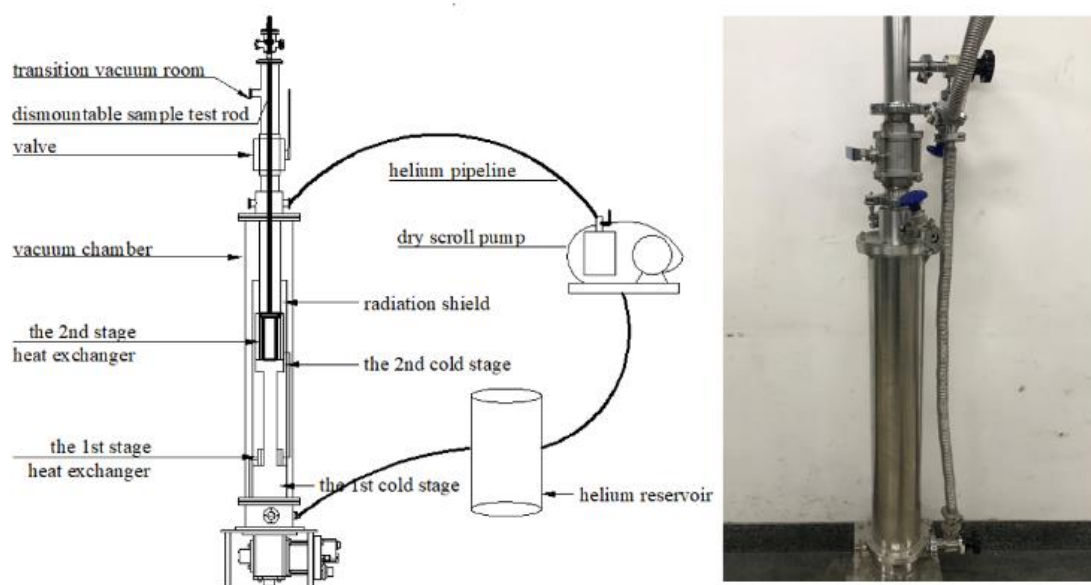

Figure 1.   The schematic of cryogen-free system

3.1 Helium refrigeration circulation

Different from the conventional cryogenic testing equipment, helium gas is used as cold medium. Thus there is no direct thermal contact between sample testing room and crycooler. Helium circulation loop includes helium reservoir, helium pipeline, heat exchanger, dry scroll pump, and helium gas circled in the loop and a G-M crycooler. The cooling power of the described crycooler is 40W at 43K in the first stage cold head and 1W at 4.2K in the second stage cold head. [6] The helium gas outflows from the reservoir, then be cooled in the first stage heater exchanger. The cold helium gas will be cooled and condensed again in the second stage heater exchanger before exchanging heat with the thermal couple ring. Vaporized helium gas will reflow to the reservoir after heat transfer process.

3.2 Independent vacuum parts

The vacuum environment of dismountable sample test rod is independent with the cryogenic unit. To guarantee the rapid replacement of sample, a transition vacuum chamber, which consists of sample change chamber and valve, is set between the two parts. With the introduction of transition vacuum chamber and dismountable sample test bar, the replacement of sample can be operated without the destruction of the vacuum environment of whole system.
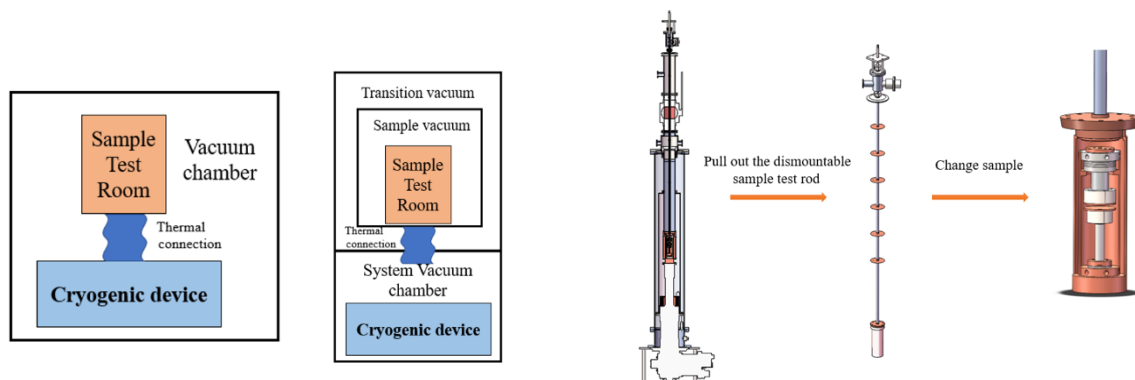

Figure 2. The schematic of transition vacuum (left) and flow chart of sample replacement (right)

3.3 Dismountable sample test rod

Dismountable sample test rod is inserted into the test tube and mounted in the second stage cold head. The schematic is shown in figure 3.

