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The Measured Permeability to Nitrogen Tetroxide of Some Potential Bladder Materials

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The objective of the permeability test program was to investigate the resistance of some prospective expulsion bladder materials to permeation and chemical attack by nitrogen tetroxide (N_2O_4). The short-term (24-hr) tests were performed under standard laboratory conditions at an ambient temperature of 70°F.

Obvious chemical attack and changes in flexibility due to contact with N_2O_4 have been noted.

Author

I. INTRODUCTION

A bipropellant liquid rocket engine, to function satisfactorily, must normally be supplied continuous, bubble-free streams of fuel and oxidizer. Assurance that these conditions prevail in a free-fall space environment requires that the propellants be restrained from mixing with the pressurizing gas. This may be accomplished either by applying g-loading or by using an expulsion device. When the latter system is used, the expulsion device, usually in the form of a bag or bladder, becomes an important part of the propulsion system, and the material from which the bladder is made must meet many special requirements. The bladder material must be both compatible with and impermeable to the propellants involved. It must be flexible enough to expel nearly all of the propellant and durable enough to withstand several cycles of operation. It must also yield to fabrication into required shapes which are usually spherical or cylindrical.

To investigate all of the material-selection criteria simultaneously was considered prohibitive. It was therefore decided to base the initial screening of material on permeability and compatibility testing, and to use nitrogen tetroxide as the test fluid. This decision was made for the following reasons:

1. The Jet Propulsion Laboratory's Advanced Liquid Propulsion System (ALPS) program, of which this bladder material development is a part, uses hydrazine and nitrogen tetroxide as one of the candidate propellant combinations. In this system, both bladders must be compatible with both propellants.

2. Nitrogen tetroxide is 100 to 1000 times more active as a permeating fluid than hydrazine (Ref. 1).

3. Permeability testing is effective in measuring short-term compatibility because it requires actual contact between the test fluid and the tested material; consequently, two purposes can be served by a single test procedure.

Most unsatisfactory materials can be eliminated by the tests with nitrogen tetroxide. Those candidate materials withstanding nitrogen tetroxide will have to be tested with hydrazine at a future time.

II. TEST EQUIPMENT AND PROCEDURE

All of the permeability tests were performed[1] with the equipment described and the test procedure outlined in Ref. 1.

In brief, this equipment (Fig. 1) includes two bell-shaped glass chambers of approximately 1¼-in. D, between which the test sample is clamped. The upper chamber is filled with nitrogen tetroxide through a neck which can be capped. Within the lower chamber, an inner cup, which just clears the underside of the installed test sample, is connected through the outer chamber wall to a cold trap. The outlet of the cold trap is connected to a bubble counter which is filled with Fluorolube oil. Both the inlet and the outlet of the cold trap are fitted with stopcocks. A short glass tube is fitted to the outer wall of the lower chamber to serve as a nitrogen (N_2) gas inlet.

Ten ml of nitrogen tetroxide (at approximately 32°F) are injected into the upper chamber. The cap is then fitted to the neck and restrained by a spring strong enough to maintain the vapor pressure of N_2O_4 at 70°F or approximately 15 psia. An N_2 source is attached to the N_2 gas inlet, and gas is allowed to flow through the system at a rate of 60 ml/min. The N_2 gas sweeps across the underside of the test sample and picks up and carries into the cold trap any N_2O_4 which permeates through the test sample. As soon as possible after the flow of gas is started, the cold trap is inserted into a Dewar bottle filled with liquid nitrogen (LN_2) which, because of boil-off, must be replenished at two-hour intervals. The test is considered to begin with the LN_2 filling, and may be terminated at any time by closing the inlet and outlet stopcocks on the cold trap. After termination, the trap is evacuated to 10^{-3} mm Hg, while still in the LN_2. It is then removed and allowed to warm to room temperature. The entrapped material is removed, and the total amount of nitrogen tetroxide is determined by titration (Ref. 1). Evacuation is imperative in this case as a safety precaution to remove residual N_2 and O_2 gas, which can cause the trap to rupture as it is warmed.

