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Outgassing properties of low temperature thermal insulators

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Introduction

This investigation was conducted in order to determine the vacuum properties of some of the low temperature thermal insulators used in vacuum and in the construction of super insulated dewars, which by proper insulation, could hold cryogenic fluids with negligible loss for about 2–3 months. The outgassing rate of these non-standard insulators are not readily available and hence we took up the study of outgassing rate as well as the species that are released from the insulators at ambient and higher temperatures. The knowledge of the nature of the gases released from the insulators is necessary to ensure better control of outgassing by proper pre-treatments[1–5]. Two sets of polyester films (being manufactured by M/s Garware Plastics and Polyesters Ltd, Nasik, India) received in the form of rolled tapes of 75 mm width and 25 and 12 μ thickness and fibre discs received in circular pieces of ~ 20 cm dia were utilized for the experiment. These were supplied by L'Air Liquide' France. Samples were made out of artificial and natural fibres on a trial basis and, hence, no commercial grade or name is available. The variation of outgassing rate has been plotted, tabulated and discussed in detail.

Test apparatus

The apparatus used in the outgassing experiment and the accompanying gas analysis experiment is shown in Figure 1, which is self explanatory[6]. At any moment the gas throughput across the conductance C (8.67 l s^{-1}) is given by the product of the conductance and pressure difference across C. A quartz tube with nichrome tape wound around it was hung in the chamber so that *in situ* heating of the sample could be done. A thermocouple was connected for measuring the temperature rise. However, an error of 5% in temperature is estimated in the arrangement. A Centronic AIG 50 model partial pressure analyser providing scanning from 1–50 amu was used to analyse the outgassing products. Vacuum of the order of 1×10^{-6} torr was maintained in the system by a 120 l oil diffusion pump backed by a 50 l rotary pump. Vacuum was maintained at all times in the quadrupole mass analyser.

Test procedure

Initially a blank run for 4–5 h was done and the system background of outgassing worked out for ambient condition. Then the heater was operated with a constant voltage of 35 V and the outgassing at different temperatures noted. Subsequently 200 cm each of the polyester films having a very smooth surface were cleaned with a tissue paper and loaded in a loose roll with copper wire inside the heater and the outgassing characteristics and gas species released at ambient and an appropriate high temperature (130°C) were worked out. Separate samples were used for each experiment for the obvious reason that the vacuum treatment could have effected some change in the polyester film. The pressure difference was noted every 2 or 3 min at the initial stage, so that the temperature at which maximum outgassing occurs could be recorded, and later every 15 min. After reaching the maximum temperature the heating was continued for 30 min and then switched off. The outgassing rate was worked out by

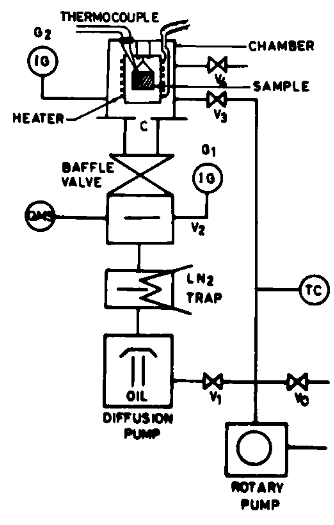

Figure 1. Experimental setup.

subtracting the corresponding outgassing of the system background at different temperatures and calculated for unit surface area.

Secondly two fibre discs were evaluated. These being porous and thicker materials the outgassing rate was higher. In this case samples were heated only up to 65–70°C. The experimental procedure was the same as that for the polyester films. During the investigation the ambient temperature varied between 25 ± 3°C and relative humidity from 60 ± 5%.

Results

The variation of outgassing rate of the polyester films of 25 and 12 μ with time and temperature is plotted in Figure 2 and tabulated in Table 1.

The results of evaluation of fibre discs A and B are given in Figure 3, and Table 2.

Figure 4 shows the micrographs of the fibre discs.

