

# Thermal conductivity of aerogel blanket insulation under cryogenic-vacuum conditions in different gas environments

J E Fesmire<sup>1</sup>, J B Ancipink<sup>1</sup>, A M Swanger<sup>1</sup>, S White<sup>2</sup> and D Yarbrough<sup>3</sup>

<sup>1</sup> NASA Kennedy Space Center, Cryogenics Test Laboratory, UB-R1, KSC, FL 32899

<sup>2</sup> Aspen Aerogels, Inc., 30 Forbes Rd, Bldg. B, Northborough, MA 01532 USA

<sup>3</sup> R&D Services, Inc., PO Box 2400, Cookeville, TN 38501 USA

E-mail: james.e.fesmire@nasa.gov

**Abstract.** Thermal conductivity of low-density materials in thermal insulation systems varies dramatically with the environment: cold vacuum pressure, residual gas composition, and boundary temperatures. Using a reference material of aerogel composite blanket (reinforcement fibers surrounded by silica aerogel), an experimental basis for the physical heat transmission model of aerogel composites and other low-density, porous materials is suggested. Cryogenic-vacuum testing between the boundary temperatures of 78 K and 293 K is performed using a one meter cylindrical, absolute heat flow calorimeter with an aerogel blanket specimen exposed to different gas environments of nitrogen, helium, argon, or CO<sub>2</sub>. Cold vacuum pressures include the full range from  $1 \times 10^{-5}$  torr to 760 torr. The soft vacuum region, from about 0.1 torr to 10 torr, is complex and difficult to model because all modes of heat transfer – solid conduction, radiation, gas conduction, and convection – are significant contributors to the total heat flow. Therefore, the soft vacuum tests are emphasized for both heat transfer analysis and practical thermal data. Results for the aerogel composite blanket are analyzed and compared to data for its component materials. With the new thermal conductivity data, future applications of aerogel-based insulation systems are also surveyed. These include Mars exploration and surface systems in the 5 torr CO<sub>2</sub> environment, field joints for vacuum-jacketed cryogenic piping systems, common bulkhead panels for cryogenic tanks on space launch vehicles, and liquid hydrogen cryofuel systems with helium purged conduits or enclosures.

## 1. Introduction

Following the initial development of flexible aerogel blanket material in the early 1990s, this novel composite has seen adoption in many industries for thermal insulation applications [1]. Commercially, for the last 15 years, Aspen Aerogels has produced a number of different types and formulations of silica-based aerogels for industry use. These include Spaceloft, Cryogel, and Pyrogel, each with variants for different technical requirements, as indicated in Table 1. The basic cryogenic-vacuum thermal performance data for Cryogel has been previously reported [2]. An advantage of the sol-gel processing routes of manufacture is that an infinite number of material and chemical combinations can be employed and tailored to specific requirements. New materials and short-scale runs of specialized aerogel blankets continue to be developed for both space and terrestrial applications, some seeing deployment to provide solutions where none exist with conventional insulations.

The use of aerogel-based thermal insulation in different gas environments has a number of practical applications in cryogenic engineering and other fields. The availability of thermal data for certain conditions is often scarce and underscores the need to understand heat transmission through complex composite materials with its nano-sized internal pore structure surrounding a network of microfibers.



Thermal conductivity of low-density materials in thermal insulation systems varies dramatically with the environment: cold vacuum pressure, residual gas composition, and boundary temperatures. The soft vacuum region, from about 0.1 torr to 10 torr, is particularly complex and difficult to model because all modes of heat transfer – solid conduction, radiation, gas conduction, and convection – can be significant contributors to the total heat flow [3-4]. Although many research efforts have developed analytical approaches and models to heat transfer in aerogels, few have addressed composites of aerogels within a fiber matrix [5]. To provide a sense of scale for the interplay of heat transmission through an aerogel-and-fiber network, a single fiber with a diameter of 15  $\mu\text{m}$  diameter represents an equivalent length of roughly 800 pores of aerogel. The models for the radiation and gas conduction components are particularly complex for aerogels alone, even without any consideration for a fiber matrix within.

