ADVANCES IN MICROSPHERE INSULATION SYSTEMS

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ABSTRACT

Microsphere insulation, typically consisting of hollow glass bubbles, combines in a single material the desirable properties that other insulations only have individually. The material has high crush strength, low density, is noncombustible, and performs well in soft vacuum. Microspheres provide robust, low-maintenance insulation systems for cryogenic transfer lines and dewars. They also do not suffer from compaction problems typical of perlite that result in the necessity to reinsulate dewars because of degraded thermal performance and potential damage to its support system. Since microspheres are load bearing, autonomous insulation panels enveloped with lightweight vacuum-barrier materials can be created. Comprehensive testing performed at the Cryogenics Test Laboratory located at the NASA Kennedy Space Center demonstrated competitive thermal performance with other bulk materials. Test conditions were representative of actual-use conditions and included cold vacuum pressure ranging from high vacuum to no vacuum and compression loads from 0 to 20 psi. While microspheres have been recognized as a legitimate insulation material for decades, actual implementation has not been pursued. Innovative microsphere insulation system configurations and applications are evaluated.

INTRODUCTION

Cryogenic insulation systems that employ glass microspheres in evacuated powder form offer significant advantages over traditional materials for many practical applications. The best insulation material for a cryogenic system is the one that offers the optimal combination of thermal performance, low cost, light weight, durability, and minimal or no maintenance. The traditional cryogenic insulations – multilayer insulation (MLI), perlite, and foam (polyurethane or glass) – do not provide a balance of these key properties. Microsphere insulation systems enable energy efficient, cost effective storage and transfer of cryogens. A micrograph of microspheres is displayed in FIGURE 1.

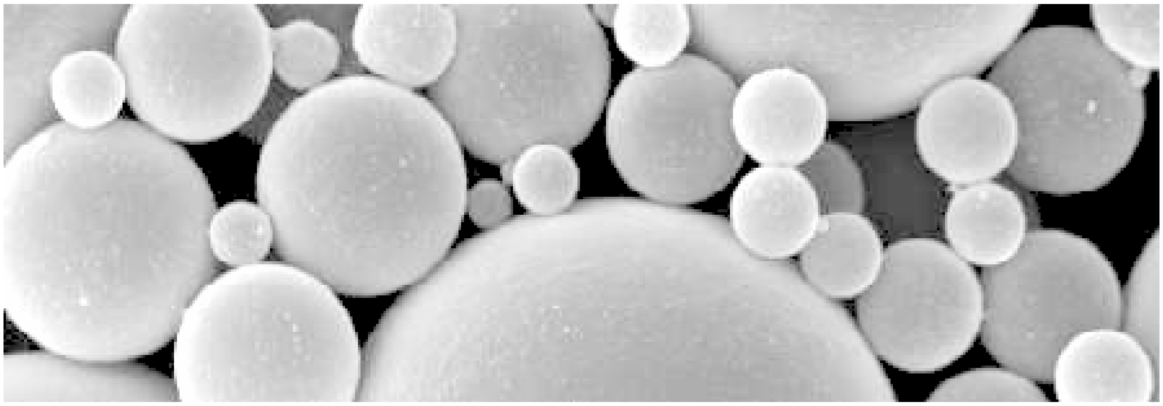

FIGURE 1. Microspheres magnified 500x

Most bulk cryogenic storage tanks (dewars) are insulated with either MLI or perlite. The process of wrapping inner vessels with MLI is costly and time-consuming. Actual thermal performance is also greatly dependent on the quality of fabrication and the vacuum level maintained during operation. Often the vacuum level degrades to the point where the potential performance of MLI is lost and could even be worse than a cryogenic tank insulated with perlite. Though inexpensive to utilize as a bulk evacuated insulation, perlite compacts and settles under the inner vessel during thermal cycling. The compaction increases heat leak to the inner vessel and can damage internal piping and supports. Water adsorption is also a major problem for perlite-insulated cryogenic tanks. Often the perlite insulation adsorbs water to the point that it is impossible to achieve an acceptable vacuum level. The result of both the compaction and water adsorption problems are increased evaporation rate with age, and eventually, costly replacement of bulk insulation and possible repair of internal components. Microsphere insulation's inherent properties of high crush strength, ability to flow, and reduced sensitivity to vacuum level combine to remedy the problems encountered with MLI, perlite, and foam insulation systems.

