Qualification of a vertical cryostat for MLI performances tests between 20 K and 60 K to 4.2 K

V Venturi 1,2, T Koettig 2, M Chorowski 1, J Polinski 1, V Parma 2, A Kopecky 1
1 WUST, Wroclaw University of Science and Technology, Wroclaw, Poland
2 CERN, Organisation Europeen pour la Recherche Nucleaire, Geneva, Switzerland
valentina.venturi@cern.ch

Abstract. Boil-off calorimetric measurements of Multilayer Insulation (MLI) performance from 20 K-60 K to 4.2 K are being performed at CERN, extending the range of measurements of MLI to cover cryostat applications where thermal shields are operated at lower temperatures than that of LN2. Possible applications include future large accelerators like the Future Circular Collider (FCC). The tests are carried out in an existing vertical test cryostat shielded with a liquid nitrogen cooled vessel and with an inner cylindrical liquid helium tank carrying the wrapped MLI sample. The cryostat has been modified to insert an additional intermediate thermal shield maintained at controlled temperatures in the 20 K-60 K range, by means of regulated power input from an electrical heater. This paper describes the test cryostat and the MLI performance measurement methodology as well as experimental results of the commissioning and calibration of the instrument.

1. Introduction
The technology which has been used since decades for thermal insulation of cryogenic devices is Multilayer Insulation (MLI), also referred to as superinsulation. MLI consists of a set of thin reflective layers, typically aluminized polyamide (Mylar® or Kapton®) interleaved with thin insulating layers, typically polyester nets. MLI operates in insulation vacuum, wrapped around the cold surfaces, protecting them from thermal radiation heat loads from higher temperatures. The performance of MLI systems has been under investigation since the 1970s and a rich literature of thermal performance can be found for applications between room temperature and 77K and between 77 K and 4.2 K [1], [2]. It is however rare to find experimental data between temperatures lower than 77 K and 4.2 K.
An investigation campaign is ongoing at CERN to explore thermal performance of MLI at 4.2 K with radiation from temperatures of 60 K and down to 20 K, especially in view of the possible exploitation of these temperature ranges for thermal shielding in large accelerators like the Future Circular Collider (FCC), for which heat loads to 4.2 K need to be kept to a minimum.

2. Cryostat description
The experimental setup is based on a test cryostat, see Figure 1, first developed by to Wroclaw University of Science and Technology, previously used to measure performances of MLI between 293
K and 77 K and between 77 K and 4.2 K [3], and subsequently modified for the purpose of these new tests. The cryostat is composed of an inner test vessel around which the MLI sample is wrapped. A double LH<sub>e</sub> guard vessel ensures insulation from other heat sources and an external LN<sub>2</sub> tank provides shielding from room temperature thermal radiation. The pipes of the guard and test vessel are double walled to aid in insulation. The boil-off gas in the test vessel is measured by a mass flow meter with an outlet to a helium recovery balloon with a pressure monitoring to measure and correct boil-off effects due to atmospheric pressure changes.

For the purpose of this study a modification to the test cryostat was made at CERN in order to allow intermediate temperature levels for the warm boundary to be fixed between 20 K and 60 K. A 1 mm thick aluminium thermal screen (ATS) has been designed and placed on a copper support in weak contact with the guard vessel. The aim is to establish, in steady-state conditions, an equilibrium temperature of the ATS between the inner tank at 4.2 K and the external tank at 77 K. 20 K is reached with no additional heat applied. In order to set the temperature of the ATS, a 60 Ω electrical resistance heater which provides power in the 5-6 W range, has been placed to provide a heat source controlled by four CERNOX® temperature sensors, which are distributed along its length.

Initial simulations of the system using ANSYS® show that the optimal position for the heater is on the bottom part of the ATS in order to minimize the temperature gradients. An important feature of the test cryostat is the measure of the pressure underneath the MLI blanket through an integrated pipe connecting the sample vessel to the outside flange. This information is important to check the residual gas pressure from outgassing of the MLI sample that is directly related to molecular conduction within its layers.

A process and distribution diagram of the system is shown in Figure 2.

**Figure 1.** Schematics of test inner part of the cryostat for MLI sample performance measurement (left) and pictures of the closed (centre) and open cryostat (right).
Figure 2. Process and distribution diagram of the system showing the instrumentation separation of the two helium circuits and the instrumentation used for the experiments.

3. Commissioning

3.1. Cryostat performance

Once the cryostat is closed, with the test blanket installed, it takes approximately 3 days to reach a vacuum level of 2E-7 mbar and a stable temperature of the thermal screen.

The ATS reaches a temperature of 20 K with no additional power applied with a gradient within 1 K along its length. The power to be applied on the ATS heater to reach 60 K is approximately 6 W and the gradient remains within 1 K.

