

Table 3 Points of $ds/dM = 0$ for subsonic to supersonic flow, $k = 1.4$

f/a	$1/k$	0.8	0.9	1.0	2.0	4.0	10.0	∞
M	0	0.577	0.828	1.0	1.73	2.15	2.42	2.65
$(s - s^*/R)_{\max}$	0.455	0.131	0.026	0	0.498	1.25	1.94	∞

Fig. 6 that the value of f has little effect on the flow in moderately divergent duct. For duct half-angles greater than 6° , f/a will be less than 0.1. This is a further indication that since the absolute value of f is an unknown factor, an experiment can be designed which will pass from supersonic to subsonic flow.

At larger values of f/a or in ducts with very little divergence, the effect of friction becomes much more prominent. In fact, at $f/a = 1/k$, T_0 is constant, see Eq. (13). This case is, of course, the variable A and f process described in Fig. 5. It is also worth noting that $f/a = 0$ corresponds to the variable A and T_0 process, and that $f/a = \infty$ corresponds to the variable T_0 and f process in Fig. 5. The variations in A and T_0 with between $f/a = 0$ and $f/a = 0.10$ are too small to be seen in Fig. 6. The variations in A and T_0 are 1% or less and 2% or less, respectively, between Mach 0.5 to 3.0.

At $f/a \geq 1/k$, $ds/dM = 0$ at some point in the flow. This behavior is also similar to that predicted by the Crocco functions. Heating or increase in T_0 is required at $M < M_{s\max}$, and vice versa. This point is illustrated in Fig. 6 for $f/a = 1.0$. For $f/a > 1$, the point of $ds/dM = 0$ occurs at $M > 1$. The values of M and entropy at the $ds/dM = 0$ point is presented in Table 3 for various values of f/a .

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Outgassing Behavior of Multilayer Insulation Materials

A. P. M. GLASSFORD*

Lockheed Missiles & Space Company, Palo Alto, Calif.

MULTILAYER insulation systems show minimum effective thermal conductivity when they are operated with an interlayer gas pressure below 10^{-5} torr, at which pressure the gaseous heat conduction is negligible. In service this low pressure is reached by evacuation of the interlayer gas to space or by vacuum pump. As the gas pressure is reduced, however, outgassing from the insulation surfaces can seriously prolong the evacuation period. This Note reports some ex-

perimental outgassing data¹ for multilayer insulation materials under conditions appropriate to use in cryogenic fuel tanks. For such a system, the multilayer insulation would be purged before launch with a gas not condensable at the fuel temperature, such as helium. With the purge gas pressure held at 1 atm the fuel tank would be filled, and the insulation wrap would assume a temperature between ambient and the fuel temperature. During and after launch the purge helium gas would be evacuated to space.

The data are reported as recommended by the American Vacuum Society's AVS-9,² according to which the solid involved is to be described only by its gross characteristics of manufacturing history, pretreatment, etc., and the data are to be obtained and presented as mass released per unit time per unit solid apparent surface area or bulk volume as a function of time for constant experimental temperature and negligible experimental pressure. Guidelines are proposed by AVS-9 for apparatus sizing and design to ensure that the background outgassing due to the apparatus itself is negligible, and that the experimental gas pressure is negligible with respect to the ambient pressure. Because of the peculiarities of the sample geometry and the required preconditioning, these two guidelines could not be followed strictly in this work, but preliminary experiments were conducted to confirm that the intent of the guidelines had been met.

