CRYOPUMPING IN CRYOGENIC INSULATIONS FOR A REUSABLE LAUNCH VEHICLE

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ABSTRACT

Testing at cryogenic temperatures was performed to verify the material characteristics and manufacturing processes of reusable propellant tank cryogenic insulations for a Reusable Launch Vehicle (RLV). The unique test apparatus and test methods developed for the investigation of cryopumping in cryogenic insulations are described. Panel level test specimens with various types of cryogenic insulations were subjected to a specific thermal profile where the temperature varied from -262°C to 21°C. Cryopumping occurred if the interior temperature of the specimen exhibited abnormal temperature fluctuations, such as a sudden decrease in temperature during the heating phase.

KEY WORDS: Cryogenic, Characterization, Insulation, Foams, Testing/Evaluation, Thermal Conductivity

1. INTRODUCTION

One of the goals for the next generation of launch vehicles is an order of magnitude reduction in the cost of delivering a payload to orbit. Studies on space transportation by the National Aeronautics and Space Administration (NASA) [1,2] indicate that a Single-Stage-To-Orbit (SSTO) Reusable Launch Vehicle (RLV), fueled by Liquid Hydrogen (LH₂) and Liquid Oxygen (LOX) has the potential to reach this goal. These technologies are being pursued to develop an RLV that has efficient, and airline-like operation with 7-day refurbishment cycles between missions to reduce the operational costs and increase safety, thereby reducing the cost to place a payload in orbit.

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1 This paper is declared a work of the U.S. Government and is not subject to copyright protection in the United States.
Reusable cryogenic propellant tanks for an RLV are required to contain the LH\textsubscript{2} and LOX. The fabrication of these tanks is one of the significant technical challenges to be overcome to develop an operable RLV [3]. Large (8.38 m.) expendable cryogenic tanks have been made for launch vehicles, but the performance of flight-weight reusable cryogenic propellant tanks with the required insulation systems have not been demonstrated [4]. Cryogenic insulation development is critical for an RLV because the tanks must function as primary structure and pressure vessels at both cryogenic temperatures and elevated re-entry temperatures [4]. The insulation maintains the quality and quantity of propellant during ground-hold and ascent, and acts as a thermal barrier during re-entry and thermal soak through after landing. Candidate cryogenic insulations must be durable, easy to maintain, easy to repair, and reusable for the life of the vehicle [5].

Cryopumping occurs when a void in a material or structure is at a low enough temperature to densify a contained volume of gases [6]. As the gases are condensed, a vacuum is created. If there is a pathway to the surface or other voids, additional gases are pulled or pumped into the void. When the material or structure is heated, the densified gases expand and are expelled or pumped from the void. During this expansion phase of cryopumping, the most damage in the material can be incurred. Cryopumping can be problematic for cryogenic materials and structures if the gases are densified into a void, then the void is rapidly heated, but the flow of gases from the void is restricted, causing a rapid pressure buildup. Cryopumping in the proof testing of the liquid hydrogen tank for the X-33 contributed to the failure of the honeycomb tank wall [7].

During uniaxial tension tests [3,4] of 30.5 cm. by 61 cm. (1 ft. by 2 ft.) panels to evaluate the durability of neat TEEK cryogenic insulations, temperature drops were observed at the mid-plane of the cryogenic insulation after the cryogen flow was stopped. Cryopumping [6] inside the cryogenic insulation was determined to contribute to the temperature drop. The thermal profile test, presented in this paper, was developed to measure the temperature drop and investigate cryopumping in cryogenic insulations. The objective of the thermal profile test was to measure the temperature change caused by cryopumping at various levels through-the-thickness of candidate coated cryogenic insulations. The objective of this paper is to provide details on the newly developed test methods and identify the susceptibility of nine candidate cryogenic insulations to cryopumping.