[1]The tests were conducted by John B. Krasinsky at The Jet Propulsion Laboratory, Pasadena, California (JPL).

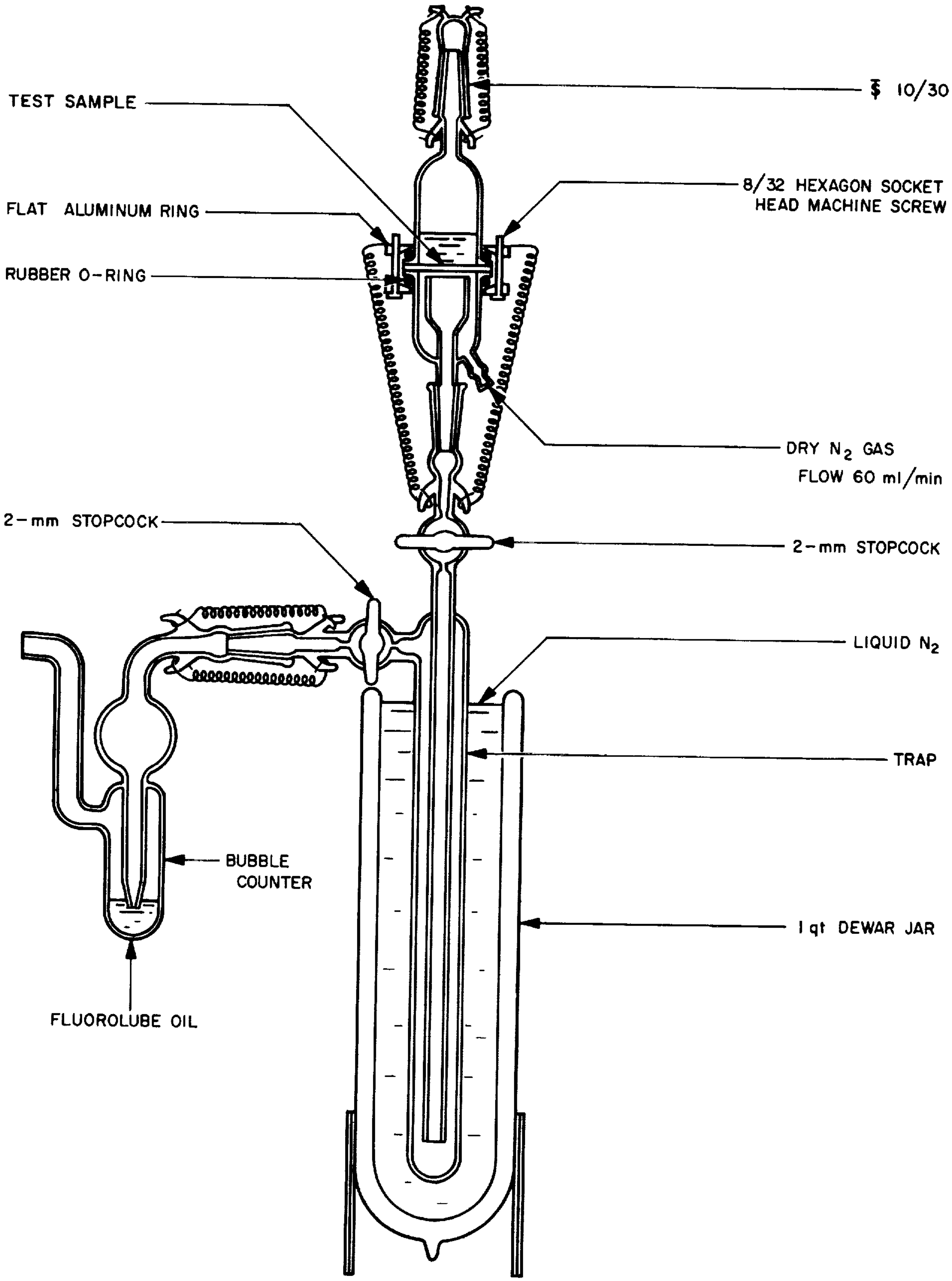

Fig. 1. Permeability test apparatus

III. TEST RESULTS

Results of the permeability tests are shown in Tables 1 through 5.