The pumpdown technique selected consists of evacuating in turn and under the same conditions an empty chamber and then the chamber plus insulation sample. The sample outgassing rate is found from the difference in the slopes of the two pressure histories at a given chamber pressure. The mass balance equations for the empty chamber and the chamber plus samples are, respectively,

$$-(V/RT)(dP/dT)_e = SP/RT - Q_e A_e \quad (1)$$

$$-(V/RT)(dP/dT)_s = (SP/RT) - Q_e A_e - Q_s A_s \quad (2)$$

If the chamber outgassing rate Q_e is negligible, the volumetric pumping speed S is constant or a function only of P over the experimental pressure range, and the temperature T is constant during the experiment, then the sample outgassing rate Q_s may be found by subtracting Eq. (2) from Eq. (1) at constant pressure. This gives

$$Q_s = -V/A_s RT [(dP/dt)_e - (dP/dt)_s]_{P=\text{constant}} \quad (3)$$

This value of Q_s is assigned to the time at which $(dP/dt)_s$ was measured.

Outgassing quantities are presented here in the customary units of pressure times volume. These data can be converted to mass quantities by dividing by the gas constant times the measurement temperature, which was 297°K for the reported tests.

Apparatus

The insulation sample chamber (Fig. 1) is 6 in. in diameter and 1.5 in. high, and its top is closed by the underside of a tank 6 in. diam and 3 in. deep. The joint is sealed by a vacuum flange using either a copper gasket at the lower temperatures or an O-ring gasket at temperatures closer to ambient. The chamber and most other components communicating with it are made from polished stainless steel. The tank can be filled with a boiling or melting mixture to provide a constant temperature heat sink or source. A sample tray within the sample chamber supports the insulation sample. The insulation sample sheets, cut 5.4 in. o.d. and 0.5 in. i.d., are stacked on the tray to a height such that when the chamber is closed, the underside of the tank exerts very little pressure on the stack, establishing its height at 0.40 in. Thermocouples were positioned within the sample stack at three

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* Research Scientist, Engineering Sciences Laboratory.

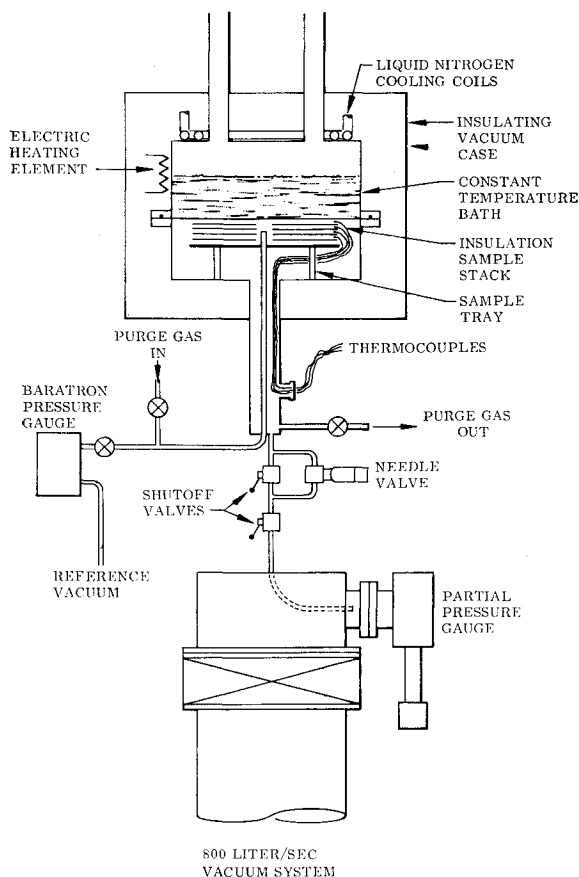


Fig. 1 Schematic of outgassing apparatus.

vertical positions and about half-way between center and circumference.

The chamber is exhausted through a 1-in. diam, 14-in. long, thin-walled tube entering its underside. Between the downstream end of this tube and the inlet to the diffusion pump is a section of 0.25-in. diam tubing about 9 in. long, which provides the principal flow resistance in the evacuation system. The line, valving, and sample chamber volume are sized so that with $P = 1$ atm initially, the entire pumpdown can be performed through the diffusion pump without hazard. The chamber pumping speed is 5.4 l/sec, and the total enclosed volume of the chamber is 1.13 l.

