# **COST-EFFICIENT STORAGE OF CRYOGENS**

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# ABSTRACT

NASA's cryogenic infrastructure, which supports launch vehicle operations and propulsion testing, is reaching an age when major refurbishment is required. Key elements of this infrastructure are the large double-walled cryogenic storage tanks used for both space vehicle launch operations and rocket propulsion testing at various NASA field centers. Perlite powder has historically been the insulation material of choice for these applications, but new bulk-fill insulation materials, including glass bubbles and aerogel beads, have been shown to provide improved thermal and mechanical performance. Research was conducted on thermal performance to identify operational considerations and risks associated with using these new materials in large cryogenic storage tanks. The program was divided into three main areas: material testing (thermal conductivity and physical characterization), tank demonstration testing (liquid nitrogen and liquid hydrogen), and system studies (thermal modeling, granular physics, and insulation changeout). This research showed that more energy-efficient insulation solutions are possible for large-scale cryogenic storage tanks worldwide and summarized the operational requirements that should be considered for these applications.

**KEYWORDS:** Cryogenic tanks, thermal insulation, granular materials, liquid hydrogen boiloff, glass bubbles, perlite

## INTRODUCTION

Cryogenic storage tanks with a capacity of 189,271 liters and larger typically have thermal insulation systems consisting of a double-wall tank with perlite powder insulation filling the annular space. For liquid hydrogen (LH<sub>2</sub>) tanks, this annular space must also be evacuated to a high vacuum level to keep boiloff losses at an acceptable level. For large liquid oxygen (LO<sub>2</sub>) tanks, the annular space is often kept at ambient pressure with a nitrogen purge to keep the insulation material dry.

This paper summarizes the research testing program, *Cost-Efficient Storage and Transfer of Cryogens* (CESAT), led by the Cryogenics Test Laboratory at NASA's Kennedy Space Center. Glass bubbles and aerogel beads were studied as potential alternatives to using perlite powder in insulation systems. We quantified the energy-efficiency improvements of these insulation materials in realistic tank configurations and addressed the engineering challenges of large-scale tank applications. Several materials were tested for a number of applications, including piping and tanks, but the program focused on using the glass bubbles for evacuated tank applications.

The research program was arranged along three lines of work: materials research, tank demonstration testing, and system studies. Materials research tasks were designed to characterize the physical, chemical, and thermal properties of the insulation materials under representative tank conditions. Tank demonstration tests used 1,000-liter spherical research tanks as well as cylindrical industrial tanks to evaluate the performance of the insulation in a near-operational environment. Systems studies covered key operational issues including safety, technical, and economic considerations.

#### **Future Operational Needs**

At Launch Complex 39 (LC-39) at the Kennedy Space Center, two launch pads, A and B, are used for Space Shuttle launch operations and will be reconfigured in the near future for the new Ares launch vehicle. Plans call for reusing the existing  $LH_2$  and  $LO_2$  storage tanks that have been in continuous operation since the Apollo program in the 1960s. The benefits and risks associated with changing the insulation material must be understood to make good engineering decisions regarding tank refurbishment or rehabilitation.

Based on its current performance, the LH<sub>2</sub> tank at LC-39B is a candidate for a retrofit with glass bubble insulation. This 3,200,000-liter capacity tank is shown in FIGURE 1. Built by Chicago Bridge & Iron in 1965, the vacuum-jacketed spherical tank has a 21.3-m diameter carbon steel outer shell and an 18.3-m diameter stainless steel inner tank. The 1.5-m thick annular space is filled with approximately 1,883,000 liters of high density perlite powder and maintains a vacuum level in the range of 15 millitorr. The daily hydrogen boiloff of 3,407 liters per day from this tank is approximately three times as much as for the identical tank at LC-39A and 50% higher than the design specification. In addition, the tank has undergone three full thermal cycles, and the tank manufacturer recommends replacing the insulation after five thermal cycles.



**FIGURE 1.** Past, present, and future views of the LH<sub>2</sub> storage tank at LC-39: Apollo Saturn V (left), Space Shuttle (center), Ares (right).

