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**Multilayer Insulation Testing at Variable Boundary Temperatures**

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MULTILAYER INSULATION TESTING AT VARIABLE BOUNDARY TEMPERATURES

By

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In dedication to my birth mother and adopted father:
Cynthia Anne Hurd (November 11, 1963 - June 10, 2010)
Warren Porter Hudson (March 28, 1957 - October 26, 2012)
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ABSTRACT

The National Aeronautics and Space Agency (NASA) is working toward going back to the Moon and beyond. Currently they are developing a dewar that can store liquid hydrogen (LH\textsubscript{2}) and liquid oxygen (LO\textsubscript{2}) in space so the capsule may refuel past the moon in order to continue toward Mars. However, thermal radiation in space threatens to boil the cryogenic fuels, which exist at 20 K (LH\textsubscript{2}) and 90 K (LO\textsubscript{2}), before they may be utilized. This is why NASA has been testing MLI samples to determine the most suitable insulation to protect the cryogenic storage vessel from the thermal radiation.

Florida State University (FSU) Cryogenics Group has a specialized calorimeter, known as the Multilayer Insulation Thermal Conductivity Experiment (MIKE), that can test multilayer insulation (MLI) at variable boundary temperatures without the use of cryogens such as liquid nitrogen (LN\textsubscript{2}) or liquid hydrogen. MIKE is particularly beneficial due to its ability to test at liquid hydrogen temperatures without any LH\textsubscript{2}, which is explosive and requires special safety considerations. NASA has requested the Cryogenics group at FSU to test several samples of MLI on MIKE so they may know how these samples would behave under proposed operating conditions. The calorimeter is made from two concentric copper cylinders that are cooled by a cryogenic refrigerator with their respective temperatures actively controlled with though Minco heaters. A thermal support rod suspends the inner cylinder and has a calibrated thermal conductance so it may also measure the heat load through the MLI.

Two samples of a new Load Bearing MLI (LB-MLI) developed by Quest Thermal Group (questthermal.com) and three samples of traditional MLI with a Velcro\textsuperscript{TM} (tMLI) seam developed by YetiSpace, Inc (Huntsville, AL) were sent to FSU to be tested. First the LBMLI and the tMLI were tested at boundary temperatures of 20 K and 90 K. Afterward the 5-layer sample of LBMLI was tested at boundary temperatures of 77 K and 293 K to compare results on the same material tested at NASA Kennedy Space Center.
CHAPTER 1
INTRODUCTION

There was a difference of 66 years between the Wright Brothers first flight and Neil Armstrong putting the first footprints on the surface of the Moon. Recently there has been a massive push go back to the Moon. The main propellant used in these rockets that ignite with an amazing explosion to take man out of the stratosphere is liquid hydrogen (LH₂) oxidized with liquid oxygen (LO₂), both of which are in a group of fluids known as cryogenic liquids. Cryogenics is the study of material properties at temperature lower than 120 K[2]. At these temperature ranges, elements that are gases at room temperature (300 K) are either solid or liquid at cryogenic temperatures.

The National Aeronautics and Space Administration (NASA) has come up with an idea for launching a mid space refueling station between Earth and the destination to lighten the weight of the fuel per launch. LH₂ in its saturated liquid form at atmospheric pressure is 20 K and LO₂ is roughly 90 K, which causes the concern for boil off of these cryogens due to thermal radiation in space. The idea is to have a storage tank that uses a radiation shield at 90 K to reduce the heat transfer to the liquid hydrogen that NASA needs to store at the refueling station.

The motivation behind the present experiment is to test cryogenic insulation samples at boundary temperatures that NASA cannot because of limitations to the experimental apparatuses at Kennedy Space Center (KSC). They have requested the testing facilities of the Multilayer Insulation Thermal Conductivity Experiment, also known as MIKE, at Florida State University (FSU). Multilayer insulation is considered the best passive form of insulation in high vacuum environments.

1.1 Modes of Heat Transfer

Heat transfer is a form of energy movement as a result of difference in temperature. The movement of heat is studied in order to increase the efficiency of heat engines or form good performing insulation. There are three modes of heat transfer that must be considered when studying thermal transport: conduction, convection, and radiation.
1.1.1 Conduction Heat Transfer

Conduction is the energy transfer through a medium by molecular motion or in the case of solid lattice vibrations given a temperature difference across the medium[1]. Typically conduction is most important in solid media as there is no induced movement with the exception of lattice vibration. When a heat source, such as an electric heater, is placed on one side of a solid body a thermal gradient forms through the body toward the lower temperature. Figure 1.1 shows a simplified one dimensional conduction scenario in which heat flows from the higher temperature (T_H) side toward the lower temperature side (T_C).

The heat (energy) diffuses through the material at different rates depending material properties. Every material has different thermal properties resulting from their atomic structure, electronic structure, crystalline structure, and density. Thermal properties to consider are the specific heat, thermal conductivity, and thermal diffusivity. Specific heat refers to the amount of energy necessary to raise one kilogram of a material one degree Kelvin. Thermal conductivity is the rate of unit heat transfer through a thickness of material per unit area [3]. Mathematically, the conduction process in one dimension is described by Fourier’s law:

\[ Q = kA \frac{dT}{dx} \approx kA \frac{\Delta T}{L} \]  \hspace{1cm} (1)

where \( Q \) is the heat load, \( k \) is the thermal conductivity, and \( \frac{dT}{dx} \) is the thermal gradient across the length or thickness of the material. However, the thermal conductivity can also be a function of temperature, meaning that \( k = k(T) \). Therefore, a better representation overall for the overall heat conduction would be:

\[ Q = k(T)A \frac{dT}{dx} \]  \hspace{1cm} (2)

Equation 1 can typically be used in very small temperature differences when an approximate answer satisfies the question. Equation 2 is used for a more finite \( \Delta T \) when a known temperature dependant thermal conductivity is known.
1.1.2 Convection

Convection is a combination of conduction heat transfer and advection, the transport mechanism through bulk fluid flow [4]. The mathematical equation to describe convection is known as Newton's law of cooling:

\[ Q = hA(T - T_\infty) \]  \hspace{1cm} (3)

where \( A \) is the area of the surface, \( T_s \) is the temperature of a surface, \( T_\infty \) is the ambient temperature, and \( h \) is known as the convective heat transfer coefficient. Due to its complexity and dependence on numerous variables, the heat transfer coefficient is usually determined experimentally. There exists two types of convection: forced and natural. Forced convection occurs when an external source such as a fan induces the fluid flow [3]. Natural convection induces fluid flow with the combination of gravity and density gradients within the fluid.