Sample chamber pressure is measured by an MKS Baratron pressure gage via a 0.25-in. diam tube that passes up through the evacuation tubing and through the center of the sample tray into the cavity formed by the center holes of the sample sheets. During the preconditioning phase the same tube is used to introduce purge gas, which flows radially outward through the sample's layers and leaves the system to atmosphere through a valve at the bottom of the 1-in. diam evacuation tube. The pressure gage had a remote control box and two sensing heads, covering the pressure range of 1000 to 10^{-5} torr. Utterback and Griffith³ show that this instrument is accurate to a few percent at pressures near 10^{-4} torr, making it more reliable than its electronic alternatives. Further, it is insensitive to the nature of the gas measured and is more convenient to use. However, its use required that the chamber pressure P be kept at least one order of magnitude higher than would be desirable, and it was necessary to establish that the data were insensitive to P . This was done by testing a typical sample of as-received double-aluminized Mylar using about half the pumping speed used in the main tests, by adjustment of the needle valve, resulting in a higher value of P at given time. For typical evacuation times the Q_e 's determined by the two methods differed by less than 2%, which was the order of the sample-to-sample variation, and

thus the system pressure was concluded to be negligible. Certain tests called for the sample to be cooled, but the instrumentation and certain other connecting components had to be located remotely so as to be maintained at ambient temperature. This feature added appendages to the apparatus which could have raised the background outgassing rate above a negligible value. For this reason preliminary tests were made in which Q_e was determined by comparing the empty chamber evacuation rate with the known system pumping speed. The measured Q_e was less than 1% of a typical Q_e after 10 min pumping and was considered to be negligible. The major background sources were the pressure gage heads which operate at 323° K, and its outgassing rate would thus be expected to decay faster than that of the samples.

The outgas was analyzed qualitatively by a Varian partial pressure gage attached to the exhaust plenum chamber. This downstream location was necessary because of the need to cool the sample chamber. However, the gage had an upper operating pressure limit of 10^{-3} torr and could be operated earlier in the pumpdown at this location than if it were attached to the chamber. Further, it permitted the background gases in the gage and pumping system to be analyzed separately and discounted accordingly. The gage had an upper limit of mass number of 70 amu, so that the heavier molecules were not detectable. However, the organic samples had been exposed to a vacuum coating process since manufacture and any solvents remaining could be considered to have been removed at that time. The outgas components were expected therefore to be principally the atmospheric components of water vapor, with small amounts of CO_2 and N_2 , for which the partial pressure gage was totally adequate.

Experimental Procedure

The materials tested are described in Table 1. The following standard procedure was adopted for opening and closing the sample chamber between tests to establish repeatability and to minimize the exposure of the internal surfaces to the atmosphere. The sample chamber was raised to 1 atm pressure by admitting helium through the system. The chamber was reclosed after exactly 10 min.

Each outgassing test consisted of one empty chamber and one full chamber pumpdown. (Preliminary testing showed that reproducibility was high enough so that only one empty test was needed in cases where similar conditions had been used previously.) Since the gas analyses in all the reported tests showed $\geq 98\%$ water vapor, $\sim 1\%$ CO_2 , and only traces of other gases, gas analyses were made only in a random manner after the initial tests in order to insure that no exception to this conclusion would escape notice.

Reproducibility of $P(t)$ data was about $\pm 2\%$, based upon comparison of tests on similar samples taken from the same roll. Empty chamber data reproducibility was about $\pm 1\%$.

