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A calorimeter for measurements of multilayer insulation at variable cold temperature

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Abstract

An improved calorimeter cryostat for MLI thermal performance measurements has been designed and put into operation at the TU Dresden. Based on a liquid helium cooled flow cryostat, it allows the setting of any cold level temperature between approx. 30 K and ambient temperature. Thermal shields and all-embracing radiation guards at both ends can be kept at nearly identical temperature. This is done by means of two separate cooling circuits. Both the actual cold test surface temperature and the cooling of the mechanical support and radiation shields can be independently controlled.

Insulation specimens are wrapped around a test cylinder with a surface of 0.9 m². The heat transfer through the MLI is measured by recording the mass flow and the inlet and outlet temperature of the cooling fluid. Measurements both in horizontal and vertical orientation can be performed or compared, respectively. Moreover the effect of an additional vacuum degradation as it might occur by decreasing getter material performance in real systems at elevated temperatures can be studied by controlled inlet of an elective gas. It is reported about the design and the equipment of this cryostat and measurements of a 10 layer MLI specimen.

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1. Introduction

Cryogenic devices use necessarily very sophisticated thermal insulation. Various approaches for many applications have been investigated. Since many years multilayer insulation now is established as state of the art for cryogenic devices at liquid helium or liquid hydrogen temperature (Scurlock et al. (1976)). Typical applications are cryostats for superconducting magnets or cryogenic storage vessels. Characteristic of these applications is a fixed cold temperature, defined by the boiling temperature of the respective cryogenic liquid inside. Consequently existing calorimeters for MLI performance measurements are commonly based on LN₂ or LHe bath cryostats. Extensive investigation results have been already published regarding e.g. an optimized number of layers, optimum MLI density, different seam methods or the effect of a residual gas pressure. Degrading effects like geometrical discontinuities in real apparatuses (edges and deflections or breaks in the isothermal layers) have been investigated as well.

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On the other hand, the operational temperature e.g. in cryo-compressed storage vessels using MLI in the vacuum insulation stretches out from 30 K over 300 K. Those systems are currently investigated for automotive hydrogen storage solutions (Acheves (2010)). MLI performance data for such a wide temperature span are not available to date.

Also superconducting systems can contain MLI in different temperature ranges, i.e. utilizing a cryocooler. The performance of MLI at intermediate temperatures between temperatures other than saturation of cryogens is of interest for these kind of applications (Celik (2013)), (Funke (2012)).

The calorimeter cryostat presented in this paper allows MLI performance measurements at variable cold temperature. In the following it is reported on its design and the experimental setup as well as on experimental results.

Nomenclature

\dot{Q}	heat flux on the test cylinder in [W]
T_1	inlet temperature of the cooling fluid
T_2	outlet temperature of the cooling fluid
\dot{m}	mass flow of the cooling fluid

2. Conceptual Design

The calorimeter cryostat was designed to measure the heat flux through applied MLI packages at variable cold temperatures. Therefore a flow cryostat was chosen as working principle. The MLI is mounted on a test cylinder. The effective area is confined by the cylindrical part of this test area. The cylinder is held at the operating temperature by means of a cooling fluid. Preferably liquid helium (LHe) respectively helium cold gas is used. An external heater can warm up the fluid in order to achieve the desired temperature for the test cylinder. In Fig. 1 the flow scheme of the cryostat is shown, including temperature sensors.

The integral value of the heat load on this test cylinder is measured. It is predominated by the heat flux through the MLI, plus some minor, inevitable parasitic heat fluxes. The chosen geometries and dimensions allow measurements for various MLI configurations, mounting techniques and test conditions.

Before entering the actual test cryostat, the inlet flow is heated up to a temperature T_1 by means of a controlled heater. The isothermal cylinder is kept at a slightly higher preselected operating temperature T_2 by this cryogenic fluid flow during the whole measurement.

Inside the cryostat the preconditioned mass flow is divided in two cooling paths (\dot{m}_c , \dot{m}_s). One for the test cylinder and the other for the thermal shields. The escaping mass flow is passively warmed up. A mass flow controller for the test cylinder circuit and a fine control valve plus a mass flow meter for the shield cooling circuit complete the external accessories.

The heat load onto the test cylinder then can be easily and quite precisely derived from the cooling fluid mass flow, its heat capacity and the temperatures T_1 and T_2 , using equation (1) for the total heat load and equation (2) for the heat flux related to the cold surface area:

$$\dot{Q} = \dot{m} \cdot \int_{T_1}^{T_2} c_p dT \quad (1)$$

$$\dot{Q} = \frac{\dot{m}}{A} \cdot \int_{T_1}^{T_2} c_p dT \quad (2)$$

Some examples for typical parameters at two different operating points are presented using the fluid database REFPROP (2013). In these calculations two mass flows of 0.05 g/s and 0.03 g/s with helium as cooling fluid are considered. The heat flux through the MLI is assumed to account for $\dot{q} = 1 \text{ W/m}^2$ according to Lehmann (2000).

