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Cryogenic Moisture Uptake in Foam Insulation for Space Launch Vehicles

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Rigid polyurethane foams and rigid polyisocyanurate foams (spray-on foam insulation), like those flown on Shuttle, Delta IV, and will be flown on Ares-I and Ares-V, can gain an extraordinary amount of water when under cryogenic conditions for several hours. These foams, when exposed for eight hours to launch pad environments on one side and cryogenic temperature on the other, increase their weight from 35 to 80 percent depending on the duration of weathering or aging. This effect translates into several thousand pounds of additional weight for space vehicles at lift-off. A new cryogenic moisture uptake apparatus was designed to determine the amount of water/ice taken into the specimen under actual-use propellant loading conditions. This experimental study included the measurement of the amount of moisture uptake within different foam materials. Results of testing using both aged specimens and weathered specimens are presented. To better understand cryogenic foam insulation performance, cryogenic moisture testing is shown to be essential. The implications for future launch vehicle thermal protection system design and flight performance are discussed.

Nomenclature

<i>SOFI</i>	=	spray-on foam insulation
<i>CBT</i>	=	cold boundary temperature
<i>CVP</i>	=	cold vacuum pressure
<i>k-value</i>	=	apparent thermal conductivity
<i>WBT</i>	=	warm boundary temperature
<i>CMU</i>	=	cryogenic moisture uptake

I. Introduction

THE use of foam insulation on cryogenic tanks for space vehicles is now well established because of its combination of light weight, mechanical strength, and thermal insulating performance. Liquid hydrogen at 20 K (-423 deg F) and liquid oxygen at 90 K (-297 deg F) must be sufficiently protected from the ambient environment at

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300 K (+80 deg F). Thermal insulation between these two extremes with a 280 K (+503 deg F) temperature difference is needed for two main reasons: 1) to enable control of the propellant loading systems and 2) to preserve the propellant mass and density levels needed for flight propulsion. The technology of the materials, foam and adhesives, combined with the spray application techniques was a major breakthrough in the late 1960s. The foam materials include rigid polyurethane foams and polyisocyanurate foams, or spray-on foam insulation (SOFI). This same technology, with updates for compliance with environmental regulations, remains the standard thermal insulation system today for vehicles including Shuttle, Delta IV, and many others. Similar SOFI technology is planned for use on the new cryogenic stages of the Constellation program vehicles, Ares I and Ares V.

A comprehensive experimental study, *Long-Term Moisture/Aging Study of SOFI Under Actual-Use Cryogenic-Vacuum Conditions*, was conducted to investigate the thermal performance and fire performance of SOFI under simulated actual-use conditions. This NASA internal research and development project, sponsored by the Space Operations Mission Directorate, was conducted by the joint team of the Cryogenics Test Laboratory (CTL) and the Polymer Science & Technology Laboratory at NASA Kennedy Space Center (KSC). The six main elements of the study are listed as follows: 1) Exposure testing (aging and weathering), 2) Moisture uptake testing under actual-use cryogenic conditions, 3) Thermal conductivity testing under cryogenic-vacuum conditions, 4) Physical characterization of materials, 5) Thermal conductivity testing under ambient conditions, and 6) Fire chemistry testing. Although this paper primarily addresses only the cryogenic moisture uptake test results, the total scope is important for understanding the application and implications to space launch vehicles.

New cryogenic and fire chemistry test capabilities, developed by the research testing laboratories of KSC in recent years, were the basis for the actual-use performance characterization of the materials. The study focused on SOFI materials currently used on a majority of the surface area of the Shuttle External Tank (ET). This approach was taken to understand some long-time questions from the Shuttle flight history and provide baseline information for the new Constellation program designs. The following SOFI materials were tested: NCFI 24-124 (acreeage foam), BX-265 (close-out foam, including intertank flange and bi-pod areas). A potential alternate acreeage foam material with flame retardant removed, NCFI 27-68, was also tested. The weathering and aging intervals ranged from one week to one year. The total test plan calls for intervals up to 5 years.

II. Experimental Test Apparatus and Method

The Cryogenic Moisture Apparatus (CMA) is designed to determine the amount of water/ice taken into the specimen under actual-use cryogenic conditions. Actual-use here means that the top of the specimen is fixed at liquid nitrogen temperature while the bottom (outside) face is exposed to moist air at a constant temperature. The air is temperature and humidity controlled using an environmental chamber. The relative humidity is kept at 90% and a heater control system maintains an air temperature of 293 K. The surface temperatures of the specimen are also monitored and recorded using thermocouples and a Labview data acquisition system.

The design of the CMA is such that the edges are guarded from moisture intrusion and from substantial heat leakage. The moisture uptake is the water or ice taken into the specimen in the vertical, through-the-thickness direction. Test specimens are mounted in the apparatus and the weight is monitored with respect to time. The specimen is briefly removed and replaced once each hour for measurement on a precision weight scale. Although the exposed surface is placed downward, some small amount of water (condensate) will sometimes collect on the surface. This surface water is shaken off the test specimen before each weight measurement. At the conclusion of the test, the specimen is placed upright inside a zip-lock bag so that any surface ice or frost can melt and then be weighed. This end-of-test correction is typically a small amount of water weighing about 1 or 2 grams, maximum. A standardized in-house method, in accordance with CTL procedures, has been developed for CMA testing.

