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OUTGASSING OF SOLID MATERIAL INTO VACUUM
THERMAL INSULATION SPACES

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ABSTRACT

Many cryogenic storage tanks use vacuum between inner and outer tank for thermal insulation. These cryogenic tanks also use a radiation shield barrier in the vacuum space to prevent radiation heat transfer. This shield is usually constructed by using multiple wraps of aluminized mylar and glass paper as inserts. For obtaining maximum thermal performance, a good vacuum level must be maintained with the insulation system. It has been found that over a period of time solid insulation materials will vaporize into the vacuum space and the vacuum will degrade. In order to determine the degradation of vacuum, the rate of outgassing of the insulation materials must be determined.

Outgassing rate of several insulation materials obtained from literature search were listed in tabular form.

SUMMARY

1. Outgassing rate of different insulation materials for cryogenic tanks are listed in Tabular form. (All data obtained through literature search)
2. Pressure increased in vacuum space of a tank during a five year period is provided.
3. An example outgassing problem is worked.

1. INTRODUCTION

Many cryogenic storage vessels use inner and outer tanks with a vacuum pumped between the tanks to prevent convection heat transfer from the wall of the outer tank to the wall of inner tank. Most cryogenic tanks also use a multilayer insulation in the vacuum space to protect from radiation heat transfer. Multilayer insulation is used extensively in applications where high thermal resistance is required, such as the insulation of cryogenic tanks. Multilayer insulation consists of many thermal radiation shields arranged in series, usually interleaved with a low conductance spacer material to reduce thermal conduction between shields. To achieve maximum thermal resistance a great many shields in series are required. Many of the shields are made by vacuum depositing of aluminum on one or both sides of mylar substrates with nominal thickness in the range 0.25 - 2.0 mils. A spacer material is used between the reflective aluminum mylar shields, commonly made of thin glass fiber. Because of the many layers required, the total surface area can be extremely high, making the multilayer insulation a significant outgas source. Multilayer insulation outgassing can affect overall system performance. It has been found that over a period of time multilayer materials and the tank material will vaporize into a vacuum space in the form of hydrogen. Newer tanks use palladium monoxide to convert the hydrogen to water which may be absorbed as a getter. Older tanks do not have a method of removing the out gassed hydrogen and the vacuum degrade with time. In order to estimate the vacuum level on old tanks without vacuum gages, outgassing rate of the insulation materials and the tank material must be determined.

2. OBJECTIVE AND TASKS OF PROJECT

2.1 Objective

The objective of this task was to make literature searches into vacuum technology and attempt to derive data and/or equations to determine or estimate the rate outgassing for insulation materials used in cryogenic tanks. This will enable determining the degradation of vacuum levels of tanks which never had palladium monoxide installed.

3. RESULTS AND DISCUSSIONS

3.1 Principle materials investigated

3.1.1 Insulation materials

- a. Aluminum foil or aluminized mylar
- b. Glass fiber paper

3.1.2 Tank material

- a. Stainless steel (Such as series 304)

3.2 Outgassing rate of vacuum space materials collected from literature search

The literature search include a view of 1476 technical documents. Useful data was found in only seven documents listed as references.

Outgassing rate of different materials, their sample preparations, test conditions, references, and remarks are listed in Table 1.