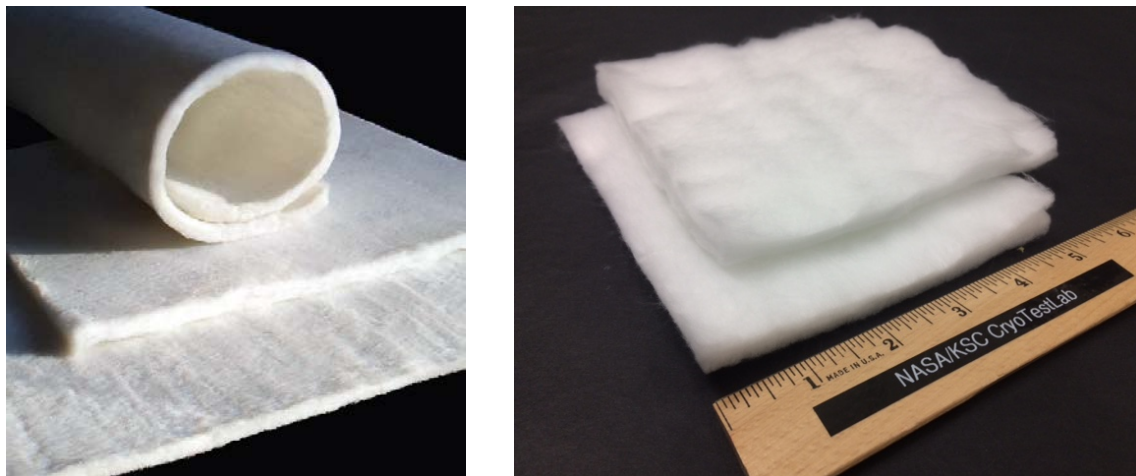
## 2. Experimental approach and set-up

Using a reference material of aerogel composite blanket (reinforcement fibers surrounded by silica aerogel), an experimental basis for the physical heat transmission model of aerogel composites and other low-density, porous materials is the goal. This experimental study focused on one material, Cryogel®, from the family of flexible aerogel composite blanket materials manufactured by Aspen Aerogels. In addition, the fiber matrix reinforcement material used in Cryogel was also tested. The basic specifications are listed in table 1. The test specimens, all taken from the same lot of material, consisted of two layers of 10-mm material (see figure 1) for a total thickness of 20 mm. With precision butt-joint installation on each layer, an overlapped longitudinal seam, and compression straps for uniform thickness from top to bottom, a 1-m tall cylindrical test specimen was built for the Cryostat-100 test instrument. Other test specimens included two layer stacks of 204-mm diameter rounds (for the Cryostat-500 instrument) or 305-mm squares (for the heat flux meter instrument).

**Table 1.** Commercial aerogel blanket insulation products by Aspen Aerogels.

Type	Thickness	*Thermal Conductivity	Nominal Density	Maximum Use Temperature
		ASTM C177	ASTM C167	
	mm	mW/m-K	g/cc	°C
Cryogel® x201	5 or 10	17.0	0.16	200
Spaceloft®	5 or 10	16.5	0.16	200
Spaceloft® Subsea	5 or 10	14.5	0.16	200

\*Thermal conductivity at a mean temperature of 273 K, a 13.8 kPa compressive load, and standard atmospheric pressure.



**Figure 1.** Aerogel composite blanket material (Cryogel) (left) and fiber matrix (right) both in 10-mm thicknesses.

Cryogenic-vacuum testing between the boundary temperatures of 78 K and 293 K was performed using a one meter cylindrical, absolute heat flow calorimeter with an aerogel blanket specimen exposed to different gas environments of nitrogen, helium, argon, or carbon dioxide (CO<sub>2</sub>). Cold vacuum pressures include the full range from  $1 \times 10^{-5}$  torr to 760 torr. The primary instrument for testing of cylindrical specimens is the Cryostat-100 as shown in figure 2. This apparatus is guarded on top and bottom for absolute thermal performance measurement [6]. A cold mass assembly, including the top and bottom guard chambers and a middle test chamber, is suspended from a domed lid atop the vacuum canister, as shown in figure 2. Vacuum instrumentation typically includes three capacitance manometers, an ion gage, and two full-range transducers for backup. The vacuum pumping system includes a directly connected turbo-pump and a separately plumbed mechanical pump. A gaseous supply system provides purging and residual gas pressure control to vacuum levels as low as  $5 \times 10^{-5}$  torr.

The principle of heat rate measurement for Cryostat-100 is based on LN<sub>2</sub> boiloff calorimetry, following the guidelines of ASTM C1774, Annex A1. In addition, the fiber matrix material was tested in parallel with the Cryogel material using the guarded flat plate boiloff calorimeter, Cryostat-500, per C1774, Annex A3. The steady-state heat flow rate ( $Q$ ) is the basis for calculating the thermal properties, including effective thermal conductivity ( $k_e$ ) and heat flux ( $q$ ).