1.1.3 Radiation

Life would not exist on Earth without thermal radiation. It is the mode of energy transfer that brings warmth from the sun. Thermal radiation is transferred via the electromagnetic spectrum, light, therefore no medium is required to move energy from a hot body to a cold body. Light is emitted as a result of energy transition of molecules within a substance. Radiation is typically negligible around room temperature (300 K) in ambient air since its effect on a body is usually orders of magnitude lower when compared to convection, however it always exists.

When a body has an emissivity of unity it is considered a black body whose emitted radiation energy can be calculated as:

\[ E_b(T) = \sigma T^4 \]  \hspace{1cm} (4)

\( T \) representing the temperature of the body and \( \sigma \) representing the Stephen-Boltzman's constant which is equal to \( 5.670 \times 10^{-8} \frac{W}{m^2K^4} \) [3]. Radiation between two or more bodies depends on a number of factors such as the temperature difference of each body, its view factor, the geometries and emissivity. View factor, also known as the shape factor or the angle factor, is a ratio from zero to one on how a radiating body "sees" another [3]. The view factor is a purely a
geometrical factor. Net radiation heat flux between two infinitely large parallel plates with a view factor equal is:

\[ q_{12} = \sigma(T_1^4 - T_2^4) \text{ if } \varepsilon = 1 \]  

(5)

If \( \varepsilon < 1 \) then net radiation heat flux is,

\[ q_{12} = \frac{\sigma(T_1^4 - T_2^4)}{\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1} \text{ if } \varepsilon < 1 \]  

(6)

**1.2 Overview of Cryogenic Insulation**

There exists various forms of insulation to reduce the boil off rate of cryogenic liquids whose temperature is below 120 K. Liquid helium, hydrogen, oxygen, argon, nitrogen, etc are extremely cold substances that would evaporate rapidly at room temperature if they are not properly stored. It takes a lot of energy to condense a room temperature gas into a liquid so in order to store these cryogen effectively they must be insulated from the outside environment. Some insulations are more effective than others but each one has its own advantages and disadvantages.

**1.2.1 Multilayer Insulation (MLI)**

MLI is considered to be the most effective form of cryogenic insulation. It consists of multiple layers of Mylar with a thin coating of aluminum or gold with low emissivity in order to reflect most of the thermal radiation and minimize the heat exchange (equation 5). A material of low thermal conductivity is installed between each reflective layer to reduce the conduction heat transfer from layer to layer.

Despite MLI being a very effective form of insulation it does have some disadvantages. First, there needs to be a hard vacuum, below 1\( \times 10^{-3} \) torr, to be fully effective. Maintaining pressures of this range is often a challenge for large systems. Another drawback is the often tedious installation of MLI, which requires careful attention in order to reduce any thermal linking between layers. MLI will be discussed in greater detail in Chapter 2.
1.2.2 Powder Insulations

Another form of cryogenic insulation is microscopic glass powder insulations consisting of glass microspheres with diameters of 10-1000 μm [2]. Powder insulations are cheaper, easier to install, and a hard vacuum is not required for them to work effectively for cryogenic storage. However in comparison, powder insulations are inferior to MLI. Powder insulation do still have fluid between the particles so convective or gas conduction heat transfer is still present. Fluid can be removed, however it is difficult to filter the powder insulation from being sucked into the vacuum pump. Also, powder insulations increase the outer area of the cylinder which effectively increases conductive heat transfer before reaching a critical thickness. Once this critical thickness is attained, the powder insulation does serve a purpose. Typically powder insulations are used to insulate liquid natural gas, liquid nitrogen, liquid oxygen or liquid hydrogen for NASA KSC storage tank.

1.3 Scope of Thesis

The scope of this work is to use an original type of calorimeter (MIKE) at the National High Magnetic Field Lab (NHMFL) to test the effective thermal conductivity of two differing types of MLI at nominal boundary temperature of 20 K and 90 K. The first type is a new self supporting MLI known as Load Bearing MLI (LB-MLI). Traditional MLI needs support and when mounted its effectiveness is compromised when a compressive load is placed upon it. Instead of using a polyester netting in between layers, LB-MLI has polymeric spacers in between layers that prevents the outer layers from touching and slipping relative to one another. The second type of MLI has a Velcro™ seam to mount the MLI without the need of aluminized Mylar tape. Velcro seam MLI is considered to be a type of traditional MLI in that there is a polyester separator.

Chapter 2 will begin with an MLI review discussing work that has been described in the past and methods used to measure heat flux through the MLI. Theory behind the analysis of MLI heat transfer will be explored followed by an explanation of the experimental apparatus in detail. Finally, the results of the experiment will be discussed and compared to theory.
CHAPTER 2

MLI REVIEW AND THEORY

All three heat transfer modes must be considered when designing an insulation system. Conduction and convection are normally the only modes considered when dealing with normal room temperature systems. However, heat transfer can be greatly reduced when reducing a number of factors to eliminate either modes of heat transfer. One method is to remove all interstitial gas in the system. Convection heat transfers reduces to zero once all fluid is removed. Conduction heat transfer can be reduced by eliminating contact between surfaces however this is hard to do outside if there is a load between two surfaces. Thermal radiation can be greatly reduced by placing reflective shields between radiating bodies.

