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# Thermal Performance of Biological Substance Systems in Vitro Under Static and Dynamic Conditions at the Cryogenic Test Laboratory, NASA Kennedy Space Center, USA

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#### INTRODUCTION

Cryogenics is fundamentally about energy, and thermal insulation is about energy conservation. The technological developments of this century have led to insulation systems that have approached the ultimate limit of performance. More technologies and markets for rapid expansion into the 21<sup>st</sup> century will require, in many cases, not super insulation but more efficient systems for a wide variety of cryogenic applications. Although bulk storage and delivery of cryogens such as liquid nitrogen, argon, oxygen, hydrogen, and helium are routinely accomplished, cryogenics is still considered a specialty. As ice usage was a specialty in the 19<sup>th</sup> century (not becoming commonplace until the 20<sup>th</sup> century), our goal is to make cryogen usage commonplace in the early 21<sup>st</sup> century. To make liquid nitrogen "flow like water," superior methods of thermal insulation are needed. The development of efficient, robust cryogenic insulation systems has been a targeted area of research for a number of years. Improved methods of characterization, testing, and evaluation of complex biological substance systems for cryosurgery and cryobiology are the focus of this paper.

#### ORIGINS AND HISTORY OF THERMAL INSULATION

From the beginning of history, humankind has used thermal insulation (e.g., animal furs to keep warm or cellars lined with straw to keep perishable foods cool). The measure "R-value" comes from the heat resistance of a 1-inch-thick oak board. Before John Gorrie (Flynn, 1997) invented the first ice-making machine around 1850, ice farms existed in Massachusetts where large slabs were cut, stacked in thick-walled warehouses, and packed in sawdust for later export. Ice shipments were made to hotter countries around the world. At times, the demand for ice was high enough to raise the price to an exorbitant \$2.50 per kilogram in Florida in 1855 (Flynn, 1997). The industrial revolution

brought increased demands for energy efficiency in boilers and metalworking processes, and thus thermal insulation development was started. As the industry proceeded to a chemical and process revolution corresponding to the first liquefaction of key gases from 1877 to 1908, development of insulation for low temperatures began.

The development of insulation for modern cryogenic storage tanks can be traced through three patents issued in the United States. The first, "insulated container for liquefied gases and the like" filed by Dana (1939), gave details for a double-walled tank with the annular space evacuated to a level below 4 torr and filled with finely divided solid material. The second patent, "radiation shield supports in vacuum insulated containers" filed by Cornell (1947), gave details for radiation shielding of containers by use of multiple polished tank walls within the outer tank. The third patent, "thermal insulation" filed by Matsch (1956), outlines the fundamental approach to multiplayer insulation (MLI), which is now the industry standard.

Conventional insulation materials for cryogenic applications can be divided into three categories of apparent thermal conductivity (k-value): around 30 milliwatts per meter – kelvin (mW/m-K) for materials at ambient pressure, about 1.5 mW/m-K for bulk materials at good vacuum (below  $10^{-3}$  torr), and below 0.1 mW/m-K for MLI and similar systems at high vacuum (below  $10^{-4}$  torr) (for boundary temperatures of about 300 K and 77 K). Thermal performance of MLI degrades rapidly for vacuum levels above  $10^{-3}$  torr. In addition to the high-vacuum requirement, other drawbacks of MLI are its high inplane of heat conduction, sensitivity to compressive loads and edge effects, the extreme care needed during installation, and its limitation to more simple structures. Furthermore, the steps of evacuation, heating, and vacuum retention are costly and time-consuming. It is important to recognize that there are three levels of thermal performance: ideal, laboratory, and actual. Actual system performance is typically several times worse than the laboratory performance and often 10 times worse than the ideal.

An insulation system that performs well in soft vacuum fills the performance gap between high vacuum (0.1 mW/m-K) and ambient pressure systems (30 mW/m-K), representing a substantial new market area.