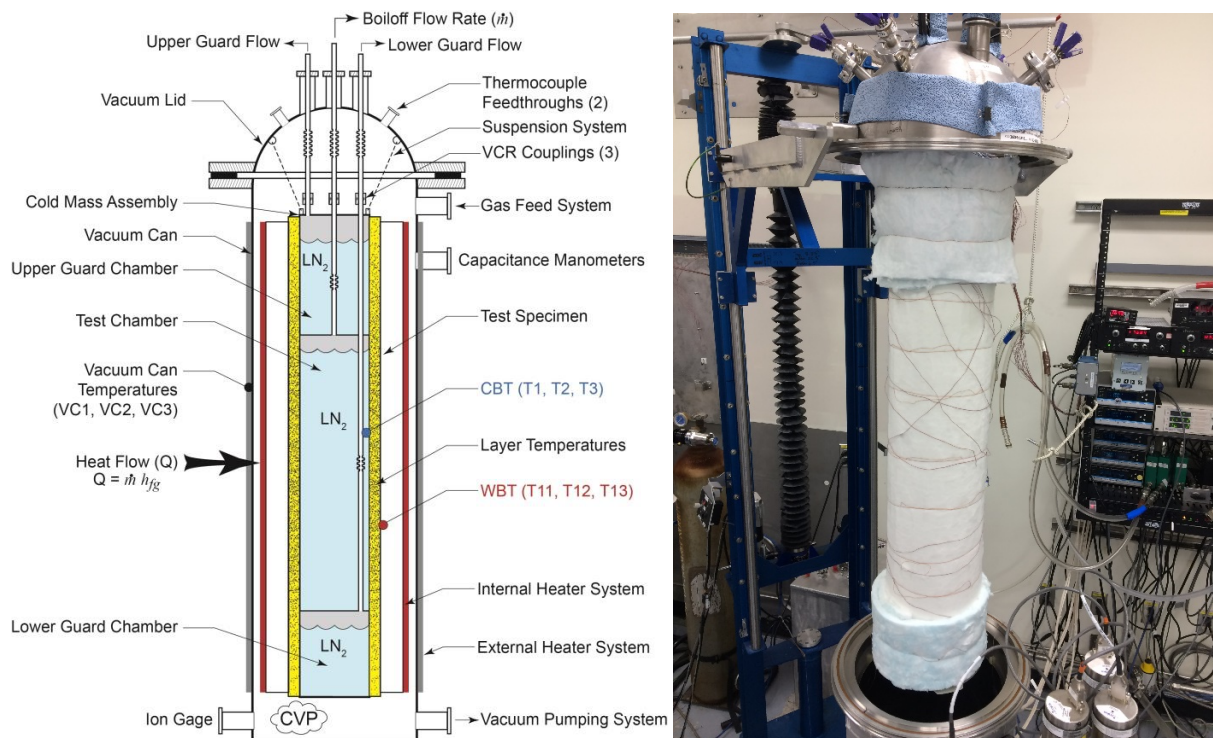
Materials from the same lots were also tested for apparent thermal conductivity ( $\lambda$ ) at ambient conditions in air using a heat flux meter apparatus in accordance with ASTM C518. The 305-mm square test specimens were also two layers and 20 mm total thickness.

The test specimen was prepared by evacuation and moderate heating (323 K or less) to achieve a vacuum level of less than  $1 \times 10^{-4}$  torr. The cryostat instrument was then cooled and filled with liquid nitrogen. After a baseline test at the high vacuum level, the gas supply was added for the desired set-point of cold vacuum pressure (CVP) from 1 millitorr up to ambient pressure as desired for a given test. The cryostat was typically returned to moderate vacuum for cold-keeping between tests. The residual gas pressure control naturally depended on the liquefaction (or sublimation point) of the gas being supplied to the cryostat system operating at a mean temperature of 186 K (between 293 K and 78 K boundary conditions). Gas properties are given in table 2 for reference in regard to both test methodology and insulation system performance.

**Table 2.** Thermal conductivity of different gases at mean temperatures from 150 K to 300 K; densities at standard temperature & pressure conditions (273 K and 1 atm).