# **Insulation Materials**

Glass bubbles were studied for use in tanks with an evacuated annular space and aerogel beads for use under ambient pressure. Micrographs of the materials are shown in FIGURE 2. The study focused on Series K1 glass bubbles by 3M, which are widely available and cost less. The K1 microsphere is a thin-walled hollow lime borosilicate glass sphere with residual gas in the interstitial volume. The mean diameter is 65 microns, and the bulk density is about 70 kg/m<sup>3</sup>. The aerogel material studied was a Nanogel product of Cabot Corporation. The aerogel beads are 1-mm spherical particles with a bulk density of about 80 kg/m<sup>3</sup>. The perlite powder material used was cryogenic-grade Ryolex Grade #39, produced by Silbrico Corporation. The bulk density of the perlite powder is nominally 112 kg/m<sup>3</sup>, but can vary from 60 kg/m<sup>3</sup> to 220 kg/m<sup>3</sup> with handling.

# **MATERIALS RESEARCH**

# **Thermal Conductivity**

Thermal conductivity testing was performed using three different methods. The Cryogenics Test Laboratory and the National High Magnetics Field Laboratory used custom-designed cryostats to measure heat transmission. Marshall Space Flight Center performed thermal conductivity testing by a third complementary method using ASTM C177. Results confirm that glass bubbles are advantageous for high-vacuum (<1 millitorr) applications while aerogel beads are preferred for ambient pressure applications. Thermal conductivity testing are given in separate papers [1-4].



**FIGURE 2.** Microscope comparison of three bulk-fill insulations: 65 µm glass bubbles (left, 200X), 600 µm perlite powder (center, 100X), and 2000 µm aerogel beads (right, 100X).

	Apparent Thermal Conductivity	
Insulation Material	(mW-m/K)	
	High Vacuum	Ambient Pressure
Perlite Powder	0.9	36
Glass Bubbles	0.6	27
Aerogel Beads	1.8	14

## **Mechanical Settling and Vibration Testing**

Comparative mechanical settling testing was conducted using vertical tubes to provide insight into how each of the materials behaves under both dynamic (filling/draining) and static conditions. A test apparatus was devised to evaluate the material packing and flow characteristics expected during tank operations and material installation. The apparatus consisted of vertical clear plastic tubes filled with different insulation materials and subjected to various external load conditions. Results showed that the measured density of the glass bubbles consistently fell within the published bulk density range of 72 kg/m<sup>3</sup> to 85 kg/m<sup>3</sup> while the measured density of the perlite varied greatly depending on the external loads applied. The glass bubbles also exhibited improved flow characteristics over perlite in a series of draining tests [5].

# **Vacuum Pumping and Retention**

Vacuum pumping and retention tests were performed using small vacuum chambers as well as the 1,000-liter research tank. Initial testing in the small chambers showed that the pumpdown and retention characteristics of the bubbles and perlite were comparable under controlled laboratory conditions [6]. Further evacuation testing was conducted with the tank demonstration testing. Results showed that the pumpdown of glass bubbles in larger volumes was somewhat more difficult due to the smaller particles, but that the vacuum retention was much better. An additional observation was that additional filtration was necessary with the bubbles to fully protect the vacuum pumping system.

#### **Structural Integrity Testing**

Structural integrity of the bubbles is of major interest in determining the long-term reliability of the insulation system. Series K1 glass bubbles have a specified crush strength of 1,724 kPa (250 psi), obtained by a nitrogen isostatic test procedure in which the bubbles are subjected to a uniform hydraulic pressure. An additional test was devised to determine a more representative crush strength due to the point-to-point contact between individual bubbles. Pneumatic pressurization tests from 172 kPa to 20,684 kPa were performed by placing the material in a chamber and pressurizing it for 5 minutes. Visual and microscope examinations, as well as a particle size analysis, were conducted to quantify microsphere strength and breakage limits. Microscope inspection indicated breakage in the 517 kPa to 689 kPa range (see FIGURE 3) and catastrophic damage at pressures at and above 3,447 kPa.

Based on these results, which showed that the pneumatic crush strength is lower than the isostatic strength, another test was devised to evaluate the strength of the bubbles with point-to-point mechanical loading. A moving-wall test fixture was constructed consisting of a steel box in which one of the vertical 254-mm by 178-mm walls was compressed 75 mm using a hydraulic jack and load cell to measure the force. Measurable breakage of the glass microspheres starts to occur above 345 kPa, as indicated in FIGURE 4. A 20% change in the volume of the glass bubbles is required to create this pressure. This displacement is substantially higher than any change in volume associated with a cryogenic tank thermal cycle [7].