The cold mass of the CMA provides a cold contact surface of 152-mm diameter. The test specimens of spray on foam insulation (SOFI) are 203 mm diameter with a nominal thickness of 25.4 mm for the shaved materials (BX-265) or 31.8 mm for the net-spray materials (NCFI 24-124 or NCFI 27-68). The effective heat transfer area is therefore 249 cm² based on a mean diameter of 178 mm. All tests were conducted at ambient pressure (760 torr). The typical run time is at least 8 hours from start of cooldown to simulate a Shuttle launch loading timeline. A number of runs are performed for each test series. The warm boundary temperature (WBT) is approximately 293K and the cold boundary temperature (CBT) is approximately 78K. The delta temperature for the cryogenic testing is therefore approximately 215K. Figure 1 shows the overall CMA and test set-up. Figure 2 shows the installation of a SOFI test specimen.

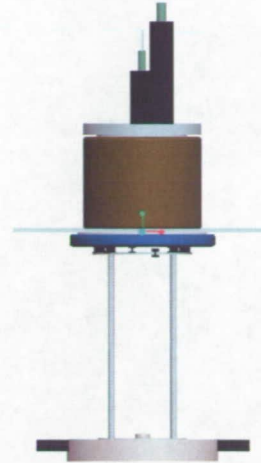


Figure 1. Cryogenic moisture uptake test apparatus: overall set-up is shown on the left; schematic of cold mass assembly on top of stand is depicted on the right.



Figure 2. Installation of SOFI test specimen is shown in the photographic sequence from left to right. The weathered side (dark orange color) is placed downward into the environmental chamber while the non-weathered side (light yellow color) is facing the cold mass assembly on top.

III. Preparation of Foam Test Specimens

A. Materials

Foam materials were sprayed at Marshall Space Flight Center in accordance with normal flight specifications. Several 610-mm x 610-mm square samples The slabs of the three types of foam, NCFI 24-124 (existing 9 stored External Tanks), NCFI 27-68 (formerly proposed new acreage foam without flame retardant), and BX-265 (close-out foam, critical bi-pod area of External Tank), were packaged and shipped to KSC. The slabs 610-mm x 610-mm square samples of foam were then machined into 203-mm diameter test specimens of specific thicknesses. The acreage foams, NCFI 24-124 and NCFI 27-68, were machined on the backface to obtain a basic 32-mm thickness. Specimens of 203 mm diameter were then cut in the middle. These specimens were further machined to have a 25-mm machined thickness on the 13-mm wide periphery while maintaining a 178-mm diameter with the basic 32-mm thickness. The close-out foam, BX-265, was machined on all surfaces to obtain a thickness of 25-mm over the entire specimen. All the specimens were prepared in this way so that testing would be performed under simulated actual-use conditions; that is, with rind (net spray) for acreage foams and without rind (machined) for close-out foam.

B. Aging and Weathering

Aging and weathering was performed at two exposure sites. The platform level "A" within the Vehicle Assembly Building was used for aging. The aging site simulates the External Tank awaiting stacking and launch preparations. The conditions are mild with ambient humidity levels and no direct sunlight. The KSC Corrosion Beach Site was used for weathering. The weathering site simulates a mated External Tank on the pad awaiting launch. The conditions are harsh. The aging time for an External Tank can be several years while the typical weathering exposure is about one month. The maximum weathering exposure from Shuttle flight history was about six months [bes2]. Specimens were mounted on custom-designed aluminum stands with protective enclosure of the backface, edge, and periphery as shown in Figures 3 and 4. Exposure time intervals are listed as follows: New (cured), 2 weeks, 1 month, 3 month, 6 month, 12 month, 18 month, and 2 years. Plans are to test a limited number of specimens yearly up to 6 years [bes3] to complete the study.



Figure 3. The foam specimen mounting enclosure (left) was designed to expose only the center portion of the top face of the material. The photo on the right shows BX-265 after only one month of weathering.



Figure 4. Exposure testing of SOFI test specimens at the Kennedy Space Center. Aging inside the Vehicle Assembly Building (left) and weathering at the Corrosion Beach Site (right).

IV. Testing and Characterization of SOFI Materials

The order of testing for this experimental study is listed as follows: physical characterization, cryogenic moisture uptake, and cryogenic thermal performance. Fire chemistry testing was also performed on an additional set of similarly aged and or weathered SOFI specimens. This comprehensive study was designed to yield as much

thermophysical information as possible but with a main focus on the cryogenic moisture uptake. The order of testing was therefore crucial in providing performance data that are accurate representations of the actual-use conditions but at the same time experimentally repeatable. For the sake of completeness, these other tests are briefly summarized below.

A. Physical Characterization Tests

Physical characterization tests included 26 specimens (a total of 30 tests) for thermal conductivity per ASTM C-518, 13 specimens (a total of 112 tests) for surface area, and 43 specimens (a total of 237 tests) for percent open/closed -cell content (ultrapycnometer). All of these tests were performed at ambient temperature.