Vacuum chamber and testing chamber holder are fixed in the thermal copper ring with bolts. The bottom bulk of sample testing chamber thermally joins the testing chamber holder with thermal contact and so with the copper radiation shield. The absorber holder is linked to the bottom bulk of sample testing chamber by the thermal resistor, with two layers (a cooper layer to install the thermal resistor and an epoxy layer to insulate the heat diffusion) inserted. The top bulk of sample testing chamber is attached to the copper radiation shield with bolts. The radiator holder is linked to the top bulk of sample testing chamber by the thermal insulation tube. There are five stainless steel pieces between locating block in the radiation shield and the top bulk. The distance between absorber and radiator can be adjusted through changing the quantity of the pieces. To diminish the radiation heat transfer, the aluminium shield is installed in the backside of both radiator and absorber. The mechanical thermal switch is inserted into the central vacuum tube and actuator of the heat switch is set in the top plate of the dismountable sample

test rod. Five radiation shields are fixed in the central vacuum tube to supress the radiation heat transfer between the testing chamber and the surrounding environment.

To measure the temperatures in the testing room, three calibrated thermometers (Lake Shore Cernox^{TM} 1010) were used as temperature detector. The testing range of sensors is 1.4K to 325K and sensitivity is 4mk at 1.4K. The radiator is heated by a resistance wire heater. To obtain a microwatt heat power, Lake shore DT-670 is chosen as the heater of the absorber. Lake Shore 320 is used as temperature and heat power controller.
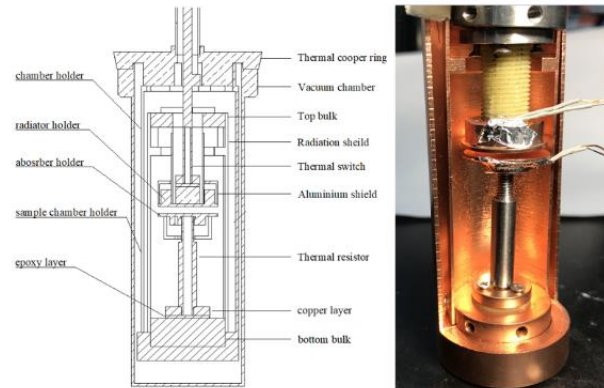

Figure 3. The schematic of dismountable sample test bar

4. Experimental Procedure and results

4.1 Experimental Procedure

To measure the emissivity by the steady calorimetric method described in this paper, the thermal resistor should be calibrated first. To obtain the calibration curve, the radiator is maintained at the temperature of the test chamber room. The temperature of absorber and stabilization can be changed with the adjusting of absorber heater power. The relationship of the temperature gradient of T_A-T_S and calibration heat power Q_C is dependent on the parameters of the thermal resistor. Different types of thermal resistor were chosen to meet the different sample measure requirements.

After the purification of cryogen-free system, emissivity measurement experiment can be operated. The temperature of radiator can be adjusted by the resistance wire heater controlled by Lake Shore 320. After sample reached the testing temperature, the temperature of radiator T_R, absorber T_A and stabilization T_S will be record. The corresponding radiation transfer power can be found in the calibration curve. With these parameters, emissivity of sample can be calculated from the equation 1.

4.2 Cooling simulation and experimental result

Before the construction of the cryogen-free system, numerical simulation of sample text room cooling was carried out. Figure 4 shows the contour plots of the temperature distribution of the sample testing room when the testing chamber reaches thermal equilibrium between GM crycooler and system heat leakage. From this figure, it can be seen that the temperature of radiator is 11.1K and the temperature of absorber and stabilization are 9K and 8.7K when the thermal cooper ring been cool down to 4.2K.

Cooling down experiment was carried out in the cryogen-free system with the construction of cryogen-free apparatus finished. Typical cooling curve is shown in Figure 5. It costs 160 minutes to obtain thermal equilibrium when radiator, absorber and stabilization reach 15.4K, 8.1K and 7.9K from

the room temperature. The results of numerical simulation are in good agreement with the experiment measurements. It indicates that contribution of elements except thermal conductivity and thermal radiation which simulation model contained is negligible to the temperature distribution of sample test room.
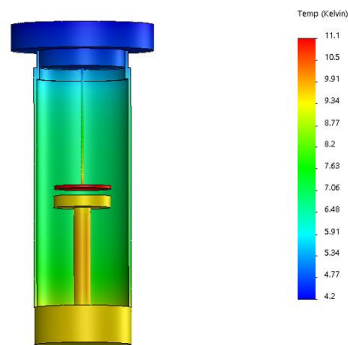

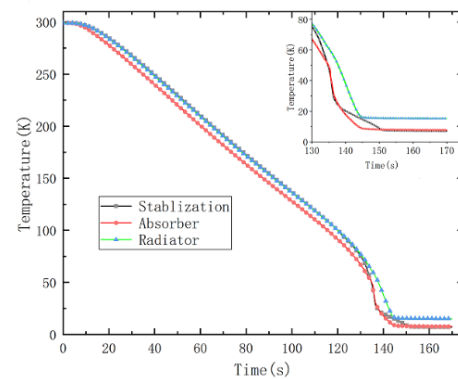**Figure 4.** Numerical simulation of temperature distribution of sample test room

Figure 5. Cooling down curve of sample test room

5. Conclusion

A cryogen-free system for rapid emissivity measurement was presented in this paper. With the introduction of dismountable sample test rod, sample can be changed conveniently without destruction of the vacuum environment of whole system. The cooling process has been simulated and in general the results of experiment and simulation are in good agreement. Sample can be cooled down to 15.4K in 160 minutes based on a two-stage Gifford-McMahon cryocooler. Experimental data of emissivity of different materials will be published in the future.

6. References

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