Current cryogenic transfer line insulation technologies are either expensive (MLI) or degrade quickly (foam). Both are maintenance-intensive to maintain the original performance level. Vacuum-jacketed transfer lines insulated with MLI also suffer from the same fabrication and vacuum level issues as tanks, particularly for flexible vacuum-jacketed pipe. Other cryogen lines are foam-insulated, which degrades starting with the first thermal cycle and is in poor condition within three to five years. Microsphere insulation can be applied to cryogenic piping either within a traditional vacuum jacket or externally applied to bare piping (that may have been previously insulated with foam) in the form of autonomous evacuated insulated panels.

Technology Applications, Inc. (TAI) has been working on two NASA SBIR (Small Business Innovation Research) programs to develop and demonstrate microsphere cryogenic insulation systems to meet NASA and commercial requirements. TAI has a patent pending for microsphere insulation systems.

MICROSPHERE PROPERTIES

Microspheres were studied and tested for their properties and behavior under vacuum conditions in order to confirm viability as a replacement for MLI, evacuated perlite, and non-evacuated foam insulations. The 3M Type K1 Scotchlite™ Glass Bubbles were selected for use in this project because of low cost, light weight, and ready availability. These microspheres are also the same type studied by previous investigators [1].

Thermal Performance

The apparent thermal conductivity of Type K1 microspheres was measured by the Cryogenics Test Laboratory of NASA Kennedy Space Center and is described in the paper by Fesmire et al [2]. The test measurements were made with 78 K cold boundary temperature and 293 K warm boundary temperature and included the full cold-vacuum pressure range. The key vacuum levels that are appropriate to compare the thermal performance of microspheres to MLI, perlite, and non-evacuated foam insulations are $1x10^{-3}$ torr, $1x10^{-1}$ torr, and 760 torr, respectively. TABLE 1 displays the apparent thermal conductivity at these vacuum levels and comparative thermal performance to these traditional cryogenic insulation materials. While the thermal performance of microspheres falls considerably short of MLI, it is important to note that the vacuum space of a commercial vacuum-jacketed cryogenic tank or transfer line is usually only partially filled with MLI in order to enable nesting of the insulated inner vessel within the vacuum shell. The complete filling of the vacuum space with microspheres can improve the comparative thermal performance of microspheres to MLI to within a factor of two. The complete filling of the vacuum space with microspheres can improve the comparative thermal performance of microspheres to MLI to within a factor of two. When microspheres are used in place of perlite or foam, insulation thickness can be significantly reduced while retaining equivalent thermal performance. Additional thermal conductivity tests performed on autonomous evacuated insulation panels are described in a subsequent section below.

Physical Properties and Composition

The 3M Type K1 microspheres are manufactured from soda-lime-borosilicate glass and is the most economical 3M microsphere product at about $0.40 per liter. TABLE 2 contains selected properties of Type K1 microspheres. Trapped within the microspheres are residual gases consisting of a 2:1 ratio of SO_2 and O_2 at an absolute pressure of about 1/3 atmosphere. Amorphous silica is added at 2% to 3% by weight to the microspheres to prevent caking if exposed to water. Caking of the bulk microspheres is caused by bridging of residual salts from the manufacturing process that have condensed on the surface of the microspheres. Amorphous silica, commonly used as a desiccant, has a very high specific surface area. The relatively small percentage of amorphous silica actually makes up the majority of the overall specific surface area and causes the bulk material to have a greater capacity for adsorbed water that must be dried out before or during the evacuation process. The affect on vacuum retention following exposure of microspheres and perlite to atmospheric conditions without a drying process prior to evacuation follows this section.

Alternative glass bubbles to the Type K1 microspheres are produced by 3M and also by Emerson & Cuming. Options include a floating process that skims off low density (weak) bubbles and removes a portion of the condensed salts. A coating of methacrylaic chromic chloride is then applied that minimizes water pickup. The overall specific surface area is about half that of the Type K1 microspheres, which may allow reduced bake-out requirements due to lower water adsorption capacity. The use of thicker-walled bubbles will benefit applications where microspheres are exposed to intense localized forces.