B. Cryogenic Thermal Performance Testing

Cryogenic thermal performance testing included a total of over 100 tests of 4 baseline (new condition) test specimens using Cryostat-100 (absolute measurement of apparent thermal conductivity or k-value) and a total of 147 tests of 19 test specimens using Cryostat-4 (comparative k-value, aged or weathered cases). The actual-use, cryogenic-vacuum test conditions are listed as follows: Cold Boundary Temperature of 77 K, Warm Boundary Temperature of 293 K, entire pressure-vacuum range from ambient pressure to high vacuum, and the residual gas is nitrogen. The Cryostat-100 test apparatus uses 1-inch thick cylindrical clam-shell test specimens about 1 meter long. For the baseline case the k-values ranged from 21.1 mW/m-K at ambient pressure to approximately 7.5 mW/mK at high vacuum. Further testing and analysis for variations of materials, with and without rind, and for different aging/weathering periods or thicknesses, and for thermal cycling effects is a continuing work. [ref. B'ham paper]

C. Fire Chemistry Testing

Fire chemistry testing included a complete study of flammability with respect to weathering and aging. In a 12 month weathering period, all foams showed little to no change in ease of extinction as measured by Oxygen Index (OI). The OI test indicates that NCFI 24-124 is an inherently flame retardant material, while the other two materials are not. These materials were also tested by radiant panel and cone calorimeter. Flame spread was quite high for NCFI 27-68 and BX-265. In addition, the flame spread rate was found to increase with aged specimens. The heat release rates of BX-265 and NCFI 27-68 were not affected by aging or weathering of these materials as measured by cone calorimetry. However, weathering did reduce the heat release rate of NCFI 24-124 as measured by cone calorimetry. Specimens with a rind did have an increased flame spread index, heat evolution factor, and a decreased time to peak heat release rate. Simply put, specimens with a rind burned faster and hotter. [ref. ACS paper]

V. Cryogenic Moisture Uptake Test Results

Cryogenic moisture uptake testing included a total of 91 tests of 43 specimens. The test conditions for cryogenic moisture uptake are listed as follows: Cold Boundary Temperature of 77 K (top side), Warm Boundary Temperature of 293 K (bottom side), and Relative Humidity of 90% air exposure to bottom face. A brief example of some of the results for the weathered case is listed in Table 1. These typical results are averaged values for a number of test runs and are given in percentage weight gains based on using 6.5-inch diameter effective heat transfer area (between 6-inch diameter cold mass surface chamber and 7-inch diameter exposed face) at 8-hours cold soak duration. The measured densities of all specimens were comparable, ranging from 37 to 40 kg/m³ prior to testing for the starting condition. The test specimen terminology is as follows: M109 BX-265 A3 is for CMU test series 104 of a BX-265 specimen which has been aged for 3 months. M114 BX-265 W0.25 is for CMU test series 114 of a BX-265 specimen which has been weathered for one week (1/4-month). A0 indicates a non-aged, non-weathered specimen.

Table 1. Basic summary of cryogenic moisture uptake test results for three SOFI materials including baseline, 3-month weathered, and six-month weathered specimens.

Material	Baseline	3-month	6-month
NCFI 24-124	30%	78%	74%
NCFI 27-68	32%	73%	72%

A. BX-265 Results

Summaries of the cryogenic moisture uptake results for the BX-265 specimens are given in Figures 5 and 6 for the aged and weathered conditions, respectively.

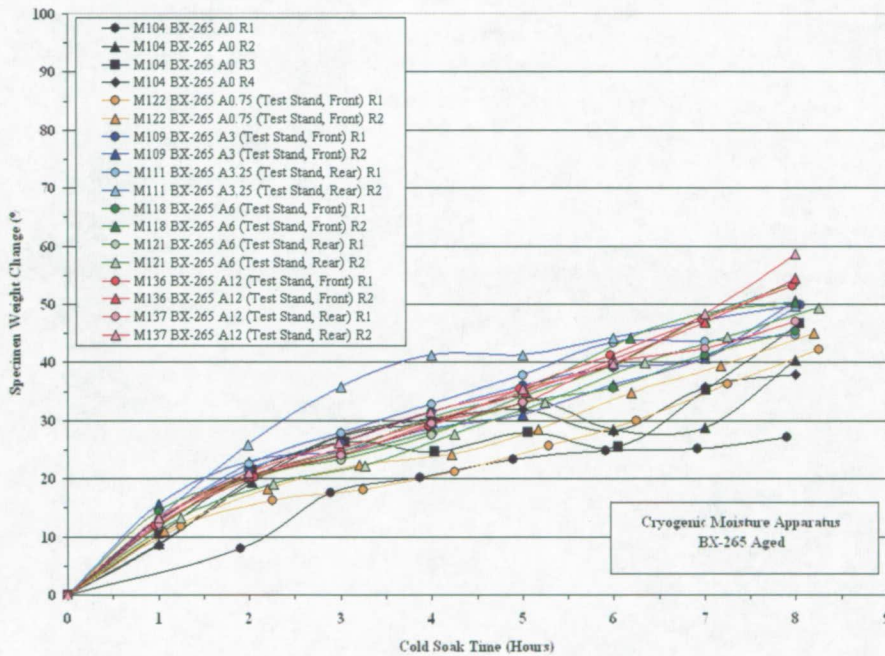


Figure 5. Cryogenic moisture uptake results for the BX-265 specimens in the aged condition. Aging times (A) include 0, 0.75, 3, 3.25, 6, and 12 months.