TABLE 1. OUTGASSING RATE OF VACUUM MATERIALS

<u>Ref. No.</u>	<u>Material</u>	<u>Outgassing Rate*</u>	<u>Sample, condition, and Remarks</u>
1	Plain double aluminized mylar as received	8.87 x 10 ⁻⁸ (0.13 - 19.0) 5.6 x 10 ⁻¹⁰ (10 - 19.9)	Mylar film, 0.00025-in. thick; with a vacuum deposited 500 A film of aluminum on each side; made by Norton Metallized Products Division. Tested at 297 K. (Outgassing rate range - from 5.00 x 10 ⁻⁷ to 4.00 x 10 ⁻¹²).
1	Crinkled double-aluminized mylar. As received	2.38 x 10 ⁻⁷ (0.1 - 13.17) 5.0 x 10 ⁻¹¹ (9.17 - 13.17)	Mylar film 0.00015-in. thick; with a vacuum deposited 500 A film of aluminum on each side; crinkled to reduce contact area and maintain separation; made by National Metallizing Division, Standard Packaging Corp. Tested at 297 K. (Outgassing range - from 1.25 x 10 ⁻⁶ to 2.00 x 10 ⁻¹¹).
1	Mylar, as received	1.24 x 10 ⁻⁷ (.017 - 25) 4.43 x 10 ⁻¹¹ (11.67 - 25)	Polyethylene terephthalate film, 0.00025-in. thick; density, 0.051 lb/in ³ ; made by E. I. du Pont de Nemours, Inc. Tested at 197 K. (Outgassing rate range - from 5.40 x 10 ⁻⁷ to 1.00 x 10 ⁻¹¹).
1	Superfloc	3.16 x 10 ⁻⁷ (0.15 - 13) 8.80 x 10 ⁻¹¹ (9.67 - 13)	Mylar film 0.00025-in. thick with a vacuum-deposited 500A film of aluminum on both sides; small tufts of Dacron epoxied to on side at about 0.5-in. spacing. (Outgassing rate range - from 1.60 x 10 ⁻⁶ to 4.00 x 10 ⁻¹²).
2	Double aluminized mylar	5.43 x 10 ⁻⁸ (0.1 - 1000) 4.27 x 10 ⁻¹² (60 - 1000)	Sample, 182.9 cm. x 3,048 cm., with a total surface area of 1.115 x 10 ⁶ cm. ² . Weight of sample is 455.97 gram. Tested at 296 K. (Outgassing rate range - from 9.00 x 10 ⁻⁷ to 5.50 x 10 ⁻¹³).
3	Aluminum foil	1.85 x 10 ⁻¹¹	Testing temperature - 900 F, Testing pressure - 10 ⁻¹⁰ Torr
3	Glass fiber	0	Testing temperature - 1000 F, Testing pressure - 10 ⁻³ Torr
4	Mylar	2.3 x 10 ⁻¹⁰	24 h at 95% Relative humidity
5	Crinkled single-aluminized mylar	2.43 x 10 ⁻⁹	Sample, 1-in. thick and 6-in. diameter disk; Purge gas, nitrogen; test chamber pressure 10 ⁻⁵ - 10 ⁻⁶ Torr

Table 1 (Continued)

<u>Ref. No.</u>	<u>Material</u>	<u>Outgassing Rate*</u>	<u>Sample, condition, and Remarks</u>
6	304 stainless steel	2.00×10^{-12}	Sample cleaned by glass-bead shot blasting; baked 30 h at 250 C; outgassing rate after 24 h at room temperature.
7	304 stainless steel	2.00×10^{-10}	Test chamber material; testing pressure, 1 atmosphere to 10^{-7} Torr

- * 1.) Unit of outgassing rate is in Torr-L./sec.cm.². Outgassing rate, $q = \text{Volume of gas at a known pressure and temperature that passes a plane in a known time/unit area,}$

$$q = pV/A$$

Where $p = \text{pressure (Torr)}$, $V = \text{volume of gas flow/unit time (L./sec.)}$
and $A = \text{unit area (cm.²)}$

- 2.) Numbers in parenthesis indicate data average between evacuation hours.

3.2.1 Outgassing characteristics of the vacuum materials

3.2.1.1 Aluminum foil

The work by R. L. Reid in 1969⁽³⁾ was the only document found on aluminum foil. Sample was tested at 900 F and 10^{-10} Torr. Reid measured an outgassing rate of 1.8×10^{-11} Torr-L./sec.cm.². This value will be chosen for outgassing rate of aluminum foil.

