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RHIC PROJECT

Brookhaven National Laboratory

Outgassing Rate of Reemay Spunbonded Polyester and Dupont Double Aluminized Mylar

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ABSTRACT

This paper presents the outgassing rates of two commercially available multi-layer insulation (*MLI*) materials commonly used in cryogenic applications. Both Reemay Spunbonded Polyester and Dupont Double Aluminized Mylar (*DAM*) were studied for outgassing species and respective rates, and the total amount of outgassed material. Measurements were made using a *Fixed Aperture* Technique. A sample was pumped on through an aperture of known size with a turbomolecular pump. Pressure vs. time was plotted for both Reemay and *DAM*, as well as the baseline system, and data conveniently extrapolated to ≈1000 hrs. A quadrupole residual gas analyzer was used to measure the outgassing species.

I. INTRODUCTION

MLI is extensively used in the **RHIC** (Relativistic Heavy Ion Collider) cryostats. This insulating material comprises *MLI* blankets which are wrapped around the superconducting cold-mass and other surfaces within the cryostat. A cross section of a typical dipole cryostat and magnet is shown in Fig. 1.0. Twelve of these cryostats measure 480 meters in length.

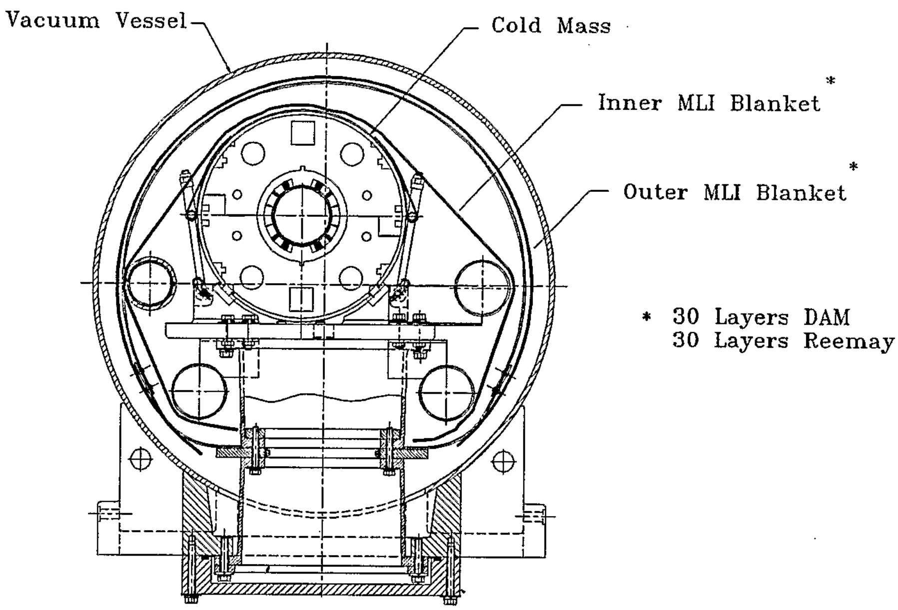

Figure 1.0 Cross Section of Dipole Magnet and Cryostat

Due to the large amounts of *MLI* in each 480 meter cryostat, ≈ $4x10^9$ cm^2, its outgassing rate has an adverse effect on the pumpdown of the insulating vacuum as well as delaying effective leak checking of the cryostat. Outgassing rate information must be obtained so suitable rough pump systems can be configured to handle the estimated gas load.

II. EXPERIMENTAL

The test apparatus, represented in Fig. 2.0, consisted of a test chamber with an area of $1.92x10^3$ cm^2, into which the sample was installed. This chamber was pumped on through an orifice of diameter 0.612 cm by a turbomolecular pump with S_{water}=30 l/s. The

0.612 cm orifice gave an effective pumping speed at the vessel of 3.36 l/s for water vapor. Two Varian UHV-24 Bayard-Albert Gauges, controlled by a Granville Phillips 271 controller were used, as well as an Inficon Quadrex 200 QRGA. The Reemay sample was 144.8 cm x 3,048 cm, comprising a total surface area of $8.83 \times 10^5 cm^2$. The *DAM* sample was 182.9 cm x 3,048 cm, with a total surface area of 1.115×10^6 cm^2. Weights of each sample were 685.50 grams and 455.97 grams respectively. Each sample was loosely rolled and inserted into the test chamber.