Cryopumping was observed in the current tests by monitoring thermocouples embedded in the out-of-plane direction (Through-the-thickness) of the cryogenic insulations. If a temperature drop was indicated in the interior of the cryogenic insulation after the specimen had achieved a steady state temperature condition and the cryogen flow had been turned off, cryopumping was assumed to have occurred inside of the cryogenic insulation near the substrate. For these tests, a new thermal profile test rig and test method were developed, the existence of cryopumping in candidate cryogenic insulations for RLV propellant tanks for NASA and Industry was determined, and the results were addressed to the closed cell content of the candidate cryogenic insulations.
2. TEST SPECIMEN AND EXPERIMENTS

2.1 Specimen Description  The specimen used in the thermal profiles test consisted of a 28 cm. by 30.5 cm. laminated composite substrate with one of the first nine 28 cm. by 28 cm. cryogenic insulations listed in Table 1 bonded on the upper surface. The (+45/-45/90/0)_S quasi-isotropic composite substrates were processed from IM7/PETI-5 [8,9] tape prepreg for all tests. Thermocouples were located through-the-thickness of the cryogenic insulation, on the tank wall (Inner) surface; in the bondline, and on the upper cryogenic insulation (External) surface to accurately monitor temperature change over three planes of the specimen during the test, (See Figures 1 through 3). The through-the-thickness thermocouples were located at approximately 0.424 cm. intervals to measure temperature change inside the specimen during the tests.

Table 1. Nine cryogenic insulations and Korex™ and Nomex™ honeycomb cores used in the tests.

<table>
<thead>
<tr>
<th>Spec. No.</th>
<th>Cryogenic Insulation</th>
<th>Chemical Description</th>
<th>Average Density (g/cc)</th>
<th>Closed Cell Content (%)</th>
<th>k-Value At 760 Torr [10] (mW/m-K)</th>
<th>Tg (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Marcore™ Polyisocyanurate</td>
<td>Polyisocyanurate</td>
<td>0.0958</td>
<td>83.65</td>
<td>27.62</td>
<td>315</td>
</tr>
<tr>
<td>2</td>
<td>TEEK/Korex™ (With a gap) Polyimide</td>
<td>Polyimide</td>
<td>0.102</td>
<td>18.21</td>
<td>51.56</td>
<td>245</td>
</tr>
<tr>
<td>3</td>
<td>SF-38 (Spray Foam) Polyurethane</td>
<td>Polyurethane</td>
<td>0.0879</td>
<td>95.57</td>
<td>24.24</td>
<td>85</td>
</tr>
<tr>
<td>4</td>
<td>BPCI/Nomex™ Polyurethane</td>
<td>Polyurethane</td>
<td>0.0766</td>
<td>88.17</td>
<td>27.44</td>
<td>85</td>
</tr>
<tr>
<td>5</td>
<td>BPCI/TEEK/Nomex™ Polyimide/Polyurethane</td>
<td>Polyimide/Polyurethane</td>
<td>0.071</td>
<td>47.01</td>
<td>28.32</td>
<td>85*</td>
</tr>
<tr>
<td>6</td>
<td>Divinylcell™ PVC base IPN with Polyurea &amp; Polyimide</td>
<td>PVC base IPN with Polyurea &amp; Polyimide</td>
<td>0.0461</td>
<td>85.02</td>
<td>25.26</td>
<td>85</td>
</tr>
<tr>
<td>7</td>
<td>Airex™ Polyetherimide</td>
<td>Polyetherimide</td>
<td>0.0623</td>
<td>22.57</td>
<td>27.27</td>
<td>160</td>
</tr>
<tr>
<td>8</td>
<td>Neat TEEK Polyimide</td>
<td>Polyimide</td>
<td>0.0415</td>
<td>23.97</td>
<td>25.36</td>
<td>245</td>
</tr>
<tr>
<td>9</td>
<td>TEEK/Nomex™ Polyimide</td>
<td>Polyimide</td>
<td>0.072</td>
<td>17.83</td>
<td>27.83</td>
<td>245</td>
</tr>
<tr>
<td>10</td>
<td>Korex™ Aramide/Phenolic</td>
<td>Aramide/Phenolic</td>
<td>0.032</td>
<td>-</td>
<td>67.58</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td>Nomex™ Aramide/Phenolic</td>
<td>Aramide/Phenolic</td>
<td>0.032</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

*Maximum Tg is based on BPCI.

BPCI – Boeing Polyurethane Cryogenic Insulation
PVC – Polyvinyl Chloride
IPN – Interpenetrating Material
Closed Cell Content – Based on manufacture’s data
k-Value – Thermal Conductivity
Tg – Glass transition temperature, Based on manufacture’s data

2.2 Specimen Preparation  Five 0.038-cm.-diameter holes were drilled into the cryogenic insulation to accommodate thermocouples. The holes were drilled at an angle of 45° using a drill press with digital display and following the hole pattern in the schematic shown in Figure 1. This hole pattern was followed for all of the neat cryogenic

2 The use of trademarks or names of manufacturers in this report is for accurate reporting and does not constitute an official endorsement, either expressed or implied, of such products or manufacturers by NASA.
insulation specimens. For the foam/honeycomb specimens, the depth and angle were identical, however, the 2.54 cm. spacing was altered in order to place the thermocouple beads at the center of the honeycomb cells.

