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Technical Report 32-1491

*Spacecraft Polymeric Material Interactions During
Decontamination, Sterilization and Thermal
Vacuum Exposures*

W. D. Roper

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JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

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Preface

The work described in this report was performed by the Engineering Mechanics Division of the Jet Propulsion Laboratory.

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Abstract

An investigation was made of twelve pairs of typical spacecraft polymeric products to determine possible incompatibilities between the paired products which may occur during a spacecraft sterilization process and a spacecraft thermal-vacuum environment. The paired products were each placed in intimate contact and then given exposure to the following three environments:

- (1) Ethylene oxide-Freon 12 decontamination
- (2) Thermal sterilization
- (3) Long term thermal-vacuum exposure

Following the sequence of exposures, the products were given mechanical and electrical property tests in order to establish possible material degradation occurring during the exposures.

Of the twelve material combinations studied, two were found to be incompatible. The incompatibility appeared to be directly attributed to an interaction between the paired products. The incompatible combinations involved a polyester film paired with a polyurethane product and the same polyester paired with a silicone elastomeric compound. A possible mechanism for the degradation process is presented.

Spacecraft Polymeric Material Interactions During Decontamination, Sterilization and Thermal Vacuum Exposures

I. Introduction

One of the major scientific gains to be achieved from planetary exploration is the investigation of the existence of extraterrestrial life forms. To accomplish this it has been well recognized that a planetary quarantine must be imposed and that some form of preflight biological sterilization must be performed on the spacecraft in any entry/lander mission. This requirement also exists in any case where there is some possibility of accidental landing of mission hardware at any extraterrestrial location except the moon.

As a result of this planetary quarantine requirement, all the materials within a spacecraft must therefore be compatible with the preflight sterilization process. This is particularly true of the many polymeric products used in spacecraft fabrication. These include such products as adhesives, films, sealants, encapsulant, etc. The Jet Propulsion Laboratory (JPL) specified preflight sterilization process includes decontamination by treatment with a gaseous mixture of Freon and ethylene oxide (ETO), followed by a thermal sterilization in a dry inert atmosphere.

Additionally, the polymeric products must be compatible with the spacecraft environment existing during the particular mission. In general, this environment has been defined as the thermal-vacuum condition existing within the spacecraft or satellite, usually pressure less than 10^{-5} torr and temperature less than 135°C (Ref. 1).

Several studies (Refs. 1-8) have been made to establish those polymeric products which are compatible with the sterilization and spacecraft thermal-vacuum environments. In these studies, the interaction between the exposure environment and the individual product was the prime concern. No studies were made to establish incompatibilities arising out of intimate contact between dissimilar products during the sterilization and thermal-vacuum exposures. Such contact is a very real situation in any spacecraft design. Contact between films and structural polymeric products and between films and polymeric coatings are only two of the many possible contacts that may exist within the spacecraft.

Interactions between the dissimilar materials may be the result of physical or chemical processes occurring as

a result of their close contact. For example, low molecular weight polymer migration or exchange may occur between the materials with the resultant degradation in properties of either or both materials. At the same time the interactions may be chemical in nature and follow the diffusion of constituents from the one product into the other. It is likely that an interaction process could be very pronounced during the rather rigorous sterilization exposures to which the products are subjected. Hydrolytic effects during decontamination could be expected to accelerate any degrading interaction. The space thermal-vacuum environment could also be expected to promote these processes due to volatility considerations and the temperature involved.

It was therefore the purpose of the present study to explore, to a limited extent, some of the material combinations which were thought to be potentially incompatible. The products selected were typical spacecraft materials. They were, in general, materials which have been found to be, by themselves, compatible to the sterilization and space thermal-vacuum exposures. The products selected for evaluation in this study are those shown in Table 1. Each product was combined with a second material and while in intimate contact the two were subjected to the following sequence of exposures:

- (1) Six decontamination cycles of a humidified ETO-Freon 12 atmosphere at 50°C for 30 h each.

- (2) Six thermal-sterilizing cycles of a dry nitrogen atmosphere at 135°C for 96 h each.
- (3) One thermal-vacuum exposure for 500 h under conditions of 135°C temperature and 10⁻⁶ torr pressure.

By the performance of physical, mechanical, and electrical property tests on each product, both before and after the exposures, a measure of the stability of the product was obtained. Gross degrading interactions between products would be reflected in the degree of retention of their key properties.