TABLE 1. Thermal performance of 3M Type K1 microspheres

COLD VACUUM PRESSURE (torr)	APPARENT THERMAL CONDUCTIVITY (mW/m-K)	COMPARATIVE THERMAL PERFORMANCE
$1x10^{-3}$	0.7	7.0 times worse than MLI
$1x10^{-1}$	1.4	3.3 times better than perlite
760	22	1.5 times better than polyurethane

TABLE 2. Selected properties of 3M Type K1 microspheres

True density	0.125 g/cc (7.8 lb/ft^3)
Bulk density (@ 60% packing factor)	0.075 g/cc (4.7 lb/ft^3)
Particle size (mean / range)	65 / 15–125 microns
Isostatic crush strength	1.7 MPa (250 psi)
Maximum operating temperature	600^{o}C
Specific surface area	0.2 m^2/cc of bulk volume

Vacuum Retention

Vacuum retention testing was performed at TAI on equal volume samples of microspheres and perlite insulation following five days of exposure to ambient conditions. Subsequently, the samples were evacuated for eight hours without the application of heat during vacuum pumping or prior bake-out conditioning. The results of the eight-hour evacuation are that the microspheres reached a vacuum level of 3.7×10^{-4} torr and perlite reached 7.9×10^{-4} torr. The vacuum pump was then isolated from the chamber to initiate vacuum retention testing. FIGURE 2 shows how the microspheres were stabilizing at a vacuum level of 1×10^{-2} torr after 36 hours, but the perlite vacuum level exceeded 2×10^{-1} torr and continued to climb. The vacuum retention testing confirms that microspheres are less sensitive to water vapor adsorbed from the atmosphere than perlite and can support a stable vacuum more easily and quickly. Pumpdown time with respect to perlite condition has been studied by Kropshot and Burgess [3]. Their conclusion was that perlite must be dry, preferably protected or filled immediately upon exit from the expansion furnace. Otherwise, perlite evacuation may require several weeks, if it can be accomplished at all.