![Thermocouple Location and Numbering](image)

Figure 1. Schematic of the through-the-thickness thermocouple location (Dimensions are in cm.) and numbering (In italics).

2.2.1 Cryogenic Insulations The nine neat, and foam/honeycomb cryogenic insulations listed in Table 1 were from NASA Langley Research Center (LaRC) and Industry. The chemical description, average density, closed cell content, thermal conductivity, and glass transition temperature of the nine cryogenic insulations are listed in Table 1. The Korex™ and Nomex™ honeycomb core used in the cryogenic insulations had a cell size of 0.034 cm. (3/16 in.) and 0.068 cm. (3/8 in.), respectively. The average density of the Korex™ and Nomex™ honeycomb cores and the thermal properties of the Korex™ honeycomb core are listed in Table 1.

2.2.2 Bonding of Cryogenic Insulation Seven, type E thermocouples were taped to the composite substrate surface shown in the schematic in Figure 2 to monitor the bondline temperature. Both the composite substrate and cryogenic insulation surface were coated with the Hysol EA9394 epoxy (Epoxy) for every specimen except the TEEK filled Korex™ honeycomb with a gap. Only the substrate was coated with epoxy for the TEEK filled Korex™ honeycomb specimen since there was gap on the honeycomb surface. The specimen was then bagged and held under full vacuum (100 kPa) at room temperature for 24 hours. This procedure was consistent for all specimens except the neat TEEK cryogenic insulation, which would be crushed under the 100 kPa of pressure. Pressure was applied on the neat TEEK material by placing 27.2 kg of dead weight on top of a load distribution plate placed over the cryogenic insulation.

2.2.3 Thermocouples The bonded specimens were instrumented with type E, type T, and type K thermocouples according to the patterns shown in the schematics in Figures 2 and

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3 The proportion of closed to open cells in a given foam material quantifies the resistance of the foam to the flow of gases through the foam and the insulation characteristics of the foam. Foams that have high closed cell content inhibit cryopumping or the flow of gases [6]. The glass transition temperature is the possible upper use temperature, if the material is lightly loaded.
3. The thermocouples were bonded to the composite substrate using General Electric RTV adhesive 157 (RTV) and aluminum tape. The RTV was applied in 0.635 cm. diameter drops along the thermocouple wires leaving the bead exposed. A typical instrumentation pattern on a specimen is shown in the photograph in Figure 4. The thermocouples were bonded to the cryogenic insulation surface side using drops of RTV only. The beads were left exposed for the type K wires, (See Figure 5). The type T thermocouples were inserted into the holes drilled into the cryogenic insulation. The embedded length of wire was measured with a caliper to ensure proper placement. A drop of RTV was placed at the point where the thermocouples exited the cryogenic insulation surface to fill the hole and prevent cryopumping and conduction along the length of the thermocouple wire.

**Figure 2.** Schematic of the thermocouple location (Dimensions are in cm.) and numbering (In italics) pattern for the tank wall (Inner) surface and bondline.

### 2.2.4 Coatings
The cryogenic insulation surface and sides were coated using polyurethane sealant. The coating served as a barrier to prevent absorption and expiration of the surrounding atmosphere by the cryogenic insulation. The polyurethane coating was applied liberally using a small acid brush. A second coat was applied 24 hours after the first application and allowed to dry 24 hours prior to testing. This procedure was followed for all of the specimens except the TEEK filled Korex™ honeycomb (With a gap). In this case, three coats of spray polyurethane were used. Each coat was allowed 24 hours to dry before the next coat was applied.

### 2.3 X-ray Non-Destructive Evaluation
X-ray Non-Destructive Evaluation (NDE) was conducted on all specimens before testing to ensure that the drilled holes were at the
correct angle and depth as specified in the schematics in Figure 3. The x-ray NDE also verified the location of the thermocouple wire.