Discussion

Polyester film. From Figure 2 it is seen that the outgassing rate of the polyester films is very low and comparable to metals. The nature of the curve shows that loosely adsorbed gases only are present. Moreover the heat treatment does not change the pattern of the curve except for a small gas burst around 50°C. Usually the main constituent to the outgassing of most of the materials is water vapour in fairly large quantities and to a lesser extent carbon monoxide, carbon dioxide, hydrocarbons etc. Also the temperature at which the water vapour is released from the sample will depend upon the binding energy with which the water molecules have been sorbed, by the material. The background spectrum at 50°C shows about 3.5×10^{-7} torr and 9×10^{-8} torr of mass nos 18 and 17 respectively and traces of mass nos 16 and 28. With the samples the partial pressures of mass nos 18 and 17 are about 2.8×10^{-6} and 7×10^{-7} torr for polyester (12 μ) and 1.7×10^{-6}

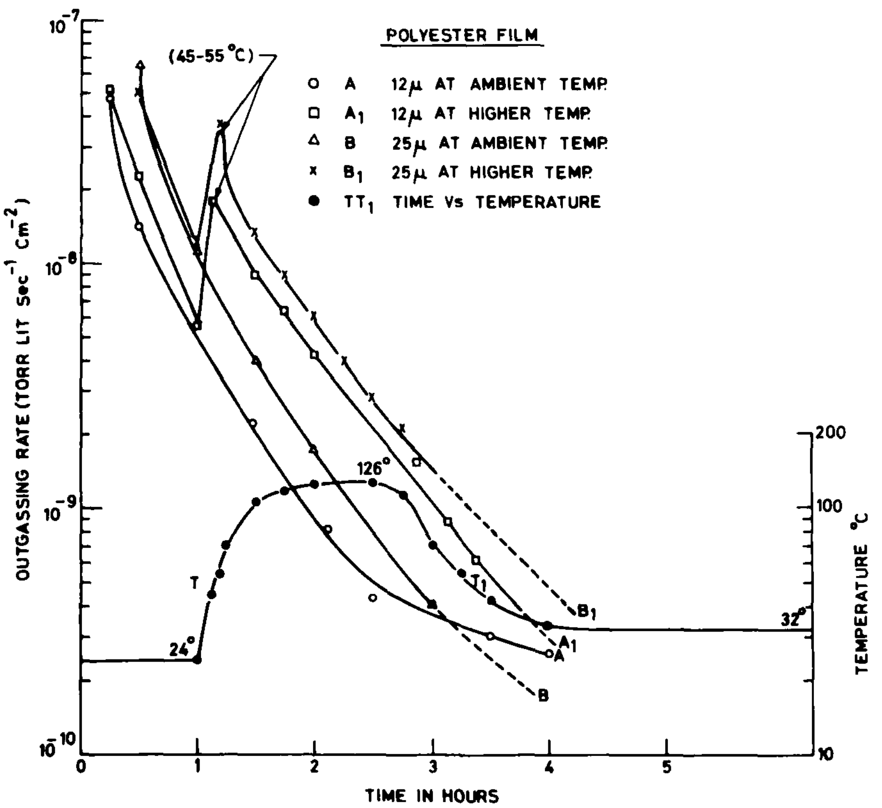

Figure 2. Variation of outgassing rate with time and temperature.

Table 1. Variation of outgassing rate at ambient and higher temperatures

Polyester film										
	Time (min)									
Ambient	15	30	60	90	120	150	180	210	240	
(a) 12 μ	48	14	4.8	2.2	0.8	0.6	0.4	0.3	0.25	
(b) 25 μ	390	65	11	4.0	1.7	1.2	0.4			
Higher temperatures										
	15 (25°C)	30 (25°C)	60 (25°C)	70 (54°C)	75 (70°C)	90 (106°C)	120 (124°C)	135 (126°C)	150 (126°C)	175 (74°C)
(a) 12 μ	52	23	5.5	16	15	10	4.2	3	2.2	1.1
(b) 25 μ	280	50	12	37	19	13	6.0	3.9	2.8	1.5

Outgassing rates in units of torr l s^{-1} $cm^{-2} \times 10^{-9}$.
Figures in brackets denote temperature.