Gas	Normal Boiling Point	Density @ STP	Thermal Conductivity (mW/m-K)			
			100 K	200 K	300 K	400 K
	<i>K</i>	<i>g/L = kg/m<sup>3</sup></i>				
Xenon (Xe)	165.0	5.76	2.0	3.6	5.5	7.3
Krypton (Kr)	119.9	3.749	3.3	6.4	9.5	12.3
*Carbon dioxide (CO <sub>2</sub> )	194.8 (sublimates)	1.96	-	9.6	16.8	25.1
Argon (Ar)	87.3	1.78	6.2	12.4	17.9	26.8
Oxygen (O <sub>2</sub> )	90.2	1.419	9.3	18.4	26.3	33.7
Air	78.8	1.275	9.4	18.4	26.2	33.3
Nitrogen (N <sub>2</sub> )	77.4	1.25	9.8	18.7	26.0	32.3
Methane (CH <sub>4</sub> )	111.7	0.716	-	22.5	34.1	49.1
Helium (He)	4.2	0.179	75.5	119.3	156.7	190.6
Hydrogen (H <sub>2</sub> )	20.3	0.090	68.6	131.7	186.9	230.4

\*Note: thermal conductivity of solid CO<sub>2</sub> is as follows: 419 mW/m-K at 210 K, 500 @ 186, 622 @ 150, and 1,168 @ 80 K [7].



**Figure 2.** Cryostat-100: basic schematic (left) and installation of aerogel blanket test specimen (right).

### 3. Thermal testing results

A graphical summary of the cylindrical Cryostat-100 test results for Cryogel in different gas environments is presented in figure 3. The variation of  $k_e$  with cold vacuum pressure (CVP) is reported for the boundary temperatures of approximately 293 K and 78 K and the residual gas type is as indicated. With nitrogen gas as the baseline, the  $k_e$  of Cryogel ranges from approximately 1.5 mW/m-K at high vacuum (approaching the ideal performance high-density perlite powder in bulk-fill applications) to 12.3 mW/m-K at ambient pressure (about half the thermal conductivity of polyurethane foams). The gases argon and CO<sub>2</sub> are seen to be below the nitrogen gas baseline up to about 100 millitorr CVP. The helium line trends above the nitrogen line starting at about 50 millitorr which corresponds to the typical transition from free molecular to continuum gas conduction.

To be able to test another material in parallel with the long-term testing of another, the flat plate boiloff calorimeter, Cryostat-500, was used. A plot of the Cryostat-500 test results for fiber matrix material in comparison with data for Cryogel is given in figure 4. An overall summary of cryostat test results for both the Cryogel and fiber matrix material are summarized in table 3.

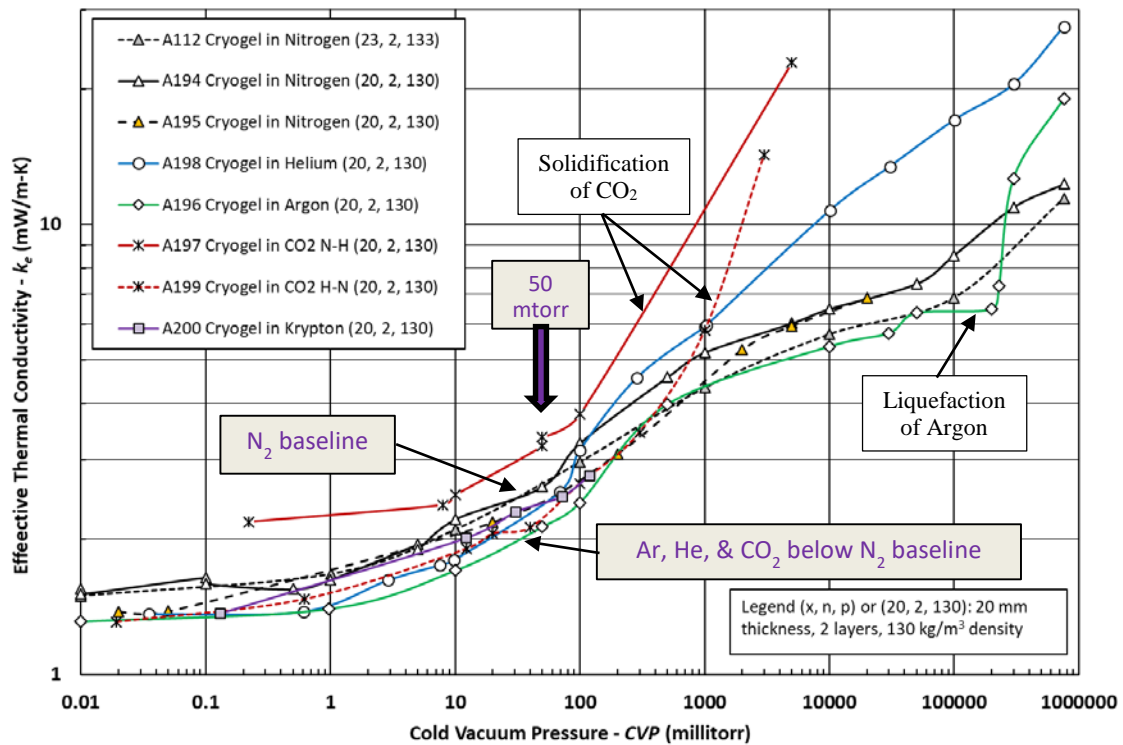
Two curves for CO<sub>2</sub> are presented. One was testing in the No-Vacuum to High-Vacuum (N-H) direction, while the other was for testing in the High-Vacuum to No-Vacuum (H-N) direction. The CO<sub>2</sub> was found to predominately load into the material on the cold side at CVP above 1 torr, particularly when approached from the No-Vacuum direction. A different result was obtained when starting at High-Vacuum and proceeding toward soft vacuum, but the 5 torr point was about the limit of the heat flow measurement capability of the Cryostat-100.