3.2.1.2 Aluminized mylar

Outgassing rate of 10^{-12} Torr-L./sec.cm.² for aluminized mylar was chosen from recent work by Todd⁽²⁾ (1993). It was an average between 60 and 1000 hour range.

3.2.1.3 Glass fiber paper

According to the work of Reid⁽³⁾, the outgassing rate of glass fiber paper was assigned to be zero.

3.2.1.4 Stainless steel

From the work of Young⁽⁶⁾, and Erikon⁽⁷⁾, the outgassing rates of stainless steel were documented as 10^{-10} Torr-L./sec.cm.² for unbaked sample, and 10^{-12} Torr-L./sec.cm.² after baked for 30 hours at 250 C. An average value of 10^{-11} Torr-L./sec.cm.² will be chosen for stainless steel.

The following outgassing rates were chosen for calculation of vacuum level of tank;

- 1.) Aluminum foil = approximately 10^{-11} Torr-L./sec.cm.².
- 2.) Aluminized mylar = approximately 10^{-12} Torr-L./sec.cm.².
- 3.) Glass fiber paper = approximately 10^{-11} Torr-L./sec.cm.².
- 4.) Stainless steel = approximately 10^{-11} Torr-L./sec.cm.².

Except stainless steel these averaged outgassing rates are very much agreed with Ludlow's⁽⁹⁾ recommendation.

3.3 Calculation example

The following problem example is given to illustrate how the outgassing rates found in the literature searches can be used to estimate the vacuum pressure decrease in a cryogenic tank.

3.3.1 Sample problem

A cryogenic tank has an inner tank diameter of 2.5 feet and an outer tank diameter of 3 feet. The inner tank length is 20 feet. The tank uses 25 layers of multilayer insulation containing double aluminized mylar and glass fiber paper. The aluminized mylar is 0.00025 inches thick. The thickness of the glass fiber paper used as spacers is 0.0006 inches providing a total thickness of one layer of insulation material of 0.00085 inches. No getter is used in the vacuum space. Neglecting the thickness of the tank walls, estimate the outgassing after a five year period.

3.3.2 Solution

Outgassing of the 3-ft. tank is calculated as follows:

1. Surface area of outer tank = $\pi(3.0 \text{ ft.})(20 \text{ ft.}) = 188.5 \text{ ft.}^2$
2. Surface area of inner tank = $\pi(2.5 \text{ ft.})(20 \text{ ft.}) = 157 \text{ ft.}^2$
3. Vacuum space volume = $(\pi/4)(D_o^2 - D_i^2)(L.) = 0.7854[(3)^2 - (2.5)^2](20)$
 $= 43.2 \text{ ft.}^3$
4. Volume of insulation materials
 $= \pi(2.5 \text{ ft.})(0.00085 \text{ in.})(1 \text{ ft./12 in.})(25)(20 \text{ ft.})$
 $= 0.278 \text{ ft.}^3$
5. Net volume of vacuum space = $43.2 \text{ ft.}^3 - 0.278 \text{ ft.}^3 = 42.92 \text{ ft.}^3$
 $= 1,216 \text{ L.} \quad (1 \text{ cu ft.} = 28.33 \text{ L.})$

6. Outgassing from Mylar in the vacuum space

$$\text{Inner area of tank} = 188.5 \text{ ft.}^2 = (188.5 \text{ ft.}^2)(929 \text{ cm.}^2/\text{ft.}^2) = 175,117 \text{ cm.}^2$$

$$\text{Surface area of 25 layers of Mylar (2 sides)} = 2 \times 25 \times 175,117 \text{ cm.}^2$$

$$= 8,755,825 \text{ cm.}^2$$

Outgassing of mylar for five years period (10^{-12} Torr-L./sec.cm.² was chosen for aluminized mylar, see Table 1, Ref. 2)

$$= 8,755,825 \text{ cm.}^2(10^{-12} \text{ Torr-L./sec.cm.}^2)(3.15 \times 10^7 \text{ sec./year})(5 \text{ year})$$