2.1 Multilayer Insulation

MLI was first developed by Peterson in 1958 and later improved over the decades with over 120 research papers added to the Advances to Cryogenic Engineering from 1960-1980 [10]. Over the years MLI has been researched in many formats, styles, and configurations, as discussed in the next section. A majority of the research occurred during the Apollo program, peaking in the 1960's and decreased in the late 1970's. Multilayer insulation is considered the best passive cryogenic insulation method to reduce the overall heat transfer. However, MLI as stated previously, works best under high vacuum conditions with pressure below $10^{-3}$ torr.

2.1.1 Radiation Shields

The thermal radiation shields are made of either metal foil or a metal coated polymer, typically Mylar, depending on the needs of the system. If the insulation will experience temperatures greater than 300° F, the pure metal foils are used to avoid melting of the polymer. If extreme temperature are not expected then metal coated polymers are chosen since they are cheaper, lighter, and have lower thermal conductivity compared to pure metal foils [9]. Several metals are considered for radiation shields based on emissivity and cost; these include aluminum, copper, silver, and gold. Aluminum is cheaper and easily vaporized compared to the other metals so the manufacturing process can have a better quality than silver or gold. Gold has a lower emissivity but costs more compared to aluminum.
One parameter that is not taken into account in these equations is the thickness of the metallic coating on the Mylar. The coating level is typically 500 Å at minimum and applied on one side of the Mylar or occasionally on both sides. When aluminum coating is on both sides it is known as Double Aluminized Mylar (DAM). [2]. Literature, however, seems to disagree on the effects of the thickness of the metallic coating. Flynn and Green mention that the thickness does matter in that below 500 Å the thermal shields can be transparent to the radiation, especially at lower temperatures. Green states that the thickness of the metallic coating should increase with decreasing temperature (i.e. 300 Å at 300 K and 300,000 Å at 4 K) [9,11]. However a test done by Borski, Nicol, and Schoo at Fermi National Accelerator Laboratory shows that this may not be true. Refer to Figure 2.1

![Figure 2.1 Heat flux measurements comparing aluminized Mylar thickness [11]](image)

Fermi National Lab tested two 10 layer samples of DAM MLI of the same geometry with a warm boundary temperature of $T_C = 20$ K and $T_H = 80$K. The only difference between the two samples was the thickness of the aluminum coating, one being 600 Å and the other 350 Å. When comparing the two data sets it can be seen that there is not any difference between the two thickness. This argues against Flynn’s statement that thicknesses below 500 Å are transparent to the radiation.
2.1.2 Insulation Spacer

The insulating spacers serve to prevent layer-to-layer conduction eliminating thermal shorting. Several low conductivity materials are used: Polyethylene terephthalate (Dacron), glass paper, silk, paper, fiberglass, nylon and foam. [2,3,10,11]. Each insulation spacer material is chosen based upon its thermal stability, thickness, and tensile strength when applicable. There are several techniques for mounting the insulating spacers. The first technique is total coverage between layers with fiberglass paper, silk screens, nylon netting, etc. These forms of MLI must be installed carefully when layers are not connected or sewn together to prevent slipping. Another technique is to use point contact spacers to minimize the total area of contact between spacers and radiation shields. Spherical or embossed spacers are glued to the radiation shield in a grid pattern in order to prevent slippage between the layers using a stepped seam to make installation easier. [9,13]. Another benefit of using point contact spacers over fibrous sheets is a larger gap for gas evacuation thus reducing out gassing or pump down time.

2.1.3 Layer Density

Another factor that is considered in the development of MLI is the layer density, the number of layers per unit thickness. As with all thermal insulation the amount needed can be optimized to find a balance between decreasing modes of heat transfer. Figure 2.2 from Leung shows a graph for optimization between conduction heat transfer and thermal radiation [14]:

![Figure 2.2: Layer density optimization from Leung [14]](image-url)
Leueng found for his particular sample of MLI the optimal density was about 0.4 layers per millimeter. A less dense material's heat transfer would be dominated by radiation alone while more the 0.4 layers per millimeter would be dominated by solid conduction. For less dense materials there is not a lot of protection against the incoming thermal which would leak through more easily. On the other hand, the more dense the material the more contact exists from layer-layer allowing heat to flow easily from one layer to another by conduction. Therefore, depending on the emissivity of the radiation shield and the thermal conductivity of the insulating spacer, an optimization needs to be determined.

2.2 Heat Transfer Analysis

As previously stated all three modes of heat transfer need to be considered in predicting how well a sample of MLI will work. Heat leaks through multilayer insulation is dominated by radiation at low layer densities and conduction at high layer densities so the most ideal scenario would be the floating shield theory:

\[ q_{12} = \frac{\sigma(T_H^4 - T_C^4)}{\left(\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1\right) * N} \]  

(7)

where equation 7 is the thermal radiation equation divided by the number of shields (N). However in real systems it is difficult to create a "floating shield" without connecting to a supporting body. Layer to layer heat conduction is introduced as a result of the need to support the individual layer requiring an insulation spacer to be introduced making the sample more effective. The downside is the samples effectiveness is degraded with more layers. Convective heat transfer is minimal but in the kinetic theory-free molecular range gas conduction heat transfer but should still be considered. In order to predict the heat transfer there are two models that are used: The Layer by Layer model developed by McIntosh[16] and the Lockheed equation[15].