The cryostat is primarily designed for helium as cooling fluid. Alternatively, i.e. for higher surface temperatures, nitrogen can be used. The measurements are carried out under high vacuum conditions or at defined degraded vacuum conditions. Therefore a needle valve is installed to vent the vacuum space with small amounts of a preselected gas.

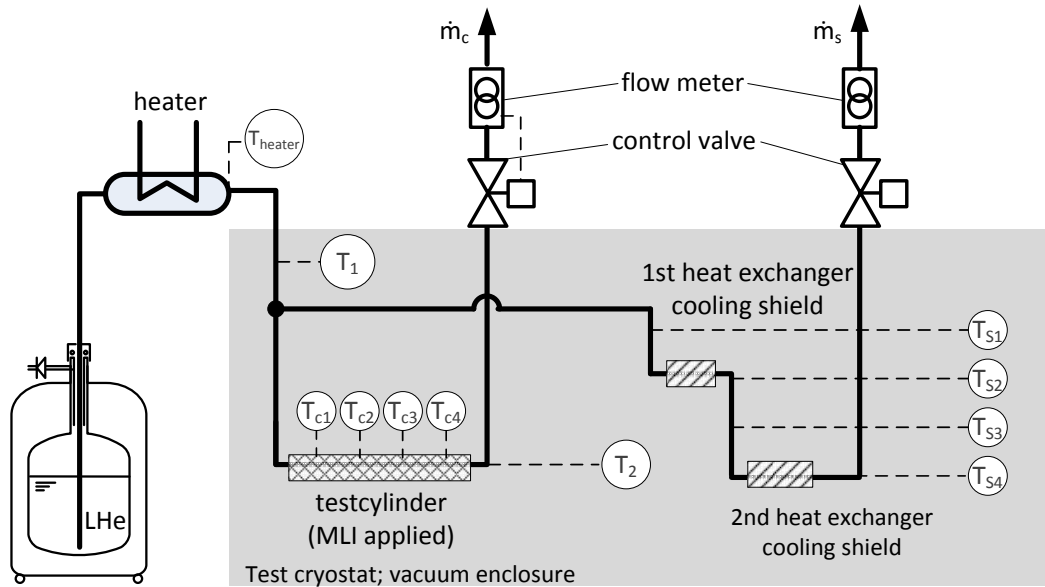


Fig. 1. Schematic design of the flow cryostat with LHe supply, external heater and the cryostat itself

Besides the variable cold temperature gravimetric effects on the thermal performance of MLI shall be investigated. Therefore the cryostat insert is designed for operation in tilted orientation as well. All ducts and connections are mounted in a way to allow an inclination up to 90° .

Table 1. Calculated operating parameters, cooling fluid Helium \dot{m}_c @ 1.5 bar absolute pressure

mass flow	0.05 g/s	0.03 g/s
Desired cold temperature	80 K	80 K
Temperature test cylinder inlet T_1	76.6	74.3 K

3. Calorimeter description and Instrumentation

The calorimeter is of concentric cylinder type and is tiltable from vertical to horizontal orientation. All inner parts are mounted at the top flange of the cylindrical vacuum vessel (Fig. 2). The cold cylinder is suspended on top and bottom via GFRP rods. The vacuum jacket represents the warm boundary.

The major components and their dimensions are as follows: The cold cylinder is made of 219 mm outer diameter, 1300 mm long and 3 mm thick seamless copper pipe. Which yields to a outer surface of 0.89 m^2 . The whole surface area is nickel plated and high gloss polished to reduce the emissivity on the outer and inner surface. On the upper and lower ends are thermal shields to minimize undesired heat loads. Each shield is actively cooled via the preconditioned cooling fluid. The first heat exchanger is mounted on the top shield the second on the lower end. They are connected in series.

Two Si-Diodes with a accuracy of 250 mK up to 100 K are installed. These two sensors are CU package and in four wire configuration. A total of 10 standard Pt100 temperature sensors are installed for monitoring purpose as well. Sensor number $T_{c1} \dots T_{c4}$ are mounted on the inside of the test cylinder to check for isothermal conditions. They are mounted according to the manufacturers standards in a copper bobbin. All wires are thermally anchored at the upper radiation shield.