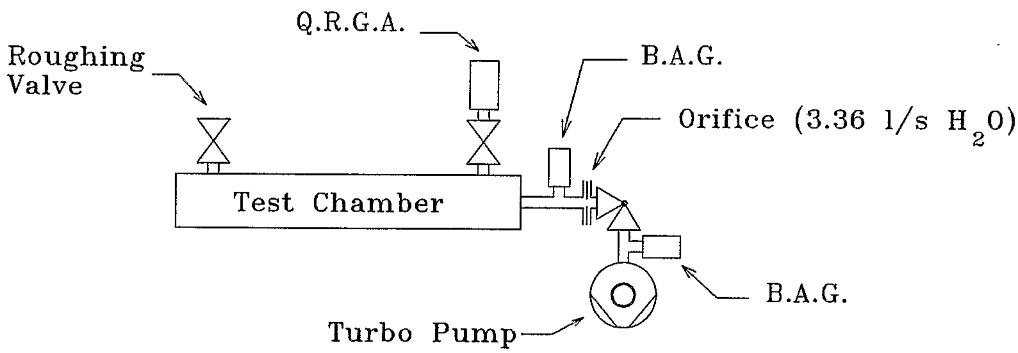

Figure 2.0 System Block Diagram

Baseline data of the system was taken for 24 hours prior to tests with *MLI* to establish system outgassing. The system was initially rough pumped through V_r by a cryosorption pump to a pressure of 100 microns. At this time the Turbo pump was valved in running at 100% speed and V_r subsequently closed. The rate of pumpdown was recorded for 100 hours or more for each sample until a pressure decay slope could be accurately charted.

The basis for measuring the outgassing rate is as follows:

$$Q(t)=P(t)\times S \qquad (1)$$

where **s** in our case is the effective speed at the vessel for water at 296°K, and is a constant. A *pumpdown curve* was generated for pressure vs. time for each material. This plot is useful in that it reveals anomalies that may exist in the outgassing rate at lower pressures. Knowing the effective pumping speed, the *pumpdown curve* was converted to a graph of **q** vs. t, where **q** is the outgassing rate per unit area and **t** is time in hours.

The total amount of outgassed material per unit $Area_{surface}$ can be expressed by:

$$\int Q(t)\,dt = S\int P(t)\,dt \qquad (2)$$

These data are readily obtained by graphically integrating the plots of **q** vs time shown in Figs. 3.1 and 3.2.