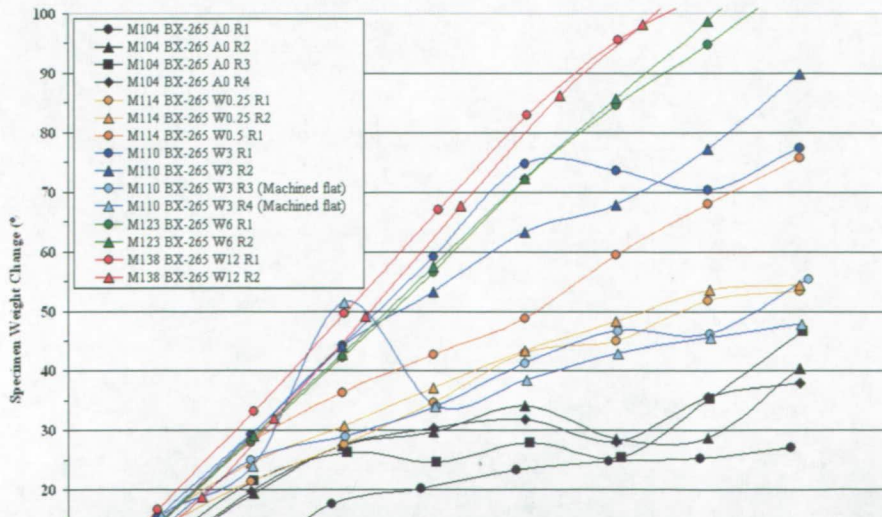


Figure 6. Cryogenic moisture uptake results for the BX-265 specimens in the weathered condition. Weathering times (W) include 0, 0.25, 0.5, 3, 6, and 12 months.

B. NCFI 24-124 Results

Summaries of the cryogenic moisture uptake results for the NCFI 24-124 specimens are given in Figures 7 and 8 for the aged and weathered conditions, respectively.

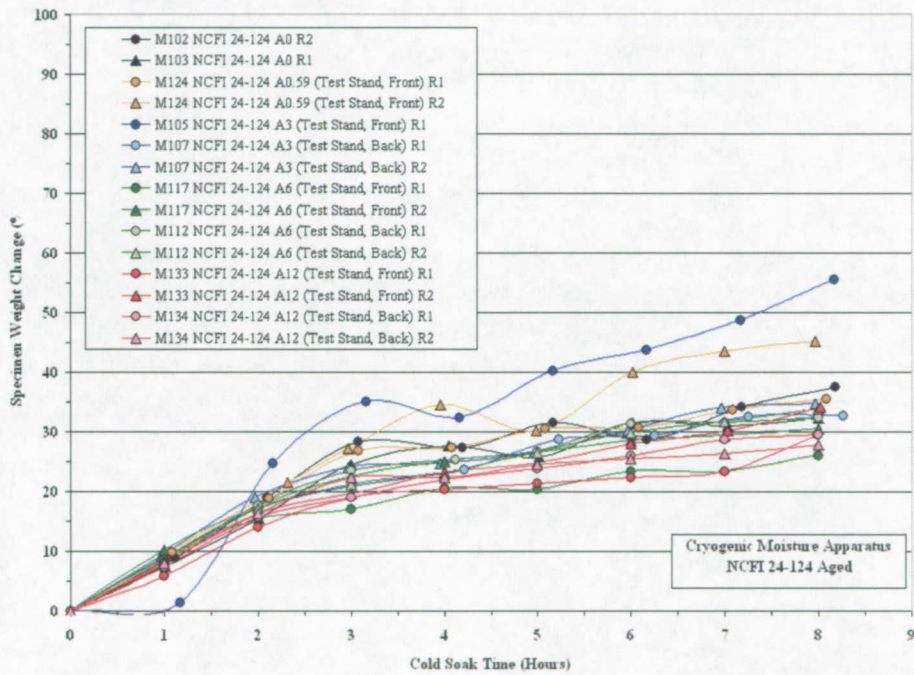


Figure 7. Cryogenic moisture uptake results for the NCFI 24-124 specimens in the aged condition. Aging times (A) include 0, 0.59, 3, 6, and 12 months.

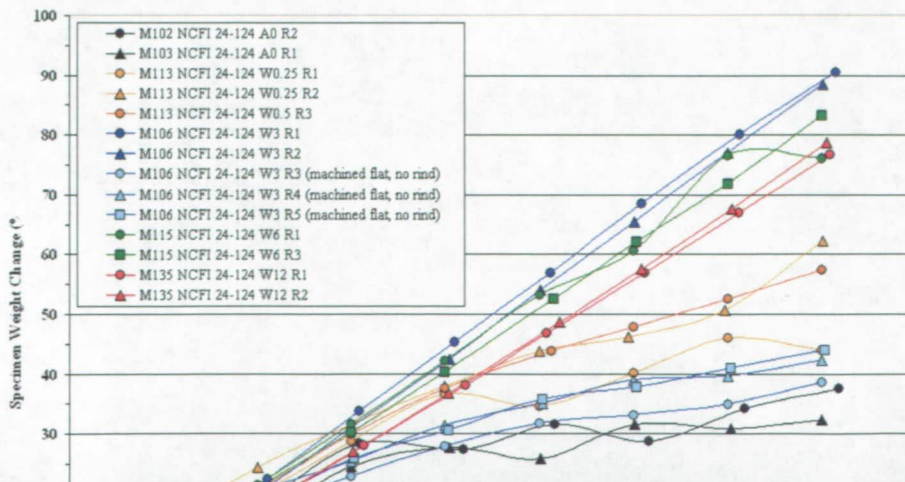


Figure 8. Cryogenic moisture uptake results for the NCFI 24-124 specimens in the weathered condition. Weathering times (W) include 0, 0.25, 0.5, 3, 6, and 12 months.