Table 1. Permeability of Teflon-TFE and FEP

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.²/hr	Remarks
Teflon—FEP Extruded film Type A	Nominal 0.010 Total 0.011[a]	21	0.480	—
	Nominal 0.010 Total 0.011	21	0.420	—
	Nominal 0.005 Total 0.005	24	1.510	Very slight distortion
	Nominal 0.010 Total 0.010	24	0.510	No change
	Nominal 0.020 Total 0.020	24	0.080	
	Nominal 0.030 Total 0.032	18	0.000	
	Nominal 0.020 Total 0.020	24	0.098	
	Nominal 0.020 Total 0.020	24	0.017	
Teflon—FEP Extruded film Type 506	Nominal 0.030 Total 0.032	18	0.000	No change
		18	0.000	
		144	0.146	
		18	0.000	
		24	0.003	
		28	0.007	
		48	0.031	
		18	0.000	
		24	0.002	
		28	0.004	
		48	0.031	
	Nominal 0.020 Total 0.020	24	0.117	No change
Teflon—FEP Extruded film Type 506		24	0.105	
	Nominal 0.010 Total 0.009	24	0.877	
		24	0.728	
	Nominal 0.010 Total 0.011	24	0.930	
		24	0.580	
Teflon—TFE Sprayed dispersion	Total 0.014	24	1.980	No change
		23.5	2.090	
	Total 0.014	24	2.770	
		23.5	2.980	
	Total 0.014	24	2.770	
		23.5	1.810	
	Total 0.018	25.5	2.900	
Teflon—TFE + FEP Sprayed dispersion	Nominal TFE 0.003 Nominal FEP 0.003 Total 0.007	23.5	0.880	No change
		23.5	0.990	
	Nominal TFE 0.003 Nominal FEP 0.003 Total 0.0075	24	1.070	
	Nominal TFE 0.003 Nominal FEP 0.003 Total 0.008	24	1.220	
	Nominal TFE 0.003 Nominal FEP 0.003 Total 0.008	24	1.010	
	Nominal TFE 0.012-0.015 Nominal FEP 0.003-0.004 Total 0.015	24	0.551	Slight bleaching effect

[a] Measured thickness

Table 2. Permeability of metal-plated Teflon

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.^{2}/hr	Remarks
Teflon—TFE Sprayed dispersion Nickel-plated on both sides	Nickel plate 0.0001 Total 0.014	25.5	1.050	Nickel 100% removed from N_2O_4 side
	Nickel plate 0.0001 Total 0.015	25.5	1.720	Nickel 100% removed from both sides
	Nickel plate 0.0002 Total 0.014	25.5	0.050	Nickel 20% removed, Teflon shows through
	Nickel plate 0.005 Total 0.016	25.5	0.000	No apparent change
	Nickel plate 0.0005 Total 0.0175	25.5	0.010	Some etching of metal surface
Teflon—TFE Sprayed dispersion Gold on nickel both sides	Nickel and gold 0.0002 Total 0.0145	24	0.090	Plating 30% removed
	Nickel and gold 0.0002 Total 0.0165	24	0.020	Plating 50% removed
	Nickel and gold 0.0002 Total 0.017	24	0.020	Plating 30% removed
Teflon—TFE Sprayed dispersion Gold plated on both sides	Gold 0.0001 Total 0.013	24	1.880	No change
	Gold 0.0001 Total 0.014	24	1.120	No change
Teflon—TFE Sprayed dispersion Aluminum plated on one side	Total 0.014	24	1.740	Plating 75% removed
	Total 0.014	24	1.590	Plating 75% removed
	Aluminum < 0.0001 Total 0.016	24	1.720	Discolored, turned iridescent blue
	Aluminum < 0.0001 Total 0.015	24	1.700	Discolored, became dark
Teflon—TFE Sprayed dispersion Aluminum plated on both sides	Aluminum < 0.0001 Total 0.017	24	1.240	Discolored, became dark
Teflon—TFE Sprayed dispersion Chemical plated on both sides with nickel, gold and aluminum	Plate/side 0.00015 Total 0.017	24	0.560	Discolored
	Plate/side 0.00015 Total 0.0175	24	0.021	Discolored
	Plate/side 0.00015 Total 0.016	24	0.033	Discolored
Teflon—TFE Sprayed dispersion Chemical plated on both sides with nickel and aluminum	Plate/side 0.0002 Total 0.016	24	0.064	Slightly discolored
Teflon—TFE Sprayed dispersion Chemical plated on both sides with gold and aluminum	Plate/side 0.00001 Total 0.015	24	1.650	Discolored
	Plate/side 0.0001 Total 0.016	24	1.310	Discolored
Teflon—TFE + FEP Sprayed dispersion Chemical plated gold in multiple laminate	TFE 0.009-0.011 FEP 0.003-0.004 Gold 0.002-0.003	24	0.235	No change
	TFE 0.009-0.011 FEP 0.003-0.004 Gold 0.002-0.003	24	0.333	
	Total 0.015	24	0.380	
	Total 0.0145	24	0.420	
	Total 0.013	24	0.430	
	Total 0.013	24	0.430	
	Total 0.012	24	0.410	