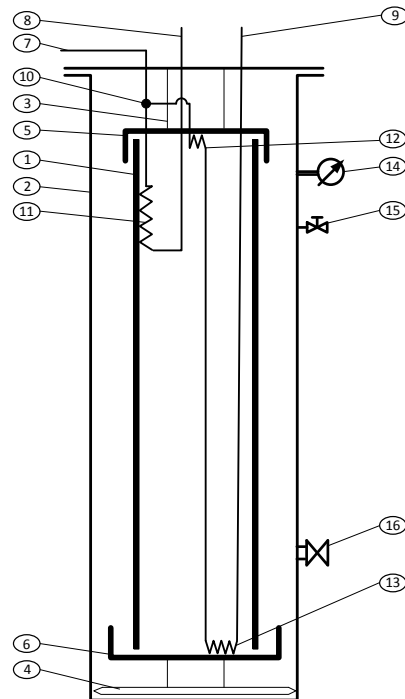


Fig. 2. Schematic of the calorimeter: 1 - cold cylinder, 2 - warm cylinder, 3 - upper support rod, 4 - lower support rod, 5 - upper radiation shield, 6 - lower radiation shield, 7 - cooling fluid inlet, 8 - cooling fluid outlet test cylinder circuit, 9 - cooling fluid outlet shield circuit, 10 - T-section of inlet line, 11 - heat exchanger test cylinder, 12 - 1st heat exchanger shield circuit, 13 - 2nd heat exchanger shield circuit, 14 - vacuum gauge, 15 - needle valve, 16 - vacuum port

There are two heat exchanger to cool the shield and support structure. Their inlet and outlet temperature is measured and represented by $T_{S1} \dots T_{S4}$. The external heater uses another two sensors, where T_{heater} is the set temperature.

The Helium dewar is externally pressurized at a constant level of 1.5 bar absolute pressure. The mass flow through the test cylinder is controlled by a Bronkhorst F-112AC flow meter and a control valve type F-004AC-LUU-49-V from the same manufacturer. The mass flow of the shield circuit is adjusted manually and a thermal mass flow meter type Sierra ML100 is used to measure the flow rate. Both flow meters are placed at ambient temperature of about 21 °C.

The data acquisition system consists of a LakeShore 224 temperature monitor and a thyristor controller built by CryoVac (Germany) and using an Eurotherm controller unit for the electrical heater. A turbomolecular pump is used to maintain a proper vacuum inside the cryostat of about 10^{-5} mbar, unless intentional gas is injected to degrade the vacuum to a certain level. A wide range vacuum gauge type Pfeiffer PKR 251 is mounted on the upper part of the cryostat.

4. Experimental details

The MLI samples to be tested are first wrapped around the cylinder. The calorimeter is then placed in the cryostat vessel and the insulation space is evacuated utilizing a turbomolecular pump. After 12-24 h a pressure level of about 10^{-5} mbar is achieved. Meanwhile the 100 l LHe dewar is connected via vacuum insulated transfer line to the electrical heater, which is placed and also vacuum insulated connected to the cryostat inlet.

For a fast cooldown high mass low rates up to 0.2 g/s are set. The temperature controller to heat up the inlet stream is brought in line when the desired cold temperature of the test cylinder is achieved. Using an estimated value for the

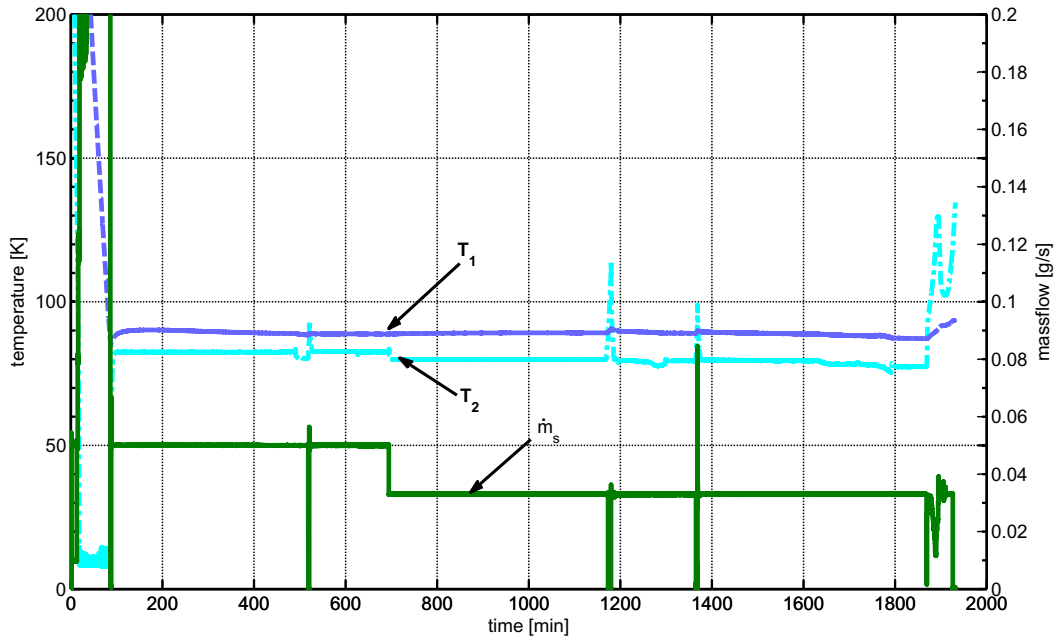


Fig. 3. Temperatures T_1 , T_2 and the mass flow through the test cylinder over time

total heat load, the inlet temperature T_1 is precalculated and set by the external heater. The cylinder temperature T_2 adapts very slowly. Hence the inlet temperature is adjusted according to the temperature slope of the test cylinder.