For comparison, ambient testing for apparent thermal conductivity ( $\lambda$ ) in air using a Lasercomp Model 304 Heat Flow Meter gave the following results at a mean temperature of 297 K (23.9°C):

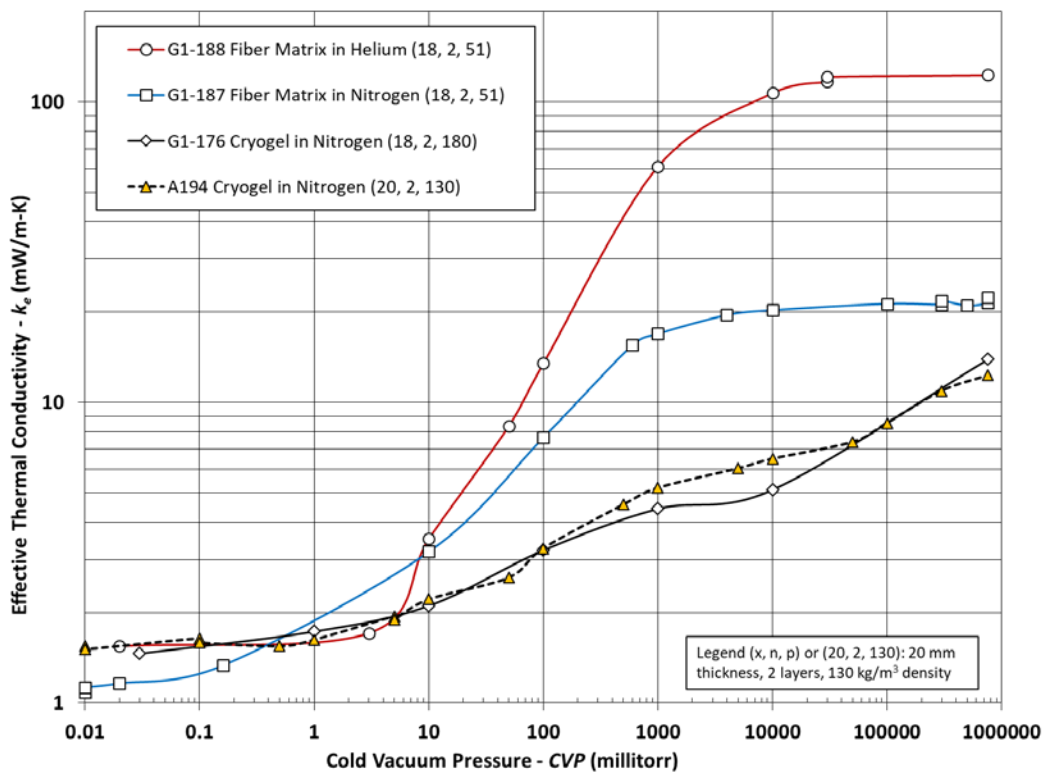
- Cryogel (density of 164 kg/m<sup>3</sup>): apparent thermal conductivity ( $\lambda$ ) of 19.1 mW/m-K (thermal resistance of 0.526 m<sup>2</sup>-K/W).
- Fiber Matrix (density of 51 kg/m<sup>3</sup>): apparent thermal conductivity ( $\lambda$ ) of 30.6 mW/m-K (thermal resistance of 0.327 m<sup>2</sup>-K/W).