McIntosh's Layer-by-Layer model is the summation of all three heat fluxes. The flux due to radiation is the same as equation 4, however the heat flux due to conduction and convection are special cases [16]:

\[ Q_{\text{gasconduction}} = C_1 * P * \alpha * (T_H - T_C) \]  

(8)
P: gas pressure  
$T_H, T_C$: hot and cold boundary temperatures  
$\alpha$: accommodation coefficient  
$C_1: \frac{\gamma+1}{\gamma-1} \left[ \frac{R}{8 \pi^2 M T} \right]^{0.5} = 1.1666$ for air  
R: Universal Gas constant  
$\gamma$: heat capacity ratio  
M: molecular weight of gas  
T: temperature of the vacuum gauge  
$q_{\text{conduction}} = \frac{C_2 f k}{DX} (T_H - T_C)$  
(9)  
$C_2$: is an empirical value for the insulating spacer  
f: separator density  
k: spacer thermal conductivity  
DX: thickness of the spacer material  

The heat flux due to gas conduction differs from the convection equation in Chapter 1 because the gas is in the free molecular regime. Again this equation is for the Layer-by-Layer model so it does not take into account layer density, which is taken into account in the Modified Lockheed equation:

$$q_{\text{total}} = \frac{A(N^\#)^{\text{fit}} T_{\text{fit}} (T_H - T_C)}{N} + \frac{B \sigma (T_H^{4.67} - T_C^{4.67})}{N} + \frac{C P(x,T) (T_H^{m+1} - T_C^{m+1})}{N}$$  
(10)  
$q_{\text{total}} = q_{\text{conduction}} + q_{\text{radiation}} + q_{\text{convection}}$  
N: number of layers  
$N^\#$: layer density  
A, B, C, m, n: are derived for the particular insulation system, and interstitial gas  
P(x,T): pressure based on temperature and position along MLI
CHAPTER 3
EXPERIMENTAL DESIGN AND PROCEDURE

3.1 Experimental Design

The main goal of this project was to measure the performance of a Load Bearing MLI (LB-MLI) developed by the Quest Thermal Group (Denver, CO) for the purposes of NASA Kennedy Space Center (KSC) in Cape Canaveral, FL. Another form of MLI developed by YetiSpace (Huntsville, AL) using a Velcro® seam was also tested in this project. NASA is currently developing a dewar that has a cryogenic refrigerator keeping an active thermal shield at 90 K [17]. However, researchers at KSC are limited to the boil off calorimetry for MLI measurements using liquid nitrogen (LN$_2$) with boundary temperature of $T_C = 77$ K and $T_H = 300$ K and above. Thus, KSC requested additional measurements using the specialized calorimeter developed by the FSU Cryogenics group [18] to test MLI samples at real performance boundary temperature, $T_C = 20$ K and $T_H = 90$K. Afterward a measurement at $T_C = 77$ K and $T_H = 300$ K was made in order to benchmark our method used at against the boil off calorimetry technique used at NASA KSC.

The Multi-layer Insulation Thermal Conductivity Experiment, also known as MIKE, is a calorimeter composed of two concentric copper cylinders, shown in Figure 3.1. The warm cylinder is suspended from a room temperature flange, while the cold cylinder is suspended from the warm cylinder top plate. The warm cylinder is 1524 mm in length, 2.3 mm thick copper sheet rolled to a diameter of 272 mm while the cold cylinder is 1219 mm in length, 3.2 mm thick copper pipe with a outside diameter of 191 mm. Each cylinder is capped off with copper plates 12.7 mm thick. [18] Both top plates are suspended from their respective points with three stainless steel threaded rods each cut in half and bonded with Stycast into G10 epoxy tubing in order to minimize the heat flow from the higher temperature bases. A small hole is drilled in the G10 tube so any residual air may be removed when the system is evacuated. The cold cylinder is connected to its top plate with a one inch diameter rod that is threaded on both ends and screwed to each connection point. This rod is the critical piece when measuring the heat loads and will be discussed later on in this section.
MIKE differs from other MLI performance test apparatus in that the only cryogen necessary is (LN$_2$) for the cooling jacket on the outside of the cryostat. The main cooling component is a cryogenic refrigerator mounted on the top flange and connected to the top cold plate with a flexible thermal link. Cryogenic refrigerators are systems that compress and expand helium gas to cause a cooling effect. A Cryomech Gifford-McMahon (GM) AL-60 cryogenic refrigerator is used for the 77 K - 300 K tests while a Cryomech Pulse Tube (PT) 810 is used for the 20 K - 90 K tests. The PT-810 is a two stage pulse tube cryogenic refrigerator whose capacity was able to cool the cold cylinder via the second stage and the warm cylinder via the first stage. The AL-60 cryogenic refrigerator was used for the 77 K to 293 K measurements. The PT-810 which has much more cooling power was used for the 20 to 90 K tests. The cryogenic refrigerators are attached to their respective cylinders by means of a flexible thermal link. This thermal link is made by soft soldering copper braided wire on to small copper plates that are
bolted to their respected surfaces, the cold head and the cold top plate. The temperature of each cylinder is controlled through its respective top plates.

The heat load is measured via a calibrated thermal link that connects the cold cylinder to its top plate. As radiation emits through the MLI sample to the inner copper cylinder, that heat moves toward the center and continues up the thermal link toward the top plate. In Figure 3.2 the thermal link can be seen attached to two copper support flanges, the top flange being the top support and the bottom being the support flange that two 3.2 mm thick copper pipes were brazed to complete the inner cylinder. The two attach points are the only contact the cold cylinder has with the cold plates so all heat flow must move through the this thermal link. Any heat load from there can be measured taking advantage of Fourier's Law which was integrated to

\[ Q = \theta(T) \Delta T \]  

(11)

where \( \theta(T) = \frac{kA}{l} \)  

(12)
where $Q$ is the heat load, $k$ is the thermal conductivity, $A$ is the cross sectional area and $\frac{\Delta T}{L}$ is the average thermal gradient over a given length. Two rods were used for the purposes of this experiment: Aluminum 6061-T6 was installed for the 20-90 K measurements and an oxygen free high thermal conductivity (OFHC) copper rod was used for the 77- 300 K measurements. The support rod for the previous experiments between 300 K and 60 K was made of copper to allow the heat to easily flow to the cold base due to the high thermal conductivity. In that case, the total heat load was on the order of 1 W [3]. However, it was estimated that the current boundary temperatures( 90 K and 20 K) would lead to a low heat load, estimated to be of order 100 mW. Low thermal resistance coupled with a low heat load leads to too small a temperature difference to measure accurately. Below 77 K the Cernox sensors have an instrumental error of ±16 mK leading to a larger uncertainty of the measurements. Therefore a different material with a larger thermal resistance was needed to be able to take measurements within the resolution of the sensors to obtain approximately the correct thermal resistance. An Aluminum 6061 rod of the same dimensions was chosen to replace the copper support rod [13].