II. Experimental Section

A. Sample Preparation and Testing

Twelve combinations or pairs of materials were selected for investigation in these studies. The pairs were selected on the basis of their potential for incompatibility due to interactions. The material combinations are shown in Table 2. In each pair one material, designated as the test material, was placed in close contact with the secondary material. The contacting materials were then given exposure to the decontamination, sterilization and the thermal vacuum environments. Close contact between the two materials was accomplished by sandwiching test specimens of the test material between sheets of the

Table 1. Tested products

Product (trade name)	Polymer type	Manufacturer	Use
Epon 828/Z	Epoxy	Shell Chemical Co., Pittsburg, Calif.	Adhesive
Solithane 113/300	Polyurethane	Thiokol Chemical Corp., Trenton, N.J.	Coating
DC93-500	Silicone	Dow Corning Corp., Midland, Mich.	Encapsulant
Mylar 100A	Polyester	E. I. duPont de Nemours and Co. (Inc.), Wilmington, Del.	Film
Kapton 200 X H667	Polyimide	E. I. duPont de Nemours and Co. (Inc.), Wilmington, Del.	Film
PPO 534-801	Polyphenylene oxide	General Electric Co., Pittsfield, Mass.	Hardware and structural parts
Micarta 20201-2	Silicone	Westinghouse Electric Corp., Micarta Div., Hampton, S.C.	Hardware and structural parts
Micarta H5834	Phenolic	Westinghouse Electric Corp., Micarta Div., Hampton, S.C.	Hardware and structural parts

Table 2. Material test matrix

Test material	Secondary material	ASTM test standard						
		D1002 for adhesive lap-shear strength	D2294 for adhesive creep	D882 for tensile strength	D1004 for tear strength	D638 for tensile strength	D257 for volume resistivity	D149 for dielectric strength (short time)
Epon 828/Z	Solithane 113/300	✓	✓					
Epon 828/Z	DC93-500	✓	✓					
Mylar 100A	Solithane 113/300			✓	✓		✓	✓
Mylar 100A	DC93-500			✓	✓		✓	✓
Mylar 100A	Micarta H5834			✓	✓		✓	✓
Kapton 200X H667	Micarta H5834			✓	✓		✓	✓
Kapton 200X H667	Solithane 113/300			✓	✓		✓	✓
Kapton 200X H667	DC93-500			✓	✓		✓	✓
PPO 534-801	DC93-500					✓	✓	✓
Micarta H5834	Solithane 113/300					✓	✓	✓
Micarta 20201-2	Solithane 113/300					✓	✓	✓
Micarta H5834	DC93-500					✓	✓	✓

secondary material. Intimate, uniform contact was obtained by clamping the polymer sandwich between perforated stainless steel plates (37% open area, 0.083-in. diam perforations) using a torque of 5 in.-lb on 3/8-in. stainless steel bolts and spring washers. Figure 1 shows the construction of a typical assembly. This technique was not used with the Epon 828/Z adhesive combinations. In these cases, the lap shear and creep test specimens of the adhesive were coated with 5-10 mil of the secondary material (Solithane 113/200 or DC 93-500).

Table 2 also shows the types of specimens that were prepared of each test material. These specimens were prepared in accordance with sizes and shapes specified in the particular ASTM¹ test method used. For each test method a minimum of six specimens (three controls and three exposed specimens) were prepared.

The sequence of the exposures and testing is shown in Figure 2. As shown in this sequence the exposed specimens received ETO decontamination, thermal sterilization and finally a long term thermal-vacuum exposure. They received all of these exposures while they were