While the fiber matrix itself is a quite effective thermal insulation material, these results show that it is about 60% higher thermal conductivity compared to the aerogel blanket.





**Figure 3.** Summary of Cryostat-100 test results for Cryogel in different gas environments: variation of  $k_e$  with CVP. Boundary temperatures: 293 K/78 K; residual gas: as indicated.



**Figure 4.** Summary of Cryostat-500 test results for fiber matrix material in comparison with Cryogel: variation of  $k_e$  with CVP. Boundary temperatures: 293 K/78 K.

**Table 3.** Summary of cryostat thermal performance test results for Cryogel aerogel blanket composite and fiber matrix material.

Cryostat Test Specimen*	$k_e$ (mW/m-K) for select <i>CVP</i> (torr)				
	$<10^{-4}$	0.01	0.1	1	760
A112 Cryogel in Nitrogen 2007	1.49	2.10	2.96	4.34	11.4
A194 Cryogel in Nitrogen	1.51	2.21	3.27	5.19	12.3
A196 Cryogel in Argon	1.06	1.70	2.41	4.10	18.8 <sup>†</sup>
A197 Cryogel in CO <sub>2</sub> (N > H)	---	2.50	3.79	15.0 <sup>†</sup>	---
A199 Cryogel in CO <sub>2</sub> (H > N)	1.31	1.90	2.65	5.81	---
A198 Cryogel in Helium	1.23	1.80	3.15	5.96	27.5
A200 Cryogel in Krypton	1.37	2.01	2.77	---	---
G1-187 Fiber Matrix in Nitrogen	1.13	3.19	7.65	16.9	22.4
G1-188 Fiber Matrix in Helium	1.55	3.52	13.5	61.1	123

\*Boundary temperatures 293 K / 78 K; residual gas as indicated; Cryostat-100 or Cryostat-500 boiloff calorimeter apparatus.

<sup>†</sup>Liquefaction of argon or solidification of carbon dioxide causes dramatic increase in heat transmission through material.