3.2 Hardware and Software

3.2.1 Temperature and Pressure Measurement

Two types of Lakeshore Cryotonics temperature sensors were used for this experiment. The first type were six Cernox® 1070 copper bobbin Thermal Resistance Detector (RTD) positioned on the thermal link, the top plates, and outside the warm cylinder. These Cernox 1070 sensors are mounted with a brass 4-40 screw placed though the sensor and bolted to its surface. A small amount of copper grease is placed on the joining surfaces in order to cut the contact resistance as much as possible. The second type of sensor was four Cernox 1050 surface mount sensors that were mounted in between MLI sheets. These 1050s were mounted by taping them to the surface with Aluminized Mylar tape. Locations for the 1050s were as follows: 1 on the inner layer, two on the third layer counting from the inner layer one 5 inches deep and one 1 inch deep, and one sensor on the outer layer. Each sensor, excluding the 1070s located on the warm cylinder, was calibrated between 15- 300 K. The calibration process is described in Section in 3.3. The RTD sensors uses the four wire scheme by keeping either a constant voltage or a constant current and measuring the resistance, which in turn is converted into a temperature from
the calibration. For the current experiment the sensors were excited a constant current of 3.16 μA.

The pressure for the experiment was measured using an Edwards wide range vacuum gauge connected at the top of the room temperature flange. For this experiment the vacuum level was requested to be $10^{-6}$ torr or better.

### 3.2.2 Data Acquisition and Temperature Control

In order to keep track of all the temperature sensors throughout the system a Lakeshore® Alternating Current Resistance Bridge 370 was used to power and read each sensor individually with the exception of the temperature sensors used to control the temperatures within the system. The sensor located on the warm and cold top plates were powered and measured by a Lakeshore® 340 Temperature Controller. Additionally the Lakeshore® 335 Temperature Controller controls the radiation shield above and below the cold cylinder. Occasionally, raising the temperature of the cold head required a much higher heat load than the temperature controllers are able to give so extra heaters were added to independently heat the cold head of the cryogenic refrigerator. For example the AL-60 has a cooling capacity of 59 W at reach 77 K, as seen in the capacity curve in Figure 3.5. However the temperature controller Lakeshore 340 is only capable of 47 Watts. It is not desirable to operate temperature controller at 100% capacity as the local heat flux may leak toward the thermal link disrupting the measurement. A block diagram for the electrical set up is shown in Figure 3.3. In the block diagram, the black arrows represent the resistance reading from each temperature sensor being sent to the Labview® computer program. The dashed lines represent feedback in terms of heat in order to control the temperature based upon the reading. Therefore, independent heat sources were needed in order to run experiments at the higher temperatures. All of these processes are controlled remotely from a Labview program. Labview reads every channel on the Lakeshore 370 AC Bridge and the temperature controllers and records all measurements into one string of data. Once the resistances are recorded, each measurement is then sent through their respective calibration curves and the heat load is calculated and recorded. Each sensor was allowed to settle for 45 seconds as using two different types of sensors caused an impedance requiring enough settling time to obtain a proper reading.
Figure 3.3: Block diagram of electrical set up
Figure 3.4 Experimental electrical set up

Figure 3.5: Capacity curve for the AL60 cryogenic refrigerator
3.3 Calibration

3.3.1 Temperature Sensors

The temperature sensors were calibrated using a liquid helium boil off cryostat and compared to the reading of a calibrated sensor (Lakeshore Cryotronics). All the sensors were placed in this cryostat, cooled in a bath of liquid helium and allowed to slowly warm to room temperature while receiving a constant current supply of 3μA from a Lakeshore Model 100 direct current source. Every thirty seconds a voltage measurement of each sensor was recorded.

Figure 3.6 Capacity curve for PT-810 cryogenic refrigerator
Knowing the current and measuring the voltage drop the resistance at that moment was calculated using Ohm's law. These resistances were then compared to the temperature measured by the calibrated sensor at the same moment. These data then gave a temperature versus resistance curve that is used to formulate a calibration equation to calculate the temperature given a resistance. The Chebychev method was used to fit the data for each sensor. (See Appendix A for the Chebychev method).

### 3.3.2 Thermal Rod

Both rods needed to be calibrated for the inverse thermal resistance as a function of temperature, $\theta(T)$:

\[ \theta = \frac{kA}{L}(T) \]  

(13)

\[ Q = \theta \Delta T \]  

(14)

at various temperatures in the measurement region as the thermal conductivity is dependent on temperature and. Richard Klimas calibrated the copper thermal rod in 2011 for use between 300 K and 60 K. The Al6061-T6 rod was calibrated early 2013 for 20 K to 90 K heat loads (~0.1 W) The thermal rod calibration set up is shown in Figure 3.6 and the results are in Figure 3.7 for the copper rod and Figure 3.8 for the aluminum rod.