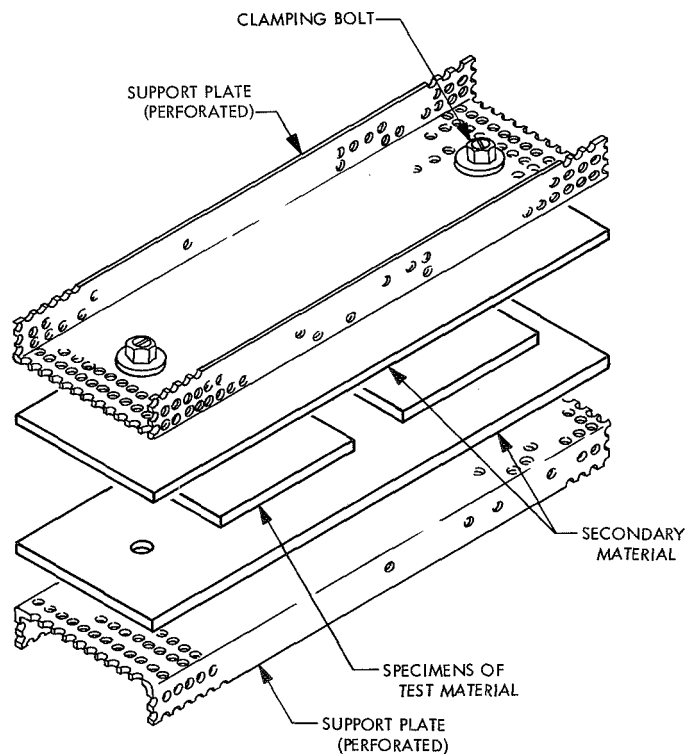


Fig. 1. Typical test specimen assembly

¹American Society for Testing Materials.

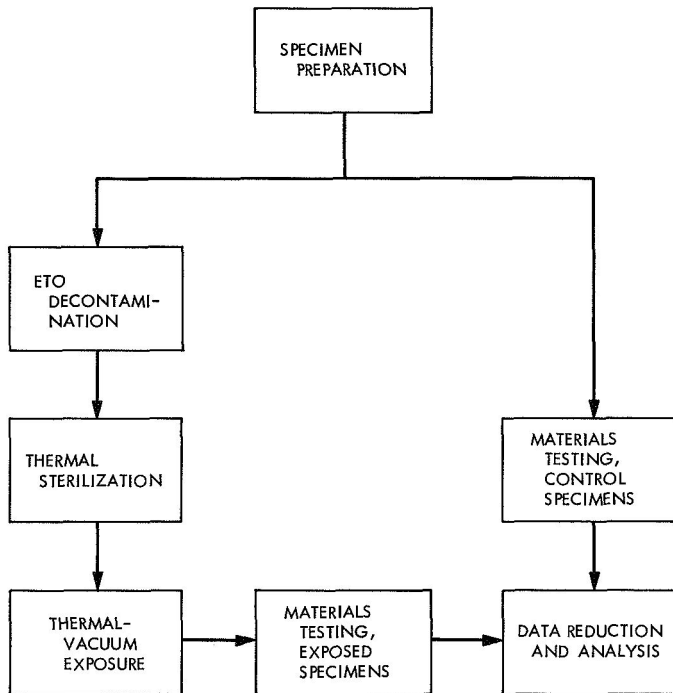


Fig. 2. Exposure and test sequence

assembled in close contact with the secondary materials as described above. After the exposures, ASTM tests were performed on the control and exposed specimens. The total effect of the secondary material and the exposure sequence could then be determined on each test material.

B. Exposure Equipment and Procedures

The exposure equipment used included an ETO-Freon 12 decontamination chamber, thermal sterilization ovens and a thermal vacuum exposure unit.

1. ETO-Freon 12 decontamination. The decontamination chamber used is shown in Fig. 3. This unit was a specially constructed gas sterilizer which was designed to perform the ETO-Freon 12 decontamination procedure as detailed in the JPL specifications.

Each complete decontamination process consisted of six identical exposure cycles. Each of these were automatically performed in the following steps:

- (1) Humidification, at $50 \pm 5\%$ relative humidity (RH), and heating of the test specimens in clean filtered air for approximately 1 h. The final chamber temperature after the conditioning was controlled at $50 \pm 2^\circ\text{C}$.