Table 1 Sample insulation materials tested

Mylar:	polyethylene terephthalate film, 0.00025-in. thick; density, 0.051 lb/in. ³ ; made by E. I. du Pont de Nemours, Inc.
Plain double-aluminized Mylar:	Mylar film, 0.00025-in. thick, with a vacuum-deposited 500 Å film of aluminum on each side; made by Norton Metallized Products Division.
Crinkled double-aluminized Mylar:	Mylar film 0.00015-in. thick with a vacuum-deposited 500 Å film of aluminum on each side; crinkled to reduce contact area and maintain separation; made by National Metallizing Division, Standard Packaging Corp.
Tissuglas:	Borosilicate glass fiber paper, 0.0006-in. nominal thickness, 0.0081 lb/in. ³ nominal density; made by Pallflex Inc.
Superfloc:	Mylar film 0.00025-in. thick with a vacuum-deposited 500 Å film of aluminum on both sides; small tufts of Dacron epoxied to one side at about 0.5-in. spacing; Dacron tufts made by E. I. du Pont de Nemours, Inc., are attached by General Dynamics Corp. to a basic film made by National Metallizing Division, Standard Packaging Corp.

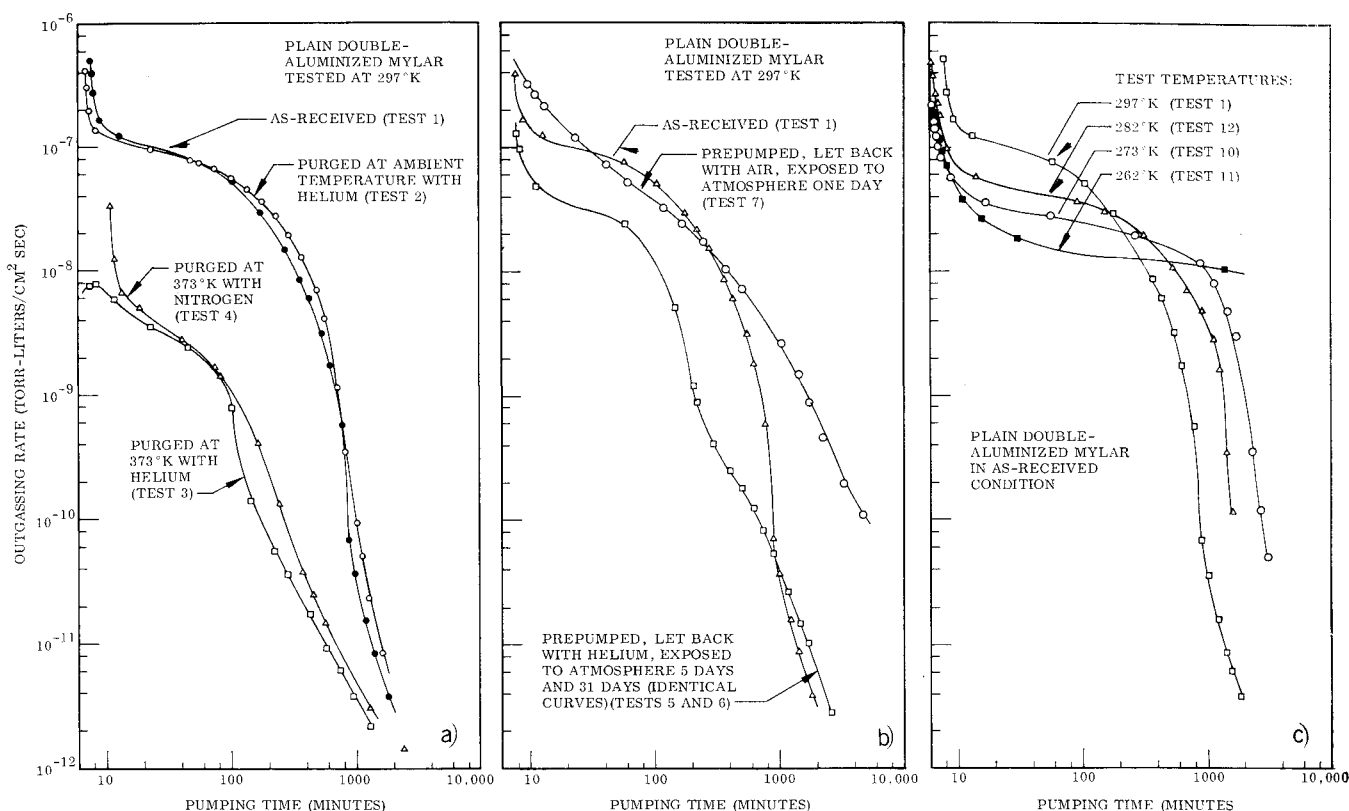


Fig. 2 Outgassing rates for plain double-aluminized Mylar: a) effect of ambient and hot purging, b) effect of pre-pumping, and c) effect of temperature.