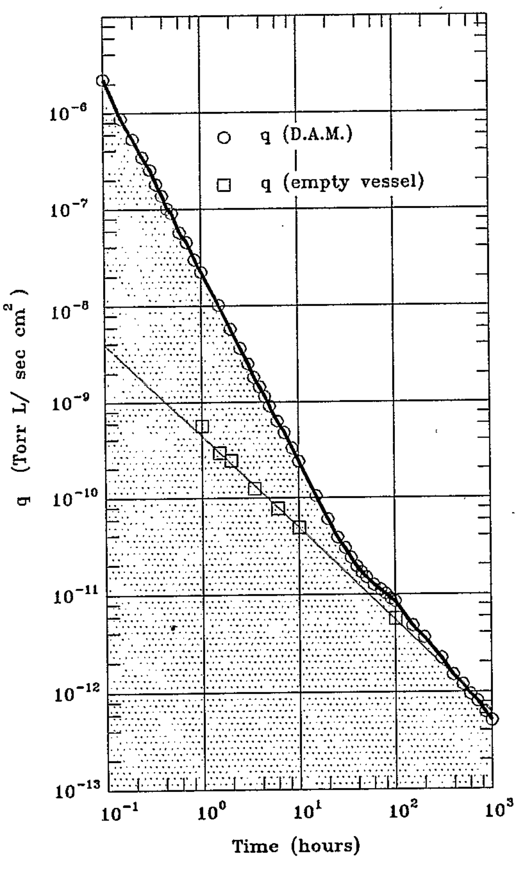

Figure 3.1 DAM Outgassing Rate vs. Time

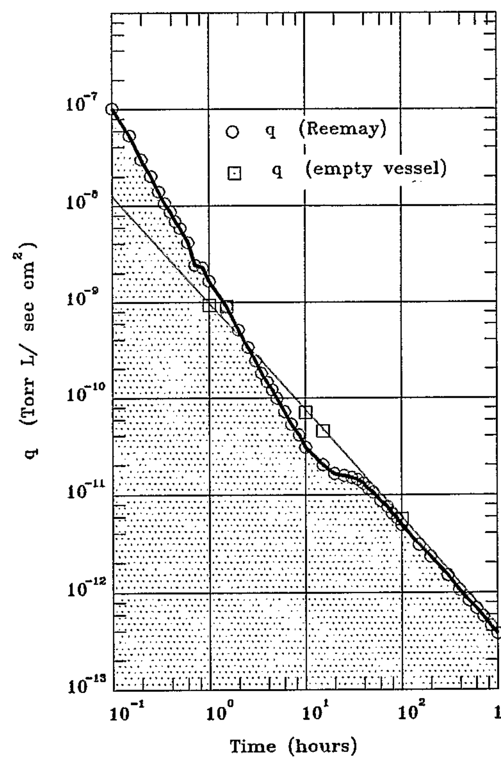

Figure 3.2 Reemay Outgassing Rate vs. Time

III. RESULTS

The outgassing rates and spectrum for both *DAM* and Reemay are shown in Figures 3 through 6. Figures 5 and 6 show that the major constituent of the outgassing is water vapor. This is true for both the *DAM* and Reemay material. It can be seen in both graphs that the baseline outgassing rate did not significantly impact on the total outgassing rate since the area of the samples were ≈ 500 times that of the test chamber. The total water contribution by the test chamber was ≈ 6.45×10^{-3} grams, or < 1% of the samples.

Two things are readily apparent from the graphs. The first being the *knee* in the outgassing slope of the Reemay. This *knee* occurs after 10 hours of outgassing where the slope goes from -1.77 to -0.33. This slope remains for roughly 40 hrs and then falls off to a new slope of -1.1. This change in slope occurs because the outgassing becomes "diffusion limited". Once most of the surface outgassing is complete, we speculate an "inter-layer" diffusion process occurs due to the long and arduous path the water vapor must take in being liberated. The initial surface outgassing changes rapidly with time (dQ/dt), while the "inter-layer" diffusion process remains more constant. After ≈ 30 hours, dQ/dt decreases to a value roughly 60% of the original surface outgassing rate. This "knee" is not representative of *MLI*, since Reemay is used as a spacer in one layer thicknesses. This effect is not as pronounced in the *DAM* where only a slight change in the slope is observed.

The second observation worth noting is the higher outgassing rate of the *DAM*. The initial **q** of the *DAM* is ≈ 23 times that of the Reemay. After one hour $q_{DAM} = 2.26\times10^{-6}$ Torr-L/sec cm^2, compared to 1.00×10^{-7} Torr-L/sec cm^2 for the Reemay.

Another major observation is the spectrum of the Reemay material. The baseline spectrum (Fig. 3.3) shows only modest amounts of CO_2 at a pressure of 1.6×10^{-6} Torr. With the Reemay inserted, the spectrum shows appreciable amounts of hydrocarbons at a total pressure of 3.5×10^{-6} Torr (Fig. 3.3.2). These hydrocarbon peaks occur at amu 39, 41, 43, and 44, with partial pressures of 5.2×10^{-8}, 9.1×10^{-8}, 1×10^{-7}, and 9.0×10^{-8} Torr respectively. Peaks of ≈ 4×10^{-8} Torr also occur at 55 and 57. These Hydrocarbons are likely a by-product of the Reemay manufacturing process. This may be noteworthy to those designing superconducting magnets who wish to pump Helium leaks with *molecular sieve* material. This material, bonded to the *cold-mass*, may become plugged due to the hydrocarbons and water present in the insulating vacuum.[1] The *DAM* spectrum (Fig. 3.4) shows no appreciable amounts of outgassed hydrocarbon material.

The total amount of outgassed water is of particular importance. This impacts on the mechanical pumps being used to evacuate the **RHIC** cryostats. Table 3.5 shows the total amount of water contained per cm^2 of material, as well as the total for a 480 meter Cryostat.