**Upper Cryogenic Insulation (Exterior) Surface**

![Upper Cryogenic Insulation (Exterior) Surface Diagram]

**Figure 3.** Schematic of the thermocouple location (Dimensions are in cm.) and numbering (In italics) pattern for the upper cryogenic insulation (Exterior) surface of the specimen.

![Photograph of composite substrate tank wall (Inner) surface]

**Figure 4.** Photograph of composite substrate tank wall (Inner) surface.

### 2.4 Thermal Profile Test Rig

The specimen was mounted in the test rig shown in the photograph in Figure 6. A seal between the composite substrate and an insulated cryogenic chamber was obtained using one layer of 1.27 cm. wide Gortex\textsuperscript{TM} Teflon\textsuperscript{TM}
sealant gasket. A 3.81-cm.-wide perimeter clamp was hand-tightened to the overhanging top surface of the specimen composite substrate to hold the specimen against the gasket on the upper edge of the cryogenic chamber. The interior of the cryogenic chamber had a U-shaped spray bar, two resistive heaters, and two vent ducts. The exterior of the chamber was enclosed in coated Rohacell™ foam. A manual cryogenic/high temperature control system regulated the temperature of the heaters and flow of LHe into the chamber. A 25.4-cm.-tall shield (Not shown in Figure 6.) placed around the cryogenic insulation of the specimen limited the exposure of the surface thermocouples to evaporating Gaseous Helium (GHe) that surrounded the specimen during the test. One of the redundant Control Thermocouples (CTs) shown in Figure 2 was connected to the cryogenic/high temperature controller. The remaining thermocouples were connected to a data acquisition system.

Figure 5. Photograph of the upper cryogenic insulation (External) surface.

2.5 Temperature Profiles The temperature profiles used in the test are shown in the temperature versus time plot in Figure 7. These profiles were selected to simulate the minimum temperature a LH₂ propellant tank would experience during ground-hold before launch, to facilitate the onset of cryopumping, and simplify the test. A short duration cycle was used for the first nine cycles. A long dwell cycle was used for the tenth cycle to ensure that a steady state temperature was achieved. Before each cycle was initiated, the CT had to reach the Set-Point (SP) temperature of 21°C and the process thermocouple (TC30) at the center on the cryogen side of the composite substrate had to reach a steady state temperature.

2.5.1 Temperature Profile Descriptions Intially, the specimen was held at a SP of 21°C for 120 seconds. After this point, the SP was lowered to –267°C. When TC30 reached –253°C, the SP was raised in value (-250°C to –262°C) to control the flow of LHe while maintaining the desired –253°C reading at TC30. The specimen was held at the minimum cryogenic temperature for 600 seconds for the first nine cycles, and for 1800 seconds for the tenth cycle. After 600 seconds, the SP was raised to –101°C. When the CT reached
–101°C, the SP was raised to 4°C. When this SP was reached, the SP was raised to 21°C. The temperature was increased incrementally to reduce the chance of a temperature overshoot on the specimen above the heating zones (Near TC32 and TC28). When the CT reached 21°C, the specimen was held at this temperature for 600 seconds. The tenth cycle was exactly the same as first nine except for the 1800 seconds dwell.

3. RESULTS

3.1 Observations The polyimide cryogenic insulation surfaces absorbed the polyurethane coating when the specimen was being assembled. During cycle 2, the neat TEEK cryogenic insulation surface cracked along the length, parallel to the rig clamps. The crack was located on top of TC5. Then, during the 1800 second dwell in cycle 10, a second crack formed in the cryogenic insulation surface running perpendicular to the clamped edges (And the first crack). The crack extended from TC5 to TC8.

3.2 X-Ray NDE The x-ray photographs of thermocouples wires shown in Figures 8 and 9 are for the neat Airex™ and TEEK/Korex™ cryogenic insulations, respectively. The accuracy of the drill holes for the thermocouples was within ± 0.175 cm. of the target depth. The variances in depth are listed in Table 2. The x-ray photograph in Figure 9 shows that TC5 (Left most thermocouple in the TEEK/Korex™ panel) appeared to be located at the edge of a honeycomb cell.