Table 2. Permeability of metal-plated Teflon (Cont'd)

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.^{2}/hr	Remarks
Teflon—TFE + FEP Sprayed dispersion Chemical plated gold in multiple laminate	Total 0.012	24	0.340	No change
	Total 0.012	24	0.280	
	Total 0.015	24	0.190	
	Total 0.015	24	0.150	
Sprayed FEP-stainless steel composite	Total 0.007	24	0.960	No change
Sprayed FEP-aluminum composite 6-layer	Total 0.011	24	0.340	No change
Sprayed FEP-aluminum composite 6-layer	Total 0.011	24	0.250	No change
Sprayed FEP-aluminum composite 4-layer	Total 0.011	24	0.280	No change

Table 3. Permeability of Teflon-metal foil laminates

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.²/hr	Remarks
Teflon-aluminum laminate polyester adhesive	Teflon 0.001 Aluminum 0.001 Total 0.003	18	0.000	—
	Teflon 0.001 Aluminum 0.00018 Total 0.003		All came through	Adhesive turned yellow, reaction with N_2O_4
	Teflon 0.001 Aluminum 0.00035 Total 0.002	18	0.259	Adhesive turned yellow, reaction with N_2O_4
Teflon-aluminum laminate epoxy adhesive	Teflon 0.001 Aluminum 0.001 Total 0.006	18	0.000	
	Teflon 0.001 Aluminum 0.00018 Total 0.005		All came through	Adhesive turned yellow, reaction with N_2O_4
	Teflon 0.001 Aluminum 0.00035 Total 0.004	18	0.000	
Teflon-aluminum laminate FEP—Teflon heat bonded to both sides of aluminum foil	Teflon 0.005 Aluminum 0.0007 Total 0.011	24	0.007	Complete separation of Teflon film from aluminum
	Teflon 0.002 Aluminum 0.001 Total 0.006		All came through	Some separation of Teflon film
	Teflon 0.002 Aluminum 0.001 Total 0.006		All came through	Some separation of Teflon film
	Teflon 0.005 Aluminum 0.0007 Total 0.011	24	0.003	Some separation of Teflon from aluminum
	Teflon 0.00025 both sides Aluminum 0.001 Total 0.0015	24	0.000	Slight shrinkage and wrinkling
	Teflon 0.00025 both sides Aluminum 0.001 Total 0.0015	24	0.000	Slight shrinkage and wrinkling
Teflon-aluminum laminate FEP—Teflon heat bonded to one side of aluminum foil	Teflon 0.005 Aluminum 0.0015 Total 0.007	24	No measurement taken	Complete separation of Teflon film from aluminum
	Teflon 0.005 Aluminum 0.0007 Total 0.0065	21	0.000	No change
	Teflon 0.005 Aluminum 0.0007 Total 0.006	24	0.008	
	Teflon 0.005 Aluminum 0.001 Total 0.0065	18	0.074	
	Teflon 0.005 Aluminum 0.0015 Total 0.007	24	0.004	
		21	0.000	
	Teflon 0.005 Aluminum 0.002 Total 0.007	18	0.000	
	Teflon 0.0005 Aluminum 0.0005 Total 0.001	24	0.000	Some wrinkling due to shrinkage of Teflon
		24	0.000	Some wrinkling due to shrinkage of Teflon
Teflon-aluminum laminate—aluminum foil heat bonded to both sides of FEP Teflon	Teflon 0.005 Aluminum 0.0007 Total*	24	0.000	No change
		24	0.005	
		48	0.009	
	Teflon 0.005 Aluminum 0.001 Total 0.0075	18	0.000	
	Teflon 0.005 Aluminum 0.0015 Total 0.008	24	0.004	—
	Teflon 0.005 Aluminum 0.002 Total 0.0085	24	0.006	—