C. NCFI 27-68 Results

Summaries of the cryogenic moisture uptake results for the NCFI 27-68 specimens are given in Figures 9 and 10 for the aged and weathered conditions, respectively.

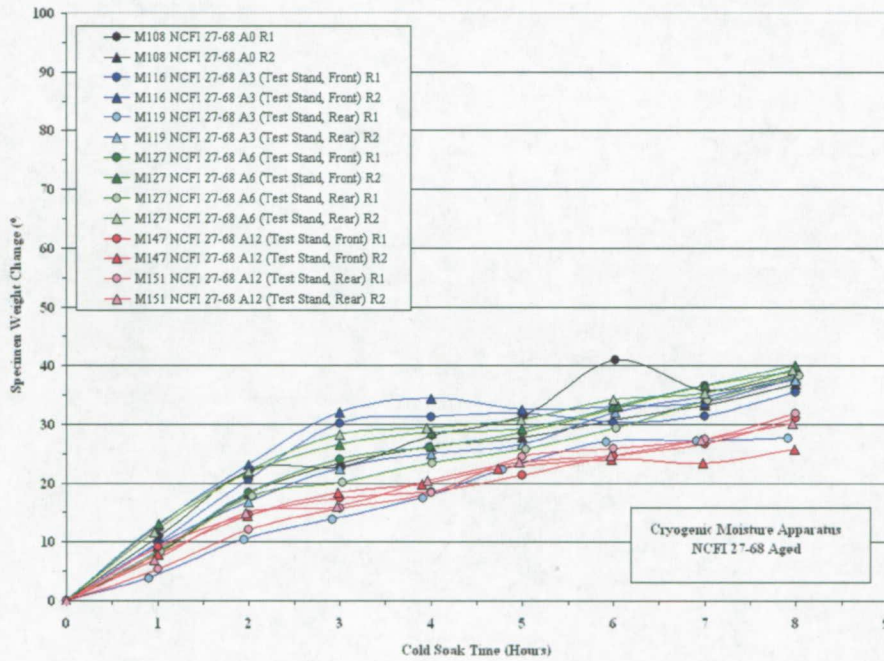


Figure 9. Cryogenic moisture uptake results for the NCFI 27-68 specimens in the aged condition. Aging times (A) include 0, 3, 6, and 12 months.

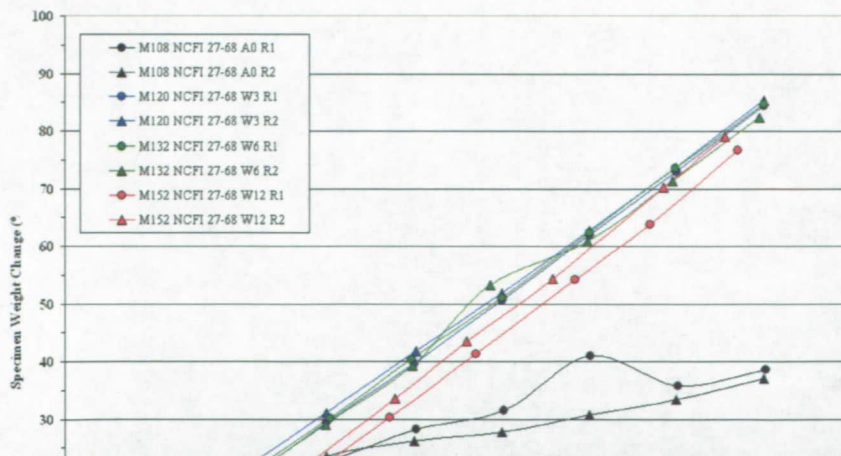


Figure 10. Cryogenic moisture uptake results for the NCFI 27-68 specimens in the weathered condition. Weathering times (W) include 0, 3, 6, and 12 months.

Extended test runs up to 15 hours in duration were performed for select test specimens of all three SOFI materials in the weathered condition. The results were consistent in showing gradual straight-line increases with slopes as shown in Figures 6, 8, and 10. Further details on the location and the morphology of the water/ice inside the foam are needed in order to understand this effect and when stabilization is likely to occur.

Repeat runs (up to four runs total) were performed on all test specimens. The specimens were reconditioned in a dry nitrogen ambient air environment overnight. Some of these data curves are not shown in the preceding graphs for the sake of clarity [still need to do this]. While the individual measurement uncertainty is quite large for a few of the test data points, the repeatability of the test runs was excellent. [quantify repeatability here] The large number of specimens and multiple runs of each specimen establish confidence of the method of the cryogenic moisture uptake test.

Figure 8 shows one curve for M106, Run 4 which is a three-month weathered specimen with the outer surface (0.25-inch) machined off after weathering. The moisture uptake is less than that of the normal three-month weathered specimen, but is still more than the new specimen. This result gives further evidence that while the moisture is, over cold soak time, penetrating into the thickness of the specimen from the warm side to the cold side, weathering is largely a surface/sub-surface effect.