3.3 Thermal Profile Data The results from the thermal profile testing are presented in the form of temperature versus time plots. Observations during the test and the corresponding points in the temperature versus time plots at three depths through-the-thickness of the cryogenic insulation will be described.
3.3.1 Near the Upper Surface of the Cryogenic Insulation  A plot of the temperature versus time measured from thermocouple TC5 for all nine specimens is shown in Figure 10. Thermocouple TC5 was located at an approximate depth of 0.424 cm. below the upper surface. All the specimens had an initial temperature reading of 21°C and upon cooling experienced a temperature change of approximately -96°C with the TEEK/Korex™ having the largest temperature change of -140°C. All of the specimens appeared to reach steady state temperature conditions during the 1800 second hold except the Airex™, TEEK/Korex™ and neat TEEK which continued to decrease in temperature.
throughout the 1800 second hold. Upon heating, the neat TEEK, TEEK/Nomex™, and TEEK/Korex™, the temperature dropped to –157°C at 2200 seconds. Thermocouple TC5 appeared to be located at the edge of a honeycomb cell possibly causing the thermocouple to monitor the temperature of the honeycomb cell wall or a heat short\(^4\) caused by the honeycomb cell wall.

### Table 2. X-ray NDE of the measured thermocouple depth and variance.

<table>
<thead>
<tr>
<th>Spec. No.</th>
<th>Specimen Cryogenic Insulation</th>
<th>Measured Depth from the Top of the Substrate, (cm.)</th>
<th>[±Variance in Thermocouple Depth], (cm.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Marcore™</td>
<td>0.424</td>
<td>0.846</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.270</td>
<td>1.694</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.118</td>
<td>2.118</td>
</tr>
<tr>
<td>2</td>
<td>TEEK/Korex™ (With a gap)</td>
<td>0.434</td>
<td>0.886</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.346</td>
<td>1.732</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.169</td>
<td>2.169</td>
</tr>
<tr>
<td>3</td>
<td>SF-38 (Spray foam)</td>
<td>0.427</td>
<td>0.838</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.250</td>
<td>1.669</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.057</td>
<td>2.057</td>
</tr>
<tr>
<td>4</td>
<td>BPCI/Nomex™</td>
<td>0.401</td>
<td>0.803</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.250</td>
<td>1.666</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.966</td>
<td>1.966</td>
</tr>
<tr>
<td>5</td>
<td>BPCI/TEEK/Nomex™</td>
<td>0.249</td>
<td>0.696</td>
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<td></td>
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<td>1.273</td>
<td>1.646</td>
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<td></td>
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<td>2.075</td>
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<tr>
<td>6</td>
<td>Divinylcell™</td>
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<td>0.889</td>
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<td></td>
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<td>1.341</td>
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<td></td>
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<td>2.136</td>
<td>2.136</td>
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<tr>
<td>7</td>
<td>Airex™</td>
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<td>0.777</td>
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<td>1.288</td>
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<td></td>
<td></td>
<td>2.134</td>
<td>2.134</td>
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<tr>
<td>8</td>
<td>Neat TEEK</td>
<td>0.439</td>
<td>0.831</td>
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<td></td>
<td></td>
<td>1.262</td>
<td>1.732</td>
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<td></td>
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<td>2.179</td>
<td>2.179</td>
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<td>9</td>
<td>TEEK/Nomex™</td>
<td>0.434</td>
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<td>1.374</td>
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<tr>
<td></td>
<td></td>
<td>2.136</td>
<td>2.136</td>
</tr>
</tbody>
</table>

BPCI – Boeing Polyurethane Cryogenic Insulation

**3.3.2 At the Mid-Plane of the Cryogenic Insulation** Thermocouple TC7 was at a depth of 1.270 cm. below the top surface. The temperature measurements from thermocouple TC7 for all nine specimens were compared in the plot in Figure 11. Thermocouples in each specimen were at an initial temperature of 21°C and cooled to a temperature ranging from –59°C to –140°C. X-ray NDE of TC7 location indicated a variation in thermocouple vertical height by as much as 0.104 cm. With such a small variation in thermocouple placement, the large temperature variation at cryogenic temperatures was not anticipated. Several of the cryogenic insulations reached steady state temperature conditions during the 1800 second hold with a nearly constant temperature obtained by the Boeing Polyurethane Cryogenic Insulation (BPCI)/Nomex™, Divinylcell™, BPCI/TEEK, and SF-38. When the cryogen flow was turned off, internal cooling occurs inside the neat TEEK, TEEK/Nomex™, and to a lesser extent on the TEEK/Korex™ at 2200 seconds.