*Not available

Table 3. Permeability of Teflon-metal foil laminates (Cont'd)

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.^{2}/hr	Remarks
Teflon-aluminum overlap seam, heat bonded Teflon joint	Teflon 0.010 Aluminum 0.001 Total 0.0225	96	0.003	50% separation of Teflon film from aluminum
		96	0.001	90% separation of Teflon film from aluminum
Teflon-tantalum laminate FEP Teflon heat bonded to tantalum foil	Teflon 0.002 Tantalum 0.003 Total 0.005	24	0.000	N_2O_4 against Teflon delaminated
		24	0.000	N_2O_4 against tantalum No change

Table 4. Permeability of metal-to-metal seals made by ultrasonic welding

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.²/hr	Remarks
Seam in aluminum foil—ultrasonic weld	0.001	24	0.00	—
			0.00	—
			0.00	—
			0.17	Leaked at welded seam (no pinholes found by photo-check)[a]
			0.00	
			0.07	Apparently leaked through pinhole (pinhole found by photo-check)[a]
			0.07	
Advertising sample ultrasonic weld	0.001	24	0.07	— —
Aluminum foil ultrasonic cross weld—heat bonded to TFE both sides	Aluminum 0.0025 TFE 0.003	—	Liquid N_2O_4 came through	Very poor sample of ultrasonic welding
Aluminum foil (hard condition)	0.0025	24	0.01	Too stiff
Aluminum foil (hard condition)	0.0025	24	0.05	Too stiff

[a]A method of locating pinholes in aluminum foil using photographic paper and a high-intensity light source.

Table 5. Permeability of miscellaneous materials

Material	Thickness, in.	Duration of test, hr	Permeability rate, mg/in.²/hr	Remarks
Teflon—FEP 120 on glass-electrical	—	18	3.83	—
High temperature H-Film	—	—	All came through	Material ruptured
Mylar on aluminum foil	Total 0.002	24	1.04	Very wrinkled after test
Aclar	Total 0.001	—	All came through	Material intact but distorted
Aclar	Total 0.005	—	All came through	Material intact but distorted
Teflon—FEP vapor-deposited gold	Total 0.002	18	7.58	Gold 100% removed, film distorted
	Total 0.005	24	1.87	Gold 30% removed, slight distortion of film
Teflon—FEP Butyl rubber one side	Total 0.012	24	1.09	Rubber removed under O-ring. Blister form, slight distortion
	Total 0.021	24	0.154	Slight rubber removal and blistering, slight distortion
Kynar	Total 0.014	23.5	2.41	Sample distorted
	Nominal 0.009 Total 0.010	23.5	4.23	Sample distorted
	Nominal 0.006 Total 0.007	23.5	10.72	Sample distorted
Armalon TFE cloth FEP film vapor-deposited aluminum	Total 0.011	24	0.73	Some bleaching, aluminum 100% removed

IV. DISCUSSION

Previous permeability tests have proven that some elastomeric materials are clearly not compatible, and hence are subject to deterioration by N_2O_4 or the nitric acid (HNO_3) formed when N_2O_4 combines with water or water vapor from the atmosphere. Other materials were known to swell excessively in N_2O_4, indicating absorption and subsequent permeability. Known facts of polymer chemistry indicate that polymeric materials with other than perfluorinated carbon chains are not likely to be compatible for extended periods of time in N_2O_4, regardless of permeability characteristics. Some materials of this kind were examined when it was determined that no previous tests had been conducted.