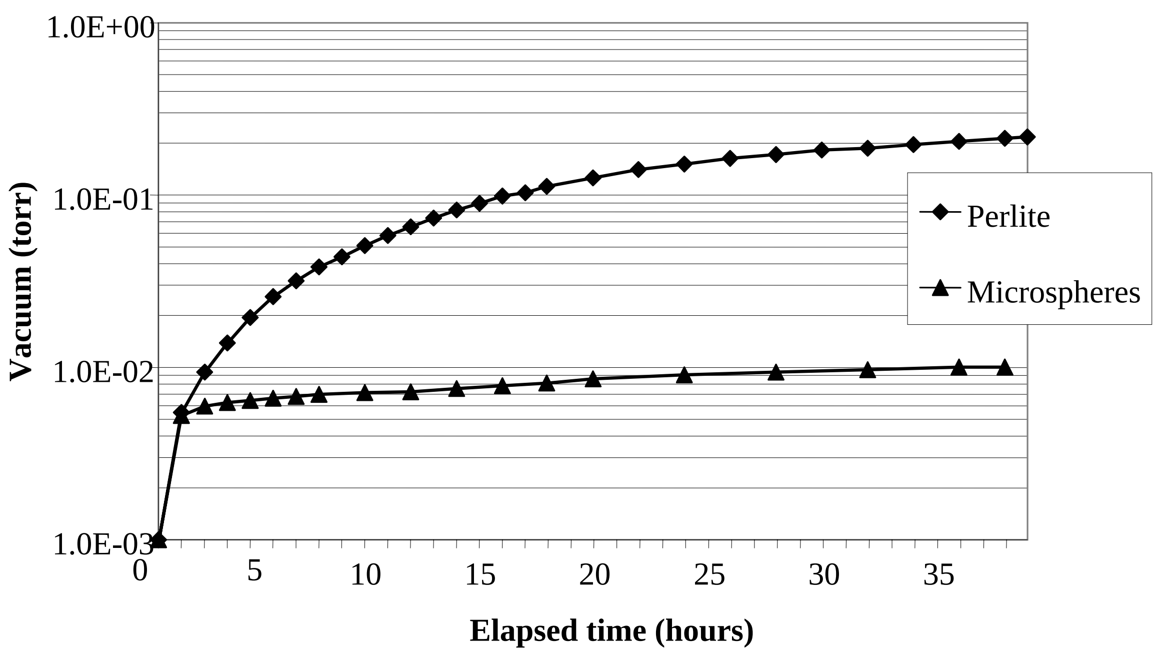

FIGURE 2. Vacuum decay comparison

MICROSPHERE INSULATION APPLICATIONS

Vacuum-Jacketed Storage Tanks

In addition to the thermal conductivity and vacuum retention tests described earlier, tests were performed to demonstrate that microspheres do not experience the compaction problem that commonly develops in perlite-insulated cryogenic storage tanks caused by repeated contraction-expansion cycles of the inner vessel. Subscale testing utilizing lab dewars verified that microspheres act essentially as an incompressible fluid within the vacuum space to preclude compaction. The testing at the Cryogenics Test Laboratory utilized two 10-liter lab dewars, one insulated with 3M Type K1 microspheres and the other with Silbrico grade #39 perlite. The objective of the testing was to compare the insulation level of both insulation systems before and after each of ten thermal cycles from 300 K to 77 K in order to detect the occurrence of compaction. The vacuum pressure was maintained below 1×10^{-3} torr. Vibration was also applied to the dewars during the cold state of each thermal cycle to accelerate the compaction effect of the inner vessel contraction and expansion.

The insulation level change from the initial condition following each thermal/vibration cycle is illustrated in FIGURE 3. The test results clearly confirm expectations regarding insulation compaction experienced by each material. The perlite insulation level showed a steady decline for a total of 5 mm after 10 cycles. This was in contrast to the microsphere insulation that actually “fluffed up” during evacuation from the initial fill level and had a final level 8 mm above the original level at the conclusion of the test. Accelerated life testing of microspheres and perlite in a mid-sized cryogenic storage tanks (500-5000 gal) is planned during 2004 following the award of a follow-on NASA SBIR Phase II program.

Vacuum-Jacketed Transfer Lines

A key benefit of microsphere insulation is that performance does not degrade as rapidly as MLI when vacuum level decays. The result is far less vacuum maintenance performed on transfer lines than those insulated with MLI. Microspheres provide superior

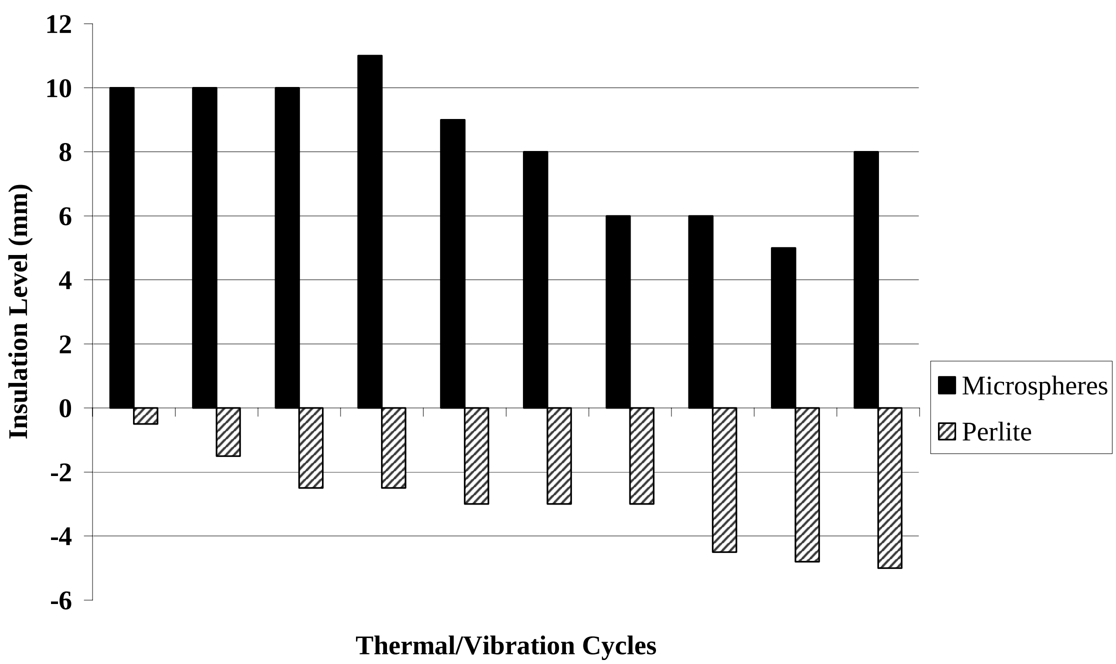

FIGURE 3. Microsphere and perlite insulation level change following thermal/vibration cycles