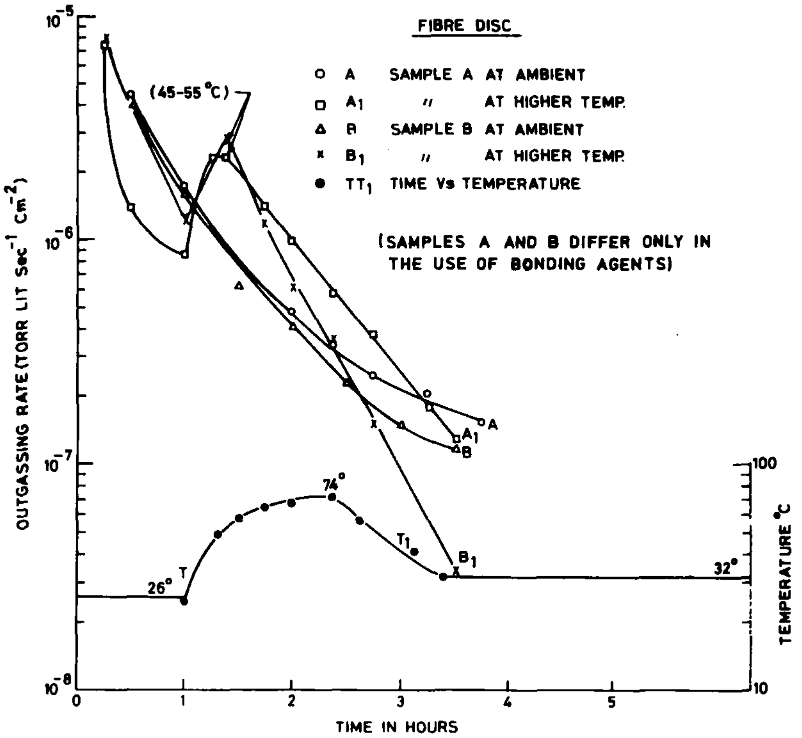

Figure 3. Variation of outgassing rate with time and temperature. (Samples A and B differ only in the use of bonding agents.)

Table 2. Variation of outgassing rate at ambient and higher temperatures

Fibre discs	Time (min)									
Ambient	30	60	90	120	150	180	210	240		
(a) A	43	17	7.7	4.7	3.3	3.4	2.0	1.5		
(b) B	40	16	6.2	4.1	2.3	2.2	1.5	1.4		
Higher temperatures	15 (25°C)	30 (25°C)	60 (25°C)	80 (52°C)	100 (66°C)	115 (70°C)	135 (74°C)	155 (58°C)	185 (42°C)	200 (32°C)
(a) A	74	14	8.7	24	14	10	5.8	3.8	1.8	1.3
(b) B	79	40	12	29	12	6.1	3.6	1.5	0.4	0.33

Outgassing rate in units of torr l s^{-1} $cm^{-2} \times 10^{-7}$.
Figures in brackets denote temperature.
Samples A and B differ only in the use of bonding agents.

and 6×10^{-7} torr for polyester (25 μ) and traces of mass nos 16 and 28. The reason for higher partial pressure of H_2O in the thinner film is being investigated further. Within a short while the water peaks reach the background value. This shows that very little water is adsorbed on the sample and this is released within 10–15 min after heating. This shows the hydrophobic nature of the polyester film. The scanning electron micrograph of the polyester film showed that there was no structure and that it is completely amorphous. The implication of this has to be investigated further. Curve A, A_1 in Figure 2 shows a lower outgassing rate due to the smaller sample thickness.

The polyester film could withstand a much higher temperature of 150°C in vacuum without decomposition and change in physical characteristics even though it is meant for low temperature thermal insulation.

Fibre discs. Figure 3 shows the outgassing curve of fibre discs A and B at ambient and higher temperatures. The nature of the curves, A, B also shows the loosely adsorbed state of gases. Even though the initial outgassing rate is high the fall rate is so fast that it reaches 1.5×10^{-7} torr l s^{-1} cm^{-2} in 4 h. On heating, the fibre disc A behaves very similar to polyester film with only a small increase in the outgassing rate around 50°C and thereafter following the ambient curve.