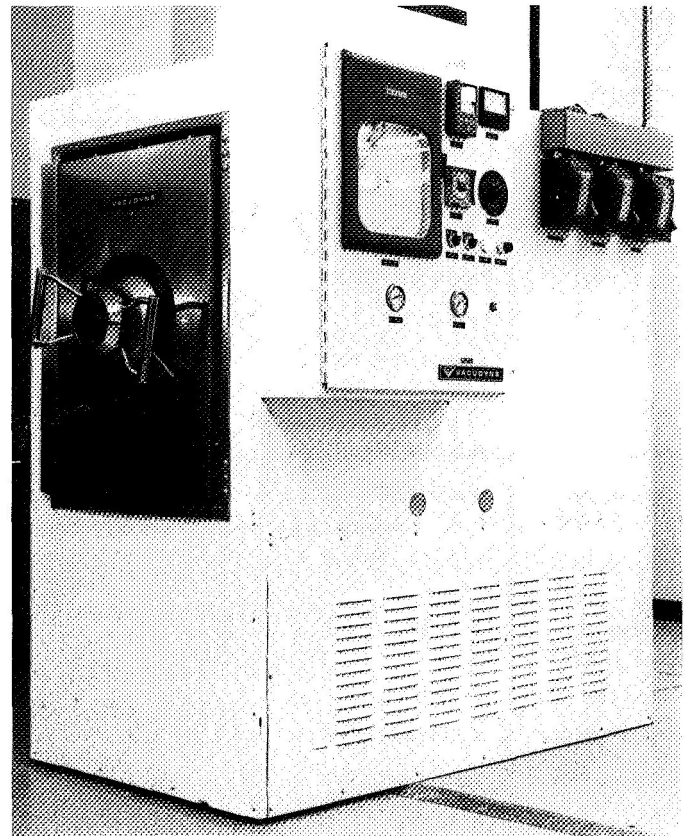


Fig. 3. Ethylene oxide decontamination unit

- (2) Evacuation of the chamber to a vacuum of greater than 27 in. Hg (within approximately 10 min).
- (3) ETO-Freon 12 decontamination for 28 h at 600 ± 50 mg/l ETO concentration, $50 \pm 2^\circ\text{C}$ and $50 \pm 5\%$ RH.
- (4) Chamber cooling to $20\text{--}25^\circ\text{C}$ (within approximately 45 min) followed by re-evacuation to greater than 27 in. Hg vacuum (approximately 10 min).
- (5) Application of an air wash with filtered ambient air for 25 min.

It should be noted that each material combination was exposed separately inside the chamber during a decontamination process.

2. Thermal sterilization. The thermal sterilization was performed in either one of three ovens: two were National Appliance Model 5830, and the third a Blue M Co. Model POM-16 VB. Each oven was fitted with a nitrogen manifold system that permitted a continuous dry nitrogen purge through the sterilization chamber

during the specimen exposures. Each was also fitted with a power rheostat which controlled the chamber warmup rate within the limits specified in JPL sterilization specification. An automatic timing device controlled the duration of each sterilization period.

The thermal sterilization process consisted of six separate 96-h cycles at 135°C. All material combinations were sterilized separately. A typical cycle is shown in Fig. 4. The following procedure was followed in each cycle:

- (1) Test specimens were placed in the sterilization chamber which was stabilized within a temperature range of 20–25°C.
- (2) Dry nitrogen was continuously purged through the chamber at either 3 ft³/h (Blue M oven) or 1.7 ft³/h (National Appliance oven). The chamber was heated at the approximate rate of 56°C/h until a temperature of 135 ± 2°C was attained.
- (3) When the chamber temperature reached 135°C, the sterilization period was started.
- (4) After 92 h of sterilization, the chamber was allowed to cool to 20–25°C (within approximately 2 h). During this time the nitrogen purge was maintained.
- (5) The sterilization cycle was repeated six times per load of specimens. Following cycle No. 6, the specimens were removed from the chamber.

3. Thermal-vacuum exposure. The thermal-vacuum exposure apparatus consisted of a multiple-cell test unit accommodating 30 sample cells. This unit is shown in Figs. 5 and 6. The test unit was so designed that each cell was isolated to eliminate all possibilities of cross contamination. A primary manifold that was responsive to

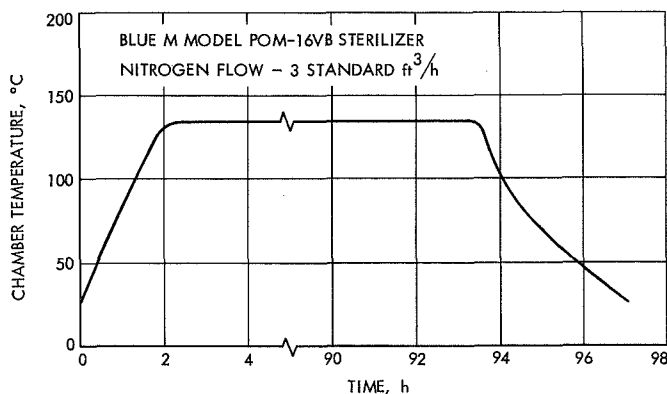


Fig. 4. Typical thermal sterilization cycle

the 30 cell units led to the vacuum system via a 6-in. duct. The vacuum system included a fore-pump, a 6-in. diffusion pump, a water-cooled chevron baffle, and a gate valve. Heating of the cells was achieved by enclosure in two electrically heated ovens in which circulation was maintained by a blower system. The design details of this unit are described in Ref. 7.