Data

The $P(t)$ data were smoothed graphically. Values of dP/dt from the smoothed curves were used in Eq. (3) to find Q_s .

Figure 2a shows outgassing data for as-received, plain double-aluminized Mylar with several types of preconditioning. Test 1 is for the sample in as-received condition. Several regimes of outgassing are evident. At early times a fast-decaying rate due to the removal of loosely bonded molecules gives way to a slower decaying rate more characteristic of gas diffusing out of the bulk material. In test 2 helium was flowed through the insulation sample sheets at 4.2 l/min for 30 min before pumpdown. The data show that ambient temperature purging has a negligible effect on outgassing rate. In test 3 the chamber and sample were heated to 373°K by boiling water in the upper tank and electrically heating the

lower portion. Helium was again flowed through the sample at 4.2 l/min for 30 min before cooling and pumpdown at ambient temperature. Test 4 was similar to test 3 except that the purge gas was nitrogen. The results for the hot purging for the two gases are very similar, showing a reduction in Q_s of between one and two orders of magnitude.

Figure 2b shows four tests performed with the same sample of plain double-aluminized Mylar. At the conclusion of test 1, P was raised back to 1 atm with He. The sample was removed, exposed to the laboratory atmosphere (about 35% humid) for 31 days and repumped as test 5. The outgassing rate is reduced by a factor of about three. The curve is similar in shape to that for the as-received sample. The sample was again let back to 1 atm with He, exposed to the laboratory atmosphere for 5 days and repumped as test 6. The data are indistinguishable from those for test 5 and both tests are represented by the same curve. The sample was then let back to 1 atm with atmospheric air and was repumped after one day's exposure to the laboratory atmosphere as test 7. This test showed an outgassing comparable to that in as-received condition test, but the curve has a different shape, indicating probably that equilibrium sorption conditions had not been reattained. Tests 1, 5, 6, and 7 suggest that pre-pumping is an effective form of preconditioning, at least for short periods, if the sample is let back to 1 atm with He.

Tests 8-12 were conducted on as-received, plain double-aluminized Mylar pumped at various reduced temperatures. No outgassing data were obtained in tests 8 (90°K) and 9 (239°K), because the $P(t)$ histories for the empty and full chambers were indistinguishable due to virtually total suppression of outgassing. The data from tests 10 (273°K), 11 (262°K), 12 (282°K), and 1 (297°K) are presented in Fig. 2b. The coolants used for tests 8-12 were boiling N_2 melting water-acetone solution, melting ice, melting water-ethylene glycol solution, and melting water-glycerine solution, respectively. It is difficult to assess the effect of temperature on Q_s at early times, because the characteristics are somewhat

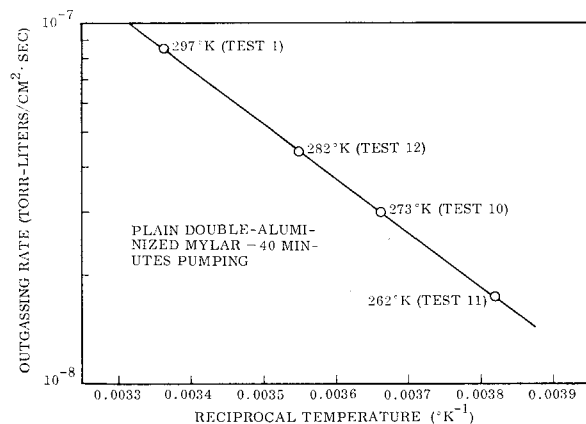


Fig. 3 Variation of outgassing rate of plain double-aluminized Mylar with temperature after 40 min of pumping [a cross plot of Fig. 2c].