thermal performance to MLI above $3x10^{-2}$ torr. Vacuum-jacketed transfer lines in actual use are often not maintained unless there is a visual indication of high heat leak, such as frost on the outer jacket. Soft vacuum conditions, sufficient to avoid a visual indication of poor performance, often go unnoticed. This condition results in a higher boil-off rate and corresponding operational cost. In the case where vacuum is lost in a microsphere-insulated segment, thermal performance will remain comparable to foam insulation. Flexible transfer lines experience significantly degraded thermal performance under bending conditions [4]. The incompressible nature of microspheres may alleviate this problem.

Evacuated Insulation Panels

Significant effort has been focused on the development of evacuated microsphere insulation panels (MIP) at TAI during the past two years. The load bearing capability of the microspheres provides structural support for a thin, flexible vacuum-barrier film. The barrier material used is Mylar® 250SBL300, a multilayer polyester-based laminate that contains several non-foil layers to provide a super-barrier to atmospheric gases and water vapor permeation. This film was specifically developed for the non-cryogenic vacuum insulation panel (VIP) market and has been in commercial use for several years. FIGURE 4 shows the vacuum chamber used by Thermal Visions, Inc. to fabricate MIP for TAI. The chamber contains special tooling to heat-seal the film edge seams while under vacuum at $<1x10^{-1}$ torr. The thermal performance of flexible vacuum-barrier MIP is about two times better than polyurethane foam based on ASTM C 518 and C 177 thermal conductivity tests.

FIGURE 5 shows a clamshell-shaped MIP used to insulate cryogenic transfer lines in place of foam insulation. While Mylar® 250SBL300 barrier film has a projected life in excess of 20 years, the MIP clamshells applied to cryogenic transfer lines will be over-wrapped with a vapor barrier to prevent water vapor from collecting between the MIP and pipe and then protected by a weatherproof PVC jacket. Prototype testing of 1-m lengths of the MIP pipe insulation system has been performed on a 3-in IPS pipe test apparatus at TAI that is liquid nitrogen cooled. Field demonstration of an MIP-insulated transfer line at a NASA facility is anticipated to begin late in 2003.

Rigid-barrier MIP development at TAI has resulted in the successful fabrication of flat panels for an above-ambient temperature application for Bell Helicopter Textron Inc., but continues to face technical challenges for a clamshell configuration to insulate cryogenic transfer lines. The development issues for rigid-barrier cryogenic MIP involve the compensation of dimensional changes and related stresses caused by thermal expansion and the identification of cost-effective fabrication methods.