These tests, together with a practical consideration of availability, limited the field to the following materials: TFE Teflon, FEP Teflon, Aclar, Kynar, Mylar, aluminum foil, various metals plated on Teflon, and combinations of the above materials with miscellaneous items which became available as testing progressed.

A. Teflon-TFE and FEP

TFE Teflon (polytetrafluoroethylene) has been, for a number of years, the accepted expulsion bladder material for use with corrosive storable propellants. It was used, although less than satisfactorily, because it had the advantage of compatibility and could be fabricated into seamless bags of any shape, for which an aluminum mandrel could be made by a dispersion spraying and sintering process. Two serious drawbacks have been excessive stiffness (which causes failure on cycling), and permeability (especially to the nitrogen containing oxidizers). Permeability rates with N_2O_4 average 3 mg/in.^{2}/hr for sprayed dispersion material 0.010-in. thick. While at first glance this may not seem excessive, it amounts to approximately 160 l of oxidizer during a period of one year from a hemispherical bladder of ALPS size (61-in. D) assuming uniform permeation over the whole surface and throughout the entire time. Any such loss can be serious, for the amount that collects on the outside of the bladder is unavailable for use when needed and must be considered to be dead weight.[2] In the ALPS system, this condition is doubly serious since the permeating propellant vapors can freely migrate from one propellant bag compartment to the other with obvious undesirable results.

In 1961, the dispersion of FEP Teflon (fluoroethylene propylene), a thermoplastic copolymer of tetrafluoroethylene and hexafluoropropylene, became available. This material, which sinters at a lower temperature (550°F) and into a more cohesive film than TFE Teflon (650°F-700°F), proved to be less permeable to N_2O_4 than films made from TFE dispersion. The flexibility and compatibility characteristics remained effectively the same as those of TFE Teflon, but the FEP material lacked strength. By combining TFE and FEP dispersions into a multilayered laminate coating over a soluble mandrel, film ranging from 0.004 to 0.020-in. thick was made. This film incorporated the favorable characteristics of both types of Teflon[3]. The permeability rate to N_2O_4 was at least equivalent to that of FEP (in the order of 1 mg/in.^{2}/hr for 24-hr periods). Although this was a significant improvement over TFE alone, it was not considered adequate as a bladder material for use in the long-term storage of N_2O_4. Films impermeable to N_2O_4 were required, and therefore it was obvious that polymers alone could not be used. With this in mind, efforts were initiated to combine compatible polymers with metals in ways designed to further reduce or even eliminate permeability.

B. Metal Plate on Teflon

Tests indicated that certain metal foils offered the necessary impermeability to N_2O_4. However, in addition to obvious fabrication problems, they lacked strength and resistance to tearing. It therefore became desirable to combine the favorable qualities of metal impermeability with the flexibility and toughness of polymeric films. One method of joining these materials was to chemically plate various metals and combinations of metals directly onto Teflon film until an impervious barrier was achieved; the other was to laminate Teflon-FEP to a metal foil (usually aluminum) either by means of adhesives or heat bonding.

The plating technique was investigated, under contract to JPL, by the Joclin Manufacturing Company of Wal-

[2]It is believed that N_2O_4 will continue to permeate through TFE Teflon indefinitely until equilibrium is reached on the downstream side. To what point permeation continues, and where a state of equilibrium may be reached in a tank and bladder system where reactions with other chemical elements can occur, has not been proven at this writing.

[3]Details of this process are considered proprietary by some vendors and will not be discussed here.

lingford, Conn. This effort involved the chemical plating of such metals as nickel, gold, and aluminum onto TFE Teflon in various thicknesses and combinations, and the development of a film, comprised of multiple layers of colloidal gold alternated with layers of TFE and FEP Teflon. The plating technique was thought to be promising because it utilized the existing, one-piece bladder fabrication methods, with the addition of only the various plating operations.