# **Electrostatic Property Testing**

Series K1 glass bubbles are classified as statically dissipative under room humidity conditions (~50% humidity) and insulating at lower humidity (~20%) while perlite is statically dissipative under both humidity conditions. These results agree with measurements of the dielectric constant. Corona charge dissipation testing showed that the glass bubbles have the ability to dissipate charge under both ambient and low-humidity conditions. Performing discharge incendivity testing on perlite showed that this material would not charge an insulating material enough to cause a large discharge during a filling operation. This testing could not be performed on the glass bubbles due to their fluidic behavior. They could not be contained in the experimental test setup because when they came in contact with other glass bubbles, they generated sufficient electrostatic charge to agglomerate particles. The final series of tests measured the electrostatic charge formed on the particles in contact with a variety of materials (stainless steel, aluminum, copper, PTFE, plastic, and glass). Results showed that minimal electrostatic charge was generated on the glass bubbles with any pipe material tested [8].



FIGURE 3. Micrographs (100X) of pneumatic pressurization tests at 344 kPa (left) and 689 kPa (right).



**FIGURE 4**. Experimental test results for glass bubbles in the moving wall test fixture [1 psi = 6.89 kPa].

# **Material Compatibility Testing**

Autogenous ignition temperature (AIT) testing in a high-pressure oxygen-enriched environment was conducted on all three insulation materials in accordance with ASTM G 72-83. No autogenous ignition was observed up to 449 °C (840 °F). Limited oxygen index (LOI) testing in accordance with ASTM D2863 showed the LOI for perlite and glass bubbles to be more than 99.5% and found that these materials could not be ignited in a 100% oxygen environment. The LOI for the aerogel beads was found to be 28.1%, which is well above the concentration of oxygen in air [9]. Minimum ignition energy (MIE) testing was also conducted on both perlite powder and glass bubbles, showing that both materials could not be ignited and that more than 10 joules would be required to ignite aerosols comprised of either material [8].

### **Corrosion Testing**

During manufacturing of the bubbles, fine particles of glass are heated enough for the glass to flow easily and surface tension to cause the particles to become spherical. A latent blowing agent within the glass then evolves to the gaseous state, blowing the bubble into its hollow form. As a result, some sulfur dioxide (SO<sub>2</sub>) is trapped in the glass bubble interstitial space. Accelerated environmental testing was performed to better understand the corrosive effects of SO<sub>2</sub> on both carbon and stainless steel materials used in the LC-39 tank. The results showed no corrosion on either the carbon or stainless steel under dry conditions representative of the evacuated tank even when the materials were subjected to SO<sub>2</sub> levels higher than the levels expected due to minor bubble breakage. Results of this testing showed that corrosion of the tank should not be an issue under normal operations; however, special efforts to keep the insulation dry and to remove the SO<sub>2</sub> through purging should be practiced whenever the tank vacuum is broken [10].

# TANK DEMONSTRATION TESTING

Tank demonstration testing was designed to evaluate insulation performance from a total system perspective. As FIGURE 5 shows, three levels of testing were performed: using 10-liter dewars, 6,000-gallon industrial tanks, and 1,000-liter research tanks. Extensive dewar testing and subscale experiments validated the benefits of the new insulation systems using glass bubbles and aerogel beads (which had improved thermal performance and no compaction problems) [11]. Liquid nitrogen (LN<sub>2</sub>) field testing of industrial tanks was performed in 2005. Two 22,700-liter (6,000-gallon) vertical cylindrical tanks were tested at ACME Cryogenics under the technical direction of Technology Applications, Inc. This work, part of a Phase II NASA research project, provided a comparative test of perlite powder and glass bubbles. The LN<sub>2</sub> boiloff and thermal testing showed that the glass bubbles were easy to install, reduced boiloff losses, and did not break over time [12].



FIGURE 5. Views of test articles: 10-liter dewars, 6,000-gallon industrial tanks, 1,000-liter research tanks.

Demonstration testing of the 1,000-liter research tanks was performed with  $LN_2$  and  $LH_2$  in 2005 and 2006, respectively. Two identical vacuum-jacketed tanks, each with a capacity of 1,000 liters, were designed and constructed. The very-low-heat-leak design is a  $1/15^{th}$  scale version of the spherical  $LH_2$  storage tanks at LC-39 and includes multipurpose viewports, feedthroughs, and flange mounts. Instrumentation included: diode temperature sensors, liquid level sensors, full-vacuum-range pressure transducers, load cells, tri-axial accelerometers, and mechanical displacement indicators. These research tanks offer an extensive thermal and mechanical test capability. System fabrication and an extensive series of  $LH_2$  tests were performed at PHPK Technologies. Further  $LN_2$  testing and thermal cycling were then conducted at the Cryogenics Test Laboratory. Test results show that the bubbles reduce boiloff and do not break or compact with thermal cycling. The details of this testing are presented in a separate paper [13].