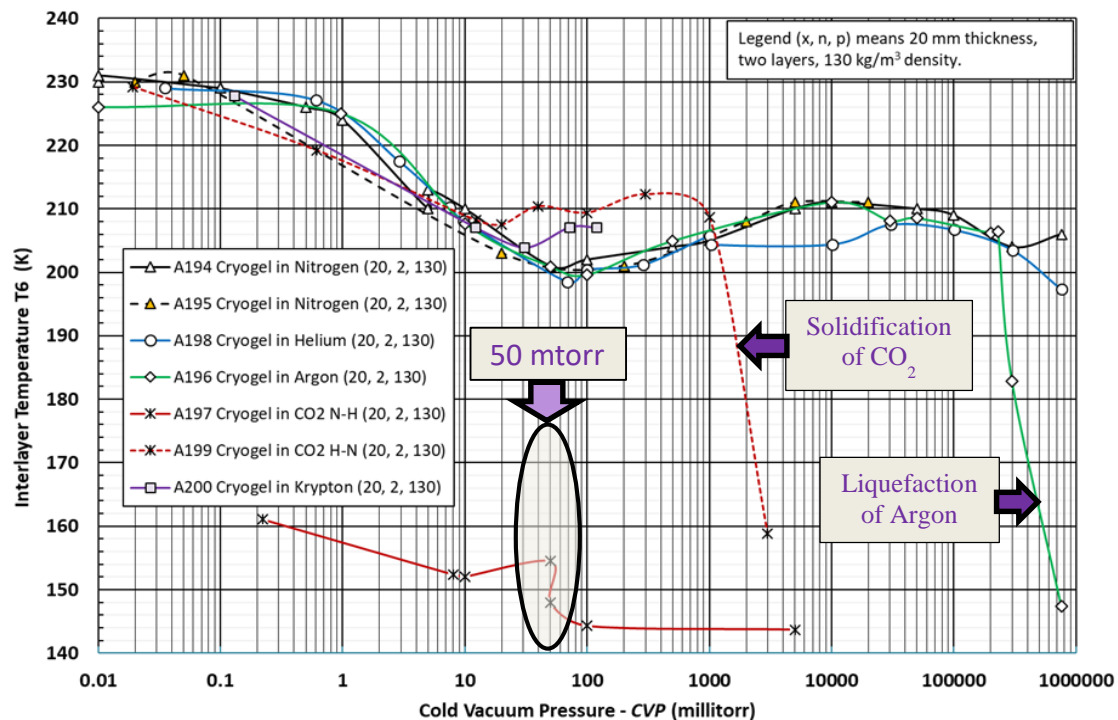
#### 4. Discussion and analysis

The soft vacuum region, from about 0.1 torr to 10 torr, is complex and difficult to model because all modes of heat transfer – solid conduction, radiation, gas conduction, and convection – are significant contributors to the total heat flow. The situation is further complicated by the physical nature of a complex composite of nano-porous aerogel within a micro-fibrous matrix. Therefore, the soft vacuum tests are emphasized for both heat transfer analysis and practical thermal data. Defining and careful execution of the gas-filling approach was critical to the thermal conductivity results obtained, as previously discussed. Similarly, in other cryogenic condensation-evacuated thermal insulation test measurements, Geisler et. al show two different gas-filling processes, warm filling and cold filling, but based on mass metering of a specific quantity of gas [8]. The current study included both warm and cold processes for gas filling, but only the cold technique is covered in this report.

Comparison between Cryogel and fiber matrix shows the dramatic effect of the heat transmission from continuum range to convective (ambient pressure). Comparing  $k_e$  at 760 torr CVP, the fiber matrix is 82% higher than the Cryogel. Conversely, the fiber matrix is 29% lower at high vacuum where gas conduction and convection are not factors. Comparison between 2007 Cryogel and 2015 Cryogel show a slight increase in  $k_e$  of about 8% between these production periods.

Special observations for the Cryogel test results are briefly summarized as follows. Helium observations at 760 torr show that  $k_e$  increases by only 124% (2.2x) compared to Cryogel in nitrogen while the corresponding increase for the gases only would be 538% (6.4x). Argon observations show that liquefaction dramatically increases above 200 torr. CO<sub>2</sub> results above about 1 torr show that aerogel blanket material can make a powerful adsorber of solid CO<sub>2</sub> within the nanoporous structures (as shown by the curve A197 proceeding from the No-Vacuum case). The interlayer temperature T6 as a function of CVP is given in figure 5. The unusual curve for A197 attests to the chaotic transitional heat transfer occurring at around 50 millitorr, the transition from free molecular to continuum gas conduction. Thermal conductivity of solid CO<sub>2</sub> at a mean temperature of 186 K is 500 mW/m-K [7]. The mass

fraction of CO<sub>2</sub> within the aerogel composite can then be estimated knowing the sublimation temperature for a given cold vacuum pressure.



**Figure 5.** Cryostat-100 test results for Cryogel in different gas environments: variation of Interlayer Temperature T6 with CVP. Boundary temperatures: 293 K/78 K.

## 5. Applications

Novel applications of the aerogel-based thermal insulation systems include field joints for vacuum-jacketed cryogenic piping systems, common bulkhead panels for cryogenic tanks on space launch vehicles, and liquid hydrogen cryofuel systems with helium purged conduits or enclosures [9-11]. Self-pumping (cryo-pumping) vacuum-jacketed (VJ) systems have been a valuable technique in many situations for decades. In the aerospace field, CO<sub>2</sub>-filled VJ systems have been used for liquid oxygen piping and Argon-filled VJ systems for liquid hydrogen piping. Use of aerogel blanket materials in such designs could provide even higher levels of thermal performance and well as structural attributes.

Finding a plausible approach to the long-term preservation of cryogenic liquid products on the Martian surface is an important goal. The current findings show that an aerogel composite blanket, when employed as a cryogenic thermal insulator, will adsorb and freeze-out carbon dioxide in a partial pressure environment such as the 5 torr ambient environment of Mars. The resulting heat leak is even worse than the case of the 760 torr air environment (Earth surface). However, potential applications of aerogel blanket on Mars include CO<sub>2</sub> capture and cryogenic storage in conjunction with lightweight, jacketed-type constructions.

In an example case of a 3800-liter liquid oxygen (LO<sub>2</sub>) tank with a 100-mm thickness of aerogel blanket for insulation, the liquid is boiled away in only 2 days on Earth [12]. The same tank of LO<sub>2</sub>, insulated with the industry standard 40 layers of foil/paper multilayer insulation (MLI), will last for about 1,400 days in high vacuum environment of the Lunar surface. Applying the new data shows that the example tank full of LO<sub>2</sub>, when located in the open Mars environment, would be boiled away in a mere 12 hours. The situation does not improve appreciably, for any typical thermal insulation material in any reasonable construction thickness, in the soft vacuum environment. The soft vacuum pressure is roughly six orders of magnitude greater than the high vacuum level required for MLI systems.