5. Results

The investigated specimen is a commercial 10 layer double side aluminized polyester film interleaved with polyester spacer material. It is butt joined and the seam is covered by a low emissivity tape. The sheet is handcut. Due to the relatively small cylinder diameter, the length between the innermost and outermost layer are cut on site.

In Fig. 3 the temperature T_1 and T_2 and the mass flow through the test cylinder are shown over time. The measurement ran over 33 h, including the cool down phase from room temperature of 20 °C.

Fig. 4 a) and b) show detail views of the measurement. Steady state conditions are widely achieved. And within relatively short time different sets of data at varied mass flow rates are acquired.

Two flow rates of 0.05 g/s and 0.03 g/s were chosen. The actual results are summarized in table 2. The calculated heat loads onto the test cylinder are within 1.5 % deviation with respect to the measurement of 1.6 W.

Table 2. Results with varied mass flow and adapted inlet temperature T_1

time	480 min	680 min	1200 min
mass flow	0.05 g/s	0.05 g/s	0.033 g/s
Temperature test cylinder inlet T_1	82.7 K	82.6 K	79.9 K
Temperature test cylinder outlet T_2	88.8 K	88.7 K	89.2 K
pressure level	1.39 bar	1.42 bar	1.40 bar
calculated total heat load	1.584 W	1.61 W	1.60 W

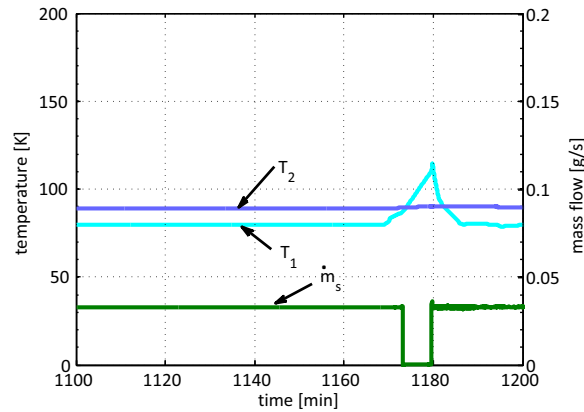


Fig. 4. Detailed view of the measurement (a)

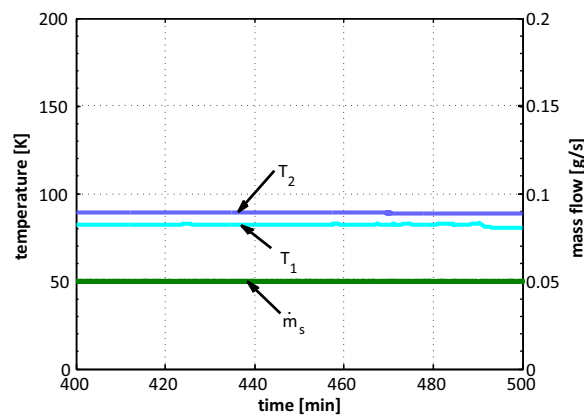


Fig. 5. Detailed view of the measurement (b)

References

- Scurlock, R. G., Saull, B., 1976. Development of multilayer insulation with thermal conductivities below $0.1 \text{ mW cm}^{-1} \text{ K}^{-1}$, *Cryogenics* 16, 303 - 311.
- REFPROP, B., Grove, A., 1965. General Relationship for the Thermal Oxidation of Silicon. *Journal of Applied Physics* 36, 37–70.
- Lehmann, W., 2000. Superisolation (SI) deren Qualitaet und Degradierung bei Anwendungen in der Kryotechnik, DKV-Tagung, Bremen.
- Salvador M. Aceves, Tobias C. Brunner, et al., High-density automotive hydrogen storage with cryogenic capable pressure vessels, *International Journal of Hydrogen Energy*, Volume 35, Issue 3, February 2010, pages 1219-1226.
- Celik D., Hurd J., Klimas R., Van Sciver S.W., 2013. A calorimeter multilayer insulation (MLI) performance measurements at variable temperature, *Cryogenics* 54-55, 73-78.
- Funke Th., Haberstroh Ch., 2012. Performance measurements of multilayer insulation at variable cold temperature. *Advances in Cryogenic Engineering*, vol. 57B. New York: American Institute of Physics, pages 12791284.