The exposure procedure used with this apparatus comprised the following steps:

- (1) One material combination was placed within each test cell at room temperature.
- (2) The test cells were heated to 135 ± 2°C and simultaneously evacuated to a maximum pressure of 10⁻⁶ torr. Stabilization at these thermal-vacuum conditions was achieved approximately 24 h from start. The specimens remained in this environment for 500 h.
- (3) At the end of the exposure period, the test cells were allowed to cool to room temperature before being vented to the atmosphere.

C. General Criteria and Rating

A rating system was necessary in order to establish the degree of compatibility between the paired materials. The rating system employed was similar to that used in previous studies (Ref. 8). In this system, the compatibility of the test material to its contacting secondary material was rated as either compatible (C), marginal (M) or noncompatible (NC), according to the degree of retention of key properties of the test material. The key properties were mechanical and/or electrical and the ratings were established as outlined in the following paragraphs.

1. Mechanical properties

- a. *Compatible.* The product retained 80% or more of its key mechanical properties.
- b. *Marginal.* The product retained 70–80% of its key mechanical properties.
- c. *Noncompatible.* The product retained less than 70% of its key mechanical properties.

2. Electrical properties

- a. *Compatible.* Decrease in product volume resistivity was less than 10⁸ Ω-cm (and threshold value of 10⁷ Ω-cm was also retained) and the product