# SYSTEM STUDIES

System studies were performed to understand the risks and benefits of using the new insulation materials in cryogenic tanks and piping systems. Several of these studies are highlighted as follows.

## Numerical Modeling of Cryogenic Tank Boiloff

Numerical modeling of both the 1,000-liter test tanks and the 3,200,000-gallon LH<sub>2</sub> tank at LC-39B was performed by Marshall Space Flight Center using the Generalized Fluid System Simulation Program (GFSSP). Numerical predictions of the LC-39B tank boiloff and ullage temperatures were produced. Modeling of the 1,000-liter research tank was also performed. Analytical and experimental results were compared to validate the heat transfer mechanisms within the tank. The predicted daily boiloff rates for perlite and glass bubbles are 977 liters and 689 liters, respectively. Details of the thermal modeling are presented in a separate paper [14].

#### **Insulation Installation Study**

The logistics of transporting, staging, and on-site material handling were evaluated. For a baseline for the logistical plan for using bubbles, techniques for installing perlite in large tanks were reviewed. Typically, mobile processing units are used to transport raw perlite material to the tank's location. Finished perlite powder is produced on-site by heating the raw perlite material to 870 °C. The perlite is then installed warm to minimize moisture absorption. Glass bubbles would be produced ready-to-use at the manufacturing plant and transported to the site in tankers. A slight purge of compressed air or nitrogen

would be used to fluidize the material inside the tankers and make it easier to use vacuum or pressure to transfer the bubbles into the annular space.

#### **Granular Physics Study**

The compaction and crushing behavior of the glass bubbles was studied to predict pressures that glass bubbles would be subjected to during a typical cryogenic tank thermal cycle. Modeling of the spherical tank geometry showed that glass bubbles in the top third of the tank would be in a frictional flow regime that would allow the them to flow easily during volume changes in the annular space while the bottom two-thirds of the tank would be in a linear elastic regime where the bubbles would be constrained from flowing and subjected to higher compressive loads. Testing was also done to determine the strength and elastic properties. Results showed that the glass bubbles in the bottom of the tank would be subjected to maximum pressures in the 14 to 21 kPa range (see FIGURE 6), much less than the 241-kPa breakage pressure measured during structural integrity testing [7].



**FIGURE 6.** Granular physics model prediction of maximum pressure of glass bubbles within annular space of spherical tanks.

# CONCLUSIONS

Glass bubbles and aerogel beads were studied as potential alternatives to using perlite powder in insulation systems. Testing has shown that glass bubbles provide the best thermal performance for an evacuated tank application while the aerogel beads material is best for nonevacuated tank applications. Detailed investigations were conducted for large, spherical, vacuum-jacketed LH<sub>2</sub> tanks. The hydrogen boiloff was found to be approximately 35% less for bubbles compared to perlite. Thermal cycling tests with liquid nitrogen confirmed that the glass bubbles do not break and that compaction does not occur. Thermal modeling of an LH<sub>2</sub> storage tank and the new 1,000-liter research tank was performed to predict boiloff rates and validate internal heat transfer mechanisms.

Current cryogenic storage tank infrastructure remains based on technology from the 1960s. A large-scale field application is being considered for the perlite-to-glass bubbles retrofit of a 190,000-liter spherical, vacuum-jacketed  $LH_2$  tank at Stennis Space Center. The handling of glass bubbles on such a large scale will be a key feature of the work. Further work in the materials research area should include thermal optimization of glass bubbles, evaluation of higher strength bubbles for transfer line applications, and granular physics modeling of glass bubbles. Life-cycle characteristics of over-the-road tanks, with vibration and environmental effects, is also needed

Demonstration testing of aerogel insulation in large  $LO_2$  or liquefied natural gas (LNG) tanks is now proceeding. This research has resulted in other work to incorporate new features such as structural instrumentation, fluid system diagnostics, load-supporting insulation materials, and integrated cryocoolers. Considering the thermal insulation system as an integral part of the total system design is essential for making advances toward truly cost-efficient, reliable cryogenic systems and for producing the "smart tanks" of the future.

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