In fibre disc B the ambient outgassing rate stabilizes around 1.5×10^{-7} torr l s^{-1} cm^{-2}. On heating the outgassing decreases

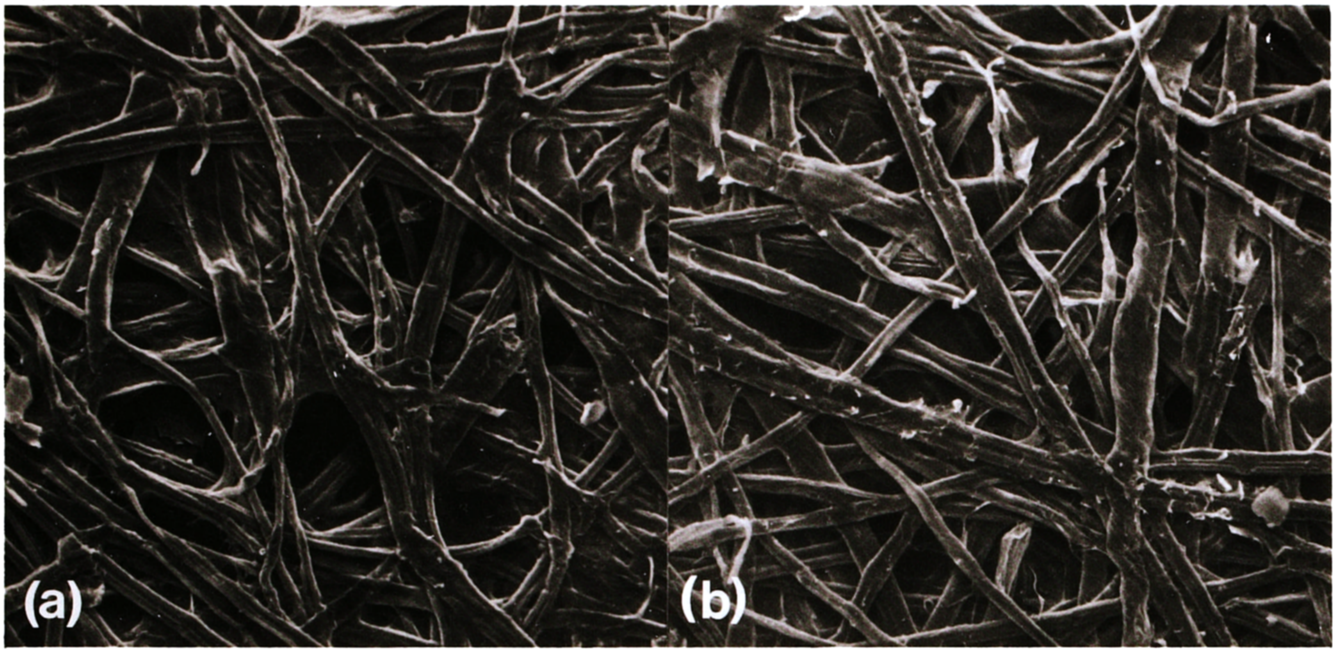

Figure 4. (a) Micrograph of surface of fibre disc A ($\times$ 200). (b) Micrograph of surface of fibre disc B ($\times$ 200).

rapidly after a small gas burst around 50°C to a much lower value of 3.2×10^{-8} torr l s^{-1} cm^{-2}. The reason is attributed to the variation in the porosities. The micrograph Figure 4 of the fibre discs shows a very porous structure which lead to easy adsorption and desorption. The partial pressure of mass nos 18 and 17 around 50°C for fibre disc A is about 4.5 and 1.2×10^{-6} torr and for fibre disc B 3.4 and 1.2×10^{-6} torr respectively with traces of mass nos 16 and 28.

Conclusion

The outgassing rate at ambient as well as at high temperatures does not differ appreciably in the polyester film which goes to prove its non-affinity for water vapour and other atmospheric constituents. The fibre discs are equally good due to their highly porous nature.

On the basis of the above it is concluded that both polyester film and fibre discs have excellent vacuum properties as far as their outgassing rate and gas release at higher temperatures are concerned.

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