A calibration of the thermal rod is necessary due to the irregularities in the geometry: flat surfaces for temperature sensors and threaded ends of the thermal rod. In order to calibrate the thermal rod, it is connected to a cryogenic refrigerator that is powerful enough to reach the range for calibration. A copper radiation shield is mounted to the cryogenic cooler to serve as an active radiation shield from any outside thermal radiation sources. Two Cernox 1070 copper bobbin sensors were bolted with 4-40 screws on two different points along the rod as seen in Figure 3.6. An electric heater is placed at the opposite end of the thermal rod to simulate a heat load from the system. Once the system has cooled to the desired temperature, a constant current flows from a Tenma 72-7245 power supply through the electric heater in order to form a thermal gradient across the rod. A Keithley 7000 Voltmeter connected in parallel to measure the voltage.
drop across the Minco heater. The added heat is then calculated using equation 15 where \( V \) is the voltage and \( I \) is the electrical current.

\[
\text{Power} = VI = Q \quad (15)
\]

While the Tenma power source does give a reading for the sensor voltage and known resistor is placed in series with the electric heater and the voltage drop is measured. From Ohm’s law, equation 15, the true current can be calculated

\[
I = \frac{V}{R} \quad (16)
\]

where \( R \) is the known resistors. Once the heat load is calculated and the thermal gradient across the thermal rod is measured, then \( \theta(T) \) may be calculated by the ratio of the heat load divided by the temperature difference. This process is repeated several times across multiple base temperatures controlled by the Lakeshore® Temperature Controller 340. After a range of \( \theta(T) \)
are measured, they are plotted over the average temperature in order to fit a calibrated curve for a specific temperature.

![Graph](image)

**Figure 3.8**: $\theta(T)$ curve for copper thermal link [15]

![Graph](image)

**Figure 3.9**: $\theta(T)$ coefficient for the aluminum thermal link [13]

### 3.4 Experimental Procedure

#### 3.4.1 Installation

As mention before, the traditional MLI it made of aluminized Mylar sheets and a polyester netting spacer to minimize radial inter-layer heat transfer via conduction. This form of MLI is effective but tedious to install to assure there is no layer-to-layer contact. LB-MLI is
different in that it uses polymeric spacers that are glued onto each layer preventing the layers
from touching or slipping. The size and material of the spacers are proprietary information so
spacer description and analysis is limited. As each layer is folded placed on its spot, a series of
small aluminized Mylar tape strips are placed in order to close the seam to outside heat sources.
LB-MLI is also able to support itself by being installed tightly around the cold cylinder. Cernox
1050 sensors are installed on each layer during installation.

The second set of MLI is a form of traditional MLI but instead of each layer being free it
is connected with a sewn seam and has a Velcro seam for its overall installation. Traditional MLI
is not self-supporting unlike the LB-MLI counterpart so dental floss had to be tied onto the
plastic tags keeping the MLI from slipping then held up by tying the other end to the G10 disk
located between the upper radiation shields. Reflective Velcro strips are placed on the inside of
the MLI, and once the dental floss suspends the sample on its on the outer reflective strip is put
in place. Installing the Cernox 1050s was done prior to the MLI installation which had to be
done carefully given the sensitivity of the 1050s and how close the each MLI sheet was. First
the sensor had to be put in its proper place after which the aluminized Mylar tape was put on the
end of a tongue depressor and placed over the sensor. Once everything was installed, pressure
was placed over the sensor on the outside of the MLI to endure the best thermal contact possible.

3.4.2 Experimental Procedure

Once the MLI is installed onto the cold cylinder, an instrumentation check is performed
making sure all sensors are working with no electrical shorting of wires before the experiment
continues. All temperature sensors are connected to the electrical blocks and room temperature
measurements are compared to previous room temperature measurements to make sure there are
no unseen electrical shorts. It is important to make sure all wires coming from the top are heat
sunk to the cold top plate to eliminate any conduction heat leak to the thermometers. Once all the
thermometry checks out, then MIKE is lowered into the warm cylinder. Four aluminum brackets
on the inside are bolted down with connecting copper tabs for good thermal contact between the
warm top plate and its outer cylinder. Again, copper grease is applied between the two surfaces
to insure good thermal contact. Afterward the outer heaters and temperature sensors are
connected to its connecting block. Extra traditional MLI is installed on the warm cylinder when
performing the 20 K- 90 K measurement to reduce any radiative heat leaks from the outside of the cryostat, but for the 77 K- 300 K the extra MLI is unnecessary.

After doing the previously stated tasks, MIKE is lowered into the cryostat and bolted down. Once the instrumentation is checked, a turbo molecular vacuum system is connected to the designated port and evacuation of the system is started with all electronics online. Pressure levels are monitored and once the system reaches $10^{-5}$ torr, typically after 12-24 hours, the cryogenic refrigerators are started. LN$_2$ must be filled into the cryostat's jacket when doing to 20 K-90 K experiments in order to reduce radiation heat transfer from the outside walls. The system is cooled to a few Kelvin below testing parameters to ensure steady state criterion. Once in steady state the Labview program was launched and testing was carried out for approximately three days.

As soon as testing is completed the cryogenic refrigerators were shut down, the temperature controllers set to warm to 280 K and addition heaters connected to a power source in order to warm up quickly. MIKE can be brought out of the cryostat as soon as the temperature controllers read 0% capacity.
CHAPTER 4

RESULTS AND CONCLUSION

This chapter reports the results from the experiment and compares them to previous tests performed by other labs on the same material that section then followed by conclusions and suggestions for future work. Some of the results presented here were published in *Advances in Cryogenic Engineering*, Proceedings from the Cryogenic Engineering Conference 2013 in Anchorage, AK.