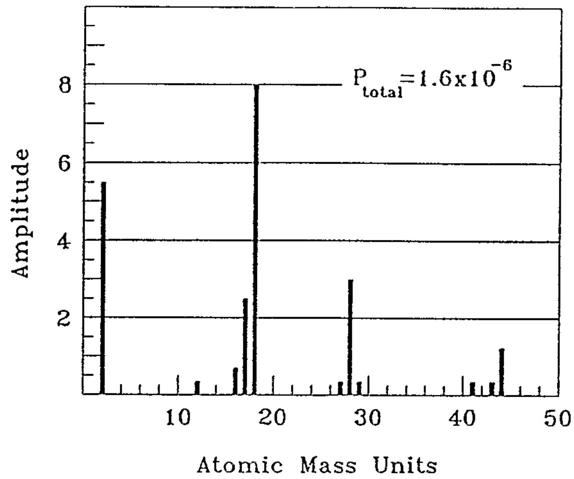

Figure 3.3 Empty Vessel Spectrum @18 hours

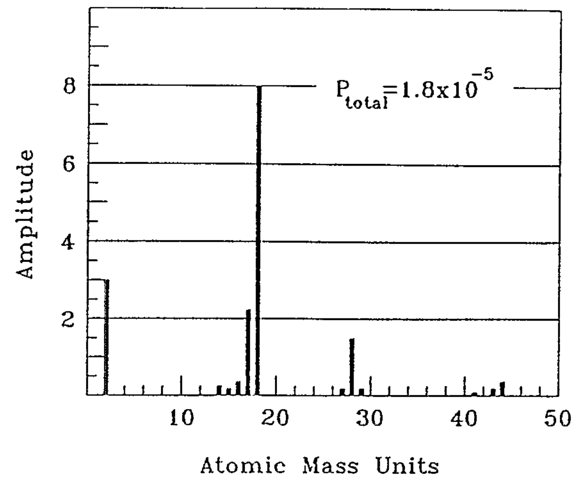

Figure 3.3.1 Spectrum @ 7.5 Hours (Reemay)

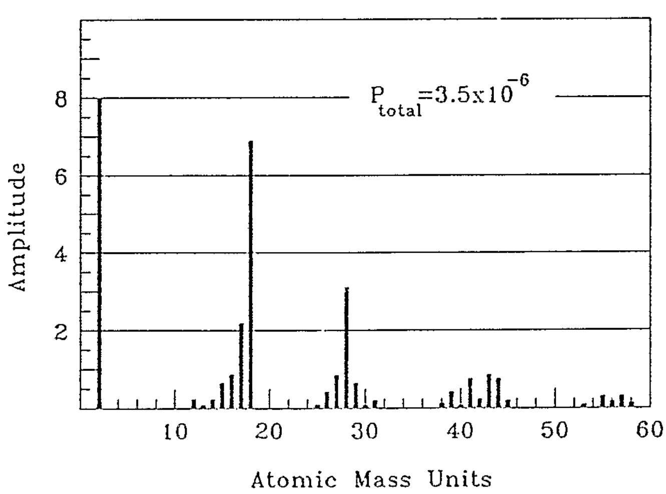

Figure 3.3.2 Spectrum @ 56 Hours (Reemay)

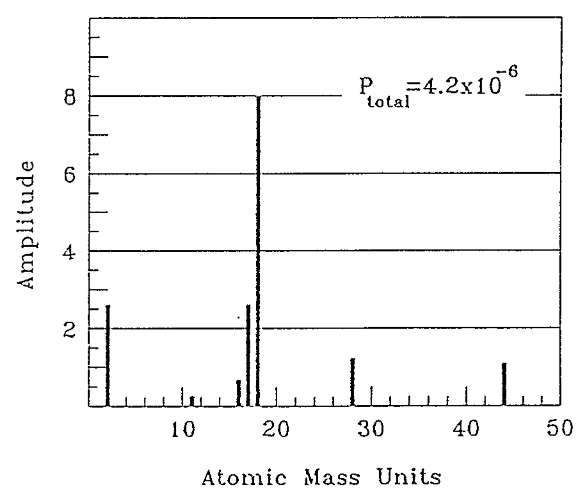

Figure 3.4 Spectrum @ 75 Hours (D.A.M.)