The Mars and Earth environments are comparable, in the heat transmission sense, compared to the vastly different high vacuum environment of the Moon (or space in general). This recognition runs

counter to past assumptions in determining the central electrical power requirement for future missions – the cryogenic refrigeration plant – that rely on MLJ systems with  $q$  of 1 W/m<sup>2</sup> ( $k_e$  of around 1 mW/m-K) that are far from the reality of soft vacuum [13]. Potential applications of soft vacuum super insulation on Mars include habitats, space suits, scientific instruments, life support systems, environmental control systems, and cryogen-based systems for oxygen, methane, and hydrogen. The obvious challenge here, with the Mars environment roughly the same as the Earth environment in regard to heat leak into cryogenic systems, is that massive steel vacuum-jacketed equipment is not an option.

## 6. Conclusion

Results for the aerogel composite blanket (Cryogel) tested under cryogenic-vacuum conditions, and in different gas environments, are analyzed and compared to data for its component materials. The variations in effective thermal conductivity ( $k_e$ ) with cold vacuum pressure (CVP) are reported for the gases nitrogen, argon, helium, carbon dioxide, and krypton. With nitrogen gas as the baseline, the  $k_e$  of Cryogel ranges from approximately 1.5 mW/m-K at high vacuum (approaching the ideal performance high-density perlite powder in bulk-fill applications) to 12.3 mW/m-K at ambient pressure (about half the thermal conductivity of polyurethane foams). The Earth and Mars environments are shown to be comparable, in the heat transmission sense, in comparison to the high vacuum environment of the Moon (or space in general). Potential applications of aerogel-based insulation systems include Mars exploration and surface systems in the soft vacuum CO<sub>2</sub> environment, welded field joint enclosures for vacuum-jacketed cryogenic piping systems, common bulkhead panels for cryogenic tanks on space launch vehicles, and liquid hydrogen cryofuel systems with helium purged conduits or enclosures. These thermal performance data provide some experimental underpinnings for future work in the physical modeling of complex composite materials such as the Cryogel aerogel blanket and other aerogel-based thermal insulation systems.

## 7. References

- [1] Fesmire J, Rouanet S and Ryu J 1998 Aerogel-based cryogenic superinsulation *Advances in Cryogenic Engineering* **44** (Plenum Press, New York) pp 219-226
- [2] Coffman B, Fesmire J, Augustynowicz S, Gould G and White S 2010 Aerogel blanket insulation materials for cryogenic applications *Adv. in Cryo. Engr.* **1218** pp 913-920
- [3] Fricke J, H'ummer E, Morper H and Scheuerpflug P 1989 Thermal properties of silica aerogel *Journal de Physique Colloques* **50** (C4) pp 87-97
- [4] Wei G, Liu Y and Zhang X 2012 Gaseous conductivity study on silica aerogel and its composite insulation materials *Journal of Heat Transfer* **134** Issue 4
- [5] Zhao J, Duan Y, Wang X and Wang B 2012 Analytical model for combined radiative and conductive heat transfer in fiber-loaded silica aerogels *J. Non-Crystalline Solids* **358**-10
- [6] Fesmire J, Johnson W, Meneghelli B and Coffman B 2015 Cylindrical boiloff calorimeters for testing of thermal insulations *IOP Conf. Series: Materials Science & Engineering* **101**
- [7] Manzhelii V et al. 1999 *Structure and Thermodynamic Properties of Cryocrystals Handbook* (Begell House Inc. Publishers, New York) pp 173-176
- [8] Geisler M, Wachtel J, Hoffman J and Ebert H 2010 Condensation-evacuated cryogenic thermal insulation systems: deposited filling gases *AIP Conference Proceedings* **1218** p 755
- [9] Johnson W, Fesmire J and Meneghelli B 2012 Cryopumping field joint can testing *AIP Conference Proceedings* **1434** pp 15-27
- [10] Perkins P, Dengler R, Niendorf L and Nies G 1968 Self-evacuated multilayer insulation of lightweight prefabricated panels for cryogenic storage tanks NASA TN D-4375
- [11] Bheekham N, Talib A and Hassan M 2013 Aerogels in aerospace: an overview *Advances in Materials Science and Engineering* Article ID 406065
- [12] Fesmire J 2016 Energy considerations and calculations for cryogenic propellant storage on the Moon and Mars *Living off the Land in Space Seminar* (Flor. Institute of Tech., Melbourne FL)
- [13] Ash R, Dowler W and Varsi G 1978 Feasibility of rocket propellant production on Mars *Acta Astronautica* **5** Issue 9 pp 705-724