4.1 Load Bearing MLI at 20 K and 90 K

NASA KSC tests samples of MLI using the boil-off calorimetric method, which is limited to the temperature of the boil off cryogen (usually LN\(_2\)). MIKE has the ability to hold different boundary temperature based upon testing requirements. The goal for NASA is to set up a refueling station midway between Earth and a new destination, so minimizing boil-off of liquid hydrogen is critical. The Quest Thermal Group (Denver, CO) sent two samples of their MLI known as load bearing MLI (LBMLI) since it is able to take a compressive load. The LB-MLI samples are 5-layer and 10-layer samples with a density of 5 layers per centimeter. A unique feature of this MLI is its closing seam which allows for an easy installation by focusing on one layer at a time. Unfortunately, a picture cannot be shown as this LB-MLI is proprietary and this forum cannot discuss descriptive detail any further. The stability of MIKE and heat transfer can be observed for the 5- and 10-layers samples respectively (Figure 4.1 and Figure 4.2 respectively). Each sample was tested over a period of three days once MIKE was determined to be near steady state. When observing at the steady state heat transfer in Figure 4.1 and Figure 4.2, there is a slight sinusoidal progression. This is can be attributed to a couple factors. First, cryogenic refrigerators work best when the cooling water is efficiently flowing through the system at low temperatures. The National High Magnetic Field Lab runs on one main cooling water system so the peaks could be a sign of multiple users in the building using the cooling water. Another observation is that there is a slight decrease in heat flux over time. The natural set up of MLI can trap air as the system is brought to lower pressures allowing some gas conduction heat transfer. However continuously pumping of the system will pull residual trapped air, a process known as out gassing. When out gassing is coupled with the filling of the liquid
nitrogen jacket the ambient pressure will decrease resulting in a lower gas conduction heat transfer. Tables 1 and 2 shows the maximum, minimum, and average per one thousand minutes:

Figure 4.1: 5-layer Load Bearing MLI at 20 K and 90K

Figure 4.2: 10-layer Load Bearing MLI at 20 and 90 K
TABLE 4.1. 5-layer Load Bearing MLI at 20 K and 90 K

<table>
<thead>
<tr>
<th>Heat Load (W/m²)</th>
<th>Day 1</th>
<th>Day 2</th>
<th>Day 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>0.213</td>
<td>0.212</td>
<td>0.206</td>
</tr>
<tr>
<td>Minimum</td>
<td>0.205</td>
<td>0.194</td>
<td>0.197</td>
</tr>
<tr>
<td>Average</td>
<td>0.210</td>
<td>0.201</td>
<td>0.201</td>
</tr>
</tbody>
</table>

TABLE 4.2. 10-layer Load Bearing MLI at 20 K and 90 K

<table>
<thead>
<tr>
<th>Heat Load (W/m²)</th>
<th>Day 1</th>
<th>Day 2</th>
<th>Day 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>0.159</td>
<td>0.157</td>
<td>0.151</td>
</tr>
<tr>
<td>Minimum</td>
<td>0.148</td>
<td>0.143</td>
<td>0.143</td>
</tr>
<tr>
<td>Average</td>
<td>0.153</td>
<td>0.152</td>
<td>0.148</td>
</tr>
</tbody>
</table>

The system appears to be cooling at a rate of around 1-2 mW per square meter per day for the 10 layer sample settling around 0.201 watts per square meter. The 10 layer sample did not cool further than what is displayed. Over all the 10 layer sample reduced heat transfer better than the 5 layer sample by roughly 50 mW per square meters or 25%, although theory states the heat transfer should have been reduced by 50%. This discrepancy may be attributed to the MLI seam or another major heat leak. Several techniques were used to try to find the excess heat leak source, however, no heat leak was found. The main technique included adding a known amount of heat through Minco resistance heater placed upon the inner cylinder and a constant current power source. Once the system came to a steady state the difference of heat load measurements with and without the additional heat were compared to the product of the voltage and current displayed by the power source. The result was only a difference of ±2 mW.

4.2 Traditional MLI at 20 K and 90 K

While the seam of the LBMLI is easy to install, removing the LBMLI can be destructive to the sample and depending on the damage may not be reusable. Yetispace (Huntsville, AL) makes a traditional MLI (tMLI) sample that has the layers quilted with a Velcro® seam. This seam allows simple, quick installation and non destructive removal in case repairs are needed on
the storage system. These samples were tested only at the 20-90 K temperature conditions for of NASA KSC. The results over time for 5-, 10-, and 15-layers are shown below (Figures 4.3-4.5):
Immediately it can be seen that the 5-layer tMLI and the 15 layer tMLI did reach a steady state while the 10-layer did not. It should be noted that after testing the LB-MLI samples, the cryogenic refrigerator developed a small leak. The leak would slowly drop pressure in the system causing an automatic shutdown when the pressure differential became too small. This limited testing time to a few days including cool down time to steady state, roughly four days. The 10 layer experiment took longer to cool so this explains Figure 4.4. In addition, the heat leaks through the 5-layer and 15-layer tMLI are roughly the same. The 3 day statistics are in the tables below (Table 3- Table 5)

<table>
<thead>
<tr>
<th>Heat Load (W/m²)</th>
<th>Day 1</th>
<th>Day 2</th>
<th>Day 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>0.227</td>
<td>0.227</td>
<td>0.219</td>
</tr>
<tr>
<td>Minimum</td>
<td>0.218</td>
<td>0.209</td>
<td>0.208</td>
</tr>
<tr>
<td>Average</td>
<td>0.224</td>
<td>0.219</td>
<td>0.214</td>
</tr>
</tbody>
</table>

Figure 4.5: 15-layer Traditional MLI at 20 K and 90 K
A large amount of outgassing is present for the tMLI, more than would be present for the LB-MLI, as the Nylon netting traps more air between the layers. An outstanding observation is that the heat fluxes through each sample is that they are all roughly equal. A few factors can be responsible for this outcome. First the seam on the tMLI was sealed by two reflective strips along the length of the straight seam. So a high heat leak can come from this seam. Another factor is the thickness of the aluminum MLI sheets. These reflective sheet were thinner compared to the LB-MLI, which could have made them transparent to the radiation heat flux from layer to layer. If the layers were transparent then the number of layers per sample would not make a difference.