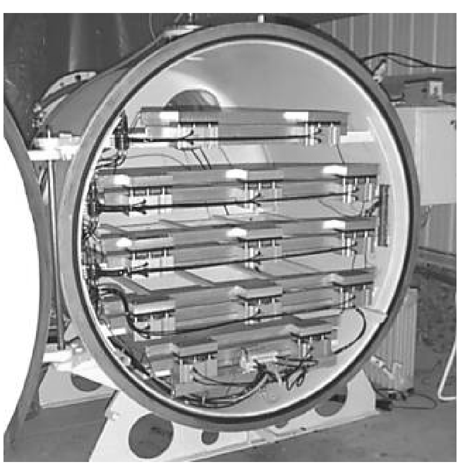

FIGURE 4. Vacuum process chamber

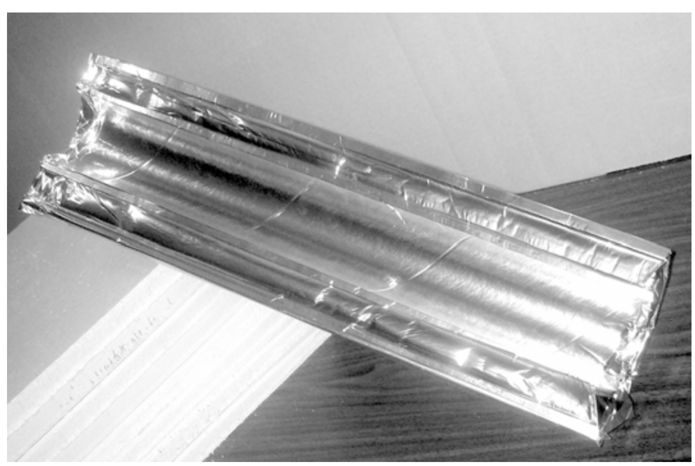

FIGURE 5. Clamshell-shaped MIP

A special test panel jointly developed by TAI and the Cryogenics Test Laboratory was used to measure the effect of compressive loads on the apparent thermal conductivity (k-value) of flexible vacuum-barrier MIP. Tests were performed at the Cryogenics Test Laboratory at vacuum levels from 1×10^{-4} torr to 760 torr. Conditions included no load, medium load (9 psi), high load (20 psi), and elevated temperature (boundary temperatures of 347 K and 240 K resulting in a mean temperature of 293 K). A simplified schematic of the test assembly is given in FIGURE 6. The calibrated k-values are given in FIGURE 7.

The thermal performance was found to degrade, as expected, with increasing load. Under high vacuum, the k-value increased about 60% comparing the 20 psi load case to the no load case, while soft vacuum and no vacuum conditions resulted in little difference. The final test series performed at elevated temperature, with mean temperature near ambient, was in full agreement with previous ASTM C 518 testing performed by the Cryogenics Test Laboratory [2]. Note that an additional heat load occurs for test series L102 – L105 relative to L101 due to a parallel thermal path through the test panel G-10 fiberglass walls.

CONCLUSIONS

A better understanding of microsphere insulation properties obtained through recent sub-scale testing has provided a clearer vision for applications that can benefit from this technology. Microspheres offer a superior bulk powder-type insulation material to replace perlite in cryogenic storage tanks and provide advantages over MLI for low-maintenance tanks and transfer lines. For the cryogenic tank manufacturer, microspheres can be handled in much the same way as perlite, utilizing similar equipment and processes. Testing at TAI has shown microsphere insulation is evacuated more quickly than perlite. The end-user can expect an insulation system that is longer-lasting since microspheres preclude the perlite compaction problem and require less maintenance to retain desired thermal performance than was previously possible, equating to significant cost savings through reduced vacuum maintenance, repair costs, and lower cryogen loss.

Microsphere insulation systems are unlikely to entirely displace MLI, but perlite insulation for cryogenic storage tanks and foam insulations for transfer lines may become obsolete. Review of this technology and recent tests with NASA and commercial customers has yielded considerable interest in full-scale performance demonstrations. The high potential of microsphere insulation technology to solve long-standing problems faced by NASA and industry presents a compelling case for further development and adoption.

Surface Temperature Measurement

Sensor	Location
T1, T3	Bottom of Cold Mass (CBT)
T2	Top of Cold Mass
T4, T5	Top of Load Cell
T6, T7, T8	Top of G-10 Plate (WBT)
VC1, VC2, VC3	Vacuum Chamber Exterior
VC4, VC5, VC6	Heater Temperature

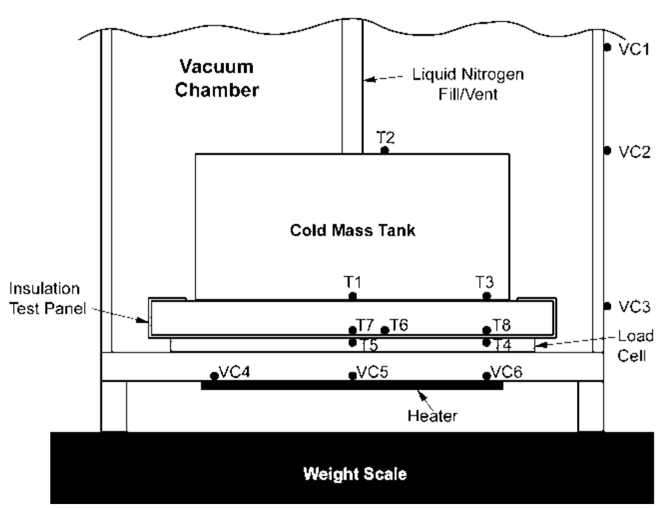

FIGURE 6. Simplified schematic of Cryostat-5 test assembly showing temperature sensor locations