The main problems, aside from the inherent inflexibility and tendency toward porosity of plated metal, were to sensitize the Teflon surface for plating and to obtain adequate cohesive strength between Teflon and the plated metal. The technique was claimed by Joclin as proprietary information at the initiation of the contract. Many samples were produced using this technique and then tested for permeability by Joclin and JPL. Joclin measured permeability with helium, using the technique and equipment described in American Society for Testing Materials D-1434-58. JPL used the system previously described in this report. Figure 2 presents a comparison of data by the two test methods on identical samples, and indicates that reasonably good correlation exists between the two methods.

In general, external plating proved unsatisfactory. Although some very low permeability rates were achieved, the necessary plate thickness made the laminate too stiff to be considered for expulsion bladder material. Nickel was the most impermeable, but it proved to be incompatible with N_2O_4 or the HNO_3 formed upon contact with moisture in the atmosphere, and was generally attacked and removed. Plate combinations which included nickel reacted in much the same way. Gold by itself adhered well, but seemed to decrease permeability only slightly. An adequate cohesion between Teflon and aluminum was not obtained.

Multiple lamination of TFE-FEP and gold appears promising. Permeability rates were lowered to 0.2 to 0.4 mg/in.^{2}/hr (Table 2), and the material is tough and no less flexible than an equal thickness of TFE-FEP dispersion. At this writing, no further improvements have been made with this material, although the contract with Joclin has not yet expired.

C. *Teflon-Metal Foil Laminates*

The other method of creating a metallic barrier involved the lamination of aluminum foil in several thicknesses with various thicknesses of FEP Teflon film. Two methods of bonding were attempted by two different vendors.

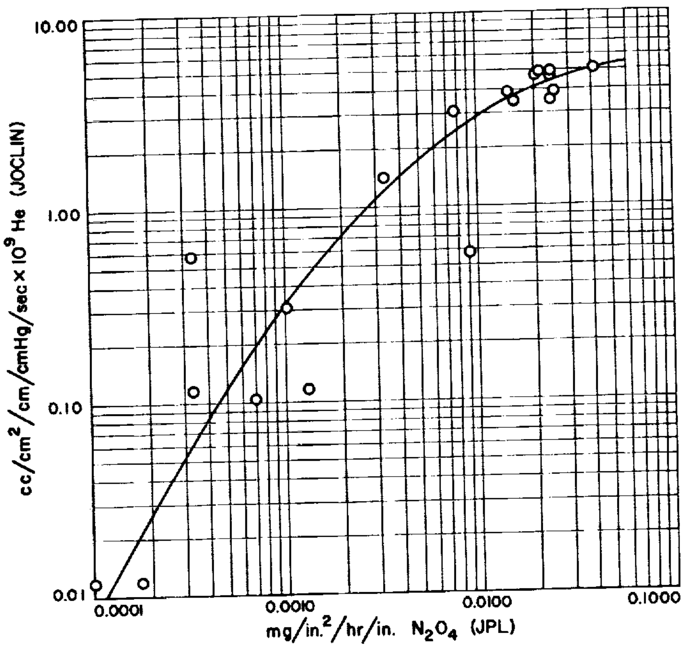

Fig. 2. Correlation of ASTM D-1434-58 standard helium permeation test results as reported by Joclin Mfg. Co. with results of JPL N_2O_4 permeation tests on samples from identical materials. (Teflon or metal plate on Teflon)

The G. T. Schjeldahl Company of Northfield, Minnesota, under contract to JPL, produced samples using 0.001-in. FEP Teflon and 0.00018 to 0.001-in. aluminum foil, bonded by both epoxy and polyester adhesives. These bonds were successful and the samples remained impervious to N_2O_4 as long as the aluminum foil had no pinholes or ruptures. When pinholes were present, both the epoxy and polyester resins were attacked by the N_2O_4, and extensive damage resulted. From tests, it was determined, however, that aluminum foil as thin as 0.00018 in. could be an impervious barrier to N_2O_4. This method of bonding Teflon and aluminum foil was abandoned because of the incompatibility of the adhesives.