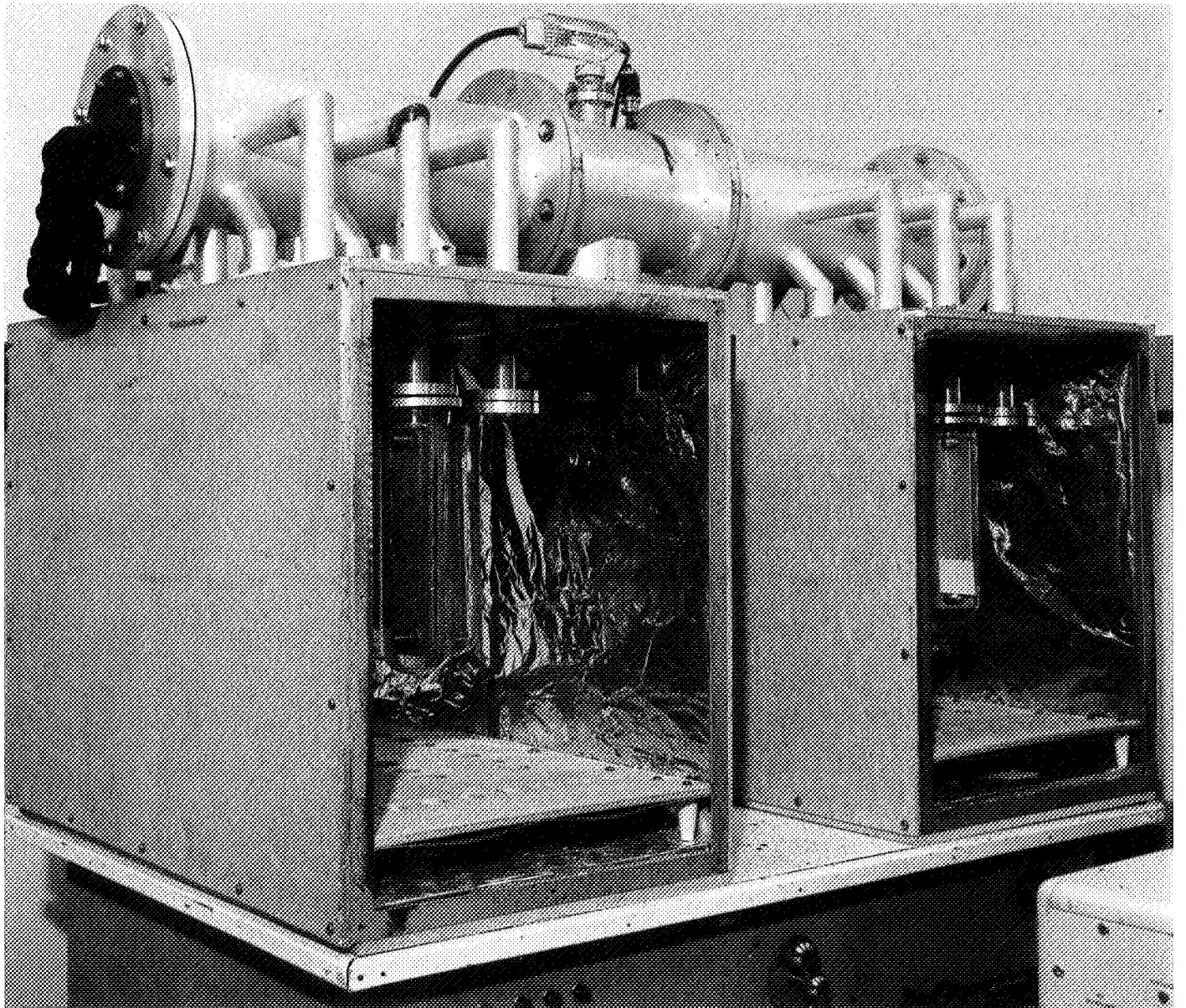


Fig. 5. Thermal-vacuum exposure unit

retained more than 75% of its dielectric strength (and threshold value of 200 V/mil was also retained).

- b. Noncompatible.* The decrease in product volume resistivity was greater than $10^3 \Omega\text{-cm}$ (or the threshold value of $10^7 \Omega\text{-cm}$ was not retained) or the product retained less than 75% of its dielectric strength (or threshold value of 200 V/mil was not retained).

III. Results and Discussion

The data obtained on the adhesive system tested in these studies are shown in Table 3. No incompatible material combinations were found. The adhesive, Epon 828/Z, was not affected by contact with either the polyurethane or the silicone polymeric materials.

The data obtained on the film type materials are given in Table 4. In these data the Mylar combinations are of particular interest. The combinations of Mylar with Solithane and Mylar with DC93-500 were both rated noncompatible (NC) combinations. These ratings were based on the loss in tensile strength and ultimate elongation that occurred in the Mylar as a result of the exposure treatment. The loss in tensile strength was of the order of 35% for each material combination. Ultimate elongation of the Mylar was also significantly reduced in both combinations. The reduction ranged from 28 to 50%.

In the Mylar/Solithane combination it was also observed that the Solithane tended to strongly adhere to the surface of the Mylar after the series of exposures. The surface of the Mylar was contaminated with Solithane particles to such an extent that electrical measure-

ments were impossible. It is also important to note that the Solithane darkened very severely as a result of the exposures. This discoloration of Solithane has been previously reported (Ref. 8).

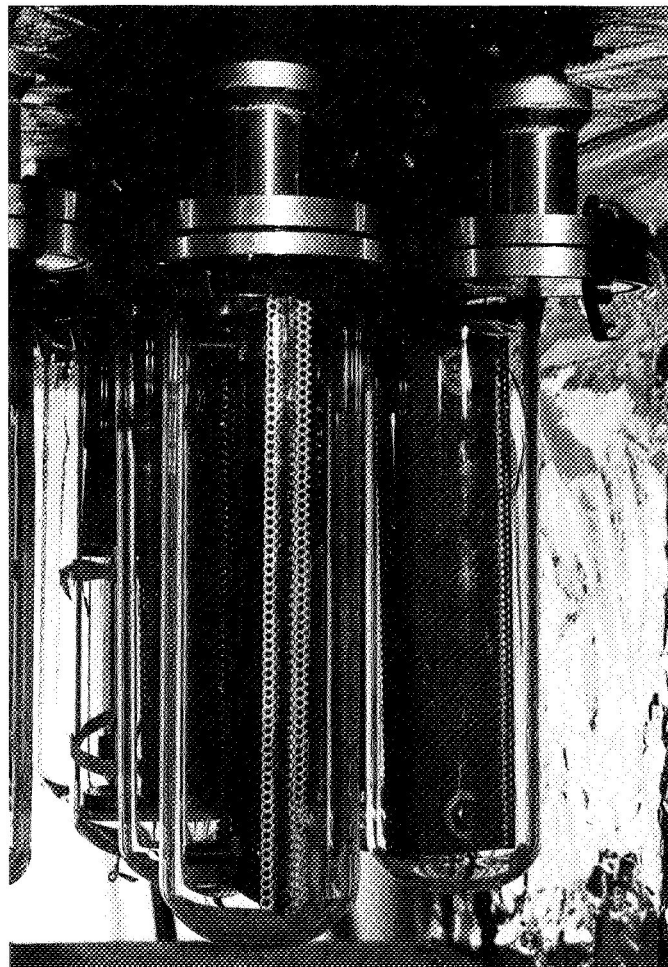


Fig. 6. Thermal-vacuum exposure cells

Table 3. Summary of test results (adhesives)

Test material (cure) ^a	Secondary material (cure) ^a	Test material specimen type	Tests		Compatibility rating
			Adhesive shear strength (D1002), psi	Adhesive creep (D2294), mil	
Epon 828/Z (2/200 + 2/275)	Solithane 113/300 (2/75 + 5/130)	Control	1950	None	C
		Exposed	2060	None	
Epon 828/Z (2/200 + 2/275)	DC93-500 (168/75)	Control	2420	None	C
		Exposed	2250	None	

^aMaterial mix ratios and cures were in accordance with the applicable JPL Material Specification. Cure used as shown (h/°F).