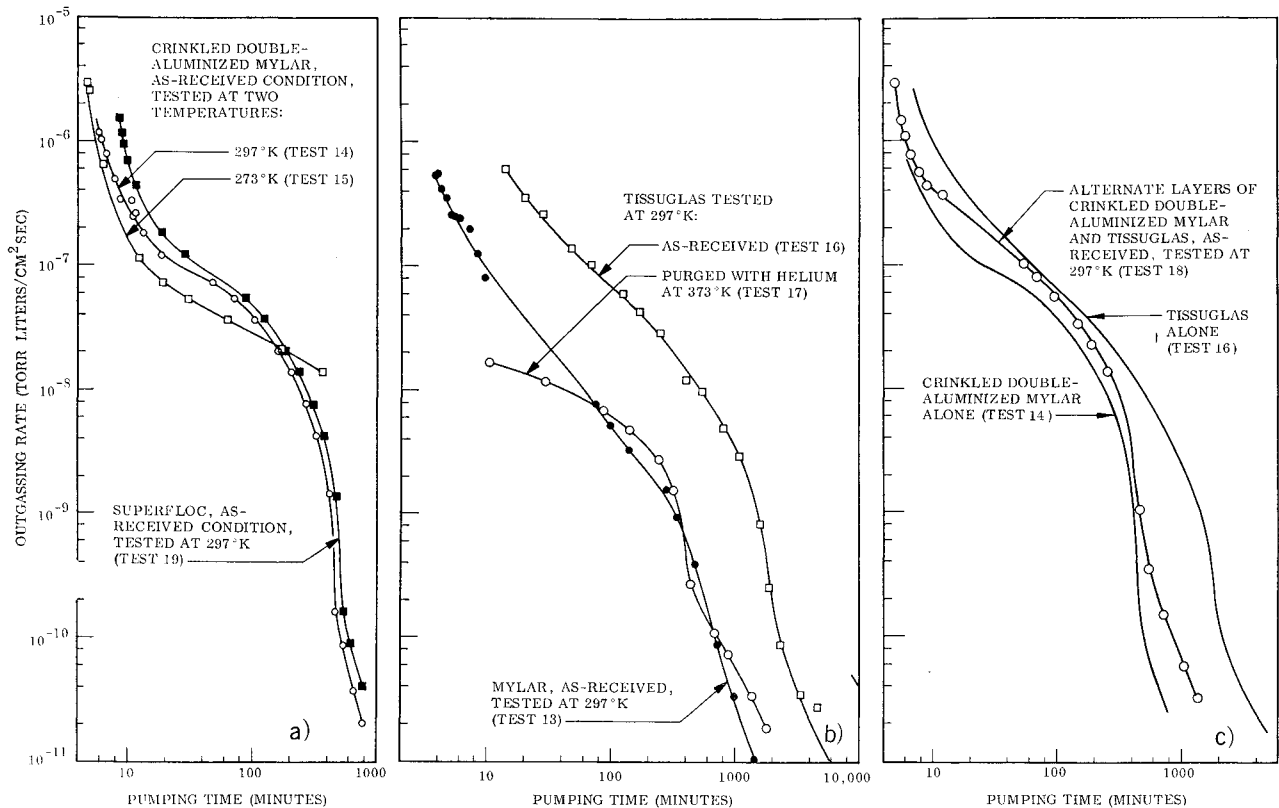


Fig. 4 Outgassing rates of various materials: a) crinkled double-aluminized Mylar at two temperatures, and Superfloc, b) Tissuglas in as-received and hot-purged condition, and plain Mylar, and c) mixed Tissuglas/crinkled double-aluminized Mylar, and the separate components.