MATERIAL	TOTAL OUTGASSED WATER	
	per unit area	per 480 meter cryostat
Reemay spunbonded polyester	9.78×10^{-8} g/cm^2	2.27×10^{2} grams
double aluminized mylar	1.63×10^{-6} g/cm^2	3.15×10^{3} grams
ambient air @ 70% rel. humidity	PP ≈ 10 Torr	1.2×10^{3} grams
steel vacuum vessel	3.38×10^{-6} g/cm^2	≈ 5 grams

Table 3.5 Total Water Accumulations

The *DAM* contains ≈ 17 times the amount of water per unit area than the Reemay material. The total amount of water contained in the *DAM* and Reemay is ≈ 3.38 liters. The water contained in the air within the insulating vacuum prior to roughing is approximately 1.2 liters, while the amount on the vessel walls is negligible. The total water in each 480 meter cryostat prior to roughing is 4.58 liters. This will be shared equally by four pumps, each having to handle 1.15 liters. A cold trap of adequate size will be used to handle this load. This will aid in leak checking the Cryostats through the rough pumps by reducing the partial pressure of water at the leak detector. It will also reduce contamination of the rough pump oil.

The results of the test are in agreement with previous work done with **MLI.**[2]Published work on the outgassing rate of *DAM* reported totals of 1.1×10^{-6} g/cm$^2_{\text{surf. area}}$, and 2.6×10^{-3} g/g$_{\text{sample mass}}$. These results, compared to 1.63×10^{-6} g/cm^2, and 3.99×10^{-3} g/g, are ≈ 50% lower. This is attributed to the room humidity at the time the sample was installed in the test chamber.

IV. CONCLUSIONS

The outgassing rates of *DAM* and Reemay have been established, and the amount of water from each determined.It is evident that the *DAM* carries much more bulk water than the Reemay spacer material. A total of 4.48 liters of water are contained in each

RHIC Cryostat. With this information, properly sized cold traps can be installed on the rough pumps.

The way in which the Reemay outgasses is also of particular interest. Once bulk outgassing is complete, a diffusion limited process takes place. This effect may show itself in application since the **RHIC** insulating blankets comprise 30 layers at a length of 10 meters.

Hydrocarbons were noted to outgass from the Reemay material. This release could have irreversible plugging affects on *sieve* material in proximity to the *MLI*.

1.Welch, K.M., CAPTURE PUMPING TECHNOLOGY(Pergamon Press Inc.,New York,1991),p.280.

2. Glassford, A.P.M., Liu, C.K., "Outgassing Rate of Multilayer Insulation",J. Vac. Sci. Technol.,17(3),696(1980).

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ADDENDUM TO THE TOTAL OUTGASSED WATER OF REEMAY AND DAM

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When generating the graphs of outgassing rate vs. time for the *Reemay* and *DAM*, data was collected after one hour of pumping and extrapolated to a lower and upper limit of 10^{-1} and 10^{3} hour respectively.

In calculating the total outgassed water of the *MLI*, the graphs of outgassing rate vs. time (Fig. 3.1, 3.2) were graphically integrated over the interval 10^{-1} to 10^{3} hours. These total accumulations are given in Table 3.5. However, significant outgassing occurs prior to 10^{-1} hour. This additional accumulation was not included in the integration. The graphs have been subsequently integrated over the interval 10^{-2} to 10^{3} and total accumulations adjusted accordingly. The updated data are given in the following table. The estimated total for a 480 meter cryostat is ≈ 34.1 kg.

MATERIAL	TOTAL OUTGASSED WATER	
	per unit area	per 480 meter cryostat
Reemay spunbonded polyester	4.00×10^{-6} g/cm^2	9.27×10^{3} grams
double aluminized mylar	1.23×10^{-5} g/cm^2	2.36×10^{4} grams
ambient air @ 70% rel. humidity	PP ≈ 10 Torr	1.2×10^{3} grams
steel vacuum vessel	3.38×10^{-6} g/cm^2	≈ 5 grams

Table 3.5 Total Water Accumulations