#### **TESTING APPARATUS AND RESULTS**

A unique research program, including a comprehensive study of the thermal performance at cryogenic vacuum insulation systems, was performed at the NASA Kennedy Space Center. The main goal was to develop a new soft vacuum system (from 1 torr to 10 torr) that provides an intermediate level of performance (k-value below 4.8 mW/m-K). Liquid nitrogen boil-off methods were used to test conventional materials, novel materials, and certain combinations. The test articles included combinations of aluminum foil, fiberglass paper, polyester fabric, silica aerogel composite blanket, fumed silica, silica aerogel powder, and syntactic foam. A new layered composite insulation (LCI) system was developed at the Cryogenics Test Laboratory at KSC. This system performs exceptionally well at soft vacuum levels and nearly as good as an MLI at high vacuum levels. Apparent thermal conductivities for the LCI range from 2 mW/m-K at soft vacuum to 0.1 mW/m-K at high vacuum.

Several cryostats were designed, constructed, and calibrated by the Cryogenics Test Laboratory as part of this research program. The cryostat test apparatus is a liquid nitrogen boil-off calorimeter system for direct measurement of the apparent thermal conductivity at a fixed vacuum level between  $5 \times 10^{-5}$  and 760 torr. Fesmire and Augustynowicz further describe the system. Continuously rolled materials are installed around a cylindrical cold mass using a wrapping machine built by the Cryogenics Test Laboratory. High accuracy sensors are placed between layers of the insulation to obtain temperature-thickness profiles. The temperatures of the cold mass [cold boundary temperature (CBT) and the insulation outer surface warm boundary temperature (WBT)] and the vacuum chamber (maintained at 313 K by thermal shroud) are measured.

The advantages of using cryogen boil-off calorimeters for measuring the thermal performance of insulation systems assembled in a cylindrical manner were previously described. The main challenge in the execution of this technique is to obtain stability of the cryogen inside the cold mass and thermal guards with stability of the boundary conditions in the vacuum space. That is, the liquid inside each of the three chambers of the cold mass must be brought to very near saturated conditions relative to the ambient pressure, while the vacuum level and temperatures across the insulation thickness are maintained at constant values. The main problem with testing high performance materials such as MLI is the extreme care that must be exercised in the fabrication and installation of such highly anisotropic test articles. Inconsistency in MLI wrapping techniques is the dominant source of error and poses a basic problem in the comparison of such insulating materials.

Improper treatment of the ends or seams, for example, can render a measurement that is several times worse than predicted. Localized compression effects, sensor installation, and out-gassing are further complications. To eliminate the seam and minimize these other problems, a sleeve method of fabricating and testing insulation was developed.

The steady-state measurement of the insulation performance is made when all temperatures and the boil-off flow are stable. The k-value of the insulation is directly computed from measuring the boil-off rate and temperature difference (WBT-CBT) and knowing the latent heat of vaporization and the geometry of the insulation. All measurements of temperatures, pressures, and flows are recorded on a custom-built 32-channel data acquisition system using LabView software.

Other cryostats for both calibrated and direct measurement of thermal performance have also been built. These cryostats and their corresponding methodologies are used for the full range of material systems including flat plate, bulk fill, composites, and other forms.

The following different low-temperature thermal tests are build, developed, and in operation at the Cryogenics Test Laboratory:

Cryostat-1, 295 tests for 34 insulation systems Cryostat-2, 172 tests for 24 insulation systems Cryostat-3, 14 tests for 2 insulation systems Cryostat-4, 79 tests for 12 insulation systems Totals Cryostats: 560 tests, 72 insulation systems, ~13,500 hours Dewar Test Apparatus Pipeline Test Apparatus

All these technologies use liquid nitrogen boil-off methods for precise measurement of thermal performance. Thermal conductivities of materials and material systems in

cylindrical, flat, bulk, and layered forms are determined from these steady-state calorimetric techniques. Environments include high vacuum to no vacuum with boundary temperatures from 77 K to 373 K.