confused in this region. The later-time outgassing appears to respond to temperature variation in two respects—the effective initial concentration and the rate of decay of the outgassing rate are both reduced. Figure 3 shows the magnitude of the temperature effect at a pumping time of 40 min, when all four characteristics seem to be in the same regime. Comparison with other data⁴ suggests that this regime probably represents the release of bulk sorbed gas, in which case the slope of the linear characteristic would reflect the temperature dependence of the diffusion coefficient. Quantitatively, the slope should be equal to $-E_d/2R_0$,⁵ where E_d is the activation energy for diffusion, giving a value of E_d equal to 14 kcal/mole. This figure agrees satisfactorily with available data⁵ for the diffusion of water vapor in organic materials.

Figure 4a shows data for as-received, crinkled double-aluminized Mylar, tests 14 and 15 at 297°K and 273°K, respectively. The magnitude of the temperature effect is comparable to that found for plain double-aluminized Mylar. Also shown in Fig. 4a are the data for as-received Superfloc, test 19. Superfloc has a thicker Mylar substrate and is not crinkled. However, the manufacturer is the same—National Metallizing—and it can be seen that the outgassing characteristics are very similar. The major difference is at early times where the outgassing contribution of the Dacron tufts on the Superfloc would be expected to be greatest, due to its high-surface-to-volume ratio.

Figure 4b shows data for as-received and hot-purged Tissuglas, tests 16 and 17. The Q_0 is considerably higher than that of the aluminized Mylar, due mainly to the very high ratio of actual-to-apparent surface area. Hot purging the Tissuglas appears to have roughly the same effect on outgassing rate as was observed for the aluminized Mylar. Also shown on Fig. 4b are data for plain Mylar, obtained from the metallizer, Norton Metallized Products Division, test 13. Approximately the same total quantity of gas is desorbed as in the case of the aluminized Mylar, but the shape of

the curve is different in that there is no well-defined diffusion-controlled region.

Figure 4c shows that the outgassing curve for a mixed sample of Tissuglas and crinkled double-aluminized Mylar lies between the curves for the single components.

Discussion

The data reported are generally comparable to available outgassing data.⁶ No unusual behavior is noted. The outgas was $\geq 98\%$ water vapor.

The outgassing rate can be significantly reduced by preconditioning, with hot purging being more effective than prepumping. However, prepumping is in effect performed during the vacuum deposition process. It would seem that a substantial reduction in as-received outgassing rate could be obtained at little extra cost if, after deposition, the vacuum chamber were raised to 1 atm using an inert gas, dry nitrogen or even dry air, rather than the customary ambient air. Such a possibility should be investigated further.

The results of the prepumping tests also suggest that preconditioning may be performed some time in advance of duty evacuation with no loss of effectiveness. It would be valuable to know just how far in advance of launch preconditioning could be performed. The advantages of preconditioning during assembly or even before lay-up rather than on the launch pad are self-evidently substantial.

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Predicted Effects of Motor Parameters on Solid Propellant Extinguishment

R. L. COATES* AND M. D. HORTON*

Brigham Young University, Provo, Utah

Nomenclature

- A = nozzle area
 B = constant
 c = heat capacity
 C^* = characteristic velocity
 E = activation energy
 K_n = (burning area)/(throat area)
 k = thermal conductivity
 L^* = (chamber volume)/(throat area)
 n = burning rate exponent = $d(\ln \dot{r})/d(\ln p)$
 p = pressure
 r = burning rate
 T = temperature
 x = distance
 α = thermal diffusivity
 γ = ratio of specific heats
 ρ = density
 (\quad) = bar denotes initial steady state

Subscripts

- c = chamber average
 f = flame, final value
 g = gas
 i = initial value
 p = value at p
 s = surface, solid
 ∞ = far beneath surface

Introduction

THEORETICAL studies¹⁻⁴ have shown that burning solid propellants can be extinguished by rapidly reducing the pressure in the combustion chamber. Early experimental studies⁵ showed that if the depressurization process were characterized by either $t_{1/2}$, the time required to reduce the pressure to one-half of the initial steady-state value, or the corresponding average depressurization rate during the time $t_{1/2}$, a reproducible marginal extinguishment condition could be measured. Extinguishment will occur if $t_{1/2}$ is less than some critical value. The models of extinguishment also predict this behavior.