3.2. Calibration curve with zero radiation

The cryostat’s background heat load, providing a systematic error, was measured by reducing the ATS temperature, via a special support, down to about 11 K, thus cancelling its radiation heat contribution. The test vessel was covered with a 10 layer MLI blanket with a packing density of 20 layers/cm. A calibration curve was built in these conditions in order to directly correlate the mass flowmeter signal in volts to the external heat load coming into the tank. A four wire measurement of the electrical heating power in the test vessel allows to directly correlate the signal of the flowmeter to the heat flux. The effect of the gaseous helium filling the volume created by the evaporation of liquid is therefore taken
into account as well as effects from the tubes precooling by the exhaust flow. As a result, a systematic background error heat load of 20 mW was extrapolated, as can be seen in Figure 3.

![Figure 3](image)

Figure 3. Calibration curve made with ATS at 11K: several electrical heater heat loads are applied to be able to extrapolate the residual heat value of -20 mW at 0 V signal.

3.3. Data acquisition and data reduction
Relevant data recorded during the experiments include the helium exhausted mass-flow out of the test vessel and its pressure and the temperature along the ATS. A level meters is also present in both LHe vessels and temperature sensors are on the LN$_2$ vessel.

Data is recorded when the system is in steady state. By experience it has been found that stable conditions appear after 1 h after setting of the heaters. A sampling rate every 20 seconds has been found to be sufficient for the time response of the system. For each test configuration, a measurement window of 10 minutes at steady state provides a sample of 30 measurement points of which a mean value is calculated. The mass flow controller (Brooks® 5850E) data indicates an accuracy of ±1 % of the reading on the full range, but measurement campaigns have shown an overall statistical error of ±12 % of the mean value is present over the range exploited for the experiments.

In order to enhance the precision of the measurements, as the flowmeter is used in its low range, for each configuration a set of measurements is taken by imposing some heat in steps on the test vessel. The points are interpolated to reduce uncertainty and a new voltage at zero heat applied is calculated from the fitting curve to correct the mean value found with the direct measurement. A correction of ±2.5 % is found in average. The value found is then converted to heat flux through the curve at zero radiation.

3.4. First tests results and discussion
First tests were performed with three different blankets with ten layers of aluminized Mylar of the type used in LHC with three different packing densities (10, 20 and 30 layers/cm). Tests have been repeated for each configuration in order to evaluate reproducibility of results and are presented in Figure 4 after having corrected the systematic and statistical errors.

The results seem to be uninfluenced by the temperature of the screen below 60 K when a blank aluminium ATS surface is used. The overall signal is too low and difficult to be reproduced for the same configurations. It seems that remaining variations are not covered in the extensive calibration procedure. After performing some tests with the aluminium screen at low emissivity, it was found that the noise-to-signal ratio was too big for a direct comparison of different blankets.

In order to increase the measurement sensitivity it was decided to increase the emissivity of the inner part of the shield by black coating it with a vacuum compatible varnish (DAG 502®). No MLI insulation was applied on the test tank for a first test which could be compared with a simple radiation heat transfer between two cylindrical surfaces. The test was performed to check the heat transfer for a higher emissivity warm boundary. Results show to be influenced on the temperature of the screen. The
comparison with a radiant heat theoretical curve suggests that the background heat load is around 40 mW/m² (8 mW in total) higher in this configuration.

![Figure 4](image)

**Figure 4.** Results on MLI performance for 10 layer blankets

4. Conclusions
A cryostat for thermal insulation performance tests was calibrated in order to start a test campaign for 4.2 MLI boil-off thermal performance measurement from temperatures between 20 K to 60 K. The calibration was performed with a bare test vessel (0.2 m² surface) and a hot boundary at 11 K allowing to find the systems’ background heat load by applying heat in steps on the test vessel. The calibration curve found is used for the correlation between the signal of the thermal flowmeter and the heat applied. The cryostat was found to have a background heat load of 20 mW. The results of tests on 10 layers MLI blankets show a big dispersion and the heat transfer doesn’t seem to be influenced on the hot boundary temperature, neither on the packing densities. Qualitative, the higher packing density would have shown a higher heat flux, as well as the higher temperature of the ATS. Being the setting unable to show this, the screen has been black coated to increase the signal and was tested without MLI. The black coated screen will be used for future experiments as the extra heat transfer due to the high emissivity of the screen shows a direct influence on the signal. The result of the test campaign will be used to check the performances of MLIs systems on thermal screens at these low temperatures. Test on degraded vacuum conditions will also be performed.

References
[1] Jacob S, 1993, *Multilayer Insulation in Cryoequipment – a Study of Reference Literature*, ISSN 0303-4003, Kernforschungszentrum-Karlsruhe-GmbH.
[2] Wiedemann C, Biering B, Lura F, *Multilayer Insulation Literature*, German Aerospace Centre
[3] Polinski J, 2006, *Cryostat For thermal measurements of Cryogenic Insulation with Evaporative Method*, Proceedings of the Twenty First International Cryogenic Engineering Conference.
[4] Barron R F, 1999, *Cryogenic heat transfer*, ISBN 1.56032.551.8, Tylor and Francis, Louisiana