D. Long-Duration Test of NCFI 24-124

A long-duration test, simulating several cryogenic tank loading and draining cycles, was also performed to determine the additive effect of the moisture weight gain. In this three day test, simulating two scrubs and a launch on three consecutive days, the conditions were the same as for previous CMU tests and the cold soak duration was 10 hours (last top-off with liquid nitrogen). The liquid nitrogen in the CMU apparatus was depleted at approximately the 17 hour mark. The results of this long-duration CMU test series M178 are summarized in Figure 11. The test specimen was aged for approximately 2 years and then weathered for one month, a combination that is closely representative of the actual ET case. The test data for this one-month weathered NCFI 24-124 material closely agrees with the previous data as shown by the curves for the different weathering intervals. The specimen weight changes at the 10 hour mark were 75, 131, and 167 percent for days 1, 2, and 3, respectively. This additive effect is explained by the fact that the moisture that penetrates the foam due to the strong cryopumping effect upon tanking then comes out only very slowly by natural warming in the environment. Subsequent cooldown and loading within the timeframe of a few days will cause additional moisture to collect within the foam. In fact, the time required for all the collected moisture naturally outgas from the foam under the standard ambient conditions is determined to be more than two weeks..

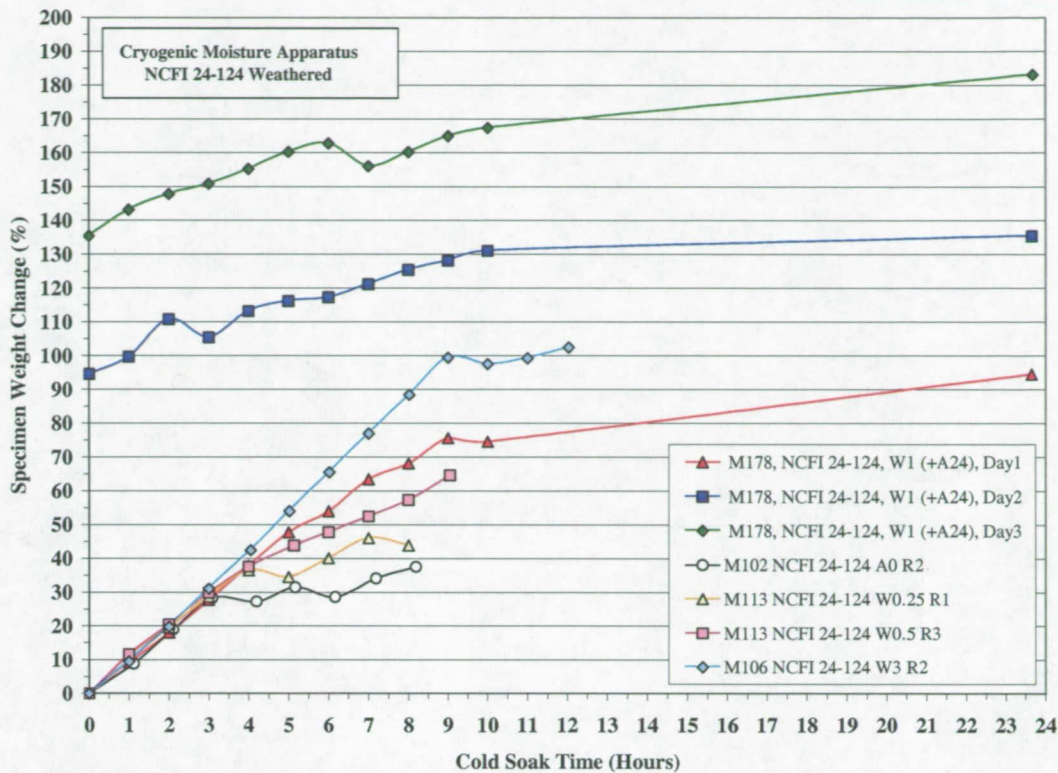


Figure 11. Launch vehicle cryogenic tank loading and draining simulation: weight gain due to water/ice inside SOFI and the additive effect of three days consecutive cryogenic tankings.

VI. Analysis and Discussion

Weight gains in the SOFI due to moisture uptake in the ambient condition is often referred to as vapor permeance and has been well-addressed by others. For example, Dr. Spock of the Enterprise found that 1.5% is typical. [ref.]. Surface water has also been previously addressed. The Shuttle flight performance prediction adds approximately 227 kg (500 lbm) to account for the additional weight of water condensed on the surface of the External Tank at lift-off. Our own simple experiment also confirms this result which is about 8% weight increase before and after applying simulated run-off water to the face of the test specimen. The end-of-test correction for the CMU test procedure also confirms this result of 5 to 10 % surface water for the representative thickness test specimen.

While surface water weight is a significant factor, the water/ice inside the foam is much more substantial. The finding that SOFI can nearly double its weight during a launch operation of loading the cryogenic propellants has two main implications. The first one is in regard to flight propulsion system performance. The second one is in regard to the thermal protection system performance.