Table 4. Summary of test results (films)

Test material	Secondary material (cure) ^a	Test material specimen type	Tests						Compatibility rating
			Tensile strength (D882), psi	Ultimate elongation (D882), %	Tear strength (D1004), lb/in. thickness	Specimen thickness, mil	Dielectric strength (D149), V/mil	Volume resistivity (D257), Ω -cm	
Mylar 100A	Solihane 113/300 (2/75 + 5/130)	Control	24,400	60	2250	1	4560	3.6×10^{16}	NC
		Exposed	16,000	43	2400	1	— ^b	— ^b	
Mylar 100A	DC93-500 (168/75)	Control	24,400	60	2250	1	4560	3.6×10^{16}	NC
		Exposed	16,200	30	2530	1	5180	1.4×10^{17}	
Kapton 200 XH667	Solihane 113/300 (2/75 + 5/130)	Control	22,600	46	1510	2	3620	3.2×10^{16}	C
		Exposed	24,000	46	1780	2	3740	4.1×10^{16}	
Kapton 200 XH667	DC93-500 (168/75)	Control	22,600	46	1510	2	3620	3.2×10^{16}	C
		Exposed	24,200	45	1430	2	3770	1.3×10^{17}	
Mylar 100A	Micaarta H5834	Control	24,400	60	2250	1	4560	3.6×10^{16}	C
		Exposed	22,000	50	2500	1	5210	8.1×10^{16}	
Kapton 200 XH667	Micaarta H5834	Control	22,600	46	1510	2	3620	3.2×10^{16}	C
		Exposed	23,800	50	1680	2	3860	4.7×10^{16}	

^aMaterial mix ratios and cures were in accordance with the applicable JPL Material Specification. Cure used as shown (h/°F).

^bNot tested.

In contrast with the preceding Mylar combinations, the combination of Mylar with Micarta H5834 was found compatible. In this combination, the loss of tensile strength and elongation was considerably less than with the other combinations.

It was significant to note that previous studies (Ref. 8) have shown Mylar to be only slightly affected when it was given the same series of exposures without a second contacting material (Mylar tensile strength loss was less than 10%). Consequently it appeared that Mylar suffered additional degradation when it was exposed while in contact with a second material.

The type of reaction involved in the Mylar degradation can only be speculated. In this reported work the degradation occurred when the second material was either a polyurethane (Solithane) or a silicone (DC93-500) material. In both cases the resultant degradation was the same: a loss in both tensile strength and elongation occurred. This degradation in mechanical strength has been reported to be indicative of hydrolysis occurring in the Mylar (Ref. 9). When this takes place, the ester linkages in the Mylar polymer are broken; this then results in a loss of mechanical strength of the material.

For hydrolysis to occur, moisture must be present as well as trace amounts of impurities capable of catalytically promoting the hydrolysis. In the series of exposures employed in this work, considerable moisture was made available during the ETO decontamination. At the same time it was quite probable that quantities of impurities (acidic or free radical) were available within

the contacting secondary material as either decomposition products or original product impurities. It was, therefore, suspected that hydrolysis was the mechanism of the Mylar degradation. Definitive tests to confirm this hypothesis were not run since this was beyond the scope of the investigation.

With regard to the test data obtained on the Kapton polyimide film, there was no apparent degradation in properties of the Kapton as a result of the exposures. The Kapton combinations were therefore all rated as compatible.

Table 5 shows the test data that was obtained with structural type materials. All the micarta materials were found compatible with their contacting secondary material. The only combination that was found to be non-compatible was the thermoplastic polyphenylene oxide (PPO534-801). The PPO showed a significant loss in tensile strength (44%) and ultimate elongation (55%), indicating considerable embrittlement. According to previous studies (Ref. 5) PPO was compatible with both the ETO and the thermal sterilization exposures. However, no long term thermal vacuum exposure data was obtained. It was therefore impossible to establish whether the degradation observed in these studies was due to the 500-h thermal-vacuum exposure or the result of the second contacting material. It has been reported (Ref. 10) that PPO is subject to crosslinking and embrittlement when aged above 120°C. It was possible that this was the mode of PPO degradation in these studies due to the extended aging involved in the final thermal vacuum exposure.