Swedlow Inc., of Los Angeles, under contract to JPL, has been able to heat bond Teflon and aluminum foil in various thicknesses and combinations that have proven impervious to N_2O_4 under the test conditions previously discussed. Initially, some difficulty was encountered with separation of the Teflon film from the aluminum foil when the Teflon was in contact with N_2O_4. This condition was corrected by an adequate cleaning of the aluminum surface.[4] Handling and inspecting the aluminum foil is a critical part of the process, because a barrier impervious to N_2O_4 is created only when the aluminum foil is free from pinholes or other defects.

Teflon-FEP bonded to 0.003-in. thick tantalum proved to be impervious to N_2O_4, but adhesive qualities were poor, and the laminate separated. It is felt that a sufficient number of samples were not available to adequately test this combination, and that further experimentation could solve the separation problem.

D. Seams and Joints

The ultrasonic welding technique was investigated as a means of fabricating the seams where the segments of a Teflon-aluminum foil laminate bladder are joined. Tests with samples of 0.001-in. thick aluminum foil have proven that a continuous metal-to-metal weld can be made that is impervious to N_2O_4, within the limits of the test method used (Ref. 1). Eight of the ten samples tested showed no detectable permeation. The problems inherent with this fabrication process will become apparent when it is applied to the construction of bladders, for the welding must be done between a weld head or horn and an anvil located beneath the work. A satisfactory method of positioning and manipulating this anvil while welding the closing seam of a bladder is yet to be developed.

Heat-bonded seams in Teflon aluminum laminate proved unsatisfactory because of their tendency to separate. Further developmental work could probably eliminate this problem. However, seams produced by this method which approached satisfactory permeability rates tended to be very stiff. This method was abandoned in favor of the ultrasonic welding technique. See Table 4 for permeability data.

E. Miscellaneous

Several other materials were tested. These included Mylar, Kynar (polyvinylidene fluoride), impregnated Teflon, laminates of FEP Teflon and butyl rubber, laminates of TFE woven cloth, FEP film and vapor deposited aluminum, and polypropylene. Varying results were obtained, none of which was satisfactory. Near the end of testing, some newly developed composite materials of finely divided aluminum or stainless steel (mixed and sprayed in layers of FEP dispersion) became available. Initial tests have shown permeability rates of 0.2 to 0.3 mg/in^2./hr, but flexibilities are less than those of equivalent thicknesses of TFE-FEP dispersion film. These materials appear interesting in that their application to bladder fabrication should not complicate current manufacturing techniques. See Table 5 for permeability data.

[4]It has been determined that proper preparation of the aluminum surface is essential to obtaining a satisfactory bond between FEP Teflon and aluminum foil. Boeing Aircraft Company metal surface treatment Spec. 5755 has been recommended. Subsequent testing by Swedlow and JPL has shown that most standard aluminum degreasing and cleaning procedures will provide surface conditions adequate for a Teflon-aluminum bond that will not separate upon contact with N_2O_4 for 24-hr periods.

Attempts to bond Aclar to aluminum foil were initially unsuccessful and were not pursued, since no advantage over Teflon-aluminum laminate was apparent. See Table 3 for permeability data.

V. CONCLUSION

None of the polymeric materials or combinations covered in this report are sufficiently impermeable to be used as bladders for long-term storage of N_2O_4.

Chemical metal plating can be applied to Teflon, but in thicknesses that appreciably lower the permeability rate, the material becomes too inflexible and brittle for use in expulsion bladders.

Teflon-aluminum laminates, which are impermeable to N_2O_4 within the limitations of the test described in this report, can be fabricated by the heat-bonding process.

A continuous hermetic seam in aluminum foil (0.001-in. thick) can be generated by the ultrasonic welding process.

Teflon-aluminum laminates, produced by the adhesive bonding technique, will not be satisfactory until adhesives which are unaffected by N_2O_4 and N_2H_4 become available.

REFERENCE

1. Vango, Stephen P., *Determination of Permeability of Cast Teflon Sheet to Nitrogen Tetroxide and Hydrazine,* Technical Memorandum No. 33-55, Jet Propulsion Laboratory, Pasadena, August 25, 1961.