$$= 1,379 \text{ Torr-L.}$$

7. Outgassing from stainless steel in the vacuum space

$$\text{Total surface area of stainless steel in the vacuum space} = 188.5 \text{ ft.}^2 + 157 \text{ ft.}^2$$

$$= 345.5 \text{ ft.}^2$$

$$= 320,970 \text{ cm.}^2$$

Outgassing of stainless steel for five years period

$$= 320,970 \text{ cm.}^2(10^{-11} \text{ Torr-L./sec.cm.}^2)(3.15 \times 10^7 \text{ sec./year})(5 \text{ year})$$

$$= 508.8 \text{ Torr-L.}$$

8. The total outgassing of the sample tank vacuum containing double aluminized mylar is

$$1,379.0 + 508.8 = 1,888 \text{ Torr-L.}$$

9. Pressure increase in the tank with a 1,219.4 L. vacuum volume during 5 years period

$$= 1,888 \text{ Torr-L.} / 1,219.4 \text{ L.} = 1.548 \text{ Torr or } 1,548 \text{ microns}$$

4. CONCLUSIONS

This study resulted in the following conclusions:

1. For a 3-ft. diameter and 20 ft. long tank, the pressure increase in the vacuum space for a time period of five years was calculated to be 1.548 Torr or 1,548 microns.
2. The calculated results were based on the estimated outgassing rates, so that the accuracy of actual pressure rise in the vacuum space can not be predicted.
3. Outgassing is controlled by almost 10 different parameters, it is quite obvious that a theoretical prediction of outgassing is very difficult and almost impossible. On this ground, to derive a mathematical equation to estimate the outgassing rate was not attempted during the summer project.
4. For more reliable results on outgassing rate, a laboratory test at KSC is highly recommended.

REFERENCES

1. Glassford, A. P. M., "Outgassing Behavior of Multilayer Insulation Materials," *Journal of Spacecraft*, Vol. 7, No. 12, 1964, pp.1464-1468.
2. Todd, Robert J., Pate, David, and Welch, Kimo M., "Outgassing Rate of Reemay Spunbonded Polyester and DuPont Double Aluminized Mylar," BNL-49345, August 1993.
3. Reid, R. L., DeWitt, W. D., and Gibbon, N. C., "Outgassing of Multifoil Insulation Materials in Sealed Vacuum Systems," *Journal of Vacuum Science and Technology*, Vol. 6, No. 1, 1969, pp. 217-221.
4. Elsey, R. J., "Outgassing of Vacuum Materials - II," *Vacuum*, Vol.25, No. 8, 1975, pp. 347-361.
5. Lin, J. T. "Analysis of Gas Flow through a Multilayer Insulation System," *AIAA Journal*, Vol. 11, No. 7, 1973, pp. 995-1000.
6. Young, R. J., "Outgassing Characteristics of Stainless Steel and Aluminum with Different Surface Treatments," *Journal of Vacuum Science and Technology*, Vol. 6, No. 3, 1968, pp. 398-400.
7. Erikson, E. D., Beat, T. G., Berger, D. D., and Frazier, B. A., "Vacuum Outgassing of Various Materials," UCRL-89299 Rev.- 1, *Vacuum Society & Technology*, December 20, 1983.
8. Perkins, W. G., "Permeation and Outgassing of Vacuum Materials," *Journal of Vacuum Science and Technology*, Vol. 10, No. 4, July/Aug. 1973, pp. 543-556.
9. Ludtke, Paul R., "A Study of the Vacuum Insulated Transfer Lines at Kennedy Space Center," *National Bureau of Standard Report 10705*, September 22, 1971.
10. Glassford, A. P. M. and Liu, C. K., "Outgassing Rates of Multilayer Insulation," NASA-N79-25051, 1978.
11. Zwaal, A., "Outgassing Measurements on Materials in Vacuum Using a Vacuum Balance and Quartz Crystal Balances," NASA-N79-25053, 1978.