4.3 Load Bearing MLI at 77 K and 293 K
Along with the 20-90 K testing, NASA KSC requested a benchmark test of MIKE at 77 K- 293 K, the temperatures KSC test with the boil off calorimetry method. The 5 layer LB-MLI sample was tested to provide an experimental comparison (Figure 4.6). The 77 K - 293 K test was allowed to cool for four days to steady state and data acquisition lasted for three days, as was the model for the previous tests. Over the three day period the test was very stable. Table 6 shows that the heat was typically 3.6 watts per square meter.
### TABLE 4.6. 5-layer Load Bearing MLI at 77 K - 293 K

<table>
<thead>
<tr>
<th>Heat Load (W/m²)</th>
<th>Day 1</th>
<th>Day 2</th>
<th>Day 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum</td>
<td>3.594</td>
<td>3.607</td>
<td>3.609</td>
</tr>
<tr>
<td>Minimum</td>
<td>3.540</td>
<td>3.571</td>
<td>3.545</td>
</tr>
<tr>
<td>Average</td>
<td>3.569</td>
<td>3.587</td>
<td>3.564</td>
</tr>
</tbody>
</table>

### 4.4 Comparison to Theory and Parallel Testing

KSC did parallel testing on the LB-MLI and presented the results at the 2013 Cryogenic Engineering Conference. The results are posted below in Table 4.7 with the results obtained at FSU. The model heat flux was determined by an Excel® program written by Wesley Johnson of KSC using the Layer-to-Layer model in order to determine the expected heat flux which made an assumption of each layer having an emissivity of 0.03. Clearly our results are higher by a factor of two or more when compared to the results from KSC. From this it can be inferred that there is a discrepancy in the measurements. Several factors can be in place such as the geometrical difference between the two systems. The Layer-to-Layer program was programmed based on the cryostats at KSC. Another factor could be the differences in size of the coupons...
used by KSC. While the thermal radiation flux should be similar, the conduction from the increased area could have affected the amount of heat measured in MIKE.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>FSU</td>
<td>5</td>
<td>77</td>
<td>293</td>
<td>3.60</td>
<td>1.751</td>
</tr>
<tr>
<td>FSU</td>
<td>5</td>
<td>20</td>
<td>90</td>
<td>0.200</td>
<td>0.086</td>
</tr>
<tr>
<td>FSU</td>
<td>10</td>
<td>20</td>
<td>90</td>
<td>0.150</td>
<td>0.043</td>
</tr>
<tr>
<td>KSC</td>
<td>5</td>
<td>78</td>
<td>293</td>
<td>1.77</td>
<td>1.678</td>
</tr>
<tr>
<td>KSC</td>
<td>9</td>
<td>78</td>
<td>293</td>
<td>0.924</td>
<td>0.932</td>
</tr>
<tr>
<td>KSC</td>
<td>20</td>
<td>77</td>
<td>292</td>
<td>0.410</td>
<td>0.471</td>
</tr>
</tbody>
</table>

**4.5 Conclusions and Future Work**

The required tests have been done for several samples of MLI at variable temperatures using MIKE. While the system can reach a stable temperature to measure the heat flux through MLI samples, the measured heat flux is about double of what is expected. More work needs to be done on the system in order to determine the source of the higher heat load. In addition, more data should be taken and compared with other accepted heat flux measurement techniques. However, the system has proven to reach various temperature ranges allowing for the multiple boundary tests to be taken. Several of the discrepancies with the heat load could come from an unseen thermal short, geometrical factors, or improper shielding from the top and bottom thermal radiation shields. One area that should be researched more is the effects of the metal coating thickness and look for a critical thickness that is temperature dependent.

A model should be developed in parallel with the KSC program based upon the geometry of MIKE in order to determine if there is any discrepancies with the differing geometries. Also the electrical wiring should be reorganized and have more guarded set up to prevent any future electrical shorting. An unnecessary but beneficial addition would be to add a third thermometer.
in the middle of the thermal rod to measure a more precise \( \theta \) and heat load measurement by checking if the temperature gradient is truly linear across the rod but also allow for higher heat load measurements.
APPENDIX A

CHEBVIYCHEV FIT

One method of forming a fit line the temperature equations is to use the Chebychev formulation that normalizes results on a log scale between -1 and 1. When calibrating a temperature sensors the raw data are the resistances at various temperatures, in our case the temperatures compared were of a sensor calibrated by the Lakeshore Cryotronics below 4 K. Depending on the sensitivity of the sensors there can be a wide range of resistance from 4 K to 300 K. Sometimes the resistance versus temperature is too curved so that a proper fit cannot be made without needing several fit equations resulting discontinuities. The Chebychev method helps to make eliminate the need for multiple fits.

The first step of the Chebychev fit is to take the log of the resistance temperatures shown in equation A and find the maximum and minimum values for $R^*$ which will be denoted as $Z_H$ and $Z_L$ respectively

$$R^* = \log_{10}(R) \quad (A.1)$$

The next step is to normalize these equations so we have a number of values between 1 and -1 by equation A.2:

$$Z^* = \frac{(R^* - Z_H) - (Z_L - R^*)}{Z_H - Z_L} \quad (A.2)$$

Finally, plot $Z^*$ versus temperature into a curve fitting tool to fit the calibration curve to equation A.3.

$$T = a^*(\cos(0^*\text{acos}(Z^*))) + b^*(\cos(1^*\text{acos}(Z^*))) + d^*(\cos(2^*\text{acos}(Z^*))) + e^*(\cos(3^*\text{acos}(Z^*))) + \ldots + f^*(\cos(4^*\text{acos}(Z^*))) + g^*(\cos(5^*\text{acos}(Z^*))) + h^*(\cos(6^*\text{acos}(Z^*))) + k^*(\cos(7^*\text{acos}(Z^*)))) \quad (A.3)$$
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