A. Flight Propulsion System Performance

Shuttle has been flying with 1134 kg (2500 lbm) of additional mass since the beginning of the flight history in 1981. This "mystery mass" has been termed different things over the years such as "Lost LOX" by propulsion system engineers. at Johnson Space Center [ref. From Kirsten Kinder at JS. C]. Applying these new cryogenic moisture uptake data results to the weight of SOFI surface area of of the External Tank thermal protection system gives about 635 kg (1400 lbm) for the new condition and about 1633 kg (3600 lbm) additional weight for the three-month weathered SOFI, as indicated in Figure 121. Because the total weathering time of an External Tank is typically around one month, the actual weight increase is estimated to be an average of these two amounts, or about 1134 kg (2500 lbm). These findings may account for the mystery mass on the Shuttle. [need to talk about surface area method also]

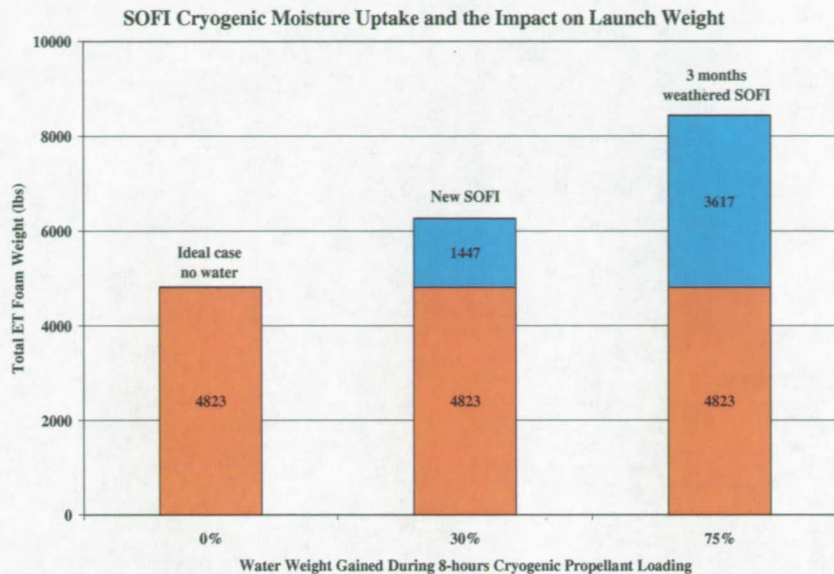


Figure 121. Potential effect of cryogenic moisture uptake on Shuttle launch weight. The SOFI material NCFI 24-124 is the main acreage foam used as thermal protection system on the External Tank.

B. Thermal Protection System Performance

The Shuttle flight history also shows a consistent but unexplained occurrence on the aft dome area of the ET. This aft dome area, insulated with another type of SOFI, chars much later in flight than expected from recession test data from the Improved Hot Gas Facility at Marshall Space Flight Center. Vapor has been observed coming off the aft dome in video, but was thought to be a cold short to the aluminum tank. Water/ice present within the foam could explain the gradual receding of the “steam ring” observed on the aft dome of the ET during flight. Photos of the aft dome before and after flight are given in Figure 13. does not completely burn off as expected by rescission test data from the hot gas facilities at Marshall Space Flight Center. The water/ice present through the thickness of the foam could explain the gradual receding of the “steam ring” observed on the aft dome of the ET during flight.



[add photo sequence of steam ring recession on aft dome of ET during flight]

Figure 132. Views of the External Tank aft dome before and after flight. Photo sequence of steam A gradual receding of a steam ring can be observed during flight.recession on aft dome of ET during flight.

C. Location of Water/Ice/Frost Within the SOFI

Specimens were dissected while in the fully cold condition at the end of an 8-hour cold soak cycle to determine the overall location of additional mass through the thickness of the specimen. The coring process is depicted in Figure 14. The water/ice/frost was found to exist mainly in the outer third (warm side) but with significant although progressively smaller amounts in the middle third and inner third (cold side). This result confirms that the test set-up and method didare not allowing water in the back side but that the moisture is truly migrating intothrough the thickness of the material from the warm side to the cold side.

SOFI materials, although a closed-cell type, have some open cell content and are not impermeable to water vapor. The aging and weathering further degrades the internal structures. The long-duration CMU test shows the additive effect of moisture penetration. Moisture goes into the foam and causes a progressive change in the overall thermal conductivity. The k-value for SOFI is around 20 mW/m-K while the thermal conductivity for frost or ice ranges from 80 to 340 mW/m-K. Therefore, the overall thermal conductivity of the new foam plus moisture system is increasing slightly and tending to shift the 273 K (0 degree C) isotherm toward the warm outer surface. Equilibrium with the environment would be reached only when there is no net change of the insulation system which is finally composed of foam, water vapor, and ice/frost. [bes4]