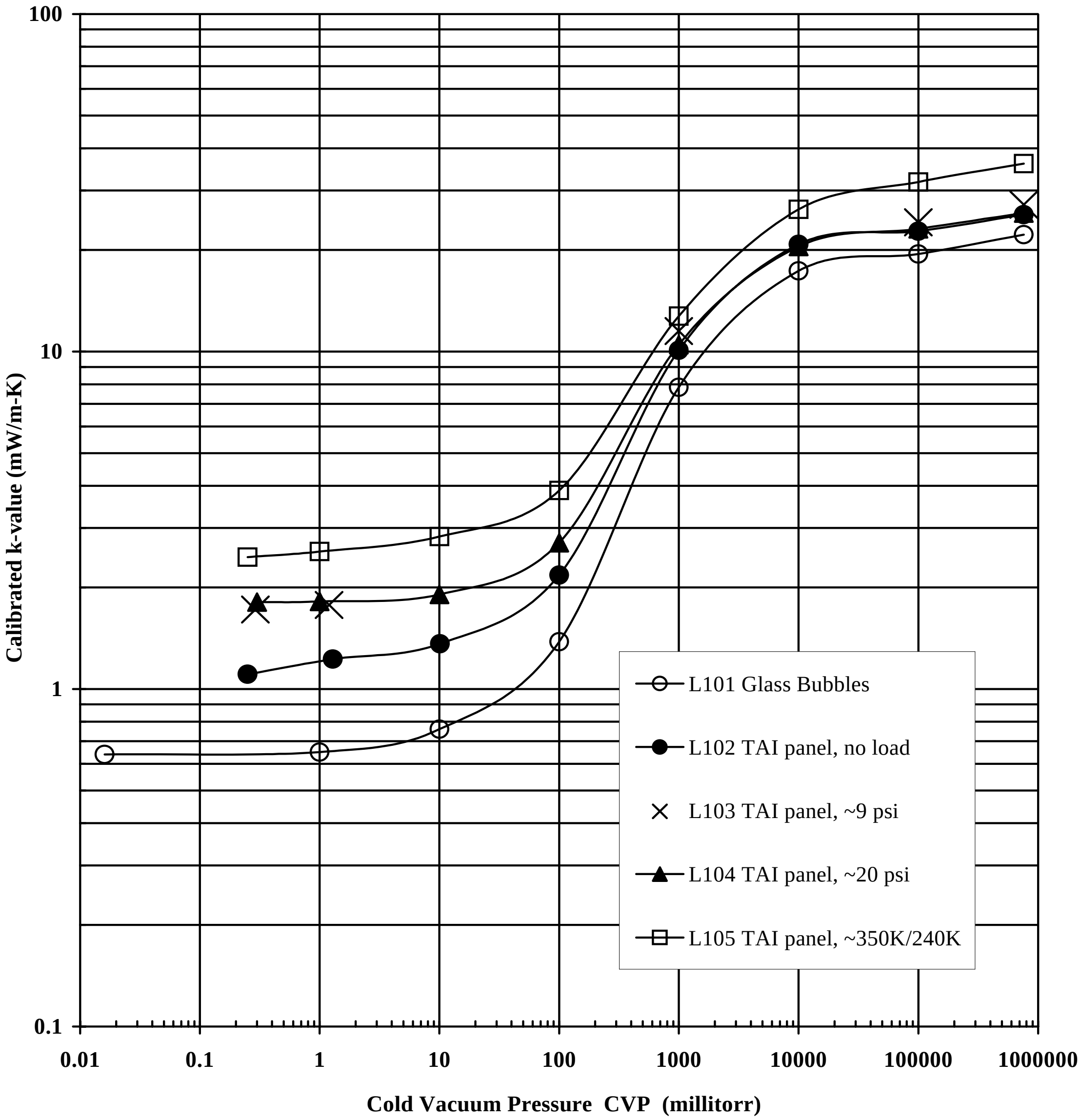

FIGURE 7. Calibrated k-value variation with vacuum pressure. Boundary temperatures are 293 K and 78 K

ACKNOWLEDGEMENTS

The authors thank Mr. Edward Myers and Dr. David Daney of Technology Applications, Inc.; Mr. Dwight Musgrave of Thermal Visions, Inc.; and Mr. Wayne Heckle of Sierra Lobo, Inc. This work was supported with NASA SBIR funding under contracts NAS10-01008, NAS10-02013, and NAS10-03016.

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