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\(^4\) A heat short occurs when one material has a higher thermal conductivity than adjacent materials. The material with the higher thermal conductivity rises or falls in temperature at a faster rate than adjacent materials and possibly heats or cools adjacent material when heated by the same heat source.
3.3.3 Near the Composite Substrate of the Cryogenic Insulation  Temperature measurements from thermocouple TC9, which was at a depth of 2.118 cm. from the upper surface, were compared in the plot in Figure 12. Nearly all of the cryogenic insulation specimens achieved steady state temperature conditions during the 1800 second dwell. The minimum temperature that was indicated by these thermocouples was –157°C. When the cryogen flow was turned off, the interior of the neat TEEK and TEEK/Korex™ cooled to a minimum temperature of –184°C at 2200 seconds. In addition, the BPCI/TEEK/Nomex™ cryogenic insulation also slightly cooled internally with a change in temperature of 11°C.

![Figure 10. Comparison of TC5 thermocouple temperature measurement for a 1800 second dwell cycle (Cycle 10).](image)

4. DISCUSSION

4.1 Cryogenic Insulations  Neat TEEK had the lowest average density (0.415 g/cc) compared to all of the cryogenic insulations listed in Table 1, while TEEK/Korex™ had the highest average density (0.102 g/cc) due to the 0.034 cm. (3/16 in.) cell size of the Korex™ honeycomb core. The polyurethane cryogenic insulations had the highest closed cell content (Greater than 88%) in Table 1 but also had the lowest glass transition temperature of 85°C. The polyimide cryogenic insulations had the lowest closed cell content (Less then 23%) but had the highest glass transition temperature of 245°C. The polyimide cryogenic insulations have had higher values of closed cell content reported [11]. The low closed cell content can be attributed to possible manufacturing process defects. All of the cryogenic insulations had a similar thermal conductivity at an atmospheric pressure of 760 Torr except for the TEEK/Korex™, which had twice the
thermal conductivity of the other insulations. This high thermal conductivity was due to the small cell size and the high thermal conductivity of the Korex™ honeycomb core.

![Comparison of TC7 thermocouple temperature measurement for a 1800 second dwell cycle (Cycle 10).](image)

**Figure 11.** Comparison of TC7 thermocouple temperature measurement for a 1800 second dwell cycle (Cycle 10).

4.2 X-Ray NDE  The observations made in Table 2 documents the variance in the location of the thermocouples through-the-thickness of the cryogenic insulation. This provides insight into the less than consistent results obtained during the thermal profile testing. The variation in the depth of the thermocouples could lead to a temperature profile that is not consistent from specimen to specimen.

4.3 Coatings  Two coats of polyurethane were hand-brushed on all of the specimens except the TEEK/Korex™ with a gap, which was coated three times with aerosol polyurethane. Due to the nature of the application of these coatings, it was difficult to control the thickness of the polyurethane layer on each specimen. In fact, the polyurethane appeared to be absorbed into the neat TEEK, TEEK/Korex™, TEEK/Nomex™, and BPCI/TEEK/Nomex™ cryogenic insulation. The absorption of polyurethane in these specimens may be due to the low closed cell content of the TEEK cryogenic insulations. The coating may not have served as a sufficient barrier for the polyimide cryogenic insulations in the prevention of the absorption and expiration of the surrounding atmosphere at cryogenic temperatures.

4.4 Thermal Profile Data  The use of heaters in the cryogenic chamber resulted in the cryogenic insulations to be tested under severe conditions that represented a cryogenic propellant tank that was heated from the inside out. Thus, the plotted data in Figures 10 through 12 compare the worst-case scenario for the 1800 second hold on all nine specimens.
4.4.1 Polyimide Cryogenic Insulations  An indication of cryopumping occurred in the neat TEEK, TEEK/Korex™, and TEEK/Nomex™ cryogenic insulations when densified air at the interface of the composite substrate and the insulation quickly flashed back to a gas and caused a pressure differential which forced colder gases away from the substrate and toward the exterior of the cryogenic insulation. As the cold gas exited the cryogenic insulation, a temperature drop was detected in the lower levels of the cryogenic insulation of the specimen with interconnecting cells. The interconnecting cells in these cryogenic insulations allowed gas transfer. The level of interconnecting cells can be inferred by the degree of closed cell content of the foam and/or damage that occurred to the foam during thermal cycling. The temperature drop after the cryogen flow was turned off was more severe the further the thermocouple was away from the substrate.