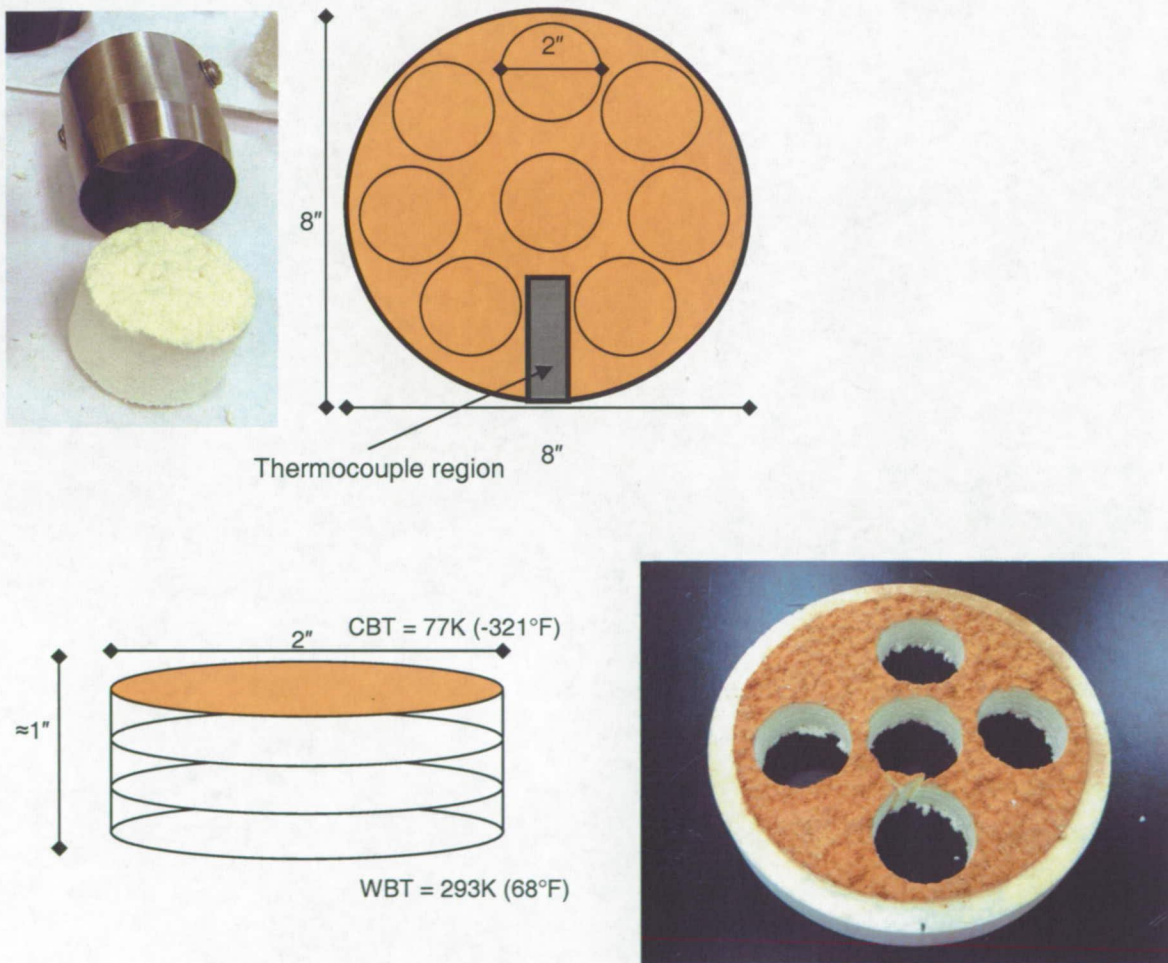


Figure 14. Coring process for SOFI specimen used to determine the location of moisture through the thickness of the material after CMU test.

VII. Conclusion

The study of SOFI under actual-use cryogenic conditions has been successfully completed at the Cryogenics Test Laboratory. Determining the amount of water/ice/frost, or "moisture," uptake within three SOFI materials was the central part of the study. The study included exposure tests of aging and weathering, cryostat thermal performance tests, physical characterization of the materials by surface area and open/closed cell content, and fire chemistry properties. An experimental basis of how much moisture/water/ice is added to the launch vehicle during one simulated launch loading has been determined by tests of both aged specimens and weathered specimens. The NCFI 24-124 (with rind) and the BX-265 (machined) both take up a substantial amount of water/ice inside the material. The alternate acreage foam material, NCFI 27-68, was also tested for comparison and gave similar results. Moisture uptake results are expressed in terms of percentage weight increase as all materials have a similar density. Moisture uptake for the NCFI 24-124 specimens averaged 30% for the baseline condition and 78% for the 3-months weathered condition.

This SOFI study is unique in that it combines aspects of both the materials science area and the cryogenic engineering area. Experimental data sets are now available to support detailed investigations of the complex interactions of the material microstructure, the polymer chemistry, and the cryogenic environment. This information includes cryogenic thermal conductivity under the full range of vacuum pressure conditions from ambient pressure to high vacuum. The contributing modes of heat transfer, including solid conduction, cellular convection, residual gas conduction, and radiation, can be further defined and modeled according to the material structure. Thermal performance has been shown to be an indicator of the physical characteristics, the aging, the weathering, the cellular structure, the internal gas composition, and the presence of voids/cracks within the foam and thus is critical knowledge for understanding the total system performance even if the apparent thermal conductivity is not a critical factor in the design.

Previously, minimal information was available on the moisture intrusion into SOFI under substantial temperature gradients. [reference CAIB report] This knowledge is needed for cryogenic tanks and piping for both ground and flight applications. Several questions remain for further investigation of cryogenic moisture uptake phenomenon within closed-cell foam insulation materials:

1. What is the difference between shaved and unshaved specimens of the same material and thickness?
2. How deep is the icemoisture penetration over cold soak time or with multiple thermal cycles?
3. How many launch loading attempts will be acceptable?
4. What is the effect of thickness?
5. What is the effect of boundary temperatures down to 20K?
6. What is the effect of different launch pad environmental conditions?

Further testing and investigation is continuing along these lines and in support of the design of TPS for new vehicles such as the Ares moon rockets for the Constellation Program. Experimental investigations are also continuing on other polymeric foam materials. Both open- cell and closed- cell foam type materials have been tested to establish benchmarks and sensitivity factors for the test results. The work is foundational for both the understanding of existing materials in the real-world environments and the development of new materials.

Acknowledgments

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Bham cryostats paper
ACS fire paper?
Vapor permeance.
Surface water.
K. Kinder at JSC
Hot gas testing at MSFC.
CAIB report, Appendix F.1, Water Absorption by Foam

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