Some of the current research projects at the Cryogenics Test Laboratory include the following:

- a. Comparative study cryogenic-vacuum insulation systems
- b. Development of LCI systems
- c. Spray-on aerogel composite insulation for cryogenic tanks
- d. Long flexible cryostats for high-temperature superconducting power cables
- e. Mars surface storage of cryogens
- f. Low-cost, high-efficiency pipelines for long-distance transfer of cryogenic propellants
- g. Evacuated microsphere insulation panels
- h. Application of aerogel beads for thermal insulation
- i. Testing of structural insulating panels for common bulkhead tanks
- j. Quick cooling and filling through a single port

#### CONCLUSION

The Cryogenics Test Laboratory has experience with all practical aspects of thermometry, measurements procedure, data acquisition systems, data recording and storage, and control system. We are also familiar with control and monitoring system development (concept, hardware, software, packaging, building block use, or commercial off-the-shelf equipment use).

We can improve the method and methodology for cryosurgery and cryobiology application looking for heat transfer studies on experimental bases in two ways:

- a. characterization of substances
- b. testing and evaluation of complex substance systems

We can develop and build Cryosurgery Cryostat Systems (CCS) for the following:

- a. Static study (steady-state methods) of substances (thermal conductivity of specimens, heat flux)
- b. Dynamic study (transient methods) of substance systems (cooling, freezing and thawing rates, heat flux ratio mapping for elemental surface area, temperature profile measurements)

A new Cryosurgery Cryostat System can be established for cryobiology and cryosurgery at the Cryogenics Test Laboratory of NASA Kennedy Space Center.

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#### SUMMARY

A unique research program, including a comprehensive study of the thermal performance at cryogenic vacuum insulation systems, was performed at the NASA Kennedy Space Center. The main goal was to develop a new soft vacuum system (from 1 torr to 10 torr) that provides an intermediate level of performance (k-value below 4.8 mW/m-K). Liquid nitrogen boil-off methods were used to test conventional materials, novel materials, and certain combinations. The test articles included combinations of aluminum foil, fiberglass paper, polyester fabric, silica aerogel composite blanket, fumed silica, silica aerogel powder, and syntactic foam. A new LCI system was developed at the Cryogenics Test Laboratory. This system performs exceptionally well at soft vacuum levels and nearly as good as an MLI at high vacuum levels. Apparent thermal conductivities for the LCI range from 2 mW/m-K at soft vacuum to 0.1 mW/m-K at high vacuum.

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The development of efficient, robust cryogenic insulation systems has been a targeted area of research for a number of years. Improved methods of characterization, testing, and evaluation of complex biological substance systems for cryosurgery and cryobiology are the focus of this paper.



## Table 1. Thermal Insulating Performance of Various Materials

Table 2. Experimental Apparent Thermal Conductivity Values for Different Materials

Material and Density	High Vacuum <10 <sup>-4</sup> torr	Soft Vacuum 1 torr	No Vacuum 760 torr
Vacuum, polished surfaces	0.5 to 5		
Nitrogen gas at 200 K			18.7
Fiberglass, 16 kg/m <sup>3</sup>	2	14	22
PU foam, 32 kg/m <sup>3</sup>			21
Cellular glass foam, 128 kg/m <sup>3</sup>			33
Perlite powder, 128 kg/m <sup>3</sup>	1	16	32
Aerogel beads, 80 kg/m <sup>3</sup>	1.1	5.4	11
Aerogel composite blanket, 125 kg/m <sup>3</sup>	0.6	3.4	12
MLI, foil and paper, 60 layers, 79 kg/m <sup>3</sup>	0.09	10	24
LCI, 30 layers, 78 kg/m <sup>3</sup> (new system)	0.09	1.6	14



Figure 1. Early Insulation System Designs for Cryogenic Tanks – Double-Walled Tank With Evacuated Powder



Figure 2. Early Insulation System Designs for Cryogenic Tanks – Vacuum-Insulated Container With Multiple Radiation Shields



Figure 3. Simplified Schematic of the Cryostat Test Apparatus Indicating Key System Measurements



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Figure 4. Diagram of Temperature Sensor Locations for a Typical Insulation Test Configuration