![Graph](image.png)

**Figure 12. Comparison of TC9 thermocouple temperature measurement for a 1800 second dwell cycle (Cycle 10).**

4.4.1.1 Damage  An investigation of the neat TEEK cryogenic insulation upon completion of the tests indicated that a crack had formed on the top surface, however, no de-bonding had occurred. The procedure utilized to manufacture the foam was determined to be outside of the documented procedure and may have contributed to the failure.

4.4.1.2 Cryopumping in the TEEK/Korex™ Cryogenic Insulation  The TEEK/Korex™ had thermocouples near the cell walls as shown by x-ray NDE in Figure 9. The heat shorts caused by this contact may have caused the low measured temperature displayed in the three plots in Figures 10 through 12. The Korex™ had a small honeycomb cell size of 0.034 cm. (3/16 in.), a high thermal conductivity, and was permeable [7]. These three factors, plus the thermocouples contacting the core made the TEEK/Korex™ behave like a poor insulator in these tests. The permeability of the Korex™ core, possibly
contributed to cryopumping by wicking gases from the atmosphere to the substrate surface and allowing mass flow between cells.

4.4.1.3 Cryopumping in the TEEK/Nomex™ Cryogenic Insulation  The 0.068 cm. (3/8 in.) cell honeycomb in the TEEK/Nomex™ cryogenic insulation increased the thermal efficiency by restricting the mass flow of cooled gases throughout the plane of the foam and from the substrate by channeling gases through an individual honeycomb cell. The honeycomb cells in effect, permitted the neat foam portion to act as efficient insulator even though the TEEK foam cryopumped gases from the atmosphere to the substrate.

4.4.2 Polyurethane Cryogenic Insulations  Based upon the data reported for the 1800 second dwell, the SF-38, BPCI/Nomex™, and BPCI/TEEK/Nomex™ were more thermally efficient insulators than the other cryogenic insulations tested under similar conditions. This was attributed mainly to the high closed cell content of these cryogenic insulations and low thermal conductivity. The BPCI in the BPCI/TEEK/Nomex™ hybrid minimized cryopumping at the substrate even though the TEEK/Nomex™ portion had low closed cell content. The higher operating temperature of the TEEK/Nomex™ would enable the external surface to be exposed to higher temperatures while the BPCI/Nomex™ cryogenic insulation at the substrate would limit cryopumping. The remaining cryogenic insulations Marcore™, Divinycell™, and Airex™ did not cryopump but were adequate insulators.

5. CONCLUDING REMARKS

A new thermal profile test rig and test methods were developed to investigate cryopumping in candidate cryogenic insulations for a Reusable Launch Vehicle (RLV) cryogenic propellant tank applications. The test methods were simple to use and determined whether a candidate cryogenic insulation would cryopump. The occurrence of cryopumping was related to measured temperature changes through-the-thickness of the cryogenic insulation. Nine coated cryogenic insulations were tested in the test rig to Liquid Helium (LHe) temperatures for ten cycles with the tenth cycle containing a 1800 second dwell at LHe temperatures.

As evidenced from the results shown in the temperature versus time plots, there was a noticeable variation in the minimum temperature achieved by each insulation system. However, the variation could be attributed to the difference in thermal conductivity and closed cell content of the cryogenic insulations. All of the cryogenic insulations had no visible degradation during the ten cycles except for the neat TEEK, which cracked upon cooling to LHe temperatures. The crack was possibly due to manufacturing defects in the foam. The three polyurethane cryogenic insulations SF-38, Boeing Polyurethane Cryogenic Insulation (BPCI)/TEEK/Nomex™, and BPCI/Nomex™ were more thermally efficient insulators than the other cryogenic insulations and did not cryopump. Marcore™, Divinycell™, and Airex™ did not cryopump. The polyimide cryogenic insulations neat TEEK, TEEK/Korex™, and TEEK/Nomex™ exhibited evidence of
cryopumping. Manufacturing defects may have caused the low closed cell content. The cryopumping and lower temperatures in the TEEK/Korex™ may have been caused by the thermocouple placement on the honeycomb cell wall or by the Korex™ core. The results supports that small cell honeycomb core should not be used in foam/honeycomb core cryogenic insulations. The polyurethane coating applied to all of the cryogenic insulations as a sealant, may not have benefited the TEEK insulations since the foam absorbed the coating.

6. REFERENCES