Table 5. Summary of test results (structural materials)

Test material	Secondary material (cure) ^a	Test material specimen type	Tests					Compatibility rating
			Tensile strength (D638), psi	Ultimate elongation (D638), %	Specimen thickness, in.	Volume resistivity (D257), Ω-cm	Dielectric strength (D149), V/mil	
PPO534-801	DC93-500 (168/75)	Control exposed	11,100 6,640	20 9	0.30 0.30	7.9×10^{15} 7.4×10^{15}	>200 >200	NC
Micarta H5834	Solithane 113/300 (2/75 + 5/130)	Control exposed	45,200 43,500	9 9	0.13 0.13	1.9×10^{15} 7.4×10^{15}	296 294	C
Micarta H5834	DC93-500 (168/75)	Control exposed	45,200 41,900	9 9	0.13 0.13	1.9×10^{15} 7.2×10^{15}	296 272	C
Micarta 20201-2	Solithane 113/300 (2/75 + 5/130)	Control exposed	26,400 28,900	7 7	0.13 0.13	6.4×10^{15} 1.4×10^{16}	265 252	C

^aMaterial mix ratios and cures were in accordance with the applicable JPL Material Specification. Cure used as shown (h/°F).

In general, the results obtained from this study are not too surprising. In the initial selection of the material combinations, there was some concern held with regard to the stability of polyester materials. It was believed that these materials could be quite susceptible to interactions with other materials due to the rather liable nature of the ester linkages within the polymer. The instability subsequently found in Mylar was therefore not too surprising and did justify the initial concern in polyester materials.

It was significant however that the polyester/phenolic resin combination (Mylar/Micarta H5834) was found compatible. It was initially suspected that residual alkali within the phenolic resin would cause hydrolytic degradation of the polyester (Mylar). This did not occur which indicates that the resin does not contain any detrimental constituents.

It was surprising that the adhesive tested was not affected by the presence of a secondary material. It had been initially felt that bond failure might occur due to migration of constituents of the secondary material into the adhesive at the bond line. It was believed that a silicone secondary material would be particularly active in this type of interaction. However, no degradation occurred with either the silicone or the polyurethane secondary material. It appears that the epoxy adhesives of the type tested in this work (polyamine cure) are resistant to the kind of degradation anticipated.

If one considers the number of combinations investigated and the number found noncompatible, it might be interpreted that there is a high incidence of incompatibility between spacecraft materials; in the present work, this probability amounted to 1 in 6 (17%). However, this interpretation should not be made because the combinations investigated in this work were "worst case" selections; it is not felt that many other incompatible combinations exist.

It is consequently believed that material interactions in spacecraft polymeric materials, although of some con-

cern, should not be considered as a major problem. The designer should nevertheless be aware of its possible occurrence and should be prepared to test any questionable combination. In general, the epoxy, polyimide and phenolic materials appear quite inert to interactions with the silicones and polyurethanes (secondary materials). The reverse cannot be stated as no evaluation of the secondary materials was made. However, the polyester materials do show some material interaction instability. These materials should therefore be used with caution in any spacecraft application where sterilization is required.

IV. Conclusions

Of the twelve material combinations tested, two were found to be incompatible combinations and this directly attributed to some material interaction. Both of these cases involved the combination of the polyester film Mylar with a second material type. The Mylar suffered considerable degradation in mechanical properties when it was given sterilization and thermal-vacuum exposures while in contact with a typical polyurethane and a typical silicone material. The degradation was believed to be attributed to a hydrolysis of the Mylar ester linkages which is a process that may be promoted by the presence of a second contacting material. Since Mylar is used to a considerable extent in spacecraft design, these results are significant. Mylar is used in areas of thermal control (multilayer insulation), electronic components and in particular tape recorder tape substrates. The degradation results observed in these studies should be considered during detailed design, particularly where spacecraft sterilization is required.

Another material, polyphenylene oxide, was found to be grossly degraded under the test conditions of these studies. Degradation of this material by direct interaction with a contacting second material was not established. It may be concluded however that this material deserves closer evaluation prior to its full acceptance as a spacecraft material.

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