Much of the experimental study of extinguishment by rapid depressurization has been devoted to studying the effect of propellant composition on $t_{1/2}$.^{1,6-8} In addition, the effects of initial pressure and ambient pressure have been investigated. The most recent studies^{4,8} have shown that motor configura-

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* Associate Professor, Chemical Engineering Department.

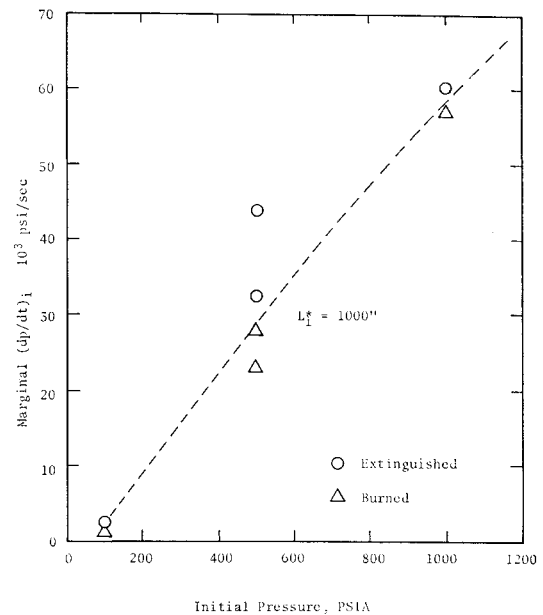


Fig. 1 Predicted effect of varying initial pressure.

tion can have an important effect. This Note presents the results of further theoretical investigation of the effect of motor configuration, in particular the effect of varying the L^* of the motor.

Theory

The transient combustion process leading to extinguishment is assumed to be represented by a one-dimensional temperature profile, with a planar boundary separating the unburned solid and the combustion gases.⁴ The solid is divided into finite-difference elements, and the following energy balance, taken on an element of the solid, is assumed to describe the transient heat conduction beneath the burning surface:

$$\frac{dT_i}{dT} = \alpha \frac{T_{i-1} - 2T_i + T_{i+1}}{(\Delta x)^2} + r \frac{T_{i+1} - T_{i-1}}{2\Delta x} \quad (1)$$

The simplified combustion theory of Denison and Baum⁹ is followed to compute the heat flux to the solid. The flux is related to the burning rate, surface temperature, and flame

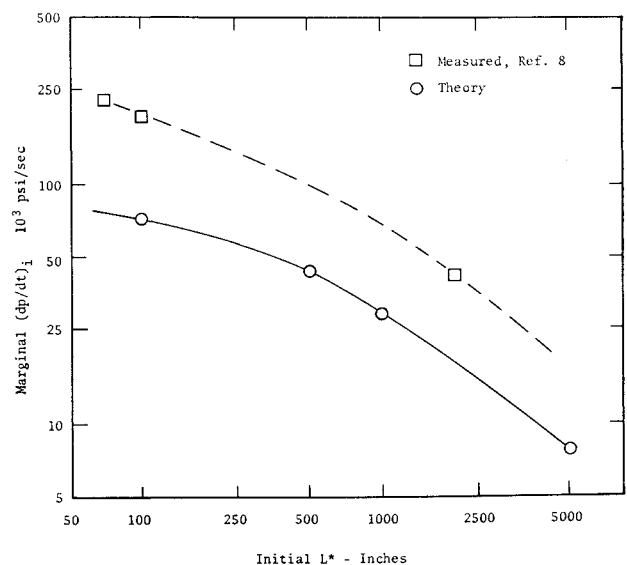


Fig. 2 Predicted effect of varying initial L^* ; recent experimental data are also shown for comparison.